

Patent Searching for Engineers and Scientists

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February 10, 2012

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Outline

- Brief Discussion about U.S. Patents
- 7-Step Patent Search Strategy
- USPTO Patent Full-Text and Full-Image Database
 - Demonstrate the 7-Step Patent Search Strategy
 - Search by patent number
 - Accessing the full-text of the patent
- Google Patents
- Using Kirk-Othmer and Ullmann's for Patents

What is a Patent?

“A patent is a grant from a government that confers upon an inventor the right to exclude others from making, using, selling, importing, or offering an invention for sale for a fixed period of time.”

(Pressman, 2009, p. 9)

Types of Patents:

Utility - Most common type. New and useful process, machine, article of manufacture, compositions, or any new and useful improvement.

Design - New, original, and ornamental design for an article of manufacture.

Plant – Plants that can be reproduced through cuttings or grafting.

Pressman, D. (2009). Patent it yourself: Your step-by-step guide to filing at the U.S. patent office. Berkeley, CA: Nolo.

Legal Requirements for a Utility Patent

- Must fit one of the five statutory classes
 - processes, machine, articles of manufacture, compositions of matter, and “new uses” of the above.
- Useful
- Novel
- Non-obvious

Where Would You Encounter Patents?

- As citations in the scientific or technical literature
- When searching the “prior art” for a patent application
- Evaluate the state-of-the-art of an industry or intellectual property of a company

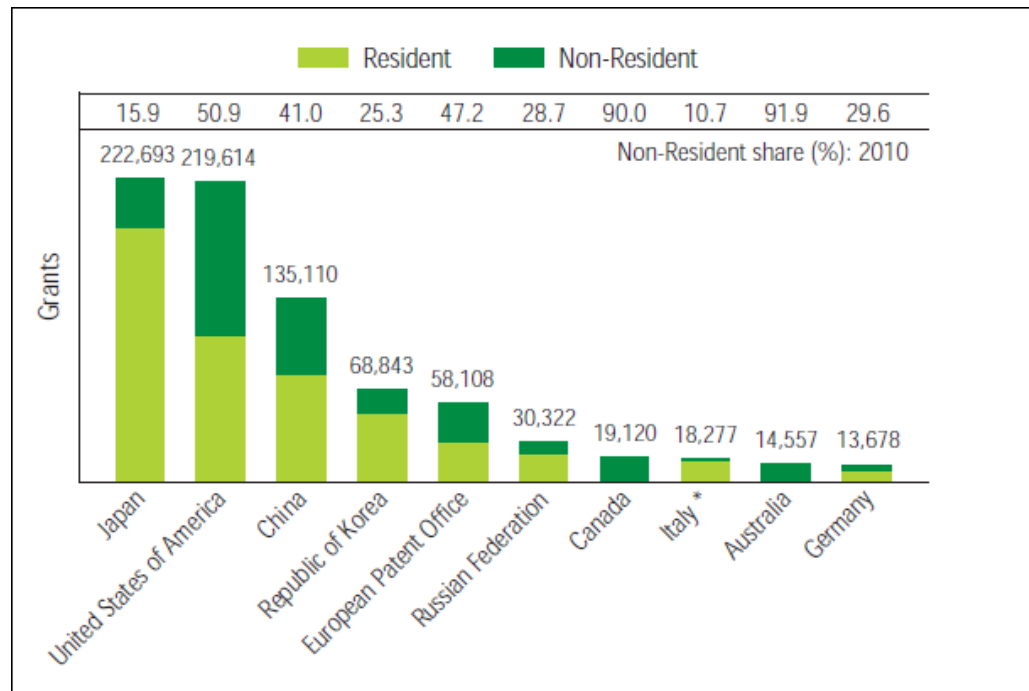
Why Search the Patent Literature?

- Technical Information
 - Find solutions to technical problems
 - Locate information that isn't published in journals
- Legal Information
 - Prepare a patent application
 - Avoid patent infringement
- Business Information
 - Identify key innovators/companies
 - Identify and monitor technology trends

Patent Statistics

- Over 8,100,000 U.S. patents issued since 1836
- U.S. and Japanese account for 48% of in force patents

Patents granted at the top 10 offices, 2010



Source: Data from 2010: World Intellectual Property Organization. (2011). *World Intellectual Property Indicators*. Retrieved February 6, 2012, from http://www.wipo.int/export/sites/www/freepublications/en/intproperty/941/wipo_pub_941_2011.pdf.

The 7-Step Patent Search Strategy

Classification

1. Brainstorm keywords related to the purpose, use and composition of the invention.
2. Look up the words in the [Index to the U.S. Patent Classification](#) to find potential class/subclasses.
3. Verify the relevancy of the class/subclasses by using the [Classification Schedule](#) in the [Manual of Classification](#).
4. Read the [Classification Definitions](#) to verify the scope of the subclasses and note "see also" references.

