No. 22-1827

UNITED STATES COURT OF APPEALS FOR THE FEDERAL CIRCUIT

CELANESE INTERNATIONAL CORPORATION,
CELANESE (MALTA) COMPANY 2 LIMITED, CELANESE SALES U.S. LTD.,

Appellants,

v.

INTERNATIONAL TRADE COMMISSION,

Appellee,

ANHUI JINHE INDUSTRIAL CO., LTD., JINHE USA LLC,

Intervenors.

Appeal from the United States International Trade Commission,
Investigation No. 337-TA-1264.

CELANESE INTERNATIONAL CORPORATION, CELANESE (MALTA) COMPANY 2 LIMITED, AND CELANESE SALES U.S. LTD.'S OPENING BRIEF

AARON G. FOUNTAIN
MORRISON & FOERSTER LLP
701 Brazos Street, Suite 1100
Austin, TX 78701

LENA H. HUGHES
MORRISON & FOERSTER LLP
250 West 55th Street
New York, NY 10019

DEANNE E. MAYNARD
BRIAN R. MATSUI
SETH W. LLOYD
MORRISON & FOERSTER LLP
2100 L Street NW, Suite 900
Washington, DC 20037
Telephone: (202) 887-8740
DMaynard@mofo.com

Counsel for Appellants Celanese International Corporation, Celanese (Malta) Company 2 Limited, Celanese Sales U.S. LTD.

OCTOBER 21, 2022

U.S. Patent No. 10,023,546 Claims 1, 11, 15, and 27

1. A process for producing a finished acesulfame potassium composition, the process comprising the steps of:

(a) contacting a cyclizing agent and a solvent selected from the group consisting of halogenated aliphatic hydrocarbons, esters of carbonic acid with lower aliphatic alcohols, nitroalkanes, alkyl-substituted pyridines, aliphatic sulfones, acetone, acetic acid, and dimethylformamide to form a cyclizing agent composition;

(b) reacting an acetoacetamide salt with the cyclizing agent in the cyclizing agent composition to form a cyclic sulfur trioxide adduct; and

(c) forming from the cyclic sulfur trioxide adduct the finished acesulfame potassium composition comprising non-chlorinated acesulfame potassium and less than 35 wppm 5-chloro-acesulfame potassium;

wherein contact time from the beginning of step (a) to the beginning of step (b) is less than 60 minutes.

11. The process of claim 1, wherein the finished acesulfame potassium composition comprises from 0.001 wppm to 2.7 wppm 5-chloro-acesulfame potassium.

15. The process of claim 1, wherein the reacting is conducted for a cyclization reaction time, from the start of the reactant feed to the end of the reactant feed, less than 35minutes.

27. The process of claim 1, wherein the process yields at least 100 grams of finished acesulfame potassium composition per hour.

CERTIFICATE OF INTEREST

Counsel for Celanese International Corporation, Celanese (Malta) Company 2 Limited, and Celanese Sales U.S. Ltd. certify under Federal Circuit Rule 47.4 that the following information is accurate and complete to the best of their knowledge:

1. **Represented Entities.** Provide the full names of all entities represented by undersigned counsel in this case.

Celanese International Corporation; Celanese (Malta) Company 2 Limited; Celanese Sales U.S. Ltd.

2. **Real Parties in Interest.** Provide the full names of all real parties in interest for the entities. Do not list the real parties if they are the same as the entities.

None.

3. **Parent Corporations and Stockholders.** Provide the full names of all parent corporations for the entities and all publicly held companies that own 10% or more stock in the entities.

Celanese (Malta) Company 2 Limited is a wholly owned, indirect subsidiary of Celanese Corporation, a publicly held company. Celanese Sales U.S. Ltd. and Celanese International Corporation are wholly-owned subsidiaries of Celanese Corporation. No publicly held corporation holds 10% or more of Celanese Corporation's stock.

4. **Legal Representatives.** List all law firms, partners, and associates that (a) appeared for the entities in the originating court or agency or (b) are expected to appear in this court for the entities. Do not include those who have already entered an appearance in this court.

DLA PIPER LLP: Susan Krumplitsch, Helena Kiepura, Erin McLaughlin, Ellen Scordino, Kristin Beale

5. **Related Cases.** Provide the case titles and numbers of any case known to be pending in this court or any other court or agency that will directly affect or be directly affected by this court's decision in the pending appeal. Do not include the originating case number(s) for this case.

Proceedings in *Celanese International Corporation, et al. v. Anhui Jinhe Industrial Co. Ltd. et al.*, No. 2:21-CV-03070-AB-KS (C.D. Cal.) are stayed until the International Trade Commission's determination becomes final. Proceedings in *Celanese International Corporation, et al. v. Anhui Jinhe Industrial Co. Ltd., et al.*, No. 1:20-cv-1775-RGA (D. Del.) are stayed pending resolution of this appeal.

6. **Organizational Victims and Bankruptcy Cases.** Provide any information required under Fed. R. App. P. 26.1(b) (organizational victims in criminal cases) and 26.1(c) (bankruptcy case debtors and trustees).

Not applicable.

Dated: October 21, 2022

/s/ Deanne E. Maynard
Deanne E. Maynard

TABLE OF CONTENTS

TABLE OF AUTHORITIES

Page(s)

Cases

Statutes

Other Authorities

STATEMENT OF RELATED CASES

Celanese International Corporation, Celanese (Malta) Company 2 Limited, and Celanese Sales U.S. Ltd. (collectively "Celanese") initiated this proceeding against Anhui Jinhe Industrial Co., Ltd. and Jinhe USA LLC (collectively "Jinhe") in the International Trade Commission ("ITC"). No appeal from this proceeding has previously been before this Court or any other court.

Celanese also initiated proceedings against Jinhe in *Celanese International Corporation, et al. v. Anhui Jinhe Industrial Co. Ltd. et al.*, No. 2:21-CV-03070-AB-KS (C.D. Cal.), and these proceedings are related in the sense that they are stayed pursuant to 28 U.S.C. § 1659 until the ITC's determination becomes final. And proceedings in *Celanese International Corporation, et al. v. Anhui Jinhe Industrial Co. Ltd., et al.*, No. 1:20-cv-1775-RGA (D. Del.), are related in the sense that they have been stayed pending resolution of this appeal. Counsel for Celanese know of no other cases pending in this Court or any other court that will directly affect or be affected by this Court's decision in this appeal.

JURISDICTIONAL STATEMENT

The administrative law judge made an initial determination on January 11, 2022. Appx5-20. On April 1, 2022, the International Trade Commission issued its final determination not to review the initial determination. Appx1-4. Celanese timely appealed and petitioned for review on May 24, 2022. This Court has jurisdiction under 19 U.S.C. § 1337(c) and 28 U.S.C. § 1295(a)(6).

INTRODUCTION

This case presents a legal question of first impression: whether, under the Leahy-Smith America Invents Act ("AIA"), the sale of a product made by a secret process invalidates a subsequently filed patent on the process by placing the process "on sale." Under the plain language, structure, and purposes of the AIA, the answer is "no." The AIA's on-sale provision unambiguously requires the "claimed invention" itself—not a product made by using the claimed invention—to be "on sale." 35 U.S.C. § 102(a)(1).[1] Congress covered sales of end products of inventive processes elsewhere in the patent laws but chose not to do so in § 102(a)(1). Reading the AIA's on-sale provision to nonetheless cover such sales not only contradicts that choice but also creates anomalies throughout the AIA.

Here, the administrative law judge ("ALJ") disregarded all this because he concluded that the Supreme Court's *Helsinn* decision required him to apply this Court's interpretation of the pre-AIA on-sale bar. *Helsinn Healthcare S.A. v. Teva Pharmaceuticals USA, Inc.*, 139 S. Ct. 628 (2019). Under this Court's pre-AIA interpretation, a patentee's sale of products made by a secret process created a bar to the process's patentability. Because Celanese sold sweeteners that were made

[1] Citations to 35 U.S.C. § ___ refer to the current version of the Code unless otherwise noted. The addendum to this brief includes relevant pre-AIA and AIA statutory provisions.

using Celanese's inventive process, the ALJ determined that *Helsinn* required him to apply the AIA's on-sale provision.

But *Helsinn* requires no such thing. The Supreme Court merely "determine[d] that Congress did not alter the meaning of 'on sale' when it enacted the AIA"—a meaning under which an invention is on sale when it is the subject of a commercial offer for sale and is ready for patenting. *Helsinn*, 139 S. Ct. at 630, 634. And thus *Helsinn* concluded that, under the AIA, a commercial offer to sell a claimed invention that is ready for patenting precludes a patent even if the invention were sold under a contractual obligation to keep its details secret. *Id*. Applied here, *Helsinn*'s holding supports Celanese. Celanese's claimed invention—its process for making high-potency sweeteners—was never the subject of a commercial offer for sale, only sweeteners produced through that process were.

Helsinn nowhere suggests that the AIA enshrined this Court's gloss on the pre-AIA on-sale bar that a patentee's commercial offer to sell something *other than* the claimed invention places that invention on sale. And the AIA's text, structure, purpose, and legislative history all confirm that Congress did not do so. This Court should thus apply the AIA's on-sale provision as intended—that is, as written—and reverse.

STATEMENT OF THE ISSUE

Whether the ALJ legally erred in concluding that Celanese's sales of products made by its secret, inventive process invalidate its patent claims on that process under the on-sale provision of the AIA.

STATEMENT OF THE CASE

A. Legal Background

1. This Court interpreted pre-AIA § 102(b)'s on-sale bar to cover sales by patentees of products made by secret inventive processes

Until recently, the United States patent system granted patents to the first inventor rather than to the first inventor to file for a patent—making the United States patent system almost unique in the world. Under this system, an inventor was entitled to a patent on his or her invention if, among other things, the invention was novel at the time of invention. Specifically, under 35 U.S.C. § 102(a), a person was "entitled to a patent unless . . . the invention was known or used by others in this country, or patented or described in a printed publication in this or a foreign country, before the invention thereof by the applicant for patent." 35 U.S.C. § 102(a) (1952).

To fall within one of these invalidating "prior art" categories in pre-AIA §102(a), this Court consistently required public accessibility. Thus, it "uniformly interpreted the 'known or used' prong of § 102(a) to mean 'knowledge or use which is accessible to the public.'" *BASF Corp. v. SNF Holding Co.*, 955 F.3d 958, 964 (Fed. Cir. 2020). Similarly, it had held that "[i]n order to qualify as a

printed publication within the meaning of § 102, a reference 'must have been sufficiently accessible to the public interested in the art.'" *In re Lister*, 583 F.3d 1307, 1311 (Fed. Cir. 2009). And it had held "that section 102(a) contains a requirement that a foreign patent be disclosed in order to qualify as prior art under section 102(a)." *In re Carlson*, 983 F.2d 1032, 1037 (Fed. Cir. 1992).

Even if an invention were novel at the time of invention and otherwise patentable, an inventor could lose her right to a patent if she delayed in applying for one. Section 102 was thus entitled "Conditions for patentability; novelty and *loss of right to patent*." Pub. L. 82-593, 66 Stat. 792, 797 (1952) (emphasis added). In the patent statute previously in effect, § 102(b) was one of those loss-of-right provisions, creating a one-year filing deadline under certain circumstances. *BASF*, 955 F.3d at 963. Specifically, pre-AIA § 102(b) precluded a person from obtaining a patent if "the invention was patented or described in a printed publication in this or a foreign country or in public use or *on sale* in this country, more than one year prior to the date of the application for patent in the United States." 35 U.S.C. § 102(b) (1952) (emphasis added).

The Supreme Court interpreted the words "on sale" in this pre-AIA on-sale bar in *Pfaff v. Wells Electronics, Inc.*, 525 U.S. 55 (1998). *Pfaff* held that for an inventive product to be "on sale" under this provision, "the product must be the subject of a commercial offer for sale" and "the invention must be ready for

patenting." *Id.* at 67. In so holding, the Supreme Court rejected this Court's interpretation of the pre-AIA on-sale bar—under which an invention could be on sale if it were "substantially complete"—because "such a rule finds no support in the text of the statute." *Id.* at 60, 65-66.

The Supreme Court never addressed whether a sale of a product made by a secret, patented process triggered the bars in pre-AIA § 102(b). But "in a number of cases it indicated by way of dicta that use under 'injunction of secrecy' might not constitute public use." 2A Chisum on Patents § 6.02[5][b] (citing *Electric Storage Battery Co. v. Shimadzu*, 307 U.S. 5, 19-20 (1939); *Egbert v. Lippman*, 104 U.S. 333, 336-37 (1881)).

Nevertheless, the Second Circuit held that the patentee's sale of a product made by a secret process did bar a patent on the process. *Metallizing Eng'g Co. v. Kenyon Bearing & Auto Parts Co.*, 153 F.2d 516 (2d Cir. 1946). In *Metallizing*, the inventor secretly used his inventive process to condition metal parts that he sold for more than a year before seeking a patent. *Id.* at 517. The Second Circuit recognized that the patent was for the process (not the products sold) and that the process had been kept secret. *Id.* at 520. Yet it held the patent invalid, reasoning that "it is a condition upon an inventor's right to a patent that he shall not exploit his discovery competitively after it is ready for patenting" for longer than the grace period, or else he "forfeits his right regardless of how little the public may have learned about the

invention." *Id.* The Second Circuit identified two policies motivating its rule: (1) Congress's desire "that the public shall as soon as possible begin to enjoy the disclosure" of the invention; and (2) preventing an inventor from effectively "extend[ing] the period of his monopoly." *Id.*

This Court adopted this rationale in *D.L. Auld Company v. Chroma Graphics Corporation*, 714 F.2d 1144 (Fed. Cir. 1983). It held that, even if an inventive method had not been disclosed, if the patentee "produced an emblem by the method of the invention and offered that emblem for sale before the critical date, the right to a patent on the method must be declared forfeited." *Id.* at 1147. The Court justified its conclusion on loss-of-right principles. *Id.* at 1147-48. It explained that *Metallizing* set out a "'forfeiture' theory" that "parallels the statutory scheme of 35 U.S.C. § 102(b), the intent of which is to preclude attempts by the inventor or his assignee to profit from commercial use of an invention for more than a year before an application for patent is filed." *Id.* at 1147. This Court reasoned that:

> a party's placing of the product of a method invention on sale more than a year before that party's application filing date must act as a forfeiture of any right to the grant of a valid patent on the method to that party if circumvention of the policy animating § 102(b) is to be avoided in respect of patents on method inventions.

Id. at 1148.

Later, this Court recognized a different rule for third-party sales of products made by a secret patented process—even though the text of § 102(b) drew no

distinction between sales by patentees and those by third parties. *W.L. Gore & Assocs., Inc. v. Garlock*, 721 F.2d 1540 (Fed. Cir. 1983). In *W.L. Gore*, the patentee Gore sued Garlock for infringing claims of Gore's patent covering a process for stretching TEFLON tape. *Id.* at 1544-46. Garlock defended on the ground that the patent was invalid because the invention had been "in public use [and] on sale." *Id.* at 1549 (bracketed material in original). Garlock asserted that a third party—Budd Company—had sold tape made in accordance with the claimed process more than a one year before Gore's application. *Id.* This Court rejected that defense, holding that while a *patentee's* sale of products made by a secret process more than one year before the critical date bars the inventor from patenting the process, a *third-party* sale of such products does not. *Id.*

In concluding that Budd's sales created no patentability bar, this Court applied the plain language of pre-AIA § 102(b). It explained that "[i]f Budd offered and sold anything, it was only tape, not whatever process was used in producing it." *Id.* at 1550. And "there was no evidence" that "the public could learn the claimed process by examining the tape." *Id.* There was thus "no reason or statutory basis" to find that Budd's "secret commercialization of a process, if established, could be held a bar to the grant of a patent to Gore on that process." *Id.* As support for treating third-party sales differently from patentee sales, this Court reasoned that, "[a]s between a prior inventor who benefits from a process by selling its products

but suppresses, conceals, or otherwise keeps the process from the public, and a later inventor who promptly files a patent application from which the public will gain a disclosure of the process, the law favors the latter." *Id.* That principle was codified in pre-AIA 35 U.S.C. § 102(g), which provided that a third party's prior invention prevented a later inventor from obtaining a patent on the invention only where the prior inventor "had not abandoned, suppressed, or concealed it." 35 U.S.C. § 102(g) (1952).

2. In transforming the United States patent system in the AIA, Congress rejected this Court's atextual gloss on the pre-AIA on-sale bar

In 2011, Congress enacted the AIA. Pub. L. 112-29, 125 Stat. 284 (2011). The most significant patent reform since 1952, the AIA "overhauled the patent system" in the United States, converting it from a first-to-invent system to a first-to-file system. *Return Mail, Inc. v. United States Postal Serv.*, 139 S. Ct. 1853, 1860 (2019). Congress designed the AIA "to establish a more efficient and streamlined patent system that will improve patent quality and limit unnecessary and counterproductive litigation costs." H.R. Rep. No. 112-98, pt. 1, at 40 (2011). It also sought to "promote harmonization of the United States patent system with the patent systems commonly used in nearly all other countries throughout the world with whom the United States conducts trade." AIA § 3(p).

Critical to this transformation is a new version of 35 U.S.C. § 102, which implements the first-to-file rule and redefines the novelty requirement for patentability. Under AIA § 102(a)(1), Congress defined what qualifies as "prior art" for purposes of determining a claimed invention's "novelty." Section 102(a) now provides, in part:

> § 102. Conditions for patentability; novelty
>
> (a) Novelty; Prior Art.--A person shall be entitled to a patent unless--
>
> (1) the claimed invention was patented, described in a printed publication, or in public use, on sale, or otherwise available to the public before the effective filing date of the claimed invention . . .

35 U.S.C. § 102(a)(1).

The AIA changed the purpose and structure of § 102. First, the words "loss of rights" have been eliminated from § 102's title, reflecting Congress's repeal of the loss-of-rights components of pre-AIA § 102. AIA § 3(b)(1). Next, "[t]he AIA combines pre-AIA subsections (a) and (b) into a hybrid definition of 'prior art' that is located at new subsection (a)(1)." Joe Matal, *A Guide to the Legislative History of the America Invents Act: Part I of II*, 21 FED. CIR. B.J. 435, 450 (2011). Under new § 102(a)(1), an invention's novelty is measured against the prior art existing at the time the patent application was effectively filed, rather than at the time of invention. *See* 35 U.S.C. § 102(a)(1).

New § 102(a)(1) also contains language different from pre-AIA §§ 102(a) and 102(b). It includes a new term, "claimed invention," which Congress defined to mean "the subject matter defined by a claim in a patent or an application for a patent." 35 U.S.C. § 100(j). It also adds the words "or otherwise available to the public" following the words "on sale," (35 U.S.C. § 102(a)(1)) in order to "clarify the broad scope of relevant prior art, as well as to emphasize the fact that it must be publicly accessible." H.R. Rep. No. 112-98, at 42-43 (2011). And it deletes the geographic restrictions on prior art that existed in pre-AIA §§ 102(a) and (b)—so that covered conduct anywhere in the world can now prevent patentability. *Id.*

In addition to creating a new prior-art provision, Congress also created a new grace period. Previously, § 102(b) built a one-year grace period into the on-sale bar itself, precluding a patent if "the invention was . . . on sale in this country, more than one year prior to the date of the application for patent in the United States." 35 U.S.C. § 102(b) (1952). Now, § 102(b) provides:

> (b) Exceptions.--
>
> (1) Disclosures made 1 year or less before the effective filing date of the claimed invention.--A disclosure made 1 year or less before the effective filing date of a claimed invention shall not be prior art to the claimed invention under subsection (a)(1) if--
>
> (A) the disclosure was made by the inventor or joint inventor or by another who obtained the subject matter disclosed directly or indirectly from the inventor or a joint inventor; or

> (B) the subject matter disclosed had, before such disclosure, been publicly disclosed by the inventor or a joint inventor or another who obtained the subject matter disclosed directly or indirectly from the inventor or a joint inventor. . . .

35 U.S.C. § 102(b). The House committee report explained that the new grace period would "apply to all actions by the patent owner during the year prior to filing that would otherwise create § 102(a) prior art." H.R. Rep. No. 112-98, at 42-43 (2011).

B. ITC Proceedings

1. Celanese sought an exclusion order based on Jinhe's importation of products made by infringing Celanese's patented processes

Celanese filed a complaint at the International Trade Commission alleging a violation of 19 U.S.C. § 1337 based on Jinhe's importation, sale for importation, or sale after importation of certain high-potency sweeteners. Appx63.[2] As relevant here, Celanese asserted that Jinhe's process for making the sweeteners infringed Celanese's U.S. Patent 10,023,546 ("'546 patent"), U.S. Patent 10,208,004 ("'004 patent"), and U.S. Patent 10,590,095 ("'095 patent"). Appx6; Appx86; Appx 88.

[2] Celanese's complaint also named other companies as respondents. Appx7979-7980. Most of them did not participate in the investigation and were held in default, and the investigation was ultimately terminated as to all. Appx1-2.

Each asserted patent claims improvements to a conventional method for making acesulfame potassium ("Ace-K"), an artificial sweetener used in foods, drinks, and medicines. Appx6. Each is subject to the AIA's on-sale provision. Appx7 (noting effective filing date of September 21, 2016); *see* AIA § 3(b), (n). During prosecution, Celanese disclosed to the United States Patent and Trademark Office ("PTO") that the claimed process for making Ace-K had been in secret use in Europe and that Ace-K that was made using that process had been exported and sold in the United States for more than one year before the asserted patents' effective filing dates. Appx7.

Jinhe moved for summary determination of no violation. Appx5; *see* Appx8212-8234. As relevant here, Jinhe contended that by selling products made according to the claimed process more than a year before the patents' effective filing date, Celanese triggered AIA § 102(a)(1)'s on-sale provision and the asserted claims practiced by Celanese were thus invalid. Appx5. Jinhe disclaimed reliance on § 102(a)(1)'s "public use" provision, explaining it "is not at issue here." Appx8225 n.6. And Jinhe presented no evidence that anyone could determine Celanese's process from the products themselves. Appx8212-8234.

2. The ALJ granted summary determination of no violation, holding that the AIA's on-sale provision applies to a patentee's sale of a product made by a secret patented process

The ALJ granted Jinhe's motion for summary determination, holding that Celanese's sales of products made by its secret process invalidated its patent claims on that process under the on-sale provision of AIA § 102(a)(1). Appx5-20.

The ALJ reached that conclusion without addressing the ordinary meaning of AIA § 102(a)(1). Instead, he concluded that Congress had not "overturn[ed] long-held precedent that a patentee's sale of an unpatented product made according to a secret method triggers the on-sale bar to patentability" under pre-AIA § 102(b). Appx10. Although the ALJ recognized that *Helsinn* did "not address[] the exact fact pattern arising [in] this investigation," he reasoned that "Celanese's position is contrary to the Supreme Court's decision in *Helsinn*, where the Court held that Congress did not alter the meaning of the on-sale bar provision when it enacted the AIA." Appx10-12.

Having thus held invalid the asserted claims allegedly practiced by Celanese, the ALJ found no violation of the Tariff Act and terminated the investigation. Appx17-18. The Commission declined Celanese's petition for review (Appx1-4), making the ALJ's decision that of the Commission. *See Microsoft Corp. v. ITC*, 731 F.3d 1354, 1358 (Fed. Cir. 2013).

SUMMARY OF ARGUMENT

A. The plain language and structure of 35 U.S.C. § 102, its surrounding provisions, and the AIA's objectives, all compel a single conclusion: under the AIA's on-sale provision, sales of products made by a secret process do not invalidate patent claims on the process.

Start with the text. Section 102(a)(1) unambiguously requires the "claimed invention"—rather than the product of the use of the claimed invention—to be "on sale." 35 U.S.C. § 102(a)(1). Congress made a deliberate choice not to cover sales of the end result of a patented process. That much is confirmed by Congress's use of different language to cover such sales elsewhere in the patent laws, including in the AIA itself.

Other features of the AIA similarly confirm Congress's choice. The AIA's newly created grace period, prior-user defense, and post-grant review proceedings all presuppose that a sale of a product made by the secret use of a process does not invalidate patent claims on the process.

Celanese's interpretation of § 102(a)(1) also serves the AIA's objectives. Congress enacted the AIA to harmonize United States and foreign patent laws and to reduce the costliness of litigating prior-art issues. Applying § 102(a)(1) as written—to require an offer to sell the claimed invention itself—does just that. By

contrast, the ALJ's interpretation would drive the United States patent system further away from foreign ones and multiply the need for costly fact-intensive discovery.

B. In holding the AIA's on-sale provision triggered here, the ALJ relied on this Court's interpretation of pre-AIA § 102(b), which had treated sales by patentees differently from sales by third parties. But Congress engrafted no such atextual interpretation onto the new § 102(a)(1). Instead, it unambiguously provided that "claimed invention" itself must be "on sale" to trigger the provision.

Helsinn provides no support for the ALJ's decision. There, unlike here, the claimed invention itself was on sale. Nor does the ALJ's determination gain strength from legislative history, which shows that Congress meant to discard the idiosyncratic judicial application of the on-sale bar to secret uses of inventive processes. Finally, to the extent the PTO's guidance does anything more than reiterate *Helsinn*'s holding, that guidance should carry no weight.

ARGUMENT

UNDER A CORRECT INTERPRETATION OF THE AIA, CELANESE'S SALES OF PRODUCTS MADE BY ITS SECRET PROCESS DO NOT INVALIDATE ITS PATENT CLAIMS ON THE PROCESS

The plain text of the AIA's on-sale provision covers only offers to sell claimed inventions, not offers to sell products made using claimed inventions. Although this Court interpreted the pre-AIA on-sale bar to cover such sales of products by the patentee, the AIA contains no such gloss. Every tool of statutory construction confirms that conclusion. The ALJ went astray by assuming that Congress must do more to overrule this Court's precedent than enact statutory language refuting it. He also overread the Supreme Court's *Helsinn* decision, which did not address the question presented here. This Court should apply § 102(a)(1)'s on-sale provision as written and reverse.

Standard Of Review

"Statutory interpretation is a question of law reviewed de novo." *Laerdal Med. Corp. v. ITC*, 910 F.3d 1207, 1211 (Fed. Cir. 2018).

A. The AIA's On-Sale Provision Is Not Triggered By Sales Of Products Made With A Secret Inventive Process

1. The plain language of § 102(a)(1) requires the "claimed invention" itself to be "on sale"

"[I]n any exercise of statutory construction," courts begin "with the text of the provision in question, and move on, as need be, to the structure and purpose of the Act in which it occurs." *New York State Conf. of Blue Cross & Blue Shield Plans v.*

Travelers Ins. Co., 514 U.S. 645, 655 (1995). "[C]ourts must presume that a legislature says in a statute what it means and means in a statute what it says there." *Barnhart v. Sigmon Coal Co., Inc*., 534 U.S. 438, 461-62 (2002). "When the words of a statute are unambiguous, then, this first canon is also the last: 'judicial inquiry is complete.'" *Id.* That simple rule should decide this case.

Under the plain language of § 102(a)(1), the claimed invention itself—not a product made using the claimed invention—must be on sale to trigger the AIA's on-sale provision. The AIA expressly defines "claimed invention" to mean "the subject matter defined by a claim in a patent or an application for a patent." 35 U.S.C. § 100(j); *see Dig. Realty Tr., Inc. v. Somers*, 138 S. Ct. 767, 776 (2018) ("When a statute includes an explicit definition, we must follow that definition . . ."). And as the Supreme Court recently reaffirmed, a claimed invention is "on sale" under § 102(a)(1) when "it was 'the subject of a commercial offer for sale' and 'ready for patenting.'" *Helsinn*, 139 S. Ct. at 630. Put together, § 102(a)(1)'s on-sale provision thus unambiguously instructs that:

> [a] person shall be entitled to a patent unless [the subject matter defined by a claim in a patent or an application for a patent] was . . . [the subject of a commercial offer for sale and ready for patenting] . . . before the effective filing date of the claimed invention.

35 U.S.C. § 102(a)(1) (bracketed material added). Nothing in this subsection covers commercial offers to sell the *product* of an invention's use. And "[i]t is a

fundamental principle of statutory interpretation that absent provisions cannot be supplied by the courts." *Rotkiske v. Klemm*, 140 S. Ct. 355, 360-61 (2019) (internal quotations and brackets omitted).

"Atextual judicial supplementation is particularly inappropriate when . . . Congress has shown that it knows how to adopt the omitted language or provision." *Id.* at 361. Here, 35 U.S.C. § 273 shows that the Congress that enacted the AIA knew how to refer to sales of the *product* of a claimed invention when it wanted to do so. In that section, the AIA created a prior-user infringement defense for use of "subject matter consisting of a process" that "would otherwise infringe a claimed invention." 35 U.S.C. § 273(a). The defense applies when a person "acting in good faith, commercially used the subject matter in the United States" "in connection with . . . *an actual arm's length sale . . . of a useful end result of such commercial use*" at least a year before the effective filing date of the claimed invention or a qualifying public disclosure. *Id*. (emphasis added).

That language expressly covering sales of the "useful end result" of claimed processes starkly contrasts with § 102(a)(1)'s language covering only offers to sell the "claimed invention" itself. "When Congress includes particular language in one section of a statute but omits it in another, [the Supreme] Court presumes that Congress intended a difference in meaning." *Digital Realty*, 138 S. Ct. at 777 (internal quotations, brackets, and alterations omitted). If Congress wanted sales of

the useful end result of inventive processes to trigger § 102(a)(1)'s on-sale provision, it would have said so.

Other provisions of the patent laws reinforce that § 102(a)(1)'s on-sale provision means what it says and that Congress uses different language to refer to sales of the products of inventions. Under 35 U.S.C. § 271(a), for instance, "whoever without authority makes, uses, *offers to sell*, *or sells any patented invention*, within the United States or imports into the United States any patented invention during the term of the patent therefor, infringes the patent." 35 U.S.C. § 271(a) (emphasis added).[3] Courts have long interpreted that language to mean that "[a] sale of a product made by a patented process does not itself infringe the patent." *United States v. Studiengesellschaft Kohle, m.b.H,* 670 F.2d 1122, 1127-28 (D.C. Cir. 1981). Thus, use of a patented process abroad to manufacture a product later imported to or sold within the United States is not a § 271(a) infringing use of the patent on the process. *Zoltek Corp. v. United States*, 672 F.3d 1309, 1322 (Fed. Cir. 2012) (en banc) ("Title 35 U.S.C. section 271(a) does not protect against the importation of products made by a patented process.").

[3] Although § 271(a) has been amended over the years, it has referred to one who "sells any patented invention" since its enactment in 1952. 35 U.S.C. § 271(a) (1952) ("Except as otherwise provided in this title, whoever without authority makes, uses or sells any patented invention, within the United States during the term of the patent therefor, infringes the patent.").

Instead, in 1988, Congress enacted a different provision, 35 U.S.C. § 271(g), to cover infringement of a claimed process based on domestic sales of useful end results. That subsection provides that "[w]hoever without authority imports into the United States or offers to sell, sells, or uses within the United States *a product which is made by a process patented* in the United States shall be liable as an infringer." 35 U.S.C. § 271(g) (emphasis added). It is only because of that subsection's specific reference to "a product . . . made by a process" that "a patentee holding a process patent" may sue for infringement those who have "used the patented process abroad to manufacture products, and then imported, used, or sold the products in this country." *Eli Lilly & Co. v. Am. Cyanamid Co.*, 82 F.3d 1568, 1571 (Fed. Cir. 1996). Such statutory text shows Congress has long understood how to refer to sales of the products of claimed processes. Yet it did not do so in the AIA's on-sale provision.

Congress's use of the words "or otherwise available to the public" in § 102(a)(1) further confirms that the AIA's on-sale provision excludes sales of a product that do not disclose the inventive process. 35 U.S.C. § 102(a)(1). The phrase "available to the public" must mean that the invention is available to at least one other person. *Cf. UCB, Inc. v. Watson Lab'ys., Inc.*, 927 F.3d 1272, 1289 (Fed. Cir. 2019) ("For prior art to anticipate because it has been 'used,' the use must be accessible to the public," but "[p]rior knowledge and use by a single person is

sufficient." (internal quotations omitted)). And Congress's use of the term "otherwise" indicates that placing a claimed invention "on sale" is another way of making the claimed invention so available. *See United States v. Standard Brewery*, 251 U.S. 210, 218 (1920) (holding that interpreting "beer, wine, or other intoxicating malt or vinous liquors for beverage purposes" to "include beer and wine whether intoxicating or not" would render the words "other intoxicating" "quite superfluous").[4]

Finally, the ordinary meaning of § 102(a) is reinforced by Congress's amendments to the section's title. "[T]he title of a statute and the heading of a section are tools available for the resolution of a doubt about the meaning of a statute." *Almendarez-Torres v. United States*, 523 U.S. 224, 234 (1998) (internal quotations omitted). Here, if any doubt remains, § 102(a)'s title clears it up. When Congress enacted § 102 in 1952, it entitled it "Conditions for patentability; novelty and loss of right to patent." Pub. L. 82-593, 66 Stat. 792, 797 (1952). That title made sense because the provision encompassed both novelty provisions and loss-of-right provisions. As this Court has explained, "[p]re-AIA 35 U.S.C. § 102 sets forth

[4] Although the Supreme Court in *Helsinn* concluded that the AIA's addition of "or otherwise available to the public" was too "oblique" a way for Congress to require that a sale of a claimed invention must make the details of the invention available to the public, the claimed invention there had itself been sold to another and thus fell within the express terms of the statute. *Helsinn*, 139 S. Ct. at 634; *see infra* pp. 42-45.

several conditions of patentability, including novelty (subsections (a), (e), (f), and (g)) and loss-of-right provisions which may, notwithstanding the novelty of an invention, bar a patent (subsections (b), (c), and (d)).” *BASF*, 955 F.3d at 963. In particular, this Court described its gloss on the on-sale bar for patentee’s sales of products made with inventive processes as a loss-of-right rule. *D.L. Auld*, 714 F.2d at 1147 (such sales “operate to create a *forfeiture* of any right to the grant of a valid patent on the method” (emphasis added)).

But in enacting new § 102, Congress struck out the “loss of right” language from the title, instead entitling the section “Conditions of patentability; novelty.” *Compare* Pub. L. 112-29, 125 Stat. 284, § 3(b)(1) (2011) *with* Pub. L. 82-593, 66 Stat. 792, 797 (1952). This change confirms that Congress discarded the judicial gloss on the pre-AIA version, under which a patentee lost his right to a patent by making a secret commercial use of his inventive process. It also confirms that Congress retained the “novelty” component of pre-AIA § 102, under which prior art had to be publicly accessible to defeat novelty. *Supra* pp. 3-4.

2. The structure of the AIA shows that § 102(a)(1) excludes sales of products made with secret inventive processes

The structure of the AIA confirms the ordinary meaning of § 102(a)(1)’s on-sale provision.

a. Other AIA provisions describing § 102(a)(1)'s prior art categories as "disclosures"

Elsewhere, the AIA refers to § 102(a)'s prior-art categories as "disclosures" or has having been "disclosed." *E.g.*, 35 U.S.C. §§ 102(b), 103. The ordinary meaning of "to disclose" is "[t]o expose to view" or "[t]o make known (something heretofore kept secret)." *Disclose*, THE AMERICAN HERITAGE DICTIONARY (5th ed. 2011). Congress's reference to § 102(a) prior art as "disclosures" confirms that "§ 102's new definition of prior art consists of subject matter that has been disclosed." Robert A. Armitage, *Understanding the America Invents Act and Its Implications for Patenting*, 40 AIPLA Q.J. 1, 67 (2012) (emphasis omitted). But a sale of a product of a secret inventive process involves no disclosure of the claimed invention. The AIA's cross-references to § 102(a)'s prior art as "disclosures" thus reinforces that Congress discarded this pre-AIA gloss.

A contrary reading of § 102(a)(1)'s on-sale provision would create a mismatch between § 102(a)(1) and its grace period. Under AIA § 102(a)(1), the fact that "the claimed invention was patented, described in a printed publication, or in public use, on sale, or otherwise available to the public before the effective filing date of the claimed invention" prevents patentability. But § 102(b)(1)(A) then creates a grace period for an inventor to engage in conduct that would otherwise give rise to prior art under § 102(a)(1). Specifically, under § 102(b)(1)(A), a "disclosure" "made by the inventor" "1 year or less before the effective filing date of a claimed

invention" "shall not be prior art to the claimed invention under subsection (a)(1)." 35 U.S.C. § 102(b)(1)(A). Congress viewed this grace period as essential "to give U.S. applicants the time they need to prepare and file their applications." H.R. Rep. 112-98 at 42.

Under a plain reading of § 102(a)(1), that subsection and its grace period operate harmoniously. Under the plain language of § 102(a)(1), to qualify as prior art, the claimed invention must have been disclosed to someone—whether by a public use, an offer for sale, or otherwise. And § 102(b)(1)(A) then creates an exception for all such disclosures by the inventor that occur within a year of the patent application's effective filing date. In other words, the inventor gets a full year to use her invention in any of the ways that would otherwise create prior art under § 102(a)(1). That is precisely what Congress intended. *See* H.R. Rep. No. 112-98, at 42-43 (2011) (explaining that AIA's grace period "will apply to all actions by the patent owner during the year prior to filing that would otherwise create § 102(a) prior art").

But under the ALJ's reading, § 102(a)(1) and its grace period are mismatched. Under the ALJ's reading, a claimed invention can be "on sale" under § 102(a)(1) even when it has never been offered for sale—and thus never been disclosed—to anyone. *E.g., Metallizing*, 153 F.2d at 520 (holding patentee's sale of product of inventive process forfeits patent right "regardless of how little the public may have

learned about the invention"); *D.L. Auld*, 714 F.2d at 1147 (holding same "[w]here a method is kept secret, and remains secret after a sale of the product of the method"). Subsection 102(b)(1)(A) would then create a grace period for only a sub-category of an inventor's actions under § 102(a)(1) taken within a year of the application—namely, the inventor's actions that resulted in a "disclosure." No grace period would exist for § 102(a)(1) conduct by the inventor that involves no disclosure—that is, the inventor's secret commercialization of its claimed invention. Put another way, an inventor's sale of a product made by the secret use of the inventive process would preclude a patent on the process even if the sale took place just a day before the inventor filed her patent application. That inventor would not be given the time Congress believed she needed to prepare and file her application.

There is no logical reason Congress would have desired such a mismatched grace period. As Senator Jon Kyl, one of the AIA's sponsors, put it:

> Why would Congress create a grace period that allows an invention that has been disclosed to the world in a printed publication, or sold and used around the world, for up to a year, to be withdrawn from the public domain and patented, but not allow an inventor to patent an invention that, by definition, has not been made available to the public?

157 Cong. Rec. S5431 (daily ed. Sept. 8, 2011). "Such an interpretation of section 102 simply makes no sense, and should be rejected for that reason alone." *Id.*

b. The AIA's prior-user defense

The ALJ's reading has another structural flaw: if taken to its logical conclusion, it could render § 273's prior-user defense for sales of useful end results of commercial uses of inventive processes superfluous. As discussed, under that provision,

> [a] person shall be entitled to a defense under section 282(b) with respect to subject matter consisting of a process . . . that would otherwise infringe a claimed invention being asserted against the person if—(1) such person, acting in good faith, commercially used the subject matter in the United States, either in connection with an internal commercial use or an actual arm's length sale or other arm's length commercial transfer of a useful end result of such commercial use [within a year of the effective filing date of the claimed invention].

35 U.S.C. § 273(a)(1). But if the ALJ were correct that a sale of the end result of a claimed process *invalidates* the patent on the process under § 102(a)(1), it appears that no one would need this defense to *infringement* for such sales. By its plain terms, § 102(a)(1)'s on-sale provision applies equally to patentees' and third parties' sales. So if a patentee's sale of the product of an inventive process places that invention "on sale" within the meaning of § 102(a)(1), then a third-party's sale of such a product should too. Yet if third-party sales of the product of the claimed process place the invention "on sale" under § 102(a)(1)—thereby invalidating a patent on the process—third parties that made such sales would never need § 273's infringement defense. It is "one of the most basic interpretive canons" that "a statute

should be construed so that effect is given to all its provisions, so that no part will be inoperative or superfluous, void or insignificant." *Corley v. United States*, 556 U.S. 303, 314 (2009) (internal quotations and brackets omitted). Celanese's interpretation honors that canon; the ALJ's does not.

c. The AIA's post-grant review proceedings

Congress's exclusion of sales of products made by secret claimed processes from § 102's prior art also coheres with the post-grant review proceedings Congress created in the AIA. Congress designed those administrative proceedings to provide a "more efficient system for challenging patents that should not have issued." H.R. Rep. No. 112-98, *supra*, Pt. I, at 39-40. To meet that goal, Congress limited post-grant proceedings to patents that would be governed by the new § 102, including its new definition of prior art. *See* AIA §§ 6(f)(2)(A), 3(n)(1). The Senate Republican Policy Committee's summary of the amendment adding this limitation explained that pre-AIA, first-to-invent "patents raise discovery-intensive invention-date and secret-prior-art issues that would be difficult to address in an administrative proceeding." 157 Cong. Rec. S1366 (daily ed. Mar. 8, 2011).

The administrative review proceedings created for business-method patents similarly reflect Congress's concern that pre-AIA § 102(b) would be too unwieldly for post-grant proceedings. Like other patents, business-method patents can be challenged through the AIA's post-grant review proceedings. *See* AIA § 18(a)(1).

Unlike other patents, however, business-method patents can be challenged in AIA post-grant review proceedings even if they issued *before* the AIA. *See* AIA § 18(a)(2). But for those patents, the AIA limits a challenger's ability to invoke pre-AIA § 102, providing that only prior art under pre-AIA § 102(a) or certain conduct by the inventor that "discloses the invention" can qualify as relevant prior art. AIA § 18(a)(1)(C). Here too, the Senate Republican Policy Committee's summary explained that "prior art is limited to old 102(a), which must be publicly available, or prior art of old 102(a) scope that shall be presumed to beat old 102(a) invention-date limits but that falls outside the old 102(b) grace period (i.e., *effectively, old 102(b) prior art but limited to old 102(a)'s publicly-available prior art scope*.)." 157 Cong. Rec. S1367 (daily ed. Mar. 8, 2011) (emphasis added). In other words, for pre-AIA patents subject to post-grant review, Congress limited prior art to that which has been disclosed. In contrast, for post-AIA patents, it simply limited prior art to AIA § 102(a)—knowing that it had already built in a disclosure requirement to that provision itself.

Under the ALJ's reading, however, secret commercial uses of an inventive process anywhere in the world could render the process unpatentable. Under that view, post-grant proceedings would have to grapple with and resolve "discovery-intensive . . . secret-prior-art issues," such as clandestine uses. 157 Cong. Rec.

S1366 (daily ed. Mar. 8, 2011). That result would be contrary to Congress's intent of establishing an efficient path for resolution of patentability challenges.

3. The ordinary meaning of § 102(a)(1)'s requirement that the "claimed invention" be "on sale" serves Congress's objectives in enacting the AIA

The on-sale provision's ordinary meaning also serves Congress's objectives in enacting the AIA. Congress undertook the monumental task of transforming a first-to-invent system to a first-to-file system to "promote harmonization of the United States patent system with the patent systems commonly used in nearly all other countries throughout the world with whom the United States conducts trade." AIA § 3(p). Congress hoped to "promote greater international uniformity and certainty in the procedures used for securing the exclusive rights of inventors to their discoveries." AIA § 3(p). Congress also intended to "simplif[y] how prior art is determined, provide[] more certainty, and reduce[] the cost associated with filing and litigating patents." H.R. Rep. 112-98, pt. 1, at 42 (2011).

Applying the on-sale provision as written serves these goals. First, it aligns the United States patent system with others around the world, as no other country treats sale of a product made by secret use of an inventive process as a bar to patentability of the process. Dmitry Karshtedt, *Did Learned Hand Get It Wrong?: The Questionable Patent Forfeiture Rule Of Metallizing Engineering*, 57 VILL. L. REV. 261, 263-64, 316 (2012). In doing so, it eliminates an uncertainty about the

requirements for United States patent validity that foreign patent law experts had previously criticized. *See* Toshiko Takenaka, *Rethinking the United States First-to-Invent Principle from A Comparative Law Perspective: A Proposal to Restructure S 102 Novelty and Priority Provisions*, 39 Hous. L. Rev. 621, 636 (2002) (arguing that "inclusion of secret commercial use within the meaning of 'public use or on sale' introduces a significant uncertainty in U.S. patent validity" not present in foreign patent systems). And second, it reduces fact-intensive discovery because it eliminates the need to investigate secret uses of the claimed invention. *See* 157 Cong. Rec. S5319-21 (daily ed. Sept. 6, 2011) (Statement of Sen. Jon Kyl) ("The main benefit of the AIA public availability standard of prior art is that it is relatively inexpensive to establish the existence of events that make an invention available to the public . . . this will greatly reduce the time and cost of patent litigation and allow the courts and the PTO to operate much more efficiently.").

The ALJ's reading of § 102(a)(1) not only fails to serve Congress's goals, it could affirmatively frustrate them. That is because if the ALJ were correct, that could mean that Congress did not just reenact the judicial gloss on pre-AIA § 102(b), but instead supercharged it. Under the judicial gloss on the pre-AIA on-sale bar, a sale of a product made by an inventive process barred a patent on the process only if the sale occurred (1) in the United States (2) by patentees (not third parties) (3) more than a year before the application. But in the AIA, Congress eliminated

geographic restrictions from § 102(a)(1) so that a sale anywhere in the world can trigger the on-sale provision. Congress also eliminated all provisions of the pre-AIA law that might have provided a statutory basis for excluding third-party sales from new § 102(a)(1). *See infra* pp. 40-42. And Congress limited § 102(b)(1)'s grace period to prior art that involves "disclosures," thereby eliminating any grace period for conduct—like the secret commercial use of an inventive process—that results in no disclosure to anyone.

So, if the ALJ's reading were taken to its logical conclusion, that could mean that product sales (1) anywhere in the world (2) by anyone (3) even a day before the effective filing date of the patent application would invalidate a patent claim on an inventive process so long as the challenger could show that the invention was secretly used to make the product. That would drive the United States patent system even further apart from those of its sister states and multiply the issues subject to fact-intensive discovery. Congress could not have intended to enact such a counterproductive rule.

Meanwhile, the purposes that motivated the judicial gloss on pre-AIA § 102(b) provide no grounds to conclude Congress intended to adopt that gloss in AIA § 102(a)(1). Courts invoked the policy objectives of encouraging early disclosures and preventing inventors from effectively extending their patent monopolies. *Supra* p. 6. Those policy judgments provided a questionable basis for

judicially expanding pre-AIA § 102(b) even then. *See Burrage v. United States*, 571 U.S. 204, 218 (2014) ("The role of this Court is to apply the statute as it is written—even if we think some other approach might 'accor[d] with good policy."). But regardless, Congress addressed those policy concerns elsewhere in the AIA, eliminating any reason to think that Congress must have intended to embrace those atextual decisions when enacting § 102(a)(1).

The desire to motivate early disclosure is addressed by the AIA's move away from a first-to-invent system. "[A] first-to-file system encourages the prompt filing of patent applications." S. Rep. 110-259, at 7 (2008). If an inventor of a secret process delays in filing a patent application, he runs the risk that another, later inventor will gain the patent monopoly. "There is no need to also require forfeiture of patents simply because the inventor has made some use of the invention that has not made the invention available to the public." 157 Cong. Rec. S1371 (2011) (Statement of Sen. Jon Kyl) (cited by H.R. Rep. No. 112-98, pt. 1, at 43 n.20 (2011)).

The concern with allowing inventors to effectively extend their patent monopolies is similarly misplaced as applied to the AIA. "Ordinary sales in competition do not yield monopoly profits, and the longer an inventor sells his product without seeking a patent, the more competition there is likely to be." *In re Mahurkar Double Lumen Hemodialysis Catheter Patent Litig.*, 831 F. Supp. 1354, 1369 (N.D. Ill. 1993) (Easterbrook, C.J.). So the only risk of functionally extending

a patent monopoly comes from the possibility that "[f]ear of losing their investment may discourage rivals" from competing "and enable the inventor to collect supra-competitive profits before obtaining a patent." *Id.* But the AIA accounts for that risk too: by enabling a rival to secure the patent monopoly by filing first (35 U.S.C. § 102(a)), or if not, to invoke a prior-user defense to protect certain investments (35 U.S.C. § 273).[5]

In short, every tool of statutory interpretation confirms that the AIA's on-sale provision means what it says: it is triggered only by sales of a claimed invention, not by sales of products made by a secret inventive process.

B. The ALJ's Reasons Cannot Sustain His Atextual Reading

The ALJ never considered the ordinary meaning of § 102(a)(1)'s text, let alone the AIA's structure and purpose. Instead, he concluded that Congress had not overturned this Court's precedent interpreting pre-AIA § 102(b) as covering sales of a product made by a secret inventive process and that the Supreme Court's *Helsinn* decision had said as much. He also pointed to legislative history and PTO guidance.

[5] A third purpose underlying pre-AIA § 102(b)—a policy against allowing an inventor to remove existing knowledge from public use—has never been served by the judicial gloss on pre-AIA law because "the secret inventions it operates against are never in the public domain, and are not even disclosed to third parties such as prospective buyers." Karshtedt, 57 VILL. L. REV. at 326.

But none of the ALJ's reasons for disregarding § 102(a)(1)'s plain and ordinary meaning withstand scrutiny.

1. The reenactment canon does not apply

The ALJ ignored the text of § 102(a)(1) because he believed Congress had not "overturn[ed] long-held precedent that a patentee's sale of an unpatented product made according to a secret method triggers the on-sale bar to patentability." Appx10. But that gets the inquiry backwards: Congress enacted a new statute, and it is that statute's language that governs. Although Congress's reenactment of preexisting statutory language can sometimes imply that it carried forward prior judicial constructions of that language, that canon only applies when certain well-established conditions are met. None of them is satisfied here.

First, "the reenactment canon does not override clear statutory language." *Oklahoma v. Castro-Huerta*, 142 S. Ct. 2486, 2498 (2022); *BP P.L.C. v. Mayor of Baltimore*, 141 S. Ct. 1532, 1541 (2021) (holding reenactment canon inapplicable to circuit courts' interpretation of statutory language where "the text and structure of the statute are to the contrary" (citation omitted)). Here, the statute clearly states that a person is entitled to a patent unless "the claimed invention was . . . on sale," not unless the product of the claimed invention was on sale. 35 U.S.C. § 102(a)(1). The AIA's structure also contradicts the prior interpretation—the grace period, prior-user defense, and post-grant review proceedings that Congress created at the

same time all presuppose that sales of unpatented products made by undisclosed processes do not trigger § 102(a)(1). *Supra* pp. 22-29.

Second, the reenactment canon is inapplicable here because the judicial gloss on pre-AIA § 102(b) was not a "settled construction." *Jama v. Immigration and Customs Enforcement*, 543 U.S. 335, 349 (2005). Even though courts of appeals had held that sales of a product made by a secret inventive process place the process on sale, the Supreme Court had never so held and, its decisions had instead "indicated by way of dicta that use under 'injunction of secrecy' might not constitute public use." 2A Chisum on Patents § 6.02[5][b].

Although the Second Circuit cited the Supreme Court's decision in *Pennock v. Dialogue*, that decision provides no support for *Metallizing*'s judicial gloss on the pre-AIA on-sale bar. *Pennock* interpreted the patent act then in effect, which authorized a patent only if the invention was "not known or used before the application." *Pennock v. Dialogue*, 27 U.S. 1, 17 (1829) (emphasis omitted). There, the inventors' licensee had made and sold the patented invention—a leather hose—publicly for seven years before the patent application. *Id.* at 9. The inventors' argument was *not* that their invention had not been used or sold. Rather, they argued that, because the use and sales had been made with their permission, they had not forfeited their patent right. *Id.* at 5 (counsel arguing "[t]hat the use of an invention, however public, if it be by the permission and under the continual exclusive claim

of the inventor; does not take away his right, except after an unreasonable lapse of time, or gross negligence, in applying for a patent"). The Supreme Court disagreed: "If such a public use is not a use within the meaning of the statute, what other use is?" *Id.* at 21. The Court thus interpreted the statute to mean that "the first inventor cannot acquire a good title to a patent; if he suffers *the thing invented* to go into public use, or to be publicly sold for use, before he makes application for a patent." *Id.* at 23-24 (emphasis added).

Pennock thus offers no support for interpreting the pre-AIA on-sale bar to invalidate patent claims to a secret inventive process, for several reasons. First, contrary to the Second Circuit's assumption, the invention at issue was not "for a process of making hose." *Metallizing*, 153 F.2d at 518. Rather, both the Supreme Court and circuit court decisions in *Pennock* indicate that the patented invention was the leather hose itself. *E.g., Pennock*, 27 U.S. at 15 (quoting jury instruction stating that "it makes no difference in the principle, that *the article so publicly used, and afterwards patented*, was made by a particular individual, who did so by the private permission of the inventor" (emphasis added)); *Pennock v. Dialogue*, 19 F. Cas. 171, 173 (C.C.E.D. Pa. 1825) (No. 10,941) ("The hose invented by Mr. Bedford more nearly resembles that which the plaintiffs have patented."). Second, even if "the thing invented" were the process, it had not been kept secret. Rather, it been "used

by others" as the Supreme Court construed the statutory bar to require, because the inventors had licensed it to Samuel Jenkins. *Pennock*, 27 U.S. at 3-4, 19.

Nor does the Supreme Court's decision in *Pfaff* suggest that the Supreme Court has endorsed the circuit-level gloss of pre-AIA § 102(b). *Contra Helsinn Healthcare S.A. v. Teva Pharams. USA, Inc.*, Nos. 16-1284, 16-1787, 2018 WL 1583031, at *2 n.1 (Jan. 16, 2018) (O'Malley, J., concurring in denial of panel rehearing). *Pfaff* zeroed in on the text of the patent act—rejecting atextual interpretations along the way. *Supra* pp. 4-5. *Pfaff* found that "the two essential conditions of the on-sale bar" had been met because the inventor had made commercial offers to sell "sockets contain[ing] all the elements of the invention claimed" at a time when his "invention was ready for patenting." 525 U.S. at 68-69. Although *Pfaff* quoted language from *Metallizing*, the Supreme Court was merely making the point that the judgment the Supreme Court was affirming found "support not only in the text of the statute but also in the basic policies underlying the statutory scheme, including § 102(b)." *Id.* at 68. Nowhere did the Supreme Court suggest that the "essential conditions of the on-sale bar" would be satisfied if the inventor had sold something containing *no* elements of the claimed invention at all.

Appellate decisions adopting the pre-AIA gloss on § 102(b) did not settle the law either. The Supreme Court has found it "most unlikely . . . that a smattering of lower court opinions could ever represent the sort of 'judicial consensus so broad

and unquestioned that we must presume Congress knew of and endorsed it.'" *BP*, 141 S. Ct. at 1541. The intermediate court decisions concluding that sales of a product made by an inventive process place the process "on sale" are just that. After *Metallizing*, the courts to adopt its reasoning did so with little to no legal analysis of their own. Karshtedt, 57 VILL. L. REV. at 289.

The pre-AIA gloss in question also cannot be a "settled construction" of pre-AIA § 102(b)'s "on sale" language because it contradicts other interpretations this Court gave the same language. A sale of a product made by a secret claimed invention was a sale when made by the patentee, but not a sale when made by a third party. *Compare Quest Integrity USA, LLC v. Cokebusters USA Inc.*, 924 F.3d 1220, 1227 (Fed. Cir. 2019) ("Sale of a product . . . produced by performing a claimed process implicates the on-sale bar.") (patentee), *with W.L. Gore*, 721 F.2d at 1550 ("If Budd offered and sold anything, it was only tape, not whatever process was used in producing it.") (third party). But the Supreme Court has held that a single phrase in a single statutory provision cannot have different meanings in different situations. *Clark v. Martinez*, 543 U.S. 371, 378 (2005) ("To give these same words a different meaning for each category would be to invent a statute rather than interpret one."). Since this Court never settled on a single conclusion about whether the sale of a product made with a secret inventive process places the invention "on sale," there was no "well-settled" interpretation for Congress to adopt.

Third, the reenactment canon is inapplicable because the AIA did not "reenact" pre-AIA § 102(b) "without change." *Jama*, 543 U.S. at 349. Instead, Congress combined pre-AIA § 102(a) and pre-AIA § 102(b) to create a single novelty provision, rather than a loss-of-right provision. *Supra* pp. 9-10. That is significant because the reasoning for the pre-AIA gloss hinged on loss-of-right principles. As this Court explained in *D.L. Auld*, the "concept explicated in *Metallizing*" is that

> a party's placing of the product of a method invention on sale more than a year before that party's application filing date must act as a forfeiture of any right to the grant of a valid patent on the method to that party if circumvention of the policy animating § 102(b) is to be avoided in respect of patents on method inventions.

D.L. Auld, 714 F.2d at 1148. In opting to create a unified novelty provision (rather than a loss-of-right provision), Congress directly undermined the foundation for the pre-AIA atextual gloss. The new provision advances no loss-of-right policy, so there is no circumvention of that policy to prevent.

Congress also repudiated the judicial gloss on pre-AIA § 102(b) in the text of § 102(a)(1) itself. Congress introduced a new term, "claimed invention," as the subject of the on-sale provision, and expressly defined it to mean "the subject matter defined by a claim in a patent or an application for a patent." 35 U.S.C. § 100(j). In doing so, it refuted the premise of this Court's pre-AIA cases holding the on-sale bar can be triggered when the thing sold is not an embodiment of the claimed invention

itself. Congress also added the catch-all, "or otherwise available to the public," to emphasize the fact that prior art under § 102(a)(1) "must be publicly accessible." H.R. Rep. No. 112-98, at 42-43 (2011). This change likewise rejects the pre-AIA gloss on § 102(b) because sales of a product made by a secret inventive process disclose the claimed invention to no one at all.

Several other changes in the AIA similarly confirm that Congress intended its new patent system to operate without the judicial gloss on the pre-AIA on-sale bar. In the AIA, Congress created a new grace period, prior-user defense, and post-grant review proceedings. As described above, each of these additions works harmoniously with § 102(a)(1)'s language as written, but chafes against a reading of § 102(a)(1) that attempts to squeeze back in the pre-AIA judicial gloss. *Supra* pp. 22-29.

Finally, Congress made material changes by eliminating pre-AIA § 102(c) and § 102(g). As noted above, pre-AIA § 102(b) itself provided no statutory basis for treating sales of products by patentees differently from such sales by third parties. *Supra* pp. 4, 6-7. So the only possible foundation for the pre-AIA asymmetrical interpretation of § 102(b) came, indirectly, from the perceived policies of other provisions. For instance, under pre-AIA § 102(c), a person was not entitled to a patent if he had "abandoned the invention." 35 U.S.C. § 102(c) (1952); *see* 2A Chisum on Patents § 6.02[5][b] (positing that pre-AIA § 102(c) may be the statutory

foundation for pre-AIA treatment of patentee sales of a product made by a secret inventive process). And under pre-AIA § 102(g), another's prior invention prevented a later inventor from obtaining a patent on the invention only where the prior inventor "had not abandoned, suppressed, or concealed" it. 35 U.S.C. § 102(g) (1952). Section 102(g) codified the principle that "the law prefers and will reward earlier disclosure over earlier invention." *Horwath v. Lee*, 564 F.2d 948, 950 (C.C.P.A. 1977). And as noted above (*supra* pp. 6-8), this Court relied on that principle to hold that third-party sales of products of secret inventive processes did not trigger the pre-AIA on-sale bar. *W.L. Gore*, 721 F.2d at 1550. The Court reasoned that "[e]arly public disclosure is a linchpin of the patent system" as demonstrated by the fact that "[a]s between a prior inventor who benefits from a process by selling its product but suppresses, conceals, or otherwise keeps the process from the public, and a later inventor who promptly files a patent application from which the public will gain a disclosure of the process, the law favors the latter." *Id.* (citing *Horwath*).

Together, pre-AIA §§ 102(c) and 102(g) arguably suggested that the identity of the individual making the sale should matter. A patentee's decision to keep an invention secret while profiting from it evinced something akin to abandonment of the patent right (pre-AIA § 102(c)). In contrast, because the law favored the inventor who disclosed the invention over someone who had concealed it (pre-AIA § 102(g)),

a third party's secret commercial use of an inventive process should not prevent an unrelated inventor from obtaining a patent. But Congress repealed both subsections in the AIA, removing these potential statutory hooks for an inventor-only forfeiture rule.

The ALJ resisted this conclusion on the ground that nothing in the legislative history shows that Congress intended to harmonize the rules applicable to first-party and third-party sales. Appx14. But "it would be a strange canon of statutory construction that would require Congress to state in committee reports or elsewhere in its deliberations that which is obvious on the face of a statute." *Harrison v. PPG Indus., Inc.*, 446 U.S. 578, 592 (1980). Congress enacted an on-sale bar that applies to first-party and third-party sales alike, while also eliminating provisions that might have provided grounds for treating such sales differently. Because Congress could not have intended to create a supercharged rule that covers all product sales—by anyone, anywhere in the world, even a day before the filing of the patent application on the secret inventive process used to make the product (*supra* pp. 30-31)—it must have intended § 102(a)(1) to have its plain meaning.

2. Helsinn *does not support the ALJ's atextual interpretation*

The ALJ also reasoned that "Celanese's position is contrary to the Supreme Court's decision in *Helsinn*." Appx10. Not so: the claimed invention in *Helsinn* was on sale; the claimed invention here was not.

In *Helsinn*, the Supreme Court held that "an inventor's sale of an invention to a third party who is obligated to keep the invention confidential can qualify as prior art under §102(a)." 139 S. Ct. at 634. Helsinn had obtained a patent covering a fixed dose of palonosetron. *Id.* at 631. Before filing its patent application, however, Helsinn entered into a supply and purchase agreement with MGI Pharma, Inc., under which Helsinn agreed to supply (and MGI agreed to purchase) any palonosetron product approved by the FDA. *Id.* As this Court held and Helsinn did not challenge in the Supreme Court, "the Supply and Purchase Agreement constituted a commercial sale or offer for sale" and Helsinn's invention was "ready for patenting." *Helsinn Healthcare S.A. v. Teva Pharms. USA, Inc.*, 855 F.3d 1356, 1367, 1371 (Fed. Cir. 2017). In the Supreme Court, Helsinn instead argued that its invention was not "on sale" within the meaning of § 102(a)(1) because MGI was contractually obligated to keep the invention confidential and the sale thus did not make the details of the invention available to the public. Brief for Petitioner at 47, *Helsinn*, 139 S. Ct. 628 (No. 17-1229). The Supreme Court disagreed, holding that Congress's enactment of § 102(a) did not disturb the well-settled meaning of "on sale" in pre-AIA § 102(b), under which a sale need not "make the details of the invention available to the public." *Helsinn*, 139 S. Ct. at 630.

In relying on *Helsinn* here, the ALJ emphasized this conclusion about the well-settled meaning of "on sale." Appx10-12. But that established meaning

disproves the ALJ's decision. As *Helsinn* explained, "[m]ore than 20 years ago, [the Supreme Court] determined that an invention was 'on sale' within the meaning of an earlier version of § 102(a) when it was 'the subject of a commercial offer for sale' and 'ready for patenting,'" and "the reenactment of the phrase 'on sale' in the AIA did not alter this meaning." 139 S. Ct. at 630. Applied here, Celanese's "invention" was not "the subject of a commercial offer for sale," only the products made by the invention were.

Contrary to the ALJ's suggestion, *Helsinn* offers no support for the different proposition that, in enacting § 102(a)(1), Congress intended to incorporate every judicial gloss on pre-AIA § 102(b)—no matter how atextual. In fact, "[a]s [this Court] stated in [its] panel opinion" in *Helsinn*, "the rule in *Metallizing* simply is not implicated by the facts of this case." *Helsinn*, 2018 WL 1583031, at *2 n.1 (O'Malley, J., concurring in denial of panel rehearing); *Helsinn*, 855 F.3d at 1369 (stating "[t]his public use issue is not before us, and we decline to address it"). Rather, the Supreme Court in *Helsinn* decided only that "the sale of an invention to a third party who is contractually obligated to keep the invention confidential places the invention 'on sale' within the meaning of § 102(a)." 139 S. Ct. at 630. In doing so, the Court emphasized that it had already held that "an offer for sale could cause an inventor to lose the right to patent, without regard to whether the offer discloses each detail of the invention." *Id*. at 633. "[I]mplicit" in the Supreme Court's

precedent was the principle that "a sale or offer of sale need not make an invention available to the public," and this Court's precedent had made that point explicit. *Id*. at 633.

Here, in contrast, no Supreme Court precedent had implied that an invention is "on sale" under pre-AIA § 102(b) when the invention is not "the subject of a commercial offer for sale" at all. To the contrary, as *Helsinn* noted, Supreme Court precedent had "focus[ed] on whether *the invention* had been sold." *Id.* at 633 (emphasis added). Celanese's invention—its process for producing Ace-K—has not.

3. The legislative history of the AIA supports Celanese's interpretation, not the ALJ's

The ALJ also looked to legislative history, concluding it supported his reading of § 102(a)(1). Appx15-16. Although there is no need to consult legislative history given the clarity of the text, structure, and purpose of the AIA, that history confirms Celanese's interpretation, not the ALJ's.