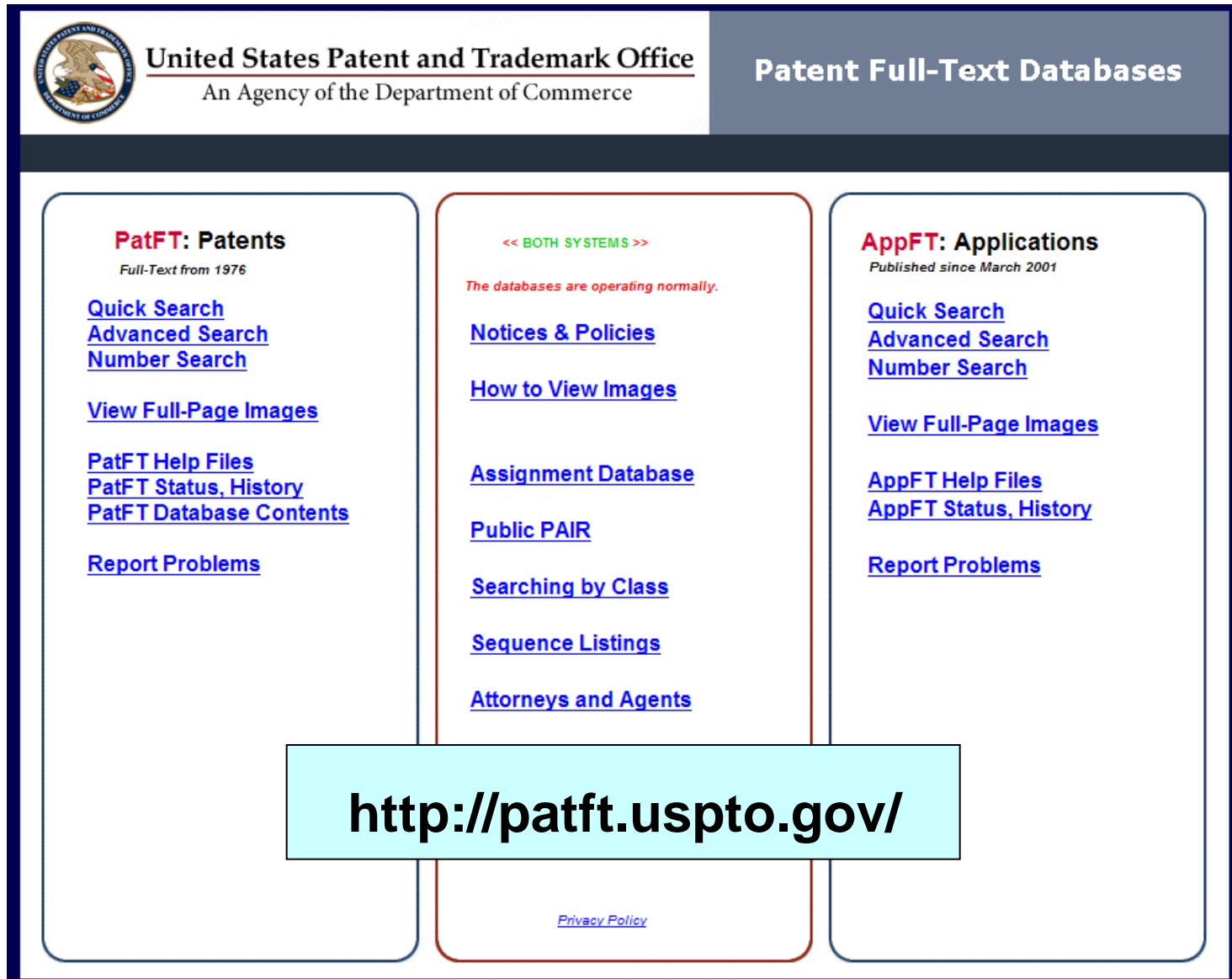
Access Full-Text

5. Search the [Issued Patents](#) and the [Published Applications](#) databases by "Current US Classification" and access full text patents and published applications.


Review and References

6. Review the claims, specifications and drawings of documents retrieved for relevancy.
7. Check all references and note the "U.S. Cl." and "Field of Search" areas for additional class/subclasses to search.

Accessing the USPTO Patent Full-Text and Image Database



The screenshot shows the USPTO Patent Full-Text Databases website. At the top left is the USPTO logo. The header includes the text "United States Patent and Trademark Office" and "An Agency of the Department of Commerce". The main title is "Patent Full-Text Databases". The page is divided into three columns. The left column is for "PatFT: Patents" (Full-Text from 1976) and includes links for Quick Search, Advanced Search, Number Search, View Full-Page Images, PatFT Help Files, PatFT Status, History, PatFT Database Contents, and Report Problems. The middle column is for "<< BOTH SYSTEMS >>" and includes a status message "The databases are operating normally." and links for Notices & Policies, How to View Images, Assignment Database, Public PAIR, Searching by Class, Sequence Listings, and Attorneys and Agents. The right column is for "AppFT: Applications" (Published since March 2001) and includes links for Quick Search, Advanced Search, Number Search, View Full-Page Images, AppFT Help Files, AppFT Status, History, and Report Problems. A large light blue box at the bottom center contains the URL "http://patft.uspto.gov/". A "Privacy Policy" link is located at the bottom center of the page.

 **United States Patent and Trademark Office**
An Agency of the Department of Commerce

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Searching by the U.S. Patent Classification System

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Click "Searching by Class"

Step 2 - Use the U.S. Patent Classification Index

A. Access Classification Info by Class/Subclass [HELP](#)

1. Enter a US Patent Classification...

 /

Class (required)/Subclass (optional)
e.g., 704/1 or 482/1

2. Select what you want...

- Class Schedule (HTML)
- Printable Version of Class Schedule (PDF)
- Class Definition (HTML)
- Printable Version of Class Definition (PDF)
- US-to-IPC8 Concordance (HTML)
- US-to-IPC8 Concordance (PDF)
- US-to-Locarno Concordance

3.

B. Classification Information

1. Index to the U.S. Patent Classification System ([Preface](#))

A B C D E F G H I J K L M N O P Q R S T U V W X Y Z

2. [Classification Orders](#)
3. [Classification Orders Index](#)
4. [Classes Under Reclaim](#)
5. [Classes Within the U.S. Classification System \(Arranged by Related Subjects\)](#)
6. [Classes Arranged Numerically With Art Unit and Search Room Locations](#)
7. [Classes Arranged in Alphabetical Order](#)
8. [Classes Arranged by Art Unit](#)
9. [Information on E-Subclasses](#)

Select the USPC Index

C. Search USPTO

Look in: Index to Classification ▾
Search for:

Don't use this search

D. Search USA.Gov (General Search and Advanced Search)

1. Click the Search USA.gov logo below to initiate a general search at USA.gov:



2. Click the Advanced Search USA.gov link below to initiate an advanced search at USA.gov:

[Advanced Search USA.gov](#)



Index to the United States Patent Classification (USPC) System

Select the format and section (by letter) ...

Index in HTML

A B C D E F G H I J K L M N O P Q R S T U V W X Y Z

Index in PDF

A B C D E F G H I J K L M N O P Q R S T U V W X Y Z

KEY: =online business system =fees =forms =help =laws/regulations =definition (glossary)

The Inventors Assistance Center is available to help you on patent matters. Send questions about USPTO programs and services to the USPTO Contact Center (UCC). You can suggest USPTO webpages or material you would like featured on this section by E-mail to the webmaster@uspto.gov. While we cannot promise to accommodate all requests, your suggestions will be considered and may lead to other improvements on the website.

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Last Modified: 12/01/2009 12:58:45

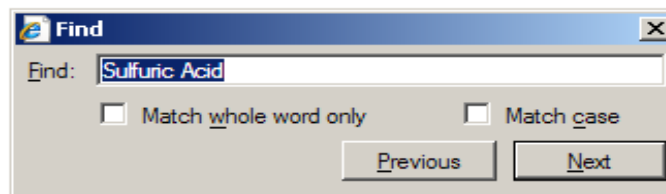
**Looking for "Sulfuric Acid"
Select the letter "S" from the Index in HTML**



A B C D E F G H I J K L M N O P Q R S T U V W X Y Z

View as PDF

Saber	30 / 165+
Sabots	102 / 517+
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Preparation from carbohydrates	562 / 515
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Fermentation	435
Saccharimeter	
Chemical analysis	422 / 68.1+
Fermentative test	435 / 4+
Hydrometer	73 / 32 R+
Optical analysis	356 / 366
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Cleaning with air	15 / 304
Closing	53 / 285+
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Filling and packing machines	53 / 570+
Holders	248 / 95+
Sewing	112
Suspensory bandage	602 / 67
Separable	602 / 68
Turners	414 / 754+
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Sacramental Receptacle	206 / 19
Saddlebags	224 / 191+



Use the Find function (Ctrl + F) to search page for "Sulfuric Acid"