The legislative history demonstrates that Congress intended to clear away any judicial gloss that would treat as "prior art" something that disclosed the claimed invention to no one. The sole committee report issued in connection with the AIA stated that § 102 "modifies the prior-art sections of the patent law." H.R. Rep. No. 112-98, at 42-43 (2011). "Prior art will be measured from the filing date of the application and will typically include all art that publicly exists prior to the filing

date, other than disclosures by the inventor within 1 year of filing." *Id.* at 42. It explained that "the phrase 'available to the public' is added to clarify the broad scope of relevant prior art, as well as to emphasize the fact that it must be publicly accessible." *Id.*

The report also stated that the AIA's new grace period (in § 102(b)) would cover "all actions by the patent owner during the year prior to filing that would otherwise create § 102(a) prior art" (a statement that, as noted above, would not be true under the ALJ's interpretation). *Id.* at 43 & n.20. In support, it cited a colloquy between Senators Leahy and Hatch (the Senate majority and minority sponsors of the AIA), in which Senator Leahy stated that "[o]ne of the implications of the point we are making is that subsection 102(a) was drafted in part to do away with precedent under current law that private offers for sale or private uses or secret processes practiced in the United States that result in a product or service that is then made public may be deemed patent-defeating prior art." 157 Cong. Rec. S.1496-97 (daily ed. March 9, 2011) (cited at H.R. Rep. No. 112-98, at 43 & n.20 (2011)). It also cited Senator Jon Kyl, who stated that the new § 102(a)(1) would "limit[] all non-patent prior art to that which is available to the public." 157 Cong. Rec. S.1370-71 (daily ed. March 8, 2011) (cited at H.R. Rep. No. 112-98, at 43 & n.20 (2011)).

Despite this history, the ALJ gathered support for his contrary view from an unenacted prior bill and from a House report for a later, also unenacted bill. Appx16

(quoting H.R. Rep. No. 110-314, at 57 (2007)). That history has no bearing here for at least three reasons.

First, it relates to unenacted laws and is contrary to the legislative history of the law actually enacted. To the extent any legislative history matters, it is the history of the law Congress passed. *Star Athletica, LLC v. Varsity Brands, Inc.*, 137 S. Ct. 1002, 1015 (2017) (rejecting petitioner's reliance on "history of failed legislation" because "'[c]ongressional inaction lacks persuasive significance' in most circumstances." (brackets in original)).

Second, at most, the legislative history cited by the ALJ suggests that Congress wanted to keep the categories of "public use" and "on sale," rather than scrap them entirely. H.R. Rep. No. 110-314, at 57 (2007). But that does not suggest it intended to incorporate peripheral interpretations of those bars that contradict § 102(a)'s new text. After all, "on sale" did have a settled meaning—it meant that an invention had to be "the subject of a commercial offer for sale and ready for patenting." *Helsinn*, 139 S. Ct. at 630 (internal quotations omitted). That Congress did not want to throw out that settled meaning hardly suggests Congress meant to adopt interpretations—like the one the ALJ accepted here—that contradict it.

Third, the House report cited by the ALJ described the bill before it was revised, during the Senate's July 2007 markup, to provide that the claimed invention must be "in public use, on sale, or *otherwise available to the public*." Patent Reform

Act of 2007, S. 1145, 110th Cong., 2d Sess. sec. 2, § 102(a)(1) (Jan. 24, 2008) (emphasis added); *compare with* Patent Reform Act of 2007, H.R. 1908, 110th Cong., 1st Sess., sec. 3, § 102(a)(1) (Sept. 11, 2007). The contemporaneous committee report explained that the revision "added the phrase 'otherwise available to the public' to § 102 to make clear that secret collaborative agreements, which are not available to the public, are not prior art." S. Rep. No. 110-259, at 39 (2008). It also explained that "the phrase 'available to the public' is added to clarify the broad scope of relevant prior art, as well as to emphasize the fact that it must be publicly available." S. Rep. No. 110-259, at 9 (2008). So even assuming Congress thought that unenacted bill would incorporate pre-AIA on-sale caselaw lock, stock, and barrel, that says nothing about the meaning of the different language Congress ultimately chose to adopt.

The ALJ was also mistaken that "the final language of § 102 in the AIA was adopted over the objections of senators who wanted to get rid of the very rule being advanced by Jinhe here." Appx16. To the contrary, the senators and representatives who spoke out against pre-AIA precedent on secret prior art were also ones who supported § 102's final language. For instance, Senator Kyl, a sponsor of the AIA, celebrated "[t]he present bill's elimination of the patent forfeiture doctrines in favor of a general public availability standard" because "[t]he current forfeiture doctrines have become traps for unwary inventors and impose extreme results to no real

purpose." 157 Cong. Rec. S1370-71 (daily ed. Mar. 8, 2011). Similarly, Senator Leahy, one of the law's namesakes, took the position that the new § 102(a) had done "away with" precedent holding that "private uses or secret processes practiced in the United States that result in a product or service that is then made public may be deemed patent-defeating prior art." 157 Cong. Rec. S1496 (daily ed. Mar. 9, 2011). And Representative Smith, the law's other namesake, stated that "contrary to current precedent, in order to trigger the bar in the new 102(a) in our legislation, an action must make *the patented subject matter* 'available to the public' before the effective filing date." 157 Cong. Rec. H4429 (daily ed. June 22, 2011) (emphasis added).

In short, the legislative history strongly supports that Congress intended courts to give § 102(a)(1) its plain meaning—a meaning that contradicts the pre-AIA judicial gloss that patentee sales of products made by a secret, inventive process place the invention "on sale."

4. The PTO's guidance provides no support for the ALJ's decision

Finally, the ALJ pointed to recent guidance from the PTO to patent examiners regarding the on-sale provision. Appx16-17. That guidance provides:

> The pre-AIA 35 U.S.C. 102(b) "on sale" provision has been interpreted as including commercial activity even if the activity is secret. *See* MPEP § 2133.03(b), subsection III.A. AIA 35 U.S.C. 102(a)(1) uses the same "on sale" term as pre-AIA 35 U.S.C. 102(b) and is treated as having the same meaning. In *Helsinn Healthcare S.A. v. Teva Pharmaceuticals USA, Inc.*, 139 S. Ct 628 (2019),

> the Supreme Court "determine[d] that Congress did not alter the meaning of 'on sale' when it enacted the AIA, [and held] that an inventor's sale of an invention to a third party who is obligated to keep the invention confidential can qualify as prior art under [AIA 35 U.S.C.] § 102(a)." *Id.* at 634. Thus, a sale or offer for sale that does not disclose the subject matter of an invention or make the invention available to the general public may nevertheless qualify as prior art in an anticipation or obviousness rejection, regardless of whether the application or patent under consideration is subject to the FITF [first inventor to file] provisions of the AIA or the first to invent provisions of pre-AIA law.

Manual of Patent Examining Procedure § 2152.02(d).

The PTO's guidance provides no support for the ALJ's decision. First, to the extent the guidance merely reiterates the holding of *Helsinn*, that holding only supports Celanese's position for the reasons discussed above. *Supra* pp. 42-45. Second, to the extent the guidance suggests more broadly that secret commercial activity that does not involve a commercial offer to sell the claimed invention will nonetheless trigger the AIA's on-sale provision, that interpretation is entitled to no deference. The PTO has no authority to declare substantive patent law. *See Animal Legal Defense Fund v. Quigg*, 932 F.2d 920, 930 (Fed. Cir. 1991); *Merck & Co., Inc. v. Kessler*, 80 F.3d 1543, 1550 (Fed. Cir. 1996). And even if it did, the statutory language Congress enacted unambiguously requires the claimed invention to be on sale, so the PTO would have no license to say differently.

C. Because The AIA's On-Sale Provision Does Not Invalidate Celanese's Patent Claims On Its Inventive Process, The Decision Should Be Reversed And The Case Remanded

Based on his conclusion that all of the patent claims Celanese allegedly practices were invalidated by the AIA's on-sale provision, the ALJ terminated this Tariff Act investigation because Celanese could not establish that it practices any valid claim. Appx17-18; *see Broadcom Corp. v. ITC*, 28 F.4th 240, 249-50 (Fed. Cir. 2022) (establishing a violation of 19 U.S.C. § 1337 requires the complainant to establish, among other things, that it "practices at least one claim of the asserted patent"). So once the ALJ's invalidity determination is reversed, the case must be remanded for further proceedings on Celanese's complaint.

CONCLUSION

The decision of the Commission should be reversed and the case remanded for further proceedings.

Dated: October 21, 2022

Respectfully submitted,

AARON G. FOUNTAIN
MORRISON & FOERSTER LLP
701 Brazos Street, Suite 1100
Austin, TX 78701

LENA H. HUGHES
MORRISON & FOERSTER LLP
250 West 55th Street
New York, NY 10019

/s/ Deanne E. Maynard
DEANNE E. MAYNARD
BRIAN R. MATSUI
SETH W. LLOYD
MORRISON & FOERSTER LLP
2100 L Street NW, Suite 900
Washington, DC 20037
Telephone: (202) 887-8740
DMaynard@mofo.com

Counsel for Appellants Celanese International Corporation, Celanese (Malta) Company 2 Limited, Celanese Sales U.S. LTD.

ADDENDUM WITH ORDERS AND PATENTS

CELANESE INTERNATIONAL CORPORATION, CELANESE (MALTA) COMPANY 2 LIMITED, CELANESE SALES U.S. LTD.,

V.

INTERNATIONAL TRADE COMMISSION, ANHUI JINHE INDUSTRIAL CO., LTD., JINHE USA LLC.

No. 22-1827 (Fed. Cir.)

ADDENDUM WITH ORDERS AND PATENTS TABLE OF CONTENTS

UNITED STATES INTERNATIONAL TRADE COMMISSION
Washington, D.C.

In the Matter of **CERTAIN HIGH-POTENCY SWEETENERS, PROCESSES FOR MAKING SAME, AND PRODUCTS CONTAINING SAME**	**Investigation No. 337-TA-1264**

NOTICE OF A COMMISSION DETERMINATION NOT TO REVIEW AN INITIAL DETERMINATION GRANTING SUMMARY DETERMINATION OF NO VIOLATION OF SECTION 337; TERMINATING THE INVESTIGATION

AGENCY: U.S. International Trade Commission.

ACTION: Notice.

SUMMARY: Notice is hereby given that the U.S. International Trade Commission ("Commission") has determined not to review an initial determination ("ID") (Order No. 29) of the presiding administrative law judge granting summary determination of no violation of section 337. This investigation is terminated.

FOR FURTHER INFORMATION CONTACT: Benjamin S. Richards, Esq., Office of the General Counsel, U.S. International Trade Commission, 500 E Street S.W., Washington, D.C. 20436, telephone (202) 708-5453. Copies of non-confidential documents filed in connection with this investigation may be viewed on the Commission's electronic docket (EDIS) at *https://edis.usitc.gov*. For help accessing EDIS, please email EDIS3Help@usitc.gov. General information concerning the Commission may also be obtained by accessing its Internet server at *https://www.usitc.gov*. Hearing-impaired persons are advised that information on this matter can be obtained by contacting the Commission's TDD terminal on (202) 205-1810.

SUPPLEMENTARY INFORMATION: The Commission instituted this investigation on May 14, 2021. 86 FR 26544-45 (May 14, 2021). The complaint, as supplemented, was filed by complainants Celanese International Corporation of Irving, Texas; Celanese (Malta) Company 2 Limited of Qormi, Malta; and Celanese Sales U.S. Ltd. of Irving, Texas (collectively "Celanese") and alleged violations of section 337 of the Tariff Act of 1930, as amended, 19 U.S.C. 1337, in the importation into the United States, the sale for importation, or the sale within the United States after importation of certain high-potency sweeteners, processes for making same, and products containing same by reason of infringement of certain claims of U.S. Patent No. 10,023,546, U.S. Patent No. 10,208,004, U.S. Patent No. 10,590,098, U.S. Patent No. 10,233,163, and U.S. Patent No. 10,590,095. *Id.* The complaint further alleged that a domestic industry exists. *Id.* The Commission's notice of investigation named twelve respondents, including Anhui Jinhe Industrial Co., Ltd. and Jinhe USA LLC ("Jinhe"). *Id.* On August 6,

2021, the Chief Administrative Law Judge ("CALJ") issued an ID granting a motion by Celanese to add eleven additional respondents to the investigation. Order No. 14, *unreviewed by* Comm'n Notice (Aug. 23, 2021). On August 26, 2021, Celanese filed an amended complaint adding the eleven additional respondents. The Office of Unfair Import Investigations ("OUII") is also participating in this investigation. 86 FR at 26544.

On September 2, 2021, respondent Jinhe filed a motion for summary determination of no violation based on the contention that all of the asserted patent claims that Celanese relied on to satisfy the technical prong of the domestic industry requirement are invalid under the "on-sale bar" provisions of 35 U.S.C. 102(a)(1). On September 13, 2021, Celanese filed a brief in opposition. OUII filed a brief in support of Jinhe's motion on the same day. The CALJ held oral argument on Jinhe's motion on September 28, 2021.

The CALJ issued the subject ID granting Jinhe's motion on January 11, 2022. Specifically, the ID found that the on-sale bar applied to invalidate all of the remaining claims that Celanese relied on to establish a domestic industry. Accordingly, the ID found that the investigation should be terminated with a finding of no violation of section 337 due to Celanese's inability to satisfy the domestic industry requirement of section 337. Celanese petitioned for review of the ID on January 21, 2022. Jinhe and OUII submitted responses opposing Celanese's petition on January 28, 2022.

Having examined the record of this investigation, including the ID, the petition for review, and the responses thereto, the Commission has determined not to review the ID. This investigation is terminated in its entirety.

The Commission vote for this determination took place on April 1, 2022.

While temporary remote operating procedures are in place in response to COVID-19, the Office of the Secretary is not able to serve parties that have not retained counsel or otherwise provided a point of contact for electronic service. Accordingly, pursuant to Commission Rules 201.16(a) and 210.7(a)(1) (19 CFR 201.16(a), 210.7(a)(1)), the Commission orders that the Complainant(s) complete service for any party/parties without a method of electronic service noted on the attached Certificate of Service and shall file proof of service on the Electronic Document Information System (EDIS).

The authority for the Commission's determination is contained in section 337 of the Tariff Act of 1930, as amended (19 U.S.C. 1337), and in Part 210 of the Commission's Rules of Practice and Procedure (19 CFR Part 210).

By order of the Commission.

Lisa R. Barton
Secretary to the Commission

Issued: April 1, 2022

CERTIFICATE OF SERVICE

I, Lisa R. Barton, hereby certify that the attached document has been served via EDIS upon the Commission OUII Investigative Attorney and the following parties as indicated, upon the date listed below.

Document	Security	Document Type	Official Rec'd Date	Title
767156	Public	Notice	04/01/2022 12:43 PM	Commission Determination Not to Review an Initial Determination Granting Summary Determination of...

Service Date: April 01, 2022

/s/

Lisa R. Barton
U.S. International Trade Commission
500 E Street, S.W.
Suite 112
Washington, D.C. 20436

Service Date: April 01, 2022 PDF Generated on: April 01, 2022

CERTIFICATE OF SERVICE

On behalf of Complainant Celanese (Malta) Company 2 Limited;Celanese International Corporation;Celanese Sales U.S. Ltd. :

Susan Krumplitsch
DLA Piper LLP (US)
2000 University Avenue
East Palo Alto, California 94303-2214, United States
Electronic Service

On behalf of Respondent Anhui Jinhe Industrial Co., Ltd.;Jinhe USA LLC:

Gary M. Hnath
Mayer Brown LLP
1909 K Street, NW
Washington, District of Columbia 20006, United States
Electronic Service

On behalf of Respondent Prinova US LLC:

Arthur Gasey
Vitale Vickrey Niro Solon & Gasey LLP
1225 N. Belmont
Arlington Heights, Illinois 60004, Unspecified
gasey@vvnlaw.com
Electronic Service

On behalf of Respondents Batory Foods:

No designated lead counsel

1700 Higgins Road
Suite 300
Des Plaines, 60018, United States of America
Paper Service

On behalf of Respondents V-Chem Trading Ltd.:

No designated lead counsel

No. 290 Wanda Plaza
Yixing City
Jiangsu, China
Paper Service

On behalf of U.S. International Trade Commission :

Yoncha Kundupoglu
OUII Investigative Attorney
500 E Street, S.W.
Washington, D.C. 20436
Internal Service

Service Date: April 01, 2022 PDF Generated on: April 01, 2022

UNITED STATES INTERNATIONAL TRADE COMMISSION

Washington, D.C.

In the Matter of **CERTAIN HIGH-POTENCY SWEETENERS, PROCESSES FOR MAKING SAME, AND PRODUCTS CONTAINING SAME**	**INV. NO. 337-TA-1264**

ORDER NO. 29: INITIAL DETERMINATION GRANTING RESPONDENTS' MOTION FOR SUMMARY DETERMINATION THAT THE ENTIRE INVESTIGATION BE TERMINATED DUE TO INVALIDITY OF THE ASSERTED PATENTS

(January 11, 2022)

On September 2, 2021, respondent Jinhe[1] filed a motion (Mot.) for summary determination of no violation based on a contention all patent claims asserted in this investigation are invalid. Motion Docket No. 1264-007. The motion alleges complainant Celanese[2] sold products produced according to the patent claims more than a year before the effective filing date of the patents, triggering the on-sale bar provision of 35 U.S.C. § 102(a)(1). *Id.* On September 13, 2021, Celanese filed a brief in opposition (Opp'n) and a disputed chart of material facts (DCMF).

[1] "Jinhe" refers collectively to respondents Anhui Jinhe Industrial Co., Ltd. and Jinhe USA LLC.

[2] "Celanese" refers collectively to complainants Celanese International Corporation, Celanese (Malta) Company 2 Limited, and Celanese Sales U.S. Ltd.

The Commission Investigative Staff filed a brief supporting Jinhe's motion on September 13, 2021.[3] I held oral argument on the motion on September 28, 2021.[4]

I. BACKGROUND

As listed in the table below, Celanese presently asserts three patents in this investigation (the Asserted Patents):

U.S. Patent Number	Asserted Claims
10,023,546 (the '546 patent)	11, 15, and 27
10,208,004 (the '004 patent)	7, 11, 28, and 33
10,590,095 (the '095 patent)	1, 19, and 34

See 86 Fed. Reg. 26544 (May 14, 2021); Order No. 20 (Sept. 21, 2021), *unreviewed*, Comm'n Notice (Oct. 14, 2021); Order No. 25 (Nov. 23, 2021), *unreviewed*, Comm'n Notice (Dec. 21, 2021); Order No. 28 (Jan. 10, 2022) (pending Commission review).

The Asserted Patents are grouped into two families: (1) the '546 and '004 patent family and (2) the '095 patent family. The '546 and '004 patent share a single specification. Mot. Ex. 2 ('004 patent) at 1:8-12 (the '004 patent is a continuation of the '546 patent). Each Asserted Patent has an effective filing date of September 21, 2016, and claims improvements to a conventional method for making acesulfame potassium (Ace-K), an artificial sweetener used in foods, drinks, and medicines. DCMF 4; *see* Mot. Exs. 2 and 6.

[3] Subsequently, Jinhe moved for leave to submit a reply brief (EDIS Doc. ID 751927) and Celanese moved for leave to submit a sur-reply brief (EDIS Doc. ID 752185). Motion Docket Nos. 1264-009 and -011. Neither motion for leave was opposed. Unopposed Motion Nos. 1264-009 and -011 for leave are granted.

[4] The transcript of the oral argument is available on EDIS as Doc. ID 752887 and is hereinafter referred to as "Tr."

During prosecution of the Asserted Patents, Celanese disclosed to the Patent Office that the claimed process for making Ace-K had been in secret use in Europe and that Ace-K made using that process had been exported and sold in the United States for more than one year before the Asserted Patents' effective filing date. DCMF Nos. 6-12. In other words, Celanese had produced and sold Ace-K before the critical date of September 21, 2015. It is undisputed that Celanese's method of making Ace-K has not changed in any material way since 2011. DCMF Nos. 16-17.

The Asserted Patents all claim priority to provisional applications that were filed after the effective date of amendments to 35 U.S.C. § 102 made by the Leahy-Smith America Invents Act (AIA). Therefore, the AIA version of the on-sale bar recited in § 102(a) governs the pending motion. *Valve Corp. v. Ironburg Inventions Ltd.*, 8 F.4th 1364, 1370 n.3 (Fed. Cir. 2021) (*citing* Leahy-Smith America Invents Act, Pub L. 112-29 § 3(b), (n),[5] 125 Stat. 284, 285-86, 293 (2011)). As explained in more detail below, this motion turns on language found in the AIA version of the on-sale bar that is not present in the pre-AIA statute. The AIA presently defines the on-sale bar as follows:

> (a) A person shall be entitled to a patent unless—
>
> > (1) the *claimed* invention was patented, described in a printed publication, or in public use, on sale, *or otherwise available to the public* before the effective filing date of the claimed invention

35 U.S.C. § 102(a)(1) (emphasis added).

[5] Amendments made to 35 U.S.C. § 102 took effect upon expiration of the 18-month period beginning on the date the AIA was enacted. The AIA was enacted on September 16, 2011.

By contrast, the pre-AIA version of the on-sale bar, which remained in effect up to March 16, 2013, did not include the phrase "claimed invention" or the phrase "or otherwise available to the public"; it provided:

> A person shall be entitled to a patent unless —
>
> . . .
>
> (b) the invention was patented or described in a printed publication in this or a foreign country or in public use or on sale in this country, more than one year prior to the date of the application for patent in the United States

35 U.S.C. § 102(b) (pre-AIA).

Both before and after the AIA amendments, courts were and are in agreement that the on-sale bar applies when two conditions are satisfied before a claim's effective filing date. *Pfaff v. Wells Elecs., Inc.*, 525 U.S. 55, 67 (1998); 35 U.S.C. § 102(a)(1); *see also Helsinn Healthcare S.A. v. Teva Pharmas. USA, Inc.*, 139 S. Ct. 628, 633 (2019). First, the invention itself must the subject of a commercial offer for sale. *Pfaff*, 525 U.S. at 67-68; *BASF Corp. v. SNF Holding Co*., 955 F.3d 958, 969 (Fed. Cir. 2020). Second, the invention must be ready for patenting, which can be shown by proof of reduction to practice. *Pfaff*, 525 U.S. at 67-68.

The Federal Circuit has recognized that a patented process presents particular considerations with respect to the on-sale bar because a process invention consists of acts rather than a tangible item. *BASF*, 955 F.3d at 969 (*citing In re Kollar*, 286 F.3d 1326, 1332 (Fed. Cir. 2002)). In certain circumstances, a patentee's sale of a product made by a later-patented process is considered a sale of the invention, invoking the on-sale bar. *Id*. (*citing Metallizing Eng'g Co., Inc. v. Kenyon Bearing & Auto Parts Co*., 153 F.2d 516 (2d Cir. 1946)); *Medicines Co. v. Hospira, Inc.*, 827 F.3d 1363, 1376 (Fed. Cir. 2016) ("we have held that the sale of products made using patented methods triggers the on-sale bar, even though title to the claimed method itself did not pass"); *D.L. Auld Co. v. Chroma Graphics Corp*., 714 F.2d 1144, 1147-48 (Fed. Cir. 1983)

(placing the product of a method invention on sale more than one year before filing a patent application bars grant of a valid patent on the method).

Under the pre-AIA version of the on-sale bar, it was well settled that a patentee's sale of an unpatented product made according to a secret method triggered the on-sale bar to patentability. The Federal Circuit explained that even "where a patented method is kept secret and remains secret after a sale of the unpatented product of the method," a sale of a product made by the secret method "prior to the critical date is a bar if engaged in by the patentee or patent applicant . . ." *In re Caveney*, 761 F. 2d 671, 675 (Fed. Cir. 1985). Thus, Celanese's pre-2015 U.S. sales of Ace-K made according to its secret method, which it later claimed in the Asserted Patents, would have triggered the pre-AIA on-sale bar.

Celanese contends that when Congress changed the statute by adding the word "claimed" as a modifier of "invention" and making other amendments it intended to change existing law and allow patent protection for products made by the patentee using a secret process. *See* Opp'n at 15-17. This motion turns, therefore, on whether the AIA changed the meaning of the on-sale bar provision such that Celanese's pre-2015 sales of Ace-K do not invalidate the Asserted Patents.

II. UNDISPUTED FACTS

I find the following facts are not in dispute.

Celanese's process to make Ace-K claimed in the Asserted Patents has been in secret use in Europe since before the undisputed critical date, which is September 21, 2015. DCMF Nos. 6-12. The Ace-K product made using Celanese's process has been exported and sold in the United States since before September 21, 2015. DCMF Nos. 6-12. Celanese's method of making Ace-K has not changed in any material way since 2011. DCMF Nos. 16-17.

Celanese's process to make Ace-K practices (at least) the following asserted claims:

- '546 patent: claims 11 and 27;

- ’004 patent: claims 7, 28, and 33;
- ’095 patent: claims 1, 19, and 34.

Mot. Exs. 10-11 (Celanese’s domestic industry technical prong contention charts for the ’546 and ’004 patents); Mot. Ex. 14 (Celanese’s domestic industry technical prong contention charts for the ’095 patent); *see* DCMF No. 38.[6]

III. ANALYSIS

Summary determination is appropriate when there is no genuine issue as to any material fact and the moving party is entitled to a determination as a matter of law. *See* 19 C.F.R. § 210.18. In determining whether there is a genuine issue of material fact, “the evidence must be viewed in the light most favorable to the party opposing the motion with doubts resolved in favor of the non-movant.” *Crown Operations Int’l, Ltd. v. Solutia, Inc.*, 289 F.3d 1367, 1374 (Fed. Cir. 2002) (citations omitted).

Celanese contends that the AIA’s amendments to § 102 overturn long-held precedent that a patentee’s sale of an unpatented product made according to a secret method triggers the on-sale bar to patentability. As discussed below, Celanese’s position is contrary to the Supreme Court’s decision in *Helsinn*, where the Court held that Congress did not alter the meaning of the on-sale bar provision when it enacted the AIA. 139 S. Ct. at 628.

Helsinn, a pharmaceutical company, licensed the sale of its patented chemotherapy drug at a specific dose but required licensees to keep the dosage information confidential. 139 S. Ct. at 631. Helsinn subsequently filed a provisional patent application covering the specific drug dose more than two years after it had entered into the sales agreement with its licensee. *Id.* Helsinn

[6] Celanese does not contend that it practices the process in asserted claim 15 of the ’546 patent or the process in asserted claim 11 of the ’004 patent when it makes Ace-K. Mot. Exs. 10-11; DCMF No. 38.

asserted the resulting '219 patent in an enforcement suit against generic drug manufacturer Teva, who raised an on-sale bar defense to infringement. *Id.* Specifically, Teva asserted that the '219 patent was invalid because the specific dose claimed in the patent was "on sale" more than one year before Helsinn filed the provisional patent application that matured into the '219 patent. *Id.*

The district court that first heard the dispute between Helsinn and Teva determined that the AIA's on-sale bar provision did not render the '219 patent invalid. 139 S. Ct. at 632 (citing *Helsinn Healthcare S.A. v. Dr. Reddy's Labs. Ltd.*, 387 F. Supp. 3d 439 (D.N.J. 2016)). The district court concluded that, "under the AIA, an invention is not 'on sale' unless the sale or offer in question made the claimed invention available to the public." *Id.* As the sale from Helsinn to its licensee did not disclose the specific dose claimed in the '219 patent, the district court found that the claimed invention was not "on-sale" before the '219 patent's critical date. *Id.*

The Federal Circuit reversed the district court's holding that the on-sale bar did not apply. *Helsinn Healthcare S.A. v. Teva Pharmas. USA, Inc.*, 855 F.3d 1356, 1360 (Fed. Cir. 2017). It concluded that "if the existence of the sale is public, the details of the invention need not be publicly disclosed in the terms of sale" to fall within the AIA's on-sale bar. *Id.* at 1371. Because the sale between Helsinn and its licensee was publicly disclosed, the Federal Circuit held that the on-sale bar applied. *Id.* at 1364, 1371.

The Supreme Court granted certiorari to determine "whether, under the AIA, an inventor's sale of an invention to a third party who is obligated to keep the invention confidential qualifies as prior art for purposes of determining the patentability of the invention." 139 S. Ct. at 632. The Court's opinion reviews the constitutional and philosophical underpinnings of the federal patent system and notes that "[e]very patent statute since 1836 has included an on-sale bar." *Id.* at 633 (citing *Pfaff*, 119 S. Ct. at 304). The opinion further notes that "Congress enacted the AIA in 2011

against the backdrop of a substantial body of law interpreting § 102's on-sale bar" and identifies Federal Circuit precedents holding that "secret sales" can invalidate a patent. *Id.* (citing *Woodland Trust v. Flowertree Nursery, Inc.*, 148 F.3d 1368, 1370 (1998) ("Thus an inventor's own prior commercial use, albeit kept secret, may constitute a public use or sale under § 102(b), barring him from obtaining a patent."). In view of "this settled pre-AIA precedent on the meaning of 'on sale,'" the *Helsinn* court concluded that "when Congress reenacted the same language in the AIA, it adopted the earlier judicial construction of that phrase" and affirmed the Federal Circuit's determination that an inventor's sale of an invention to a third party who is obligated to keep the invention confidential can trigger the on-sale bar under § 102(a). *Id.* at 633-34; *see also id.* at 634 ("[W]e determine that Congress did not alter the meaning of 'on sale' when it enacted the AIA.").

Thus, the Supreme Court's *Helsinn* opinion, although not addressing the exact fact pattern arising this investigation, supports a conclusion that Congress's enactment of the AIA did not overturn long-established precedent holding that a patentee's sale of an unpatented product made according to a secret method triggers the on-sale bar to patentability under § 102. *See, e.g.*, *Caveney*, 761 F. 2d at 675.

Celanese contends otherwise, arguing that textual changes to § 102 enacted with the AIA overturned the long history of judicial precedent interpreting the on-sale bar. Specifically, Celanese takes the position that the AIA's use of the phrase "claimed invention" in the on-sale bar provision, in contrast to the pre-AIA version's use of the standalone word "invention," means that the on-sale bar can now only be triggered by the public sale or use of the claimed invention itself, and not by the public sale or use of a product made according to a claimed method. *See, e.g.*, Tr. at 44:22-45:17, 49:1-10, 50:2-6. Celanese's argument lacks merit, however, because pre-AIA precedent already recognized the distinction that Celanese contends was created by the

amendment. Pre-AIA cases recognized that the product of a claimed method was distinct from the steps of a method invention, but precedents also recognized that a product could embody commercialization of a method invention sufficiently to trigger the on-sale bar. *See, e.g.*, *D.L. Auld*, 714 F.2d at 1148 ("a party's placing of ***the product of a method invention*** on sale more than a year before that party's application filing date must act as a forfeiture of any right to the grant of a valid patent on the method to that party if circumvention of the policy animating §102(b) is to be avoided in respect of patents on method inventions") (emphasis added). The AIA's addition of the word "claimed" to modify "invention"—with no indication in other statutory text or legislative history about what change was intended—"would be a fairly oblique way of attempting to overturn" a settled body of law that a patentee's sale of a product made by its use of a secret process bars the patenting of that process. *Cf. Helsinn*, 139 S. Ct. at 634 (quoting with approval *amicus* United States, who argued the AIA amendment adding the words "or otherwise available to the public" did not change the previous interpretation of the on-sale bar). Following the lead of the Supreme Court in *Helsinn*, I decline to interpret the AIA as working a change in the on-sale bar as applied to these facts.

Celanese also contends that the pre-AIA § 102(g) "codified the legal principle that the sale by another of a product made by a secret process was not a bar to patentability under pre-AIA § 102(b)," and that the AIA's elimination of § 102(g) "repeal[ed] any distinction between an inventor's own activities and those of another with regard to use and sale of the invention." *See* Opp'n at 10. In Celanese's view, the change to § 102(g) "demonstrates Congress's intention to treat the secret use of processes that result in commercialized products by patentees and third parties the same." *See id*. at 11. Celanese's argument fails to recognize the distinct policies motivating the pre-AIA on-sale bar and pre-AIA § 102(g). The Federal Circuit described "the

intent" behind the pre-AIA on-sale bar was "to preclude attempts by the inventor or his assignee to profit from commercial use of an invention for more than a year before an application for patent is filed," including the sale of the product of a method. *See D.L. Auld*, 714 F.2d 1144, 1147 (Fed. Cir. 1983). Pre-AIA § 102(g), in contrast, operated "to ensure that a patent is awarded only to the 'first' inventor," even if a different applicant was the first to file a patent application concerning the invention. *Apotex USA, Inc. v. Merck & Co.*, 254 F.3d 1031, 1035 (Fed. Cir. 2001). The legislative history of the AIA is express that the change to § 102(g) was driven by the congressional preference to convert the U.S. patent system to a "first-inventor-to-file" system. *See* 157 Cong. Rec. S5402-02 (daily ed. Sept. 8, 2011) (statement of Sen. Patrick Leahy) ("One of the key provisions of the legislation transitions the United States patent system from a first-to-invent system to a first-inventor-to-file system."); *see also id.* (statement of Sen. Roy Blunt) (elimination of § 102(g) was a result of the change to a first-inventor-to-file system). There is no indication in the text of the new statute or in its legislative history that the elimination of § 102(g) was intended to harmonize treatment of patentees and what Celanese calls "third parties" with respect to the on-sale bar.

Celanese contends the AIA's expansion of prior user rights under § 273 also demonstrates that the secret use of a process by a patentee no longer creates a statutory bar under the AIA version of § 102. *See* Opp'n at 11-15 (examining 35 U.S.C. § 273). As enacted in the AIA, § 273 provides a personal defense to individuals accused of patent infringement if the following criteria are met: (1) commercial use of the patented subject matter in the United States in connection with an internal commercial use or in connection with a sale or transfer of the end result of the foregoing commercial use and (2) the commercial use occurred more than one year before the effective date of the claimed invention. *See* 35 U.S.C. § 273. Celanese argues that the prior use protection of

§ 273 added by the AIA would be unnecessary if such a use would also be invalidating art under the AIA version of § 102(a)(1). Opp'n at 13. But Celanese's argument again conflates two distinct issues. Section 273 provides an infringement defense to one using a method prior to the patenting of that method by another; the question of whether the same operative facts will invalidate the patent is entirely distinct. *See BASF Corp.*, 955 F.3d at 968 (noting that "Congress has considered the implications of patenting secret processes" when enacting the AIA and a successful prior-use defense under § 273 "does not necessarily establish invalidity"). A patentee may very well retain a valid patent even after successful invocation of the § 273 prior use defense by an accused infringer. Thus, the prior use defense of § 273 is entirely consistent with the *Caveney* rule that states "where a patented method is kept secret and remains secret after a sale of the unpatented product of the method[,] [the] sale prior to the critical date is a bar if engaged in by the patentee or patent applicant, but not if engaged in by another." *See* 761 F.2d at 675.

Celanese also contends that certain passages from the legislative history of the AIA demonstrate Congress's intent that the sale of a product made by a secret process should no longer be a bar to the patentability of that process under § 102(a)(1). Opp'n at 15-17. In particular, Celanese cites the following passage from the House Committee Report on H.R. 1249 (the AIA) in support of its position:

> Prior art will be measured from the filing date of the application and will typically include all art that ***publicly exists*** prior to the filing date, other than disclosures by the inventor within 1 year of filing. Prior art also will no longer have any geographic limitations. Thus, in section 102 the "in this country" limitation as applied to "public use" and "on sale" is removed, and the phrase "available to the public" is added to clarify the broad scope of relevant prior art, as well as ***to emphasize the fact that it must be publicly accessible***.

H.R. Rept. No. 112-98 at 42-43 (2011) (emphases added by Celanese at Opp'n at 15). Celanese also relies on statements made by Senators Kyl and Leahy in support of its position. Opp'n at 15-17.

The legislative history cited by Celanese must be evaluated in context. As described an Amici Curiae brief submitted by 45 intellectual property law professors in connection with the *Helsinn* case before the Supreme Court,[7] the original bill leading to the AIA was introduced in Congress in 2005. It would have eliminated the former prior art categories of "public use" and "on sale" altogether, defining prior art as only things "patented, described in a printed publication, or otherwise publicly known." H.R. 2795, 109th Cong. § 3 (2005). But that language was not the language Congress adopted.

During the course of six years of congressional debate, Congress added the terms "public use" and "on sale" back into the definition of prior art. The House Report accompanying the 2007 bill that reintroduced those terms stated the bill used "the current § 102(b) as the template from which to define the scope of prior art in the Act, primarily because of how the terms 'in public use' and 'on sale' have been interpreted by the courts." H.R. Rep. No. 110-314, at 57 (2007). That—coupled with the fact that the final language of § 102 in the AIA was adopted over the objections of senators who wanted to get rid of the very rule being advanced by Jinhe here—suggests that Congress did not deliberately throw out the understanding of the on-sale bar as it had existed for decades, even if a few senators wished it were otherwise.

This interpretation of the legislative history is also consistent with guidance given by the U.S. Patent and Trademark Office to patent examiners determining whether or not to reject a patent application based on an on-sale bar:

> The pre-AIA 35 U.S.C. 102(b) "on sale" provision has been interpreted as including commercial activity even if the activity is secret. *See* MPEP § 2133.03(b), subsection III.A. AIA 35 U.S.C. 102(a)(1) uses the same "on sale" term as pre-AIA 35 U.S.C. 102(b) and is treated as having the same meaning. In *Helsinn Healthcare S.A. v. Teva Pharmaceuticals USA, Inc.*, 139 S.Ct. 628 (2019), the

[7] The amicus brief submitted by the intellectual property law professors is attached as Exhibit 1 to Jinhe's reply brief, EDIS Doc. ID 751927.

> Supreme Court "determine[d] that Congress did not alter the meaning of 'on sale' when it enacted the AIA, [and held] that an inventor's sale of an invention to a third party who is obligated to keep the invention confidential can qualify as prior art under [AIA 35 U.S.C.] § 102(a)." *Id.* at 634. Thus, a sale or offer for sale that does not disclose the subject matter of an invention or make the invention available to the general public may nevertheless qualify as prior art in an anticipation or obviousness rejection, regardless of whether the application or patent under consideration is subject to the FITF provisions of the AIA or the first to invent provisions of pre-AIA law.

Manual of Patent Examining Procedure § 2152.02(d).

In sum, the AIA did not alter the pre-AIA on-sale bar as set forth in *Caveney*: a patentee's sale of an unpatented product made according to a secret method triggers the on-sale bar to patentability.

It is undisputed that Celanese sold in the United States, more than one year before the effective filing date, Ace-K manufactured according to the inventions in the following claims:

- '546 patent: claims 11 and 27;
- '004 patent: claims 7, 28, and 33;
- '095 patent: claims 1, 19, and 34.

I therefore determine that the claims listed above are invalid pursuant to the on-sale bar provision of 35 U.S.C. § 102(a)(1):

Apart from these invalid claims, Celanese contends that Jinhe infringes the following two claims, which have not been shown to have been practiced more than one year before each claim's effective filing date:

- '546 patent: claim 15;
- '004 patent: claim 11.

Celanese does not contend, however, that its current production of Ace-K satisfies the technical prong of the domestic industry requirement by practicing either of these two claims. Mot. Exs.

10-11; DCMF No. 38. As discussed at oral argument, the parties agreed that I could decide in the context of the pending motion whether the technical prong of the domestic industry requirement has been satisfied on this record. *See* Tr. at 90:6-15. I therefore determine that Celanese does not practice any valid claim of the Asserted Patents and therefore has not met its burden to show satisfaction of the technical prong of the domestic industry requirement. Accordingly, I determine that no violation of section 337 of the Tariff Act of 1930 can be proved based on the undisputed facts and summary determination to that effect is appropriate.

IV. CONCLUSION

For the reasons set forth above, it is my initial determination that Motion No. 1264-007 is granted with a finding of no violation of section 337. This initial determination, along with supporting documentation, is hereby certified to the Commission.

Pursuant to 19 C.F.R. § 210.42(h), this initial determination shall become the determination of the Commission unless a party files a petition for review of the initial determination pursuant to 19 C.F.R. § 210.43(a), or the Commission, pursuant to 19 C.F.R. § 210.44, orders on its own motion a review of the initial determination or certain issues herein.

All pending hearings and deadlines set forth in the procedural schedule issued as Order No. 10 on June 23, 2021, and all subsequent modifications to that schedule made by order are hereby stayed pending a final resolution by the Commission of the issues addressed in this initial determination. All other motions pending in this investigation are denied as moot.

SO ORDERED.

Clark S. Cheney
Acting Chief Administrative Law Judge

CERTIFICATE OF SERVICE

I, Lisa R. Barton, hereby certify that the attached document has been served via EDIS upon the Commission OUII Investigative Attorney and the following parties as indicated, upon the date listed below.

Document	Security	Document Type	Official Rec'd Date	Title
760224	Public	ID/RD - Other Than Final on Violation	01/11/2022 01:22 PM	Initial Determination Granting Respondents' Motion for Summary Determination That the Entire Investigation Be Terminated Due to Invalidity of the Asserted Patents

Service Date: January 11, 2022

/s/

Lisa R. Barton
U.S. International Trade Commission
500 E Street, S.W.
Suite 112
Washington, D.C. 20436

Service Date: January 11, 2022 PDF Generated on: January 11, 2022

CERTIFICATE OF SERVICE

On behalf of Complainant Celanese (Malta) Company 2 Limited;Celanese International Corporation;Celanese Sales U.S. Ltd. :

Susan Krumplitsch
DLA Piper LLP (US)
2000 University Avenue
East Palo Alto, California 94303-2214, United States
Electronic Service

On behalf of Respondent Anhui Jinhe Industrial Co., Ltd.;Jinhe USA LLC:

Gary M. Hnath
Mayer Brown LLP
1909 K Street, NW
Washington, District of Columbia 20006, United States
Electronic Service

On behalf of Respondent Prinova US LLC:

Arthur Gasey
Vitale Vickrey Niro Solon & Gasey LLP
1225 N. Belmont
Arlington Heights, Illinois 60004, Unspecified
gasey@vvnlaw.com
Electronic Service

On behalf of Respondents V-Chem Trading Ltd.:

No designated lead counsel

No. 290 Wanda Plaza
Yixing City
Jiangsu, China
Paper Service

On behalf of Respondents Batory Foods:

No designated lead counsel

1700 Higgins Road
Suite 300
Des Plaines, 60018, United States of America
Paper Service

On behalf of U.S. International Trade Commission :

Yoncha Kundupoglu
OUII Investigative Attorney
500 E Street, S.W.
Washington, D.C. 20436
Internal Service

Service Date: January 11, 2022 PDF Generated on: January 11, 2022


US010023546B2

(12) **United States Patent**
Mollenkopf et al.

(10) **Patent No.: US 10,023,546 B2**
(45) **Date of Patent: Jul. 17, 2018**

(54) **ACESULFAME POTASSIUM COMPOSITIONS AND PROCESSES FOR PRODUCING SAME**

(71) Applicant: **CELANESE INTERNATIONAL CORPORATION**, Irving, TX (US)

(72) Inventors: **Christoph Mollenkopf**, Frankfurt am Main (DE); **Peter Groer**, Babenhausen (DE); **Arvind Yadav**, Hessen (IN)

(73) Assignee: **Celanese International Corporation**, Irving, TX (US)

(*) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 0 days.

(21) Appl. No.: **15/704,457**

(22) Filed: **Sep. 14, 2017**

(65) **Prior Publication Data**

US 2018/0079736 A1 Mar. 22, 2018

Related U.S. Application Data

(60) Provisional application No. 62/397,540, filed on Sep. 21, 2016.

(51) **Int. Cl.**

C07D 209/06	(2006.01)
A23L 27/30	(2016.01)
C07D 291/06	(2006.01)
C07C 235/74	(2006.01)

(52) **U.S. Cl.**

CPC ***C07D 291/06*** (2013.01); ***A23L 27/30*** (2016.08); ***C07C 235/74*** (2013.01); *A23V 2002/00* (2013.01)

(58) **Field of Classification Search**

CPC C07D 209/06; A23L 27/30
USPC 544/200
See application file for complete search history.

(56) **References Cited**

U.S. PATENT DOCUMENTS

4,563,521	A	1/1986	Clauss et al.
4,607,100	A	8/1986	Clauss et al.
4,638,063	A	1/1987	Clauss et al.
4,649,209	A	3/1987	Geering
4,695,629	A	9/1987	Clauss et al.
4,804,755	A	2/1989	Reuschling et al.
4,806,639	A	2/1989	Reuschling et al.
4,876,341	A	10/1989	Schiitz et al.
5,011,982	A	4/1991	Clauss et al.
5,084,180	A	1/1992	Boateng
5,103,046	A	4/1992	Clauss et al.
5,334,397	A	8/1994	Ream et al.
5,744,010	A	4/1998	Roscher et al.
5,808,159	A	9/1998	Giebeler
7,408,059	B2	8/2008	Kobayashi et al.
7,977,514	B2	7/2011	Peters et al.
8,182,756	B2	5/2012	Liu et al.
8,303,921	B2	11/2012	Brietzke et al.
8,309,048	B2	11/2012	Brietzke et al.
8,496,905	B2	7/2013	Brietzke et al.
8,658,830	B2	2/2014	Brietzke et al.
9,024,016	B2	5/2015	Bayer et al.
2003/0065172	A1	4/2003	Tian et al.
2003/0065218	A1	4/2003	Mollenkopf
2008/0076919	A1	3/2008	Liu et al.
2009/0318685	A1	12/2009	Saito et al.
2009/0318686	A1	12/2009	Saito et al.
2010/0274057	A1	10/2010	Peters et al.
2011/0256045	A1	10/2011	Brietzke et al.
2011/0256046	A1	10/2011	Brietzke et al.
2013/0062192	A1	3/2013	Brietzke et al.
2013/0331565	A1	12/2013	Bayer et al.
2018/0079733	A1	3/2018	Mollenkopf et al.
2018/0079734	A1	3/2018	Mollenkopf et al.
2018/0079735	A1	3/2018	Mollenkopf et al.

FOREIGN PATENT DOCUMENTS

CA	1273923	9/2009
CN	85104277	6/1985
CN	85104277 A	12/1986
CN	1883790	12/2006
CN	200949088	9/2007
CN	101124981	2/2008
CN	101124981 A	2/2008
CN	101148300	3/2008
CN	101157666	4/2008
CN	101787001	7/2010
CN	101787001 A	7/2010

(Continued)

OTHER PUBLICATIONS

Boehshar, Manfred & Burgard, Andreas. (2003). 5-Chloroacesulfame K—a characteristic indicator for application of the "sulfur trioxide" process in the manufacture of acesulfame K. Research Disclosure. 477036.

D. Mayer, et al., Acesulfame-K (Food Science and Technology), Jun. 28, 1991.

Linkies, et al., Synthesis, 1990, (5), pp. 405-406.

Suenaga, T., "Ethylene-amine salt recovery—by converting the hydrochloride into the sulphate, and reacting with ammonia in aq. Solvent to ppte. Ammonium sulphate", WPI/Thompson, vol. 1979, No. 16, XP 002598345 (See JP54032406).

Sunnett Brochure, "Acesulfame Potassium", Celanese, Apr. 2014.

Xiangsheng, et al., "Synthesis of Acesulfame Potassium", Fine Chemicals, vol. 13, 1996, pp. 1-15.

International Search Report received in the related International PCT Patent application No. PCT/US2007/051509, dated Nov. 9, 2017.

International Search Report received in the related International PCT Patent application No. PCT/US2007/051512, dated Nov. 9, 2017.

(Continued)

Primary Examiner — Kahsay Habte
(74) *Attorney, Agent, or Firm* — Dority & Manning, P.A.

(57) **ABSTRACT**

Improved processes for producing high purity acesulfame potassium. In one embodiment, the process comprises the steps of contacting a solvent, e.g., dichloromethane, and a cyclizing agent, e.g., sulfur trioxide, to form a cyclizing agent composition and reacting an acetoacetamide salt with the cyclizing agent in the composition to form a cyclic sulfur trioxide adduct. The contact time is less than 60 minutes. The process also comprises forming from the cyclic sulfur trioxide adduct composition a finished acesulfame potassium composition comprising non-chlorinated, e.g., non-chlorinated, acesulfame potassium and less than 35 wppm 5-halo acesulfame potassium, preferably less than 5 wppm.

30 Claims, 1 Drawing Sheet

(56) **References Cited**

FOREIGN PATENT DOCUMENTS

CN	101913898		12/2010
CN	201921689		8/2011
CN	102225333		10/2011
CN	102359926		2/2012
CN	102380226		3/2012
CN	102380266	A	3/2012
CN	202221403	U	5/2012
CN	102866042		1/2013
CN	103018368		4/2013
CN	103130743		6/2013
CN	103351294		10/2013
CN	103570592		2/2014
CN	103570592	A	2/2014
CN	103588728		2/2014
CN	103588728	A	2/2014
CN	103613566		3/2014
CN	103613566	A	3/2014
CN	103960558		8/2014
CN	104193625		12/2014
CN	104209052		12/2014
CN	104225956		12/2014
CN	104292181		1/2015
CN	104292181	A	1/2015
CN	204320227		5/2015
CN	105085160	A	11/2015
CN	105111166	A	12/2015
CN	105152446		12/2015
CN	105198778		12/2015
CN	106262665	A	1/2017
CN	106267879	A	1/2017
CN	106346138	A	1/2017
CN	106349009	A	1/2017
CN	106349190		1/2017
CN	106349191		1/2017
CN	106349300	A	1/2017
CN	106365952		2/2017
CN	106496159	A	3/2017
CN	206001191	U	3/2017
CN	206001201	U	3/2017
CN	206001439	U	3/2017
CN	206121215	U	4/2017
DE	1249262	B	10/1966
DE	1268141	B	5/1968
DE	3522470		1/1987
DE	3531357		3/1987
DE	3531357	A1	3/1987
DE	3545196		6/1987
EP	0155634	A2	9/1985
EP	0159516	A1	10/1985
EP	0215347	A1	3/1987
EP	0217024	A	4/1987
EP	0218076	A1	4/1987
JP	54032406		3/1979
JP	S 5432406	A	3/1979
WO	93/19055	A1	9/1993
WO	WO 2011/133468	A1	10/2011
WO	2013/182651	A1	12/2013

OTHER PUBLICATIONS

International Search Report received in the corresponding International PCT Patent application No. PCT/US2007/051507, dated Nov. 10, 2017.

International Search Report received in the related International PCT Patent application No. PCT/US2017/051514, dated Nov. 9, 2017.

ASTM E 313-05, "Standard Practice for Calculating Yellowness and Whiteness Indices from Instrumentally Measured Color Coordinates," Oct. 1, 2005, 6 pages.

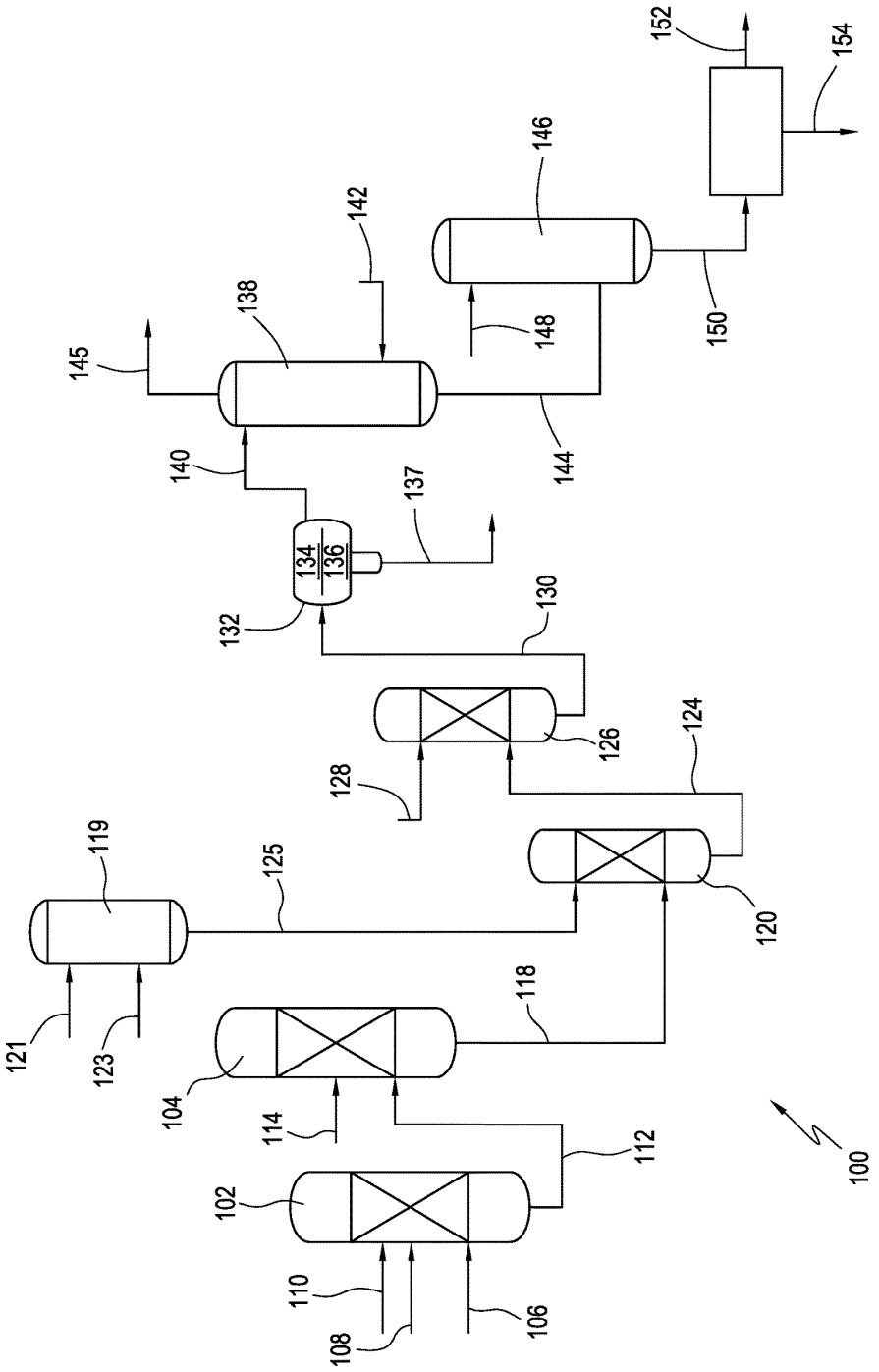
Opinion—"Re-evaluation of acesulfame K with reference to the previous SCF opinion of 1991," European Commission, Health & Consumer Protection Directorate-General, Directorate B—Scientific Health Opinions, Unit B3—Management of Scientific Committees II, Scientific Committee on Food, Mar. 13, 2000, 8 pages.

Official Journal of the European Communities, "Commission Direction 95/31/EC of Jul. 5, 1995 laying down specific criteria of purity concerning sweeteners for use in foodstuffs," Jul. 28, 1995, 19 pages.

Official Journal of the European Union, "Commission Direction 2008/60/EC of Jun. 17, 2008 laying down specific purity criteria concerning sweeteners for use in foodstuffs," Jun. 18, 2008, 40 pages.

Official Journal of the European Union, "Regulations—Commission Regulation (EU) No. 231/2012 of Mar. 9, 2012 laying down specifications for food additives listed in Annexes II and II to Regulation (EC) No. 1333/2008 of the European Parliament and of the Council," Mar. 22, 2012, 295 pages.

Product Information on "Acesulfame Potassium," 2001, 57th JECFA (joint FAO/WHO Expert Committee on Food Additives), FNP 52 Add 9, 2 pages.


152
154
146
142
138
148
150
145
144
140
137
134
136
132
130
126
124
128
119
125
120
118
121
123
104
114
112
100
102
110
108
106

ACESULFAME POTASSIUM COMPOSITIONS AND PROCESSES FOR PRODUCING SAME

CROSS-REFERENCE TO RELATED APPLICATION

This application claims priority to U.S. Provisional Patent Application No. 62/397,540, filed Sep. 21, 2016, the disclosure of which is incorporated herein by reference in its entirety.

FIELD OF INVENTION

The present invention relates generally to acesulfame potassium and to processes for producing acesulfame potassium. More specifically, the present invention relates to processes for producing high purity acesulfame potassium.

BACKGROUND OF THE INVENTION

Acesulfame potassium has an intense, sweet taste and has been used in many food-related applications as a sweetener. In conventional acesulfame potassium production processes, sulfamic acid and an amine, e.g., triethylamine, are reacted to form an amidosulfamic acid salt, such as a trialkyl ammonium amidosulfamic acid salt. The amidosulfamic acid salt is then reacted with diketene to form an acetoacetamide salt. The acetoacetamide salt may be cyclized, hydrolyzed, and neutralized to form acesulfame potassium. U.S. Pat. Nos. 5,744,010 and 9,024,016 disclose exemplary acesulfame potassium production processes.

Typically, the acetoacetamide salt intermediate is cyclized by reaction with sulfur trioxide in an inorganic or organic solvent to form a cyclic sulfur trioxide adduct. The solvent routinely utilized in this reaction is an organic solvent such as a halogenated, aliphatic hydrocarbon solvent, for example, dichloromethane. The adduct formed by this reaction is subsequently hydrolyzed and then neutralized with potassium hydroxide to form acesulfame potassium.

Acesulfame potassium and the intermediate compositions produced by conventional methods contain undesirable impurities, such as 5-chloro-acesulfame potassium. Limits for the content of various impurities are often set by governmental regulations or customer guidelines. Due to their similar chemical structures and properties, separation of 5-chloro-acesulfame potassium from the desired non-chlorinated acesulfame potassium, using standard purification procedures such as crystallization has proven difficult, resulting in consumer dissatisfaction and the failure to meet regulatory standards.

The need exists for an improved process for producing high purity acesulfame potassium compositions in which the formation of 5-chloro-acesulfame potassium during synthesis is reduced or eliminated.

All of the references discussed herein are hereby incorporated by reference.

SUMMARY OF THE INVENTION

The application discloses processes for producing a finished acesulfame potassium composition, the process comprising the steps of: contacting a solvent and a cyclizing agent to form a cyclizing agent composition, reacting an acetoacetamide salt with the cyclizing agent in the cyclizing agent composition to form a cyclic sulfur trioxide adduct, and forming from the cyclic sulfur trioxide adduct the finished acesulfame potassium composition comprising non-chlorinated acesulfame potassium and less than 35 wppm 5-chloro-acesulfame potassium, e.g., from 0.001 wppm to 2.7 wppm 5-chloro-acesulfame potassium. Contact time from the beginning of contacting step to the beginning of the reacting step is less than 60 minutes. The forming of the finished acesulfame potassium composition may comprise: hydrolyzing the cyclic sulfur trioxide adduct to form an acesulfame-H composition comprising acesulfame-H, neutralizing the acesulfame-H in the acesulfame-H composition to form a crude acesulfame potassium composition comprising non-chlorinated acesulfame potassium and less than 35 wppm 5-chloro-acesulfame potassium, and forming the finished acesulfame potassium composition from the crude acesulfame potassium composition. The finished acesulfame potassium composition may comprise from 0.001 wppm to 5 wppm 5-chloro-acesulfame potassium. In some cases, the contact time is less than 15 minutes and the crude acesulfame potassium composition comprises from 0.001 wppm to 5 wppm 5-chloro-acesulfame potassium and the finished acesulfame potassium composition comprises from 0.001 wppm to 5 wppm 5-chloro-acesulfame potassium. In one embodiment, the contact time is less than 5 minutes and the crude acesulfame potassium composition comprises from 0.001 wppm to 5 wppm 5-chloro-acesulfame potassium and the finished acesulfame potassium composition comprises from 0.001 wppm to 2.7 wppm 5-chloro-acesulfame potassium. The finished acesulfame potassium composition may comprise at least 90% by weight of the 5-chloro-acesulfame potassium present in the crude acesulfame potassium composition. In some case, the hydrolyzing comprises adding water to the cyclic sulfur trioxide adduct to form a hydrolysis reaction mixture, and wherein the temperature of the hydrolysis reaction mixture is maintained at a temperature ranging from −35° C. to 0° C. The finished acesulfame potassium composition may comprise from 0.001 wppm to 5 wppm organic impurities and/or from 0.001 wppm to 5 wppm heavy metals. Preferably, the process further comprises the steps of reacting sulfamic acid and an amine to form an amidosulfamic acid salt, and reacting the amidosulfamic acid salt and acetoacetylating agent to form the acetoacetamide salt. The cyclizing agent composition may comprise less than 1 wt % of compounds selected from chloromethyl chlorosulfate, methyl-bis-chlorosulfate, and mixtures thereof. The reacting is conducted for a cyclization reaction time, from the start of the reactant feed to the end of the reactant feed, less than 35 minutes. The weight ratio of solvent to cyclizing agent in the cyclizing agent composition may be at least 1:1. The processes may further comprise cooling the cyclizing agent composition to a temperature less than 15° C. Preferably, the cyclizing agent comprises sulfur trioxide and the solvent comprises dichloromethane. In one embodiment, the processes comprise the steps of: reacting sulfamic acid and triethylamine to form an amidosulfamic acid salt, reacting the amidosulfamic acid salt and diketene to form the acetoacetamide salt, contacting dichloromethane and a sulfur trioxide to form a cyclizing agent composition (optionally cooling the cyclizing agent composition to a temperature less than 15° C.), reacting the acetoacetamide salt with the sulfur trioxide in the cyclizing agent composition to form a cyclic sulfur trioxide adduct, hydrolyzing the cyclic sulfur trioxide adduct to form an acesulfame-H composition, and neutralizing the acesulfame-H to form the finished acesulfame potassium composition comprising non-chlorinated acesulfame potassium and less than 10 wppm 5-chloro-acesulfame potassium, and contact time from the beginning of step (a) to the beginning

of step (b) may be less than 10 minutes. The application also describes crude, intermediate, and finished acesulfame potassium composition produced by the processes described herein, e.g., a finished acesulfame potassium composition comprising non-chlorinated acesulfame potassium, from 0.001 wppm to 2.7 wppm 5-chloro acesulfame potassium, and from 0.001 wppm to 5 wppm heavy metals.

BRIEF DESCRIPTION OF THE DRAWINGS

The invention is described in detail below with reference to the appended drawing.

FIG. **1** is a process flow sheet of an acesulfame potassium production process in accordance with one embodiment of the present invention.

DETAILED DESCRIPTION OF THE INVENTION

Introduction

Conventional processes for producing acesulfame potassium involve reacting sulfamic acid and an amine in the presence of acetic acid to form an amidosulfamic acid salt. The amidosulfamic acid salt is then reacted with an acetoacetylating agent, e.g., diketene, to form an acetoacetamide salt. The acetoacetamide salt is reacted with a cyclizing agent, e.g., sulfur trioxide, to form a cyclic sulfur trioxide adduct. The cyclic sulfur trioxide adduct is then hydrolyzed and neutralized via conventional means to form a crude acesulfame potassium composition comprising acesulfame potassium. This composition is phase separated into aqueous and organic phases. Most of the acesulfame potassium separates into the aqueous phase. As used herein, the term "crude acesulfame potassium composition" refers to the initial product of the neutralization reaction or to the aqueous phase that is formed from the phase separation step (without any further purification). The crude acesulfame potassium composition comprises at least 5 wt % acesulfame potassium. The crude acesulfame potassium composition may be optionally treated to form an "intermediate acesulfame potassium composition" and/or a "finished acesulfame potassium composition," which are discussed below.

Conventional acesulfame potassium compositions have been shown to comprise several undesirable impurities, among them 5-chloro-acesulfame potassium and acetoacetamide. Content limits for these compounds in the finished acesulfame potassium composition are often determined by industry purity standards and/or by standards established for particular end use products that utilize acesulfame potassium as a sweetener. In some cases, limits for these impurities are determined by governmental regulations. For most applications, high acesulfame potassium purity levels are preferred. Because the chemical structure of 5-chloro-acesulfame potassium is similar to that of non-chlorinated acesulfame potassium, separation of 5-chloro-acesulfame potassium using standard purification procedures such as crystallization has proven difficult.

Without being bound by theory, it has now been discovered that the reaction of the cyclizing agent with the acetoacetamide salt to form the cyclic sulfur trioxide adduct may also involve side reactions that form the 5-chloro-acesulfame potassium impurity.

The use of specific reaction parameters, however, may advantageously reduce or eliminate 5-chloro-acesulfame potassium formation or the formation of its precursor, 5-chloro-acesulfame-H. In particular, it has now been discovered that limiting contact time, as discussed below, surprisingly reduces or eliminates 5-chloro-acesulfame potassium formation in the crude, intermediate, and/or finished acesulfame potassium compositions. In addition, the reduced impurity levels in these acesulfame potassium compositions reduce or eliminate the need for additional purification steps, resulting in overall improved process efficiency.

It is postulated that the contacting of the cyclizing agent, the solvent, and optionally other components may lead to the formation of chlorine/chloride-containing compounds. Exemplary cyclizing agent/solvent reaction products include halogen-containing compounds such as chlorine/chloride-containing compounds, e.g., chlorosulfates. These compounds, in turn, may react to chlorinate the acesulfame precursor acid, acesulfame-H, sometimes referred to as sweetener acid, or its precursors, e.g., acetoacetamide-N-sulfonate. By limiting the contact time, lower amounts of chlorine/chloride-containing compounds are formed, e.g., chlorosulfates, are formed (as compared to the amount formed when longer contact times are employed). That is, shorter contact times have now been shown to retard the formation of chlorine/chloride-containing compounds, e.g., chlorosulfates. As a result of the shorter contact times, in one embodiment, the cyclizing agent composition may have a low chlorine/chloride-containing compound content, e.g., a low chlorosulfate content, as discussed herein. The reduction or elimination of chlorine/chloride-containing compounds directly leads to the formation of higher purity crude acesulfame potassium compositions discussed herein, thereby simplifying subsequent treatment operations for forming the intermediate or finished acesulfame potassium compositions. The process also advantageously leads to the formation of intermediate and finished acesulfame potassium compositions having low 5-chloro-acesulfame potassium content.

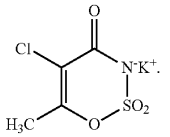
Additional specific terms that are used herein are now defined. "Contact time," as used herein, refers to the time period that the solvent contacts the cyclizing agent before formation of the cyclic sulfur trioxide adduct. Thus, contact time begins when at least some of the solvent contacts at least some the cyclizing agent to form the cyclizing agent/solvent mixture ("cyclizing agent composition"), and contact time ends when the acetoacetamide salt first contacts the cyclizing agent in the cyclizing agent composition.

"Residence time," as used herein, refers to the time period that a composition (or stream) to be treated, e.g., a crude acesulfame potassium composition, remains in a particular treatment operation. Residence time begins when the composition to be treated enters the treatment operation, and residence time ends when the resultant compositions (formed via the treatment) exit the treatment operation. As one particular example, residence time for a concentrating operation, e.g., evaporation, refers to the time from when a crude acesulfame potassium composition enters the evaporator until the intermediate acesulfame potassium composition exits the evaporator. As another example, residence time for a separating operation, e.g., crystallization, refers to the time from when a crude acesulfame potassium composition enters the crystallizer until the intermediate acesulfame potassium composition exits the crystallizer.

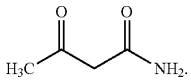
"Cyclization reaction time," as used herein, refers to the time from the start of the acetoacetamide salt feed to the termination of the acetoacetamide salt feed. In some cases, if indicated, the cyclization reaction time may include addi-

tional time past the termination of the acetoacetamide salt feed, e.g., an extra 5 minutes or an extra minute.

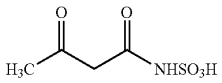
"5-chloro-acesulfame potassium," as used herein, refers to the following molecule:



"Acetoacetamide," as used herein, refers to the following molecule:



"Acetoacetamide-N-sulfonic acid" as used herein, refers to the molecule shown below. In some cases, acetoacetamide-N-sulfonic acid may be a degradation product of acesulfame potassium or acesulfame-H. The term "acetoacetamide-N-sulfonic acid," as used herein, also includes salts of acetoacetamide-N-sulfamic acid, e.g., potassium, sodium, and other alkali metal salts.



An "intermediate acesulfame potassium composition" refers to a composition resulting from the concentrating of the crude acesulfame potassium composition, e.g., the removal of water from the crude acesulfame potassium composition. The intermediate acesulfame potassium composition comprises at least 10 wt % acesulfame potassium, based on the total weight of the intermediate acesulfame potassium composition, and has an acesulfame potassium weight percentage that is higher than that of the crude acesulfame potassium composition.

A "finished acesulfame potassium composition" refers to a composition (preferably directly) resulting from the separating, e.g., crystallizing and/or filtering, of the intermediate acesulfame potassium composition, e.g. no further process steps are preferably conducted after the separating of the intermediate acesulfame potassium composition in order to obtain the finished acesulfame potassium composition. The finished acesulfame potassium composition comprises at least 15 wt % acesulfame potassium, based on the total weight percentage of the finished acesulfame potassium composition, and has an acesulfame potassium weight percentage that is higher than that of the intermediate acesulfame potassium composition.

"Wppm" and "wppb," as used herein, mean weight parts per million or weight parts per billion, respectively. These are based on the total weight of the respective composition, e.g., the total weight of the entire crude acesulfame potassium composition or the entire finished acesulfame potassium composition.

Acesulfame Potassium Formation (Contact Time)

Processes for producing acesulfame potassium exhibiting high purity levels are described herein. In one embodiment, the process comprises the steps of contacting a solvent and a cyclizing agent to form a cyclizing agent composition and reacting an acetoacetamide salt with the cyclizing agent (in the cyclizing agent composition) to form a cyclic sulfur trioxide adduct. Importantly, the contact time is less than 60 minutes. The process also comprises forming a finished acesulfame potassium composition from the cyclic sulfur trioxide adduct composition.

The contacting of the solvent and the cyclizing agent is contemplated broadly. In some embodiments, the contacting methods include the addition of solvent to cyclizing agent, the addition of cyclizing agent to solvent. The components may be fed, e.g., simultaneously fed to a vessel. Addition/mixing and/or co-feeding (optionally simultaneous) of these components are contemplated.

The reaction of the acetoacetamide salt and the cyclizing agent may be conducted by contacting the two reactants. The reactants may be fed, e.g., simultaneously fed to a vessel. In one embodiment, the acetoacetamide salt may be added to the cyclizing agent in the cyclizing agent composition. The cyclizing agent in the cyclizing agent composition may be added to the acetoacetamide salt. Addition/mixing and/or co-feeding (optionally simultaneous) of the reactants are also contemplated. In one embodiment, the cyclizing agent composition may be contained in a vessel and the acetoacetamide salt may be added to the cyclizing agent composition, e.g., added drop-wise to the cyclizing agent composition.

In some embodiments, contact time is less than 60 minutes, e.g., less than 45 minutes, less than 30 minutes, less than 15 minutes, less than 10 minutes, less than 8 minutes, less than 5 minutes, less than 3 minutes, or less than 1 minute. In one embodiment, the solvent and cyclizing agent are mixed and immediately reacted with the acetoacetamide salt. In terms of ranges, contact time may range from 1 second to 60 minutes, e.g., from 1 second to 45 minutes, from 1 second to 30 minutes, from 1 second to 15 minutes, from 1 second to 10 minutes, from 1 minute to 45 minutes, from 1 minute to 30 minutes, from 1 minute to 15 minutes, from 1 minute to 10 minutes, from 10 seconds to 45 minutes, from 10 seconds to 30 minutes, from 30 seconds to 30 minutes, from 1 minute to 10 minutes, from 3 minutes to 10 minutes, or from 5 minutes to 10 minutes. Contact time may be at least 1 second, e.g., at least 5 seconds, at least 30 seconds, at least 1 minute, at least 5 minutes, at least 10 minutes, at least 15 minutes, or at least 30 minutes.

By limiting the contact time as discussed herein, fewer cyclizing agent/solvent reaction products, e.g., chlorosulfates, are formed. The cyclizing agent composition, for example, may have a low cyclizing agent/solvent reaction product content, e.g., a low chlorosulfate content. For example, the cyclizing agent composition may comprise less than 1 wt % cyclizing agent/solvent reaction product, e.g., less than 0.75 wt %, less than 0.5 wt %, less than 0.25 wt %, less than 0.1 wt %, less than 0.05 wt %, or less than 0.01 wt %. In terms of ranges, the cyclizing agent composition may comprise from 1 ppm to 1 wt % cyclizing agent/solvent reaction products, e.g., from 10 ppm to 1 wt %, from 10 ppm to 0.75 wt %, from 10 ppm to 0.5 wt %, from 10 ppm to 0.25 wt %, from 100 ppm to 0.75 wt %, from 100 ppm to 0.5 wt %, or from 100 ppm to 0.25 wt %. These ranges and limits apply to cyclizing agent/solvent reaction products generally and to specific reaction products generally, e.g., chloromethyl chlorosulfate, methyl-bis-chlorosulfate, and combinations thereof.

Exemplary chlorosulfates include chloromethyl chlorosulfate and methyl-bis-chlorosulfate. These reaction products may be formed when a chlorine-containing solvent is employed. In one embodiment, the cyclizing agent composition comprises less than 1 wt % chloromethyl chlorosulfate and/or methyl-bis-chlorosulfate, e.g., less than 0.75 wt %, less than 0.5 wt %, less than 0.25 wt %, less than 0.1 wt %, less than 0.05 wt %, or less than 0.01 wt %. In one embodiment, the cyclizing agent composition comprises less than 1 wt % chloromethyl chlorosulfate, e.g., less than 0.75 wt %, less than 0.5 wt %, less than 0.25 wt %, less than 0.1 wt %, less than 0.05 wt %, or less than 0.01 wt %. In one embodiment, the cyclizing agent composition comprises less than 1 wt % methyl-bis-chlorosulfate, e.g., less than 0.75 wt %, less than 0.5 wt %, less than 0.25 wt %, less than 0.1 wt %, less than 0.05 wt %, or less than 0.01 wt %.

In one embodiment, the solvent and cyclizing agent are combined in a first vessel, e.g., a first reactor, to form a cyclizing agent composition, which optionally may be cooled. The cyclizing agent composition may then be added to the acetoacetamide salt in a second reactor. In one embodiment, the first vessel is chilled, e.g., to temperature below 35° C., prior to combining the solvent and cyclizing agent. In some cases, the cyclizing agent and the solvent are cooled individually and then fed to the reaction with the acetoacetamide salt, optionally followed by additional cooling. In one embodiment, the first vessel itself is chilled, e.g., to a temperature below 15° C., prior to contacting the solvent and cyclizing agent, which leads to the cooling of the solvent and the cyclization that may be added to the first vessel. In some cases, the cyclizing agent and the solvent are cooled individually and then combined and fed to the reaction with the acetoacetamide salt.

In some cases, the process comprises the steps of providing a cyclic sulfur trioxide adduct composition comprising less than 1 wt % cyclizing agent/solvent reaction products, e.g., chloromethyl chlorosulfate and/or methyl-bis-chlorosulfate, and forming the acesulfame potassium composition from the cyclic sulfur trioxide adduct composition. The providing of the cyclic sulfur trioxide adduct composition may vary widely as long as the cyclic sulfur trioxide adduct composition has the required cyclizing agent/solvent reaction product content. The cyclic sulfur trioxide adduct composition optionally is formed using any of the methods described herein.

In some embodiments, the cyclizing agent composition is provided at a low temperature and/or is cooled to yield a cooled cyclizing agent composition having a low temperature. The cooling or the providing of the low temperature cyclizing agent composition may be achieved through any of a variety of different cooling techniques. For example, the cooling step may be achieved by using one or more heat exchangers, refrigeration units, air cooling units, water cooling units, or a cooling medium, such as liquid nitrogen or other cryogenics. If heat exchangers are employed, a water/glycol mixture is a preferable exchange medium, with brine being a suitable alternative.

In some embodiments, the cyclizing agent composition is provided at or is cooled to a temperature less than 15° C., e.g., less than 12° C., less than 11° C., less than 10° C., less than 8° C., less than 5° C., less than 3° C., less than 1° C., or less than 0° C. In terms of ranges, the cyclization agent composition is cooled to a temperature ranging from −20° C. to 15° C., e.g., from −15° C. to 15° C., from −10° C. to 12° C., from −8° C. to 10° C., or −8° C. to 5° C. In some embodiments, the cooling step reduces the temperature of the cyclizing agent composition (as provided), e.g., by at least 2° C., at least 3° C., at least 5° C., at least 10° C., at least 15° C., at least 20° C., or at least 25° C.

In one embodiment, only the cyclizing agent (e.g., without solvent) is cooled, and then the cooled cyclizing agent is mixed with the solvent to form the cyclizing agent composition, which is then reacted with the acetoacetamide salt. That is, in some cases, the solvent (if present) may not be cooled in the same manner as the cyclizing agent is cooled. In other embodiments, the solvent is cooled prior to being mixed with the cyclizing agent to form the cyclizing agent composition, optionally followed by additional cooling of the resulting cyclizing agent composition.

In some cases, the cooling is implemented via multiple cooling steps. For example, the solvent may be cooled to a first temperature, then combined with the cyclizing agent to form the cyclizing agent composition, which is then further cooled to a second temperature, which is less than the first temperature. In some embodiments, the cyclizing agent is cooled to a first temperature, the solvent is cooled to a second temperature, and the cooled cyclizing agent and the cooled solvent are combined and optionally cooled to a third temperature, which is less than the first and second temperatures. These cooling schemes are merely exemplary and are not intended to limit the scope of the cooling step.

It has also been discovered that if cyclization reaction time is minimized, the formation of impurities, e.g., organic impurities, such as 5-chloro-acesulfame potassium, is reduced or eliminated. In some embodiments, the cyclization reaction is conducted for a cyclization reaction time, less than 35 minutes, e.g., less than 30 minutes, less than 25 minutes, less than 20 minutes, less than 15 minutes, or less than 10 minutes. In terms of ranges, the cyclization reaction may be conducted for a cyclization reaction time ranging from 1 second to 35 minutes, e.g., from 10 seconds to 25 minutes, from 30 seconds to 15 minutes, or from 1 minute to 10 minutes.

The cyclic sulfur trioxide adduct may be subjected to one or more steps to form the finished acesulfame potassium composition. In some cases, the formation of the finished acesulfame potassium composition comprises the steps of hydrolyzing (at least some of) the cyclic sulfur trioxide adduct to form an acesulfame-H composition comprising acesulfame-H and neutralizing the acesulfame-H in the acesulfame-H composition to form a crude acesulfame potassium composition.

Crude acesulfame compositions may be treated to form intermediate acesulfame potassium compositions and (subsequently) finished acesulfame compositions, and this treatment operation may include one or more concentrating or separating operations.

For example, the treatment operation may comprise concentrating the crude acesulfame potassium composition to form a water stream and an intermediate acesulfame potassium composition and then separating the intermediate acesulfame potassium composition to form the finished acesulfame potassium composition comprising acesulfame potassium, e.g., via filtration and/or crystallization.

Acesulfame Potassium Compositions

The crude acesulfame potassium composition is formed by hydrolyzing the cyclic sulfur trioxide adduct to form an acesulfame-H composition and neutralizing the acesulfame-H in the acesulfame-H composition to form the crude acesulfame potassium composition, as discussed herein. The product of the neutralization step is phase separated into aqueous and organic phases. The crude acesulfame potassium composition may be obtained from the aqueous phase (without any further purification). The crude acesulfame

potassium composition preferably comprises a mixture of acesulfame potassium, e.g., non-chlorinated acesulfame potassium, and less than 35 wppm 5-chloro-acesulfame potassium, e.g., less than 30 wppm, less than 25 wppm, less than 20 wppm, less than 15 wppm, less than 12 wppm, less than 10 wppm, less than 7 wppm, less than 5 wppm, less than 3 wppm, or less than 1 wppm. In some cases the crude acesulfame potassium composition is free of 5-chloro-acesulfame potassium, e.g., substantially free of 5-chloro-acesulfame potassium (undetectable). In terms of ranges, the crude acesulfame potassium composition may comprise from 1 wppb to 35 wppm 5-chloro-acesulfame potassium, e.g., from 1 wppb to 20 wppm, from 1 wppb to 10 wppm, from 1 wppb to 5 wppm, from 1 wppb to 2.7 wppm, from 10 wppb to 20 wppm, from 10 wppb to 19 wppm, from 10 wppb to 15 wppm, from 10 wppb to 12 wppm, from 10 wppb to 10 wppm, from 10 wppb to 5 wppm, from 100 wppb to 15 wppm, from 100 wppb to 10 wppm, or from 100 wppb to 5 wppm.

The finished acesulfame potassium compositions, which are typically suitable for end consumer usage, are formed by treating the crude acesulfame potassium composition to remove impurities, as discussed herein. This finished acesulfame potassium composition preferably comprises a mixture of acesulfame potassium, e.g., non-chlorinated acesulfame potassium, and less than 35 wppm 5-chloro-acesulfame potassium, e.g., less than 30 wppm, less than 25 wppm, less than 20 wppm, less than 15 wppm, less than 12 wppm, less than 10 wppm, less than 7 wppm, less than 5 wppm, less than 3 wppm, or less than 1 wppm. In some cases the finished acesulfame potassium composition is free of 5-chloro-acesulfame potassium, e.g., substantially free of 5-chloro-acesulfame potassium (undetectable). In terms of ranges, the finished acesulfame potassium composition may comprise from 1 wppb to 35 wppm 5-chloro-acesulfame potassium, e.g., from 1 wppb to 20 wppm, from 1 wppb to 10 wppm, from 1 wppb to 5 wppm, from 1 wppb to 2.7 wppm, from 10 wppb to 20 wppm, from 10 wppb to 19 wppm, from 10 wppb to 15 wppm, from 10 wppb to 12 wppm, from 10 wppb to 10 wppm, from 10 wppb to 5 wppm, from 100 wppb to 15 wppm, from 100 wppb to 10 wppm, or from 100 wppb to 5 wppm. The shorter contact times reduce or eliminate 5-chloro-acesulfame potassium formation, resulting in both a crude acesulfame potassium composition and a finished acesulfame potassium composition having low 5-chloro-acesulfame potassium content.

In some embodiments, the finished acesulfame potassium compositions comprise acesulfame potassium and less than 33 wppm acetoacetamide, e.g., less than 32 wppm, less than 30 wppm, less than 25 wppm, less than 20 wppm, less than 15 wppm, less than 12 wppm, less than 10 wppm, less than 7 wppm, less than 5 wppm, less than 3 wppm, less than 1 wppm, less than 0.8 wppm, less than 0.5 wppm, or less than 0.3 wppm. In some cases the finished acesulfame potassium composition is free of acetoacetamide, e.g., substantially free of acetoacetamide (undetectable). In terms of ranges, the finished acesulfame potassium composition may comprise from 1 wppb to 33 wppm acetoacetamide, e.g., from 10 wppb to 32 wppm, from 10 wppb to 25 wppm, from 10 wppb to 15 wppm, from 10 wppb to 12 wppm, from 10 wppb to 10 wppm, from 10 wppb to 7 wppm, from 10 wppb to 5 wppm, from 10 wppb to 3 wppm, from 100 wppb to 15 wppm, from 100 wppb to 10 wppm, or from 100 wppb to 5 wppm. In some cases, acetoacetamide-N-sulfonic acid may also be present in the finished acesulfame potassium compositions in the aforementioned amounts. These impurities may be formed by side reactions and degradation of the acesulfame potassium and acesulfame-H molecules, e.g., during treatment of the specific crude acesulfame potassium compositions discussed herein.

The 5-chloro-acesulfame potassium content may be measured in the crude and/or finished acesulfame potassium compositions (as well as any intermediate compositions) via high performance liquid chromatography (HPLC) analysis, based on European Pharmacopoeia guidelines (2017), based on European Pharmacopoeia guidelines for thin layer chromatography (2017) and adapted for HPLC. A particular measurement scenario utilizes an LC Systems HPLC unit from Shimadzu having a CBM-20 Shimadzu controller and being equipped with a CC 250/4.6 Nucleodur 100-3 C18 ec (250×4.6 mm) MACHEREY NAGEL column. A Shimadzu SPD-M20A photodiode array detector can be used for detection (at 234 nm wavelength). Analysis may be performed at 23° C. column temperature. As an eluent solution, an aqueous solution of tetra butyl ammonium hydrogen sulfate (optionally at 3.4 g/L and at 60% of the total solution) and acetonitrile (optionally at 300 mL/L and at 40% of the total solution) may be employed. Elution may be isocratic. The overall flow rate of total eluent may be approximately 1 mL/min. The data collection and calculations may be performed using Lab Solution software from Shimadzu.

The acetoacetamide-N-sulfonic acid and/or the acetoacetamide content may be measured in the crude, intermediate, or finished acesulfame potassium compositions via HPLC analysis, based on European Pharmacopoeia guidelines for thin layer chromatography (2017) and adapted for HPLC. A particular measurement scenario utilizes an LC Systems HPLC unit from Shimadzu having a CBM-20 Shimadzu controller and being equipped with an IonPac NS1 ((5 μm) 150×4 mm) analytical column and an IonPac NG1 guard column (35×4.0 mm). A Shimadzu SPD-M20A photodiode array detector can be used for detection (at 270 nm and 280 nm wavelength). Analysis may be performed at 23° C. column temperature. As a first eluent solution, an aqueous mixture of tetra butyl ammonium hydrogen sulfate (3.4 g/L), acetonitrile (300 mL/L), and potassium hydroxide (0.89 g/L) may be employed; as a second eluent solution, an aqueous mixture of tetra butyl ammonium hydrogen sulfate (3.4 g/L) and potassium hydroxide (0.89 g/L) may be employed. Elution may be conducted in gradient mode according to the following second eluent flow profile:

0 to 3 minutes: constant 80% (v/v)
3 to 6 minutes: linear reduction to 50% (v/v)
6 to 15 minutes: constant at 50% (v/v)
15 to 18 minutes: linear reduction to 0%
18 to 22 minutes: constant at 0%
22 to 24 minutes: linear increase to 80% (v/v)
24 to 35 minutes constant at 80% (v/v).

Overall flow rate of eluent may be approximately 1.2 mL/min. The data collection and calculations may be performed using Lab Solution software from Shimadzu.

As noted above, the crude acesulfame potassium composition is formed by the aforementioned contacting of the solvent and the cyclizing agent to form a cyclizing agent composition; cyclic sulfur trioxide adduct composition formation reaction, and forming from the cyclic sulfur trioxide adduct the finished acesulfame potassium composition (for example via hydrolysis, neutralization, and treatment). In preferred embodiments, the contact time may be less than 60 minutes, e.g., less than 45 minutes, less than 30 minutes, less than 15 minutes, less than 10 minutes, less than 8 minutes, less than 5 minutes, less than 3 minutes, or less than 1 minute (optionally ranging from 1 second to 60 minutes, e.g., from 1 second to 45 minutes, from 1 second to 30 minutes, from

1 second to 15 minutes, from 1 second to 10 minutes, from 1 minute to 45 minutes, from 1 minute to 30 minutes, from 1 minute to 15 minutes, from 1 minute to 10 minutes, from 10 seconds to 45 minutes, from 10 seconds to 30 minutes, from 30 seconds to 30 minutes, from 1 minute to 10 minutes, from 3 minutes to 10 minutes, or from 5 minutes to 10 minutes); the crude acesulfame potassium composition may comprise from 1 wppb to 35 wppm 5-chloro-acesulfame potassium, e.g., from 1 wppb to 20 wppm, from 1 wppb to 10 wppm, from 1 wppb to 5 wppm, from 1 wppb to 2.7 wppm, from 10 wppb to 20 wppm, from 10 wppb to 19 wppm, from 10 wppb to 15 wppm, from 10 wppb to 12 wppm, from 10 wppb to 10 wppm, from 10 wppb to 5 wppm, from 100 wppb to 15 wppm, from 100 wppb to 10 wppm, or from 100 wppb to 5 wppm (optionally less than 35 wppm 5-chloro-acesulfame potassium, e.g., less than 30 wppm, less than 25 wppm, less than 20 wppm, less than 15 wppm, less than 12 wppm, less than 10 wppm, less than 7 wppm, less than 5 wppm, less than 3 wppm, or less than 1 wppm); and the finished acesulfame potassium composition may comprise from 1 wppb to 35 wppm 5-chloro-acesulfame potassium, e.g., from 1 wppb to 20 wppm, from 1 wppb to 10 wppm, from 1 wppb to 5 wppm, from 1 wppb to 2.7 wppm, from 10 wppb to 20 wppm, from 10 wppb to 19 wppm, from 10 wppb to 15 wppm, from 10 wppb to 12 wppm, from 10 wppb to 10 wppm, from 10 wppb to 5 wppm, from 100 wppb to 15 wppm, from 100 wppb to 10 wppm, or from 100 wppb to 5 wppm (optionally less than 35 wppm 5-chloro-acesulfame potassium, e.g., less than 30 wppm, less than 25 wppm, less than 20 wppm, less than 15 wppm, less than 12 wppm, less than 10 wppm, less than 7 wppm, less than 5 wppm, less than 3 wppm, or less than 1 wppm).

In a particular embodiment, the contact time is less than 15 minutes, the crude acesulfame potassium composition comprises from 0.001 wppm to 5 wppm 5-chloro-acesulfame potassium, and the finished acesulfame potassium composition comprises from 0.001 wppm to 5 wppm 5-chloro-acesulfame potassium.

In another particular embodiment, the contact time is less than 5 minutes, the crude acesulfame potassium composition comprises from 0.001 wppm to 5 wppm 5-chloro-acesulfame potassium, and the finished acesulfame potassium composition comprises from 0.001 wppm to 2.7 wppm 5-chloro-acesulfame potassium.

In another particular embodiment, the contact time ranges from 1 second to 10 minutes, the crude acesulfame potassium composition comprises from 1 wppb to 35 wppm 5-chloro-acesulfame potassium, and the finished acesulfame potassium composition comprises from 1 wppb to 35 wppm 5-chloro-acesulfame potassium.

In another particular embodiment, the contact time ranges from 1 second to 10 minutes, the crude acesulfame potassium composition comprises from 1 wppb to 5 wppm 5-chloro-acesulfame potassium, and the finished acesulfame potassium composition comprises from 1 wppb to 5 wppm 5-chloro-acesulfame potassium.

In another particular embodiment, the contact time ranges from 1 second to 30 minutes, the crude acesulfame potassium composition comprises from 10 wppb to 10 wppm 5-chloro-acesulfame potassium, and the finished acesulfame potassium composition comprises from 10 wppb to 10 wppm 5-chloro-acesulfame potassium.

The acesulfame potassium compositions (crude and/or finished) may, in some cases, comprise other impurities. Exemplary impurities include, inter alia, acetoacetamide, acetoacetamidesulfonate, and acetoacetamide-N-sulfonic acid. The acesulfame potassium compositions (crude and/or finished) also may comprise heavy metals. The organic impurities and/or heavy metals may be present in an amount ranging from 1 wppb to 25 wppm, based on the total weight of the respective acesulfame potassium composition, crude or finished, e.g., from 100 wppb to 20 wppm, from 100 wppb to 15 wppm, from 500 wppb to 10 wppm, or from 1 wppm to 5 wppm. Heavy metals are defined as metals with relatively high densities, e.g., greater than 3 g/cm^3 or greater than 7 g/cm^3. Exemplary heavy metals include lead and mercury. In some cases, the crude or finished acesulfame potassium composition may comprise mercury in an amount ranging from 1 wppb to 25 wppm, e.g., from 100 wppb to 20 wppm, from 100 wppb to 15 wppm, from 500 wppb to 10 wppm, or from 1 wppm to 5 wppm. In terms of limits, the crude or finished acesulfame potassium composition may comprise less than 25 wppm mercury, e.g., less than 20 wppm, less than 15 wppm, less than 10 wppm, or less than 5 wppm. In some cases, the crude or finished acesulfame potassium composition may comprise lead in an amount ranging from 1 wppb to 25 wppm, e.g., from 100 wppb to 20 wppm, from 100 wppb to 15 wppm, from 500 wppb to 10 wppm, or from 1 wppm to 5 wppm. In terms of limits, the crude or finished acesulfame potassium composition may comprise less than 25 wppm lead, e.g., less than 20 wppm, less than 15 wppm, less than 10 wppm, or less than 5 wppm. In some cases, when potassium hydroxide is formed via a membrane process, the resultant crude or finished acesulfame potassium composition may have very low levels of mercury, if any, e.g., less than 10 wppm, less than 5 wppm, less than 3 wppm, less than 1 wppm, less than 500 wppb, or less than 100 wppb.

In some embodiments, the acesulfame potassium compositions (crude, intermediate, and/or finished) may comprise acetoacetamide-N-sulfonic acid, e.g., less than 37 wppm acetoacetamide-N-sulfonic acid, e.g., less than 35 wppm, less than 30 wppm, less than 25 wppm, less than 20 wppm, less than 15 wppm, less than 12 wppm, less than 10 wppm, less than 7 wppm, less than 5 wppm, less than 3 wppm, less than 1 wppm, less than 0.8 wppm, less than 0.5 wppm, or less than 0.3 wppm. In some cases the finished acesulfame potassium composition is substantially free of acetoacetamide-N-sulfonic acid, e.g., free of acetoacetamide-N-sulfonic acid. In terms of ranges, the finished acesulfame potassium composition may comprise from 1 wppb to 37 wppm acetoacetamide-N-sulfonic acid, e.g., from 10 wppb to 35 wppm, from 10 wppb to 25 wppm, from 10 wppb to 15 wppm, from 10 wppb to 12 wppm, from 10 wppb to 10 wppm, from 10 wppb to 7 wppm, from 10 wppb to 5 wppm, from 10 wppb to 3 wppm, from 100 wppb to 15 wppm, from 100 wppb to 10 wppm, or from 100 wppb to 5 wppm. Acetoacetamide-N-sulfonic acid may be formed in side reactions. The use of the aforementioned temperature (and optionally contact time) parameters also provide for low amounts of acetoacetamide-N-sulfonic acid.

In some embodiments, the crude acesulfame potassium composition is treated to achieve the finished acesulfame potassium composition. In some cases, however, treatment steps may not provide for removal of 5-chloro-acesulfame potassium, perhaps due to the chemical similarities of 5-chloro-acesulfame potassium and acesulfame potassium. Surprisingly, the use of the process steps disclosed herein advantageously provides for the reduction or elimination of impurities during the reaction scheme, before purification of the crude acesulfame potassium composition. Accordingly, the need to rely on purification of the crude acesulfame potassium composition to remove 5-chloro-acesulfame

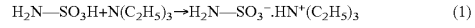
potassium is beneficially reduced. In some embodiments, the acesulfame potassium compositions (crude and/or finished) comprise at least 90% of the 5-chloro-acesulfame potassium present the crude acesulfame potassium composition, e.g., at least 93%, at least 95%, or at least 99%.

Intermediate Reaction Parameters

The reactions for production of high purity acesulfame potassium are described in more detail as follows.

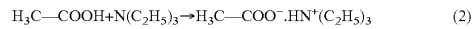
Amidosulfamic Acid Salt Formation Reaction

In a first reaction step, sulfamic acid and an amine are reacted to form sulfamic acid salt. An exemplary reaction scheme that employs triethylamine as the amine and yields triethyl ammonium sulfamic acid salt is shown in reaction (1), below.



$$H_2N—SO_3H+N(C_2H_5)_3 \rightarrow H_2N—SO_3^-.HN^+(C_2H_5)_3 \quad (1)$$

Acetic acid is also present in the first reaction mixture and reacts with the amine, e.g., triethylamine, to form an ammonium acetate, e.g., triethylammonium acetate, as shown in reaction (2), below.



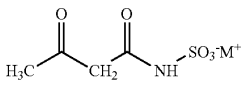
$$H_3C—COOH+N(C_2H_5)_3 \rightarrow H_3C—COO^-.HN^+(C_2H_5)_3 \quad (2)$$

The amine employed in these reactions may vary widely. Preferably, the amine comprises triethylamine. In one embodiment, the amine may be selected from the group consisting of trimethylamine, diethylpropylamine, tri-n-propylamine, triisopropylamine, ethyldiisopropylamine, tri-n-butylamine, triisobutylamine, tricyclohexylamine, ethyldicyclohexylamine, N,N-dimethylaniline, N,N-diethylaniline, benzyldimethylamine, pyridine, substituted pyridines such as picoline, lutidine, cholidine or methylethylpyridine, N-methylpiperidine, N-ethylpiperidine, N-methylmorpholine, N,N-dimethylpiperazine, 1,5-diazabicyclo[4.3.0]-non-5-en, 1,8-diazabicyclo-[5.4.0]-undec-7-en, 1,4-diazabicyclooctane, tetramethylhexamethylendiamine, tetramethylethylendiamine, tetramethylpropylendiamine, tetramethylbutylendiamine, 1,2-dimorpholylethan, pentamethyldiethyltriamine, pentaethyldiethylentriamine, pentamethyldipropylentriamine, tetramethyldiaminomethane, tetrapropyldiaminomethane, hexamethyltriethylentetramine, hexamethyltripropylenetetramine, diisobutylentriamine, triisopro-pylentriamine, and mixtures thereof.

Acetoacetamide Salt Formation Reaction

Once formed in reaction (1), the sulfamic acid salt is reacted with the acetoacetylating agent to form the acetoacetamide salt, preferably acetoacetamide-N-sulfonate triethylammonium salt. Preferably, the acetoacetylating agent comprises diketene, although other acetoacetylating agents may be employed, either with or without diketene.

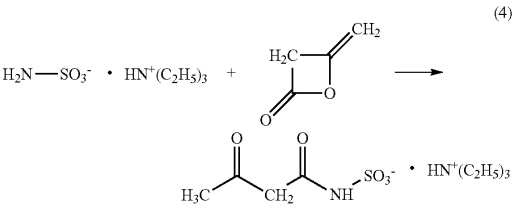
In one embodiment, the resultant acetoacetamide salt corresponds to the following formula (3).



(3)

wherein M^+ is an appropriate ion. Preferably, M^+ is an alkali metal ion or $N^+R_1R_2R_3R_4$. R_1, R_2, R_3 and R_4, independently of one another, may be organic radicals or hydrogen, preferably H or C_1-C_8 alkyl, C_6-C_{10} cycloalkyl, aryl and/or aralkyl. In a preferred embodiment, R_1 is hydrogen, and R_2, R_3 and R_4 are alkyl, e.g., ethyl.

An exemplary reaction scheme for forming an acetoacetamide salt employs a trialkyl ammonium amidosulfamic acid salt and diketene as reactants and yields an acetoacetamide triethylammonium salt is shown in reaction (4), below.



(4)

In one embodiment, the reaction is conducted in the presence of a catalyst, which may vary widely. In some embodiments, the catalyst comprises one or more amines and/or phosphines. Preferably, the catalyst comprises triethylamine. In some cases trimethylamine serves as both a catalyst and a reactant.

In one embodiment wherein the amidosulfamic acid salt formation reaction and the acetoacetamide salt formation reaction are conducted in separate reactors, a second reaction mixture comprises the amidosulfamic acid salt, the diketene, and the catalyst, e.g., triethylamine. Preferably, catalyst from the first reaction is carried through to the reaction mixture of the second reaction. The second reaction mixture is then subjected to conditions effective to form the acetoacetamide salt.

In one embodiment, the composition of the second reaction mixture may be similar to that of the first reaction mixture. In a preferred embodiment, the reaction product of the amidosulfamic acid salt formation reaction provides the amidosulfamic acid salt component of the second reaction mixture. In addition to the above-mentioned components, the second reaction mixture may further comprise reaction by-products from the first reaction or non-reacted starting materials.

In one embodiment, the amount of acetoacetylating agent, e.g., diketene, should be at least equimolar to the reactant amidosulfamic acid salt that is provided. In one embodiment, the process may utilize a diketene in excess, but preferably in an excess less than 30 mol %, e.g., less than 10 mol %. Greater excesses are also contemplated.

The amidosulfamic acid salt formation reaction and/or the acetoacetamide salt formation reaction may employ an organic solvent. Suitable inert organic solvents include any organic solvents that do not react in an undesired manner with the starting materials, cyclizing agent, final products and/or the catalysts in the reaction. The solvents preferably have the ability to dissolve, at least partially, amidosulfamic acid salts. Exemplary organic solvents include halogenated aliphatic hydrocarbons, preferably having up to 4 carbon atoms such as, for example, methylene chloride, chloroform, 1,2-dichlorethane, trichloroethylene, tetrachloroethylene, trichlorofluoroethylene; aliphatic ketones, preferably those having 3 to 6 carbon atoms such as, for example, acetone, methyl ethyl ketone; aliphatic ethers, preferably cyclic aliphatic ethers having 4 or 5 carbon atoms such as, for example, tetrahydrofuran, dioxane; lower aliphatic carboxylic acids, preferably those having 2 to 6 carbon atoms such as, for example, acetic acid, propionic acid; aliphatic nitriles, preferably acetonitrile; N-alkyl-substituted amides of carbonic acid and lower aliphatic carboxylic acids, preferably amides having up to 5 carbon atoms such as, for example,

tetramethylurea, dimethylformamide, dimethylacetamide, N-methylpyrrolidone; aliphatic sulfoxides, preferably dimethyl sulfoxide, and aliphatic sulfones, preferably sulfolane.

Particularly preferred solvents include dichloromethane (methylene chloride), 1,2-dichloroethane, acetone, glacial acetic acid and dimethylformamide, with dichloromethane (methylene chloride) being particularly preferred. The solvents may be used either alone or in a mixture. In one embodiment, the solvent is a halogenated, aliphatic hydrocarbon solvent, preferably the solvent is dichloromethane. Chloroform and tetrachloromethane are also exemplary solvents.

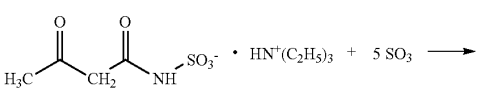
In one embodiment, the acetoacetamide salt formation reaction is conducted a temperature ranging from −30° C. to 50° C., e.g., from 0° C. to 25° C. The reaction pressure may vary widely. In preferred embodiments, the reaction is carried out at atmospheric pressure, although other pressures are also contemplated. The reaction time may vary widely, preferably ranging from 0.5 hours to 12 hours, e.g., from 1 hour to 10 hours. In one embodiment, the reaction is carried out by introducing the amidosulfamic acid salt and metering in the diketene. In another embodiment, the reaction is carried out by introducing diketene and metering in the amidosulfamic acid salt. The reaction may be carried out by introducing the diketene and amidosulfamic acid and metering in the catalyst.

Once formed, each reaction product is optionally subjected to one or more purification steps. For example, the solvent may be separated from the reaction product, e.g., via distillation, and the residue (mainly acetoacetamide-N-sulfonate) may be recrystallized from a suitable solvent such as, for example, acetone, methyl acetate or ethanol.

Generally speaking, the steps of reacting sulfamic acid and triethylamine to form an amidosulfamic acid salt, reacting the amidosulfamic acid salt and diketene to form the acetoacetamide salt, and contacting dichloromethane and a sulfur trioxide to form a cyclizing agent composition are performed in no particular order. Each of these steps may be performed independently of one another. In some cases, these steps may be performed in any order as long as they are performed before the cyclization reaction, e.g., the reaction of the acetoacetamide salt with sulfur trioxide to form a cyclic sulfur trioxide adduct.

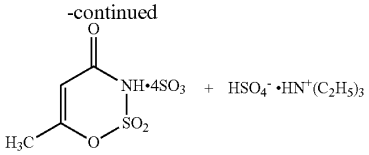
Cyclization and Hydrolyzation

The acetoacetamide salt is reacted with cyclizing agent, e.g., cyclizing agent in the cyclizing agent composition, in the presence of a solvent to form the cyclic (sulfur trioxide) adduct composition, which contains cyclic sulfur trioxide adduct and, in some cases, impurities. In some cases, a cooling step occurs before the cyclic sulfur trioxide adduct formation reaction. In one embodiment, the cyclization is achieved by using at least an equimolar amount of the cyclizing agent. The cyclizing agent may be dissolved in an inert inorganic or organic solvent. The cyclizing agent is generally used in a molar excess, e.g., up to a 20 fold excess, or up to a 10 fold excess, based on the total moles of acetoacetamide salt. An exemplary cyclization reaction using sulfur trioxide as the cyclizing agent is shown in reaction (5), below.



(5)

-continued



In one embodiment, the weight ratio of solvent to cyclizing agent in the cyclizing agent composition is at least 1:1, e.g., at least 2:1, or at least 5:1. In one embodiment, the weight ratio of solvent to cyclizing agent in the cyclizing agent composition ranges from 1:1 to 25:1, e.g., from 1:1 to 10:1, from 2:1 to 10:1, or from 5:1 to 10:1.

A cyclizing agent may be any compound that initiates the ring closure of the acetoacetamide salt. Although sulfur trioxide is a preferred cyclizing agent, the employment of other cyclizing agents is contemplated.

Suitable inert inorganic or organic solvents are those liquids which do not react in an undesired manner with sulfur trioxide or the starting materials or final products of the reaction. Preferred organic solvents include, but are not limited to, halogenated aliphatic hydrocarbons, preferably having up to four carbon atoms, such as, for example, methylene chloride (dichloromethane), chloroform, 1,2-dichloroethane, trichloroethylene, tetrachloroethylene, trichlorofluoroethylene; esters of carbonic acid with lower aliphatic alcohols, preferably with methanol or ethanol; nitroalkanes, preferably having up to four carbon atoms, in particular nitromethane; alkyl-substituted pyridines, preferably collidine; and aliphatic sulfones, preferably sulfolane. Particularly preferred solvents for the cyclization reaction include dichloromethane (methylene chloride), 1,2-dichloroethane, acetone, glacial acetic acid and dimethylformamide, with dichloromethane (methylene dichloride) being particularly preferred. Other solvents, e.g., other solvents mentioned herein, may also be suitable as solvents. The solvents may be used either alone or in a mixture. In one embodiment, the solvent is a halogenated, aliphatic hydrocarbon solvent, preferably the solvent is dichloromethane. The processes may employ these solvents alone or in mixtures thereof.

In some cases, the solvent in the cyclizing agent composition may be selected from 1) concentrated sulfuric acid, 2) liquid sulfur dioxide, or 3) an inert organic solvent.

In a preferred embodiment, the same solvent is used in both the acetoacetamide salt formation reaction and the cyclization reaction. As one benefit, the solution obtained in the acetoacetamide salt formation reaction, without isolation of the acetoacetamide salt formation reaction product, may be used immediately in the cyclization.

In one embodiment, the reaction temperature for the cyclization reaction ranges from −70° C. to 175° C., e.g., from −40° C. to 60° C. The pressure at which the reaction is conducted may vary widely. In one embodiment, the reaction is conducted at a pressure ranging from 0.01 MPa to 10 MPa, e.g., from 0.1 MPa to 5 MPa. Preferably, the reaction is conducted at atmospheric pressure.

The acetoacetamide salt may be introduced to the cyclization reactor and the cyclizing agent composition, e.g., a solution of cyclizing agent optionally in solvent, may be metered into the reactor. In preferred embodiments, both reactants (acetoacetamide salt and cyclizing agent) are simultaneously fed into the reactor. In one embodiment, the cyclizing agent composition is initially introduced into the reactor and the acetoacetamide salt is added. Preferably, at

least part of the cyclizing agent composition is introduced into the reactor and, either continuously or in portions, acetoacetamide salt and (additional) cyclizing agent are then metered in, preferably while maintaining the temperature as described above.

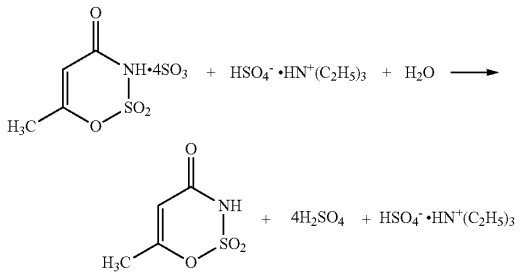
The acetoacetamide salt may be introduced to the reactor and the cyclizing agent composition may be metered into the reactor. In preferred embodiments, both reactants are simultaneously fed into the reactor. In one embodiment, the cyclizing agent composition is initially introduced into the reactor and the acetoacetamide salt is added. Preferably, at least part of the cyclizing agent composition is introduced into the reactor and, either continuously or in portions, acetoacetamide salt and (additional) cyclizing agent are then metered in, preferably while maintaining the temperature as described above.

The formation of the crude acesulfame potassium composition from the cyclic sulfur trioxide adduct composition, in some embodiments, comprises the steps of hydrolyzing the cyclic sulfur trioxide adduct to form an acesulfame-H composition; neutralizing the acesulfame-H in the acesulfame H composition to form a crude acesulfame potassium composition; and forming the acesulfame potassium composition from the crude acesulfame potassium composition.

The cyclic sulfur trioxide adduct may be hydrolyzed via conventional means, e.g., using water. Thus, the forming step may comprise the steps of hydrolyzing the cyclic sulfur trioxide adduct to form an acesulfame-H composition. Acesulfame-H is referred to as sweetener acid.

An exemplary hydrolysis reaction scheme is shown in reaction (6), below.

(6)



The addition of the water leads to a phase separation. The majority of the sweetener acid, acesulfame-H (6-methyl-3,4-dihydro-1,2,3-oxathiazin-4-one 2,2-dioxide), which is formed via the hydrolysis, is present in the organic phase, e.g., at least 60 wt %, at least 70%, at least 80%, or at least 90%. The remainder of the sweetener acid is in the water phase and can be extracted and optionally added to the sweetener acid in the organic phase. In cases where dichloromethane is used as the reaction medium, water or ice may be added, e.g., in a molar excess, based on the sulfur trioxide, to the cyclic sulfur trioxide adduct/sulfur trioxide solution.

In some cases, the hydrolysis step comprises adding water to the cyclic sulfur trioxide adduct. In preferred embodiments, the weight ratio of water to acetoacetamide salt is greater than 1.3:1, e.g., greater than 1.5:1, greater than 1.7:1, greater than 2:1 or greater than 2.2:1. Employment of these ratios may lead to decreases in acetoacetamide-N-sulfonic acid and/or acetoacetamide formation in the neutralized crude acesulfame potassium composition, e.g., the crude acesulfame potassium composition may comprise acetoacetamide-N-sulfonic acid in the amounts discussed herein.

It was surprisingly discovered that the temperature at which the water is initially fed to the hydrolysis reaction may have beneficial effects on impurity production, e.g., organic production or 5-chloro-acesulfame potassium production as well as reaction parameters, e.g., temperature. At lower temperatures, e.g., lower than approximately −35° C. or lower than −22° C., ice tends to build up in the reaction mixture. As this ice melted, it led to the onset of additional reaction, which caused the temperature to rise quickly. This rise in temperature surprisingly led to a product that contained much higher levels of impurities. In some cases, the hydrolyzing comprises adding hydrolysis water to the cyclic sulfur trioxide adduct to form a hydrolysis reaction mixture and reacting the mixture to from the acesulfame-H composition. In some embodiments, the temperature of the hydrolysis reaction mixture or the temperature at which the hydrolysis water is fed to the reactor is maintained at a temperature greater than −35° C., e.g., greater than −30° C., greater than −25° C., greater than −24° C., greater than −23° C., greater than −22° C., greater than −21.5° C., greater than −21° C., or greater than greater than −20° C. In terms of ranges, the temperature of the hydrolysis reaction mixture or the temperature at which the hydrolysis water is fed to the reactor optionally is maintained at a temperature ranging from −35° C. to 0° C., e.g., from −30° C. to −5° C., from −20° C. to −5° C., from −30° C. to −20° C., from −25° C. to −21° C., or −25° C. to −21.5° C.

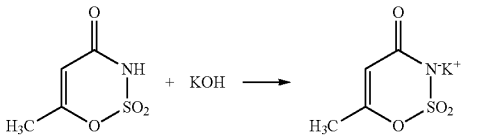
After the addition of water, the reaction solvent, e.g., dichloromethane, may be removed by distillation, or the acesulfame-H that remains in the organic phase may be extracted with a more suitable solvent. Suitable solvents are those which are sufficiently stable towards sulfuric acid and which have a satisfactory dissolving capacity. Other suitable solvents include esters of carbonic acid such as, for example dimethyl carbonate, diethyl carbonate and ethylene carbonate, or esters of organic monocarboxylic acids such as, for example, isopropyl formate and isobutyl formate, ethyl acetate, isopropyl acetate, butyl acetate, isobutyl acetate and neopentyl acetate, or esters of dicarboxylic acids or amides which are immiscible with water, such as, for example, tetrabutylurea, are suitable. Isopropyl acetate and isobutyl acetate are particularly preferred.

The combined organic phases are dried with, for example, Na_2SO_4, and are evaporated. Any sulfuric acid which has been carried over in the extraction may be removed by appropriate addition of aqueous alkali to the organic phase. For this purpose, dilute aqueous alkali may be added to the organic phase until the pH reached in the aqueous phase corresponds to that of pure 6-methyl-3,4-dihydro-1,2,3-oxathiazin-4-one 2,2-dioxide at the same concentration in the same two-phase system of extracting agent and water.

Neutralization

The neutralization of the acesulfame-H yields a non-toxic salt of acesulfame-H, e.g., acesulfame potassium. In one embodiment, neutralization is carried out by reacting the acesulfame-H with an appropriate base, e.g., potassium hydroxide, in particular a membrane-produced potassium hydroxide. Other suitable bases include, for example, KOH, $KHCO_3$, K_2CO_3, and potassium alcoholates. An exemplary reaction scheme using potassium hydroxide as a neutralizing agent is shown in reaction (7), below.

(7)



In some cases, the neutralization is conducted or maintained at a low pH levels, which may advantageously further result in a reduction or elimination of the formation of impurities, e.g., acetoacetamide salts. In this context, "conducted" means that the neutralization step begins at a low pH level, and "maintained" means that steps are taken to ensure that the pH stays within a low pH range throughout the entire neutralization step. In one embodiment, the neutralization step is conducted or maintained at a pH below 10.0, e.g., below 9.5, below 9.0, below 8.5, below 8.0, below 7.5, below 7.0, or below 6.5. In terms of ranges, the neutralization step is preferably conducted or maintained at a pH between 6.0 and 10.0, e.g., between 6.5 and 9.5, between 7.0 and 9.0, or between 7.5 and 8.5.

In some cases, the pH in the neutralizing step may be maintained within the desired range by managing the components of the neutralization reaction mixture, which comprises acesulfame-H and neutralizing agent (and also solvent). For example, the composition of the neutralization reaction mixture may include from 1 wt % to 95 wt % neutralizing agent, e.g., from 10 wt % to 85 wt % or from 25 wt % to 75 wt %, and from 1 wt % to 95 wt % acesulfame-H, e.g., from 10 wt % to 85 wt % or from 25 wt % to 75 wt %. These concentration ranges are based on the mixture of neutralization agent and acesulfame-H (not including solvent).

In one embodiment, the acesulfame-H may be neutralized and extracted directly from the purified organic extraction phase using an aqueous potassium base. The acesulfame potassium then precipitates out, where appropriate after evaporation of the solution, in the crystalline form, and it can also be recrystallized for purification.

In one embodiment, the process is not a small-scale batch process or a laboratory-scale process. For example, the inventive process for producing a finished acesulfame potassium composition may yield at least 50 grams of finished acesulfame potassium composition per batch, e.g., at least 100 grams per batch, at least 500 grams per batch, at least 1 kilogram per batch, or at least 10 kilograms per batch. In terms of rates, the inventive process may yield at least 50 grams of finished acesulfame potassium composition per hour, e.g., at least 100 grams per hour, at least 500 grams per hour, at least 1 kilogram per hour, or at least 10 kilograms per hour.

FIG. **1** shows an exemplary acesulfame potassium process **100** in accordance with the process described herein. Process **100** comprises amidosulfamic acid salt formation reactor **102** and acetoacetamide salt formation reactor **104**. Although FIG. **1** shows separate reactors for the two intermediate formation reactions, other configurations, e.g., a one reactor process, are within the contemplation of the present process. Sulfamic acid is fed to amidosulfamic acid salt formation reactor **102** via sulfamic acid feed line **106**. One or more amines, preferably triethylamine, are fed to amidosulfamic acid salt formation reactor **102** via amine feed line **108**. In addition to sulfamic acid and amine(s), acetic acid is also fed to amidosulfamic acid salt formation reactor **102** (via feed line **110**). The resultant reaction mixture in amidosulfamic acid salt formation reactor **102** is as discussed above. In amidosulfamic acid salt formation reactor **102**, the sulfamic acid and the amine (in the presence of the acetic acid) are reacted to yield a crude amidosulfamic acid salt composition, which exits reactor **102** via line **112**. Although not shown, a reaction solvent, e.g., dichloromethane may also be present in the amidosulfamic acid salt formation reactor **102**.

The crude amidosulfamic acid salt composition in line **112** is directed to acetoacetamide salt formation reactor **104**. Diketene is fed to acetoacetamide salt formation reactor **104** via feed line **114**. In acetoacetamide salt formation reactor **104**, the amidosulfamic acid salt and the diketene are reacted to yield a crude acetoacetamide salt composition, which exits reactor **104** via line **118**. Although not shown, dichloromethane may also be present in the acetoacetamide salt formation reactor **104**.

Cyclizing agent (sulfur dioxide) and solvent (dichloromethane) are fed to vessel **119** via feed lines **121** and **123**. Vessel **119** is preferably a cooling vessel wherein the cyclizing agent composition (as discussed above) is formed. The cyclizing agent composition exits vessel **119** via line **125**.

The crude acetoacetamide salt composition is directed to cyclization reactor **120** via line **118**. The cooled cyclizing agent composition is also directed to cyclization reactor **120** (via line **125**). Line **125** is preferably made of a material and in such a size and shape to facilitate the residence times discussed herein. In cyclization reactor **120**, the acetoacetamide salt in the crude acetoacetamide salt composition in line **118** is cyclized and a cyclic sulfur trioxide adduct stream exits via line **124**.

The cyclic sulfur trioxide adduct in line **124**, is directed to hydrolysis reactor **126**. Water is fed to hydrolysis reactor **126** via water feed **128**. In hydrolysis reactor **126**, the cyclic sulfur trioxide adduct is hydrolyzed to yield a crude acesulfame-H composition, which exits hydrolysis reactor **126** via line **130** and is directed to phase separation unit **132**. Phase separation unit **132** separates the contents of line **130** into organic phase **134** and aqueous phase **136**. Organic phase **134** comprises a major amount of the acesulfame-H in line **130** as well as solvent, e.g., methylene chloride. Aqueous phase **136** exits via line **137** and comprises triethylammonium sulfate, and optionally sulfuric acid and minor amounts of acesulfame-H. This aqueous phase may be further purified to separate and/or recover the acesulfame-H and/or the triethylammonium sulfate. The recovered acesulfame-H may be combined with the acesulfame from the organic phase (not shown).

Organic phase **134** exits phase separation unit **132** and is directed to extraction column **138** (via line **140**). Water is fed to extraction column **138** via water feed **142**. The water extracts residual sulfates from the contents of line **140** and a purified acesulfame-H composition exits extraction column **138** via line **144**. The extracted sulfates exit extraction column **138** via line **145**.

The organic phase exits phase separation unit **132** and is directed to extraction column **138** (via line **140**). Water is fed to extraction column **138** via water feed **142**. The water extracts residual sulfates from the contents of line **140** and a purified acesulfame-H stream exits extraction column **138** via line **144**. The extracted sulfates exit extraction column **138** via line **145**.

The purified acesulfame-H composition in line **144** is directed to neutralization unit **146**. Potassium hydroxide is also fed to neutralization unit **146** (via line **148**). The potassium hydroxide neutralizes the acesulfame-H in the

purified acesulfame-H composition to yield a product comprising acesulfame potassium, dichloromethane, water, potassium hydroxide, and impurities, e.g., 5-chloro-acesulfame potassium, which exits neutralization unit **146** via line **150**. This product may be considered a crude acesulfame potassium composition.

The product in line **150** is directed to phase separation unit **160**. Phase separation unit **160** separates the product in line **150** into organic phase **162** and an aqueous phase **164**. Aqueous phase **164** comprises a major amount of the acesulfame potassium in line **150** as well as some impurities. Organic phase **162** comprises potassium hydroxide, dichloromethane, and water and may be further treated to recover these components. Aqueous phase **164** (without any further treatment) may be considered a crude acesulfame potassium composition. Aqueous phase **164** may be optionally treated to form a finished acesulfame potassium composition.

Aqueous phase **164** is directed to treatment unit **156** via line **166**. In treatment unit **156**, aqueous phase **164** is treated to obtain finished acesulfame potassium composition (product that may be sold), which is shown exiting via stream **152**. In addition to the finished acesulfame potassium composition, dichloromethane and potassium hydroxide may be separated. These components exit treatment unit **156** via line **154**. The contents of stream **154** may be recovered and/or recycled to the process.

The crude acesulfame potassium product stream comprises acesulfame potassium, dichloromethane, water, and potassium hydroxide. The crude acesulfame potassium product stream in line **150** may be directed to further processing to recover purified acesulfame potassium, which is shown exiting via stream **152**. In addition to the purified acesulfame potassium, dichloromethane and potassium hydroxide may be separated from the crude acesulfame potassium product stream, as shown by stream **154**. The contents of stream **154** may be recovered and/or recycled to the process.

The invention relates also to the following aspects:

Aspect 1: A process for producing a finished acesulfame potassium composition, the process comprising the steps of:

(a) contacting a solvent and a cyclizing agent to form a cyclizing agent composition;

(b) reacting an acetoacetamide salt with the cyclizing agent in the cyclizing agent composition to form a cyclic sulfur trioxide adduct; and

(c) forming from the cyclic sulfur trioxide adduct the finished acesulfame potassium composition comprising non-chlorinated acesulfame potassium and less than 35 wppm 5-chloro-acesulfame potassium;

wherein contact time from the beginning of step (a) to the beginning of step (b) is less than 60 minutes.

Aspect 2: The process of aspect 1, wherein the forming comprises:

hydrolyzing the cyclic sulfur trioxide adduct to form an acesulfame-H composition comprising acesulfame-H;

neutralizing the acesulfame-H in the acesulfame-H composition to form a crude acesulfame potassium composition comprising non-chlorinated acesulfame potassium and less than 35 wppm 5-chloro-acesulfame potassium; and

forming the finished acesulfame potassium composition from the crude acesulfame potassium composition.

Aspect 3: The process of any one of the preceding aspects, wherein the finished acesulfame potassium composition comprises from 0.001 wppm to 5 wppm 5-chloro-acesulfame potassium.

Aspect 4: The process of any one of the preceding aspects, wherein the contact time is less than 15 minutes and the crude acesulfame potassium composition comprises from 0.001 wppm to 5 wppm 5-chloro-acesulfame potassium and the finished acesulfame potassium composition comprises from 0.001 wppm to 5 wppm 5-chloro-acesulfame potassium.

Aspect 5: The process of any one of the preceding aspects, wherein the contact time is less than 5 minutes and the crude acesulfame potassium composition comprises from 0.001 wppm to 5 wppm 5-chloro-acesulfame potassium and the finished acesulfame potassium composition comprises from 0.001 wppm to 2.7 wppm 5-chloro-acesulfame potassium.

Aspect 6: The process of any one of the preceding aspects, wherein the crude acesulfame potassium composition comprises less than 35 wppm 5-chloro-acesulfame potassium.

Aspect 7: The process of any one of the preceding aspects, wherein the finished acesulfame potassium composition comprises at least 90% by weight of the 5-chloro-acesulfame potassium present in the crude acesulfame potassium composition.

Aspect 8: The process of any one of the preceding aspects, wherein the hydrolyzing comprises adding water to the cyclic sulfur trioxide adduct to form a hydrolysis reaction mixture, and wherein the temperature of the hydrolysis reaction mixture is maintained at a temperature ranging from −35° C. to 0° C.

Aspect 9: The process of any one of the preceding aspects, wherein the finished acesulfame potassium composition comprises from 0.001 wppm to 5 wppm organic impurities.

Aspect 10: The process of any one of the preceding aspects, further comprising:

reacting sulfamic acid and an amine to form an amidosulfamic acid salt; and

reacting the amidosulfamic acid salt and acetoacetylating agent to form the acetoacetamide salt.

Aspect 11: The process of any one of the preceding aspects, wherein the finished acesulfame potassium composition comprises from 0.001 wppm to 2.7 wppm 5-chloro-acesulfame potassium.

Aspect 12: The process of any one of the preceding aspects, wherein the finished acesulfame potassium composition comprises from 0.001 wppm to 5 wppm organic impurities.

Aspect 13: The process of any one of the preceding aspects, wherein the finished acesulfame potassium composition comprises from 0.001 wppm to 5 wppm heavy metals.

Aspect 14: The process of any one of the preceding aspects, wherein the cyclizing agent composition comprises less than 1 wt % of compounds selected from chloromethyl chlorosulfate, methyl-bis-chlorosulfate, and mixtures thereof.

Aspect 15: The process of any one of the preceding aspects, wherein the reacting is conducted for a cyclization reaction time, from the start of the reactant feed to the end of the reactant feed, less than 35 minutes.

Aspect 16: The process of any one of the preceding aspects, wherein the weight ratio of solvent to cyclizing agent in the cyclizing agent composition is at least 1:1.

Aspect 17: The process of any one of the preceding aspects, further comprising cooling the cyclizing agent composition to a temperature less than 15° C.

Aspect 18: The process of any one of the preceding aspects, wherein the cyclizing agent comprises sulfur trioxide and the solvent comprises dichloromethane.

Aspect 19: A finished acesulfame potassium composition produced or producible by, or obtainable or obtained from the process of any one of aspects 1 to 18.

Aspect 20: The finished acesulfame potassium composition of aspect 19, comprising:

non-chlorinated acesulfame potassium;

from 0.001 wppm to 2.7 wppm 5-chloro acesulfame potassium; and

from 0.001 wppm to 5 wppm heavy metals.

Aspect 21: A process for producing a finished acesulfame potassium composition, the process comprising the steps of:

reacting sulfamic acid and triethylamine to form an amidosulfamic acid salt;

reacting the amidosulfamic acid salt and diketene to form the acetoacetamide salt;

contacting dichloromethane and a sulfur trioxide to form a cyclizing agent composition;

reacting the acetoacetamide salt with the sulfur trioxide in the cyclizing agent composition to form a cyclic sulfur trioxide adduct;

hydrolyzing the cyclic sulfur trioxide adduct to form an acesulfame-H composition; and

neutralizing the acesulfame-H to form the finished acesulfame potassium composition comprising non-chlorinated acesulfame potassium and less than 10 wppm 5-chloro-acesulfame potassium,

wherein contact time from the beginning of step (a) to the beginning of step (b) is less than 10 minutes.

Aspect 22: The process of aspect 21, further comprising cooling the cyclizing agent composition to a temperature less than 15° C.

Aspect 23: An acesulfame potassium composition comprising non-chlorinated acesulfame potassium and less than 35 wppm, preferably 0.001 wppm to 2.7 wppm 5-chloro-acesulfame potassium.

Aspect 24: The acesulfame potassium composition of aspect 23, further comprising less than 37 wppm, preferably 1 wppb to 5 wppm acetoacetamide-N-sulfonic acid.

Aspect 25: The acesulfame potassium composition of any one of the preceding aspects, further comprising 0.001 wppm to 5 wppm organic impurities and/or 0.001 wppm to 5 wppm of at least one heavy metal.

Aspect 26: The acesulfame potassium composition of any one of the preceding aspects, wherein the at least one heavy metal is selected from the group consisting of mercury, lead and both.

Aspect 27: The acesulfame potassium composition of any one of the preceding aspects, wherein the mercury is present in an amount of 1 wppb to 20 wppm.

Aspect 28: The acesulfame potassium composition of any one of the preceding aspects, wherein the lead is present in an amount of 1 wppb to 25 wppm.

EXAMPLES

Example 1

Liquid sulfur trioxide and dichloromethane were continuously fed, contacted (to form a cyclizing agent composition), and cooled into a static mixer at 1220 kg/h and 8000 kg/h, respectively. The temperature of the cooled cyclizing agent composition was 11° C. The mixture was held in the static mixture for less than 5 minutes and then fed into a cyclization reactor, thus contact time was less than 5 minutes. In the cyclization reactor the sulfur trioxide/dichloromethane composition was reacted with a solution of acetoacetamide-N-sulfonate triethylammonium salt (acetoacetamide salt) in dichloromethane. The resultant cyclized product was hydrolyzed and worked up to yield a crude acesulfame potassium composition comprising (non-chlorinated) acesulfame potassium. Testing for 5-chloro-acesulfame potassium content was performed using the HPLC equipment and techniques discussed herein. In particular, the HPLC analysis was performed using an LC Systems HPLC unit from Shimadzu having a CBM-20 Shimadzu controller and being equipped with a CC 250/4.6 Nucleodur 100-3 C18 ec (250×4.6 mm) MACHEREY NAGEL column. A Shimadzu SPD-M20A photodiode array detector was used for detection (at 234 nm wavelength). Analysis was performed at 23° C. column temperature. As an eluent solution, an aqueous solution of tetra butyl ammonium hydrogen sulfate (3.4 g/L and 60% of the total solution) and acetonitrile (HPLC grade) (300 mL/L and 40% of the total solution) was employed. Elution was isocratic. The overall flow rate of total eluent was approximately 1 mL/min. The data collection and calculations were performed using Lab Solution software from Shimadzu. With a detection limit of 1 wppm, no 5-chloro-acesulfame potassium was detected.

Comparative Example A

528 mmol of sulfur trioxide in dichloromethane was prepared and stored for 20 days at 20° C. The sulfur trioxide/diclhloromethane composition was fed to a stirred vessel. 100 mmol acetoacetamide-N-sulfonate triethylammonium salt in dichloromethane was reacted with the sulfur trioxide/diclhloromethane composition by continuous feeding into the stirred vessel for 30 minutes. Contact time was 20 days. After additional stirring for two minutes, the reaction mixture was hydrolyzed by the addition of 50 ml water and worked up as described herein. Testing for 5-chloro-acesulfame potassium content was performed using the HPLC equipment and techniques discussed above. The crude acesulfame potassium had an impurity content of 4960 wppm 5-chloro-acesulfame potassium.

Example 2 and Comparative Examples B and C

100 mmol of 99.5% pure sulfamic acid was suspended in 50 mL dichloromethane in a flask with reflux. Under continuous agitation, 105 mmol of trimethylamine was added within approximately 3 minutes. During this time, temperature increased due to acid/base exothermal reaction up to about 42° C. (the boiling point of dichloromethane). This reaction mixture was stirred for approximately 15 additional minutes, until no solid sedimentation was seen in the flask. Then, 10 mmol of acetic acid was added to the first reaction mixture and was stirred for approximately 15 additional minutes. At this point, within 7 minutes of the addition of the acetic acid, 110 mmol of diketene was added dropwise to form a second reaction mixture. After the addition of all of the diketene was added to the second reaction mixture and approximately 15 minutes of reaction time, this second reaction mixture was cooled. The resultant cooled second reaction mixture contained approximately 30% acetoacetamide N-sulfonate triethylammonium salt. Additional batches of cooled second reaction mixture were prepared as necessary. The acetoacetamide N-sulfonate triethylammonium salt was used as discussed below.