Sulfur


Acids and acid anhydrides	423 / 512.1+
Electrolytic synthesis	205 / 554
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Burner	422 / 160+
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Carboxylic acids	
Acyclic	562 / 512+
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Alicyclic esters	560 / 125
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Removed from gaseous mixture	423 / 242.1+
Rendering harmless in fuel	208 / 177+
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Sulfureted hydrogen	423 / 363
Sulfuric acid	423 / 522+
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Concentrating	422 / 160+
Trains	422 / 160
Electrolytic synthesis	205 / 554
Esters	558 / 20+
Mineral oil sludge source	208 / 13

Located "Sulfuric Acid"

**Class/subclass is 423/522
Make note of the number
and write it down**

**Verify the relevancy of the
Class/subclass by clicking
on "423"**

Step 3 – Verify the Relevancy of the Class / Subclass using the Classification Schedule

 **United States Patent and Trademark Office**
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Patents > Guidance, Tools, and Manuals > Classification > Class Schedule

Class Numbers & Titles | Class Numbers Only | USPC Index | International | HELP

Class 423 CHEMISTRY OF INORGANIC COMPOUNDS

Click here for a printable version of this file

Turn Outline

Select Largest Indent Level to be Displayed

- [A](#) [P](#) 700 **ZEOLITE**
- [A](#) [P](#) 701
 - Organic compound used to form zeolite
- [A](#) [P](#) 702
 - Organic template used
- [A](#) [P](#) 703
 - Mixed template
- [A](#) [P](#) 704
 - Nitrogen containing
- [A](#) [P](#) 705
 - Amine
- [A](#) [P](#) 706
 - Cyclic
- [A](#) [P](#) 707
 - Hydroxyl
- [A](#) [P](#) 708
 - Diamine
- [A](#) [P](#) 709
 - Seed used
- [A](#) [P](#) 710
 - Aging to induce zeolite formation from inorganic mixture
- [A](#) [P](#) 711
 - With physical treatment
- [A](#) [P](#) 712
 - Synthesized from naturally occurring product
- [A](#) [P](#) 713
 - Isomorphic metal substitution
- [A](#) [P](#) 714
 - Acid treatment
- [A](#) [P](#) 715
 - Halogen containing acid
- [A](#) [P](#) 716
 - With change of synthesized zeolite morphology
- [A](#) [P](#) 717
 - Physical treatment
- [A](#) [P](#) 718
 - Structure defined X-ray diffraction pattern
- [A](#) [P](#) 1 **TREATING MIXTURE TO OBTAIN METAL CONTAINING COMPOUND**

This is the Classification Schedule for Class 423 Chemistry of Inorganic Compounds

Clicking on "Class 423" would provide the Classification Definition

Class Schedule – Only Small Portions are Hierarchical

	A	P		
-	A	P	511	SULFUR OR COMPOUND THEREOF
-	A	P	512.1	· Oxygen containing
-	A	P	513	.. Persulfate
-	A	P	514	.. Thiosulfate
-	A	P	515	.. Dithionite
-	A	P	516	... Employing amalgam as reactant
-	A	P	517	.. Metal and ammonium containing
-	A	P	518	.. Plural metal containing
-	A	P	519	.. Bisulfite
-	A	P	519.2	.. Sulfite
-	A	P	520	... Bisulfate
-	A	P	521	.. Ternary compound containing hydrogen
-	A	P	522	... Sulfuric acid
-	A	P	523 Nitrogenous impurity or utilizing nitrogenous catalyst or reactant
-	A	P	524 Lead chamber process
-	A	P	525 Starting material includes organic or carbonaceous impurity
-	A	P	526 Utilizing pressure or vacuum on mixture
-	A	P	527 Adding organic compound to mixture
-	A	P	528 Contacting mixture with gas, steam, or vapor
-	A	P	529 Subjecting reactants to pressure, vacuum, or steam
-	A	P	530 Utilizing metal sulfate
-	A	P	531 Purifying acid or reactant
-	A	P	532	.. Sulfur trioxide
-	A	P	533	... Utilizing catalyst in reaction
-	A	P	534 Promoter or successive diverse catalysts
-	A	P	535 Catalyst contains oxygen, vanadium, and another metal
-	A	P	536 Platinum catalyst
-	A	P	537 With sulfate or asbestos carrier
-	A	P	538 Oxygen containing catalyst
-	A	P	539	.. Sulfur dioxide

Scanning down the schedule we find "Sulfuric Acid" by following the schedule hierarchy from least specific to most specific

Check the definition for subclass "522" by clicking the link

Step 4 – Read the Classification Definitions and Verify Scope

519 Bisulfite

This subclass is indented under subclass 512.1. Products or processes wherein the compound contains the bisulfite or acid sulfite radical (HSO_3^-).

519.2 Sulfite:

This subclass is indented under subclass 512.1. Products and processes wherein the compound is a sulfite; i.e., includes a (SO_3^{2-}) radical.

520 Bisulfate

This subclass is indented under subclass 512.1. Products or processes wherein the compound is a bisulfate or acid sulfate radical (SO_4^{2-}).

Definition for 423/522

521 Ternary compound containing hydrogen

This subclass is indented under subclass 512.1. Products or processes in which the compound is ternary and consists of sulfur, oxygen and hydrogen only.

522 Sulfuric acid

This subclass is indented under subclass 521. Products or processes in which the ternary compound is sulfuric acid (H_2SO_4).

(1) Note. This subclass provides for sulfuric acid (H_2SO_4) including fuming sulfuric acid which is sometimes termed oleum or Nordhausen acid for which sometimes the formula $\text{H}_2\text{S}_2\text{O}_7$ may be designated. However, this compound is actually H_2SO_4 with SO_3 dissolved therein and is classified in this subclass.

523 Nitrogenous impurity or utilizing nitrogenous catalyst or reactant

This subclass is indented under subclass 522. Processes in which during the manufacture of the sulfuric acid a compound of nitrogen is used as a catalyst or as a reactant, or is present as an impurity which requires removal.

524

Click the red P link to see all the patents for a particular subclass

Sulfuric acid is produced from sulfur dioxide oxygen (air) and water or steam by means of nitrogen oxides

on of sulfur dioxide by burning sulfur or sulfur compounds. Sulfuric acid is produced in the lead chamber means of nitrogen oxides (catalysts or reactants). The gases leaving the reaction chamber contain practically the oxidation of SC_2 to SO_3 . These nitrogen oxides are recovered by absorption in H_2SO_4 in so called towers is conveyed to the beginning of the system where it is denitrated in so called Glover towers by

means of the entering hot gases containing sulfur dioxide.

Step 5 – Search Issued Patents and Access Full-Text

USPTO PATENT FULL-TEXT AND IMAGE DATABASE

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Searching US Patent Collection...

Results of Search in US Patent Collection db for:

CCL/423/522: 440 patents.

Hits 1 through 50 out of 440

Next 50 Hits

Jump To

Refine Search

CCL/423/522

Using the "Refine Search" we could narrow our search by adding keywords.