Sulfur trioxide/dichloromethane compositions (cyclizing agent compositions) were prepared by contacting approximately 15 wt % sulfur trioxide and approximately 85 wt % dichloromethane with one another in a flask.

For each of Example 2 and Comparative Examples B and C, a reaction flask (a 4 necked round bottom flask equipped with mechanical stirrer, thermometer, and feed vessels) was placed into a cooling bath containing a mixture of isopro-

panol and dry ice. Approximately 200 g of the acetoacetamide-N-sulfonate triethylammonium salt solution and approximately 577 g of the sulfur trioxide/dichloromethane compositions were measured. The compositions were held for various time periods before the start of the cyclization reaction. Contact times for the respective examples are shown in Table 1.

TABLE 1

Contact Times

Example	Contact Time
Ex. 2	1 hour
Comp. Ex. B	4 days
Comp. Ex. C	5 days

For each example, the flask was placed into a cooling bath containing a mixture of isopropanol and dry ice. Approximately 15 wt % of the total sulfur trioxide/dichloromethane composition (approximately 87 g) was initially fed to the reaction flask under continuous agitation by mechanical stirrer. When the temperature of the reaction flask contents reached −35° C. (due to the cooling batch), the remainder of the sulfur trioxide/dichloromethane composition and all of the acetoacetamide-N-sulfonate triethylammonium salt solution were fed into the reaction flask. The time period that the solvent contacted the cyclizing agent before formation of the cyclic sulfur trioxide adduct, e.g., before the acetoacetamide-N-sulfonate triethylammonium salt solution was fed to the reaction flask, was less than an hour. The feed rate was controlled in such a way that the temperature of the reaction flask contents remained between −25° and −35° C. during the feeding/cyclization reaction. After the reactants were fed, the reaction was allowed to proceed for approximately one additional minute. The cooling bath was then removed.

After approximately one minute, the temperature of the reaction flask contents reached approximately −22° C. At this time, hydrolysis was initiated by feeding deionized water to the reaction flask. Water was fed over 10 minutes. The hydrolysis reaction was exothermic. Water was added slowly so as to maintain temperature between −20° C. and −5° C. After addition of water, reaction mixture was allowed to reach room temperature.

The hydrolyzed product was phase separated via a separating funnel. A heavier organic sweetener acid-dichloromethane phase (acesulfame-H composition) was separated out, and the remaining aqueous phase was discarded.

The acesulfame-H in the acesulfame-H composition was neutralized with a 10% potassium hydroxide solution. Neutralization was carried out at 25° C.±1° C. Potassium hydroxide addition was completed within 20 minutes.

After completion of the neutralization step, an additional phase separation was performed using a separating funnel to yield an aqueous phase containing acesulfame potassium (and some impurities) and an organic phase. The aqueous phase is considered a crude acesulfame potassium composition. The aqueous phase analyzed for impurities, e.g., 5-chloro acesulfame potassium. Testing for 5-chloro-acesulfame potassium content was performed using the HPLC equipment and techniques discussed above. The remaining dichloromethane phase was discarded.

The results of the impurity analysis of Examples 1 and 2 and Comparative Examples A-C are shown in Table 2.

TABLE 2

5-chloro Ace-K Content

	Contact Time	5-chloro Ace-K (in crude), wppm
Ex. 1	<5 min.	Not detectable
Ex. 2	1 hour	32
Comp. Ex. A	20 days	4960
Comp. Ex. B	4 days	54
Comp. Ex. C	5 days	78

As shown in the Examples, the 5-chloro-acesulfame potassium content was affected by contact time. When a contact time of greater than 1 hour was employed (Comparative Examples A-C), significant amounts of 5-chloro acesulfame potassium were present in the crude acesulfame potassium composition. Importantly, when contact time was kept below 1 hour (Example 2), e.g., below 5 minutes (Example 1), then crude acesulfame potassium composition comprised much smaller amounts of 5-chloro acesulfame potassium.

Only minor and simple additional treatment of the crude acesulfame composition was necessary to form the finished acesulfame potassium compositions.

Approximately 50% of water was evaporated out of the crude acesulfame potassium compositions in roti vapor at reduced pressure. The resultant concentrated acesulfame potassium composition is considered an intermediate acesulfame potassium composition and was then cooled in a refrigerator at +5° C., which led to precipitation of crude crystals containing mostly acesulfame potassium.

The crude crystals were then dissolved in enough water and this resultant solution was heated to 70° C. Activated carbon powder was then added to the solution. The solution (with the added activated carbon) was then filtered.

The filtrate that was yielded from the filtration was cooled to room temperature, which led to the formation of crystals containing mostly acesulfame potassium. These crystals were dissolved in sufficient water and heated to 70° C. in a water bath.

Activated carbon was added to this solution of crystals and activated carbon. This solution was then filtered. When filtrate was cooled down to room temperature, white-colored crystals of acesulfame potassium were formed. These crystals are considered a finished acesulfame potassium composition.

Testing for 5-chloro-acesulfame potassium content was performed using the HPLC equipment and techniques discussed above. The crystals of the finished acesulfame potassium composition contained the same amount (or slightly lower amounts) of 5-chloro-acesulfame potassium.

The treatment steps did not show a marked reduction in 5-chloro-acesulfame potassium content. It is believed that because the chemical structure of chloro-acesulfame potassium is similar to that of acesulfame potassium, separation of chloro-acesulfame potassium using standard purification procedures such as crystallization is ineffective. This analysis demonstrates the importance of reducing/eliminating the production of 5-chloro-acesulfame potassium during the steps leading to the formation of the crude acesulfame composition as described herein.

While the invention has been described in detail, modifications within the spirit and scope of the invention will be readily apparent to those of skill in the art. In view of the foregoing discussion, relevant knowledge in the art and references discussed above in connection with the Back-

ground and Detailed Description, the disclosures of which are all incorporated herein by reference. In addition, it should be understood that aspects of the invention and portions of various embodiments and various features recited above and/or in the appended claims may be combined or interchanged either in whole or in part. In the foregoing descriptions of the various embodiments, those embodiments which refer to another embodiment may be appropriately combined with other embodiments as will be appreciated by one of skill in the art. Furthermore, those of ordinary skill in the art will appreciate that the foregoing description is by way of example only, and is not intended to limit the invention.

We claim:

1. A process for producing a finished acesulfame potassium composition, the process comprising the steps of:

(a) contacting a cyclizing agent and a solvent selected from the group consisting of halogenated aliphatic hydrocarbons, esters of carbonic acid with lower aliphatic alcohols, nitroalkanes, alkyl-substituted pyridines, aliphatic sulfones, acetone, acetic acid, and dimethylformamide to form a cyclizing agent composition;

(b) reacting an acetoacetamide salt with the cyclizing agent in the cyclizing agent composition to form a cyclic sulfur trioxide adduct; and

(c) forming from the cyclic sulfur trioxide adduct the finished acesulfame potassium composition comprising non-chlorinated acesulfame potassium and less than 35 wppm 5-chloro-acesulfame potassium;

wherein contact time from the beginning of step (a) to the beginning of step (b) is less than 60 minutes.

2. The process of claim **1**, wherein the forming comprises:

hydrolyzing the cyclic sulfur trioxide adduct to form an acesulfame-H composition comprising acesulfame-H;

neutralizing the acesulfame-H in the acesulfame-H composition to form a crude acesulfame potassium composition comprising non-chlorinated acesulfame potassium and less than 35 wppm 5-chloro-acesulfame potassium; and

forming the finished acesulfame potassium composition from the crude acesulfame potassium composition.

3. The process of claim **2**, wherein the finished acesulfame potassium composition comprises from 0.001 wppm to 5 wppm 5-chloro-acesulfame potassium.

4. The process of claim **2**, wherein the contact time is less than 15 minutes and the crude acesulfame potassium composition comprises from 0.001 wppm to 5 wppm 5-chloro-acesulfame potassium and the finished acesulfame potassium composition comprises from 0.001 wppm to 5 wppm 5-chloro-acesulfame potassium.

5. The process of claim **2**, wherein the contact time is less than 5 minutes and the crude acesulfame potassium composition comprises from 0.001 wppm to 5 wppm 5-chloro-acesulfame potassium and the finished acesulfame potassium composition comprises from 0.001 wppm to 2.7 wppm 5-chloro-acesulfame potassium.

6. The process of claim **2**, wherein the crude acesulfame potassium composition comprises less than 35 wppm 5-chloro-acesulfame potassium.

7. The process of claim **5**, wherein the finished acesulfame potassium composition comprises at least 90% by weight of the 5-chloro-acesulfame potassium present in the crude acesulfame potassium composition.

8. The process of claim **2**, wherein the hydrolyzing comprises adding water to the cyclic sulfur trioxide adduct to form a hydrolysis reaction mixture, and wherein the temperature of the hydrolysis reaction mixture is maintained at a temperature ranging from −35° C. to 0° C.

9. The process of claim **8**, wherein the finished acesulfame potassium composition comprises from 0.001 wppm to 5 wppm organic impurities.

10. The process of claim **1**, further comprising:

reacting sulfamic acid and an amine to form an amidosulfamic acid salt; and

reacting the amidosulfamic acid salt and acetoacetylating agent to form the acetoacetamide salt.

11. The process of claim **1**, wherein the finished acesulfame potassium composition comprises from 0.001 wppm to 2.7 wppm 5-chloro-acesulfame potassium.

12. The process of claim **1**, wherein the finished acesulfame potassium composition comprises from 0.001 wppm to 5 wppm organic impurities.

13. The process of claim **1**, wherein the finished acesulfame potassium composition comprises from 0.001 wppm to 5 wppm heavy metals.

14. The process of claim **1**, wherein the cyclizing agent composition comprises less than 1 wt % of compounds selected from chloromethyl chlorosulfate, methyl-bis-chlorosulfate, and mixtures thereof.

15. The process of claim **1**, wherein the reacting is conducted for a cyclization reaction time, from the start of the reactant feed to the end of the reactant feed, less than 35 minutes.

16. The process of claim **1**, wherein the weight ratio of solvent to cyclizing agent in the cyclizing agent composition is at least 1:1.

17. The process of claim **1**, further comprising cooling the cyclizing agent composition to a temperature less than 15° C.

18. The process of claim **1**, wherein the cyclizing agent comprises sulfur trioxide and the solvent comprises dichloromethane.

19. The process of claim **2**, wherein the contact time ranges from 1 second to 10 minutes, the crude acesulfame potassium composition comprises from 1 wppb to 35 wppm 5-chloro-acesulfame potassium, and the finished acesulfame potassium composition comprises from 1 wppb to 35 wppm 5-chloro-acesulfame potassium.

20. The process of claim **2**, wherein the contact time ranges from 1 second to 10 minutes, the crude acesulfame potassium composition comprises from 1 wppb to 5 wppm 5-chloro-acesulfame potassium, and the finished acesulfame potassium composition comprises from 1 wppb to 5 wppm 5-chloro-acesulfame potassium.

21. The process of claim **2**, wherein the contact time ranges from 1 second to 30 minutes, the crude acesulfame potassium composition comprises from 10 wppb to 10 wppm 5-chloro-acesulfame potassium, and the finished acesulfame potassium composition comprises from 10 wppb to 10 wppm 5-chloro-acesulfame potassium.

22. The process of claim **1**, further comprising cooling the cyclizing agent composition to a temperature ranging from −20° C. to 15° C., and wherein the cooling step reduces the temperature of the cyclizing agent composition by at least 10° C.

23. The process of claim **2**, wherein the finished acesulfame potassium composition comprises at least 90% by weight of the 5-chloro-acesulfame potassium present in the crude acesulfame potassium composition.

24. The process of claim **2**, wherein the hydrolyzing step comprises adding water to the cyclic sulfur trioxide adduct, and wherein the weight ratio of water to acetoacetamide salt in the hydrolyzing step is greater than 1.3:1.

25. The process of claim **24**, wherein the weight ratio of water to acetoacetamide salt in the hydrolyzing step (b) is greater than 1.7:1, and wherein the finished acesulfame potassium composition comprises less than 10 wppm acetoacetamide-N-sulfonic acid and less than 10 wppm acetoacetamide.

26. The process of claim **23**, wherein the finished acesulfame potassium composition comprises from 1 wppb to 5 wppm 5-chloro-acesulfame potassium.

27. The process of claim **1**, wherein the process yields at least 100 grams of finished acesulfame potassium composition per hour.

28. The process of claim **26**, wherein the reacting is conducted for a cyclization reaction time, from the start of the reactant feed to the end of the reactant feed, less than 35 minutes.

29. The process of claim **2**, wherein the forming of the finished acesulfame potassium composition from the crude acesulfame potassium composition comprises the steps of:

concentrating the crude acesulfame composition to form an intermediate acesulfame potassium composition comprising at least 10 wt % acesulfame potassium; and

separating the intermediate acesulfame potassium composition to form the finished acesulfame potassium composition comprising at least 15 wt % acesulfame potassium.

30. The process of claim **29**, wherein the intermediate acesulfame potassium composition comprises acetoacetamide-N-sulfonic acid and acetoacetamide in an amount ranging from 10 wppb to 25 wppm.

* * * * *

(12) **United States Patent**
Mollenkopf et al.

(10) **Patent No.: US 10,208,004 B2**
(45) **Date of Patent: *Feb. 19, 2019**

(54) **ACESULFAME POTASSIUM COMPOSITIONS AND PROCESSES FOR PRODUCING SAME**

(71) Applicant: **Celanese International Corporation**, Irving, TX (US)

(72) Inventors: **Christoph Mollenkopf**, Frankfurt am Main (DE); **Peter Groer**, Babenhausen (DE); **Arvind Yadav**, Hessen (IN)

(73) Assignee: **Celanese International Corporation**, Irving, TX (US)

(*) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 0 days.

This patent is subject to a terminal disclaimer.

(21) Appl. No.: **16/014,552**

(22) Filed: **Jun. 21, 2018**

(65) **Prior Publication Data**

US 2018/0297970 A1 Oct. 18, 2018

Related U.S. Application Data

(63) Continuation of application No. 15/704,457, filed on Sep. 14, 2017, now Pat. No. 10,023,546.

(60) Provisional application No. 62/397,540, filed on Sep. 21, 2016.

(51) **Int. Cl.**

C07D 291/06	(2006.01)
A23L 27/30	(2016.01)
C07C 235/74	(2006.01)

(52) **U.S. Cl.**

CPC ***C07D 291/06*** (2013.01); ***A23L 27/30*** (2016.08); ***C07C 235/74*** (2013.01); *A23V 2002/00* (2013.01)

(58) **Field of Classification Search**

CPC C07D 209/06; A23L 27/30
USPC 544/200
See application file for complete search history.

(56) **References Cited**

U.S. PATENT DOCUMENTS

4,563,521	A	1/1986	Clauss et al.
4,607,100	A	8/1986	Clauss et al.
4,638,063	A	1/1987	Clauss et al.
4,695,629	A	9/1987	Clauss et al.
4,804,755	A	2/1989	Reuschling et al.
4,806,639	A	2/1989	Reuschling et al.
4,876,341	A	10/1989	Schiitz et al.
5,011,982	A	4/1991	Clauss et al.
5,084,180	A	1/1992	Boateng
5,103,046	A	4/1992	Clauss et al.
5,334,397	A	8/1994	Ream et al.
5,744,010	A	4/1998	Roscher et al.
5,808,159	A	9/1998	Giebeler
7,408,059	B2	8/2008	Kobayashi et al.
7,977,514	B2	7/2011	Peters et al.
8,182,756	B2	5/2012	Liu et al.
8,303,921	B2	11/2012	Brietzke et al.
8,309,048	B2	11/2012	Brietzke et al.
8,496,905	B2	7/2013	Brietzke et al.
8,658,830	B2	2/2014	Brietzke et al.
9,024,016	B2	5/2015	Bayer et al.
10,023,546	B2 *	7/2018	Mollenkopf A23L 27/30
2003/0065172	A1	4/2003	Tian et al.
2003/0065218	A1	4/2003	Mollenkopf
2008/0076919	A1	3/2008	Liu et al.
2009/0318685	A1	12/2009	Saito et al.
2009/0318686	A1	12/2009	Saito et al.
2010/0274057	A1	10/2010	Peters et al.
2011/0256045	A1	10/2011	Brietzke et al.
2011/0256046	A1	10/2011	Brietzke et al.
2013/0062192	A1	3/2013	Brietzke et al.
2013/0331565	A1	12/2013	Bayer et al.
2018/0079733	A1	3/2018	Mollenkopf et al.
2018/0079734	A1	3/2018	Mollenkopf et al.
2018/0079735	A1	3/2018	Mollenkopf et al.

FOREIGN PATENT DOCUMENTS

CA	85104277	6/1985
CA	1273923	9/2009
CN	85104277 A	12/1986
CN	1883790	12/2006
CN	200949088	9/2007
CN	101124981	2/2008
CN	101124981 A	2/2008

(Continued)

OTHER PUBLICATIONS

ASTM E 313-05, Standard Practice for Calculating Yellowness and Whiteness Indices from Instrumentally Measured Color Coordinates, Oct. 1, 2005, 6 pages.

Boehshar et al., 5-Chloroacesulfame K—a characteristic indicator for application of the "sulfur trioxide" process in the manufacture of acesulfame K, Research Disclosure, 2003, 477036.

Mayer et al., Acesulfame-K, Food Science and Technology, Jun. 28, 1991.

International Search Report received in the corresponding International PCT Patent application No. PCT/U52007/051507, dated Nov. 9, 2017.

Linkies et al., Synthesis, 1990, 5, 405-406.

(Continued)

Primary Examiner — Kahsay Habte
(74) *Attorney, Agent, or Firm* — Dority & Manning, P.A.

(57) **ABSTRACT**

Improved processes for producing high purity acesulfame potassium. In one embodiment, the process comprises the steps of contacting a solvent, e.g., dichloromethane, and a cyclizing agent, e.g., sulfur trioxide, to form a cyclizing agent composition and reacting an acetoacetamide salt with the cyclizing agent in the composition to form a cyclic sulfur trioxide adduct. The contact time is less than 60 minutes. The process also comprises forming from the cyclic sulfur trioxide adduct composition a finished acesulfame potassium composition comprising non-chlorinated, e.g., non-chlorinated, acesulfame potassium and less than 35 wppm 5-halo acesulfame potassium, preferably less than 5 wppm.

43 Claims, 1 Drawing Sheet

(56) **References Cited**

FOREIGN PATENT DOCUMENTS

CN	101148300	3/2008
CN	101157666	4/2008
CN	101787001	7/2010
CN	101913898	12/2010
CN	201921689	8/2011
CN	102225333	10/2011
CN	102359926	2/2012
CN	102380226	3/2012
CN	102380266 A	3/2012
CN	202221403 U	5/2012
CN	102866042	1/2013
CN	103018368	4/2013
CN	103130743	6/2013
CN	10331294	10/2013
CN	103570592	2/2014
CN	103570592 A	2/2014
CN	103588728	2/2014
CN	103588728 A	2/2014
CN	103613566	3/2014
CN	103613566 A	3/2014
CN	103960558	8/2014
CN	104193625	12/2014
CN	104209052	12/2014
CN	104225956	12/2014
CN	104292181	1/2015
CN	104292181 A	1/2015
CN	204320227	5/2015
CN	105085160 A	11/2015
CN	105111166 A	12/2015
CN	105152446	12/2015
CN	105198778	12/2015
CN	106262665	1/2017
CN	106267879	1/2017
CN	106346138	1/2017
CN	106349009	1/2017
CN	106349190	1/2017
CN	106349191	1/2017
CN	106349300	1/2017
CN	106365952	2/2017
CN	106496159 A	3/2017
CN	206001191	3/2017
CN	206001201	3/2017
CN	206001439	3/2017
CN	206121215	4/2017
DE	1249262	10/1966
DE	1268141	5/1968
DE	3522470	1/1987
DE	3531357	3/1987
DE	3545196	6/1987
EP	0155634	9/1985
EP	0159516	10/1985
EP	0215347	3/1987
EP	0217024	4/1987
EP	0218076	4/1987
JP	54032406	3/1979
JP	S 5432406 A	3/1979
WO	WO93/19055	9/1993
WO	WO 2011/133468 A1	10/2011
WO	WO2013/182651	12/2013

OTHER PUBLICATIONS

"Commission Direction 95/31/EC of Jul. 5, 1995 laying down specific criteria of purity concerning sweeteners for use in foodstuffs," Official Journal of the European Communities, Jul. 28, 1995, 19 pages.

"Commission Direction 2008/60/EC of Jun. 17, 2008 laying down specific purity criteria concerning sweeteners for use in foodstuffs," Official Journal of the European Union, Jun. 18, 2008, 40 pages.

"Regulations—Commission Regulation (EU) No. 231/2012 of Mar. 9, 2012 laying down specifications for food additives listed in Annexes II and II to Regulation (EC) No. 1333/2008 of the European Parliament and of the Council," Official Journal of the European Union, Mar. 22, 2012, 295 pages.

Opinion—"Re-evaluation of acesulfame K with reference to the previous SCF opinion of 1991," European Commission , Health & Consumer Protection Directorate-General, Directorate B—Scientific Health Opinions, Unit B3—Management of Scientific Committees II, Scientific Committee on Food, Mar. 13, 2000, 8 pages.

Product Information on "Acesulfame Potassium," 2001, 57th JECFA (Joint FAO/WHO Expert Committee on Food Additives), FNP 52 Add 9, 2 pages.

Suenaga, "Ethylene-amine salt recovery—by converting the hydrochloride into the sulphate, and reating with ammonia in aq. Solvent to ppte. Ammonium sulphate", WPI/Thompson, 1979, No. 16, XP 002598345 (See JP54032406).

Sunnett Brochure, "Acesulfame Potassium", Celanese, Apr. 2014.

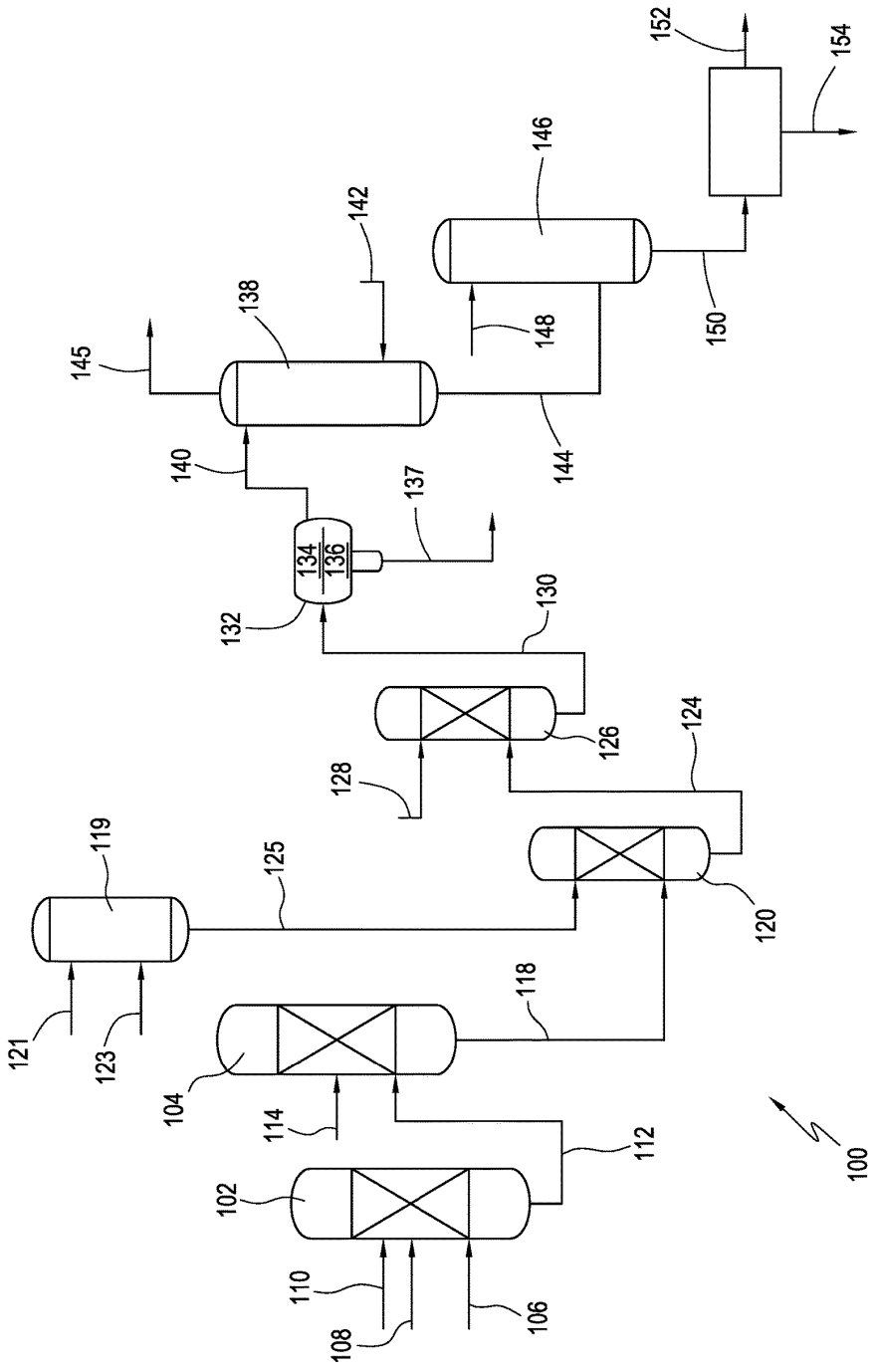
Xiangsheng et al, "Synthesis of Acesulfame Potassium", Fine Chemicals, vol. 13, 1996, 1-15.

Boehshar, Manfred & Burgard, Andreas. (2003). 5-Chloroacesulfame K—a characteristic indicator for application of the "sulfur trioxide" process in the manufacture of acesulfame K. Research Disclosure. 477036.

D. Mayer, et al., Acesulfame-K (Food Science and Technology), 1991, Intro., Chapters 15, 16, and 18, 56 pages.

Information Disclosure Statement submitted Oct. 27, 2017, 2 pages.

* cited by examiner


152
154
146
142
138
145
148
150
144
140
137
134
136
132
130
126
124
128
119
125
120
121
123
118
104
114
112
100
102
110
106
108

ACESULFAME POTASSIUM COMPOSITIONS AND PROCESSES FOR PRODUCING SAME

CROSS-REFERENCE TO RELATED APPLICATIONS

This application is a continuation of U.S. application Ser. No. 15/704,457 having a filing date of Sep. 14, 2017, which claims priority to U.S. Provisional Patent Application No. 62/397,540, filed Sep. 21, 2016, the disclosures of which are incorporated herein by reference in their entirety.

FIELD OF INVENTION

The present invention relates generally to acesulfame potassium and to processes for producing acesulfame potassium. More specifically, the present invention relates to processes for producing high purity acesulfame potassium.

BACKGROUND OF THE INVENTION

Acesulfame potassium has an intense, sweet taste and has been used in many food-related applications as a sweetener. In conventional acesulfame potassium production processes, sulfamic acid and an amine, e.g., triethylamine, are reacted to form an amidosulfamic acid salt, such as a trialkyl ammonium amidosulfamic acid salt. The amidosulfamic acid salt is then reacted with diketene to form an acetoacetamide salt. The acetoacetamide salt may be cyclized, hydrolyzed, and neutralized to form acesulfame potassium. U.S. Pat. Nos. 5,744,010 and 9,024,016 disclose exemplary acesulfame potassium production processes.

Typically, the acetoacetamide salt intermediate is cyclized by reaction with sulfur trioxide in an inorganic or organic solvent to form a cyclic sulfur trioxide adduct. The solvent routinely utilized in this reaction is an organic solvent such as a halogenated, aliphatic hydrocarbon solvent, for example, dichloromethane. The adduct formed by this reaction is subsequently hydrolyzed and then neutralized with potassium hydroxide to form acesulfame potassium.

Acesulfame potassium and the intermediate compositions produced by conventional methods contain undesirable impurities, such as 5-chloro-acesulfame potassium. Limits for the content of various impurities are often set by governmental regulations or customer guidelines. Due to their similar chemical structures and properties, separation of 5-chloro-acesulfame potassium from the desired non-chlorinated acesulfame potassium, using standard purification procedures such as crystallization has proven difficult, resulting in consumer dissatisfaction and the failure to meet regulatory standards.

The need exists for an improved process for producing high purity acesulfame potassium compositions in which the formation of 5-chloro-acesulfame potassium during synthesis is reduced or eliminated.

All of the references discussed herein are hereby incorporated by reference.

SUMMARY OF THE INVENTION

The application discloses processes for producing a finished acesulfame potassium composition, the process comprising the steps of: contacting a solvent and a cyclizing agent to form a cyclizing agent composition, reacting an acetoacetamide salt with the cyclizing agent in the cyclizing agent composition to form a cyclic sulfur trioxide adduct,

and forming from the cyclic sulfur trioxide adduct the finished acesulfame potassium composition comprising non-chlorinated acesulfame potassium and less than 35 wppm 5-chloro-acesulfame potassium, e.g., from 0.001 wppm to 2.7 wppm 5-chloro-acesulfame potassium. Contact time from the beginning of contacting step to the beginning of the reacting step is less than 60 minutes. The forming of the finished acesulfame potassium composition may comprise: hydrolyzing the cyclic sulfur trioxide adduct to form an acesulfame-H composition comprising acesulfame-H, neutralizing the acesulfame-H in the acesulfame-H composition to form a crude acesulfame potassium composition comprising non-chlorinated acesulfame potassium and less than 35 wppm 5-chloro-acesulfame potassium, and forming the finished acesulfame potassium composition from the crude acesulfame potassium composition. The finished acesulfame potassium composition may comprise from 0.001 wppm to 5 wppm 5-chloro-acesulfame potassium. In some cases, the contact time is less than 15 minutes and the crude acesulfame potassium composition comprises from 0.001 wppm to 5 wppm 5-chloro-acesulfame potassium and the finished acesulfame potassium composition comprises from 0.001 wppm to 5 wppm 5-chloro-acesulfame potassium. In one embodiment, the contact time is less than 5 minutes and the crude acesulfame potassium composition comprises from 0.001 wppm to 5 wppm 5-chloro-acesulfame potassium and the finished acesulfame potassium composition comprises from 0.001 wppm to 2.7 wppm 5-chloro-acesulfame potassium. The finished acesulfame potassium composition may comprise at least 90% by weight of the 5-chloro-acesulfame potassium present in the crude acesulfame potassium composition. In some case, the hydrolyzing comprises adding water to the cyclic sulfur trioxide adduct to form a hydrolysis reaction mixture, and wherein the temperature of the hydrolysis reaction mixture is maintained at a temperature ranging from −35° C. to 0° C. The finished acesulfame potassium composition may comprise from 0.001 wppm to 5 wppm organic impurities and/or from 0.001 wppm to 5 wppm heavy metals. Preferably, the process further comprises the steps of reacting sulfamic acid and an amine to form an amidosulfamic acid salt, and reacting the amidosulfamic acid salt and acetoacetylating agent to form the acetoacetamide salt. The cyclizing agent composition may comprise less than 1 wt % of compounds selected from chloromethyl chlorosulfate, methyl-bis-chlorosulfate, and mixtures thereof. The reacting is conducted for a cyclization reaction time, from the start of the reactant feed to the end of the reactant feed, less than 35 minutes. The weight ratio of solvent to cyclizing agent in the cyclizing agent composition may be at least 1:1. The processes may further comprise cooling the cyclizing agent composition to a temperature less than 15° C. Preferably, the cyclizing agent comprises sulfur trioxide and the solvent comprises dichloromethane. In one embodiment, the processes comprise the steps of: reacting sulfamic acid and triethylamine to form an amidosulfamic acid salt, reacting the amidosulfamic acid salt and diketene to form the acetoacetamide salt, contacting dichloromethane and a sulfur trioxide to form a cyclizing agent composition (optionally cooling the cyclizing agent composition to a temperature less than 15° C.), reacting the acetoacetamide salt with the sulfur trioxide in the cyclizing agent composition to form a cyclic sulfur trioxide adduct, hydrolyzing the cyclic sulfur trioxide adduct to form an acesulfame-H composition, and neutralizing the acesulfame-H to form the finished acesulfame potassium composition comprising non-chlorinated acesulfame potassium and less than 10 wppm 5-chloro-acesulfame potassium, and

contact time from the beginning of step (a) to the beginning of step (b) may be less than 10 minutes. The application also describes crude, intermediate, and finished acesulfame potassium composition produced by the processes described herein, e.g., a finished acesulfame potassium composition comprising non-chlorinated acesulfame potassium, from 0.001 wppm to 2.7 wppm 5-chloro acesulfame potassium, and from 0.001 wppm to 5 wppm heavy metals.

BRIEF DESCRIPTION OF THE DRAWINGS

The invention is described in detail below with reference to the appended drawing.

FIG. **1** is a process flow sheet of an acesulfame potassium production process in accordance with one embodiment of the present invention.

DETAILED DESCRIPTION OF THE INVENTION

Introduction

Conventional processes for producing acesulfame potassium involve reacting sulfamic acid and an amine in the presence of acetic acid to form an amidosulfamic acid salt. The amidosulfamic acid salt is then reacted with an acetoacetylating agent, e.g., diketene, to form an acetoacetamide salt. The acetoacetamide salt is reacted with a cyclizing agent, e.g., sulfur trioxide, to form a cyclic sulfur trioxide adduct. The cyclic sulfur trioxide adduct is then hydrolyzed and neutralized via conventional means to form a crude acesulfame potassium composition comprising acesulfame potassium. This composition is phase separated into aqueous and organic phases. Most of the acesulfame potassium separates into the aqueous phase. As used herein, the term "crude acesulfame potassium composition" refers to the initial product of the neutralization reaction or to the aqueous phase that is formed from the phase separation step (without any further purification). The crude acesulfame potassium composition comprises at least 5 wt % acesulfame potassium. The crude acesulfame potassium composition may be optionally treated to form an "intermediate acesulfame potassium composition" and/or a "finished acesulfame potassium composition," which are discussed below.

Conventional acesulfame potassium compositions have been shown to comprise several undesirable impurities, among them 5-chloro-acesulfame potassium and acetoacetamide. Content limits for these compounds in the finished acesulfame potassium composition are often determined by industry purity standards and/or by standards established for particular end use products that utilize acesulfame potassium as a sweetener. In some cases, limits for these impurities are determined by governmental regulations. For most applications, high acesulfame potassium purity levels are preferred. Because the chemical structure of 5-chloro-acesulfame potassium is similar to that of non-chlorinated acesulfame potassium, separation of 5-chloro-acesulfame potassium using standard purification procedures such as crystallization has proven difficult.

Without being bound by theory, it has now been discovered that the reaction of the cyclizing agent with the acetoacetamide salt to form the cyclic sulfur trioxide adduct may also involve side reactions that form the 5-chloro-acesulfame potassium impurity.

The use of specific reaction parameters, however, may advantageously reduce or eliminate 5-chloro-acesulfame potassium formation or the formation of its precursor, 5-chloro-acesulfame-H. In particular, it has now been discovered that limiting contact time, as discussed below, surprisingly reduces or eliminates 5-chloro-acesulfame potassium formation in the crude, intermediate, and/or finished acesulfame potassium compositions. In addition, the reduced impurity levels in these acesulfame potassium compositions reduce or eliminate the need for additional purification steps, resulting in overall improved process efficiency.

It is postulated that the contacting of the cyclizing agent, the solvent, and optionally other components may lead to the formation of chlorine/chloride-containing compounds. Exemplary cyclizing agent/solvent reaction products include halogen-containing compounds such as chlorine/chloride-containing compounds, e.g., chlorosulfates. These compounds, in turn, may react to chlorinate the acesulfame precursor acid, acesulfame-H, sometimes referred to as sweetener acid, or its precursors, e.g., acetoacetamide-N-sulfonate. By limiting the contact time, lower amounts of chlorine/chloride-containing compounds are formed, e.g., chlorosulfates, are formed (as compared to the amount formed when longer contact times are employed). That is, shorter contact times have now been shown to retard the formation of chlorine/chloride-containing compounds, e.g., chlorosulfates. As a result of the shorter contact times, in one embodiment, the cyclizing agent composition may have a low chlorine/chloride-containing compound content, e.g., a low chlorosulfate content, as discussed herein. The reduction or elimination of chlorine/chloride-containing compounds directly leads to the formation of higher purity crude acesulfame potassium compositions discussed herein, thereby simplifying subsequent treatment operations for forming the intermediate or finished acesulfame potassium compositions. The process also advantageously leads to the formation of intermediate and finished acesulfame potassium compositions having low 5-chloro-acesulfame potassium content.

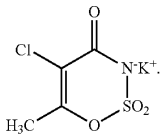
Additional specific terms that are used herein are now defined. "Contact time," as used herein, refers to the time period that the solvent contacts the cyclizing agent before formation of the cyclic sulfur trioxide adduct. Thus, contact time begins when at least some of the solvent contacts at least some the cyclizing agent to form the cyclizing agent/solvent mixture ("cyclizing agent composition"), and contact time ends when the acetoacetamide salt first contacts the cyclizing agent in the cyclizing agent composition.

"Residence time," as used herein, refers to the time period that a composition (or stream) to be treated, e.g., a crude acesulfame potassium composition, remains in a particular treatment operation. Residence time begins when the composition to be treated enters the treatment operation, and residence time ends when the resultant compositions (formed via the treatment) exit the treatment operation. As one particular example, residence time for a concentrating operation, e.g., evaporation, refers to the time from when a crude acesulfame potassium composition enters the evaporator until the intermediate acesulfame potassium composition exits the evaporator. As another example, residence time for a separating operation, e.g., crystallization, refers to the time from when a crude acesulfame potassium composition enters the crystallizer until the intermediate acesulfame potassium composition exits the crystallizer.

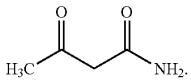
"Cyclization reaction time," as used herein, refers to the time from the start of the acetoacetamide salt feed to the termination of the acetoacetamide salt feed. In some cases, if indicated, the cyclization reaction time may include addi-

tional time past the termination of the acetoacetamide salt feed, e.g., an extra 5 minutes or an extra minute.

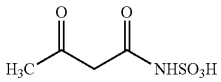
"5-chloro-acesulfame potassium," as used herein, refers to the following molecule:



"Acetoacetamide," as used herein, refers to the following molecule:



"Acetoacetamide-N-sulfonic acid" as used herein, refers to the molecule shown below. In some cases, acetoacetamide-N-sulfonic acid may be a degradation product of acesulfame potassium or acesulfame-H. The term "acetoacetamide-N-sulfonic acid," as used herein, also includes salts of acetoacetamide-N-sulfamic acid, e.g., potassium, sodium, and other alkali metal salts.



An "intermediate acesulfame potassium composition" refers to a composition resulting from the concentrating of the crude acesulfame potassium composition, e.g., the removal of water from the crude acesulfame potassium composition. The intermediate acesulfame potassium composition comprises at least 10 wt % acesulfame potassium, based on the total weight of the intermediate acesulfame potassium composition, and has an acesulfame potassium weight percentage that is higher than that of the crude acesulfame potassium composition.

A "finished acesulfame potassium composition" refers to a composition (preferably directly) resulting from the separating, e.g., crystallizing and/or filtering, of the intermediate acesulfame potassium composition, e.g. no further process steps are preferably conducted after the separating of the intermediate acesulfame potassium composition in order to obtain the finished acesulfame potassium composition. The finished acesulfame potassium composition comprises at least 15 wt % acesulfame potassium, based on the total weight percentage of the finished acesulfame potassium composition, and has an acesulfame potassium weight percentage that is higher than that of the intermediate acesulfame potassium composition.

"Wppm" and "wppb," as used herein, mean weight parts per million or weight parts per billion, respectively. These are based on the total weight of the respective composition, e.g., the total weight of the entire crude acesulfame potassium composition or the entire finished acesulfame potassium composition.

Acesulfame Potassium Formation (Contact Time)

Processes for producing acesulfame potassium exhibiting high purity levels are described herein. In one embodiment, the process comprises the steps of contacting a solvent and a cyclizing agent to form a cyclizing agent composition and reacting an acetoacetamide salt with the cyclizing agent (in the cyclizing agent composition) to form a cyclic sulfur trioxide adduct. Importantly, the contact time is less than 60 minutes. The process also comprises forming a finished acesulfame potassium composition from the cyclic sulfur trioxide adduct composition.

The contacting of the solvent and the cyclizing agent is contemplated broadly. In some embodiments, the contacting methods include the addition of solvent to cyclizing agent, the addition of cyclizing agent to solvent. The components may be fed, e.g., simultaneously fed to a vessel. Addition/mixing and/or co-feeding (optionally simultaneous) of these components are contemplated.

The reaction of the acetoacetamide salt and the cyclizing agent may be conducted by contacting the two reactants. The reactants may be fed, e.g., simultaneously fed to a vessel. In one embodiment, the acetoacetamide salt may be added to the cyclizing agent in the cyclizing agent composition. The cyclizing agent in the cyclizing agent composition may be added to the acetoacetamide salt. Addition/mixing and/or co-feeding (optionally simultaneous) of the reactants are also contemplated. In one embodiment, the cyclizing agent composition may be contained in a vessel and the acetoacetamide salt may be added to the cyclizing agent composition, e.g., added drop-wise to the cyclizing agent composition.

In some embodiments, contact time is less than 60 minutes, e.g., less than 45 minutes, less than 30 minutes, less than 15 minutes, less than 10 minutes, less than 8 minutes, less than 5 minutes, less than 3 minutes, or less than 1 minute. In one embodiment, the solvent and cyclizing agent are mixed and immediately reacted with the acetoacetamide salt. In terms of ranges, contact time may range from 1 second to 60 minutes, e.g., from 1 second to 45 minutes, from 1 second to 30 minutes, from 1 second to 15 minutes, from 1 second to 10 minutes, from 1 minute to 45 minutes, from 1 minute to 30 minutes, from 1 minute to 15 minutes, from 1 minute to 10 minutes, from 10 seconds to 45 minutes, from 10 seconds to 30 minutes, from 30 seconds to 30 minutes, from 1 minute to 10 minutes, from 3 minutes to 10 minutes, or from 5 minutes to 10 minutes. Contact time may be at least 1 second, e.g., at least 5 seconds, at least 30 seconds, at least 1 minute, at least 5 minutes, at least 10 minutes, at least 15 minutes, or at least 30 minutes.

By limiting the contact time as discussed herein, fewer cyclizing agent/solvent reaction products, e.g., chlorosulfates, are formed. The cyclizing agent composition, for example, may have a low cyclizing agent/solvent reaction product content, e.g., a low chlorosulfate content. For example, the cyclizing agent composition may comprise less than 1 wt % cyclizing agent/solvent reaction product, e.g., less than 0.75 wt %, less than 0.5 wt %, less than 0.25 wt %, less than 0.1 wt %, less than 0.05 wt %, or less than 0.01 wt %. In terms of ranges, the cyclizing agent composition may comprise from 1 ppm to 1 wt % cyclizing agent/solvent reaction products, e.g., from 10 ppm to 1 wt %, from 10 ppm to 0.75 wt %, from 10 ppm to 0.5 wt %, from 10 ppm to 0.25 wt %, from 100 ppm to 0.75 wt %, from 100 ppm to 0.5 wt %, or from 100 ppm to 0.25 wt %. These ranges and limits apply to cyclizing agent/solvent reaction products generally

and to specific reaction products generally, e.g., chloromethyl chlorosulfate, methyl-bis-chlorosulfate, and combinations thereof.

Exemplary chlorosulfates include chloromethyl chlorosulfate and methyl-bis-chlorosulfate. These reaction products may be formed when a chlorine-containing solvent is employed. In one embodiment, the cyclizing agent composition comprises less than 1 wt % chloromethyl chlorosulfate and/or methyl-bis-chlorosulfate, e.g., less than 0.75 wt %, less than 0.5 wt %, less than 0.25 wt %, less than 0.1 wt %, less than 0.05 wt %, or less than 0.01 wt %. In one embodiment, the cyclizing agent composition comprises less than 1 wt % chloromethyl chlorosulfate, e.g., less than 0.75 wt %, less than 0.5 wt %, less than 0.25 wt %, less than 0.1 wt %, less than 0.05 wt %, or less than 0.01 wt %. In one embodiment, the cyclizing agent composition comprises less than 1 wt % methyl-bis-chlorosulfate, e.g., less than 0.75 wt %, less than 0.5 wt %, less than 0.25 wt %, less than 0.1 wt %, less than 0.05 wt %, or less than 0.01 wt %.

In one embodiment, the solvent and cyclizing agent are combined in a first vessel, e.g., a first reactor, to form a cyclizing agent composition, which optionally may be cooled. The cyclizing agent composition may then be added to the acetoacetamide salt in a second reactor. In one embodiment, the first vessel is chilled, e.g., to temperature below 35° C., prior to combining the solvent and cyclizing agent. In some cases, the cyclizing agent and the solvent are cooled individually and then fed to the reaction with the acetoacetamide salt, optionally followed by additional cooling. In one embodiment, the first vessel itself is chilled, e.g., to a temperature below 15° C., prior to contacting the solvent and cyclizing agent, which leads to the cooling of the solvent and the cyclization that may be added to the first vessel. In some cases, the cyclizing agent and the solvent are cooled individually and then combined and fed to the reaction with the acetoacetamide salt.

In some cases, the process comprises the steps of providing a cyclic sulfur trioxide adduct composition comprising less than 1 wt % cyclizing agent/solvent reaction products, e.g., chloromethyl chlorosulfate and/or methyl-bis-chlorosulfate, and forming the acesulfame potassium composition from the cyclic sulfur trioxide adduct composition. The providing of the cyclic sulfur trioxide adduct composition may vary widely as long as the cyclic sulfur trioxide adduct composition has the required cyclizing agent/solvent reaction product content. The cyclic sulfur trioxide adduct composition optionally is formed using any of the methods described herein.

In some embodiments, the cyclizing agent composition is provided at a low temperature and/or is cooled to yield a cooled cyclizing agent composition having a low temperature. The cooling or the providing of the low temperature cyclizing agent composition may be achieved through any of a variety of different cooling techniques. For example, the cooling step may be achieved by using one or more heat exchangers, refrigeration units, air cooling units, water cooling units, or a cooling medium, such as liquid nitrogen or other cryogenics. If heat exchangers are employed, a water/glycol mixture is a preferable exchange medium, with brine being a suitable alternative.

In some embodiments, the cyclizing agent composition is provided at or is cooled to a temperature less than 15° C., e.g., less than 12° C., less than 11° C., less than 10° C., less than 8° C., less than 5° C., less than 3° C., less than 1° C., or less than 0° C. In terms of ranges, the cyclization agent composition is cooled to a temperature ranging from −20° C. to 15° C., e.g., from −15° C. to 15° C., from −10° C. to 12° C., from −8° C. to 10° C., or −8° C. to 5° C. In some embodiments, the cooling step reduces the temperature of the cyclizing agent composition (as provided), e.g., by at least 2° C., at least 3° C., at least 5° C., at least 10° C., at least 15° C., at least 20° C., or at least 25° C.

In one embodiment, only the cyclizing agent (e.g., without solvent) is cooled, and then the cooled cyclizing agent is mixed with the solvent to form the cyclizing agent composition, which is then reacted with the acetoacetamide salt. That is, in some cases, the solvent (if present) may not be cooled in the same manner as the cyclizing agent is cooled. In other embodiments, the solvent is cooled prior to being mixed with the cyclizing agent to form the cyclizing agent composition, optionally followed by additional cooling of the resulting cyclizing agent composition.

In some cases, the cooling is implemented via multiple cooling steps. For example, the solvent may be cooled to a first temperature, then combined with the cyclizing agent to form the cyclizing agent composition, which is then further cooled to a second temperature, which is less than the first temperature. In some embodiments, the cyclizing agent is cooled to a first temperature, the solvent is cooled to a second temperature, and the cooled cyclizing agent and the cooled solvent are combined and optionally cooled to a third temperature, which is less than the first and second temperatures. These cooling schemes are merely exemplary and are not intended to limit the scope of the cooling step.

It has also been discovered that if cyclization reaction time is minimized, the formation of impurities, e.g., organic impurities, such as 5-chloro-acesulfame potassium, is reduced or eliminated. In some embodiments, the cyclization reaction is conducted for a cyclization reaction time, less than 35 minutes, e.g., less than 30 minutes, less than 25 minutes, less than 20 minutes, less than 15 minutes, or less than 10 minutes. In terms of ranges, the cyclization reaction may be conducted for a cyclization reaction time ranging from 1 second to 35 minutes, e.g., from 10 seconds to 25 minutes, from 30 seconds to 15 minutes, or from 1 minute to 10 minutes.

The cyclic sulfur trioxide adduct may be subjected to one or more steps to form the finished acesulfame potassium composition. In some cases, the formation of the finished acesulfame potassium composition comprises the steps of hydrolyzing (at least some of) the cyclic sulfur trioxide adduct to form an acesulfame-H composition comprising acesulfame-H and neutralizing the acesulfame-H in the acesulfame-H composition to form a crude acesulfame potassium composition.

Crude acesulfame compositions may be treated to form intermediate acesulfame potassium compositions and (subsequently) finished acesulfame compositions, and this treatment operation may include one or more concentrating or separating operations.

For example, the treatment operation may comprise concentrating the crude acesulfame potassium composition to form a water stream and an intermediate acesulfame potassium composition and then separating the intermediate acesulfame potassium composition to form the finished acesulfame potassium composition comprising acesulfame potassium, e.g., via filtration and/or crystallization.

Acesulfame Potassium Compositions

The crude acesulfame potassium composition is formed by hydrolyzing the cyclic sulfur trioxide adduct to form an acesulfame-H composition and neutralizing the acesulfame-H in the acesulfame-H composition to form the crude acesulfame potassium composition, as discussed herein. The product of the neutralization step is phase separated into

aqueous and organic phases. The crude acesulfame potassium composition may be obtained from the aqueous phase (without any further purification). The crude acesulfame potassium composition preferably comprises a mixture of acesulfame potassium, e.g., non-chlorinated acesulfame potassium, and less than 35 wppm 5-chloro-acesulfame potassium, e.g., less than 30 wppm, less than 25 wppm, less than 20 wppm, less than 15 wppm, less than 12 wppm, less than 10 wppm, less than 7 wppm, less than 5 wppm, less than 3 wppm, or less than 1 wppm. In some cases the crude acesulfame potassium composition is free of 5-chloro-acesulfame potassium, e.g., substantially free of 5-chloro-acesulfame potassium (undetectable). In terms of ranges, the crude acesulfame potassium composition may comprise from 1 wppb to 35 wppm 5-chloro-acesulfame potassium, e.g., from 1 wppb to 20 wppm, from 1 wppb to 10 wppm, from 1 wppb to 5 wppm, from 1 wppb to 2.7 wppm, from 10 wppb to 20 wppm, from 10 wppb to 19 wppm, from 10 wppb to 15 wppm, from 10 wppb to 12 wppm, from 10 wppb to 10 wppm, from 10 wppb to 5 wppm, from 100 wppb to 15 wppm, from 100 wppb to 10 wppm, or from 100 wppb to 5 wppm.

The finished acesulfame potassium compositions, which are typically suitable for end consumer usage, are formed by treating the crude acesulfame potassium composition to remove impurities, as discussed herein. This finished acesulfame potassium composition preferably comprises a mixture of acesulfame potassium, e.g., non-chlorinated acesulfame potassium, and less than 35 wppm 5-chloro-acesulfame potassium, e.g., less than 30 wppm, less than 25 wppm, less than 20 wppm, less than 15 wppm, less than 12 wppm, less than 10 wppm, less than 7 wppm, less than 5 wppm, less than 3 wppm, or less than 1 wppm. In some cases the finished acesulfame potassium composition is free of 5-chloro-acesulfame potassium, e.g., substantially free of 5-chloro-acesulfame potassium (undetectable). In terms of ranges, the finished acesulfame potassium composition may comprise from 1 wppb to 35 wppm 5-chloro-acesulfame potassium, e.g., from 1 wppb to 20 wppm, from 1 wppb to 10 wppm, from 1 wppb to 5 wppm, from 1 wppb to 2.7 wppm, from 10 wppb to 20 wppm, from 10 wppb to 19 wppm, from 10 wppb to 15 wppm, from 10 wppb to 12 wppm, from 10 wppb to 10 wppm, from 10 wppb to 5 wppm, from 100 wppb to 15 wppm, from 100 wppb to 10 wppm, or from 100 wppb to 5 wppm. The shorter contact times reduce or eliminate 5-chloro-acesulfame potassium formation, resulting in both a crude acesulfame potassium composition and a finished acesulfame potassium composition having low 5-chloro-acesulfame potassium content.

In some embodiments, the finished acesulfame potassium compositions comprise acesulfame potassium and less than 33 wppm acetoacetamide, e.g., less than 32 wppm, less than 30 wppm, less than 25 wppm, less than 20 wppm, less than 15 wppm, less than 12 wppm, less than 10 wppm, less than 7 wppm, less than 5 wppm, less than 3 wppm, less than 1 wppm, less than 0.8 wppm, less than 0.5 wppm, or less than 0.3 wppm. In some cases the finished acesulfame potassium composition is free of acetoacetamide, e.g., substantially free of acetoacetamide (undetectable). In terms of ranges, the finished acesulfame potassium composition may comprise from 1 wppb to 33 wppm acetoacetamide, e.g., from 10 wppb to 32 wppm, from 10 wppb to 25 wppm, from 10 wppb to 15 wppm, from 10 wppb to 12 wppm, from 10 wppb to 10 wppm, from 10 wppb to 7 wppm, from 10 wppb to 5 wppm, from 10 wppb to 3 wppm, from 100 wppb to 15 wppm, from 100 wppb to 10 wppm, or from 100 wppb to 5 wppm. In some cases, acetoacetamide-N-sulfonic acid may also be present in the finished acesulfame potassium compositions in the aforementioned amounts. These impurities may be formed by side reactions and degradation of the acesulfame potassium and acesulfame-H molecules, e.g., during treatment of the specific crude acesulfame potassium compositions discussed herein.

The 5-chloro-acesulfame potassium content may be measured in the crude and/or finished acesulfame potassium compositions (as well as any intermediate compositions) via high performance liquid chromatography (HPLC) analysis, based on European Pharmacopoeia guidelines (2017), based on European Pharmacopoeia guidelines for thin layer chromatography (2017) and adapted for HPLC. A particular measurement scenario utilizes an LC Systems HPLC unit from Shimadzu having a CBM-20 Shimadzu controller and being equipped with a CC 250/4.6 Nucleodur 100-3 C18 ec (250×4.6 mm) MACHEREY NAGEL column. A Shimadzu SPD-M20A photodiode array detector can be used for detection (at 234 nm wavelength). Analysis may be performed at 23° C. column temperature. As an eluent solution, an aqueous solution of tetra butyl ammonium hydrogen sulfate (optionally at 3.4 g/L and at 60% of the total solution) and acetonitrile (optionally at 300 mL/L and at 40% of the total solution) may be employed. Elution may be isocratic. The overall flow rate of total eluent may be approximately 1 mL/min. The data collection and calculations may be performed using Lab Solution software from Shimadzu.

The acetoacetamide-N-sulfonic acid and/or the acetoacetamide content may be measured in the crude, intermediate, or finished acesulfame potassium compositions via HPLC analysis, based on European Pharmacopoeia guidelines for thin layer chromatography (2017) and adapted for HPLC. A particular measurement scenario utilizes an LC Systems HPLC unit from Shimadzu having a CBM-20 Shimadzu controller and being equipped with an IonPac NS1 ((5 μm) 150×4 mm) analytical column and an IonPac NG1 guard column (35×4.0 mm). A Shimadzu SPD-M20A photodiode array detector can be used for detection (at 270 nm and 280 nm wavelength). Analysis may be performed at 23° C. column temperature. As a first eluent solution, an aqueous mixture of tetra butyl ammonium hydrogen sulfate (3.4 g/L), acetonitrile (300 mL/L), and potassium hydroxide (0.89 g/L) may be employed; as a second eluent solution, an aqueous mixture of tetra butyl ammonium hydrogen sulfate (3.4 g/L) and potassium hydroxide (0.89 g/L) may be employed. Elution may be conducted in gradient mode according to the following second eluent flow profile:

0 to 3 minutes: constant 80% (v/v)
3 to 6 minutes: linear reduction to 50% (v/v)
6 to 15 minutes: constant at 50% (v/v)
15 to 18 minutes: linear reduction to 0%
18 to 22 minutes: constant at 0%
22 to 24 minutes: linear increase to 80% (v/v)
24 to 35 minutes constant at 80% (v/v).

Overall flow rate of eluent may be approximately 1.2 mL/min. The data collection and calculations may be performed using Lab Solution software from Shimadzu.

As noted above, the crude acesulfame potassium composition is formed by the aforementioned contacting of the solvent and the cyclizing agent to form a cyclizing agent composition; cyclic sulfur trioxide adduct composition formation reaction, and forming from the cyclic sulfur trioxide adduct the finished acesulfame potassium composition (for example via hydrolysis, neutralization, and treatment). In preferred embodiments, the contact time may be less than 60 minutes, e.g., less than 45 minutes, less than 30 minutes, less than 15 minutes, less than 10 minutes, less than 8 minutes,

less than 5 minutes, less than 3 minutes, or less than 1 minute (optionally ranging from 1 second to 60 minutes, e.g., from 1 second to 45 minutes, from 1 second to 30 minutes, from 1 second to 15 minutes, from 1 second to 10 minutes, from 1 minute to 45 minutes, from 1 minute to 30 minutes, from 1 minute to 15 minutes, from 1 minute to 10 minutes, from 10 seconds to 45 minutes, from 10 seconds to 30 minutes, from 30 seconds to 30 minutes, from 1 minute to 10 minutes, from 3 minutes to 10 minutes, or from 5 minutes to 10 minutes); the crude acesulfame potassium composition may comprise from 1 wppb to 35 wppm 5-chloro-acesulfame potassium, e.g., from 1 wppb to 20 wppm, from 1 wppb to 10 wppm, from 1 wppb to 5 wppm, from 1 wppb to 2.7 wppm, from 10 wppb to 20 wppm, from 10 wppb to 19 wppm, from 10 wppb to 15 wppm, from 10 wppb to 12 wppm, from 10 wppb to 10 wppm, from 10 wppb to 5 wppm, from 100 wppb to 15 wppm, from 100 wppb to 10 wppm, or from 100 wppb to 5 wppm (optionally less than 35 wppm 5-chloro-acesulfame potassium, e.g., less than 30 wppm, less than 25 wppm, less than 20 wppm, less than 15 wppm, less than 12 wppm, less than 10 wppm, less than 7 wppm, less than 5 wppm, less than 3 wppm, or less than 1 wppm); and the finished acesulfame potassium composition may comprise from 1 wppb to 35 wppm 5-chloro-acesulfame potassium, e.g., from 1 wppb to 20 wppm, from 1 wppb to 10 wppm, from 1 wppb to 5 wppm, from 1 wppb to 2.7 wppm, from 10 wppb to 20 wppm, from 10 wppb to 19 wppm, from 10 wppb to 15 wppm, from 10 wppb to 12 wppm, from 10 wppb to 10 wppm, from 10 wppb to 5 wppm, from 100 wppb to 15 wppm, from 100 wppb to 10 wppm, or from 100 wppb to 5 wppm (optionally less than 35 wppm 5-chloro-acesulfame potassium, e.g., less than 30 wppm, less than 25 wppm, less than 20 wppm, less than 15 wppm, less than 12 wppm, less than 10 wppm, less than 7 wppm, less than 5 wppm, less than 3 wppm, or less than 1 wppm).

In a particular embodiment, the contact time is less than 15 minutes, the crude acesulfame potassium composition comprises from 0.001 wppm to 5 wppm 5-chloro-acesulfame potassium, and the finished acesulfame potassium composition comprises from 0.001 wppm to 5 wppm 5-chloro-acesulfame potassium.

In another particular embodiment, the contact time is less than 5 minutes, the crude acesulfame potassium composition comprises from 0.001 wppm to 5 wppm 5-chloro-acesulfame potassium, and the finished acesulfame potassium composition comprises from 0.001 wppm to 2.7 wppm 5-chloro-acesulfame potassium.

In another particular embodiment, the contact time ranges from 1 second to 10 minutes, the crude acesulfame potassium composition comprises from 1 wppb to 35 wppm 5-chloro-acesulfame potassium, and the finished acesulfame potassium composition comprises from 1 wppb to 35 wppm 5-chloro-acesulfame potassium.

In another particular embodiment, the contact time ranges from 1 second to 10 minutes, the crude acesulfame potassium composition comprises from 1 wppb to 5 wppm 5-chloro-acesulfame potassium, and the finished acesulfame potassium composition comprises from 1 wppb to 5 wppm 5-chloro-acesulfame potassium.

In another particular embodiment, the contact time ranges from 1 second to 30 minutes, the crude acesulfame potassium composition comprises from 10 wppb to 10 wppm 5-chloro-acesulfame potassium, and the finished acesulfame potassium composition comprises from 10 wppb to 10 wppm 5-chloro-acesulfame potassium.

The acesulfame potassium compositions (crude and/or finished) may, in some cases, comprise other impurities. Exemplary impurities include, inter alia, acetoacetamide, acetoacetamidesulfonate, and acetoacetamide-N-sulfonic acid. The acesulfame potassium compositions (crude and/or finished) also may comprise heavy metals. The organic impurities and/or heavy metals may be present in an amount ranging from 1 wppb to 25 wppm, based on the total weight of the respective acesulfame potassium composition, crude or finished, e.g., from 100 wppb to 20 wppm, from 100 wppb to 15 wppm, from 500 wppb to 10 wppm, or from 1 wppm to 5 wppm. Heavy metals are defined as metals with relatively high densities, e.g., greater than 3 g/cm^3 or greater than 7 g/cm^3. Exemplary heavy metals include lead and mercury. In some cases, the crude or finished acesulfame potassium composition may comprise mercury in an amount ranging from 1 wppb to 25 wppm, e.g., from 100 wppb to 20 wppm, from 100 wppb to 15 wppm, from 500 wppb to 10 wppm, or from 1 wppm to 5 wppm. In terms of limits, the crude or finished acesulfame potassium composition may comprise less than 25 wppm mercury, e.g., less than 20 wppm, less than 15 wppm, less than 10 wppm, or less than 5 wppm. In some cases, the crude or finished acesulfame potassium composition may comprise lead in an amount ranging from 1 wppb to 25 wppm, e.g., from 100 wppb to 20 wppm, from 100 wppb to 15 wppm, from 500 wppb to 10 wppm, or from 1 wppm to 5 wppm. In terms of limits, the crude or finished acesulfame potassium composition may comprise less than 25 wppm lead, e.g., less than 20 wppm, less than 15 wppm, less than 10 wppm, or less than 5 wppm. In some cases, when potassium hydroxide is formed via a membrane process, the resultant crude or finished acesulfame potassium composition may have very low levels of mercury, if any, e.g., less than 10 wppm, less than 5 wppm, less than 3 wppm, less than 1 wppm, less than 500 wppb, or less than 100 wppb.

In some embodiments, the acesulfame potassium compositions (crude, intermediate, and/or finished) may comprise acetoacetamide-N-sulfonic acid, e.g., less than 37 wppm acetoacetamide-N-sulfonic acid, e.g., less than 35 wppm, less than 30 wppm, less than 25 wppm, less than 20 wppm, less than 15 wppm, less than 12 wppm, less than 10 wppm, less than 7 wppm, less than 5 wppm, less than 3 wppm, less than 1 wppm, less than 0.8 wppm, less than 0.5 wppm, or less than 0.3 wppm. In some cases the finished acesulfame potassium composition is substantially free of acetoacetamide-N-sulfonic acid, e.g., free of acetoacetamide-N-sulfonic acid. In terms of ranges, the finished acesulfame potassium composition may comprise from 1 wppb to 37 wppm acetoacetamide-N-sulfonic acid, e.g., from 10 wppb to 35 wppm, from 10 wppb to 25 wppm, from 10 wppb to 15 wppm, from 10 wppb to 12 wppm, from 10 wppb to 10 wppm, from 10 wppb to 7 wppm, from 10 wppb to 5 wppm, from 10 wppb to 3 wppm, from 100 wppb to 15 wppm, from 100 wppb to 10 wppm, or from 100 wppb to 5 wppm. Acetoacetamide-N-sulfonic acid may be formed in side reactions. The use of the aforementioned temperature (and optionally contact time) parameters also provide for low amounts of acetoacetamide-N-sulfonic acid.

In some embodiments, the crude acesulfame potassium composition is treated to achieve the finished acesulfame potassium composition. In some cases, however, treatment steps may not provide for removal of 5-chloro-acesulfame potassium, perhaps due to the chemical similarities of 5-chloro-acesulfame potassium and acesulfame potassium. Surprisingly, the use of the process steps disclosed herein advantageously provides for the reduction or elimination of

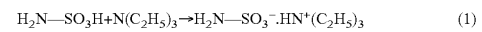
impurities during the reaction scheme, before purification of the crude acesulfame potassium composition. Accordingly, the need to rely on purification of the crude acesulfame potassium composition to remove 5-chloro-acesulfame potassium is beneficially reduced. In some embodiments, the acesulfame potassium compositions (crude and/or finished) comprise at least 90% of the 5-chloro-acesulfame potassium present the crude acesulfame potassium composition, e.g., at least 93%, at least 95%, or at least 99%.

Intermediate Reaction Parameters

The reactions for production of high purity acesulfame potassium are described in more detail as follows.