PAT. NO. Title

- 1 [7,632,479](#) [Process for producing ammonia and sulfuric acid from a stream comprising ammonium sulfate](#)
- 2 [7,632,475](#) [Process for removing contaminants from gas streams](#)
- 3 [7,595,035](#) [Process for the recovery of sulfuric acid](#)
- 4 [7,582,271](#) [Emission control system](#)
- 5 [7,455,819](#) [Apparatus for simultaneous dry desulfurization/denitrification](#)
- 6 [7,452,521](#) [Method for the removal of mercury from sulphuric acid with thiosulphate precipitation](#)
- 7 [7,442,363](#) [Hydrogen iodide manufacturing method and hydrogen iodide manufacturing apparatus](#)
- 8 [7,442,359](#) [Recovery of sulphuric acid](#)
- 9 [7,442,352](#) [Flue gas purification process using a sorbent polymer composite material](#)
- 10 [7,416,716](#) [Purification of carbon dioxide](#)
- 11 [7,404,938](#) [Emission control system](#)
- 12 [7,361,326](#) [Process for the production of sulfuric acid](#)


Clicking on these links will take you to the full-text of the patent

A Note about Accessing and Viewing Patents

Refine Search

CCL/423/522

PAT. NO. Title

251	3,933,991	 Sulfuric acid contact process
252	3,932,599	 Method of obtaining hydrogen from steam
253	3,929,972	 Production of silico-dihydrogen sulfate
254	3,923,964	 Process for the production of calcium fluoride from fluosilicates and by-product gypsum
255	3,920,421	 Removal of oxides of nitrogen from gas streams which also contain sulfur dioxide
256	3,919,402	 Petroleum oil desulfurization process
257	3,917,798	 SO.sub.2 abatement
258	3,917,519	 Process for the manufacture of electrolytic copper
259	3,914,398	 Process for making anhydrous hydrogen fluoride from fluosilicic acid
260	3,909,211	 Coal desulfurization process
261	3,907,979	 Low SO.sub.2 emission sulfuric acid process form sulfur and oxygen
262	3,898,320	 Dry absorbent composition and process for making the same
263	3,897,545	 Process for catalytically reacting gases having a high SO.sub.2 content using different catalysts
264	3,880,985	 PROCESS FOR PRODUCTION OF SULPHUR TRIOXIDE
265	3,875,294	 Process for catalytically reacting gases having a high sulfur dioxide content
266	3,873,674	 Conversion of sulfur dioxide to sulfur trioxide by peroxytitanium complexes
267	3,862,298	 PROCESS FOR THE TREATMENT OF A SALT-CONTAINING ACID SOLUTION
268	3,862,295	 METHOD FOR SORPTIVE REMOVAL OF SULFUR GASES
269	3,853,502	 METHOD OF REMOVING SO AND H SO MIST FROM A GAS STREAM
270	3,836,630	 423/243.03 423/166 423/243.07 423/522 423/555
271	3,829,560	 RECOVERY OF SULFUR DIOXIDE FROM GAS STREAMS
272	3,825,657	 423/540 423/351 423/437.1 423/522
273	3,819,816	 423/522 423/224 423/234 423/238 423/574.1

A patent displaying a "T" icon has the full-text available for viewing. A patent displaying the "picture" icon only has an image (TIFF file) available for viewing.

This 1974 patent is only available as an image (TIFF file). You must install a TIFF viewer to view these images.

Results of Clicking the Class / Subclass Search

- 20 [7,041,152](#) **II** [Method for processing elemental sulfur-bearing materials using high temperature pressure leaching](#)
- 21 [7,033,565](#) **II** [Production of sulphuric acid from a feed gas with varying concentration of sulphur dioxide](#)
- 22 [7,029,639](#) **II** [Desulfurizer comprising activated carbon and method of desulfurization](#)
- 23 [6,946,108](#) **II** [Flue gas desulfurization apparatus and flue gas desulfurization system, and method for operating flue gas desulfurization apparatus](#)
- 24 [6,893,622](#) **II** [Process for the combustion of sulphur for the preparation of oleum and sulphuric acid with reduced \(NO\)_x content](#)
- 25 [6,890,371](#) **II** [Method for processing elemental sulfur-bearing materials using high temperature pressure leaching](#)
- 26 [6,872,373](#) **II** [Flue gas processing apparatus and desulfurization method](#)
- 27 [6,790,418](#) **II** [Materials and method for the biological production of sulfuric acid](#)
- 28 [6,740,302](#) **II** [Method for producing high-purity sulphuric acid](#)
- 29 [6,689,326](#) **II** [Method and apparatus for introducing sulphur dioxide into aqueous solutions](#)
- 30 [6,635,231](#) **II** [Preparation of arsenic pentafluoride](#)
- 31 [6,627,172](#) **II** [Process for preparing sulphuric acid from gases containing SO₃ and gaseous nit](#)
- 32 [6,616,905](#) **II** [Desulfurization of exhaust gases using activated carbon catalyst](#)
- 33 [6,610,268](#) **II** [Method for the microbiological production of sulfuric acid](#)
- 34 [6,610,263](#) **II** [System and process for removal of pollutants from a gas stream](#)
- 35 [6,572,835](#) **II** [Method and apparatus for producing gaseous sulfur trioxide](#)

Scan the patents resulting from the search of Class / subclass

It looks like 6,740,302 might be worth investigating

Reading a Patent

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United States Patent
Hostalek, et al.

Title of the Invention

Patent Number

6,740,302
May 25, 2004

Method for producing high-purity sulphuric acid

Abstract of Patent

Abstract

The invention relates to a novel method for producing high-purity sulfuric acid for use in the semiconductor industry. The method comprises the addition of a hydrogen peroxide solution to an engineered column in order to reduce the SO₂ concentration, evaporation of the SO₃ and separation of acid traces. The high-purity SO₃ is then enriched with inert gas and the SO₃ is absorbed into sulfuric acid.

Inventors: Hostalek; Martin (Darmstadt, DE), Buttner; Werner (Darmstadt-Arheilgen, DE), Hafner; Rolf (Egelbach, DE), Lu; Chih-Peng (Taipei, TW), Kan; Ching-Jung (Nan-Tou, TW), Seitz; Ekkehart (Seeheim-Jungenheim, DE), Friedel; Ernst (Darmstadt, DE)

Inventor Names

Assignee: Merck Patent GmbH (Darmstadt, DE)

Assignee Information

Appl. No.: 10/168,869
Filed: June 26, 2002
PCT Filed: December 07, 2000
PCT No.: PCT/EP00/12327
PCT Pub. No.: WO01/47804
PCT Pub. Date: July 05, 2001

Foreign Application Priority Data

Dec 28, 1999 [DE]

199 63 509

Classification Information

Current U.S. Class:

Current International Class:

Field of Search:

423/523 ; 423/522; 423/531; 423/532
C01B 17/69 (20060101); C01B 17/90 (20060101); C01B 17/00 (20060101); C01B 017/69 O; C01B 017/74 O;
C01B 017/82 O; C01B 017/90 O
423/522,523,531,532

References

References Cited [Referenced By]

U.S. Patent Documents

[2562240](#)
[3760061](#)
[3948624](#)

July 1951
September 1973
April 1976

Merriam et al.
Hammond
Fornoff et al.