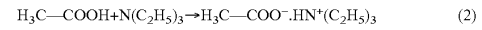
Amidosulfamic Acid Salt Formation Reaction

In a first reaction step, sulfamic acid and an amine are reacted to form sulfamic acid salt. An exemplary reaction scheme that employs triethylamine as the amine and yields triethyl ammonium sulfamic acid salt is shown in reaction (1), below.



(1)

Acetic acid is also present in the first reaction mixture and reacts with the amine, e.g., triethylamine, to form an ammonium acetate, e.g., triethylammonium acetate, as shown in reaction (2), below.



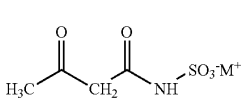
(2)

The amine employed in these reactions may vary widely. Preferably, the amine comprises triethylamine. In one embodiment, the amine may be selected from the group consisting of trimethylamine, diethylpropylamine, tri-n-propylamine, triisopropylamine, ethyldiisopropylamine, tri-n-butylamine, triisobutylamine, tricyclohexylamine, ethyldicyclohexylamine, N,N-dimethylaniline, N,N-diethylaniline, benzyldimethylamine, pyridine, substituted pyridines such as picoline, lutidine, cholidine or methylethylpyridine, N-methylpiperidine, N-ethylpiperidine, N-methylmorpholine, N,N-dimethylpiperazine, 1,5-diazabicyclo[4.3.0]-non-5-en, 1,8-diazabicyclo-[5.4.0]-undec-7-en, 1,4-diazabicyclooctane, tetramethylhexamethylendiamine, tetramethylethylendiamine, tetramethylpropylendiamine, tetramethylbutylendiamine, 1,2-dimorpholylethan, pentamethyldiethyltriamine, pentaethyldiethylentriamine, pentamethyldipropylentriamine, tetramethyldiaminomethane, tetrapropyldiaminomethane, hexamethyltriethylentetramine, hexamethyltripropylenetetramine, diisobutylentriamine, triisopropylentriamine, and mixtures thereof.

Acetoacetamide Salt Formation Reaction

Once formed in reaction (1), the sulfamic acid salt is reacted with the acetoacetylating agent to form the acetoacetamide salt, preferably acetoacetamide-N-sulfonate triethylammonium salt. Preferably, the acetoacetylating agent comprises diketene, although other acetoacetylating agents may be employed, either with or without diketene.

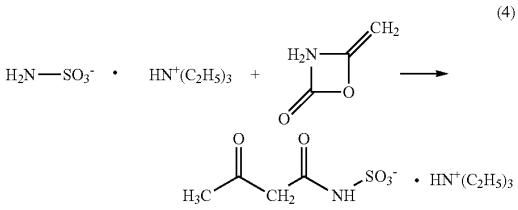
In one embodiment, the resultant acetoacetamide salt corresponds to the following formula (3).



(3)

wherein M^+ is an appropriate ion. Preferably, M^+ is an alkali metal ion or $N^+ R_1R_2R_3R_4$. R_1, R_2, R_3 and R_4, independently of one another, may be organic radicals or hydrogen, preferably H or C_1-C_8 alkyl, C_6-C_{10} cycloalkyl, aryl and/or aralkyl. In a preferred embodiment, R_1 is hydrogen, and R_2, R_3 and R_4 are alkyl, e.g., ethyl.

An exemplary reaction scheme for forming an acetoacetamide salt employs a trialkyl ammonium amidosulfamic acid salt and diketene as reactants and yields an acetoacetamide triethylammonium salt is shown in reaction (4), below.



(4)

In one embodiment, the reaction is conducted in the presence of a catalyst, which may vary widely. In some embodiments, the catalyst comprises one or more amines and/or phosphines. Preferably, the catalyst comprises triethylamine. In some cases trimethylamine serves as both a catalyst and a reactant.

In one embodiment wherein the amidosulfamic acid salt formation reaction and the acetoacetamide salt formation reaction are conducted in separate reactors, a second reaction mixture comprises the amidosulfamic acid salt, the diketene, and the catalyst, e.g., triethylamine. Preferably, catalyst from the first reaction is carried through to the reaction mixture of the second reaction. The second reaction mixture is then subjected to conditions effective to form the acetoacetamide salt.

In one embodiment, the composition of the second reaction mixture may be similar to that of the first reaction mixture. In a preferred embodiment, the reaction product of the amidosulfamic acid salt formation reaction provides the amidosulfamic acid salt component of the second reaction mixture. In addition to the above-mentioned components, the second reaction mixture may further comprise reaction by-products from the first reaction or non-reacted starting materials.

In one embodiment, the amount of acetoacetylating agent, e.g., diketene, should be at least equimolar to the reactant amidosulfamic acid salt that is provided. In one embodiment, the process may utilize a diketene in excess, but preferably in an excess less than 30 mol %, e.g., less than 10 mol %. Greater excesses are also contemplated.

The amidosulfamic acid salt formation reaction and/or the acetoacetamide salt formation reaction may employ an organic solvent. Suitable inert organic solvents include any organic solvents that do not react in an undesired manner with the starting materials, cyclizing agent, final products and/or the catalysts in the reaction. The solvents preferably have the ability to dissolve, at least partially, amidosulfamic acid salts. Exemplary organic solvents include halogenated aliphatic hydrocarbons, preferably having up to 4 carbon atoms such as, for example, methylene chloride, chloroform, 1,2-dichlorethane, trichloroethylene, tetrachloroethylene, trichlorofluoroethylene; aliphatic ketones, preferably those having 3 to 6 carbon atoms such as, for example, acetone, methyl ethyl ketone; aliphatic ethers, preferably cyclic aliphatic ethers having 4 or 5 carbon atoms such as, for example, tetrahydrofuran, dioxane; lower aliphatic carboxylic acids, preferably those having 2 to 6 carbon atoms such

as, for example, acetic acid, propionic acid; aliphatic nitriles, preferably acetonitrile; N-alkyl-substituted amides of carbonic acid and lower aliphatic carboxylic acids, preferably amides having up to 5 carbon atoms such as, for example, tetramethylurea, dimethylformamide, dimethylacetamide, N-methylpyrrolidone; aliphatic sulfoxides, preferably dimethyl sulfoxide, and aliphatic sulfones, preferably sulfolane.

Particularly preferred solvents include dichloromethane (methylene chloride), 1,2-dichloroethane, acetone, glacial acetic acid and dimethylformamide, with dichloromethane (methylene chloride) being particularly preferred. The solvents may be used either alone or in a mixture. In one embodiment, the solvent is a halogenated, aliphatic hydrocarbon solvent, preferably the solvent is dichloromethane. Chloroform and tetrachloromethane are also exemplary solvents.

In one embodiment, the acetoacetamide salt formation reaction is conducted a temperature ranging from −30° C. to 50° C., e.g., from 0° C. to 25° C. The reaction pressure may vary widely. In preferred embodiments, the reaction is carried out at atmospheric pressure, although other pressures are also contemplated. The reaction time may vary widely, preferably ranging from 0.5 hours to 12 hours, e.g., from 1 hour to 10 hours. In one embodiment, the reaction is carried out by introducing the amidosulfamic acid salt and metering in the diketene. In another embodiment, the reaction is carried out by introducing diketene and metering in the amidosulfamic acid salt. The reaction may be carried out by introducing the diketene and amidosulfamic acid and metering in the catalyst.

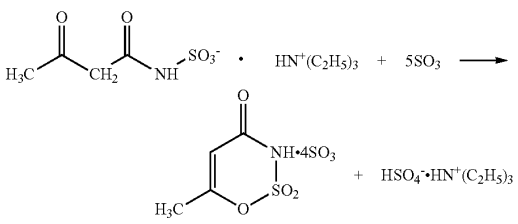
Once formed, each reaction product is optionally subjected to one or more purification steps. For example, the solvent may be separated from the reaction product, e.g., via distillation, and the residue (mainly acetoacetamide-N-sulfonate) may be recrystallized from a suitable solvent such as, for example, acetone, methyl acetate or ethanol.

Generally speaking, the steps of reacting sulfamic acid and triethylamine to form an amidosulfamic acid salt, reacting the amidosulfamic acid salt and diketene to form the acetoacetamide salt, and contacting dichloromethane and a sulfur trioxide to form a cyclizing agent composition are performed in no particular order. Each of these steps may be performed independently of one another. In some cases, these steps may be performed in any order as long as they are performed before the cyclization reaction, e.g., the reaction of the acetoacetamide salt with sulfur trioxide to form a cyclic sulfur trioxide adduct.

Cyclization and Hydrolyzation

The acetoacetamide salt is reacted with cyclizing agent, e.g., cyclizing agent in the cyclizing agent composition, in the presence of a solvent to form the cyclic (sulfur trioxide) adduct composition, which contains cyclic sulfur trioxide adduct and, in some cases, impurities. In some cases, a cooling step occurs before the cyclic sulfur trioxide adduct formation reaction. In one embodiment, the cyclization is achieved by using at least an equimolar amount of the cyclizing agent. The cyclizing agent may be dissolved in an inert inorganic or organic solvent. The cyclizing agent is generally used in a molar excess, e.g., up to a 20 fold excess, or up to a 10 fold excess, based on the total moles of acetoacetamide salt. An exemplary cyclization reaction using sulfur trioxide as the cyclizing agent is shown in reaction (5), below.

(5)



in one embodiment, the weight ratio of solvent to cyclizing agent in the cyclizing agent composition is at least 1:1, e.g., at least 2:1, or at least 5:1. In one embodiment, the weight ratio of solvent to cyclizing agent in the cyclizing agent composition ranges from 1:1 to 25:1, e.g., from 1:1 to 10:1, from 2:1 to 10:1, or from 5:1 to 10:1.

A cyclizing agent may be any compound that initiates the ring closure of the acetoacetamide salt. Although sulfur trioxide is a preferred cyclizing agent, the employment of other cyclizing agents is contemplated.

Suitable inert inorganic or organic solvents are those liquids which do not react in an undesired manner with sulfur trioxide or the starting materials or final products of the reaction. Preferred organic solvents include, but are not limited to, halogenated aliphatic hydrocarbons, preferably having up to four carbon atoms, such as, for example, methylene chloride (dichloromethane), chloroform, 1,2-dichloroethane, trichloroethylene, tetrachloroethylene, trichlorofluoroethylene; esters of carbonic acid with lower aliphatic alcohols, preferably with methanol or ethanol; nitroalkanes, preferably having up to four carbon atoms, in particular nitromethane; alkyl-substituted pyridines, preferably collidine; and aliphatic sulfones, preferably sulfolane. Particularly preferred solvents for the cyclization reaction include dichloromethane (methylene chloride), 1,2-dichloroethane, acetone, glacial acetic acid and dimethylformamide, with dichloromethane (methylene dichloride) being particularly preferred. Other solvents, e.g., other solvents mentioned herein, may also be suitable as solvents. The solvents may be used either alone or in a mixture. In one embodiment, the solvent is a halogenated, aliphatic hydrocarbon solvent, preferably the solvent is dichloromethane. The processes may employ these solvents alone or in mixtures thereof.

In some cases, the solvent in the cyclizing agent composition may be selected from 1) concentrated sulfuric acid, 2) liquid sulfur dioxide, or 3) an inert organic solvent.

In a preferred embodiment, the same solvent is used in both the acetoacetamide salt formation reaction and the cyclization reaction. As one benefit, the solution obtained in the acetoacetamide salt formation reaction, without isolation of the acetoacetamide salt formation reaction product, may be used immediately in the cyclization.

In one embodiment, the reaction temperature for the cyclization reaction ranges from −70° C. to 175° C., e.g., from −40° C. to 60° C. The pressure at which the reaction is conducted may vary widely. In one embodiment, the reaction is conducted at a pressure ranging from 0.01 MPa to 10 MPa, e.g., from 0.1 MPa to 5 MPa. Preferably, the reaction is conducted at atmospheric pressure.

The acetoacetamide salt may be introduced to the cyclization reactor and the cyclizing agent composition, e.g., a solution of cyclizing agent optionally in solvent, may be

metered into the reactor. In preferred embodiments, both reactants (acetoacetamide salt and cyclizing agent) are simultaneously fed into the reactor. In one embodiment, the cyclizing agent composition is initially introduced into the reactor and the acetoacetamide salt is added. Preferably, at least part of the cyclizing agent composition is introduced into the reactor and, either continuously or in portions, acetoacetamide salt and (additional) cyclizing agent are then metered in, preferably while maintaining the temperature as described above.

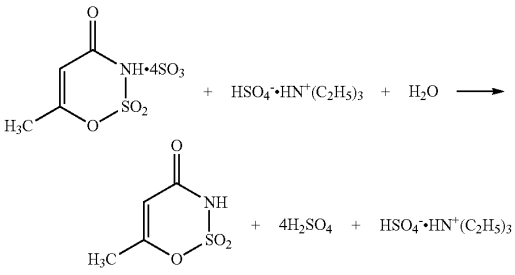
The acetoacetamide salt may be introduced to the reactor and the cyclizing agent composition may be metered into the reactor. In preferred embodiments, both reactants are simultaneously fed into the reactor. In one embodiment, the cyclizing agent composition is initially introduced into the reactor and the acetoacetamide salt is added. Preferably, at least part of the cyclizing agent composition is introduced into the reactor and, either continuously or in portions, acetoacetamide salt and (additional) cyclizing agent are then metered in, preferably while maintaining the temperature as described above.

The formation of the crude acesulfame potassium composition from the cyclic sulfur trioxide adduct composition, in some embodiments, comprises the steps of hydrolyzing the cyclic sulfur trioxide adduct to form an acesulfame-H composition; neutralizing the acesulfame-H in the acesulfame H composition to form a crude acesulfame potassium composition; and forming the acesulfame potassium composition from the crude acesulfame potassium composition.

The cyclic sulfur trioxide adduct may be hydrolyzed via conventional means, e.g., using water. Thus, the forming step may comprise the steps of hydrolyzing the cyclic sulfur trioxide adduct to form an acesulfame-H composition. Acesulfame-H is referred to as sweetener acid.

An exemplary hydrolysis reaction scheme is shown in reaction (6), below.

(6)



The addition of the water leads to a phase separation. The majority of the sweetener acid, acesulfame-H (6-methyl-3,4-dihydro-1,2,3-oxathiazin-4-one 2,2-dioxide), which is formed via the hydrolysis, is present in the organic phase, e.g., at least 60 wt %, at least 70%, at least 80%, or at least 90%. The remainder of the sweetener acid is in the water phase and can be extracted and optionally added to the sweetener acid in the organic phase. In cases where dichloromethane is used as the reaction medium, water or ice may be added, e.g., in a molar excess, based on the sulfur trioxide, to the cyclic sulfur trioxide adduct/sulfur trioxide solution.

In some cases, the hydrolysis step comprises adding water to the cyclic sulfur trioxide adduct. In preferred embodiments, the weight ratio of water to acetoacetamide salt is greater than 1.3:1, e.g., greater than 1.5:1, greater than 1.7:1, greater than 2:1 or greater than 2.2:1. Employment of these ratios may lead to decreases in acetoacetamide-N-sulfonic acid and/or acetoacetamide formation in the neutralized crude acesulfame potassium composition, e.g., the crude acesulfame potassium composition may comprise acetoacetamide-N-sulfonic acid in the amounts discussed herein.

It was surprisingly discovered that the temperature at which the water is initially fed to the hydrolysis reaction may have beneficial effects on impurity production, e.g., organic production or 5-chloro-acesulfame potassium production as well as reaction parameters, e.g., temperature. At lower temperatures, e.g., lower than approximately −35° C. or lower than −22° C., ice tends to build up in the reaction mixture. As this ice melted, it led to the onset of additional reaction, which caused the temperature to rise quickly. This rise in temperature surprisingly led to a product that contained much higher levels of impurities. In some cases, the hydrolyzing comprises adding hydrolysis water to the cyclic sulfur trioxide adduct to form a hydrolysis reaction mixture and reacting the mixture to from the acesulfame-H composition. In some embodiments, the temperature of the hydrolysis reaction mixture or the temperature at which the hydrolysis water is fed to the reactor is maintained at a temperature greater than −35° C., e.g., greater than −30° C., greater than −25° C., greater than −24° C., greater than −23° C., greater than −22° C., greater than −21.5° C., greater than −21° C., or greater than greater than −20° C. In terms of ranges, the temperature of the hydrolysis reaction mixture or the temperature at which the hydrolysis water is fed to the reactor optionally is maintained at a temperature ranging from −35° C. to 0° C., e.g., from −30° C. to −5° C., from −20° C. to −5° C., from −30° C. to −20° C., from −25° C. to −21° C., or −25° C. to −21.5° C.

After the addition of water, the reaction solvent, e.g., dichloromethane, may be removed by distillation, or the acesulfame-H that remains in the organic phase may be extracted with a more suitable solvent. Suitable solvents are those which are sufficiently stable towards sulfuric acid and which have a satisfactory dissolving capacity. Other suitable solvents include esters of carbonic acid such as, for example dimethyl carbonate, diethyl carbonate and ethylene carbonate, or esters of organic monocarboxylic acids such as, for example, isopropyl formate and isobutyl formate, ethyl acetate, isopropyl acetate, butyl acetate, isobutyl acetate and neopentyl acetate, or esters of dicarboxylic acids or amides which are immiscible with water, such as, for example, tetrabutylurea, are suitable. Isopropyl acetate and isobutyl acetate are particularly preferred.

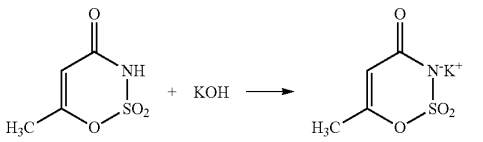
The combined organic phases are dried with, for example, Na_2SO_4, and are evaporated. Any sulfuric acid which has been carried over in the extraction may be removed by appropriate addition of aqueous alkali to the organic phase. For this purpose, dilute aqueous alkali may be added to the organic phase until the pH reached in the aqueous phase corresponds to that of pure 6-methyl-3,4-dihydro-1,2,3-oxathiazin-4-one 2,2-dioxide at the same concentration in the same two-phase system of extracting agent and water.

Neutralization

The neutralization of the acesulfame-H yields a non-toxic salt of acesulfame-H, e.g., acesulfame potassium. In one embodiment, neutralization is carried out by reacting the acesulfame-H with an appropriate base, e.g., potassium hydroxide, in particular a membrane-produced potassium hydroxide. Other suitable bases include, for example, KOH, $KHCO_3$, K_2CO_3, and potassium alcoholates. An exemplary

reaction scheme using potassium hydroxide as a neutralizing agent is shown in reaction (7), below.

(7)



In some cases, the neutralization is conducted or maintained at a low pH levels, which may advantageously further result in a reduction or elimination of the formation of impurities, e.g., acetoacetamide salts. In this context, "conducted" means that the neutralization step begins at a low pH level, and "maintained" means that steps are taken to ensure that the pH stays within a low pH range throughout the entire neutralization step. In one embodiment, the neutralization step is conducted or maintained at a pH below 10.0, e.g., below 9.5, below 9.0, below 8.5, below 8.0, below 7.5, below 7.0, or below 6.5. In terms of ranges, the neutralization step is preferably conducted or maintained at a pH between 6.0 and 10.0, e.g., between 6.5 and 9.5, between 7.0 and 9.0, or between 7.5 and 8.5.

In some cases, the pH in the neutralizing step may be maintained within the desired range by managing the components of the neutralization reaction mixture, which comprises acesulfame-H and neutralizing agent (and also solvent). For example, the composition of the neutralization reaction mixture may include from 1 wt % to 95 wt % neutralizing agent, e.g., from 10 wt % to 85 wt % or from 25 wt % to 75 wt %, and from 1 wt % to 95 wt % acesulfame-H, e.g., from 10 wt % to 85 wt % or from 25 wt % to 75 wt %. These concentration ranges are based on the mixture of neutralization agent and acesulfame-H (not including solvent).

In one embodiment, the acesulfame-H may be neutralized and extracted directly from the purified organic extraction phase using an aqueous potassium base. The acesulfame potassium then precipitates out, where appropriate after evaporation of the solution, in the crystalline form, and it can also be recrystallized for purification.

In one embodiment, the process is not a small-scale batch process or a laboratory-scale process. For example, the inventive process for producing a finished acesulfame potassium composition may yield at least 50 grams of finished acesulfame potassium composition per batch, e.g., at least 100 grams per batch, at least 500 grams per batch, at least 1 kilogram per batch, or at least 10 kilograms per batch. In terms of rates, the inventive process may yield at least 50 grams of finished acesulfame potassium composition per hour, e.g., at least 100 grams per hour, at least 500 grams per hour, at least 1 kilogram per hour, or at least 10 kilograms per hour.

FIG. **1** shows an exemplary acesulfame potassium process **100** in accordance with the process described herein. Process **100** comprises amidosulfamic acid salt formation reactor **102** and acetoacetamide salt formation reactor **104**. Although FIG. **1** shows separate reactors for the two intermediate formation reactions, other configurations, e.g., a one reactor process, are within the contemplation of the present process. Sulfamic acid is fed to amidosulfamic acid salt formation reactor **102** via sulfamic acid feed line **106**. One or more amines, preferably triethylamine, are fed to amidosulfamic acid salt formation reactor **102** via amine feed line **108**. In addition to sulfamic acid and amine(s), acetic acid is also fed to amidosulfamic acid salt formation reactor **102** (via feed line **110**). The resultant reaction mixture in amidosulfamic acid salt formation reactor **102** is as discussed above. In amidosulfamic acid salt formation reactor **102**, the sulfamic acid and the amine (in the presence of the acetic acid) are reacted to yield a crude amidosulfamic acid salt composition, which exits reactor **102** via line **112**. Although not shown, a reaction solvent, e.g., dichloromethane may also be present in the amidosulfamic acid salt formation reactor **102**.

The crude amidosulfamic acid salt composition in line **112** is directed to acetoacetamide salt formation reactor **104**. Diketene is fed to acetoacetamide salt formation reactor **104** via feed line **114**. In acetoacetamide salt formation reactor **104**, the amidosulfamic acid salt and the diketene are reacted to yield a crude acetoacetamide salt composition, which exits reactor **104** via line **118**. Although not shown, dichloromethane may also be present in the acetoacetamide salt formation reactor **104**.

Cyclizing agent (sulfur dioxide) and solvent (dichloromethane) are fed to vessel **119** via feed lines **121** and **123**. Vessel **119** is preferably a cooling vessel wherein the cyclizing agent composition (as discussed above) is formed. The cyclizing agent composition exits vessel **119** via line **125**.

The crude acetoacetamide salt composition is directed to cyclization reactor **120** via line **118**. The cooled cyclizing agent composition is also directed to cyclization reactor **120** (via line **125**). Line **125** is preferably made of a material and in such a size and shape to facilitate the residence times discussed herein. In cyclization reactor **120**, the acetoacetamide salt in the crude acetoacetamide salt composition in line **118** is cyclized and a cyclic sulfur trioxide adduct stream exits via line **124**.

The cyclic sulfur trioxide adduct in line **124**, is directed to hydrolysis reactor **126**. Water is fed to hydrolysis reactor **126** via water feed **128**. In hydrolysis reactor **126**, the cyclic sulfur trioxide adduct is hydrolyzed to yield a crude acesulfame-H composition, which exits hydrolysis reactor **126** via line **130** and is directed to phase separation unit **132**. Phase separation unit **132** separates the contents of line **130** into organic phase **134** and aqueous phase **136**. Organic phase **134** comprises a major amount of the acesulfame-H in line **130** as well as solvent, e.g., methylene chloride. Aqueous phase **136** exits via line **137** and comprises triethylammonium sulfate, and optionally sulfuric acid and minor amounts of acesulfame-H. This aqueous phase may be further purified to separate and/or recover the acesulfame-H and/or the triethylammonium sulfate. The recovered acesulfame-H may be combined with the acesulfame from the organic phase (not shown).

Organic phase **134** exits phase separation unit **132** and is directed to extraction column **138** (via line **140**). Water is fed to extraction column **138** via water feed **142**. The water extracts residual sulfates from the contents of line **140** and a purified acesulfame-H composition exits extraction column **138** via line **144**. The extracted sulfates exit extraction column **138** via line **145**.

The organic phase exits phase separation unit **132** and is directed to extraction column **138** (via line **140**). Water is fed to extraction column **138** via water feed **142**. The water extracts residual sulfates from the contents of line **140** and a purified acesulfame-H stream exits extraction column **138** via line **144**. The extracted sulfates exit extraction column **138** via line **145**.

The purified acesulfame-H composition in line **144** is directed to neutralization unit **146**. Potassium hydroxide is also fed to neutralization unit **146** (via line **148**). The potassium hydroxide neutralizes the acesulfame-H in the purified acesulfame-H composition to yield a product comprising acesulfame potassium, dichloromethane, water, potassium hydroxide, and impurities, e.g., 5-chloro-acesulfame potassium, which exits neutralization unit **146** via line **150**. This product may be considered a crude acesulfame potassium composition.

The product in line **150** is directed to phase separation unit **160**. Phase separation unit **160** separates the product in line **150** into organic phase **162** and an aqueous phase **164**. Aqueous phase **164** comprises a major amount of the acesulfame potassium in line **150** as well as some impurities. Organic phase **162** comprises potassium hydroxide, dichloromethane, and water and may be further treated to recover these components. Aqueous phase **164** (without any further treatment) may be considered a crude acesulfame potassium composition. Aqueous phase **164** may be optionally treated to form a finished acesulfame potassium composition.

Aqueous phase **164** is directed to treatment unit **156** via line **166**. In treatment unit **156**, aqueous phase **164** is treated to obtain finished acesulfame potassium composition (product that may be sold), which is shown exiting via stream **152**. In addition to the finished acesulfame potassium composition, dichloromethane and potassium hydroxide may be separated. These components exit treatment unit **156** via line **154**. The contents of stream **154** may be recovered and/or recycled to the process.

The crude acesulfame potassium product stream comprises acesulfame potassium, dichloromethane, water, and potassium hydroxide. The crude acesulfame potassium product stream in line **150** may be directed to further processing to recover purified acesulfame potassium, which is shown exiting via stream **152**. In addition to the purified acesulfame potassium, dichloromethane and potassium hydroxide may be separated from the crude acesulfame potassium product stream, as shown by stream **154**. The contents of stream **154** may be recovered and/or recycled to the process.

The invention relates also to the following aspects:

Aspect 1: A process for producing a finished acesulfame potassium composition, the process comprising the steps of:

(a) contacting a solvent and a cyclizing agent to form a cyclizing agent composition;

(b) reacting an acetoacetamide salt with the cyclizing agent in the cyclizing agent composition to form a cyclic sulfur trioxide adduct; and

(c) forming from the cyclic sulfur trioxide adduct the finished acesulfame potassium composition comprising non-chlorinated acesulfame potassium and less than 35 wppm 5-chloro-acesulfame potassium;

wherein contact time from the beginning of step (a) to the beginning of step (b) is less than 60 minutes.

Aspect 2: The process of aspect 1, wherein the forming comprises:

hydrolyzing the cyclic sulfur trioxide adduct to form an acesulfame-H composition comprising acesulfame-H;

neutralizing the acesulfame-H in the acesulfame-H composition to form a crude acesulfame potassium composition comprising non-chlorinated acesulfame potassium and less than 35 wppm 5-chloro-acesulfame potassium; and

forming the finished acesulfame potassium composition from the crude acesulfame potassium composition.

Aspect 3: The process of any one of the preceding aspects, wherein the finished acesulfame potassium composition comprises from 0.001 wppm to 5 wppm 5-chloro-acesulfame potassium.

Aspect 4: The process of any one of the preceding aspects, wherein the contact time is less than 15 minutes and the crude acesulfame potassium composition comprises from 0.001 wppm to 5 wppm 5-chloro-acesulfame potassium and the finished acesulfame potassium composition comprises from 0.001 wppm to 5 wppm 5-chloro-acesulfame potassium.

Aspect 5: The process of any one of the preceding aspects, wherein the contact time is less than 5 minutes and the crude acesulfame potassium composition comprises from 0.001 wppm to 5 wppm 5-chloro-acesulfame potassium and the finished acesulfame potassium composition comprises from 0.001 wppm to 2.7 wppm 5-chloro-acesulfame potassium.

Aspect 6: The process of any one of the preceding aspects, wherein the crude acesulfame potassium composition comprises less than 35 wppm 5-chloro-acesulfame potassium.

Aspect 7: The process of any one of the preceding aspects, wherein the finished acesulfame potassium composition comprises at least 90% by weight of the 5-chloro-acesulfame potassium present in the crude acesulfame potassium composition.

Aspect 8: The process of any one of the preceding aspects, wherein the hydrolyzing comprises adding water to the cyclic sulfur trioxide adduct to form a hydrolysis reaction mixture, and wherein the temperature of the hydrolysis reaction mixture is maintained at a temperature ranging from −35° C. to 0° C.

Aspect 9: The process of any one of the preceding aspects, wherein the finished acesulfame potassium composition comprises from 0.001 wppm to 5 wppm organic impurities.

Aspect 10: The process of any one of the preceding aspects, further comprising:

reacting sulfamic acid and an amine to form an amidosulfamic acid salt; and

reacting the amidosulfamic acid salt and acetoacetylating agent to form the acetoacetamide salt.

Aspect 11: The process of any one of the preceding aspects, wherein the finished acesulfame potassium composition comprises from 0.001 wppm to 2.7 wppm 5-chloro-acesulfame potassium.

Aspect 12: The process of any one of the preceding aspects, wherein the finished acesulfame potassium composition comprises from 0.001 wppm to 5 wppm organic impurities.

Aspect 13: The process of any one of the preceding aspects, wherein the finished acesulfame potassium composition comprises from 0.001 wppm to 5 wppm heavy metals.

Aspect 14: The process of any one of the preceding aspects, wherein the cyclizing agent composition comprises less than 1 wt % of compounds selected from chloromethyl chlorosulfate, methyl-bis-chlorosulfate, and mixtures thereof.

Aspect 15: The process of any one of the preceding aspects, wherein the reacting is conducted for a cyclization reaction time, from the start of the reactant feed to the end of the reactant feed, less than 35 minutes.

Aspect 16: The process of any one of the preceding aspects, wherein the weight ratio of solvent to cyclizing agent in the cyclizing agent composition is at least 1:1.

Aspect 17: The process of any one of the preceding aspects, further comprising cooling the cyclizing agent composition to a temperature less than 15° C.

Aspect 18: The process of any one of the preceding aspects, wherein the cyclizing agent comprises sulfur trioxide and the solvent comprises dichloromethane.

Aspect 19: A finished acesulfame potassium composition produced or producible by, or obtainable or obtained from the process of any one of aspects 1 to 18.

Aspect 20: The finished acesulfame potassium composition of aspect 19, comprising:

non-chlorinated acesulfame potassium;

from 0.001 wppm to 2.7 wppm 5-chloro acesulfame potassium; and

from 0.001 wppm to 5 wppm heavy metals.

Aspect 21: A process for producing a finished acesulfame potassium composition, the process comprising the steps of:

reacting sulfamic acid and triethylamine to form an amidosulfamic acid salt;

reacting the amidosulfamic acid salt and diketene to form the acetoacetamide salt;

contacting dichloromethane and a sulfur trioxide to form a cyclizing agent composition; reacting the acetoacetamide salt with the sulfur trioxide in the cyclizing agent composition to form a cyclic sulfur trioxide adduct;

hydrolyzing the cyclic sulfur trioxide adduct to form an acesulfame-H composition; and neutralizing the acesulfame-H to form the finished acesulfame potassium composition comprising non-chlorinated acesulfame potassium and less than 10 wppm 5-chloro-acesulfame potassium,

wherein contact time from the beginning of step (a) to the beginning of step (b) is less than 10 minutes.

Aspect 22: The process of aspect 21, further comprising cooling the cyclizing agent composition to a temperature less than 15° C.

Aspect 23: An acesulfame potassium composition comprising non-chlorinated acesulfame potassium and less than 35 wppm, preferably 0.001 wppm to 2.7 wppm 5-chloro-acesulfame potassium.

Aspect 24: The acesulfame potassium composition of aspect 23, further comprising less than 37 wppm, preferably 1 wppb to 5 wppm acetoacetamide-N-sulfonic acid.

Aspect 25: The acesulfame potassium composition of any one of the preceding aspects, further comprising 0.001 wppm to 5 wppm organic impurities and/or 0.001 wppm to 5 wppm of at least one heavy metal.

Aspect 26: The acesulfame potassium composition of any one of the preceding aspects, wherein the at least one heavy metal is selected from the group consisting of mercury, lead and both.

Aspect 27: The acesulfame potassium composition of any one of the preceding aspects, wherein the mercury is present in an amount of 1 wppb to 20 wppm.

Aspect 28: The acesulfame potassium composition of any one of the preceding aspects, wherein the lead is present in an amount of 1 wppb to 25 wppm.

EXAMPLES

Example 1

Liquid sulfur trioxide and dichloromethane were continuously fed, contacted (to form a cyclizing agent composition), and cooled into a static mixer at 1220 kg/h and 8000 kg/h, respectively. The temperature of the cooled cyclizing agent composition was 11° C. The mixture was held in the static mixture for less than 5 minutes and then fed into a cyclization reactor, thus contact time was less than 5 minutes. In the cyclization reactor the sulfur trioxide/dichloromethane composition was reacted with a solution of acetoacetamide-N-sulfonate triethylammonium salt (acetoacetamide salt) in dichloromethane. The resultant cyclized product was hydrolyzed and worked up to yield a crude acesulfame potassium composition comprising (non-chlorinated) acesulfame potassium. Testing for 5-chloro-acesulfame potassium content was performed using the HPLC equipment and techniques discussed herein. In particular, the HPLC analysis was performed using an LC Systems HPLC unit from Shimadzu having a CBM-20 Shimadzu controller and being equipped with a CC 250/4.6 Nucleodur 100-3 C18 ec (250×4.6 mm) MACHEREY NAGEL column. A Shimadzu SPD-M20A photodiode array detector was used for detection (at 234 nm wavelength). Analysis was performed at 23° C. column temperature. As an eluent solution, an aqueous solution of tetra butyl ammonium hydrogen sulfate (3.4 g/L and 60% of the total solution) and acetonitrile (HPLC grade) (300 mL/L and 40% of the total solution) was employed. Elution was isocratic. The overall flow rate of total eluent was approximately 1 mL/min. The data collection and calculations were performed using Lab Solution software from Shimadzu. With a detection limit of 1 wppm, no 5-chloro-acesulfame potassium was detected.

Comparative Example A

528 mmol of sulfur trioxide in dichloromethane was prepared and stored for 20 days at 20° C. The sulfur trioxide/dichloromethane composition was fed to a stirred vessel. 100 mmol acetoacetamide-N-sulfonate triethylammonium salt in dichloromethane was reacted with the sulfur trioxide/dichloromethane composition by continuous feeding into the stirred vessel for 30 minutes. Contact time was 20 days. After additional stirring for two minutes, the reaction mixture was hydrolyzed by the addition of 50 ml water and worked up as described herein. Testing for 5-chloro-acesulfame potassium content was performed using the HPLC equipment and techniques discussed above. The crude acesulfame potassium had an impurity content of 4960 wppm 5-chloro-acesulfame potassium.

Example 2 and Comparative Examples B and C

100 mmol of 99.5% pure sulfamic acid was suspended in 50 mL dichloromethane in a flask with reflux. Under continuous agitation, 105 mmol of trimethylamine was added within approximately 3 minutes. During this time, temperature increased due to acid/base exothermal reaction up to about 42° C. (the boiling point of dichloromethane). This reaction mixture was stirred for approximately 15 additional minutes, until no solid sedimentation was seen in the flask. Then, 10 mmol of acetic acid was added to the first reaction mixture and was stirred for approximately 15 additional minutes. At this point, within 7 minutes of the addition of the acetic acid, 110 mmol of diketene was added dropwise to form a second reaction mixture. After the addition of all of the diketene was added to the second reaction mixture and approximately 15 minutes of reaction time, this second reaction mixture was cooled. The resultant cooled second reaction mixture contained approximately 30% acetoacetamide N-sulfonate triethylammonium salt. Additional batches of cooled second reaction mixture were prepared as necessary. The acetoacetamide N-sulfonate triethylammonium salt was used as discussed below.

Sulfur trioxide/dichloromethane compositions (cyclizing agent compositions) were prepared by contacting approximately 15 wt % sulfur trioxide and approximately 85 wt % dichloromethane with one another in a flask.

For each of Example 2 and Comparative Examples B and C, a reaction flask (a 4 necked round bottom flask equipped with mechanical stirrer, thermometer, and feed vessels) was placed into a cooling bath containing a mixture of isopropanol and dry ice. Approximately 200 g of the acetoacetamide-N-sulfonate triethylammonium salt solution and approximately 577 g of the sulfur trioxide/dichloromethane compositions were measured. The compositions were held for various time periods before the start of the cyclization reaction. Contact times for the respective examples are shown in Table 1.

TABLE 1

Contact Times

Example	Contact Time
Ex. 2	1 hour
Comp. Ex. B	4 days
Comp. Ex. C	5 days

For each example, the flask was placed into a cooling bath containing a mixture of isopropanol and dry ice. Approximately 15 wt % of the total sulfur trioxide/dichloromethane composition (approximately 87 g) was initially fed to the reaction flask under continuous agitation by mechanical stirrer. When the temperature of the reaction flask contents reached −35° C. (due to the cooling batch), the remainder of the sulfur trioxide/dichloromethane composition and all of the acetoacetamide-N-sulfonate triethylammonium salt solution were fed into the reaction flask. The time period that the solvent contacted the cyclizing agent before formation of the cyclic sulfur trioxide adduct, e.g., before the acetoacetamide-N-sulfonate triethylammonium salt solution was fed to the reaction flask, was less than an hour. The feed rate was controlled in such a way that the temperature of the reaction flask contents remained between −25° and −35° C. during the feeding/cyclization reaction. After the reactants were fed, the reaction was allowed to proceed for approximately one additional minute. The cooling bath was then removed.

After approximately one minute, the temperature of the reaction flask contents reached approximately −22° C. At this time, hydrolysis was initiated by feeding deionized water to the reaction flask. Water was fed over 10 minutes. The hydrolysis reaction was exothermic. Water was added slowly so as to maintain temperature between −20° C. and −5° C. After addition of water, reaction mixture was allowed to reach room temperature.

The hydrolyzed product was phase separated via a separating funnel. A heavier organic sweetener acid-dichloromethane phase (acesulfame-H composition) was separated out, and the remaining aqueous phase was discarded.

The acesulfame-H in the acesulfame-H composition was neutralized with a 10% potassium hydroxide solution. Neutralization was carried out at 25° C.±1° C. Potassium hydroxide addition was completed within 20 minutes.

After completion of the neutralization step, an additional phase separation was performed using a separating funnel to yield an aqueous phase containing acesulfame potassium (and some impurities) and an organic phase. The aqueous phase is considered a crude acesulfame potassium composition. The aqueous phase analyzed for impurities, e.g., 5-chloro acesulfame potassium. Testing for 5-chloro-acesulfame potassium content was performed using the HPLC equipment and techniques discussed above. The remaining dichloromethane phase was discarded.

The results of the impurity analysis of Examples 1 and 2 and Comparative Examples A-C are shown in Table 2.

TABLE 2

5-chloro Ace-K Content

	Contact Time	5-chloro Ace-K (in crude), wppm
Ex. 1	<5 min.	Not detectable
Ex. 2	1 hour	32
Comp. Ex. A	20 days	4960
Comp. Ex. B	4 days	54
Comp. Ex. C	5 days	78

As shown in the Examples, the 5-chloro-acesulfame potassium content was affected by contact time. When a contact time of greater than 1 hour was employed (Comparative Examples A-C), significant amounts of 5-chloro acesulfame potassium were present in the crude acesulfame potassium composition. Importantly, when contact time was kept below 1 hour (Example 2), e.g., below 5 minutes (Example 1), then crude acesulfame potassium composition comprised much smaller amounts of 5-chloro acesulfame potassium.

Only minor and simple additional treatment of the crude acesulfame composition was necessary to form the finished acesulfame potassium compositions.

Approximately 50% of water was evaporated out of the crude acesulfame potassium compositions in roti vapor at reduced pressure. The resultant concentrated acesulfame potassium composition is considered an intermediate acesulfame potassium composition and was then cooled in a refrigerator at +5° C., which led to precipitation of crude crystals containing mostly acesulfame potassium.

The crude crystals were then dissolved in enough water and this resultant solution was heated to 70° C. Activated carbon powder was then added to the solution. The solution (with the added activated carbon) was then filtered.

The filtrate that was yielded from the filtration was cooled to room temperature, which led to the formation of crystals containing mostly acesulfame potassium. These crystals were dissolved in sufficient water and heated to 70° C. in a water bath.

Activated carbon was added to this solution of crystals and activated carbon. This solution was then filtered. When filtrate was cooled down to room temperature, white-colored crystals of acesulfame potassium were formed. These crystals are considered a finished acesulfame potassium composition.

Testing for 5-chloro-acesulfame potassium content was performed using the HPLC equipment and techniques discussed above. The crystals of the finished acesulfame potassium composition contained the same amount (or slightly lower amounts) of 5-chloro-acesulfame potassium.

The treatment steps did not show a marked reduction in 5-chloro-acesulfame potassium content. It is believed that because the chemical structure of chloro-acesulfame potassium is similar to that of acesulfame potassium, separation of chloro-acesulfame potassium using standard purification procedures such as crystallization is ineffective. This analysis demonstrates the importance of reducing/eliminating the production of 5-chloro-acesulfame potassium during the steps leading to the formation of the crude acesulfame composition as described herein.

While the invention has been described in detail, modifications within the spirit and scope of the invention will be

readily apparent to those of skill in the art. In view of the foregoing discussion, relevant knowledge in the art and references discussed above in connection with the Background and Detailed Description, the disclosures of which are all incorporated herein by reference. In addition, it should be understood that aspects of the invention and portions of various embodiments and various features recited above and/or in the appended claims may be combined or interchanged either in whole or in part. In the foregoing descriptions of the various embodiments, those embodiments which refer to another embodiment may be appropriately combined with other embodiments as will be appreciated by one of skill in the art. Furthermore, those of ordinary skill in the art will appreciate that the foregoing description is by way of example only, and is not intended to limit the invention.

We claim:

1. A process for producing a finished acesulfame potassium composition, the process comprising:

(a) providing a solvent at an initial temperature and a cyclizing agent at an initial temperature, wherein the cyclizing agent includes sulfur trioxide;

(b) contacting the solvent and the cyclizing agent to form a cyclizing agent composition;

(c) reacting an acetoacetamide salt with the cyclizing agent composition to form a cyclic sulfur trioxide adduct, wherein prior to being reacted with the acetoacetamide salt, the cyclizing agent composition has a temperature that is lower than the initial temperature of the cyclizing agent and/or the initial temperature of the solvent; and

(d) forming from the cyclic sulfur trioxide adduct the finished acesulfame potassium composition comprising non-chlorinated acesulfame potassium and less than 35 wppm 5-chloro-acesulfame potassium;

wherein contact time from the beginning of step (b) to the beginning of step (c) is less than 60 minutes.

2. The process of claim **1**, wherein the forming comprises:

hydrolyzing the cyclic sulfur trioxide adduct to form an acesulfame-H composition comprising acesulfame-H;

neutralizing the acesulfame-H in the acesulfame-H composition to form a crude acesulfame potassium composition comprising non-chlorinated acesulfame potassium; and

forming the finished acesulfame potassium composition from the crude acesulfame potassium composition.

3. The process of claim **2**, wherein the finished acesulfame potassium composition comprises from 0.001 wppm to 5 wppm 5-chloro-acesulfame potassium.

4. The process of claim **2**, wherein the hydrolyzing comprises adding water to the cyclic sulfur trioxide adduct to form a hydrolysis reaction mixture, and wherein the temperature of the hydrolysis reaction mixture is maintained at a temperature ranging from −35° C. to 0° C.

5. The process of claim **4**, wherein the finished acesulfame potassium composition comprises from 0.001 wppm to 5 wppm organic impurities.

6. The process of claim **1**, further comprising:

reacting sulfamic acid and an amine to form an amidosulfamic acid salt; and

reacting the amidosulfamic acid salt and acetoacetylating agent to form the acetoacetamide salt.

7. The process of claim **1**, wherein the finished acesulfame potassium composition comprises from 0.001 wppm to 2.7 wppm 5-chloro-acesulfame potassium.

8. The process of claim **1**, wherein the finished acesulfame potassium composition comprises from 0.001 wppm to 5 wppm organic impurities.

9. The process of claim **1**, wherein the finished acesulfame potassium composition comprises from 0.001 wppm to 5 wppm heavy metals.

10. The process of claim **1**, wherein the cyclizing agent composition comprises less than 1 wt % of compounds selected from chloromethyl chlorosulfate, methyl-bis-chlorosulfate, and mixtures thereof.

11. The process of claim **1**, wherein the reacting is conducted for a cyclization reaction time, from the start of a reactant feed to the end of the reactant feed, of less than 35 minutes.

12. The process of claim **1**, wherein the weight ratio of solvent to cyclizing agent in the cyclizing agent composition is at least 1:1.

13. The process of claim **1**, wherein the solvent comprises dichloromethane.

14. A finished acesulfame potassium composition produced from the process of claim **1**.

15. The finished acesulfame potassium composition of claim **14**, comprising:

non-chlorinated acesulfame potassium;

from 0.001 wppm to 2.7 wppm 5-chloro acesulfame potassium; and

from 0.001 wppm to 5 wppm heavy metals.

16. The finished acesulfame potassium composition of claim **14**, wherein the finished composition contains less than 37 wppm acetoacetamide-N-sulfonic acid.

17. The finished acesulfame potassium composition of claim **14**, wherein the finished composition contains from 0.001 wppm to 5 wppm organic impurities and from 0.001 wppm to 5 wppm of at least one heavy metal.

18. The finished acesulfame potassium composition of claim **17**, wherein the heavy metal is selected from the group consisting of mercury, lead and mixtures thereof.

19. The finished acesulfame potassium composition of claim **18**, wherein mercury is present in the finished composition in an amount of 1 wppb to 20 wppm.

20. The finished acesulfame potassium composition of claim **18**, wherein lead is present in the finished composition in an amount of 1 wppb to 25 wppm.

21. The process of claim **1**, wherein the temperature of the cyclizing agent composition is at least 2° C. lower than the initial temperature of the solvent and/or the initial temperature of the cyclizing agent.

22. The process of claim **1**, further comprising cooling the cyclizing agent composition.

23. The process of claim **22**, wherein the cyclizing agent composition is cooled to a temperature of less than 35° C.

24. The process of claim **22**, wherein the cyclizing agent composition is cooled to a temperature of less than 15° C.

25. The process of claim **1**, further comprising cooling the solvent prior to contact with the cyclizing agent.

26. The process of claim **25**, wherein the solvent is cooled to a temperature that is at least 2° C. less than the initial temperature of the solvent.

27. The process of claim **25**, wherein the solvent is cooled to a temperature of less than 35° C.

28. The process of claim **25**, wherein the solvent is cooled to a temperature of less than 15° C.

29. The process of claim **1**, further comprising cooling the cyclizing agent prior to contact with the solvent.

30. The process of claim **29**, wherein the cyclizing agent is cooled to a temperature that is at least 2° C. less than the initial temperature of the cyclizing agent.

31. The process of claim **29**, wherein the cyclizing agent is cooled to a temperature of less than 35° C.

32. The process of claim **29**, wherein the cyclizing agent is cooled to a temperature of less than 15° C.

33. The process of claim **1**, wherein the finished acesulfame potassium composition comprises from 0.001 wppm to 5 wppm 5-chloro-acesulfame potassium.

34. The process of claim **2**, wherein the crude acesulfame potassium composition comprises from 1 wppb to 5 wppm 5-chloro-acesulfame potassium.

35. The process of claim **1**, wherein the temperature of the cyclizing agent composition is less than 35° C. prior to reacting with the acetoacetamide salt.

36. The process of claim **1**, wherein the temperature of the cyclizing agent composition is less than 15° C. prior to reacting with the acetoacetamide salt.

37. The process of claim **1**, wherein the contact time is less than 15 minutes.

38. The process of claim **37**, wherein the temperature of the cyclizing agent composition is less than 35° C. prior to reacting with the acetoacetamide salt.

39. The process of claim **38**, wherein the finished acesulfame potassium composition comprises from 1 wppb to 5 wppm 5-chloro-acesulfame potassium.

40. The process of claim **1**, wherein the contact time is less than 5 minutes.

41. The process of claim **40**, wherein the temperature of the cyclizing agent composition is less than 35° C. prior to reacting with the acetoacetamide salt.

42. The process of claim **41**, wherein the finished acesulfame potassium composition comprises from 1 wppb to 5 wppm 5-chloro-acesulfame potassium.

43. The process of claim **1**, further comprising forming the acetoacetamide salt by a process that comprises reacting sulfamic acid and triethylamine to form an amidosulfamic acid salt, and reacting the amidosulfamic acid salt and a diketene to the form acetoacetamide salt.

* * * * *

US010590095B2

(12) **United States Patent**
Mollenkopf et al.

(10) **Patent No.: US 10,590,095 B2**
(45) **Date of Patent: *Mar. 17, 2020**

(54) **ACESULFAME POTASSIUM COMPOSITIONS AND PROCESSES FOR PRODUCING SAME**

(71) Applicant: **Celanese International Corporation**, Irving, TX (US)

(72) Inventors: **Christoph Mollenkopf**, Frankfurt am Main (DE); **Peter Groer**, Babenhausen (DE); **Arvind Yadav**, Hessen (IN)

(73) Assignee: **Celanese International Corporation**, Iving, TX (US)

(*) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 0 days.

This patent is subject to a terminal disclaimer.

(21) Appl. No.: **16/273,358**

(22) Filed: **Feb. 12, 2019**

(65) **Prior Publication Data**

US 2019/0169143 A1 Jun. 6, 2019

Related U.S. Application Data

(63) Continuation of application No. 16/014,431, filed on Jun. 21, 2018, now Pat. No. 10,233,163, which is a continuation of application No. 15/704,386, filed on Sep. 14, 2017, now Pat. No. 10,029,999.

(60) Provisional application No. 62/397,509, filed on Sep. 21, 2016.

(51) **Int. Cl.**
C07D 291/06 (2006.01)
A23L 27/30 (2016.01)

(52) **U.S. Cl.**
CPC ***C07D 291/06*** (2013.01); ***A23L 27/30*** (2016.08); *A23V 2002/00* (2013.01)

(58) **Field of Classification Search**
CPC C07D 209/06; A23L 27/30
USPC 544/200
See application file for complete search history.

(56) **References Cited**

U.S. PATENT DOCUMENTS

4,563,521	A	1/1986	Clauss et al.	
4,607,100	A	8/1986	Clauss et al.	
4,638,063	A	1/1987	Clauss et al.	
4,695,629	A	9/1987	Clauss et al.	
4,804,755	A	2/1989	Reuschling et al.	
4,806,639	A	2/1989	Reuschling et al.	
4,876,341	A	10/1989	Schiitz et al.	
5,011,982	A	4/1991	Clauss et al.	
5,084,180	A	1/1992	Boateng	
5,103,046	A	4/1992	Clauss et al.	
5,334,397	A	8/1994	Ream et al.	
5,744,010	A	4/1998	Roscher et al.	
5,808,159	A	9/1998	Giebeler	
7,408,059	B2	8/2008	Kobayashi et al.	
7,977,514	B2	7/2011	Peters et al.	
8,182,756	B2	5/2012	Liu et al.	
8,303,921	B2	11/2012	Brietzke et al.	
8,309,048	B2	11/2012	Brietzke et al.	
8,496,905	B2	7/2013	Brietzke et al.	
8,658,830	B2	2/2014	Brietzke et al.	
9,024,016	B2	5/2015	Bayer et al.	
10,023,456	B1	7/2018	Mollenkopf et al.	
10,029,998	B2	7/2018	Mollenkopf et al.	
10,029,999	B2 *	7/2018	Mollenkopf	A23L 27/30
10,030,000	B2	7/2018	Mollenkopf et al.	
10,233,163	B2 *	3/2019	Mollenkopf	A23L 27/30
2003/0065172	A1	4/2003	Tian et al.	
2003/0065218	A1	4/2003	Mollenkopf	
2008/0076919	A1	3/2008	Liu et al.	
2009/0318685	A1	12/2009	Saito et al.	
2009/0318686	A1	12/2009	Saito et al.	
2010/0274057	A1	10/2010	Peters et al.	
2011/0256045	A1	10/2011	Brietzke et al.	
2011/0256046	A1	10/2011	Brietzke et al.	
2013/0062192	A1	3/2013	Brietzke et al.	
2013/0331565	A1	12/2013	Bayer et al.	
2018/0297968	A1	10/2018	Mollenkopf et al.	

(Continued)

FOREIGN PATENT DOCUMENTS

CA	1273923	9/2009
CN	85104277	6/1985
CN	85104277 A	12/1986

(Continued)

OTHER PUBLICATIONS

Duan et al., "Synthesis of Acesulfame Potassium," *Fine Chemicals*, vol. 13, 1996, pp. 22-24.

(Continued)

Primary Examiner — Kahsay Habte

(74) *Attorney, Agent, or Firm* — Dority & Manning, P.A.

(57) **ABSTRACT**

Compositions and processes for producing high purity acesulfame potassium are described. One process comprises the steps of providing a crude acesulfame potassium composition comprising acesulfame potassium and acetoacetamide, concentrating the crude acesulfame potassium composition to form a water stream and an intermediate acesulfame potassium composition comprising acesulfame potassium and less than 33 wppm acetoacetamide, and separating the intermediate acesulfame potassium composition to form the finished acesulfame potassium composition comprising acesulfame potassium and less than 33 wppm acetoacetamide. The concentrating step is conducted at a temperature below 90° C. and the separating step is conducted at a temperature at or below 35° C.

39 Claims, 2 Drawing Sheets

(56) **References Cited**

U.S. PATENT DOCUMENTS

2018/0297969	A1	10/2018	Mollenkopf et al.
2018/0297970	A1	10/2018	Mollenkopf et al.

FOREIGN PATENT DOCUMENTS

CN	1883790		12/2006
CN	200949088		9/2007
CN	101124981		2/2008
CN	101124981	A	2/2008
CN	101148300		3/2008
CN	101157666		4/2008
CN	101787001		7/2010
CN	101913898		12/2010
CN	201921689		8/2011
CN	102225333		10/2011
CN	102359926		2/2012
CN	102380226		3/2012
CN	102380266	A	3/2012
CN	202221403	U	5/2012
CN	102866042		1/2013
CN	103018368		4/2013
CN	103130743		6/2013
CN	10331294		10/2013
CN	103570592		2/2014
CN	103570592	A	2/2014
CN	103588728		2/2014
CN	103588728	A	2/2014
CN	103613566		3/2014
CN	103613566	A	3/2014
CN	103960558		8/2014
CN	104193625		12/2014
CN	104209052		12/2014
CN	104225956		12/2014
CN	104292181		1/2015
CN	104292181	A	1/2015
CN	204320227		5/2015
CN	105085160	A	11/2015
CN	105111166	A	12/2015
CN	105152446		12/2015
CN	105198778		12/2015
CN	106262665		1/2017
CN	106267879		1/2017
CN	106346138		1/2017
CN	106349009		1/2017
CN	106349190		1/2017
CN	106349191		1/2017
CN	106349300		1/2017
CN	106365952		2/2017
CN	106496159	A	3/2017
CN	206001191		3/2017
CN	206001201		3/2017
CN	206001439		3/2017
CN	206121215		4/2017
DE	1249262		10/1966
DE	1268141		5/1968
DE	3522470		1/1987
DE	3531357		3/1987
DE	3545196		6/1987
EP	0155634		9/1985
EP	0159516		10/1985
EP	0215347		3/1987
EP	0217024		4/1987
EP	0218076		4/1987
JP	54032406		3/1979
JP	S 5432406	A	3/1979
WO	WO93/19055		9/1993
WO	WO 2011/133468	A1	10/2011
WO	WO2013/182651		12/2013

OTHER PUBLICATIONS

ASTM E 313-05, Standard Practice for Calculating Yellowness and Whiteness Indices from Instrumentally Measured Color Coordinates, Oct. 1, 2005, 6 pages.

Boehshar et al., 5-Chloroacesulfame K—a characteristic indicator for application of the "sulfur trioxide" process in the manufacture of acesulfame K, Research Disclosure, 2003, 477036.

Mayer et al., Acesulfame-K, Food Science and Technology, Jun. 28, 1991.

International Search Report received in the corresponding International PCT Patent application No. PCT/US2007/051507, dated Nov. 9, 2017.

Linkies et al., Synthesis, 1990, 5, 405-406.

"Commission Direction 95/31/EC of Jul. 5, 1995 laying down specific criteria of purity concerning sweeteners for use in foodstuffs," Official Journal of the European Communities, Jul. 28, 1995, 19 pages.

"Commission Direction 2008/60/EC of Jun. 17, 2008 laying down specific purity criteria concerning sweeteners for use in foodstuffs," Official Journal of the European Union, Jun. 18, 2008, 40 pages.

"Regulations—Commission Regulation (EU) No. 231/2012 of Mar. 9, 2012 laying down specifications for food additives listed in Annexes II and II to Regulation (EC) No. 1333/2008 of the European Parliament and of the Council," Official Journal of the European Union, Mar. 22, 2012, 295 pages.

Opinion—"Re-evaluation of acesulfame K with reference to the previous SCF opinion of 1991," European Commission , Health & Consumer Protection Directorate—General, Directorate B—Scientific Health Opinions, Unit B3—Management of Scientific Committees II, Scientific Committee on Food, Mar. 13, 2000, 8 pages.

Product Information on "Acesulfame Potassium," 2001, 57th JECFA (joint FAO/WHO Expert Committee on Food Additives), FNP 52 Add 9, 2 pages.

Suenaga, "Ethylene-amine salt recovery—by converting the hydrochloride into the sulphate, and reacting with ammonia in aq. Solvent to ppte. Ammonium sulphate", WPI/Thompson, 1979, No. 16, XP 002598345.

Sunnett Brochure, "Acesulfame Potassium", Celanese, Apr. 2014.

Xiangsheng et al., "Synthesis of Acesulfame Potassium", Fine Chemicals, vol. 13, 1996, 1-15.

Information Disclosure Statement submitted Oct. 27, 2017, 2 pages.

Boehshar, Manfred & Burgard, Andreas. (2003). 5-Chloroacesulfame K—a characteristic indicator for application of the "sulfur trioxide" process in the manufacture of acesulfame K. Research Disclosure. 477036.

D. Mayer, et al., Acesulfame-K (Food Science and Technology), 1991, Intro., Chapters 15, 16, and 18, 56 pages.

* cited by examiner

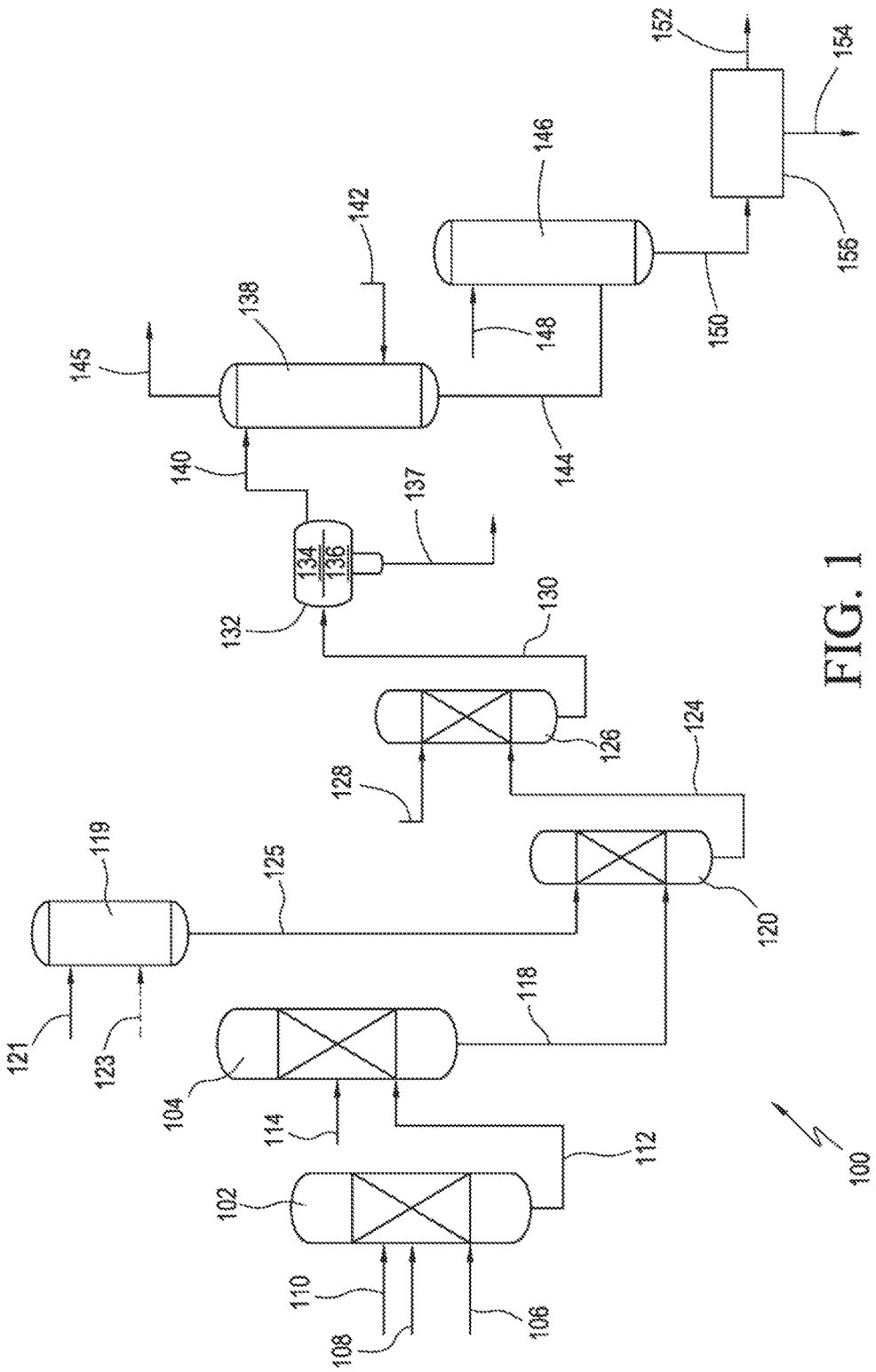

152
154
146
142
156
138
148
150
145
144
140
137
134
136
132
130
126
124
128
119
125
120
121
123
118
104
114
112
102
110
108
106
100

FIG. 1

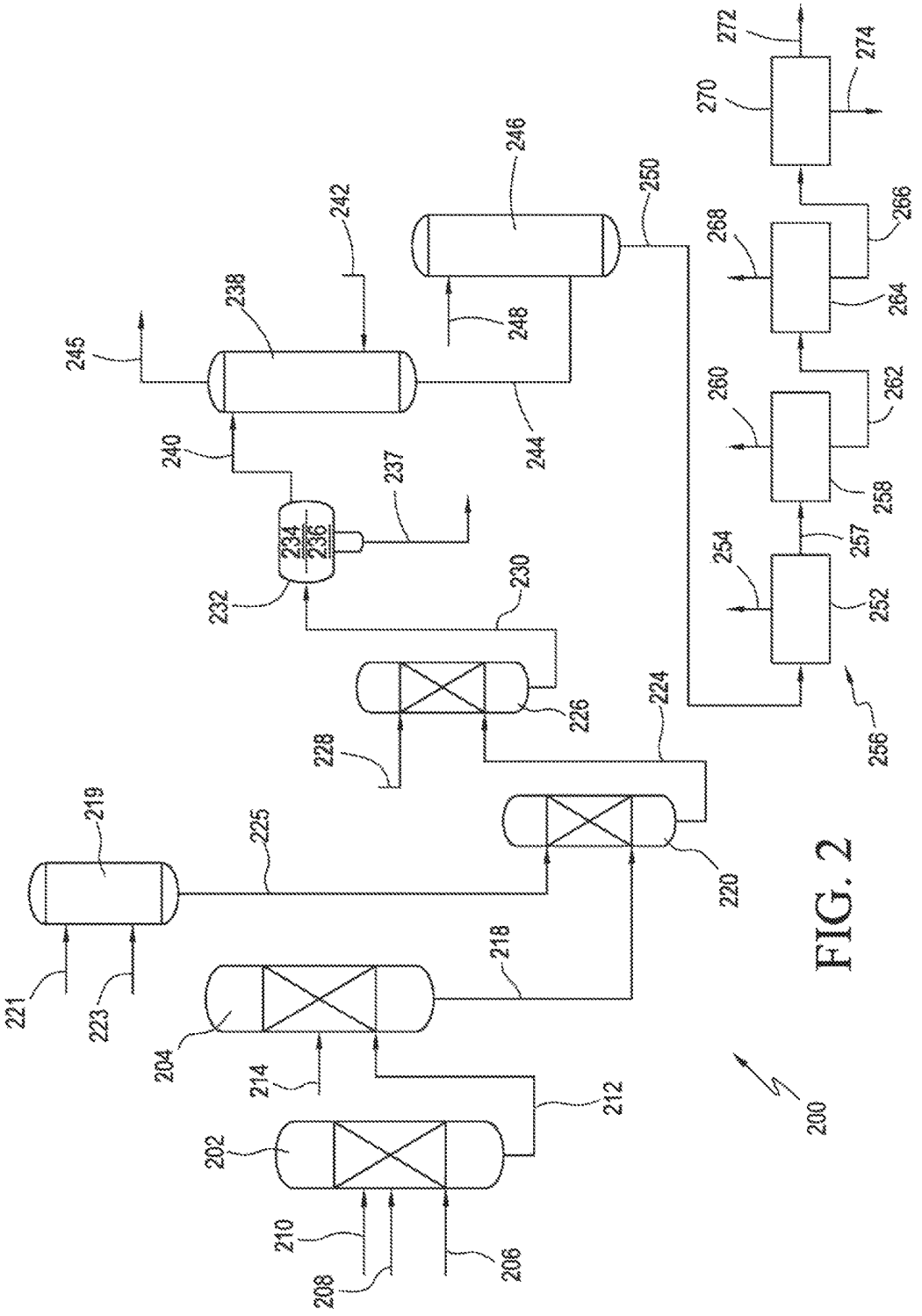

272
274
270
246
250
242
266
268
238
264
248
245
260
262
240
244
237
234
236
258
257
254
232
230
252
226
224
228
256
219
225
220
221
218
223
204
214
212
200
202
210
208
206

FIG. 2

ACESULFAME POTASSIUM COMPOSITIONS AND PROCESSES FOR PRODUCING SAME

CROSS-REFERENCE TO RELATED APPLICATIONS

This patent application is a continuation of U.S. Application Ser. No. 16/014,431 having a filing date of Jun. 21, 2018 which continuation of U.S. application Ser. No. 15/704,386 having a filing date of Sep. 14, 2017 (now U.S. Pat. No. 10,029,999), which claims priority to U.S. Provisional Patent Application No. 62/397,509, filed Sep. 21, 2016, the disclosures of which are incorporated herein by reference in their entirety.