Step 6 – Review the Claims, Description, and Drawings.

Claims Define the Unique Features of the Invention and Determine Patentability

Claims

What is claimed is:

1. A process for producing high purity sulfuric acid comprising: a) adding hydrogen peroxide solution having a concentration of 1-70% to 24-70% technical grade oleum in a sufficient amount to lower the SO₂ concentration to below 10 ppm, b) evaporating SO₃ in the oleum at 90-130 degree C. in a falling film evaporator, c) removing traces of sulfuric acid and nitrosyl sulfuric acid from the resultant SO₃ gas stream escaping from the evaporator by means of a demister, d) enriching the high purity SO₃ with inert gas, and e) absorbing the SO₃ in sulfuric acid at a concentration of 90-99% to form said high purity sulphuric acid.
2. A process according to claim 1, wherein a portion of the high purity sulfuric acid stream obtained is recycled back to the absorption step.
3. A process according to claim 1, wherein high purity deionized water is added to said high purity sulfuric acid to adjust the concentration thereof, of the high purity sulfuric acid to a desired concentration, the and wherein the concentration adjustment is closed loop controlled by conductivity measurement.
4. A process according to claim 1, wherein the high purity sulfuric acid obtained is filled into PTFE-lined storage vessels or containers.
5. A process according to claim 1, further comprising removing particles from the resultant high purity sulfuric acid using a three-stage filtration.
6. A process according to claim 5, wherein in the removal of particles by filtration PFA or PTFE filters having a pore size of 0.1 μm to 1 μm are used.
7. A process according to claim 1, wherein absorption of SO₃ is performed cocurrently in a PTFE-lined reactor containing packing elements of PFA.
8. A process according to claim 1, wherein the heat of reaction formed is removed in a tube bundle reactor made of PFA or fluorinated polyolefins under an inert gas cushion downstream of adsorption of SO₃.
9. A process according to claim 1, wherein said high purity SO₃ is enriched with ultrapure nitrogen or highly purified air as said inert gas to an inert gas concentration between 1 to 50%.
10. A process according to claim 1, wherein said demister is made of high purity PFA or fluorinated polyolefins.
11. A process according to claim 1, wherein vent gases from the absorption step are treated with pure sulfuric acid in a scrubber.

The Description provides a brief summary of the invention, description of drawings (if any), background information on the invention, and a detailed description of the invention

Description

The present invention relates to a novel process for producing high purity sulfuric acid for use in the semiconductor industry.

Pure sulfuric acid can be produced on an industrial scale by passing SO₃ into dilute sulfuric acid, by combining SO₃ and pure water or by distillation of sulfuric acid at atmospheric or reduced pressure.

The quality of the sulfuric acid produced is affected not only by the design of the plants and the quality of the raw materials used, but also by the type and quality of the materials of construction used in the plant. These have an appreciable bearing on the level of undesirable metal ions, but also on the level of particles.

It is known to produce relatively high purity concentrated sulfuric acid by having SO₃ evaporated or expelled from oleum in a distillation flask and then introduced into dilute pure sulfuric acid. In particular embodiments, the evaporation is occasionally carried out in a falling film evaporator. Generally, the equipment used for producing pure sulfuric acid is made of glass or of enamel-lined steel. Depending on the quality used, these materials may leach ionogenic and/or particulate impurities.

Existing processes have the disadvantage that, in the event of nonuniform evaporation, the gas stream may entrain drops of liquid in the form of a fine mist and any impurities present therein into the end product. This happens in particular on conducting the evaporation in falling film evaporators customarily used on an industrial scale, but also on using distillation flasks.

Another disadvantage is the SO₂ still present in the sulfuric acid after purification.

It is an object of the present invention to provide an improved, economical way of producing on an industrial scale for use in the semiconductor industry a high purity sulfuric acid that is ideally free of metal ions and SO₂, but ideally also free of particles in particular.

This object is achieved by a continuously operable process for producing high purity sulfuric acid for the semiconductor industry, which is characterized in that a) hydrogen peroxide solution having a concentration of 1-70% is added to 24-70% technical grade oleum in a sufficient amount to lower the SO₂ concentration to below 10 ppm, b) the SO₃ in the oleum is evaporated at 90-130.degree. C. in a falling film evaporator, c) sulfuric acid and nitrosylsulfuric acid traces are removed from the SO₃ gas stream escaping from the evaporator by means of a demister, for example in the form of a candle filter, d) the high purity SO₃ is enriched with inert gas, and e) the SO₃ is absorbed in sulfuric acid of a concentration of 90-99% with cooling.

High purity deionized water is added to adjust the concentration of the high purity sulfuric acid to a desired concentration, the concentration adjustment being closed loop controlled by conductivity measurement.

FIG. 1 is a schematic flow diagram of a plant according to the invention.

FIG. 2 shows the schematic construction of an oleum evaporator useful in the process of the invention.

FIG. 3 shows in turn the schematic construction of an absorber or absorption tower useful in the process of the invention.

In a particular embodiment of the process according to the invention, a portion of the high purity sulfuric acid stream obtained is recycled back into the absorption space.

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- References that cite this patent
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Note Classifications assigned and Field of Search

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Primary Examiner: Langel; Wayne A.

Attorney, Agent or Firm: Miller, White, Zelano, Branigan, P.C.

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United States Patent
Hostalek, et al.

6,740,302
May 25, 2004

Method for producing high-purity sulphuric acid

The invention relates to a novel method for producing high-purity sulphuric acid. The invention involves the use of a sulfur dioxide solution to an engineered column in order to reduce the SO₂ concentration, evaporation of the SO₃ and separation of acid traces. The high-purity SO₃ is then enriched with inert gas and the SO₃ is absorbed into sulfuric acid.

oxide solution to an engineered

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US006740302B2

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

(10) Patent No.: **US 6,740,302 B2**
(45) Date of Patent: **May 25, 2004**

(54) **METHOD FOR PRODUCING HIGH-PURITY SULPHURIC ACID**

(75) Inventors: **Martin Hostalek, Darmstadt (DE); Werner Bittner, Darmstadt/Arbldgen (DE); Rolf Hufner, Egelbach (DE); Chih-Peng Lu, Taipei (TW); Ching-Tsun Kan, Nan-Tou (TW); Ekkehart Selts, Selters/Trarbachheim (DE); Ernst Friedel, Darmstadt (DE)**

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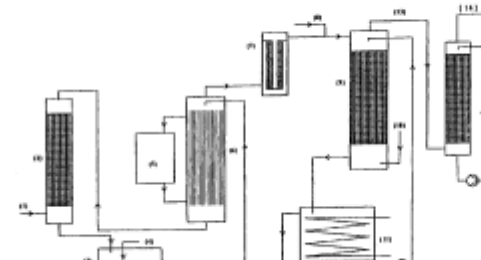
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Primary Examiner—Wayne A. Langel
(74) Attorney, Agent, or Firm—Miller, White, Zelano, Brinigan, PC.