FIELD OF INVENTION

The present invention relates generally to acesulfame potassium and to processes for producing acesulfame potassium. More specifically, the present invention relates to processes for producing high purity acesulfame potassium.

BACKGROUND OF THE INVENTION

Acesulfame potassium has an intense, sweet taste and has been used in many food-related applications as a sweetener. In conventional acesulfame potassium production processes, sulfamic acid and an amine, e.g., triethylamine, are reacted to form an amidosulfamic acid salt, such as a trialkyl ammonium amidosulfamic acid salt. The amidosulfamic acid salt is then reacted with diketene to form an acetoacetamide salt. The acetoacetamide salt may be cyclized, hydrolyzed, and neutralized to form acesulfame potassium. U.S. Pat. Nos. 5,744,010 and 9,024,016 disclose exemplary acesulfame potassium production processes.

Typically, the acetoacetamide salt intermediate is cyclized by reaction with sulfur trioxide in an inorganic or organic solvent to form a cyclic sulfur trioxide adduct. The solvent routinely utilized in this reaction is an organic solvent such as a halogenated, aliphatic hydrocarbon solvent, for example, dichloromethane. The adduct formed by this reaction is subsequently hydrolyzed and then neutralized with potassium hydroxide to form acesulfame potassium.

Acesulfame potassium product and the intermediate compositions produced by conventional methods contain undesirable impurities, such as acetoacetamide (and acetoacetamide-N-sulfonic acid). Limits for the content of various impurities are often set by governmental regulations or customer guidelines. Removal of many of these impurities using standard purification procedures such as evaporation, crystallization, and/or filtration has proven difficult, resulting in consumer dissatisfaction and the failure to meet standards.

The need exists for improved processes for producing high purity acesulfame potassium compositions in which the formation of impurities such as acetoacetamide during synthesis is reduced or eliminated.

All of the references discussed herein are hereby incorporated by reference.

SUMMARY OF THE INVENTION

The application discloses processes for producing a finished acesulfame potassium composition, the processes comprising the steps of: providing a crude acesulfame potassium composition comprising acesulfame potassium, acetoacetamide and water, concentrating the crude acesulfame potassium composition to form a water stream and an intermediate acesulfame potassium composition comprising acesulfame potassium and less than 33 wppm acetoacetamide (and optionally less than 33 wppm acetoacetamide-N-sulfonic acid), and separating the intermediate acesulfame potassium composition to form the finished acesulfame potassium composition comprising acesulfame potassium and less than 33 wppm acetoacetamide. The concentrating step is conducted at a temperature below 90° C., and the separating step is conducted at a temperature at or below 35° C. The weight percentage of acetoacetamide in the finished acesulfame potassium composition may be less than the weight percentage of acetoacetamide in the crude acesulfame potassium composition. The intermediate acesulfame potassium composition may comprise less than 33 wppm acetoacetamide-N-sulfonic acid. The providing of the crude acesulfame composition may comprise the steps of reacting sulfamic acid and an amine to form an amidosulfamic acid salt, reacting the amidosulfamic acid salt and acetoacetylating agent to form an acetoacetamide salt, reacting the acetoacetamide salt with cyclizing agent in the cyclizing agent composition to form the cyclic sulfur trioxide adduct, hydrolyzing the cyclic sulfur trioxide adduct to form an acesulfame-H composition comprising acesulfame-H, and neutralizing the acesulfame-H in the acesulfame-H composition to form the crude acesulfame potassium composition comprising acesulfame potassium and acetoacetamide. The concentrating step may comprise evaporating the crude acesulfame potassium composition to form the water stream and the intermediate acesulfame potassium composition comprising acesulfame potassium and less than 75 wt % water, and the evaporation residence time may be less than 180 minutes. The separating may comprise crystallizing the intermediate acesulfame potassium composition to form acesulfame potassium crystals and filtering the acesulfame potassium crystals to form the finished acesulfame potassium composition. Preferably, the concentrating comprises evaporating the crude acesulfame potassium composition to form a water stream and an intermediate acesulfame potassium composition comprising acesulfame potassium and less than 50 wt % water, and the separating comprises crystallizing the intermediate acesulfame potassium composition to form a crystal-containing stream comprising acesulfame potassium crystals, and filtering the crystal-containing stream to form the finished acesulfame potassium composition. The filtering may be conducted at a temperature at or below 35° C. and/or the crystallizing may be conducted at a temperature at or below 35° C. and/or may comprise at least two filtration operations. In some cases, the evaporating may be conducted at a temperature below 85° C. and the intermediate acesulfame potassium composition may comprise from 1 wppb to 33 wppm acetoacetamide (and optionally less than 33 wppm acetoacetamide-N-sulfonic acid) and the finished acesulfame potassium composition may comprise less than 33 wppm acetoacetamide. In one embodiment, the evaporating may be conducted at a temperature below 60° C. and the evaporator residence time is less than 50 minutes and the intermediate acesulfame potassium composition may comprise from 10 wppb to 25 wppm acetoacetamide (and optionally less than 30 wppm acetoacetamide-N-sulfonic acid) and the finished acesulfame potassium composition may comprise from 10 wppb to 15 wppm acetoacetamide. In one embodiment, the evaporating may be conducted at a temperature below 46° C., the evaporator residence time may be less than 30 minutes, the crystallizing may be conducted at a temperature below 35°

C., the intermediate acesulfame potassium composition may comprise from 10 wppb to 12 wppm acetoacetamide (and optionally less than 20 wppm acetoacetamide-N-sulfonic acid), and the finished acesulfame potassium composition may comprise from 10 wppb to 7 wppm acetoacetamide. In some cases, the evaporating is conducted at a temperature ranging from 20° C. to 55° C.; the evaporator residence time ranges from 1 minute to 300 minutes; the separating is conducted at a temperature ranging from −10° C. to 15° C.; the separating operation residence time ranges from 1 to 180 minutes; the crude acesulfame potassium composition comprises from 500 wppm to 2375 wppm acetoacetamide; the intermediate acesulfame potassium composition comprises 10 wppb to 20 wppm acetoacetamide and 10 wppb to 20 wppm acetoacetamide-N-sulfonic acid; and the finished acesulfame potassium composition comprises from 10 wppb to 10 wppm acetoacetamide, and from 1 wppb to 20 wppm acetoacetamide-N-sulfonic acid. The crystallizing may be conducted at a pH below 10. The crude acesulfame composition may further comprise solvent and wherein the process may further comprise removing solvent from the crude acesulfame potassium composition prior to the evaporation. The processes may comprise the step of separating from the acesulfame-H composition a transition phase comprising at least 2 wt % acetoacetamide to form a purified acesulfame-H composition, and the neutralizing may comprise neutralizing the acesulfame-H in the purified acesulfame-H composition to form the crude acesulfame potassium composition comprising acesulfame potassium and acetoacetamide. In one embodiment, the process comprises the steps of reacting sulfamic acid and triethylamine to form an amidosulfamic acid salt, reacting the amidosulfamic acid salt and diketene to form acetoacetamide salt, contacting dichloromethane and a sulfur trioxide to form a cyclizing agent composition, reacting the acetoacetamide salt with sulfur trioxide in the cyclizing agent composition to form a cyclic sulfur trioxide adduct, hydrolyzing the cyclic sulfur trioxide adduct to form an acesulfame-H composition, neutralizing the acesulfame-H to form the crude acesulfame potassium composition comprising acesulfame potassium and acetoacetamide, evaporating the crude acesulfame potassium composition to form a water stream and an intermediate acesulfame potassium composition comprising acesulfame potassium and less than 75 wt % water, crystallizing the intermediate acesulfame potassium composition to form acesulfame potassium crystals; and filtering the acesulfame potassium crystals to form the finished acesulfame potassium composition comprising acesulfame potassium and less than 10 wppm acetoacetamide. The evaporating may be conducted at a temperature below 50° C., evaporator residence time may be less than 30 minutes filtering may be conducted at a temperature below 35° C., and/or crystallizing may be conducted at a temperature below 35° C. The application also describes crude, intermediate, and finished acesulfame potassium composition produced by the processes described herein. In some cases, the application describes an acesulfame potassium composition comprising acesulfame potassium and less than 33 wppm, preferably less than 10 wppm acetoacetamide and optionally further comprises less than 33 wppm, preferably less than 10 wppm acetoacetamide-N-sulfonic acid. In some cases, the acesulfame potassium composition further comprises 0.001 wppm to 5 wppm organic impurities and/or 0.001 wppm to 5 wppm of at least one heavy metal, e.g., the at least one heavy metal being selected from the group consisting of mercury, lead and mixtures thereof. In some cases, the acesulfame potassium composition further comprises mercury present in an amount of 1 wppb to 20 wppm and/or lead present in an amount of 1 wppb to 25 wppm.

BRIEF DESCRIPTION OF THE DRAWINGS

The invention is described in detail below with reference to the appended drawing.

FIG. **1** is a process flow sheet of an acesulfame potassium production process in accordance with one embodiment of the present invention.

FIG. **2** is a process flow sheet of an acesulfame potassium production process employing one embodiment of a treatment scheme of the present invention.

DETAILED DESCRIPTION OF THE INVENTION

Introduction

Conventional processes for producing acesulfame potassium involve reacting sulfamic acid and an amine in the presence of acetic acid to form an amidosulfamic acid salt. The amidosulfamic acid salt is then reacted with an acetoacetylating agent, e.g., diketene, to form an acetoacetamide salt. The acetoacetamide salt is reacted with a cyclizing agent, e.g., sulfur trioxide, to form a cyclic sulfur trioxide adduct. The cyclic sulfur trioxide adduct is then hydrolyzed and neutralized via conventional means to form a crude acesulfame potassium composition comprising acesulfame potassium. This composition is phase separated into aqueous and organic phases. Most of the acesulfame potassium separates into the aqueous phase. As used herein, the term "crude acesulfame potassium composition" refers to the initial product of the neutralization reaction or to the aqueous phase that is formed from the phase separation step (without any further purification). The crude acesulfame potassium composition comprises at least 5 wt % acesulfame potassium. The crude acesulfame potassium composition may be optionally treated to form an "intermediate acesulfame potassium composition" and/or a "finished acesulfame potassium composition," which are discussed below.

Conventional acesulfame potassium compositions have been shown to comprise several undesirable impurities, among them acetoacetamide and acetoacetamide salts, e.g., acetoacetamide-N-sulfonate triethylammonium salt. Acetoacetamide-N-sulfonic acid and salts thereof may also be present. Content limits for these compounds in the finished acesulfame potassium composition are often determined by industry purity standards and/or by standards established for particular end use products that utilize acesulfame potassium as a sweetener. In some cases, limits for these impurities are determined by governmental regulations. For most applications, high acesulfame potassium purity levels are preferred. Thus, crude acesulfame potassium compositions typically are treated through various treatment operations to reduce the presence of these impurities. A non-limiting list of such treatment operations includes: evaporation, crystallization, and/or filtration.

Without being bound by theory, it has now been discovered that these treatment operations may create stress, e.g., thermal stress, on acesulfame potassium molecules. This thermal stress may also affect acesulfame-H, also known as sweetener acid, which is formed during the hydrolysis step and is a precursor to the acesulfame potassium. This stress on the acesulfame potassium and potentially on the acesulfame-H can result in degradation of these compounds, resulting in the formation of undesirable impurities. In some

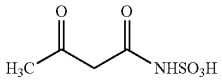
situations, this stress may cause the acesulfame potassium/acesulfame-H to degrade into its formation reaction reactants, e.g., acetoacetamide and/or salts thereof and/or acetoacetamide-N-sulfonic acid, which can lead to the formation of additional impurities.

It has also now been discovered that the use of specific treatment parameters may advantageously reduce or eliminate stress on the acesulfame potassium (or acesulfame-H) and/or reduce or eliminate product degradation, which in turn reduces or eliminates the formation of additional impurities and ultimately leads to a high-purity end product.

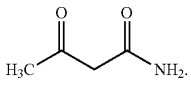
In particular, conducting the treatment (or the individual treatment steps) within certain temperature ranges or limits and/or maintaining treatment residence time within certain time ranges or limits now has been found to surprisingly reduce or eliminate acesulfame potassium (or acesulfame-H) degradation and impurity formation, examples of which include the (re)formation acetoacetamide and salts thereof. Traditionally, the treatment operations, e.g., evaporations, have been conducted at higher temperatures so as to improve process speed and by rapidly removing water. The reduced degradation of acesulfame potassium and acesulfame-H leads directly to the formation of the higher purity crude acesulfame potassium compositions discussed herein, thereby simplifying subsequent treatment operations for forming the intermediate or finished acesulfame potassium compositions. The process also advantageously leads to the formation of intermediate and finished acesulfame potassium compositions having low acetoacetamide-N-sulfamic acid and/or acetoacetamide content.

Additional specific terms that are used herein are now defined.

"Acetoacetamide-N-sulfonic acid" as used herein, refers to the molecule shown below. In some cases, acetoacetamide-N-sulfonic acid may be a degradation product of acesulfame potassium or acesulfame-H. The term "acetoacetamide-N-sulfonic acid," as used herein, also includes salts of acetoacetamide-N-sulfamic acid, e.g., potassium, sodium, and other alkali metal salts.



"Acetoacetamide," as used herein, refers to the following molecule:



Crude acesulfame compositions may be treated to form intermediate acesulfame potassium compositions and finished acesulfame compositions, and this treatment step may include one or more concentrating or separating operations.

An "intermediate acesulfame potassium composition" refers to a composition resulting from the concentrating of the crude acesulfame potassium composition, e.g., the removal of water from the crude acesulfame potassium composition. The intermediate acesulfame potassium composition comprises at least 10 wt % acesulfame potassium, based on the total weight of the intermediate acesulfame potassium composition, and has an acesulfame potassium weight percentage that is higher than that of the crude acesulfame potassium composition.

A "finished acesulfame potassium composition" refers to a composition (preferably directly) resulting from the separating, e.g., crystallizing and/or filtering, of the intermediate acesulfame potassium composition. The finished acesulfame potassium composition comprises at least 15 wt % acesulfame potassium, based on the total weight percentage of the finished acesulfame potassium composition, and has an acesulfame potassium weight percentage that is higher than that of the intermediate acesulfame potassium composition.

"Residence time," as used herein, refers to the time period that a composition (or stream) to be treated, e.g., a crude acesulfame potassium composition, remains in a particular treatment operation. Residence time begins when the composition to be treated enters the treatment operation, and residence time ends when the resultant compositions (formed via the treatment) exit the treatment operation. As one particular example, residence time for a concentrating operation, e.g., evaporation, refers to the time from when a crude acesulfame potassium composition enters the evaporator until the intermediate acesulfame potassium composition exits the evaporator. As another example, residence time for a separating operation, e.g., crystallization, refers to the time from when a crude acesulfame potassium composition enters the crystallizer until the intermediate acesulfame potassium composition exits the crystallizer.

The treatment of the crude acesulfame potassium composition may entail one or more operations, e.g., a concentrating operation and/or a separating operation. Generally, a concentrating operation is not considered a separating operation. In some embodiments, the concentrating operation(s) and the separating operation(s) make up the overall treatment of the crude acesulfame potassium composition, which results in the finished acesulfame potassium composition. In some cases, the overall concentrating operation may include multiple individual concentrating operations or units and the overall separating operation may include multiple individual separating operations or units.

"Cyclization reaction time," as used herein, refers to the time from the start of the acetoacetamide salt feed to the termination of the acetoacetamide salt feed. In some cases, if indicated, the cyclization reaction time may include additional time past the termination of the acetoacetamide salt feed, e.g., an extra 5 minutes or an extra minute.

"Wppm" and "wppb," as used herein, mean weight parts per million or weight parts per billion, respectively, and are based on the total weight of the entire respective composition, e.g., the total weight of the entire crude acesulfame potassium composition or the entire finished acesulfame potassium composition.

Acesulfame Potassium Formation

Processes for producing high purity acesulfame potassium compositions are described herein. In one embodiment, the process comprises the steps of providing a crude acesulfame potassium composition comprising acesulfame potassium and acetoacetamide (optionally present in an amount ranging from 1 wppb to 50 wppm) and treating the crude acesulfame potassium composition to form a finished acesulfame potassium composition. The treatment may comprise concentrating the crude acesulfame potassium composition to form a water stream and an intermediate acesulfame potassium composition comprising acesulfame potassium and low amounts of acetoacetamide and then separating the intermediate acesulfame potassium composition to form the finished acesulfame potassium composition comprising acesulfame potassium and low amounts of

acetoacetamide. As noted above, the crude acesulfame potassium composition may be formed by reacting sulfamic acid and an amine to form an amidosulfamic acid salt and then reacting the amidosulfamic acid salt with an acetoacetylating agent to form an acetoacetamide salt. The acetoacetamide salt may then be cyclized, hydrolyzed, and neutralized (and optionally phase separated). These steps are described in more detail below.

Importantly, in some embodiments, certain parameters of the concentrating and/or the separating operations are maintained at particular levels and/or within particular ranges. In some cases, the temperatures at which the concentrating and/or the separating operations are conducted are maintained at low levels. Also, in some embodiments, the residence times (of the crude acesulfame potassium composition in the concentrating operation or of the intermediate acesulfame potassium composition in the separating operation) are maintained at a low level. As a result, without being bound by theory, little or no additional impurities, e.g., acetoacetamide, are generated during treatment, e.g., during the concentrating and/or separating operations, which advantageously provides for a more pure finished acesulfame potassium composition. In some cases, the weight percentage of acetoacetamide in the finished acesulfame potassium composition or in the intermediate acesulfame potassium composition is less than the weight percentage of acetoacetamide in the crude acesulfame potassium composition, i.e., acetoacetamide content is actually reduced during treatment.

In some embodiments, the concentrating operation, e.g., one or more of the steps that make up the concentrating operation, is conducted at or maintained at a low temperature, e.g., a temperature below 90° C., e.g., below 88° C., below 85° C., below 83° C., below 80° C., below 78° C., below 75° C., below 73° C., below 70° C., below 65° C., below 55° C., below 50° C., or below 46° C. In some cases, the temperature of the concentrating operation may be maintained at a temperature above 0° C., e.g., above 10° C., above 15° C., above 20° C., above 22° C., above 25° C., above 35° C., above 40° C. or above 50° C. In terms of ranges, the temperature of the concentrating operation may range from 0° C. to 90° C., e.g., 25° C. to 90° C., from 55° C. to 90° C., from 10° C. to 88° C., from 10° C. to 85° C., from 75° C. to 88° C., from 80° C. to 88° C., from 15° C. to 85° C., from 75° C. to 85° C., from 20° C. to 83° C., from 20° C. to 80° C., from 22° C. to 78° C., from 25° C. to 75° C., from 25° C. to 73° C., from 15° C. to 50° C., from 25° C. to 65° C., from 22° C. to 50° C., from 20° C. to 55° C., from 25° C. to 70° C., or from 30° C. to 60° C.

In some embodiments, the separating operation, e.g., one or more of the steps that make up the separating operation, is conducted at or maintained at a low temperature, e.g., a temperature below 35° C., below 30° C., below 25° C., below 20° C., below 15° C., below 10° C., below 8° C., below 6° C., below 5° C., or below 0° C. In some cases, the temperature of the separating operation may be maintained at a temperature above −25° C., e.g., above −10° C., above 0° C., above 5° C., above 10° C., above 15° C., above 25° C., or above 30° C. In terms of ranges, the temperature of the separating operation may range from −25° C. to 35° C., e.g., −10° C. to 35° C., from 0° C. to 35° C., from 5° C. to 30° C., from −10° C. to 30° C., from −10° C. to 25° C., from −10° C. to 20° C., from −10° C. to 15° C., from 0° C. to 25° C., or from −10° C. to 30° C. The employment of the aforementioned temperatures in the treatment advantageously improves final product purity.

In some embodiments, the concentrating operation, e.g., one or more of the steps that make up the concentrating operation, is conducted at or maintained at a low residence time. In one embodiment, residence time is less than 180 minutes, e.g., less than 170 minutes, less than 150 minutes, less than 120 minutes, less than 100 minutes, less than 90 minutes, less than 75 minutes, less than 50 minutes, less than 40 minutes, less than 30 minutes, less than 20 minutes, or less than 10 minutes. In terms of lower limits, residence time may be at least 1 second, e.g., at least 10 seconds, at least 1 minute, at least 10 minutes, or at least 15 minutes. In terms of ranges, the residence time may range from 1 second to 180 minutes, e.g., from 10 seconds to 180 minutes, from 1 minute to 180 minutes, from 10 minutes to 150 minutes, from 1 minute to 50 minutes, from 1 minute to 30 minutes, from 10 minutes to 100 minutes, from 1 minute to 80 minutes, from 10 minutes to 80 minutes, from 10 minutes to 50 minutes, from 15 minutes to 90 minutes, or from 15 minutes to 75 minutes. The same residence time limits and ranges are applicable to the separating operation, e.g., one or more of the steps that make up the separating operation. The employment of residence times in the concentrating operation and/or separating operation advantageously improves final product purity.

In some embodiments, the concentrating operation, e.g., one or more of the steps that make up a concentrating operation, is conducted at or maintained at a low pH. In one embodiment, the pH of the separating is maintained below 10.0, e.g., below 9.5, below 9.0, below 8.5, below 8.0, below 7.5, below 7.0, or below 6.5. In terms of ranges, the pH of the concentrating operation is preferably maintained between 6.0 and 10.0, e.g., between 6.5 and 9.5, between 7.0 and 9.0, or between 7.5 and 8.5. The same pH limits and ranges are applicable to the separating operation, e.g., one or more of the steps that make up the separating operation. The employment of low pH levels in the concentrating operation or separating operation advantageously improves final product purity.

In cases where evaporation is utilized in the concentrating operation, the evaporation may be conducted at the aforementioned temperature limits and ranges. It has been discovered that, in addition to the aforementioned impurity reduction benefits, the utilization of lower evaporation temperatures surprisingly limits or eliminates the formation of solids in the evaporator, e.g., solid acesulfame potassium, which can lead to safety issues, e.g., unnecessary pressure build-up or explosion of the evaporator.

The aforementioned parameter limits and ranges are applicable to individual concentrating or separating operations that may make up the overall concentrating or separating operation. For example, if the concentrating operation may comprise evaporation, then the evaporation may be conducted at a temperature below 90° C., e.g., below 88° C., below 85° C., below 83° C., below 80° C., below 78° C., below 75° C., below 73° C., below 70° C., below 65° C., below 55° C., below 50° C., or below 46° C. As another example, if the concentrating operation may comprise evaporation, then the evaporation may be conducted at a residence time less than 180 minutes, e.g., less than 170 minutes, less than 150 minutes, less than 120 minutes, less than 100 minutes, less than 90 minutes, less than 75 minutes, less than 50 minutes, less than 40 minutes, less than 30 minutes, less than 20 minutes, or less than 10 minutes. As another example, if the separating operation comprises crystallization, then the crystallization may be conducted at a pH below 10.0, e.g., below 9.5, below 9.0, below 8.5, below 8.0, below 7.5, below 7.0, or below 6.5.

By performing the treatment under the temperature, pH, and/or residence time parameters discussed herein, stress on the acesulfame potassium molecules (in the crude acesulfame potassium composition) is advantageously minimized during the separating operation. As a result, less acesulfame potassium degrades into the acetoacetamide during the separating operation. Thus, the intermediate acesulfame potassium product and the finished acesulfame potassium composition advantageously contain lower amounts of impurities, e.g., acetoacetamide (if any) than would typically form from acesulfame potassium degradation.

The concentrating operation, in some embodiments, comprises the step of evaporating the crude acesulfame potassium composition to form a water stream and an intermediate acesulfame potassium composition comprising acesulfame potassium and less than 75 wt % water, e.g., less than 50 wt %, less than 40 wt %, less than 30 wt %, less than 20 wt %, less than 15 wt %, less than 10 wt %, less than 50 wt %, less than 3 wt %, or less than 1 wt %. The water stream may refer to water that is evaporated from the crude acesulfame potassium composition, e.g., water that is not present in the intermediate acesulfame potassium composition. The evaporating may be conducted at the concentrating operation parameters mentioned herein.

In some cases, the separating comprises the step of crystallizing the intermediate acesulfame potassium composition or a derivative thereof, to form the finished acesulfame potassium composition, which may be in the form of acesulfame potassium crystals (or a composition/stream comprising a the acesulfame potassium crystals). The intermediate acesulfame potassium composition may be a stream or a composition that results from the concentration of the crude acesulfame potassium composition. The crystallizing may be conducted at the separating operation parameters mentioned herein.

In some embodiments the separating comprises the step of filtering the intermediate acesulfame potassium composition (or a crystal-containing derivative thereof) to form the finished acesulfame potassium composition. A crystal-containing derivative of the intermediate acesulfame potassium composition may be a stream or a composition that results from the concentration of the crude acesulfame potassium composition and that contains crystals either in dissolved or solid form. The filtering may be conducted at the separating operation parameters mentioned herein.

In preferred embodiments, the overall treatment comprises the steps of evaporating the crude acesulfame potassium composition to form a water stream and the intermediate acesulfame potassium composition comprising acesulfame potassium and low amounts of water (see limits/ranges above), crystallizing the intermediate acesulfame potassium composition to form acesulfame potassium crystals, and filtering the acesulfame potassium crystals to form the finished acesulfame potassium composition.

In one embodiment, the process comprises the steps of providing the crude acesulfame potassium composition comprising acesulfame potassium and acetoacetamide and water and evaporating the crude acesulfame potassium composition to form the water stream and the intermediate acesulfame potassium composition (as disclosed above). In this embodiment, the residence time of the crude acesulfame potassium composition in the evaporator is less than 180 minutes, e.g., less than 170 minutes, less than 150 minutes, less than 120 minutes, less than 100 minutes, less than 90 minutes, less than 75 minutes, less than 50 minutes, less than 40 minutes, less than 30 minutes, less than 20 minutes, or less than 10 minutes. In terms of ranges, the residence time may range from 1 second to 180 minutes, e.g., from 10 seconds to 180 minutes, from 1 minute to 180 minutes, from 10 minutes to 150 minutes, from 10 minutes to 100 minutes, from 10 minutes to 80 minutes, from 10 minutes to 50 minutes, from 15 minutes to 90 minutes, or from 15 minutes to 75 minutes.

In some embodiments, the forming of the finished acesulfame potassium composition from the intermediate acesulfame potassium composition comprises crystallizing the intermediate acesulfame potassium composition to form acesulfame potassium crystals and filtering the crystal-containing stream to form the finished acesulfame potassium composition. In preferred embodiments, a falling film evaporator is employed to form the intermediate acesulfame potassium composition.

The crude acesulfame composition may further comprise solvent, and in some embodiments, the concentrating operation comprises a solvent removal step, e.g., stripping solvent from the crude acesulfame potassium composition, e.g., prior to concentrating (evaporating). The process may comprise the steps of providing the crude acesulfame potassium composition, stripping the crude acesulfame potassium composition to form a solvent stream comprising solvent and a stripped acesulfame potassium composition comprising less than 50 wt % solvent, e.g., less than 40 wt %, less than 30 wt %, less than 20 wt %, less than 15 wt %, less than 10 wt %, less than 50 wt %, less than 3 wt %, or less than 1 wt %, followed by the aforementioned concentrating operation and separating operation. It has been found that removal of solvent, e.g., methylene dichloride, surprisingly increases concentration efficiency. In addition to the stripping step, the separating operation may further comprise the evaporation, crystallization, and/or filtration steps.

In some embodiments, the separating operation comprises the step of crystallizing the intermediate acesulfame potassium composition to form a crystal-containing stream comprising acesulfame potassium crystals. The crystallization may be conducted at the pH ranges and limits discussed above. The process may further comprise the step of forming from the crystal-containing stream the finished acesulfame potassium composition. In some embodiments, the forming step comprises filtering the crystal-containing stream to form the finished acesulfame potassium composition. This embodiment may also utilize the aforementioned solvent stripping step.

If filtration is employed in the separating operation, the filtration is preferably conducted at the separating operation temperature limits and ranges discussed herein. In cases where crystallization is utilized in the separating operation, the crystallization may be conducted at the temperature limits and ranges discussed herein.

In addition to the temperature limits and ranges, the crystallization may be conducted at the pH limits and ranges discussed herein. For example, the crystallization may be conducted at a pH below 10.0, e.g., below 9.5, below 9.0, below 8.5, below 8.0, below 7.5, below 7.0, or below 6.5. In addition to the benefits of reducing acetoacetamide formation, it has also been found that conducting the crystallization also improves separation of dimers that may form in side reaction. It is postulated that the lower pH levels promote precipitation of dimers. Higher pH levels have been found to promote dimer solubility. The precipitation advantageously provides for more efficient separation thereof.

In addition to the temperature limits and ranges, the crystallization may be conducted at the residence time limits and ranges discussed herein.

In one embodiment, the provision of the crude acesulfame potassium composition (which is subsequently concentrated and separated) comprises the steps of reacting sulfamic acid and an amine to form an amidosulfamic acid salt and reacting the amidosulfamic acid salt with the acetoacetylating agent to form the acetoacetamide salt. The acetoacetamide salt may then be reacted with a cyclizing agent, optionally in the presence of a solvent, to form the cyclic (sulfur trioxide) adduct composition. In preferred embodiments, the provision of the crude acesulfame potassium composition further comprises the step of hydrolyzing the cyclic sulfur trioxide adduct to form acesulfame-H, and neutralizing the acesulfame-H to form the crude acesulfame potassium composition. In some embodiments, the neutralizing step comprises reacting the acesulfame-H (optionally in the acesulfame-H composition) with a neutralizing agent to form the acesulfame potassium composition. The reacting may comprise contacting the acesulfame-H with the neutralizing agent. The acesulfame potassium composition comprises acesulfame potassium and impurities. The formation of the cyclic sulfur trioxide adduct may yield a cyclic sulfur trioxide adduct composition that comprises the cyclic sulfur trioxide adduct and additional reaction side products and impurities. Similarly, the formation of the acesulfame-H may yield an acesulfame-H composition that comprises acesulfame-H and additional reaction side products and impurities.

The specific methods employed to implement the desired temperature and/or residence time features may vary widely. With regard to temperature, heat exchange equipment, e.g., cooling equipment, may be employed. Temperature maintenance methods are well known in the art.

With regard to residence time, the respective separation streams may be controlled to maintain the residence times discussed herein using the appropriate valves, gauges, metering devices, and piping.

The acesulfame potassium composition formed via the process(es) described herein will be a high purity acesulfame potassium composition. For example, the acesulfame potassium composition may comprise acetoacetamide salts in the amounts discussed above.

Acesulfame Potassium Compositions

The crude acesulfame potassium composition is formed by hydrolyzing a cyclic sulfur trioxide adduct to form an acesulfame-H composition and neutralizing the acesulfame-H in the acesulfame-H composition to form the crude acesulfame potassium composition, as discussed herein. The product of the neutralization step is phase separated into aqueous and organic phases. The crude acesulfame potassium composition may be obtained from the aqueous phase (without any further purification). The crude acesulfame potassium composition comprises acesulfame potassium and acetoacetamide, e.g., less than 2800 wppm acetoacetamide, e.g., less than 2700 wppm, less than 2600 wppm, less than 2500 wppm, less than 2400 wppm, less than 2000 wppm, less than 1500 wppm, less than 1000 wppm, less than 500 wppm, or less than 100 wppm. In some cases the crude acesulfame potassium composition is substantially free of acetoacetamide (undetectable), e.g., free of acetoacetamide. In terms of ranges, the crude acesulfame potassium composition may comprise from 1 wppm to 2800 wppm acetoacetamide, e.g., from 1 wppm to 2700 wppm, from 10 wppm to 2700 wppm, from 20 wppm to 2500 wppm, from 100 wppm to 2500 wppm, from 500 wppm to 2500 wppm, from 1500 to 2400 wppm, from 500 wppm to 2375 wppm, from 600 wppm to 2000 wppm, from 900 to 1900 wppm, from 300 wppm to 1500 wppm, from 400 wppm to 1400 wppm, from 600 wppm to 1200 wppm or from 700 wppm to 1100 wppm.

The crude acesulfame potassium composition may further comprise acetoacetamide-N-sulfonic acid, which may be present in the amounts discussed above with respect to acetoacetamide.

The finished acesulfame potassium compositions, which are typically suitable for end consumer usage, are formed by treating the crude acesulfame potassium composition to remove impurities, as discussed herein. This finished acesulfame potassium composition preferably comprises a mixture of acesulfame potassium and less than 33 wppm acetoacetamide, e.g., less than 32 wppm, less than 30 wppm, less than 25 wppm, less than 20 wppm, less than 15 wppm, less than 12 wppm, less than 10 wppm, less than 7 wppm, less than 5 wppm, less than 3 wppm, less than 1 wppm, less than 0.8 wppm, less than 0.5 wppm, or less than 0.3 wppm. In some cases the finished acesulfame potassium composition is substantially free of acetoacetamide, e.g., free of acetoacetamide. In terms of ranges, the finished acesulfame potassium composition may comprise from 1 wppb to 33 wppm acetoacetamide, e.g., from 10 wppb to 32 wppm, from 10 wppb to 25 wppm, from 10 wppb to 15 wppm, from 10 wppb to 12 wppm, from 10 wppb to 10 wppm, from 10 wppb to 7 wppm, from 10 wppb to 5 wppm, from 10 wppb to 3 wppm, from 100 wppb to 15 wppm, from 100 wppb to 10 wppm, or from 100 wppb to 5 wppm.

The finished acesulfame potassium composition preferably comprises acesulfame potassium and less than 33 wppm acetoacetamide-N-sulfonic acid, e.g., less than 32 wppm, less than 30 wppm, less than 25 wppm, less than 20 wppm, less than 15 wppm, less than 12 wppm, less than 10 wppm, less than 7 wppm, less than 5 wppm, less than 3 wppm, less than 1 wppm, less than 0.8 wppm, less than 0.5 wppm, or less than 0.3 wppm. In some cases the finished acesulfame potassium composition is substantially free of acetoacetamide-N-sulfonic acid, e.g., free of acetoacetamide-N-sulfonic acid. In terms of ranges, the finished acesulfame potassium composition may comprise from 1 wppb to 33 wppm acetoacetamide-N-sulfonic acid, e.g., from 10 wppb to 32 wppm, from 10 wppb to 25 wppm, from 1 wppb to 22 wppm, from 10 wppb to 22 wppm, from 1 wppb to 20 wppm, from 10 wppb to 20 wppm, from 10 wppb to 15 wppm, from 10 wppb to 12 wppm, from 10 wppb to 10 wppm, from 10 wppb to 7 wppm, from 10 wppb to 5 wppm, from 10 wppb to 3 wppm, from 100 wppb to 15 wppm, from 100 wppb to 10 wppm, or from 100 wppb to 5 wppm.

The acetoacetamide-N-sulfonic acid and/or the acetoacetamide content may be measured in the crude, intermediate, or finished acesulfame potassium compositions via high performance liquid chromatography (HPLC) analysis, based on European Pharmacopoeia guidelines for thin layer chromatography (2017) and adapted for HPLC. A particular measurement scenario utilizes an LC Systems HPLC unit from Shimadzu having a CBM-20 Shimadzu controller and being equipped with an IonPac NS1 ((5 μm) 150×4 mm) analytical column and an IonPac NG1 guard column (35×4.0 mm). A Shimadzu SPD-M20A photodiode array detector can be used for detection (at 270 nm and 280 nm wavelength). Analysis may be performed at 23° C. column temperature. As a first eluent solution, an aqueous mixture of tetra butyl ammonium hydrogen sulfate (3.4 g/L), acetonitrile (300 mL/L), and potassium hydroxide (0.89 g/L) may be employed; as a second eluent solution, an aqueous mixture of tetra butyl ammonium hydrogen sulfate (3.4 g/L)

and potassium hydroxide (0.89 g/L) may be employed. Elution may be conducted in gradient mode according to the following second eluent flow profile:

0 to 3 minutes: constant 80% (v/v)
3 to 6 minutes: linear reduction to 50% (v/v)
6 to 15 minutes: constant at 50% (v/v)
15 to 18 minutes: linear reduction to 0%
18 to 22 minutes: constant at 0%
22 to 24 minutes: linear increase to 80% (v/v)
24 to 35 minutes constant at 80% (v/v).

Overall flow rate of eluent may be approximately 1.2 mL/min. The data collection and calculations may be performed using Lab Solution software from Shimadzu.

One or more intermediate acesulfame potassium compositions may be formed, e.g., during the concentrating operation, for example via evaporation. The intermediate acesulfame potassium composition preferably comprises a mixture of acesulfame potassium and less than 33 wppm acetoacetamide, e.g., less than 30 wppm, less than 25 wppm, less than 20 wppm, less than 15 wppm, less than 12 wppm, less than 10 wppm, less than 7 wppm, less than 5 wppm, less than 3 wppm, less than 1 wppm, less than 0.8 wppm, less than 0.5 wppm, or less than 0.3 wppm. In some cases the intermediate acesulfame potassium composition is free of acetoacetamide, e.g., substantially free of acetoacetamide (undetectable). In terms of ranges, the intermediate acesulfame potassium composition may comprise from 1 wppb to 33 wppm acetoacetamide, e.g., from 10 wppb to 30 wppm, from 10 wppb to 25 wppm, from 10 wppb to 15 wppm, from 10 wppb to 12 wppm, from 10 wppb to 10 wppm, from 10 wppb to 7 wppm, from 10 wppb to 5 wppm, from 10 wppb to 3 wppm, from 100 wppb to 15 wppm, from 100 wppb to 10 wppm, from 100 wppb to 5 wppm. The intermediate acesulfame potassium composition may comprise a mixture of acesulfame potassium and acetoacetamide.

As noted above, the crude acesulfame potassium composition is formed by the aforementioned reactions, hydrolysis, and neutralization. The crude acesulfame potassium composition is concentrated to form the intermediate acesulfame composition, which is then separated to form the finished acesulfame potassium composition, as discussed herein. In preferred embodiments, the temperature at which the concentrating operation, e.g., the evaporation, is conducted is at or below 90° C., e.g., below 88° C., below 85° C., below 83° C., below 80° C., below 78° C., below 75° C., below 73° C., below 70° C., below 65° C., below 55° C., below 50° C., or below 46° C. (optionally at a temperature ranging from 0° C. to 90° C., e.g., 25° C. to 90° C., from 55° C. to 90° C., from 10° C. to 88° C., from 10° C. to 85° C., from 75° C. to 88° C., from 80° C. to 88° C., from 15° C. to 85° C., from 75° C. to 85° C., from 20° C. to 83° C., from 20° C. to 80° C., from 22° C. to 78° C., from 25° C. to 75° C., from 25° C. to 73° C., from 15° C. to 50° C., from 25° C. to 65° C., from 22° C. to 50° C., from 20° C. to 55° C., from 25° C. to 70° C., or from 30° C. to 60° C.); the concentrating operation utilizes a residence time less than 180 minutes, e.g., less than 170 minutes, less than 150 minutes, less than 120 minutes, less than 100 minutes, less than 90 minutes, less than 75 minutes, less than 50 minutes, less than 40 minutes, less than 30 minutes, less than 20 minutes, or less than 10 minutes (optionally utilizing a residence time ranging from 1 second to 180 minutes, e.g., from 10 seconds to 180 minutes, from 1 minute to 180 minutes, from 10 minutes to 150 minutes, from 1 minute to 50 minutes, from 1 minute to 30 minutes, from 10 minutes to 100 minutes, from 10 minutes to 80 minutes, from 10 minutes to 50 minutes, from 15 minutes to 90 minutes, or from 15 minutes to 75 minutes); the temperature at which the separating operation, e.g., the crystallization and/or filtration, is conducted is below 35° C., e.g., below 30° C., below 25° C., below 20° C., below 15° C., below 10° C., below 8° C., below 6° C., below 5° C., or below 0° C. (optionally at a temperature ranging from −25° C. to 35° C., e.g., −10° C. to 35° C., from 0° C. to 35° C., from 5° C. to 30° C., from −10° C. to 30° C., from −10° C. to 25° C., from −10° C. to 20° C., from −10° C. to 15° C., from 0° C. to 25° C., or from −10° C. to 30° C.); the separating operation utilizes a residence time less than 180 minutes, e.g., less than 170 minutes, less than 150 minutes, less than 120 minutes, less than 100 minutes, less than 90 minutes, less than 75 minutes, less than 50 minutes, less than 40 minutes, less than 30 minutes, less than 20 minutes, or less than 10 minutes (optionally utilizing a residence time ranging from 1 second to 180 minutes, e.g., from 10 seconds to 180 minutes, from 1 minute to 180 minutes, from 10 minutes to 150 minutes, from 10 minutes to 100 minutes, from 1 minute to 80 minutes, from 10 minutes to 80 minutes, from 10 minutes to 50 minutes, from 15 minutes to 90 minutes, or from 15 minutes to 75 minutes); the crude acesulfame potassium composition may comprise less than 2800 wppm acetoacetamide, e.g., less than 2700 wppm, less than 2600 wppm, less than 2500 wppm, less than 2400 wppm, less than 2000 wppm, less than 1500 wppm, less than 1000 wppm, less than 500 wppm, or less than 100 wppm (optionally from 1 wppm to 2800 wppm acetoacetamide, e.g., from 1 wppm to 2800 wppm, from 10 wppm to 2700 wppm, from 20 wppm to 2500 wppm, from 100 wppm to 2500 wppm, from 500 wppm to 2500 wppm, from 1500 to 2400 wppm, from 500 wppm to 2375 wppm, from 600 wppm to 2000 wppm, from 900 to 1900 wppm, from 300 wppm to 1500 wppm, from 400 wppm to 1400 wppm, from 600 wppm to 1200 wppm or from 700 wppm to 1100 wppm) (the crude acesulfame potassium composition may comprise acetoacetamide-N-sulfonic acid in the same amounts); the intermediate acesulfame potassium composition may comprise less than 33 wppm acetoacetamide, e.g., less than 32 wppm, less than 30 wppm, less than 25 wppm, less than 20 wppm, less than 15 wppm, less than 12 wppm, less than 10 wppm, less than 7 wppm, less than 5 wppm, less than 3 wppm, less than 1 wppm, less than 0.8 wppm, less than 0.5 wppm, or less than 0.3 wppm (optionally from 1 wppb to 33 wppm acetoacetamide, e.g., from 10 wppb to 32 wppm, from 10 wppb to 25 wppm, from 10 wppb to 15 wppm, from 10 wppb to 12 wppm, from 10 wppb to 10 wppm, from 10 wppb to 7 wppm, from 10 wppb to 5 wppm, from 10 wppb to 3 wppm, from 100 wppb to 15 wppm, from 100 wppb to 10 wppm, from 100 wppb to 5 wppm) (the intermediate acesulfame potassium composition may comprise acetoacetamide-N-sulfonic acid in the same amounts); and the finished acesulfame potassium composition may comprise less than 33 wppm acetoacetamide, e.g., less than 32 wppm, less than 30 wppm, less than 25 wppm, less than 20 wppm, less than 15 wppm, less than 12 wppm, less than 10 wppm, less than 7 wppm, less than 5 wppm, less than 3 wppm, less than 1 wppm, less than 0.8 wppm, less than 0.5 wppm, or less than 0.3 wppm (optionally from 1 wppb to 33 wppm acetoacetamide, e.g., from 10 wppb to 32 wppm, from 10 wppb to 25 wppm, from 10 wppb to 15 wppm, from 10 wppb to 12 wppm, from 10 wppb to 10 wppm, from 10 wppb to 7 wppm, from 10 wppb to 5 wppm, from 10 wppb to 3 wppm, from 100 wppb to 15 wppm, from 100 wppb to 10 wppm, from 100 wppb to 5 wppm) (the finished acesulfame potassium composition may comprise acetoacetamide-N-sulfonic acid in the same amounts).

In a particular embodiment, the concentrating operation, e.g., the evaporation, is conducted at a temperature below 85° C., the intermediate acesulfame potassium composition comprises from 1 wppb to 33 wppm acetoacetamide (and optionally less than 33 wppm acetoacetamide-N-sulfonic acid), and the finished acesulfame potassium composition comprises less than 33 wppm acetoacetamide (and optionally less than 33 wppm acetoacetamide-N-sulfonic acid).

In another particular embodiment, the concentrating operation, e.g., the evaporation, is conducted at a temperature below 60° C., the evaporator residence time is less than 50 minutes and the intermediate acesulfame potassium composition comprises from 10 wppb to 25 wppm acetoacetamide (and optionally less than 30 wppm acetoacetamide-N-sulfonic acid), and the finished acesulfame potassium composition comprises from 10 wppb to 15 wppm acetoacetamide (and optionally less than 30 wppm acetoacetamide-N-sulfonic acid).

In another particular embodiment, the concentrating operation, e.g., the evaporation, is conducted at a temperature below 46° C., the evaporator residence time is less than 30 minutes, the crystallizing is conducted at a temperature below 35° C., the intermediate acesulfame potassium composition comprises from 10 wppb to 12 wppm acetoacetamide (and optionally less than 20 wppm acetoacetamide-N-sulfonic acid), and the finished acesulfame potassium composition comprises from 10 wppb to 7 wppm acetoacetamide.

In another particular embodiment, the concentrating operation, e.g., the evaporation, is conducted at a temperature ranging from 25° C. to 90° C., evaporator residence time ranges from 10 seconds to 180 minutes, the separating operation, e.g., the crystallization and/or filtration, is conducted at a temperature ranging from −10° C. to 35° C., the separating operation residence time ranges from 10 seconds to 180 minutes, the crude acesulfame potassium composition comprises 1 wppm to 2800 wppm acetoacetamide and 1 wppm to 2800 wppm acetoacetamide-N-sulfonic acid, the intermediate acesulfame potassium composition comprises 1 wppb to 33 wppm acetoacetamide and 1 wppb to 33 wppm acetoacetamide-N-sulfonic acid, and the finished acesulfame potassium composition comprises 1 wppb to 33 wppm acetoacetamide, and 1 wppb to 33 wppm acetoacetamide-N-sulfonic acid.

In another particular embodiment, the concentrating operation, e.g., the evaporation, is conducted at a temperature ranging from 25° C. to 90° C., evaporator residence time ranges from 10 seconds to 180 minutes, the separating operation, e.g., the crystallization and/or filtration, is conducted at a temperature ranging from −10° C. to 35° C., the separating operation residence time ranges from 10 seconds to 180 minutes, the crude acesulfame potassium composition comprises 1 wppm to 2800 wppm acetoacetamide and 1 wppm to 2800 wppm acetoacetamide-N-sulfonic acid, the intermediate acesulfame potassium composition comprises 1 wppb to 33 wppm acetoacetamide and 1 wppb to 33 wppm acetoacetamide-N-sulfonic acid, and the finished acesulfame potassium composition comprises 1 wppb to 33 wppm acetoacetamide, and 1 wppb to 33 wppm acetoacetamide-N-sulfonic acid.

In another particular embodiment, the concentrating operation, e.g., the evaporation, is conducted at a temperature ranging from 20° C. to 55° C., evaporator residence time ranges from 1 minute to 300 minutes, the separating operation, e.g., the crystallization and/or filtration, is conducted at a temperature ranging from −10° C. to 15° C., the separating operation residence time ranges from 1 to 180 minutes, the crude acesulfame potassium composition comprises from 500 wppm to 2375 wppm acetoacetamide, the intermediate acesulfame potassium composition comprises 10 wppb to 20 wppm acetoacetamide and 10 wppb to 20 wppm acetoacetamide-N-sulfonic acid, and the finished acesulfame potassium composition comprises from 10 wppb to 10 wppm acetoacetamide, and from 1 wppb to 20 wppm acetoacetamide-N-sulfonic acid.

The acesulfame potassium compositions (crude and/or finished) may, in some cases, comprise organic impurities. Organic impurities include, inter alia, halo-acesulfame potassium. The acesulfame potassium compositions (crude and/or finished) also may comprise heavy metals. The organic impurities and/or heavy metals may be present in an amount ranging from 1 wppb to 25 wppm, based on the total weight of the respective acesulfame potassium composition, crude or finished, e.g., from 100 wppb to 20 wppm, from 100 wppb to 15 wppm, from 500 wppb to 10 wppm, or from 1 wppm to 5 wppm. Heavy metals are defined as metals with relatively high densities, e.g., greater than 3 g/cm^3 or greater than 7 g/cm^3. Exemplary heavy metals include lead and mercury. In some cases, the crude or finished acesulfame potassium composition may comprise mercury in an amount ranging from 1 wppb to 25 wppm, e.g., from 100 wppb to 20 wppm, from 100 wppb to 15 wppm, from 500 wppb to 10 wppm, or from 1 wppm to 5 wppm. In terms of limits, the crude or finished acesulfame potassium composition may comprise less than 25 wppm mercury, e.g., less than 20 wppm, less than 15 wppm, less than 10 wppm, or less than 5 wppm. In some cases, the crude or finished acesulfame potassium composition may comprise lead in an amount ranging from 1 wppb to 25 wppm, e.g., from 100 wppb to 20 wppm, from 100 wppb to 15 wppm, from 500 wppb to 10 wppm, or from 1 wppm to 5 wppm. In terms of limits, the crude or finished acesulfame potassium composition may comprise less than 25 wppm lead, e.g., less than 20 wppm, less than 15 wppm, less than 10 wppm, or less than 5 wppm. In some cases, when potassium hydroxide is formed via a membrane process, the resultant crude or finished acesulfame potassium composition may have very low levels of mercury, if any, e.g., less than 10 wppm, less than 5 wppm, less than 3 wppm, less than 1 wppm, less than 500 wppb, or less than 100 wppb.

Intermediate Reaction Parameters

The reactions for production of high purity acesulfame potassium are described in more detail below.

Amidosulfamic Acid Salt Formation Reaction

In a first reaction step, sulfamic acid and an amine are reacted to form sulfamic acid salt. An exemplary reaction scheme that employs triethylamine as the amine and yields triethyl ammonium sulfamic acid salt is shown in reaction (1), below.

$$H_2N—SO_3H+N(C_2H_5)_3 \rightarrow H_2N—SO_3^-.HN^+(C_2H_5)_3 \quad (1)$$

Acetic acid is also present in the first reaction mixture and reacts with the amine, e.g., triethylamine, to form a triethylammonium acetate, as shown in reaction (2), below.

$$H_3C—COOH+N(C_2H_5)_3 \rightarrow H_3C—COO^-.HN^+(C_2H_5)_3 \quad (2)$$

The amine employed in these reactions may vary widely. Preferably, the amine comprises triethylamine. In one embodiment, the amine may be selected from the group consisting of trimethylamine, diethylpropylamine, tri-n-propylamine, triisopropylamine, ethyldiisopropylamine, tri-n-butylamine, triisobutylamine, tricyclohexylamine, ethyldicyclohexylamine, N,N-dimethylaniline, N,N-diethylaniline, benzyldimethylamine, pyridine, substituted pyridines such

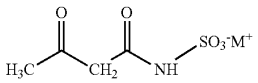
as picoline, lutidine, cholidine or methylethylpyridine, N-methylpiperidine, N-ethylpiperidine, N-methylmorpholine, N,N-dimethylpiperazine, 1,5-diazabicyclo[4.3.0]-non-5-en, 1,8-diazabicyclo-[5.4.0]-undec-7-en, 1,4-diazabicyclooctane, tetramethylhexamethylendiamine, tetramethylethylendiamine, tetramethylpropylendiamine, tetramethylbutylendiamine, 1,2-dimorpholylethan, pentamethyldiethyltriamine, pentaethyldiethylentriamine, pentamethyldipropylentriamine, tetramethyldiaminomethane, tetrapropyldiaminomethane, hexamethyltriethylentetramine, hexamethyltripropylenetetramine, diisobutylentriamine, triisopropylentriamine, and mixtures thereof.

Acetoacetamide Salt Formation Reaction

Once formed in reaction (1), the sulfamic acid salt is reacted with the acetoacetylating agent to form the acetoacetamide salt, preferably acetoacetamide-N-sulfonate triethylammonium salt. Preferably, the acetoacetylating agent comprises diketene, although other acetoacetylating agents may be employed, either with or without diketene.

In one embodiment, the resultant acetoacetamide salt corresponds to the following formula (3).

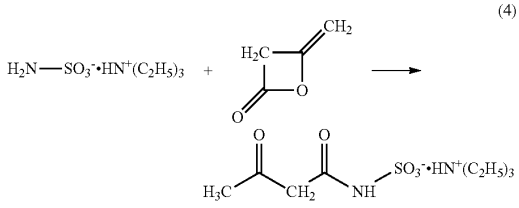
(3)



wherein M^+ is an appropriate ion. Preferably, M^+ is an alkali metal ion or $N^+R_1R_2R_3R_4$. R_1, R_2, R_3 and R_4, independently of one another, may be organic radicals or hydrogen, preferably H or C_1-C_8 alkyl, C_6-C_{10} cycloalkyl, aryl and/or aralkyl. In a preferred embodiment, R_1 is hydrogen, and R_2, R_3 and R_4 are alkyl, e.g., ethyl.

An exemplary reaction scheme for forming an acetoacetamide salt employs a trialkyl ammonium amidosulfamic acid salt and diketene as reactants and yields an acetoacetamide triethylammonium salt is shown in reaction (4), below.

(4)



In one embodiment, the reaction is conducted in the presence of a catalyst, which may vary widely. In some embodiments, the catalyst comprises one or more amines and/or phosphines. Preferably, the catalyst comprises triethylamine. In some cases trimethylamine serves as both a catalyst and a reactant.

In one embodiment, wherein the amidosulfamic acid salt formation reaction and the acetoacetamide salt formation reaction are conducted in separate reactors, a second reaction mixture comprises the amidosulfamic acid salt, the diketene, and the catalyst, e.g., triethylamine. Preferably, catalyst from the first reaction is carried through to the reaction mixture of the second reaction. The second reaction mixture is then subjected to conditions effective to form the acetoacetamide salt.

In one embodiment, the composition of the second reaction mixture may be similar to that of the first reaction mixture. In a preferred embodiment, the reaction product of the amidosulfamic acid salt formation reaction provides the amidosulfamic acid salt component of the second reaction mixture. In addition to the above-mentioned components, the second reaction mixture may further comprise reaction by-products from the first reaction or non-reacted starting materials.

In one embodiment, the amount of acetoacetylating agent, e.g., diketene, should be at least equimolar to the reactant amidosulfamic acid salt that is provided. In one embodiment, the process may utilize a diketene in excess, but preferably in an excess less than 30 mol %, e.g., less than 10 mol %. Greater excesses are also contemplated.

The amidosulfamic acid salt formation reaction and/or the acetoacetamide salt formation reaction may employ an organic solvent. Suitable inert organic solvents include any organic solvents that do not react in an undesired manner with the starting materials, cyclizing agent, final products and/or the catalysts in the reaction. The solvents preferably have the ability to dissolve, at least partially, amidosulfamic acid salts. Exemplary organic solvents include halogenated aliphatic hydrocarbons, preferably having up to 4 carbon atoms such as, for example, methylene chloride, chloroform, 1,2-dichlorethane, trichloroethylene, tetrachloroethylene, trichlorofluoroethylene; aliphatic ketones, preferably those having 3 to 6 carbon atoms such as, for example, acetone, methyl ethyl ketone; aliphatic ethers, preferably cyclic aliphatic ethers having 4 or 5 carbon atoms such as, for example, tetrahydrofuran, dioxane; lower aliphatic carboxylic acids, preferably those having 2 to 6 carbon atoms such as, for example, acetic acid, propionic acid; aliphatic nitriles, preferably acetonitrile; N-alkyl-substituted amides of carbonic acid and lower aliphatic carboxylic acids, preferably amides having up to 5 carbon atoms such as, for example, tetramethylurea, dimethylformamide, dimethylacetamide, N-methylpyrrolidone; aliphatic sulfoxides, preferably dimethyl sulfoxide, and aliphatic sulfones, preferably sulfolane.

Particularly preferred solvents include dichloromethane (methylene chloride), 1,2-dichloroethane, acetone, glacial acetic acid and dimethylformamide, with dichloromethane (methylene chloride) being particularly preferred. The solvents may be used either alone or in a mixture. In one embodiment, the solvent is a halogenated, aliphatic hydrocarbon solvent, preferably the solvent is dichloromethane. Chloroform and tetrachloromethane are also exemplary solvents.

In one embodiment, the acetoacetamide salt formation reaction is conducted a temperature ranging from −30° C. to 50° C., e.g., from 0° C. to 25° C. The reaction pressure may vary widely. In preferred embodiments, the reaction is carried out at atmospheric pressure, although other pressures are also contemplated. The reaction time may vary widely, preferably ranging from 0.5 hours to 12 hours, e.g., from 1 hour to 10 hours. In one embodiment, the reaction is carried out by introducing the amidosulfamic acid salt and metering in the diketene. In another embodiment, the reaction is carried out by introducing diketene and metering in the amidosulfamic acid salt. The reaction may be carried out by introducing the diketene and amidosulfamic acid and metering in the catalyst.

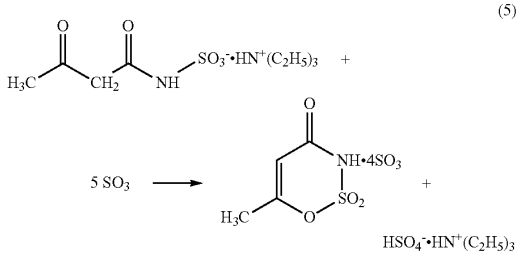
Once formed, each reaction product is optionally subjected to one or more purification steps. For example the solvent may be separated from the reaction product, e.g., via distillation, and the residue (mainly acetoacetamide-N-sul-

fonate) may be recrystallized from a suitable solvent such as, for example, acetone, methyl acetate or ethanol.

Cyclization and Hydrolyzation

The acetoacetamide salt is reacted with cyclizing agent, e.g., cyclizing agent in the cyclizing agent composition, in the presence of a solvent to form the cyclic (sulfur trioxide) adduct composition, which contains cyclic sulfur trioxide adduct and, in some cases, impurities. In some cases, a cooling step occurs before the cyclic sulfur trioxide adduct formation reaction. In one embodiment, the cyclization is achieved by using at least an equimolar amount of the cyclizing agent. The cyclizing agent may be dissolved in an inert inorganic or organic solvent. The cyclizing agent is generally used in a molar excess, e.g., up to a 20 fold excess, or up to a 10 fold excess, based on the total moles of acetoacetamide salt. An exemplary cyclization reaction using sulfur trioxide as the cyclizing agent is shown in reaction (5), below.

(5)



In one embodiment, the weight ratio of solvent to cyclizing agent in the cyclizing agent composition is at least 1:1, e.g., at least 2:1, or at least 5:1. In one embodiment, the weight ratio of solvent to cyclizing agent in the cyclizing agent composition ranges from 1:1 to 25:1, e.g., from 1:1 to 10:1, from 2:1 to 10:1, or from 5:1 to 10:1.

A cyclizing agent may be any compound that initiates the ring closure of the acetoacetamide salt. Although sulfur trioxide is a preferred cyclizing agent, the employment of other cyclizing agents is contemplated.

The cyclizing agent may be added to the reaction mixture either in the solid or the liquid form or by condensing in vapor. Suitable inert inorganic or organic solvents are those liquids which do not react in an undesired manner with sulfur trioxide or the starting materials or final products of the reaction. Preferred organic solvents include, but are not limited to, halogenated aliphatic hydrocarbons, preferably having up to four carbon atoms, such as, for example, methylene chloride (dichloromethane), chloroform, 1,2-dichloroethane, trichloroethylene, tetrachloroethylene, trichlorofluoroethylene; esters of carbonic acid with lower aliphatic alcohols, preferably with methanol or ethanol; nitroalkanes, preferably having up to four carbon atoms, in particular nitromethane; alkyl-substituted pyridines, preferably collidine; and aliphatic sulfones, preferably sulfolane. Particularly preferred solvents for the cyclization reaction include dichloromethane (methylene chloride), 1,2-dichloroethane, acetone, glacial acetic acid and dimethylformamide, with dichloromethane (methylene dichloride) being particularly preferred. Other solvents, e.g., other solvents mentioned herein, may also be suitable as solvents. The solvents may be used either alone or in a mixture. In one embodiment, the solvent is a halogenated, aliphatic hydrocarbon solvent, preferably the solvent is dichloromethane. The processes may employ these solvents alone or in mixtures thereof.

In some cases, the solvent in the cyclizing agent composition may be selected from 1) concentrated sulfuric acid, 2) liquid sulfur dioxide, or 3) an inert organic solvent.

In a preferred embodiment, the same solvent is used in both the acetoacetamide salt formation reaction and the cyclization reaction. As one benefit, the solution obtained in the acetoacetamide salt formation reaction, without isolation of the acetoacetamide salt formation reaction product, may be used immediately in the cyclization.

In one embodiment, the reaction temperature for the cyclization reaction ranges from −70° C. to 175° C., e.g., from −40° C. to 60° C. The pressure at which the reaction is conducted may vary widely. In one embodiment, the reaction is conducted at a pressure ranging from 0.01 MPa to 10 MPa, e.g., from 0.1 MPa to 5 MPa. Preferably, the reaction is conducted at atmospheric pressure.

The acetoacetamide salt may be introduced to the cyclization reactor and the cooled cyclizing agent composition, e.g., a solution of cyclizing agent optionally in solvent, may be metered into the reactor. In preferred embodiments, both reactants (acetoacetamide salt and cyclizing agent) are simultaneously fed into the reactor. In one embodiment, the cooled cyclizing agent composition is initially introduced into the reactor and the acetoacetamide salt is added. Preferably, at least part of the cyclizing agent composition is introduced into the reactor and, either continuously or in portions, acetoacetamide salt and (additional) cyclizing agent are then metered in, preferably while maintaining the temperature as described above.

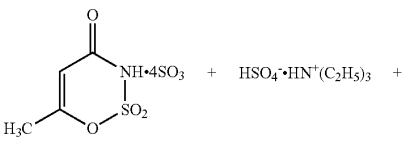
The acetoacetamide salt may be introduced to the reactor and the cyclizing agent composition may be metered into the reactor. In preferred embodiments, both reactants are simultaneously fed into the reactor. In one embodiment, the cyclizing agent composition is initially introduced into the reactor and the acetoacetamide salt is added. Preferably, at least part of the cyclizing agent composition is introduced into the reactor and, either continuously or in portions, acetoacetamide salt and (additional) cyclizing agent are then metered in, preferably while maintaining the temperature as described above.

The formation of the crude acesulfame potassium composition from the cyclic sulfur trioxide adduct composition, in some embodiments, comprises the steps of hydrolyzing the cyclic sulfur trioxide adduct to form an acesulfame-H composition; neutralizing the acesulfame-H in the acesulfame H composition to form a crude acesulfame potassium composition; and forming the acesulfame potassium composition from the crude acesulfame potassium composition.

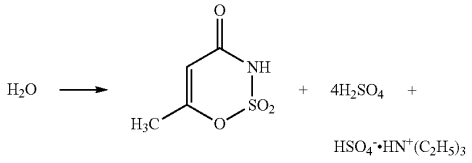
The cyclic sulfur trioxide adduct may be hydrolyzed via conventional means, e.g., using water. Thus, the forming step may comprise the steps of hydrolyzing the cyclic sulfur trioxide adduct to form an acesulfame-H composition. Acesulfame-H is referred to as sweetener acid.

An exemplary hydrolysis reaction scheme is shown in reaction (6), below.

(6)



-continued



The addition of the water leads to a phase separation. The majority of the sweetener acid, acesulfame-H (6-methyl-3,4-dihydro-1,2,3-oxathiazin-4-one 2,2-dioxide), which is formed via the hydrolysis, is present in the organic phase, e.g., at least 60 wt %, at least 70%, at least 80%, or at least 90%. The remainder of the sweetener acid is in the water phase and can be extracted and optionally added to the sweetener acid in the organic phase. In cases where dichloromethane is used as the reaction medium, water or ice may be added, e.g., in a molar excess, based on the sulfur trioxide, to the cyclic sulfur trioxide adduct/sulfur trioxide solution.

In some cases, the hydrolysis step comprises adding water to the cyclic sulfur trioxide adduct. In preferred embodiments, the weight ratio of water to acetoacetamide salt is greater than 1.3:1, e.g., greater than 1.5:1, greater than 1.7:1, greater than 2:1 or greater than 2.2:1. Employment of these ratios may lead to decreases in acetoacetamide-N-sulfonic acid and/or acetoacetamide formation in the neutralized crude acesulfame potassium composition, e.g., the crude acesulfame potassium composition may comprise acetoacetamide-N-sulfonic acid in the amounts discussed herein.

It was surprisingly discovered that the temperature at which the water is initially fed to the hydrolysis reaction may have beneficial effects on impurity production, e.g., organic production or 5-chloro-acesulfame potassium production as well as reaction parameters, e.g., temperature. At lower temperatures, e.g., lower than approximately −35° C. or lower than −22° C., ice tends to build up in the reaction mixture. As this ice melted, it led to the onset of additional reaction, which caused the temperature to rise quickly. This rise in temperature surprisingly led to a product that contained much higher levels of impurities. In some cases, the hydrolyzing comprises adding hydrolysis water to the cyclic sulfur trioxide adduct to form a hydrolysis reaction mixture and reacting the mixture to from the acesulfame-H composition. In some embodiments, the temperature of the hydrolysis reaction mixture or the temperature at which the hydrolysis water is fed to the reactor is maintained at a temperature greater than −35° C., e.g., greater than −30° C., greater than −25° C., greater than −24° C., greater than −23° C., greater than −22° C., greater than −21.5° C., greater than −21° C., or greater than greater than −20° C. In terms of ranges, the temperature of the hydrolysis reaction mixture or the temperature at which the hydrolysis water is fed to the reactor optionally is maintained at a temperature ranging from −35° C. to 0° C., e.g., from −30° C. to −5° C., from −20° C. to −5° C., from −30° C. to −20° C., from −25° C. to −21° C., or −25° C. to −21.5° C.

After the addition of water, the reaction solvent, e.g., dichloromethane, may be removed by distillation, or the acesulfame-H that remains in the organic phase may be extracted with a more suitable solvent. Suitable solvents are those which are sufficiently stable towards sulfuric acid and which have a satisfactory dissolving capacity. Other suitable solvents include esters of carbonic acid such as, for example dimethyl carbonate, diethyl carbonate and ethylene carbonate, or esters of organic monocarboxylic acids such as, for example, isopropyl formate and isobutyl formate, ethyl acetate, isopropyl acetate, butyl acetate, isobutyl acetate and neopentyl acetate, or esters of dicarboxylic acids or amides which are immiscible with water, such as, for example, tetrabutylurea, are suitable. Isopropyl acetate and isobutyl acetate are particularly preferred.

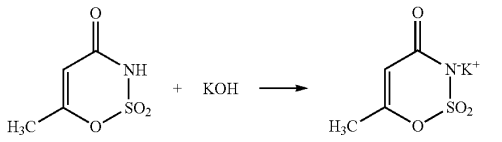
It has now been discovered that a transition phase may form in addition to the organic sweetener acid-dichloromethane phase and aqueous phase. The transition phase may contain high amounts of impurities, e.g., acetoacetamide. The transition phase may contain higher amounts of such impurities than the organic phase. Beneficially, this transition phase may be removed from the organic sweetener acid-dichloromethane phase thus significantly reducing impurity content thereof. The process may utilize the step of phase separating the acesulfame-H composition. The phase separation may form the sweetener acid-dichloromethane phase, the aqueous phase, and the aforementioned transition phase comprising at least 2 wt % impurities, e.g., at least 5 wt %, at least 10 wt %, at least 20 wt %, at least 30 wt %, or at least 50 wt %. The process may comprise separating from the acesulfame-H composition the transition phase to form a purified acesulfame-H composition. The finished acesulfame potassium composition may then be formed from the purified acesulfame-H composition, e.g., via neutralization and treatment.

The combined organic phases are dried with, for example, Na_2SO_4, and are evaporated. Any sulfuric acid which has been carried over in the extraction may be removed by appropriate addition of aqueous alkali to the organic phase. For this purpose, dilute aqueous alkali may be added to the organic phase until the pH reached in the aqueous phase corresponds to that of pure 6-methyl-3,4-dihydro1,2,3-oxathiazin-4-one 2,2-dioxide at the same concentration in the same two-phase system of extracting agent and water.

Neutralization

The neutralization of the acesulfame-H yields a non-toxic salt of acesulfame-H, e.g., acesulfame potassium. In one embodiment, neutralization is carried out by reacting the acesulfame-H with an appropriate base, e.g., potassium hydroxide, in particular a membrane-produced potassium hydroxide. Other suitable bases include, for example, KOH, $KHCO_3$, K_2CO_3, and potassium alcoholates. An exemplary reaction scheme using potassium hydroxide as a neutralizing agent is shown in reaction (7), below.

(7)



In some cases, the neutralization is conducted or maintained at a low pH levels, which may advantageously further result in a reduction or elimination of the formation of impurities, e.g., acetoacetamide salts. In this context, "conducted" means that the neutralization step begins at a low pH level, and "maintained" means that steps are taken to ensure that the pH stays within a low pH range throughout the entire neutralization step. In one embodiment, the neutralization step is conducted or maintained at a pH below 10.0, e.g., below 9.5, below 9.0, below 8.5, below 8.0, below 7.5,

below 7.0, or below 6.5. In terms of ranges, the neutralization step is preferably conducted or maintained at a pH between 6.0 and 10.0, e.g., between 6.5 and 9.5, between 7.0 and 9.0, or between 7.5 and 8.5.

In some cases, the pH in the neutralizing step may be maintained within the desired range by managing the components of the neutralization reaction mixture, which comprises acesulfame-H and neutralizing agent (and also solvent). For example, the composition of the neutralization reaction mixture may include from 1 wt % to 95 wt % neutralizing agent, e.g., from 10 wt % to 85 wt % or from 25 wt % to 75 wt %, and from 1 wt % to 95 wt % acesulfame-H, e.g., from 10 wt % to 85 wt % or from 25 wt % to 75 wt %. These concentration ranges are based on the mixture of neutralization agent and acesulfame-H (not including solvent).

In one embodiment, the acesulfame-H may be neutralized and extracted directly from the purified organic extraction phase using an aqueous potassium base. The acesulfame potassium then precipitates out, where appropriate after evaporation of the solution, in the crystalline form, and it can also be recrystallized for purification.

In one embodiment, the process is not a small-scale batch process or a laboratory-scale process. For example, the inventive process for producing a finished acesulfame potassium composition may yield at least 50 grams of finished acesulfame potassium composition per batch, e.g., at least 100 grams per batch, at least 500 grams per batch, at least 1 kilogram per batch, or at least 10 kilograms per batch. In terms of rates, the inventive process may yield at least 50 grams of finished acesulfame potassium composition per hour, e.g., at least 100 grams per hour, at least 500 grams per hour, at least 1 kilogram per hour, or at least 10 kilograms per hour.

FIG. **1** shows an exemplary acesulfame potassium process **100** in accordance with the process described herein. Process **100** comprises amidosulfamic acid salt formation reactor **102** and acetoacetamide salt formation reactor **104**. Although FIG. **1** shows separate reactors for the two intermediate formation reactions, other configurations, e.g., a one reactor process, are within the contemplation of the present process. Sulfamic acid is fed to amidosulfamic acid salt formation reactor **102** via sulfamic acid feed line **106**. Amine(s), preferably triethylamine, are fed to amidosulfamic acid salt formation reactor **102** via amine feed line **108**. In addition to sulfamic acid and amine(s), acetic acid is also fed to amidosulfamic acid salt formation reactor **102** (via feed line **110**). The resultant reaction mixture in amidosulfamic acid salt formation reactor **102** is as discussed above. In amidosulfamic acid salt formation reactor **102**, the sulfamic acid and the amine (in the presence of the acetic acid) are reacted to yield a crude amidosulfamic acid salt composition, which exits reactor **102** via line **112**. Although not shown, a reaction solvent, e.g., dichloromethane may also be present in the amidosulfamic acid salt formation reactor **102**.

The crude amidosulfamic acid salt composition in line **112** is directed to acetoacetamide salt formation reactor **104**. Diketene is fed to acetoacetamide salt formation reactor **104** via feed line **114**. In acetoacetamide salt formation reactor **104**, the amidosulfamic acid salt and the diketene are reacted to yield a crude acetoacetamide salt composition, which exits reactor **104** via line **118**. Although not shown, dichloromethane may also be present in the acetoacetamide salt formation reactor **104**.

Cyclizing agent (sulfur dioxide) and solvent (dichloromethane) are fed to vessel **119** via feed lines **121** and **123**. Vessel **119** is preferably a vessel wherein a cyclizing agent composition comprising these two components is formed. The cyclizing agent composition comprising both cyclizing agent and solvent exits vessel **119** via line **125**.

The crude acetoacetamide salt composition is directed to cyclization reactor **120** via line **118**. The cyclizing agent composition is also directed to cyclization reactor **120** (via line **125**). Line **125** is preferably made of a material and in such a size and shape to facilitate the residence times discussed herein. In cyclization reactor **120**, the acetoacetamide salt in the crude acetoacetamide salt composition in line **118** is cyclized and a cyclic sulfur trioxide adduct stream exits via line **124**.

The cyclic sulfur trioxide adduct in line **124**, is directed to hydrolysis reactor **126**. Water is fed to hydrolysis reactor **126** via water feed **128**. In hydrolysis reactor **126**, the cyclic sulfur trioxide adduct is hydrolyzed to yield a crude acesulfame-H composition, which exits hydrolysis reactor **126** via line **130** and is directed to phase separation unit **132**. Phase separation unit **132** separates the contents of line **130** into organic phase **134** and aqueous phase **136**. Organic phase **134** comprises a major amount of the acesulfame-H in line **130** as well as solvent, e.g., methylene chloride. Aqueous phase **136** exits via line **137** and comprises triethylammonium sulfate, and optionally sulfuric acid and minor amounts of acesulfame-H. The aqueous phase may be further purified to separate and/or recover the acesulfame-H and/or the triethylammonium sulfate. The recovered acesulfame-H may be combined with the acesulfame from the organic phase (not shown).

Organic phase **134** exits phase separation unit **132** and is directed to extraction column **138** (via line **140**). Water is fed to extraction column **138** via water feed **142**. The water extracts residual sulfates from the contents of line **140** and a purified acesulfame-H composition exits extraction column **138** via line **144**. The extracted sulfates exit extraction column **138** via line **145**.

The purified acesulfame-H composition in line **144** is directed to neutralization unit **146**. Potassium hydroxide is also fed to neutralization unit **146** (via line **148**). The addition of the potassium hydroxide (via line **148**) to neutralization unit **146** may be adjusted to achieve and/or maintain the desired pH levels during the neutralization, as discussed herein. The potassium hydroxide neutralizes the acesulfame-H in the purified acesulfame-H composition to yield a product comprising acesulfame potassium, dichloromethane, water, potassium hydroxide, and impurities, e.g., acetoacetamide, which exits neutralization unit **146** via line **150**. This product may be considered a crude acesulfame potassium composition.

The crude acesulfame potassium product stream in line **150** may be directed to treatment zone **156** to recover finished acesulfame potassium, which is shown exiting via stream **152**. In addition to the finished acesulfame potassium, dichloromethane and potassium hydroxide may be separated from the crude acesulfame potassium product stream, as shown by stream **154**. The contents of stream **154** may be recovered and/or recycled to the process. Treatment zone **156** may comprise one or more of the treatment steps described herein, e.g., stripping, evaporation, crystallization, and filtration.

The product in line **150** is directed to phase separation unit **160**. Phase separation unit **160** separates the product in line **150** into organic phase **162** and an aqueous phase **164**. Aqueous phase **164** comprises a major amount of the acesulfame potassium in line **150** as well as some impurities. Organic phase **162** comprises potassium hydroxide, dichlo-

romethane, and water and may be further treated to recover these components. Aqueous phase **164** (without any further treatment) may be considered a crude acesulfame potassium composition. Aqueous phase **164** may be optionally treated to form a finished acesulfame potassium composition.

Aqueous phase **164** is directed to treatment unit **156** via line **166**. In treatment unit **156**, aqueous phase **164** is treated to obtain finished acesulfame potassium composition (product that may be sold), which is shown exiting via stream **152**. In addition to the finished acesulfame potassium composition, dichloromethane and potassium hydroxide may be separated. These components exit treatment unit **156** via line **154**. The contents of stream **154** may be recovered and/or recycled to the process.

FIG. **2** shows and exemplary treatment zone. Crude acesulfame potassium product stream **250** is fed to treatment zone **256**. In particular, crude acesulfame potassium product stream **250** is fed to stripper **252** to strip solvent therefrom. Solvent in stream **254** exits stripper **252** and is directed to further processing, e.g. re-use or recycling. Stripped acesulfame potassium stream **257** comprises low amounts of solvent and exits stripper **252** and is directed to evaporator **258**. It has been found that the removal of solvent prior to evaporation unexpectedly provides the benefit of improved evaporation operations.

Evaporator **258** removes water from stripped acesulfame potassium stream in line **257** to form water stream **260** and intermediate acesulfame potassium composition stream **262**. Evaporator **258** is preferably a falling film evaporator. Intermediate acesulfame potassium composition stream **262** is directed to crystallizer **264**, which yields crystal-containing stream **266** and recycle stream **268**. Crystal-containing stream **266** is then directed to filtration unit **270**, which filters impurities to yield finished acesulfame potassium composition stream **272** and impurity stream **274**. The treatment units may beneficially be operated at the separation parameters discussed herein.

The invention relates also to the following aspects:

Aspect 1: A process for producing a finished acesulfame potassium composition, the process comprising the steps of:

(a) providing a crude acesulfame potassium composition comprising acesulfame potassium, acetoacetamide and water;

(b) concentrating the crude acesulfame potassium composition to form a water stream and an intermediate acesulfame potassium composition comprising acesulfame potassium and less than 33 wppm acetoacetamide; and

(c) separating the intermediate acesulfame potassium composition to form the finished acesulfame potassium composition comprising acesulfame potassium and less than 33 wppm acetoacetamide;

wherein the concentrating step (b) is conducted at a temperature below 90° C., and wherein the separating step (c) is conducted at a temperature at or below 35° C.

Aspect 2: The process of aspect 1, wherein the providing step (a) comprises:

reacting sulfamic acid and an amine to form an amidosulfamic acid salt;

reacting the amidosulfamic acid salt and acetoacetylating agent to form an acetoacetamide salt;

reacting the acetoacetamide salt with cyclizing agent in the cyclizing agent composition to form the cyclic sulfur trioxide adduct;

hydrolyzing the cyclic sulfur trioxide adduct to form an acesulfame-H composition comprising acesulfame-H; and

neutralizing the acesulfame-H in the acesulfame-H composition to form the crude acesulfame potassium composition.

Aspect 3: The process of any one of the preceding aspects, wherein the intermediate acesulfame potassium composition comprises less than 33 wppm acetoacetamide-N-sulfonic acid.

Aspect 4: The process of any one of the preceding aspects, wherein the concentrating comprises: evaporating the crude acesulfame potassium composition to form the water stream and the intermediate acesulfame potassium composition comprising acesulfame potassium and less than 75 wt % water.

Aspect 5: The process of any one of the preceding aspects, wherein evaporation residence time is less than 180 minutes.

Aspect 6: The process of any one of the preceding aspects, wherein the intermediate acesulfame potassium composition comprises less than 33 wppm acetoacetamide-N-sulfonic acid.

Aspect 7: The process of any one of the preceding aspects, wherein the separating comprises:

crystallizing the intermediate acesulfame potassium composition to form acesulfame potassium crystals; and

filtering the acesulfame potassium crystals to form the finished acesulfame potassium composition.

Aspect 8: The process of any one of the preceding aspects, wherein the filtering is conducted at a temperature at or below 35° C.

Aspect 9: The process of any one of the preceding aspects, wherein the crystallizing is conducted at a temperature at or below 35° C.

Aspect 10: The process of any one of the preceding aspects, wherein the crystallizing is conducted at a pH below 10.

Aspect 11: The process of any one of the preceding aspects, wherein the evaporating is conducted at a temperature below 85° C. and the intermediate acesulfame potassium composition comprises from 1 wppb to 33 wppm acetoacetamide and the finished acesulfame potassium composition comprises less than 33 wppm acetoacetamide.

Aspect 12: The process of any one of the preceding aspects, wherein the intermediate acesulfame potassium composition further comprises less than 33 wppm acetoacetamide-N-sulfonic acid.

Aspect 13: The process of any one of the preceding aspects, wherein the evaporating is conducted at a temperature below 60° C. and the evaporator residence time is less than 50 minutes and the intermediate acesulfame potassium composition comprises from 10 wppb to 25 wppm acetoacetamide and the finished acesulfame potassium composition comprises from 10 wppb to 15 wppm acetoacetamide.

Aspect 14: The process of any one of the preceding aspects, wherein the intermediate acesulfame potassium composition comprises less than 30 wppm acetoacetamide-N-sulfonic acid.

Aspect 15: The process of any one of the preceding aspects, wherein the evaporating is conducted at a temperature ranging from 20° C. to 55° C.; the evaporator residence time ranges from 1 minute to 300 minutes; the separating is conducted at a temperature ranging from −10° C. to 15° C.; the separating operation residence time ranges from 1 to 180 minutes; the crude acesulfame potassium composition comprises from 500 wppm to 2375 wppm acetoacetamide; the intermediate acesulfame potassium composition comprises 10 wppb to 20 wppm acetoacetamide and 10 wppb to 20 wppm acetoacetamide-N-sulfonic acid; and the finished acesulfame potassium composition comprises from 10 wppb

to 10 wppm acetoacetamide, and from 1 wppb to 20 wppm acetoacetamide-N-sulfonic acid.

Aspect 16: The process of any one of the preceding aspects, wherein:

(i) the evaporating is conducted at a temperature below 46° C.,

(ii) the evaporator residence time is less than 30 minutes,

(iii) crystallizing is conducted at a temperature below 35° C.,

(iv) the intermediate acesulfame potassium composition comprises from 10 wppb to 12 wppm acetoacetamide, and

(v) the finished acesulfame potassium composition comprises from 10 wppb to 7 wppm acetoacetamide.

Aspect 17: The process of any one of the preceding aspects, wherein the intermediate acesulfame potassium composition comprises less than 20 wppm acetoacetamide-N-sulfonic acid.

Aspect 18: The process of any one of the preceding aspects, wherein the crude acesulfame composition further comprises solvent and wherein the process further comprises removing solvent from the crude acesulfame potassium composition prior to the evaporation.

Aspect 19: The process of any one of the preceding aspects, wherein the weight percentage of acetoacetamide in the finished acesulfame potassium composition is less than the weight percentage of acetoacetamide in the crude acesulfame potassium composition.

Aspect 20: The process of any one of the preceding aspects, further comprising:

separating from the acesulfame-H composition a transition phase comprising at least 2 wt % acetoacetamide to form a purified acesulfame-H composition.

Aspect 21: The process of any one of the preceding aspects, wherein the neutralizing comprises neutralizing the acesulfame-H in the purified acesulfame-H composition to form the crude acesulfame potassium composition comprising acesulfame potassium and acetoacetamide.

Aspect 22: The process of any one of the preceding aspects, wherein the concentrating comprises evaporating the crude acesulfame potassium composition to form a water stream and an intermediate acesulfame potassium composition comprising acesulfame potassium and less than 50 wt % water, and the separating comprises crystallizing the intermediate acesulfame potassium composition to form a crystal-containing stream comprising acesulfame potassium crystals, and filtering the crystal-containing stream to form the finished acesulfame potassium composition.

Aspect 23: The process of any one of the preceding aspects, wherein the filtering comprises at least two filtration operations.

Aspect 24: A finished acesulfame potassium composition produced or producible by, or obtainable or obtained from the process of any one of aspects 1 to 22.

Aspect 25: A process for producing a finished acesulfame potassium composition, the process comprising the steps of:

(a) reacting sulfamic acid and triethylamine to form an amidosulfamic acid salt;

(b) reacting the amidosulfamic acid salt and diketene to form acetoacetamide salt;

(c) contacting dichloromethane and a sulfur trioxide to form a cyclizing agent composition;

(d) reacting the acetoacetamide salt with sulfur trioxide in the cyclizing agent composition to form a cyclic sulfur trioxide adduct;

(e) hydrolyzing the cyclic sulfur trioxide adduct to form an acesulfame-H composition;

(f) neutralizing the acesulfame-H to form the crude acesulfame potassium composition comprising acesulfame potassium and acetoacetamide,

(g) evaporating the crude acesulfame potassium composition to form a water stream and an intermediate acesulfame potassium composition comprising acesulfame potassium and less than 75 wt % water;

(h) crystallizing the intermediate acesulfame potassium composition to form acesulfame potassium crystals; and

(i) filtering the acesulfame potassium crystals to form the finished acesulfame potassium composition comprising acesulfame potassium and less than 10 wppm acetoacetamide, wherein the evaporating is conducted at a temperature below 50° C. and wherein evaporator residence time is less than 30 minutes.

Aspect 26: The process of aspect 25, wherein filtering is conducted at a temperature below 35° C. and crystallizing is conducted at a temperature below 35° C.

Aspect 27: A finished acesulfame potassium composition produced or producible by, or obtainable or obtained from the process of aspects 25 or 25.

Aspect 28: An acesulfame potassium composition comprising acesulfame potassium and less than 33 wppm, preferably less than 10 wppm acetoacetamide.

Aspect 29: The acesulfame potassium composition of aspect 27 or 28, further comprising less than 33 wppm, preferably less than 10 wppm acetoacetamide-N-sulfonic acid.

Aspect 30: The acesulfame potassium composition of aspect 27-29, further comprising 0.001 wppm to 5 wppm organic impurities and/or 0.001 wppm to 5 wppm of at least one heavy metal.

Aspect 31: The acesulfame potassium composition of aspect 27-30, wherein the at least one heavy metal is selected from the group consisting of mercury, lead and mixtures thereof.

Aspect 31: The acesulfame potassium composition of aspect 27-31, wherein the mercury is present in an amount of 1 wppb to 20 wppm.

Aspect 31: The acesulfame potassium composition of aspect 27-32, wherein the lead is present in an amount of 1 wppb to 25 wppm.

EXAMPLES

The following examples are included to illustrate the process and compositions and are not meant to limit the scope of the application.

Crude Acesulfame Potassium Composition Formation

100 mmol of 99.5% pure sulfamic acid was suspended in 50 mL dichloromethane in a flask with reflux. Under continuous agitation, 105 mmol of trimethylamine was added within approximately 3 minutes. During this time, temperature increased due to acid/base exothermal reaction up to about 42° C. (the boiling point of dichloromethane). This first reaction mixture was stirred for approximately 15 additional minutes, until no solid sedimentation was seen in the flask. Then, 10 mmol of acetic acid was added to the first reaction mixture and was stirred for approximately 15 additional minutes. At this point, within 7 minutes of the addition of the acetic acid, 110 mmol of diketene was added dropwise to form a second reaction mixture. After the addition of all of the diketene was added to the second reaction mixture and approximately 15 minutes of reaction time, this second reaction mixture was cooled. The resultant cooled second reaction mixture contained approximately

30% acetoacetamide N-sulfonate triethylammonium salt. Additional batches of cooled second reaction mixture were prepared as necessary.

In a separate vessel, a sulfur trioxide/dichloromethane composition comprising approximately 15 wt % sulfur trioxide and approximately 85 wt % dichloromethane was prepared by contacting the two components with one another.

A second flask (a 4 necked round bottom flask equipped with mechanical stirrer, thermometer, and feed vessels) was placed into a cooling bath containing a mixture of isopropanol and dry ice. Approximately 200 g of the acetoacetamide-N-sulfonate triethylammonium salt solution and approximately 577 g of the sulfur trioxide/dichloromethane compositions were measured. Approximately 15 wt % of the total sulfur trioxide/dichloromethane composition (approximately 87 g) was initially fed to the reaction flask under continuous agitation by mechanical stirrer. When the temperature of the flask contents reached −35° C. (due to the cooling bath), the remainder of the sulfur trioxide/dichloromethane composition and all of the acetoacetamide-N-sulfonate triethylammonium salt solution were fed into the second flask. The time period that the solvent contacts the cyclizing agent before formation of the cyclic sulfur trioxide adduct, e.g., before the acetoacetamide-N-sulfonate triethylammonium salt solution was fed to the second flask, was less than an hour. The feed rate was controlled in such a way that the temperature of the second flask contents remained between −25° and −35° C. during the feeding/cyclization reaction. After the reactants were fed, the reaction was allowed to proceed for approximately one additional minute. The cooling bath was then removed.

After approximately one minute, the temperature of the flask contents reached approximately −22° C. At this time, hydrolysis was initiated by feeding deionized water to the flask. Water was fed over 10 minutes. The hydrolysis reaction was exothermic. Water was added slowly so as to maintain temperature between −20° C. and −5° C. After addition of water, reaction mixture was allowed to reach room temperature.

The hydrolyzed product was phase separated via a separating funnel. A heavier organic sweetener acid-dichloromethane phase (acesulfame-H composition) was separated out, and the remaining aqueous phase was discarded.

The acesulfame-H in the acesulfame-H composition was neutralized with a 10% potassium hydroxide solution. Neutralization was carried out at 25° C.±1° C. Potassium hydroxide addition was completed within 20 minutes. After completion of the neutralization step, an additional phase separation was performed using a separating funnel to yield an aqueous phase containing acesulfame potassium (and some impurities) and an organic phase. The aqueous phase was considered a crude acesulfame potassium composition. This crude acesulfame potassium composition was split into two portions and treated as discussed below. The remaining dichloromethane in the organic phase was discarded.

Example 1: Evaporation/Crystallization

A first portion of the crude acesulfame potassium composition was evaporated in a rotary evaporator at 45° C. and under reduced pressure for approximately 20 minutes. As a result, approximately 50% of the water was evaporated from the crude acesulfame potassium composition. After the water was removed an intermediate acesulfame potassium composition remained. The intermediate acesulfame potassium composition was then separated, e.g., cooled to 5° C. in a refrigerator.

The cooling resulted in the precipitation of crude crystals containing mostly acesulfame potassium. These crude crystals were considered a finished acesulfame potassium composition. The crude crystals were separated from the liquid and analyzed for yield and impurities, e.g., acetoacetamide. Testing for acetoacetamide (AAA) content was performed using the HPLC equipment and techniques discussed herein. In particular, the HPLC analysis was performed using an LC Systems HPLC unit from Shimadzu having a CBM-20 Shimadzu controller and being equipped with an IonPac NS1 ((5 μm) 150×4 mm) analytical column and an IonPac NG1 guard column (35×4.0 mm). A Shimadzu SPD-M20A photodiode array detector was used for detection (at 270 nm and 280 nm wavelength). Analysis was performed at 23° C. column temperature. As a first eluent solution, an aqueous mixture of tetra butyl ammonium hydrogen sulfate (3.4 g/L), acetonitrile (300 mL/L), and potassium hydroxide (0.89 g/L) was employed; as a second eluent solution, an aqueous mixture of tetra butyl ammonium hydrogen sulfate (3.4 g/L) and potassium hydroxide (0.89 g/L) was employed. Elution was conducted in gradient mode according to the following second eluent flow profile:

0 to 3 minutes: constant 80% (v/v)

3 to 6 minutes: linear reduction to 50% (v/v)

6 to 15 minutes: constant at 50% (v/v)

15 to 18 minutes: linear reduction to 0%

18 to 22 minutes: constant at 0%

22 to 24 minutes: linear increase to 80% (v/v)

24 to 35 minutes constant at 80% (v/v).

Overall flow rate of eluent was approximately 1.2 mL/min. The data collection and calculations were performed using Lab Solution software from Shimadzu.

Comparative Example A: Evaporation/Crystallization

A second portion of the crude acesulfame potassium composition was evaporated in a rotary evaporator at 90° C. and under reduced pressure for approximately 180 minutes. As a result, approximately 50% of the water was evaporated from the crude acesulfame potassium composition. After the water was removed an intermediate acesulfame potassium composition remained. The intermediate acesulfame potassium composition was then separated, e.g., cooled to 5° C. in a refrigerator.

The cooling resulted in the precipitation of crude crystals containing mostly acesulfame potassium. These crude crystals were considered a finished acesulfame potassium composition. The crude crystals were separated from the liquid and analyzed for yield and impurities, e.g., acetoacetamide. Testing for acetoacetamide content was performed using the HPLC equipment and techniques discussed above. The results for Example 1 and Comparative Example A are shown in Table 1.

TABLE 1

ACK Impurity Testing Immediately After First Evaporation/Crystallization

Example 1	Evap. Temp., ° C.	Evap. Time, min.	Crystallization Temp., ° C.	AAA, wppm, in the finished ACK composition
Comp. Ex. A	45° C.	20 min.	5° C.	5 wppm
	90° C.	180 min.	5° C.	37 wppm

The temperature of the concentrating of the crude acesulfame potassium composition and the separating operation of the intermediate acesulfame potassium composition have an influence on acetoacetamide formation. Without being bound by theory, it is believed that acetoacetamide impurities form as a result of thermal stress during high temperature concentration of the crude acesulfame potassium composition, e.g., during the initial evaporation step. As shown, if the temperature of the concentrating operation is kept below 90° C., then additional post-crystallization separation is not needed to keep acetoacetamide content below a suitable level, e.g., less than 10 wppm.

Examples 2 and Comparative Examples B-D

The degradation effects of temperature and pH on acesulfame potassium compositions during treatment were also explored. Acesulfame potassium was diluted in sufficient water to form a 16% aqueous solution. The aqueous solution was divided into multiple portions (Example 2 and Comparative Examples B-D). These portions of aqueous solution (except for Example 2) were heated under reflux condensing to simulate the concentrating operation. In Comparative Example D, prior to heating, a small amount of 10% potassium hydroxide was added to the respective portion of aqueous solution, which brought the pH of the resultant solution to approximately 9.8. For each portion, crystals (intermediate acesulfame potassium compositions) were removed from the remaining liquid (after heating) and analyzed for and impurities formed via degradation, e.g., acetoacetamide-N-sulfonic acid (AAA-NSH). Testing for acetoacetamide-N-sulfonic acid content was performed using the HPLC equipment and techniques discussed above. The treatment conditions and resultant impurity contents are shown in Table 2.

TABLE 2

Effects of Temperature and pH on Degradation Products

	Temp., ° C.	Heating Time, min.	pH	AAA-NSH, wppm, in the int. acesulfame potassium composition
Example 2	25° C.	—	6.5	19 wppm
Comp. Ex. B	95° C.	180 min.	6.5	33 wppm
Comp. Ex. C	95° C.	360 min.	6.5	41 wppm
Comp. Ex. C	95° C.	180 min.	9.8	89 wppm

As shown in Table 2, the use of high temperature, heating time (which simulates residence time), and pH during conventional concentrating operation conditions results in degradation of acesulfame potassium (or acesulfame-H), which leads to the formation of additional impurities, e.g., acetoacetamide-N-sulfonic acid. By utilizing the concentrating operation parameters discussed herein, impurities formed via degradation are reduced or eliminated, which leads to higher purity finished acesulfame potassium compositions, i.e., finished acesulfame potassium compositions with low acetoacetamide-N-sulfonic acid content.

While the invention has been described in detail, modifications within the spirit and scope of the invention will be readily apparent to those of skill in the art. In view of the foregoing discussion, relevant knowledge in the art and references discussed above in connection with the Background and Detailed Description, the disclosures of which are all incorporated herein by reference. In addition, it should be understood that aspects of the invention and portions of various embodiments and various features recited above and/or in the appended claims may be combined or interchanged either in whole or in part. In the foregoing descriptions of the various embodiments, those embodiments which refer to another embodiment may be appropriately combined with other embodiments as will be appreciated by one of skill in the art. Furthermore, those of ordinary skill in the art will appreciate that the foregoing description is by way of example only, and is not intended to limit the invention.

We claim:

1. A process for producing a finished acesulfame potassium composition, the process comprising the steps of:

(a) providing a crude acesulfame potassium composition comprising acesulfame potassium, acetoacetamide and water;

(b) concentrating the crude acesulfame potassium composition to form a water stream and an intermediate acesulfame potassium composition; and

(c) separating the intermediate acesulfame potassium composition to form the finished acesulfame potassium composition comprising acesulfame potassium and less than 33 wppm acetoacetamide;

wherein the concentrating step (b) is conducted at a temperature below 85° C., and wherein the separating step (c) is conducted at a temperature at or below 20° C.

2. The process of claim **1**, wherein the providing step (a) comprises:

reacting sulfamic acid and an amine to form an amidosulfamic acid salt;

reacting the amidosulfamic acid salt and acetoacetylating agent to form an acetoacetamide salt;

reacting the acetoacetamide salt with cyclizing agent in the cyclizing agent composition to form the cyclic sulfur trioxide adduct;

hydrolyzing the cyclic sulfur trioxide adduct to form an acesulfame-H composition comprising acesulfame-H; and

neutralizing the acesulfame-H in the acesulfame-H composition to form the crude acesulfame potassium composition.

3. The process of claim **1**, wherein the intermediate acesulfame potassium composition comprises less than 33 wppm acetoacetamide and less than 33 wppm acetoacetamide-N-sulfonic acid.

4. The process of claim **1**, wherein the concentrating comprises: evaporating the crude acesulfame potassium composition to form the water stream and the intermediate acesulfame potassium composition comprising acesulfame potassium and less than 75 wt % water.

5. The process of claim **4**, wherein evaporation residence time is less than 180 minutes.

6. The process of claim 5, wherein the intermediate acesulfame potassium composition comprises less than 33 wppm acetoacetamide-N-sulfonic acid.

7. The process of claim 4, wherein the separating comprises:

crystallizing the intermediate acesulfame potassium composition to form acesulfame potassium crystals; and

filtering the acesulfame potassium crystals to form the finished acesulfame potassium composition.

8. The process of claim 7, wherein the filtering is conducted at a temperature at or below 20° C.

9. The process of claim 7, wherein the crystallizing is conducted at a temperature at or below 20° C.

10. The process of claim 7, wherein the crystallizing is conducted at a pH below 10.

11. The process of claim 4, wherein the evaporating is conducted at a temperature below 85° C. and the finished acesulfame potassium composition comprises less than 33 wppm acetoacetamide.

12. The process of claim 11, wherein the intermediate acesulfame potassium composition further comprises from 1 wppb to 33 wppm acetoacetamide and less than 33 wppm acetoacetamide-N-sulfonic acid.

13. The process of claim 4, wherein the evaporating is conducted at a temperature below 60° C. and the evaporator residence time is less than 50 minutes and the finished acesulfame potassium composition comprises from 10 wppb to 15 wppm acetoacetamide.

14. The process of claim 13, wherein the intermediate acesulfame potassium composition comprises from 10 wppb to 25 wppm acetoacetamide and less than 30 wppm acetoacetamide-N-sulfonic acid.

15. The process of claim 4, wherein the evaporating is conducted at a temperature ranging from 20° C. to 55° C., the evaporator residence time ranges from 1 minute to 300 minutes, the separating is conducted at a temperature ranging from −10° C. to 15° C., the separating operation residence time ranges from 1 to 180 minutes, the finished acesulfame potassium composition comprises from 10 wppb to 10 wppm acetoacetamide, and from 1 wppb to 20 wppm acetoacetamide-N-sulfonic acid.

16. The process of claim 7, wherein:

(i) the evaporating is conducted at a temperature below 46° C.,

(ii) the evaporator residence time is less than 30 minutes,

(iii) crystallizing is conducted at a temperature below 20° C. 35° C.,

and

(iv) the finished acesulfame potassium composition comprises from 10 wppb to 7 wppm acetoacetamide.

17. The process of claim 16, wherein the intermediate acesulfame potassium composition comprises from 10 wppb to 12 wppm acetoacetamide and less than 20 wppm acetoacetamide-N-sulfonic acid.

18. The process of claim 4, wherein the crude acesulfame composition further comprises solvent and wherein the process further comprises removing solvent from the crude acesulfame potassium composition prior to the evaporation.

19. The process of claim 4, wherein the weight percentage of acetoacetamide in the finished acesulfame potassium composition is less than the weight percentage of acetoacetamide in the crude acesulfame potassium composition.

20. The process of claim 2, further comprising:

separating from the acesulfame-H composition a transition phase comprising at least 2 wt % acetoacetamide to form a purified acesulfame-H composition.

21. The process of claim 20, wherein the neutralizing comprises neutralizing the acesulfame-H in the purified acesulfame-H composition to form the crude acesulfame potassium composition.

22. The process of claim 1, wherein the concentrating comprises evaporating the crude acesulfame potassium composition to form a water stream and an intermediate acesulfame potassium composition comprising acesulfame potassium and less than 50 wt % water, and the separating comprises crystallizing the intermediate acesulfame potassium composition to form a crystal-containing stream comprising acesulfame potassium crystals, and filtering the crystal-containing stream to form the finished acesulfame potassium composition.

23. The process of claim 22, wherein the filtering comprises at least two filtration operations.

24. The process of claim 2, wherein the crude acesulfame potassium composition is evaporated to form the water stream and the intermediate acesulfame potassium composition.

25. The process of claim 24, wherein the intermediate acesulfame potassium composition comprises less than 75 wt % water.

26. The process of claim 24, wherein the crude acesulfame potassium composition is evaporated at a temperature of less than 50° C. and the evaporator residence time is less than 30 minutes.

27. The process of claim 24, wherein the intermediate acesulfame potassium composition is crystallized to form acesulfame crystals.

28. The process of claim 27, wherein the intermediate acesulfame potassium composition is crystallized at a temperature of below 20° C.

29. The process of claim 27, wherein acesulfame crystals are filtered to form the finished acesulfame composition.

30. The process of claim 27, wherein acesulfame crystals are filtered at a temperature below 20° C.

31. The process of claim 1, wherein the finished acesulfame potassium composition comprises from 10 wppb to 15 wppm acetoacetamide.

32. The process of claim 1, wherein the finished acesulfame potassium composition comprises from 10 wppb to 10 wppm acetoacetamide.

33. The process of claim 1, wherein the finished acesulfame potassium composition comprises from 10 wppb to 7 wppm acetoacetamide.

34. The process of claim 1, wherein the finished acesulfame potassium composition comprises less than 33 wppm acetoacetamide-N-sulfonic acid.

35. The process of claim 1, wherein the finished acesulfame potassium composition comprises from 1 wppb to 20 wppm acetoacetamide-N-sulfonic acid.

36. The process of claim 1, wherein the finished acesulfame potassium composition comprises from 10 wppb to 10 wppm acetoacetamide and from 1 wppb to 20 wppm acetoacetamide-N-sulfonic acid.

37. The process of claim 1, wherein the finished acesulfame potassium composition comprises 0.001 wppm to 5 wppm organic impurities and/or 0.001 wppm to 5 wppm of at least one heavy metal.

38. The process of claim 1, wherein the concentrating step (b) is conducted for a residence time of less than 120 minutes.

39. The process of claim 1, wherein the separating step (c) is conducted for a residence time of from 10 to 100 minutes.

* * * * *

ADDENDUM WITH STATUTORY EXCERPTS

CELANESE INTERNATIONAL CORPORATION, CELANESE (MALTA) COMPANY 2 LIMITED, CELANESE SALES U.S. LTD.,

v.

INTERNATIONAL TRADE COMMISSION, ANHUI JINHE INDUSTRIAL CO., LTD., JINHE USA LLC

No. 22-1827

ADDENDUM WITH STATUTORY EXCERPTS

TABLE OF CONTENTS

35 U.S.C. § 100 (Current)

§ 100. Definitions

When used in this title unless the context otherwise indicates-

(a) The term "invention" means invention or discovery.

(b) The term "process" means process, art or method, and includes a new use of a known process, machine, manufacture, composition of matter, or material.

(c) The terms "United States" and "this country" mean the United States of America, its territories and possessions.

(d) The word "patentee" includes not only the patentee to whom the patent was issued but also the successors in title to the patentee.

(e) The term "third-party requester" means a person requesting ex parte reexamination under section 302 who is not the patent owner.

(f) The term "inventor" means the individual or, if a joint invention, the individuals collectively who invented or discovered the subject matter of the invention.

(g) The terms "joint inventor" and "coinventor" mean any 1 of the individuals who invented or discovered the subject matter of a joint invention.

(h) The term "joint research agreement" means a written contract, grant, or cooperative agreement entered into by 2 or more persons or entities for the performance of experimental, developmental, or research work in the field of the claimed invention.

(i)(1) The term "effective filing date" for a claimed invention in a patent or application for patent means-

> (A) if subparagraph (B) does not apply, the actual filing date of the patent or the application for the patent containing a claim to the invention; or
>
> (B) the filing date of the earliest application for which the patent or application is entitled, as to such invention, to a right of priority under section 119, 365(a), or 365(b) or to the benefit of an earlier filing date under section 120, 121, or 365(c).

(2) The effective filing date for a claimed invention in an application for reissue or reissued patent shall be determined by deeming the claim to the invention to have been contained in the patent for which reissue was sought.

(j) The term "claimed invention" means the subject matter defined by a claim in a patent or an application for a patent.

35 U.S.C. § 102 (Current)

§ 102. Conditions for patentability; novelty

(a) **NOVELTY; PRIOR ART**.-A person shall be entitled to a patent unless-

(1) the claimed invention was patented, described in a printed publication, or in public use, on sale, or otherwise available to the public before the effective filing date of the claimed invention; or

(2) the claimed invention was described in a patent issued under section 151, or in an application for patent published or deemed published under section 122(b), in which the patent or application, as the case may be, names another inventor and was effectively filed before the effective filing date of the claimed invention.

(b) **EXCEPTIONS**.-

(1) **DISCLOSURES MADE 1 YEAR OR LESS BEFORE THE EFFECTIVE FILING DATE OF THE CLAIMED INVENTION**.-

A disclosure made 1 year or less before the effective filing date of a claimed invention shall not be prior art to the claimed invention under subsection (a)(1) if-

(A) the disclosure was made by the inventor or joint inventor or by another who obtained the subject matter disclosed directly or indirectly from the inventor or a joint inventor; or

(B) the subject matter disclosed had, before such disclosure, been publicly disclosed by the inventor or a joint inventor or another who obtained the subject matter disclosed directly or indirectly from the inventor or a joint inventor.

(2) **DISCLOSURES APPEARING IN APPLICATIONS AND PATENTS**.-
A disclosure shall not be prior art to a claimed invention under subsection(a)(2) if-

(A) the subject matter disclosed was obtained directly or indirectly from the inventor or a joint inventor;

(B) the subject matter disclosed had, before such subject matter was effectively filed under subsection (a)(2), been publicly disclosed by the inventor or a joint inventor or another who obtained the subject matter disclosed directly or indirectly from the inventor or a joint inventor; or

(C) the subject matter disclosed and the claimed invention, not later than the effective filing date of the claimed invention, were owned by the same person or subject to an obligation of assignment to the same person.

(c) **COMMON OWNERSHIP UNDER JOINT RESEARCH AGREEMENTS**.-Subject matter disclosed and a claimed invention shall be deemed to have been owned by the same person or subject to an obligation of assignment to the same person in applying the provisions of subsection (b)(2)(C) if-

(1) the subject matter disclosed was developed and the claimed invention was made by, or on behalf of, 1 or more parties to a joint research agreement that was in effect on or before the effective filing date of the claimed invention;

(2) the claimed invention was made as a result of activities undertaken within the scope of the joint research agreement; and

(3) the application for patent for the claimed invention discloses or is amended to disclose the names of the parties to the joint research agreement.

(d) **PATENTS AND PUBLISHED APPLICATIONS EFFECTIVE AS PRIOR ART**.-For purposes of determining whether a patent or application for patent is prior art to a claimed invention under subsection (a)(2), such patent or application shall be considered to have been effectively filed, with respect to any subject matter described in the patent or application-

(1) if paragraph (2) does not apply, as of the actual filing date of the patent or the application for patent; or

(2) if the patent or application for patent is entitled to claim a right of priority under section 119, 365(a), or 365(b), or to claim the benefit of an earlier filing date under section 120, 121, or 365(c), based upon 1 or more prior filed applications for patent, as of the filing date of the earliest such application that describes the subject matter.

35 U.S.C. § 102 (2006)

§ 102. Conditions for patentability; novelty and loss of right to patent

A person shall be entitled to a patent unless-

(a) the invention was known or used by others in this country, or patented or described in a printed publication in this or a foreign country, before the invention thereof by the applicant for patent, or

(b) the invention was patented or described in a printed publication in this or a foreign country or in public use or on sale in this country, more than one year prior to the date of the application for patent in the United States, or

(c) he has abandoned the invention, or

(d) the invention was first patented or caused to be patented, or was the subject of an inventor's certificate, by the applicant or his legal representatives or assigns in a foreign country prior to the date of the application for patent in this country on an application for patent or inventor's certificate filed more than twelve months before the filing of the application in the United States, or

(e) the invention was described in (1) an application for patent, published under section 122(b), by another filed in the United States before the invention by the applicant for patent or (2) a patent granted on an application for patent by another filed in the United States before the invention by the applicant for patent, except that an international application filed under the treaty defined in section 351(a) shall have the effects for the purposes of this subsection of an application filed in the United States only if the international application designated the United States and was published under Article 21(2) of such treaty in the English language;[1] or

(f) he did not himself invent the subject matter sought to be patented, or

(g)

(1) during the course of an interference conducted under section 135 or section 291, another inventor involved therein establishes, to the extent permitted in section 104, that before such person's invention thereof the invention was made by such other inventor and not abandoned, suppressed, or concealed, or

[1] So in original. The semicolon probably should be a comma.

(2) before such person's invention thereof, the invention was made in this country by another inventor who had not abandoned, suppressed, or concealed it. In determining priority of invention under this subsection, there shall be considered not only the respective dates of conception and reduction to practice of the invention, but also the reasonable diligence of one who was first to conceive and last to reduce to practice, from a time prior to conception by the other.

35 U.S.C. § 102 (1952)

§ 102. Conditions for patentability; novelty and loss of right to patent.

A person shall be entitled to a patent unless-

(a) the invention was known or used by others in this country, or patented or described in a printed publication in this or a foreign country, before the invention thereof by the applicant for patent, or

(b) the invention was patented or described in a printed publication in this or a foreign country or in public use or on sale in this country, more than one year prior to the date of the application for patent in the United States, or

(c) he has abandoned the invention, or

(d) the invention was first patented or caused to be patented by the applicant or his legal representatives or assigns in a foreign country prior to the date of the application for patent in this country on an application filed more than twelve months before the filing of the application in the United States, or

(e) the invention was described in a patent granted on an application for patent by another filed in the United States before the invention thereof by the applicant for patent, or

(f) he did not himself invent the subject matter sought to be patented, or

(g) before the applicant's invention thereof the invention was made in this country by another who had not abandoned, suppressed, or concealed it. In determining priority of invention there shall be considered not only the respective dates of concept and reduction to practice of the invention, but also the reasonable diligence of one who was first to conceive and last to reduce to practice, from a time prior to conception by the other. (July 19, 1952, ch. 950, § 1, 66 Stat. 797.)

35 U.S.C. § 271 (Current)

§ 271. Infringement of patent

(a) Except as otherwise provided in this title, whoever without authority makes, uses, offers to sell, or sells any patented invention, within the United States or imports into the United States any patented invention during the term of the patent therefor, infringes the patent.

(b) Whoever actively induces infringement of a patent shall be liable as an infringer.

(c) Whoever offers to sell or sells within the United States or imports into the United States a component of a patented machine, manufacture, combination or composition, or a material or apparatus for use in practicing a patented process, constituting a material part of the invention, knowing the same to be especially made or especially adapted for use in an infringement of such patent, and not a staple article or commodity of commerce suitable for substantial noninfringing use, shall be liable as a contributory infringer.

(d) No patent owner otherwise entitled to relief for infringement or contributory infringement of a patent shall be denied relief or deemed guilty of misuse or illegal extension of the patent right by reason of his having done one or more of the following: (1) derived revenue from acts which if performed by another without his consent would constitute contributory infringement of the patent; (2) licensed or authorized another to perform acts which if performed without his consent would constitute contributory infringement of the patent; (3) sought to enforce his patent rights against infringement or contributory infringement; (4) refused to license or use any rights to the patent; or (5) conditioned the license of any rights to the patent or the sale of the patented product on the acquisition of a license to rights in another patent or purchase of a separate product, unless, in view of the circumstances, the patent owner has market power in the relevant market for the patent or patented product on which the license or sale is conditioned.

(e)

(1) It shall not be an act of infringement to make, use, offer to sell, or sell within the United States or import into the United States a patented invention (other than a new animal drug or veterinary biological product (as those terms are used in the Federal Food, Drug, and Cosmetic Act and the Act of March 4, 1913) which is primarily manufactured using recombinant DNA, recombinant RNA, hybridoma technology, or other processes involving site specific genetic manipulation techniques) solely for uses reasonably related to the development and submission

of information under a Federal law which regulates the manufacture, use, or sale of drugs or veterinary biological products.

(2) It shall be an act of infringement to submit-

(A) an application under section 505(j) of the Federal Food, Drug, and

Cosmetic Act or described in section 505(b)(2) of such Act for a drug claimed in a patent or the use of which is claimed in a patent,

(B) an application under section 512 of such Act or under the Act of March 4, 1913 (21 U.S.C. 151-158) for a drug or veterinary biological product which is not primarily manufactured using recombinant DNA, recombinant RNA, hybridoma technology, or other processes involving site specific genetic manipulation techniques and which is claimed in a patent or the use of which is claimed in a patent, or

(C)

(i) with respect to a patent that is identified in the list of patents described in section 351(l)(3) of the Public Health Service Act (including as provided under section 351(l)(7) of such Act), an application seeking approval of a biological product, or

(ii) if the applicant for the application fails to provide the application and information required under section 351(l)(2)(A) of such Act, an application seeking approval of a biological product for a patent that could be identified pursuant to section 351(l)(3)(A)(i) of such Act, if the purpose of such submission is to obtain approval under such Act to engage in the commercial manufacture, use, or sale of a drug, veterinary biological product, or biological product claimed in a patent or the use of which is claimed in a patent before the expiration of such patent.

(3) In any action for patent infringement brought under this section, no injunctive or other relief may be granted which would prohibit the making, using, offering to sell, or selling within the United States or importing into the United States of a patented invention under paragraph (1).

(4) For an act of infringement described in paragraph (2)-

(A) the court shall order the effective date of any approval of the drug or veterinary biological product involved in the infringement to be a date which

is not earlier than the date of the expiration of the patent which has been infringed.

(B) injunctive relief may be granted against an infringer to prevent the commercial manufacture, use, offer to sell, or sale within the United States or importation into the United States of an approved drug, veterinary biological product, or biological product,

(C) damages or other monetary relief may be awarded against an infringer only if there has been commercial manufacture, use, offer to sell, or sale within the United States or importation into the United States of an approved drug, veterinary biological product, or biological product, and

(D) the court shall order a permanent injunction prohibiting any infringement of the patent by the biological product involved in the infringement until a date which is not earlier than the date of the expiration of the patent that has been infringed under paragraph (2)(C), provided the patent is the subject of a final court decision, as defined in section 351(k)(6) of the Public Health Service Act, in an action for infringement of the patent under section 351(l)(6) of such Act, and the biological product has not yet been approved because of section 351(k)(7) of such Act.

The remedies prescribed by subparagraphs (A), (B), (C), and (D) are the only remedies which may be granted by a court for an act of infringement described in paragraph (2), except that a court may award attorney fees under section 285.

(5) Where a person has filed an application described in paragraph (2) that includes a certification under subsection (b)(2)(A)(iv) or (j)(2)(A)(vii)(IV) of section 505 of the Federal

35 U.S.C. § 273 (Current)

§ 273. Defense to infringement based on prior commercial use

(a) **IN GENERAL**.-A person shall be entitled to a defense under section 282(b) with respect to subject matter consisting of a process, or consisting of a machine, manufacture, or composition of matter used in a manufacturing or other commercial process, that would otherwise infringe a claimed invention being asserted against the person if-

(1) such person, acting in good faith, commercially used the subject matter in the United States, either in connection with an internal commercial use or an actual arm's length sale or other arm's length commercial transfer of a useful end result of such commercial use; and

(2) such commercial use occurred at least 1 year before the earlier of either-

(A) the effective filing date of the claimed invention; or

(B) the date on which the claimed invention was disclosed to the public in a manner that qualified for the exception from prior art under section 102(b).

(b) **BURDEN OF PROOF**.-A person asserting a defense under this section shall have the burden of establishing the defense by clear and convincing evidence.

(c) **ADDITIONAL COMMERCIAL USES**.-

(1) **PREMARKETING REGULATORY REVIEW**.-Subject matter for which commercial marketing or use is subject to a premarketing regulatory review period during which the safety or efficacy of the subject matter is established, including any period specified in section 156(g), shall be deemed to be commercially used for purposes of subsection (a)(1) during such regulatory review period.

(2) **NONPROFIT LABORATORY USE**.-A use of subject matter by a nonprofit research laboratory or other nonprofit entity, such as a university or hospital, for which the public is the intended beneficiary, shall be deemed to be a commercial use for purposes of subsection (a)(1), except that a defense under this section may be asserted pursuant to this paragraph only for continued and noncommercial use by and in the laboratory or other nonprofit entity.

(d) **EXHAUSTION OF RIGHTS**.-Notwithstanding subsection (e)(1), the sale or other disposition of a useful end result by a person entitled to assert a defense under this section in connection with a patent with respect to that useful end result shall

exhaust the patent owner's rights under the patent to the extent that such rights would have been exhausted had such sale or other disposition been made by the patent owner.

(e) **LIMITATIONS AND EXCEPTIONS**.-

(1) **PERSONAL DEFENSE**.-

(A) In general.-A defense under this section may be asserted only by the person who performed or directed the performance of the commercial use described in subsection (a), or by an entity that controls, is controlled by, or is under common control with such person.

(B) Transfer of right.-Except for any transfer to the patent owner, the right to assert a defense under this section shall not be licensed or assigned or transferred to another person except as an ancillary and subordinate part of a good-faith assignment or transfer for other reasons of the entire enterprise or line of business to which the defense relates.

(C) Restriction on sites.-A defense under this section, when acquired by a person as part of an assignment or transfer described in subparagraph (B), may only be asserted for uses at sites where the subject matter that would otherwise infringe a claimed invention is in use before the later of the effective filing date of the claimed invention or the date of the assignment or transfer of such enterprise or line of business.

(2) **DERIVATION**.-A person may not assert a defense under this section if the subject matter on which the defense is based was derived from the patentee or persons in privity with the patentee.

(3) **NOT A GENERAL LICENSE**.-The defense asserted by a person under this section is not a general license under all claims of the patent at issue, but extends only to the specific subject matter for which it has been established that a commercial use that qualifies under this section occurred, except that the defense shall also extend to variations in the quantity or volume of use of the claimed subject matter, and to improvements in the claimed subject matter that do not infringe additional specifically claimed subject matter of the patent.

(4) **ABANDONMENT OF USE**.-A person who has abandoned commercial use (that qualifies under this section) of subject matter may not rely on activities performed before the date of such abandonment in establishing a defense under this section with respect to actions taken on or after the date of such abandonment.

(5) **UNIVERSITY EXCEPTION**.-

(A) In general.-A person commercially using subject matter to which subsection (a) applies may not assert a defense under this section if the claimed invention with respect to which the defense is asserted was, at the time the invention was made, owned or subject to an obligation of assignment to either an institution of higher education (as defined in section 101(a) of the Higher Education Act of 1965 (20 U.S.C. 1001(a)),1 or a technology transfer organization whose primary purpose is to facilitate the commercialization of technologies developed by one or more such institutions of higher education.

(B) Exception.-Subparagraph (A) shall not apply if any of the activities required to reduce to practice the subject matter of the claimed invention could not have been undertaken using funds provided by the Federal Government.

(f) **UNREASONABLE ASSERTION OF DEFENSE**.-If the defense under this section is pleaded by a person who is found to infringe the patent and who subsequently fails to demonstrate a reasonable basis for asserting the defense, the court shall find the case exceptional for the purpose of awarding attorney fees under section 285.

(g**) INVALIDITY**.-A patent shall not be deemed to be invalid under section 102 or 103 solely because a defense is raised or established under this section.

19 U.S.C. § 1337 (Current)

§ 1337. Unfair practices in import trade

(a) **Unlawful activities; covered industries; definitions**

(1) Subject to paragraph (2), the following are unlawful, and when found by the Commission to exist shall be dealt with, in addition to any other provision of law, as provided in this section:

(A) Unfair methods of competition and unfair acts in the importation of articles (other than articles provided for in subparagraphs (B), (C), (D), and (E)) into the United States, or in the sale of such articles by the owner, importer, or consignee, the threat or effect of which is-

(i) to destroy or substantially injure an industry in the United States;

(ii) to prevent the establishment of such an industry; or

(iii) to restrain or monopolize trade and commerce in the United States.

(B) The importation into the United States, the sale for importation, or the sale within the United States after importation by the owner, importer, or consignee, of articles that-

(i) infringe a valid and enforceable United States patent or a valid and enforceable United States copyright registered under title 17; or

(ii) are made, produced, processed, or mined under, or by means of, a process covered by the claims of a valid and enforceable United States patent.

(C) The importation into the United States, the sale for importation, or the sale within the United States after importation by the owner, importer, or consignee, of articles that infringe a valid and enforceable United States trademark registered under the Trademark Act of 1946 [15 U.S.C. 1051 et seq.].

(D) The importation into the United States, the sale for importation, or the sale within the United States after importation by the owner, importer, or consignee, of a semiconductor chip product in a manner that constitutes infringement of a mask work registered under chapter 9 of title 17.

(E) The importation into the United States, the sale for importation, or the sale within the United States after importation by the owner, importer, or consigner, of an article that constitutes infringement of the exclusive rights in a design protected under chapter 13 of title 17.

(2) Subparagraphs (B), (C), (D), and (E) of paragraph (1) apply only if an industry in the United States, relating to the articles protected by the patent, copyright, trademark, mask work, or design concerned, exists or is in the process of being established.

(3) For purposes of paragraph (2), an industry in the United States shall be considered to exist if there is in the United States, with respect to the articles protected by the patent, copyright, trademark, mask work, or design concerned-

(A) significant investment in plant and equipment;

(B) significant employment of labor or capital; or

(C) substantial investment in its exploitation, including engineering, research and development, or licensing.

(4) For the purposes of this section, the phrase "owner, importer, or consignee" includes any agent of the owner, importer, or consignee.

(b) **Investigation of violations by Commission**

(1) The Commission shall investigate any alleged violation of this section on complaint under oath or upon its initiative. Upon commencing any such investigation, the Commission shall publish notice thereof in the Federal Register. The Commission shall conclude any such investigation and make its determination under this section at the earliest practicable time after the date of publication of notice of such investigation. To promote expeditious adjudication, the Commission shall, within 45 days after an investigation is initiated, establish a target date for its final determination.

(2) During the course of each investigation under this section, the Commission shall consult with, and seek advice and information from, the Department of Health and Human Services, the Department of Justice, the Federal Trade Commission, and such other departments and agencies as it considers appropriate.

(3) Whenever, in the course of an investigation under this section, the Commission has reason to believe, based on information before it, that a matter, in whole or in part, may come within the purview of part II of subtitle IV of this chapter, it shall promptly notify the Secretary of Commerce so that such action may be taken as is otherwise authorized by such part II. If the Commission has reason to believe that the matter before it (A) is based solely on alleged acts and effects which are within the purview of section 1671 or 1673 of this title, or (B) relates to an alleged copyright infringement with respect to which action is prohibited by section 1008 of title 17, the Commission shall terminate, or not institute, any investigation into the matter. If the Commission has reason to believe the matter before it is based in part on alleged acts and effects which are within the purview of section 1671 or 1673 of this title, and in part on alleged acts and effects which may, independently from or in conjunction with those within the purview of such section, establish a basis for relief under this section, then it may institute or continue an investigation into the matter. If the Commission notifies the Secretary or the administering authority (as defined in section 1677(1) of this title) with respect to a matter under this paragraph, the Commission may suspend its investigation during the time the matter is before the Secretary or administering authority for final decision. Any final decision by the administering authority under section 1671 or 1673 of this title with respect to the matter within such section 1671 or 1673 of this title of which the Commission has notified the Secretary or administering authority shall be conclusive upon the Commission with respect to the issue of less than- fair-value sales or subsidization and the matters necessary for such decision.

(c) **Determinations; review**

The Commission shall determine, with respect to each investigation conducted by it under this section, whether or not there is a violation of this section, except that the Commission may, by issuing a consent order or on the basis of an agreement between the private parties to the investigation, including an agreement to present the matter for arbitration, terminate any such investigation, in whole or in part, without making such a determination. Each determination under subsection (d) or (e) of this section shall be made on the record after notice and opportunity for a hearing in conformity with the provisions of subchapter II of chapter 5 of title 5. All legal and equitable defenses may be presented in all cases. A respondent may raise any counterclaim in a manner prescribed by the Commission. Immediately after a counterclaim is received by the Commission, the respondent raising such counterclaim shall file a notice of removal with a United States district court in which venue for any of the counterclaims raised by the party would exist under section 1391 of title 28. Any

counterclaim raised pursuant to this section shall relate back to the date of the original complaint in the proceeding before the Commission. Action on such counterclaim shall not delay or affect the proceeding under this section, including the legal and equitable defenses that may be raised under this subsection. Any person adversely affected by a final determination of the Commission under subsection (d), (e), (f), or (g) of this section may appeal such determination, within 60 days after the determination becomes final, to the United States Court of Appeals for the Federal Circuit for review in accordance with chapter 7 of title 5. Notwithstanding the foregoing provisions of this subsection, Commission determinations under subsections (d), (e), (f), and (g) of this section with respect to its findings on the public health and welfare, competitive conditions in the United States economy, the production of like or directly competitive articles in the United States, and United States consumers, the amount and nature of bond, or the appropriate remedy shall be reviewable in accordance with section 706 of title 5. Determinations by the Commission under subsections (e), (f), and (j) of this section with respect to forfeiture of bonds and under subsection (h) of this section with respect to the imposition of sanctions for abuse of discovery or abuse of process shall also be reviewable in accordance with section 706 of title 5.

(d) **Exclusion of articles from entry**

(1) If the Commission determines, as a result of an investigation under this section, that there is a violation of this section, it shall direct that the articles concerned, imported by any person violating the provision of this section, be excluded from entry into the United States, unless, after considering the effect of such exclusion upon the public health and welfare, competitive conditions in the United States economy, the production of like or directly competitive articles in the United States, and United States consumers, it finds that such articles should not be excluded from entry. The Commission shall notify the Secretary of the Treasury of its action under this subsection directing such exclusion from entry, and upon receipt of such notice, the Secretary shall, through the proper officers, refuse such entry.

(2) The authority of the Commission to order an exclusion from entry of articles shall be limited to persons determined by the Commission to be violating this section unless the Commission determines that,-

(A) a general exclusion from entry of articles is necessary to prevent circumvention of an exclusion order limited to products of named persons; or

(B) there is a pattern of violation of this section and it is difficult to identify the source of infringing products.

(e) **Exclusion of articles from entry during investigation except under bond; procedures applicable; preliminary relief**

(1) If, during the course of an investigation under this section, the Commission determines that there is reason to believe that there is a violation of this section, it may direct that the articles concerned, imported by any person with respect to whom there is reason to believe that such person is violating this section, be excluded from entry into the United States, unless, after considering the effect of such exclusion upon the public health and welfare, competitive conditions in the United States economy, the production of like or directly competitive articles in the United States, and United States consumers, it finds that such articles should not be excluded from entry. The Commission shall notify the Secretary of the Treasury of its action under this subsection directing such exclusion from entry, and upon receipt of such notice, the Secretary shall, through the proper officers, refuse such entry, except that such articles shall be entitled to entry under bond prescribed by the Secretary in an amount determined by the Commission to be sufficient to protect the complainant from any injury. If the Commission later determines that the respondent has violated the provisions of this section, the bond may be forfeited to the complainant.

(2) A complainant may petition the Commission for the issuance of an order under this subsection. The Commission shall make a determination with regard to such petition by no later than the 90th day after the date on which the Commission's notice of investigation is published in the Federal Register. The Commission may extend the 90-day period for an additional 60 days in a case it designates as a more complicated case. The Commission shall publish in the Federal Register its reasons why it designated the case as being more complicated. The Commission may require the complainant to post a bond as a prerequisite to the issuance of an order under this subsection. If the Commission later determines that the respondent has not violated the provisions of this section, the bond may be forfeited to the respondent.

(3) The Commission may grant preliminary relief under this subsection or subsection (f) of this section to the same extent as preliminary injunctions and temporary restraining orders may be granted under the Federal Rules of Civil Procedure.

(4) The Commission shall prescribe the terms and conditions under which bonds may be forfeited under paragraphs (1) and (2).

(f) **Cease and desist orders; civil penalty for violation of orders**

(1) In addition to, or in lieu of, taking action under subsection (d) or (e) of this section, the Commission may issue and cause to be served on any person violating this section, or believed to be violating this section, as the case may be, an order directing such person to cease and desist from engaging in the unfair methods or acts involved, unless after considering the effect of such order upon the public health and welfare, competitive conditions in the United States economy, the production of like or directly competitive articles in the United States, and United States consumers, it finds that such order should not be issued. The Commission may at any time, upon such notice and in such manner as it deems proper, modify or revoke any such order, and, in the case of a revocation, may take action under subsection (d) or (e) of this section, as the case may be. If a temporary cease and desist order is issued in addition to, or in lieu of, an exclusion order under subsection (e) of this section, the Commission may require the complainant to post a bond, in an amount determined by the Commission to be sufficient to protect the respondent from any injury, as a prerequisite to the issuance of an order under this subsection. If the Commission later determines that the respondent has not violated the provisions of this section, the bond may be forfeited to the respondent. The Commission shall prescribe the terms and conditions under which the bonds may be forfeited under this paragraph.