(57) **ABSTRACT**
The invention relates to a novel method for producing high-purity sulfuric acid for use in the semiconductor industry. The method comprises the addition of a hydrogen peroxide solution to an engineered column in order to reduce the SO₂ concentration, evaporation of the SO₂, and separation of acid traces. The high-purity SO₂ is then enriched with inert gas and the SO₂ is absorbed into sulfuric acid.

(21) Appl. No.: **10/168,569**
(22) PCT Filed: **Dec. 7, 2000**
(86) PCT No.: **PCT/EP00/12327**
§ 371 (c)(1), (2), (4) Date: **Jun. 26, 2002**
(87) PCT Pub. No.: **WO01/47804**
PCT Pub. Date: **Jul. 5, 2001**
(65) **Prior Publication Data**
US 2002/0192144 A1 Dec. 19, 2002
(30) **Foreign Application Priority Data**
Dec. 28, 1999 (DE) 199 63 509
(51) **Int. Cl.** **C01B 17/09; C01B 17/74; C01B 17/82; C01B 17/90**
(52) **U.S. Cl.** **423/523; 423/522; 423/531; 423/532**
(58) **Field of Search** **423/522, 523, 423/531, 532**

21 Claims, 3 Drawing Sheets



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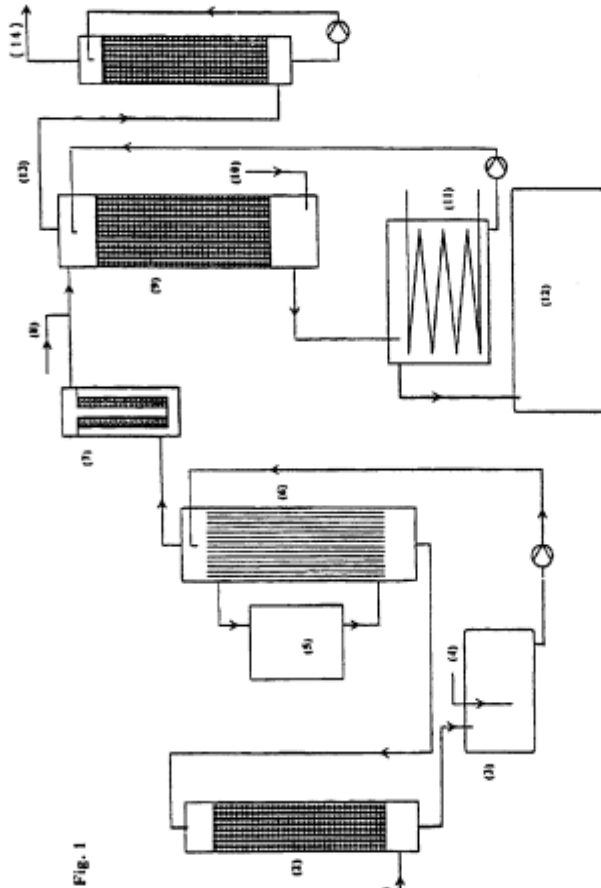


Fig. 1

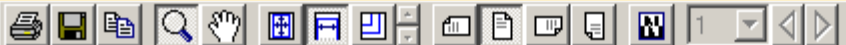
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Specifications



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US 6,740,302 B2

METHOD FOR PRODUCING HIGH-PURITY SULPHURIC ACID

The present invention relates to a novel process for producing high purity sulfuric acid for use in the semiconductor industry.

Pure sulfuric acid can be produced on an industrial scale by passing SO₂ into dilute sulfuric acid, by combining SO₂ and pure water or by distillation of sulfuric acid at atmospheric or reduced pressure.

The quality of the sulfuric acid produced is affected not only by the design of the plants and the quality of the raw materials used, but also by the type and quality of the materials of construction used in the plant. These have an appreciable bearing on the level of undesirable metal ions, but also on the level of particles.

It is known to produce relatively high purity concentrated sulfuric acid by having SO₂ evaporated or expelled from oleum in a distillation flask and then introduced into dilute pure sulfuric acid. In particular embodiments, the evaporation is occasionally carried out in a falling film evaporator. Generally, the equipment used for producing pure sulfuric acid is made of glass or enamel-lined steel. Depending on the quality used, these materials may leach isogenic and/or particulate impurities.

Existing processes have the disadvantage that, in the event of nonuniform evaporation, the gas stream may entrain drops of liquid in the form of a fine mist and any impurities present therein into the end product. This happens in particular on conducting the evaporation in falling film evaporators customarily used on an industrial scale, but also on using distillation flasks.

Another disadvantage is the SO₂ still present in the sulfuric acid after purification.

It is an object of the present invention to provide an improved, economical way of producing on an industrial scale for use in the semiconductor industry a high purity sulfuric acid that is ideally free of metal ions and SO₂, but ideally also free of particles in particular.

This object is achieved by a continuously operable process for producing high purity sulfuric acid for the semiconductor industry, which is characterized in that

- hydrogen peroxide solution having a concentration of 1-70% is added to 24-70% technical grade oleum in a sufficient amount to lower the SO₂ concentration to below 10 ppm,
- the SO₂ in the oleum is evaporated at 90-130° C. in a falling film evaporator,
- sulfuric acid and nitrosylsulfuric acid traces are removed from the SO₂ gas stream escaping from the evaporator by means of a demister, for example in the form of a candle filter,
- the high purity SO₂ is enriched with inert gas, and
- the SO₂ is absorbed in sulfuric acid of a concentration of 90-99% with cooling.

High purity deionized water is added to adjust the concentration of the high purity sulfuric acid to a desired concentration, the concentration adjustment being closed loop controlled by conductivity measurement.

FIG. 1 is a schematic flow diagram of a plant according to the invention.

FIG. 2 shows the schematic construction of an oleum evaporator useful in the process of the invention.

In a particular embodiment of the process according to the invention, a portion of the high purity sulfuric acid stream obtained is recycled back into the absorption space.

The high purity sulfuric acid obtained by the process according to the invention is collected in PTFE-lined storage vessels.

Particulate impurities formed or entrained are removed from the sulfuric acid using a one- to three-stage filtration.

The process according to the invention is preferably carried out using PFA or PTFE films having a pore size of 0.1 µm to 1 µm.

The SO₂ is advantageously absorbed cocurrently in a PTFE-lined reactor containing packing elements of PFA. The heat of reaction formed is removed in a downstream tube bundle reactor made of PFA or fluorinated polyolefins under an inert gas cushion.

The high purity SO₂ is enriched with ultrapure nitrogen or highly purified air as inert gas to an inert gas concentration between 1 to 50%.

A demister made of high purity PFA or fluorinated polyolefins is used.

Vent gases are treated with pure sulfuric acid in a scrubber.