(2) Any person who violates an order issued by the Commission under paragraph (1) after it has become final shall forfeit and pay to the United States a civil penalty for each day on which an importation of articles, or their sale, occurs in violation of the order of not more than the greater of $100,000 or twice the domestic value of the articles entered or sold on such day in violation of the order. Such penalty shall accrue to the United States and may be recovered for the United States in a civil action brought by the Commission in the Federal District Court for the District of Columbia or for the district in which the violation occurs. In such actions, the United States district courts may issue mandatory injunctions incorporating the relief sought by the Commission as they deem appropriate in the enforcement of such final orders of the Commission.

(g) **Exclusion from entry or cease and desist order; conditions and procedures applicable**

(1) If-

(A) a complaint is filed against a person under this section;

(B) the complaint and a notice of investigation are served on the person;

(C) the person fails to respond to the complaint and notice or otherwise fails to appear to answer the complaint and notice;

(D) the person fails to show good cause why the person should not be found in default; and

(E). the complainant seeks relief limited solely to that person;

the Commission shall presume the facts alleged in the complaint to be true and shall, upon request, issue an exclusion from entry or a cease and desist order, or both, limited to that person unless, after considering the effect of such exclusion or order upon the public health and welfare, competitive conditions in the United States economy, the production of like or directly competitive articles in the United States, and United States consumers, the Commission finds that such exclusion or order should not be issued.

(2) In addition to the authority of the Commission to issue a general exclusion from entry of articles when a respondent appears to contest an investigation concerning a violation of the provisions of this section, a general exclusion from entry of articles, regardless of the source or importer of the articles, may be issued if-

(A) no person appears to contest an investigation concerning a violation of the provisions of this section,

(B) such a violation is established by substantial, reliable, and probative evidence, and

(C) the requirements of subsection (d)(2) of this section are met.

(h) **Sanctions for abuse of discovery and abuse of process** The Commission may by rule prescribe sanctions for abuse of discovery and abuse of process to the extent authorized by Rule 11 and Rule 37 of the Federal Rules of Civil Procedure.

(i) **Forfeiture**

(1) In addition to taking action under subsection (d) of this section, the Commission may issue an order providing that any article imported in violation of the provisions of this section be seized and forfeited to the United States if-

(A) the owner, importer, or consignee of the article previously attempted to import the article into the United States;

(B) the article was previously denied entry into the United States by reason of an order issued under subsection (d) of this section; and

(C) upon such previous denial of entry, the Secretary of the Treasury provided the owner, importer, or consignee of the article written notice of-

(i) such order, and

(ii) the seizure and forfeiture that would result from any further attempt to import the article into the United States.

(2) The Commission shall notify the Secretary of the Treasury of any order issued under this subsection and, upon receipt of such notice, the Secretary of the Treasury shall enforce such order in accordance with the provisions of this section.

(3) Upon the attempted entry of articles subject to an order issued under this subsection, the Secretary of the Treasury shall immediately notify all ports of entry of the attempted importation and shall identify the persons notified under paragraph (1)(C).

(4) The Secretary of the Treasury shall provide-

(A) the written notice described in' paragraph (1)(C) to the owner, importer, or consignee of any article that is denied entry into the United States by reason of an order issued under subsection (d) of this section; and

(B) a copy of such written notice to the Commission.

(j) **Referral to President**

(1) If the Commission determines that there is a violation of this section, or that, for purposes of subsection (e) of this section, there is reason to believe that there is such a violation, it shall-

(A) publish such determination in the Federal Register, and

(B) transmit to the President a copy of such determination and the action taken under subsection (d), (e), (f), (g), or (i) of this section, with respect thereto, together with the record upon which such determination is based.

(2) If, before the close of the 60-day period beginning on the day after the day on which he receives a copy of such determination, the President, for policy reasons, disapproves such determination and notifies the Commission of his disapproval, then, effective on the date of such notice, such determination and the action taken under subsection (d), (e), (f), (g), or (i) of this section with respect thereto shall have no force or effect.

(3) Subject to the provisions of paragraph (2), such determination shall, except for purposes of subsection (c) of this section, be effective upon publication thereof in the Federal Register, and the action taken under subsection (d), (e), (f), (g), or (i) of this section, with respect thereto shall be effective as provided in such subsections, except that articles directed to be excluded from entry under subsection (d) of this section or subject to a cease and desist order under subsection (f) of this section shall, until such determination becomes final, be entitled to entry under bond prescribed by the Secretary in an amount determined by the Commission to be sufficient to protect the complainant from any injury. If the determination becomes final, the bond may be forfeited to the complainant. The Commission shall prescribe the terms and conditions under which bonds may be forfeited under this paragraph.

(4) If the President does not disapprove such determination within such 60-day period, or if he notifies the Commission before the close of such period that he approves such determination, then, for purposes of paragraph (3) and subsection (c) of this section such determination shall become final on the day after the close of such period or the day on which the President notifies the Commission of his approval, as the case may be.

(k) **Period of effectiveness; termination of violation or modification or rescission of exclusion or order**

(1) Except as provided in subsections (f) and (j) of this section, any exclusion from entry or order under this section shall continue in effect until the Commission finds, and in the case of exclusion from entry notifies the Secretary of the Treasury, that the conditions which led to such exclusion from entry or order no longer exist.

(2) If any person who has previously been found by the Commission to be in violation of this section petitions the Commission for a determination that the petitioner is no longer in violation of this section or for a modification or rescission of an exclusion from entry or order under subsection (d), (e), (f), (g), or (i) of this section-

(A) the burden of proof in any proceeding before the Commission regarding such petition shall be on the petitioner; and

(B) relief may be granted by the Commission with respect to such petition-

(i) on the basis of new evidence or evidence that could not have been presented at the prior proceeding, or

(ii) on grounds which would permit relief from a judgment or order under the Federal Rules of Civil Procedure.

(l) **Importation by or for United States** Any exclusion from entry or order under subsection (d), (e), (f), (g), or (i) of this section, in cases based on a proceeding involving a patent, copyright, mask work, or design under subsection (a)(1) of this section, shall not apply to any articles imported by and for the use of the United States, or imported for, and to be used for, the United States with the authorization or consent of the Government. Whenever any article would have been excluded from entry or would not have been entered pursuant to the provisions of such subsections but for the operation of this subsection, an owner of the patent, copyright, mask work, or design adversely affected shall be entitled to reasonable and entire compensation in an action before the United States Court of Federal Claims pursuant to the procedures of section 1498 of title 28.

(m) **"United States" defined**

For purposes of this section and sections 1338 and 13401 of this title, the term "United States" means the customs territory of the United States as defined in general note 2 of the Harmonized Tariff Schedule of the United States.

(n) **Disclosure of confidential information**

(1) Information submitted to the Commission or exchanged among the parties in connection with proceedings under this section which is properly designated as confidential pursuant to Commission rules may not be disclosed (except under a protective order issued under regulations of the Commission which authorizes limited disclosure of such information) to any person (other than a person described in paragraph (2)) without the consent of the person submitting it.

(2) Notwithstanding the prohibition contained in paragraph (1), information referred to in that paragraph may be disclosed to-

(A) an officer or employee of the Commission who is directly concerned with-

(i) carrying out the investigation or related proceeding in connection with which the information is submitted,

(ii) the administration of a bond posted pursuant to subsection (e), (f), or (j) of this section,

(iii) the administration or enforcement of an exclusion order issued pursuant to subsection (d), (e), or (g) of this section, a cease and desist order issued pursuant to subsection (f) of this section, or a consent order issued pursuant to subsection (c) of this section,

(iv) proceedings for the modification or rescission of a temporary or permanent order issued under subsection (d), (e), (f), (g), or (i) of this section, or a consent order issued under this section, or

(v) maintaining the administrative record of the investigation or related proceeding,

(B) an officer or employee of the United States Government who is directly involved in the review under subsection (j) of this section, or

(C) an officer or employee of the United States Customs Service who is directly involved in administering an exclusion from entry under subsection (d), (e), or (g) of this section resulting from the investigation or related proceeding in connection with which the information is submitted.

Leahy-Smith America Invents Act, 112 Pub. L. No. 112-29 Stat. 284 (2011), §§ 3, 6, 18

SEC. 3 FIRST INVENTOR TO FILE.

(a) Definitions.--Section 100 of title 35, United States Code, is amended--

(1) in subsection (e), by striking "or inter partes reexamination under section 311"; and

(2) by adding at the end the following:

"(f) The term 'inventor' means the individual or, if a joint invention, the individuals collectively who invented or discovered the subject matter of the invention.

"(g) The terms 'joint inventor' and 'coinventor' mean any 1 of the individuals who invented or discovered the subject matter of a joint invention.

"(h) The term 'joint research agreement' means a written contract, grant, or cooperative agreement entered into by 2 or more persons or entities for the performance of experimental, developmental, or research work in the field of the claimed invention.

"(i)

(1) The term 'effective filing date' for a claimed invention in a patent or application for patent means--

"(A) if subparagraph (B) does not apply, the actual filing date of the patent or the application for the patent containing a claim to the invention; or

"(B) the filing date of the earliest application for which the patent or application is entitled, as to such invention, to a right of priority under section 119, 365(a), or 365(b) or to the benefit of an earlier filing date under section 120, 121, or 365(c).

"(2) The effective filing date for a claimed invention in an application for reissue or reissued patent shall be determined by deeming the claim to the invention to have been contained in the patent for which reissue was sought.

"(j) The term 'claimed invention' means the subject matter defined by a claim in a patent or an application for a patent.".

(b) CONDITIONS FOR PATENTABILITY.--

(1) In general.--Section 102 of title 35, United States Code, is amended to read as follows:

"**§ 102. Conditions for patentability; novelty**

"(a) Novelty; Prior Art.--A person shall be entitled to a patent unless--

"(1) the claimed invention was patented, described in a printed publication, or in public use, on sale, or otherwise available to the public before the effective filing date of the claimed invention; or

"(2) the claimed invention was described in a patent issued under section 151, or in an application for patent published or deemed published under section 122(b), in which the patent or application, as the case may be, names another inventor and was effectively filed before the effective filing date of the claimed invention.

"(b) Exceptions.--

"(1) Disclosures made 1 year or less before the effective filing date of the claimed invention.--A disclosure made 1 year or less before the effective filing date of a claimed invention shall not be prior art to the claimed invention under subsection (a)(1) if--

"(A) the disclosure was made by the inventor or joint inventor or by another who obtained the subject matter disclosed directly or indirectly from the inventor or a joint inventor; or

"(B) the subject matter disclosed had, before such disclosure, been publicly disclosed by the inventor or a joint inventor or another who obtained the subject matter disclosed directly or indirectly from the inventor or a joint inventor.

"(2) Disclosures appearing in applications and patents.--A disclosure shall not be prior art to a claimed invention under subsection (a)(2) if--

"(A) the subject matter disclosed was obtained directly or indirectly from the inventor or a joint inventor;

"(B) the subject matter disclosed had, before such subject matter was effectively filed under subsection (a)(2), been publicly disclosed by the inventor or a joint inventor or another who obtained the subject matter disclosed directly or indirectly from the inventor or a joint inventor; or

"(C) the subject matter disclosed and the claimed invention, not later than the effective filing date of the claimed invention, were owned by the same person or subject to an obligation of assignment to the same person.

"(c) Common Ownership Under Joint Research Agreements.--Subject matter disclosed and a claimed invention shall be deemed to have been owned by the same person or subject to an obligation of assignment to the same person in applying the provisions of subsection (b)(2)(C) if--

"(1) the subject matter disclosed was developed and the claimed invention was made by, or on behalf of, 1 or more parties to a joint research agreement that was in effect on or before the effective filing date of the claimed invention;

"(2) the claimed invention was made as a result of activities undertaken within the scope of the joint research agreement; and

"(3) the application for patent for the claimed invention discloses or is amended to disclose the names of the parties to the joint research agreement.

"(d) Patents and Published Applications Effective as Prior Art.--For purposes of determining whether a patent or application for patent is prior art to a claimed invention under subsection (a)(2), such patent or application shall be considered to have been effectively filed, with respect to any subject matter described in the patent or application--

"(1) if paragraph (2) does not apply, as of the actual filing date of the patent or the application for patent; or

"(2) if the patent or application for patent is entitled to claim a right of priority under section 119, 365(a), or 365(b), or to claim the benefit of an earlier filing date under section 120, 121, or 365(c), based upon 1 or more prior filed applications for patent, as of the filing date of the earliest such application that describes the subject matter.".

(2) CONTINUITY OF INTENT UNDER THE CREATE ACT.--The enactment of section 102(c) of title 35, United States Code, under paragraph (1) of this subsection is done with the same intent to promote joint research activities that was expressed, including in the legislative history, through the enactment of the Cooperative Research and Technology Enhancement Act of 2004 (Public Law 108-453; the "CREATE Act"), the amendments of which are stricken by subsection (c) of this section. The United States Patent and Trademark Office shall administer section 102(c) of title 35, United States Code, in a manner consistent with the legislative history of the CREATE Act that was relevant to its administration by the United States Patent and Trademark Office.

(3) CONFORMING AMENDMENT.--The item relating to section 102 in the table of sections for chapter 10 of title 35, United States Code, is amended to read as follows:

"102. Conditions for patentability; novelty.".

(c) CONDITIONS FOR PATENTABILITY; NONOBVIOUS SUBJECT MATTER.--Section 103 of title 35, United States Code, is amended to read as follows:

"**§ 103. Conditions for patentability; non-obvious subject matter**

"A patent for a claimed invention may not be obtained, notwithstanding that the claimed invention is not identically disclosed as set forth in section 102, if the differences between the claimed invention and the prior art are such that the claimed invention as a whole would have been obvious before the effective filing date of the claimed invention to a person having ordinary skill in the art to which the claimed invention pertains. Patentability shall not be negated by the manner in which the invention was made.".

(d) REPEAL OF REQUIREMENTS FOR INVENTIONS MADE ABROAD.--Section 104 of title 35, United States Code, and the item relating to that section in the table of sections for chapter 10 of title 35, United States Code, are repealed.

(e) REPEAL OF STATUTORY INVENTION REGISTRATION.--

(1) IN GENERAL.--Section 157 of title 35, United States Code, and the item relating to that section in the table of sections for chapter 14 of title 35, United States Code, are repealed.

(2) REMOVAL OF CROSS REFERENCES.--Section 111(b)(8) of title 35, United States Code, is amended by striking "sections 115, 131, 135, and 157" and inserting "sections 131 and 135".

(3) EFFECTIVE DATE.--The amendments made by this subsection shall take effect upon the expiration of the 18-month period beginning on the date of the enactment of this Act, and shall apply to any request for a statutory invention registration filed on or after that effective date.

(f) EARLIER FILING DATE FOR INVENTOR AND JOINT INVENTOR.--Section 120 of title 35, United States Code, is amended by striking "which is filed by an inventor or inventors named" and inserting "which names an inventor or joint inventor".

(g) CONFORMING AMENDMENTS.--

(1) RIGHT OF PRIORITY.--Section 172 of title 35, United States Code, is amended by striking "and the time specified in section 102(d)".

(2) LIMITATION ON REMEDIES.--Section 287(c)(4) of title 35, United States Code, is amended by striking "the earliest effective filing date of which is prior to" and inserting "which has an effective filing date before".

(3) INTERNATIONAL APPLICATION DESIGNATING THE UNITED STATES: EFFECT.-- Section 363 of title 35, United States Code, is amended by striking "except as otherwise provided in section 102(e) of this title".

(4) PUBLICATION OF INTERNATIONAL APPLICATION: EFFECT.--Section 374 of title 35, United States Code, is amended by striking "sections 102(e) and 154(d)" and inserting "section 154(d)".

(5) PATENT ISSUED ON INTERNATIONAL APPLICATION: EFFECT.--The second sentence of section 375(a) of title 35, United States Code, is amended by striking "Subject to section 102(e) of this title, such" and inserting 'Such".

(6) LIMIT ON RIGHT OF PRIORITY.--Section 119(a) of title 35, United States Code, is amended by striking "; but no patent shall be granted" and all that follows through "one year prior to such filing".

(7) INVENTIONS MADE WITH FEDERAL ASSISTANCE.--Section 202(c) of title 35, United States Code, is amended--

(A) in paragraph (2)--

(i) by striking "publication, on sale, or public use," and all that follows through "obtained in the United States" and inserting "the 1-year period referred to in section 102(b) would end before the end of that 2-year period"; and

(ii) by striking "prior to the end of the statutory" and inserting "before the end of that 1-year"; and

(B) in paragraph (3), by striking "any statutory bar date that may occur under this title due to publication, on sale, or public use" and inserting "the expiration of the 1-year period referred to in section 102(b)".

(h) DERIVED PATENTS.--

(1) IN GENERAL.--Section 291 of title 35, United States Code, is amended to read as follows:

"**§ 291. Derived Patents**

"(a) In General.--The owner of a patent may have relief by civil action against the owner of another patent that claims the same invention and has an earlier effective filing date, if the invention claimed in such other patent was derived from the inventor of the invention claimed in the patent owned by the person seeking relief under this section.

"(b) Filing Limitation.--An action under this section may be filed only before the end of the 1-year period beginning on the date of the issuance of the first patent containing a claim to the allegedly derived invention and naming an individual alleged to have derived such invention as the inventor or joint inventor.".

(2) CONFORMING AMENDMENT.-- The item relating to section 291 in the table of sections for chapter 29 of title 35, United States Code, is amended to read as follows:

"291. Derived patents.".

(i) DERIVATION PROCEEDINGS.--Section 135 of title 35, United States Code, is amended to read as follows:

"**§ 135. Derivation proceedings**

"(a) Institution of Proceeding.--An applicant for patent may file a petition to institute a derivation proceeding in the Office. The petition shall set forth with particularity the basis for finding that an inventor named in an earlier application derived the claimed invention from an inventor named in the petitioner's application and, without authorization, the earlier application claiming such invention was filed. Any such petition may be filed only within the 1-year period beginning on the date of the first publication of a claim to an invention that is the same or substantially the same as the earlier application's claim to the invention, shall be made under oath, and shall be supported by substantial evidence. Whenever the Director determines that a petition filed under this subsection demonstrates that the standards for instituting a derivation proceeding are met, the Director may institute a derivation proceeding. The determination by the Director whether to institute a derivation proceeding shall be final and nonappealable.

"(b) Determination by Patent Trial and Appeal Board.--In a derivation proceeding instituted under subsection (a), the Patent Trial and Appeal Board shall determine whether an inventor named in the earlier application derived the claimed invention from an inventor named in the petitioner's application and, without authorization, the earlier application claiming such invention was filed. In appropriate circumstances, the Patent Trial and Appeal Board may correct the naming of the inventor in any application or patent at issue. The Director shall prescribe regulations setting forth standards for the conduct of derivation proceedings, including requiring parties to provide sufficient evidence to prove and rebut a claim of derivation.

"(c) Deferral of Decision.--The Patent Trial and Appeal Board may defer action on a petition for a derivation proceeding until the expiration of the 3-month period beginning on the date on which the Director issues a patent that includes the claimed invention that is the subject of the petition. The Patent Trial and Appeal Board also may

defer action on a petition for a derivation proceeding, or stay the proceeding after it has been instituted, until the termination of a proceeding under chapter 30, 31, or 32 involving the patent of the earlier applicant.

"(d) Effect of Final Decision.--The final decision of the Patent Trial and Appeal Board, if adverse to claims in an application for patent, shall constitute the final refusal by the Office on those claims. The final decision of the Patent Trial and Appeal Board, if adverse to claims in a patent, shall, if no appeal or other review of the decision has been or can be taken or had, constitute cancellation of those claims, and notice of such cancellation shall be endorsed on copies of the patent distributed after such cancellation.

"(e) Settlement.--Parties to a proceeding instituted under subsection (a) may terminate the proceeding by filing a written statement reflecting the agreement of the parties as to the correct inventors of the claimed invention in dispute. Unless the Patent Trial and Appeal Board finds the agreement to be inconsistent with the evidence of record, if any, it shall take action consistent with the agreement. Any written settlement or understanding of the parties shall be filed with the Director. At the request of a party to the proceeding, the agreement or understanding shall be treated as business confidential information, shall be kept separate from the file of the involved patents or applications, and shall be made available only to Government agencies on written request, or to any person on a showing of good cause.

"(f) Arbitration.--Parties to a proceeding instituted under subsection (a) may, within such time as may be specified by the Director by regulation, determine such contest or any aspect thereof by arbitration. Such arbitration shall be governed by the provisions of title 9, to the extent such title is not inconsistent with this section. The parties shall give notice of any arbitration award to the Director, and such award shall, as between the parties to the arbitration, be dispositive of the issues to which it relates. The arbitration award shall be unenforceable until such notice is given. Nothing in this subsection shall preclude the Director from determining the patentability of the claimed inventions involved in the proceeding.".

(j) ELIMINATION OF REFERENCES TO INTERFERENCES.-- (1) Sections 134, 145, 146, 154, and 305 of title 35, United States Code, are each amended by striking "Board of Patent Appeals and Interferences" each place it appears and inserting "Patent Trial and Appeal Board".

(2)(A) Section 146 of title 35, United States Code, is amended--

(i) by striking "an interference" and inserting "a derivation proceeding"; and

(ii) by striking "the interference" and inserting "the derivation proceeding".

(B) The subparagraph heading for section 154(b)(1)(C) of title 35, United States Code, is amended to read as follows:

"(C) GUARANTEE OF ADJUSTMENTS FOR DELAYS DUE TO DERIVATION PROCEEDINGS, SECRECY ORDERS, AND APPEALS.".

(3) The section heading for section 134 of title 35, United States Code, is amended to read as follows:

"**§ 134. Appeal to the Patent Trial and Appeal Board**".

(4) The section heading for section 146 of title 35, United States Code, is amended to read as follows:

"**§ 146. Civil action in case of derivation proceeding**".

(5) The items relating to sections 134 and 135 in the table of sections for chapter 12 of title 35, United States Code, are amended to read as follows:

"134. Appeal to the Patent Trial and Appeal Board.

"135. Derivation proceedings.".

(6) The item relating to section 146 in the table of sections for chapter 13 of title 35, United States Code, is amended to read as follows:

"146. Civil action in case of derivation proceeding.".

(k) STATUTE OF LIMITATIONS.--

(1) IN GENERAL.-- Section 32 of title 35, United States Code, is amended by inserting between the third and fourth sentences the following: "A proceeding under this section shall be commenced not later than the earlier of either the date that is 10 years after the date on which the misconduct forming the basis for the proceeding occurred, or 1 year after the date on which the misconduct forming the basis for the proceeding is made known to an officer or employee of the Office as prescribed in the regulations established under section 2(b)(2)(D).".

(2) REPORT TO CONGRESS.-- The Director shall provide on a biennial basis to the Judiciary Committees of the Senate and House of Representatives a report providing a short description of incidents made known to an officer or employee of the Office as prescribed in the regulations established under section 2(b)(2)(D) of title 35, United States Code, that reflect substantial evidence of misconduct before the Office but for which the Office was barred from commencing a proceeding under section 32 of title 35, United States Code, by the time limitation established by the fourth sentence of that section.

(3) EFFECTIVE DATE.-- The amendment made by paragraph (1) shall apply in any case in which the time period for instituting a proceeding under section 32 of title 35, United States Code, had not lapsed before the date of the enactment of this Act.

(l) SMALL BUSINESS STUDY.--

(1) DEFINITIONS.-- In this subsection--

(A) the term "Chief Counsel" means the Chief Counsel for Advocacy of the Small Business Administration;

(B) the term "General Counsel" means the General Counsel of the United States Patent and Trademark Office; and

(C) the term "small business concern" has the meaning given that term under section 3 of the Small Business Act (15 U.S.C. 632).

(2) STUDY.----

(A) IN GENERAL.-- The Chief Counsel, in consultation with the General Counsel, shall conduct a study of the effects of eliminating

the use of dates of invention in determining whether an applicant is entitled to a patent under title 35, United States Code.

(B) AREAS OF STUDY.-- The study conducted under subparagraph (A) shall include examination of the effects [*292] of eliminating the use of invention dates, including examining--

(i) how the change would affect the ability of small business concerns to obtain patents and their costs of obtaining patents;

(ii) whether the change would create, mitigate, or exacerbate any disadvantages for applicants for patents that are small business concerns relative to applicants for patents that are not small business concerns, and whether the change would create any advantages for applicants for patents that are small business concerns relative to applicants for patents that are not small business concerns;

(iii) the cost savings and other potential benefits to small business concerns of the change; and

(iv) the feasibility and costs and benefits to small business concerns of alternative means of determining whether an applicant is entitled to a patent under title 35, United States Code.

(3) REPORT.-- Not later than the date that is 1 year after the date of the enactment of this Act, the Chief Counsel shall submit to the Committee on Small Business and Entrepreneurship and the Committee on the Judiciary of the Senate and the Committee on Small Business and the Committee on the Judiciary of the House of Representatives a report on the results of the study under paragraph (2).

(m) REPORT ON PRIOR USER RIGHTS.--

(1) IN GENERAL.-- Not later than the end of the 4-month period beginning on the date of the enactment of this Act, the Director shall report, to the Committee on the Judiciary of the Senate and the Committee on the Judiciary of the House of Representatives, the findings and recommendations of the Director on the operation of prior user rights in selected countries in the industrialized world. The report shall include the following:

(A) A comparison between patent laws of the United States and the laws of other industrialized countries, including members of the European Union and Japan, Canada, and Australia.

(B) An analysis of the effect of prior user rights on innovation rates in the selected countries.

(C) An analysis of the correlation, if any, between prior user rights and start-up enterprises and the ability to attract venture capital to start new companies.

(D) An analysis of the effect of prior user rights, if any, on small businesses, universities, and individual inventors.

(E) An analysis of legal and constitutional issues, if any, that arise from placing trade secret law in patent law.

(F) An analysis of whether the change to a first-to-file patent system creates a particular need for prior user rights.

(2) CONSULTATION WITH OTHER AGENCIES.-- In preparing the report required under paragraph (1), the Director shall consult with the United States Trade Representative, the Secretary of State, and the Attorney General.

(n) EFFECTIVE DATE.--

(1) IN GENERAL.-- Except as otherwise provided in this section, the amendments made by this section shall take effect upon the expiration of the 18-month period beginning on the date of the enactment of this Act, and shall apply to any application for patent, and to any patent issuing thereon, that contains or contained at any time--

(A) a claim to a claimed invention that has an effective filing date as defined in section 100(i) of title 35, United States Code, that is on or after the effective date described in this paragraph; or

(B) a specific reference under section 120, 121, or 365(c) of title 35, United States Code, to any patent or application that contains or contained at any time such a claim.

(2) INTERFERING PATENTS.-- The provisions of sections 102(g), 135, and 291 of title 35, United States Code, as in effect on the day before the

effective date set forth in paragraph (1) of this subsection, shall apply to each claim of an application for patent, and any patent issued thereon, for which the amendments made by this section also apply, if such application or patent contains or contained at any time--

(A) a claim to an invention having an effective filing date as defined in section 100(i) of title 35, United States Code, that occurs before the effective date set forth in paragraph (1) of this subsection; or

(B) a specific reference under section 120, 121, or 365(c) of title 35, United States Code, to any patent or application that contains or contained at any time such a claim.

(o) SENSE OF CONGRESS.-- It is the sense of the Congress that converting the United States patent system from "first to invent" to a system of "first inventor to file" will promote the progress of science and the useful arts by securing for limited times to inventors the exclusive rights to their discoveries and provide inventors with greater certainty regarding the scope of protection provided by the grant of exclusive rights to their discoveries.

(p) SENSE OF CONGRESS.-- It is the sense of the Congress that converting the United States patent system from "first to invent" to a system of "first inventor to file" will improve the United States patent system and promote harmonization of the United States patent system with the patent systems commonly used in nearly all other countries throughout the world with whom the United States conducts trade and thereby promote greater international uniformity and certainty in the procedures used for securing the exclusive rights of inventors to their discoveries.

SEC. 6. POST-GRANT REVIEW PROCEEDINGS.

(a) INTER PARTES REVIEW.--Chapter 31 of title 35, United States Code, is amended to read as follows:

"CHAPTER 31--INTER PARTES REVIEW

"Sec.

"311. Inter partes review.

"312. Petitions.

"313. Preliminary response to petition.

"314. Institution of inter partes review.

"315. Relation to other proceedings or actions.

"316. Conduct of inter partes review.

"317. Settlement.

"318. Decision of the Board.

"319. Appeal.

"§ 311. Inter partes review

"(a) IN GENERAL.-- Subject to the provisions of this chapter, a person who is not the owner of a patent may file with the Office a petition to institute an inter partes review of the patent. The Director shall establish, by regulation, fees to be paid by the person requesting the review, in such amounts as the Director determines to be reasonable, considering the aggregate costs of the review.

"(b) SCOPE.-- A petitioner in an inter partes review may request to cancel as unpatentable 1 or more claims of a patent only on a ground that could be raised under section 102 or 103 and only on the basis of prior art consisting of patents or printed publications.

"(c) FILING DEADLINE.-- A petition for inter partes review shall be filed after the later of either--

"(1) the date that is 9 months after the grant of a patent or issuance of a reissue of a patent; or

"(2) if a post-grant review is instituted under chapter 32, the date of the termination of such post-grant review.

"§ 312. Petitions

"(a) REQUIREMENTS OF PETITION.-- A petition filed under section 311 may be considered only if--

"(1) the petition is accompanied by payment of the fee established by the Director under section 311;

"(2) the petition identifies all real parties in interest;

"(3) the petition identifies, in writing and with particularity, each claim challenged, the grounds on which the challenge to each claim is based, and the evidence that supports the grounds for the challenge to each claim, including--

"(A) copies of patents and printed publications that the petitioner relies upon in support of the petition; and

"(B) affidavits or declarations of supporting evidence and opinions, if the petitioner relies on expert opinions;

"(4) the petition provides such other information as the Director may require by regulation; and

"(5) the petitioner provides copies of any of the documents required under paragraphs (2), (3), and (4) to the patent owner or, if applicable, the designated representative of the patent owner.

"(b) PUBLIC AVAILABILITY.--As soon as practicable after the receipt of a petition under section 311, the Director shall make the petition available to the public.

"§ 313.

Preliminary response to petition

"If an inter partes review petition is filed under section 311, the patent owner shall have the right to file a preliminary response to the petition, within a time period set by the Director, that sets forth reasons why no inter partes review should be instituted based upon the failure of the petition to meet any requirement of this chapter.

"§ 314. Institution of inter partes review

"(a) THRESHOLD.-- The Director may not authorize an inter partes review to be instituted unless the Director determines that the information presented in the petition filed under section 311 and any response filed under section 313 shows that there is a reasonable likelihood that the petitioner would prevail with respect to at least 1 of the claims challenged in the petition.

"(b) TIMING.--The Director shall determine whether to institute an inter partes review under this chapter pursuant to a petition filed under section 311 within 3 months after--

"(1) receiving a preliminary response to the petition under section 313; or

"(2) if no such preliminary response is filed, the last date on which such response may be filed.

"(c) NOTICE.-- The Director shall notify the petitioner and patent owner, in writing, of the Director's determination under subsection (a), and shall make such notice available to the public as soon as is practicable. Such notice shall include the date on which the review shall commence.

"(d) NO APPEAL.-- The determination by the Director whether to institute an inter partes review under this section shall be final and nonappealable.

"§ 315. Relation to other proceedings or actions

"(a) INFRINGER'S CIVIL ACTION.—

"(1) INTER PARTES REVIEW BARRED BY CIVIL ACTION.-- An inter partes review may not be instituted if, before the date on which the petition for such a review is filed, the petitioner or real party in interest filed a civil action challenging the validity of a claim of the patent.

"(2) STAY OF CIVIL ACTION.-- If the petitioner or real party in interest files a civil action challenging the validity of a claim of the patent on or after

the date on which the petitioner files a petition for inter partes review of the patent, that civil action shall be automatically stayed until either--

“(A) the patent owner moves the court to lift the stay;

“(B) the patent owner files a civil action or counterclaim alleging that the petitioner or real party in interest has infringed the patent; or

“(C) the petitioner or real party in interest moves the court to dismiss the civil action.

“(3) TREATMENT OF COUNTERCLAIM.--A counterclaim challenging the validity of a claim of a patent does not constitute a civil action challenging the validity of a claim of a patent for purposes of this subsection.

“(b) PATENT OWNER'S ACTION.--An inter partes review may not be instituted if the petition requesting the proceeding is filed more than 1 year after the date on which the petitioner, real party in interest, or privy of the petitioner is served with a complaint alleging infringement of the patent. The time limitation set forth in the preceding sentence shall not apply to a request for joinder under subsection (c).

“(c) JOINDER.-- If the Director institutes an inter partes review, the Director, in his or her discretion, may join as a party to that inter partes review any person who properly files a petition under section 311 that the Director, after receiving a preliminary response under section 313 or the expiration of the time for filing such a response, determines warrants the institution of an inter partes review under section 314.

“(d) MULTIPLE PROCEEDINGS.--Notwithstanding sections 135(a), 251, and 252, and chapter 30, during the pendency of an inter partes review, if another proceeding or matter involving the patent is before the Office, the Director may determine the manner in which the inter partes review or other proceeding or matter may proceed, including providing for stay, transfer, consolidation, or termination of any such matter or proceeding.

“(e) ESTOPPEL.--

“(1) PROCEEDINGS BEFORE THE OFFICE.-- The petitioner in an inter partes review of a claim in a patent under this chapter that results in a final written decision under section 318(a), or the real party in interest or privy of

the petitioner, may not request or maintain a proceeding before the Office with respect to that claim on any ground that the petitioner raised or reasonably could have raised during that inter partes review.

"(2) CIVIL ACTIONS AND OTHER PROCEEDINGS.-- The petitioner in an inter partes review of a claim in a patent under this chapter that results in a final written decision under section 318(a), or the real party in interest or privy of the petitioner, may not assert either in a civil action arising in whole or in part under section 1338 of title 28 or in a proceeding before the International Trade Commission under section 337 of the Tariff Act of 1930 that the claim is invalid on any [*302] ground that the petitioner raised or reasonably could have raised during that inter partes review.

"§ 316. Conduct of inter partes review

"(a) REGULATIONS.-- The Director shall prescribe regulations--

"(1) providing that the file of any proceeding under this chapter shall be made available to the public, except that any petition or document filed with the intent that it be sealed shall, if accompanied by a motion to seal, be treated as sealed pending the outcome of the ruling on the motion;

"(2) setting forth the standards for the showing of sufficient grounds to institute a review under section 314(a);

"(3) establishing procedures for the submission of supplemental information after the petition is filed;

"(4) establishing and governing inter partes review under this chapter and the relationship of such review to other proceedings under this title;

"(5) setting forth standards and procedures for discovery of relevant evidence, including that such discovery shall be limited to--

"(A) the deposition of witnesses submitting affidavits or declarations; and

"(B) what is otherwise necessary in the interest of justice;

"(6) prescribing sanctions for abuse of discovery, abuse of process, or any other improper use of the proceeding, such as to harass or to cause unnecessary delay or an unnecessary increase in the cost of the proceeding;

"(7) providing for protective orders governing the exchange and submission of confidential information;

"(8) providing for the filing by the patent owner of a response to the petition under section 313 after an inter partes review has been instituted, and requiring that the patent owner file with such response, through affidavits or declarations, any additional factual evidence and expert opinions on which the patent owner relies in support of the response;

"(9) setting forth standards and procedures for allowing the patent owner to move to amend the patent under subsection (d) to cancel a challenged claim or propose a reasonable number of substitute claims, and ensuring that any information submitted by the patent owner in support of any amendment entered under subsection (d) is made available to the public as part of the prosecution history of the patent;

"(10) providing either party with the right to an oral hearing as part of the proceeding;

"(11) requiring that the final determination in an inter partes review be issued not later than 1 year after the date on which the Director notices the institution of a review under this chapter, except that the Director may, for good cause shown, extend the 1-year period by not more than 6 months, and may adjust the time periods in this paragraph in the case of joinder under section 315(c);

"(12) setting a time period for requesting joinder under section 315(c); and

"(13) providing the petitioner with at least 1 opportunity to file written comments within a time period established by the Director.

"(b) CONSIDERATIONS.--In prescribing regulations under this section, the Director shall consider the effect of any such regulation on the economy, the integrity of the patent system, the efficient administration of the Office, and the ability of the Office to timely complete proceedings instituted under this chapter.

"(c) PATENT TRIAL AND APPEAL BOARD.-- The Patent Trial and Appeal Board shall, in accordance with section 6, conduct each inter partes review instituted under this chapter.

"(d) AMENDMENT OF THE PATENT.--

"(1) IN GENERAL.-- During an inter partes review instituted under this chapter, the patent owner may file 1 motion to amend the patent in 1 or more of the following ways:

"(A) Cancel any challenged patent claim.

"(B) For each challenged claim, propose a reasonable number of substitute claims.

"(2) ADDITIONAL MOTIONS.-- Additional motions to amend may be permitted upon the joint request of the petitioner and the patent owner to materially advance the settlement of a proceeding under section 317, or as permitted by regulations prescribed by the Director.

"(3) SCOPE OF CLAIMS.-- An amendment under this subsection may not enlarge the scope of the claims of the patent or introduce new matter.

"(e) EVIDENTIARY STANDARDS.-- In an inter partes review instituted under this chapter, the petitioner shall have the burden of proving a proposition of unpatentability by a preponderance of the evidence.

"§ 317. Settlement

"(a) IN GENERAL.-- An inter partes review instituted under this chapter shall be terminated with respect to any petitioner upon the joint request of the petitioner and the patent owner, unless the Office has decided the merits of the proceeding before the request for termination is filed. If the inter partes review is terminated with respect to a petitioner under this section, no estoppel under section 315(e) shall attach to the petitioner, or to the real party in interest or privy of the petitioner, on the basis of that petitioner's institution of that inter partes review. If no petitioner remains in the inter partes review, the Office may terminate the review or proceed to a final written decision under section 318(a).

"(b) AGREEMENTS IN WRITING.--Any agreement or understanding between the patent owner and a petitioner, including any collateral agreements referred to in such agreement or understanding, made in connection with, or in contemplation of, the termination of an inter partes review under this section shall be in writing and a true copy of such agreement or understanding shall be filed in the Office before the termination of the inter partes review as between the parties. At the request of a party to the proceeding, the agreement or understanding shall be treated

as business confidential information, shall be kept separate from the file of the involved patents, and shall be made available only to Federal Government agencies on written request, or to any person on a showing of good cause.

"§ 318. Decision of the Board

"(a) FINAL WRITTEN DECISION.--If an inter partes review is instituted and not dismissed under this chapter, the Patent Trial [*304] and Appeal Board shall issue a final written decision with respect to the patentability of any patent claim challenged by the petitioner and any new claim added under section 316(d).

"(b) CERTIFICATE.-- If the Patent Trial and Appeal Board issues a final written decision under subsection (a) and the time for appeal has expired or any appeal has terminated, the Director shall issue and publish a certificate canceling any claim of the patent finally determined to be unpatentable, confirming any claim of the patent determined to be patentable, and incorporating in the patent by operation of the certificate any new or amended claim determined to be patentable.

"(c) INTERVENING RIGHTS.-- Any proposed amended or new claim determined to be patentable and incorporated into a patent following an inter partes review under this chapter shall have the same effect as that specified in section 252 for reissued patents on the right of any person who made, purchased, or used within the United States, or imported into the United States, anything patented by such proposed amended or new claim, or who made substantial preparation therefor, before the issuance of a certificate under subsection (b).

"(d) DATA ON LENGTH OF REVIEW.-- The Office shall make available to the public data describing the length of time between the institution of, and the issuance of a final written decision under subsection (a) for, each inter partes review.

"§ 319. Appeal

"A party dissatisfied with the final written decision of the Patent Trial and Appeal Board under section 318(a) may appeal the decision pursuant to sections 141 through 144. Any party to the inter partes review shall have the right to be a party to the appeal.".

(b) CONFORMING AMENDMENT.-- The table of chapters for part III of title 35, United States Code, is amended by striking the item relating to chapter 31 and inserting the following:

"31. Inter Partes Review .....… 311".

(c) REGULATIONS AND EFFECTIVE DATE.--

(1) REGULATIONS.-- The Director shall, not later than the date that is 1 year after the date of the enactment of this Act, issue regulations to carry out chapter 31 of title 35, United States Code, as amended by subsection (a) of this section.

(2) APPLICABILITY.----

(A) IN GENERAL.-- The amendments made by subsection (a) shall take effect upon the expiration of the 1-year period beginning on the date of the enactment of this Act and shall apply to any patent issued before, on, or after that effective date.

(B) GRADUATED IMPLEMENTATION.-- The Director may impose a limit on the number of inter partes reviews that may be instituted under chapter 31 of title 35, United States Code, during each of the first 4 1-year periods in which the amendments made by subsection (a) are in effect, if such number in each year equals or exceeds the number of inter partes reexaminations that are ordered under chapter 31 of title 35, United States Code, in the last fiscal year ending before the effective date of the amendments made by subsection (a).

(3) TRANSITION.----

(A) IN GENERAL.--Chapter 31 of title 35, United States Code, is amended--

(i) in section 312--

(I) in subsection (a)--

(aa) in the first sentence, by striking "a substantial new question of patentability affecting any claim of the patent concerned is raised by the request," and inserting "the information presented in the

request shows that there is a reasonable likelihood that the requester would prevail with respect to at least 1 of the claims challenged in the request,"; and

(bb) in the second sentence, by striking "The existence of a substantial new question of patentability" and inserting "A showing that there is a reasonable likelihood that the requester would prevail with respect to at least 1 of the claims challenged in the request"; and

(II) in subsection (c), in the second sentence, by striking "no substantial new question of patentability has been raised," and inserting "the showing required by subsection (a) has not been made,"; and

(ii) in section 313, by striking "a substantial new question of patentability affecting a claim of the patent is raised" and inserting "it has been shown that there is a reasonable likelihood that the requester would prevail with respect to at least 1 of the claims challenged in the request".

(B) APPLICATION.-- The amendments made by this paragraph--

(i) shall take effect on the date of the enactment of this Act; and

(ii) shall apply to requests for inter partes reexamination that are filed on or after such date of enactment, but before the effective date set forth in paragraph (2)(A) of this subsection.

(C) CONTINUED APPLICABILITY OF PRIOR PROVISIONS.-- The provisions of chapter 31 of title 35, United States Code, as amended by this paragraph, shall continue to apply to requests for inter partes reexamination that are filed before the effective date set forth in paragraph (2)(A) as if subsection (a) had not been enacted.

(d) POST-GRANT REVIEW.-- Part III of title 35, United States Code, is amended by adding at the end the following:

"CHAPTER 32--POST-GRANT REVIEW

“Sec.

“321. Post-grant review.

“322. Petitions.

“323. Preliminary response to petition.

“324. Institution of post-grant review.

“325. Relation to other proceedings or actions.

“326. Conduct of post-grant review.

“327. Settlement.

“328. Decision of the Board.

“329. Appeal.

“§ 321. Post-grant review

“(a) IN GENERAL.-- Subject to the provisions of this chapter, a person who is not the owner of a patent may file with the Office a petition to institute a post-grant review of the patent. The Director shall establish, by regulation, fees to be paid by the person requesting the review, in such amounts as the Director determines to be reasonable, considering the aggregate costs of the post-grant review.

“(b) SCOPE.-- A petitioner in a post-grant review may request to cancel as unpatentable 1 or more claims of a patent on any ground that could be raised under paragraph (2) or (3) of section 282(b) (relating to invalidity of the patent or any claim).

“(c) FILING DEADLINE.-- A petition for a post-grant review may only be filed not later than the date that is 9 months after the date of the grant of the patent or of the issuance of a reissue patent (as the case may be).

“§ 322. Petitions

“(a) REQUIREMENTS OF PETITION.-- A petition filed under section 321 may be considered only if--

"(1) the petition is accompanied by payment of the fee established by the Director under section 321;

"(2) the petition identifies all real parties in interest;

"(3) the petition identifies, in writing and with particularity, each claim challenged, the grounds on which the challenge to each claim is based, and the evidence that supports the grounds for the challenge to each claim, including--

"(A) copies of patents and printed publications that the petitioner relies upon in support of the petition; and

"(B) affidavits or declarations of supporting evidence and opinions, if the petitioner relies on other factual evidence or on expert opinions;

"(4) the petition provides such other information as the Director may require by regulation; and

"(5) the petitioner provides copies of any of the documents required under paragraphs (2), (3), and (4) to the patent owner or, if applicable, the designated representative of the patent owner.

"(b) PUBLIC AVAILABILITY.-- As soon as practicable after the receipt of a petition under section 321, the Director shall make the petition available to the public.

"§ 323. Preliminary response to petition

"If a post-grant review petition is filed under section 321, the patent owner shall have the right to file a preliminary response to the petition, within a time period set by the Director, that sets forth reasons why no post-grant review should be instituted based upon the failure of the petition to meet any requirement of this chapter.

"§ 324. Institution of post-grant review

"(a) THRESHOLD.-- The Director may not authorize a post-grant review to be instituted unless the Director determines that the information presented in the petition filed under section 321, if such information is not rebutted, would demonstrate that it is more likely than not that at least 1 of the claims challenged in the petition is unpatentable.

"(b) ADDITIONAL GROUNDS.--The determination required under subsection (a) may also be satisfied by a showing that the petition raises a novel or unsettled legal question that is important to other patents or patent applications.

"(c) TIMING.-- The Director shall determine whether to institute a post-grant review under this chapter pursuant to a petition filed under section 321 within 3 months after--

"(1) receiving a preliminary response to the petition under section 323; or

"(2) if no such preliminary response is filed, the last date on which such response may be filed.

"(d) NOTICE.-- The Director shall notify the petitioner and patent owner, in writing, of the Director's determination under subsection (a) or (b), and shall make such notice available to the public as soon as is practicable. Such notice shall include the date on which the review shall commence.

"(e) NO APPEAL.-- The determination by the Director whether to institute a post-grant review under this section shall be final and nonappealable.

"§ 325. Relation to other proceedings or actions

"(a) INFRINGER'S CIVIL ACTION.--

"(1) POST-GRANT REVIEW BARRED BY CIVIL ACTION.-- A post-grant review may not be instituted under this chapter if, before the date on which the petition for such a review is filed, the petitioner or real party in interest filed a civil action challenging the validity of a claim of the patent.

"(2) STAY OF CIVIL ACTION.-- If the petitioner or real party in interest files a civil action challenging the validity of a claim of the patent on or after the date on which the petitioner files a petition for post-grant review of the patent, that civil action shall be automatically stayed until either--

"(A) the patent owner moves the court to lift the stay;

"(B) the patent owner files a civil action or counterclaim alleging that the petitioner or real party in interest has infringed the patent; or

"(C) the petitioner or real party in interest moves the court to dismiss the civil action.

"(3) TREATMENT OF COUNTERCLAIM.-- A counterclaim challenging the validity of a claim of a patent does not constitute a civil action challenging the validity of a claim of a patent for purposes of this subsection.

"(b) PRELIMINARY INJUNCTIONS.-- If a civil action alleging infringement of a patent is filed within 3 months after the date on which the patent is granted, the court may not stay its consideration of the patent owner's motion for a preliminary injunction against infringement of the patent on the basis that a petition for post-grant review has been filed under this chapter or that such a post-grant review has been instituted under this chapter.

"(c) JOINDER.-- If more than 1 petition for a post-grant review under this chapter is properly filed against the same patent and the Director determines that more than 1 of these petitions warrants the institution of a post-grant review under section 324, the Director may consolidate such reviews into a single post-grant review.

"(d) MULTIPLE PROCEEDINGS.-- Notwithstanding sections 135(a), 251, and 252, and chapter 30, during the pendency of any post-grant [*308] review under this chapter, if another proceeding or matter involving the patent is before the Office, the Director may determine the manner in which the post-grant review or other proceeding or matter may proceed, including providing for the stay, transfer, consolidation, or termination of any such matter or proceeding. In determining whether to institute or order a proceeding under this chapter, chapter 30, or chapter 31, the Director may take into account whether, and reject the petition or request because, the same or substantially the same prior art or arguments previously were presented to the Office.

"(e) ESTOPPEL.--

" (1) PROCEEDINGS BEFORE THE OFFICE.-- The petitioner in a post-grant review of a claim in a patent under this chapter that results in a final written decision under section 328(a), or the real party in interest or privy of the petitioner, may not request or maintain a proceeding before the Office with respect to that claim on any ground that the petitioner raised or reasonably could have raised during that post-grant review.

"(2) CIVIL ACTIONS AND OTHER PROCEEDINGS.-- The petitioner in a post-grant review of a claim in a patent under this chapter that results in a final written decision under section 328(a), or the real party in interest or privy of the petitioner, may not assert either in a civil action arising in whole or in part under section 1338 of title 28 or in a proceeding before the International Trade Commission under section 337 of the Tariff Act of 1930 that the claim is invalid on any ground that the petitioner raised or reasonably could have raised during that post-grant review.

"(f) REISSUE PATENTS.-- A post-grant review may not be instituted under this chapter if the petition requests cancellation of a claim in a reissue patent that is identical to or narrower than a claim in the original patent from which the reissue patent was issued, and the time limitations in section 321(c) would bar filing a petition for a post-grant review for such original patent.

"§ 326. Conduct of post-grant review

"(a) REGULATIONS.-- The Director shall prescribe regulations--

"(1) providing that the file of any proceeding under this chapter shall be made available to the public, except that any petition or document filed with the intent that it be sealed shall, if accompanied by a motion to seal, be treated as sealed pending the outcome of the ruling on the motion;

"(2) setting forth the standards for the showing of sufficient grounds to institute a review under subsections (a) and (b) of section 324;

"(3) establishing procedures for the submission of supplemental information after the petition is filed;

"(4) establishing and governing a post-grant review under this chapter and the relationship of such review to other proceedings under this title;

"(5) setting forth standards and procedures for discovery of relevant evidence, including that such discovery shall be limited to evidence directly related to factual assertions advanced by either party in the proceeding;

"(6) prescribing sanctions for abuse of discovery, abuse of process, or any other improper use of the proceeding, such [*309] as to harass or to cause unnecessary delay or an unnecessary increase in the cost of the proceeding;

"(7) providing for protective orders governing the exchange and submission of confidential information;

"(8) providing for the filing by the patent owner of a response to the petition under section 323 after a post-grant review has been instituted, and requiring that the patent owner file with such response, through affidavits or declarations, any additional factual evidence and expert opinions on which the patent owner relies in support of the response;

"(9) setting forth standards and procedures for allowing the patent owner to move to amend the patent under subsection (d) to cancel a challenged claim or propose a reasonable number of substitute claims, and ensuring that any information submitted by the patent owner in support of any amendment entered under subsection (d) is made available to the public as part of the prosecution history of the patent;

"(10) providing either party with the right to an oral hearing as part of the proceeding;

"(11) requiring that the final determination in any post-grant review be issued not later than 1 year after the date on which the Director notices the institution of a proceeding under this chapter, except that the Director may, for good cause shown, extend the 1-year period by not more than 6 months, and may adjust the time periods in this paragraph in the case of joinder under section 325(c); and

"(12) providing the petitioner with at least 1 opportunity to file written comments within a time period established by the Director.

"(b) CONSIDERATIONS.-- In prescribing regulations under this section, the Director shall consider the effect of any such regulation on the economy, the integrity of the patent system, the efficient administration of the Office, and the ability of the Office to timely complete proceedings instituted under this chapter.

"(c) PATENT TRIAL AND APPEAL BOARD.-- The Patent Trial and Appeal Board shall, in accordance with section 6, conduct each post-grant review instituted under this chapter.

"(d) AMENDMENT OF THE PATENT.--

"(1) IN GENERAL.-- During a post-grant review instituted under this chapter, the patent owner may file 1 motion to amend the patent in 1 or more of the following ways:

"(A) Cancel any challenged patent claim.

"(B) For each challenged claim, propose a reasonable number of substitute claims.

"(2) ADDITIONAL MOTIONS.-- Additional motions to amend may be permitted upon the joint request of the petitioner and the patent owner to materially advance the settlement of a proceeding under section 327, or upon the request of the patent owner for good cause shown.

"(3) SCOPE OF CLAIMS.-- An amendment under this subsection may not enlarge the scope of the claims of the patent or introduce new matter.

"(e) EVIDENTIARY STANDARDS.-- In a post-grant review instituted under this chapter, the petitioner shall have the burden of proving a proposition of unpatentability by a preponderance of the evidence.

"§ 327. Settlement

"(a) IN GENERAL.-- A post-grant review instituted under this chapter shall be terminated with respect to any petitioner upon the joint request of the petitioner and the patent owner, unless the Office has decided the merits of the proceeding before the request for termination is filed. If the post-grant review is terminated with respect to a petitioner under this section, no estoppel under section 325(e) shall attach to the petitioner, or to the real party in interest or privy of the petitioner, on the basis of that petitioner's institution of that post-grant review. If no petitioner remains in the post-grant review, the Office may terminate the post-grant review or proceed to a final written decision under section 328(a).

"(b) AGREEMENTS IN WRITING.-- Any agreement or understanding between the patent owner and a petitioner, including any collateral agreements referred to in such agreement or understanding, made in connection with, or in contemplation of, the termination of a post-grant review under this section shall be in writing, and a true copy of such agreement or understanding shall be filed in the Office before the termination of the post-grant review as between the parties. At the request of a party to the proceeding, the agreement or understanding shall be treated as

business confidential information, shall be kept separate from the file of the involved patents, and shall be made available only to Federal Government agencies on written request, or to any person on a showing of good cause.

"§ 328. Decision of the Board

"(a) FINAL WRITTEN DECISION.-- If a post-grant review is instituted and not dismissed under this chapter, the Patent Trial and Appeal Board shall issue a final written decision with respect to the patentability of any patent claim challenged by the petitioner and any new claim added under section 326(d).

"(b) CERTIFICATE.-- If the Patent Trial and Appeal Board issues a final written decision under subsection (a) and the time for appeal has expired or any appeal has terminated, the Director shall issue and publish a certificate canceling any claim of the patent finally determined to be unpatentable, confirming any claim of the patent determined to be patentable, and incorporating in the patent by operation of the certificate any new or amended claim determined to be patentable.

"(c) INTERVENING RIGHTS.-- Any proposed amended or new claim determined to be patentable and incorporated into a patent following a post-grant review under this chapter shall have the same effect as that specified in section 252 of this title for reissued patents on the right of any person who made, purchased, or used within the United States, or imported into the United States, anything patented by such proposed amended or new claim, or who made substantial preparation therefor, before the issuance of a certificate under subsection (b).

"(d) Data on Length of Review.-- The Office shall make available to the public data describing the length of time between the institution of, and the issuance of a final written decision under subsection (a) for, each post-grant review.

"§ 329. Appeal

"A party dissatisfied with the final written decision of the Patent Trial and Appeal Board under section 328(a) may appeal the decision pursuant to sections 141 through 144. Any party to the post-grant review shall have the right to be a party to the appeal.".

(e) CONFORMING AMENDMENT.-- The table of chapters for part III of title 35, United States Code, is amended by adding at the end the following:

"32. Post-Grant Review .....… 321".

(f) REGULATIONS AND EFFECTIVE DATE.--

(1) REGULATIONS.-- The Director shall, not later than the date that is 1 year after the date of the enactment of this Act, issue regulations to carry out chapter 32 of title 35, United States Code, as added by subsection (d) of this section.

(2) APPLICABILITY.----

(A) IN GENERAL.-- The amendments made by subsection (d) shall take effect upon the expiration of the 1-year period beginning on the date of the enactment of this Act and, except as provided in section 18 and in paragraph (3), shall apply only to patents described in section 3(n)(1).

(B) LIMITATION.-- The Director may impose a limit on the number of post-grant reviews that may be instituted under chapter 32 of title 35, United States Code, during each of the first 4 1-year periods in which the amendments made by subsection (d) are in effect.

(3) PENDING INTERFERENCES.----

(A) PROCEDURES IN GENERAL.-- The Director shall determine, and include in the regulations issued under paragraph (1), the procedures under which an interference commenced before the effective date set forth in paragraph (2)(A) is to proceed, including whether such interference--

(i) is to be dismissed without prejudice to the filing of a petition for a post-grant review under chapter 32 of title 35, United States Code; or

(ii) is to proceed as if this Act had not been enacted.

(B) PROCEEDINGS BY PATENT TRIAL AND APPEAL BOARD.-- For purposes of an interference that is commenced before the effective date set forth in paragraph (2)(A), the Director may deem the Patent Trial and Appeal Board to be the Board of Patent Appeals and

Interferences, and may allow the Patent Trial and Appeal Board to conduct any further proceedings in that interference.

(C) APPEALS.-- The authorization to appeal or have remedy from derivation proceedings in sections 141(d) and 146 of title 35, United States Code, as amended by this Act, and the jurisdiction to entertain appeals from derivation proceedings in section 1295(a)(4)(A) of title 28, United States Code, as amended by this Act, shall be deemed to extend to any final decision in an interference that is commenced before the effective date set forth in paragraph (2)(A) of this subsection and that is not dismissed pursuant to this paragraph.

(g) CITATION OF PRIOR ART AND WRITTEN STATEMENTS.--

(1) IN GENERAL.-- Section 301 of title 35, United States Code, is amended to read as follows:

"§ 301. Citation of prior art and written statements

"(a) IN GENERAL.-- Any person at any time may cite to the Office in writing--

"(1) prior art consisting of patents or printed publications which that person believes to have a bearing on the patentability of any claim of a particular patent; or

"(2) statements of the patent owner filed in a proceeding before a Federal court or the Office in which the patent owner took a position on the scope of any claim of a particular patent.

"(b) OFFICIAL FILE.-- If the person citing prior art or written statements pursuant to subsection (a) explains in writing the pertinence and manner of applying the prior art or written statements to at least 1 claim of the patent, the citation of the prior art or written statements and the explanation thereof shall become a part of the official file of the patent.

"(c) ADDITIONAL INFORMATION.-- A party that submits a written statement pursuant to subsection (a)(2) shall include any other documents, pleadings, or evidence from the proceeding in which the statement was filed that addresses the written statement.

"(d) LIMITATIONS.-- A written statement submitted pursuant to subsection (a)(2), and additional information submitted pursuant to subsection (c), shall not be considered by the Office for any purpose other than to determine the proper meaning of a patent claim in a proceeding that is ordered or instituted pursuant to section 304, 314, or 324. If any such written statement or additional information is subject to an applicable protective order, such statement or information shall be redacted to exclude information that is subject to that order.

"(e) CONFIDENTIALITY.-- Upon the written request of the person citing prior art or written statements pursuant to subsection (a), that person's identity shall be excluded from the patent file and kept confidential.".

(2) CONFORMING AMENDMENT.-- The item relating to section 301 in the table of sections for chapter 30 of title 35, United States Code, is amended to read as follows:

"301. Citation of prior art and written statements.".

(3) EFFECTIVE DATE.-- The amendments made by this subsection shall take effect upon the expiration of the 1-year period beginning on the date of the enactment of this Act and shall apply to any patent issued before, on, or after that effective date.

(h) REEXAMINATION.—

(1) DETERMINATION BY DIRECTOR.----

(A) IN GENERAL.-- Section 303(a) of title 35, United States Code, is amended by striking "section 301 of this title" and inserting "section 301 or 302".

(B) EFFECTIVE DATE.-- The amendment made by this paragraph shall take effect upon the expiration of the 1-year period beginning on the date of the enactment of this Act and shall apply to any patent issued before, on, or after that effective date.

(2) APPEAL.----

(A) IN GENERAL.-- Section 306 of title 35, United States Code, is amended by striking “145” and inserting “144”.

(B) EFFECTIVE DATE.-- The amendment made by this paragraph shall take effect on the date of the enactment of this Act and shall apply to any appeal of a reexamination before the Board of Patent Appeals and Interferences or the Patent Trial and Appeal Board that is pending on, or brought on or after, the date of the enactment of this Act.

SEC. 18. TRANSITIONAL PROGRAM FOR COVERED BUSINESS METHOD

PATENTS.

(a) TRANSITIONAL PROGRAM.--

(1) ESTABLISHMENT.-- Not later than the date that is 1 year after the date of the enactment of this Act, the Director shall issue regulations establishing and implementing a transitional post-grant review proceeding for review of the validity of covered business method patents. The transitional proceeding implemented pursuant to this subsection shall be regarded as, and shall employ the standards and procedures of, a post-grant review under chapter 32 of title 35, United States Code, subject to the following:

(A) Section 321(c) of title 35, United States Code, and subsections (b), (e)(2), and (f) of section 325 of such title shall not apply to a transitional proceeding.

(B) A person may not file a petition for a transitional proceeding with respect to a covered business method patent unless the person or the person's real party in interest or privy has been sued for infringement of the patent or has been charged with infringement under that patent.

(C) A petitioner in a transitional proceeding who challenges the validity of 1 or more claims in a covered business method patent on a ground raised under section 102 or 103 of title 35, United States Code, as in effect on the day before the effective date set forth in section 3(n)(1), may support such ground only on the basis of--

(i) prior art that is described by section 102(a) of such title of such title (as in effect on the day before such effective date); or

(ii) prior art that--

(I) discloses the invention more than 1 year before the date of the application for patent in the United States; and

(II) would be described by section 102(a) of such title (as in effect on the day before the effective date set forth in section 3(n)(1)) if the disclosure had been made by

another before the invention thereof by the applicant for patent.

(D) The petitioner in a transitional proceeding that results in a final written decision under section 328(a) of title 35, United States Code, with respect to a claim in a covered business method patent, or the petitioner's real party in interest, may not assert, either in a civil action arising in whole or in part under section 1338 of title 28, United States Code, or in a proceeding before the International Trade Commission under section 337 of the Tariff Act of 1930 (19 U.S.C. 1337), that the claim is invalid on any ground that the petitioner raised during that transitional proceeding.

(E) The Director may institute a transitional proceeding only for a patent that is a covered business method patent.

(2) EFFECTIVE DATE.-- The regulations issued under paragraph (1) shall take effect upon the expiration of the 1-year period beginning on the date of the enactment of this Act and shall apply to any covered business method patent issued before, on, or after that effective date, except that the regulations shall not apply to a patent described in section 6(f)(2)(A) of this Act during the period in which a petition for post-grant review of that patent would satisfy the requirements of section 321(c) of title 35, United States Code.

(3) SUNSET.----

(A) IN GENERAL.-- This subsection, and the regulations issued under this subsection, are repealed effective upon the expiration of the 8-year period beginning on the date that the regulations issued under to paragraph (1) take effect.

(B) APPLICABILITY.-- Notwithstanding subparagraph (A), this subsection and the regulations issued under this subsection shall continue to apply, after the date of the repeal under subparagraph (A), to any petition for a transitional proceeding that is filed before the date of such repeal.

(b) REQUEST FOR STAY.--

(1) IN GENERAL.-- If a party seeks a stay of a civil action alleging infringement of a patent under section 281 of title 35, United States Code,

relating to a transitional proceeding for that patent, the court shall decide whether to enter a stay based on--

(A) whether a stay, or the denial thereof, will simplify the issues in question and streamline the trial;

(B) whether discovery is complete and whether a trial date has been set;

(C) whether a stay, or the denial thereof, would unduly prejudice the nonmoving party or present a clear tactical advantage for the moving party; and

(D) whether a stay, or the denial thereof, will reduce the burden of litigation on the parties and on the court.

(2) REVIEW.-- A party may take an immediate interlocutory appeal from a district court's decision under paragraph (1). The United States Court of Appeals for the Federal Circuit shall review the district court's decision to ensure consistent application of established precedent, and such review may be de novo.

(c) ATM EXEMPTION FOR VENUE PURPOSES.-- In an action for infringement under section 281 of title 35, United States Code, of a covered business method patent, an automated teller machine shall not be deemed to be a regular and established place of business for purposes of section 1400(b) of title 28, United States Code.

(d) DEFINITION.--

(1) IN GENERAL.-- For purposes of this section, the term “covered business method patent” means a patent that claims a method or corresponding apparatus for performing data processing or other operations used in the practice, administration, or management of a financial product or service, except that the term does not include patents for technological inventions.

(2) REGULATIONS.-- To assist in implementing the transitional proceeding authorized by this subsection, the Director shall issue regulations for determining whether a patent is for a technological invention.

(e) RULE OF CONSTRUCTION.-- Nothing in this section shall be construed as amending or interpreting categories of patent-eligible subject matter set forth under section 101 of title 35, United States Code.

CERTIFICATE OF COMPLIANCE

This brief complies with the type-volume limitations of Federal Circuit Rule 32(b) because it contains 13,002 words, excluding the parts of the brief exempted by Fed. R. App. P. 32(f) and Federal Circuit Rule 32(b)(2), as determined by the word-counting feature of Microsoft Word.

This brief complies with the typeface requirement of Fed. R. App. P. 32(a)(5) and the type-style requirements of Fed. R. App. P. 32(a)(6) because it has been prepared in a proportionally spaced typeface, including serifs, using Microsoft Word 2016 in Times New Roman 14-point font.

Dated: October 21, 2022 /s/ Deanne E. Maynard

NY:2411570