It has been experimentally determined that high purity sulfuric acid meeting the abovementioned criteria is produced economically on an industrial scale on subjecting the concentrated technical grade starting quality to a stagewise treatment and purification, although the overall process is per se operable continuously.

The tests which have been carried out have shown that, in an upstream step, the SO₂ content of concentrated sulfuric acid of technical grade quality (oleum having a concentration between 24-70%) can be lowered to concentrations of less than 1 ppm by the addition of small amounts of hydrogen peroxide. The SO₂ concentration in oleum is determined according to customary methods of titration representing the best in analytical technology. Such a method is described, for example, in JIS K8051^{1,1001}.

It is advantageous for this purpose to use hydrogen peroxide solutions having a concentration between 1 to 70%. It is generally sufficient to add hydrogen peroxide solution in an amount of 0.001 to 0.1%, based on the amount of concentrated sulfuric acid or oleum to be treated, to oxidize the SO₂ present therein.

The method of adding the hydrogen peroxide solution to the technical grade oleum is not critical per se. Addition may be via a dip tube installed at the bottom of the oleum buffer tank FIG. 1 (3), which is disposed downstream of the oleum tower FIG. 1 (2). Mixing takes place automatically as a consequence of the convection due to the heat released in the tank. It is also possible to add the hydrogen peroxide solution elsewhere in the tank. However, the latter option would necessitate additional internals and possibly a form of mechanical mixing, for example stirring.

Pure SO₂ free of sulfuric acid is then obtained on evaporating SO₂ from the SO₂-free oleum under mild conditions in a falling film evaporator FIG. 1 (4) equipped with a precisely controllable heating system FIG. 1 (5).

A falling film evaporator suitable for this purpose has the following properties:

- homogeneous liquid distribution,
- low gas velocity,
- pressure control by siphons, which contributes to plant accident avoidance,

Claims



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The process of the invention is particularly useful in combination with a plant for synthesizing SO₂.

FIG. 1 is a schematic flow diagram of a plant according to the invention; the components shown therein have the following meanings:

- (1) SO₂ feed
- (2) Oleum tower
- (3) Oleum buffer tank
- (4) Hydrogen peroxide feed
- (5) Heating system
- (6) Evaporator
- (7) Demister
- (8) Inert gas feed
- (9) Absorption tower
- (10) Ultrapure water feed
- (11) Cooling
- (12) Inlet product
- (13) Offgas
- (14) Purified offgas

FIG. 2 shows the schematic construction of an oleum evaporator useful in the process. The individual components have the following meanings:

- (1) Oleum entry
- (2) First overflow weir
- (3) Pipe distributor caps
- (4) Evaporator tube
- (5) Oleum outlet
- (6) SO₂ takeoff
- (7) Hot air entry
- (8) Hot air exit

FIG. 3 shows in turn the schematic construction of an absorber or absorption tower useful in the process. The components shown therein have the hereinbelow indicated meanings:

- (1) Acid distributor nozzles
- (2) Packing layer
- (3) Acid outlet to cooler
- (4) Inert outlet
- (5) SO₂ outlet

The scope of the present invention covers not just the plants described and schematically depicted. The scope is to be understood as also including generalizations or modifications that occur to a person skilled in the art or equivalent components performing the same purpose.

What is claimed is:

1. A process for producing high purity sulfuric acid comprising:

- adding hydrogen peroxide solution having a concentration of 1–70% to 24–70% technical grade oleum in a sufficient amount to lower the SO₂ concentration to below 10 ppm,
- evaporating SO₂ in the oleum at 90–130° C. in a falling film evaporator,
- removing traces of sulfuric acid and nitrosyl sulfuric acid from the resultant SO₂ gas stream escaping from the evaporator by means of a demister,
- enriching the high purity SO₂ with inert gas, and
- absorbing the SO₂ in sulfuric acid at a concentration of

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3. A process according to claim 1, wherein high purity deionized water is added to said high purity sulfuric acid to adjust the concentration thereof, of the high purity sulfuric acid to a desired concentration, the and wherein the concentration adjustment is closed loop controlled by conductivity measurement.

4. A process according to claim 1, wherein the high purity sulfuric acid obtained is filled into PTFE-lined storage vessels or containers.

5. A process according to claim 1, further comprising removing particles from the resultant high purity sulfuric acid using a three-stage filtration.

6. A process according to claim 5, wherein in the removal of particles by filtration PFA or PTFE filters having a pore size of 0.1 μm to 1 μm are used.

7. A process according to claim 1, wherein absorption of SO₂ is performed co-currently in a PTFE-lined reactor containing packing elements of PFA.

8. A process according to claim 1, wherein the heat of reaction formed is removed in a tube bundle reactor made of PFA or fluorinated polyolefins under an inert gas cushion downstream of absorption of SO₂,

9. A process according to claim 1, wherein said high purity SO₂ is enriched with ultrapure nitrogen or highly purified air as said inert gas to an inert gas concentration between 1 to 50%.

10. A process according to claim 1, wherein said demister is made of high purity PFA or fluorinated polyolefins.

11. A process according to claim 1, wherein vent gases from the absorption step are treated with pure sulfuric acid in a scrubber.

12. A process according to claim 1, wherein said demister is a candle filter.

13. A process according to claim 12, wherein said candle filter is made of high purity PFA or fluorinated polyolefin containing no cationic impurities.

14. A process according to claim 1, wherein said vent gases from the absorption step are treated with sulfuric acid at a concentration of 90–99%.

15. A process according to claim 1, further comprising removing particles from the resultant high purity sulfuric acid using a one- to three-stage filtration.

16. A process according to claim 1, wherein hydrogen peroxide solution is added in an amount of 0.001 to 0.1%, based on the amount of oleum to be treated.

17. A process according to claim 1, wherein said inert gas is ultrapure nitrogen or purified air.

18. A process according to claim 1, wherein the sulfuric acid formed by absorption is diluted with high purity water and cooled, and a portion of the resultant diluted, cooled sulphuric acid is passed back to the absorption step.

19. A process according to claim 18, wherein absorption is performed in an absorption column and cooling is performed in a subsequent heat exchanger, and said high purity water is introduced at the base of said absorption column ahead of the subsequent heat exchanger.

20. A process for producing product sulfuric acid comprising:

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United States Patent
Hostalek et al.

(10) Patent No.: US 6,740,302 B2
(45) Date of Patent: May 25, 2004

(54) METHOD FOR PRODUCING HIGH-PURITY SULFURIC ACID

(56) References Cited

(75) Inventors: Hostalek, et al.

(73) Assignee: Hostalek, et al.

(*) Not for Publication

(21) Application No. 09/921,144
(22) Filed: Aug. 11, 2002
(80) Provisional Patent Application No. 60/302,144
(87) International Publication No. WO/03/019,214
(65) Prior Art Document: US 2002/019,214 A1 Dec. 19, 2002

(30) Foreign Application Priority Data
Dec. 28, 1999 (DE) 199 63 509

(51) Int. Cl.⁷ C01B 17/06; C01B 17/04; C01B 17/82; C01B 17/90

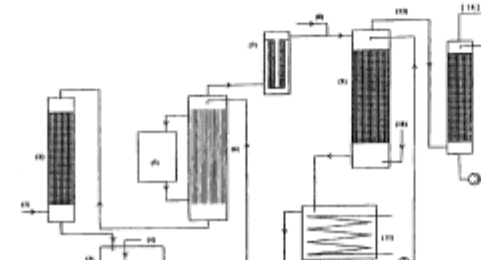
(52) U.S. Cl. 423/523; 423/522; 423/531; 423/532

(58) Field of Search 423/522, 523, 423/531, 532

(57) ABSTRACT

The invention relates to a novel method for producing high-purity sulfuric acid for use in the semiconductor industry. The method comprises the addition of a hydrogen peroxide solution to an engineered olefin in order to reduce the SO₂ concentration, evaporation of the SO₂, and separation of acid traces. The high-purity SO₂ is then enriched with inert gas and the SO₂ is absorbed into sulfuric acid.

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Reissue -- RE35,312 RE12345 RE00007

Defensive Publication -- T109,201 T855019 T100001

Statutory Invention Registration -- H001,523 H001234 H0000001

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United States Patent
Hostalek , et al.

6,740,302
May 25, 2004

Method for producing high-purity sulphuric acid

Abstract

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The invention relates to a novel method for producing high-purity sulfuric acid for use in the semiconductor industry. The method comprises the addition of a hydrogen peroxide solution to an engineered column in order to reduce the SO₂ concentration, evaporation of the SO₃ and separation of acid traces. The high-purity SO₃ is then enriched with inert gas and the SO₃ is absorbed into sulfuric acid.

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United States Patent

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Current U.S. Class:

Current International Class:

3,492,131

January 27, 1970

426/548 ; 260/1; 426/656; 530/801

A23G 3/34 (20060101); A23L 1/236 (20060101)

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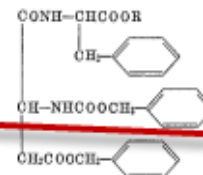
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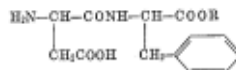
group occurs to afford the protected dipeptide of the following formula

3,492,131
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James M. Schlatter, Glenview, Ill., assignor to G. D. Searle & Co., Chicago, Ill., a corporation of Delaware
No Drawing. Filed Apr. 18, 1966, Ser. No. 543,054
Int. Cl. A231 1/26
U.S. Cl. 99-141 12 Claims



The present invention relates to novel compositions which are especially useful in view of their sweetening properties and to novel methods for the use of those compositions as sweetening agents.

The instant substances which exhibit this surprising sweetening property are dipeptide derivatives characterized by the following structural formula



wherein R is a lower alkyl radical.

Illustrative of the lower alkyl radicals denoted by the R term are methyl, ethyl, propyl, butyl, pentyl, hexyl, heptyl and the branched-chain groups isomeric therewith.

The surprisingly potent sweet taste of these dipeptide derivatives is completely unexpected and could not have been predicted from a consideration of their chemical structure. That property apparently is related to the polarity of the molecule as indicated by the fact that the compounds wherein R is hydrogen, i.e. the corresponding free carboxylic acids, are completely lacking in sweetness.

The sweetening property of the dipeptide substances is dependent also upon the stereochemistry of the individual amino acids, i.e. aspartic acid and phenylalanine, from which the dipeptides are derived. Each of the amino acids can exist in either the D or L form, but it has been determined that the L-aspartyl - L - phenylalanine esters are

wherein R is a lower alkyl radical as exemplified above. Removal of the N-benzoyloxycarbonyl and O-benzyl protecting groups is conveniently effected by hydrogenolysis at atmospheric pressure and room temperature, utilizing palladium as the catalyst. Those processes are specifically illustrated by the reaction of N-benzoyloxycarbonyl - L - aspartic acid α - p - nitrophenyl, β - benzyl diester with L-phenylalanine methyl ester to afford β - benzyl N-benzyl - oxycarbonyl - L - aspartyl - L - phenylalanine methyl ester and hydrogenolysis of that intermediate in aqueous acetic acid with palladium metal catalyst to produce L-aspartyl - L - phenylalanine methyl ester.

The instant dipeptide sweetening agents are water soluble, stable substances which can be utilized in a variety of physical forms, e.g. as powders, tablets, syrups, etc. Liquid or solid carriers such as water, glycerol, starch, sorbitol, salt, citric acid and other suitable non-toxic substances can be utilized also. These compositions are particularly valuable as sweetening agents for edible materials. Examples of such materials are fruits, vegetables, juices, meat products such as ham or bacon, sweetened milk products, egg products, salad dressings, ice creams and sherbets, icings, syrups, cake mixes and beverages such as carbonated soft drinks and wines.

The invention will appear more fully from the examples which follow. These examples are set forth by way of illustration only and it will be understood that the invention is not to be construed as limited either in spirit

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US 20090178512A1

(19) **United States**
(12) **Patent Application Publication** (10) **Pub. No.: US 2009/0178512 A1**
Partelpoeg (43) **Pub. Date: Jul. 16, 2009**

(54) **PROCESS FOR LIBERATING METALS USING DIRECT PRODUCTION OF LEACH GRADE ACID SOLUTIONS**

Related U.S. Application Data

(60) Provisional application No. 61/010,755, filed on Jan. 11, 2008, provisional application No. 61/060,872, filed on Jun. 12, 2008.

(75) Inventor: **Eric H. Partelpoeg**, Tucson, AZ (US)

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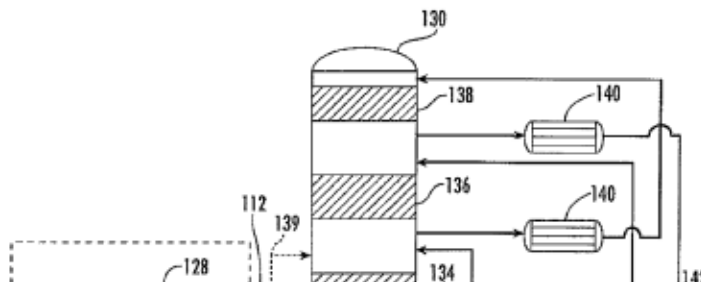
ABSTRACT

A system which uses available waste acid process streams from hydrometallurgical extraction processes as absorption media for the direct production of leach grade acid solutions for return to the hydrometallurgical processing circuit for use in liberating metals from metal-bearing materials. The produced acid may have a concentration in the range suitable for processing of metal-bearing materials

(73) Assignee: **EHP Technology, LLC**, Tucson, AZ (US)

(21) Appl. No.: **12/246,250**

(22) Filed: **Oct. 6, 2008**



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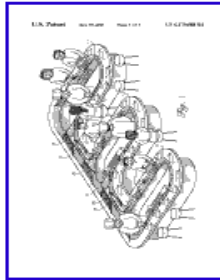
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