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

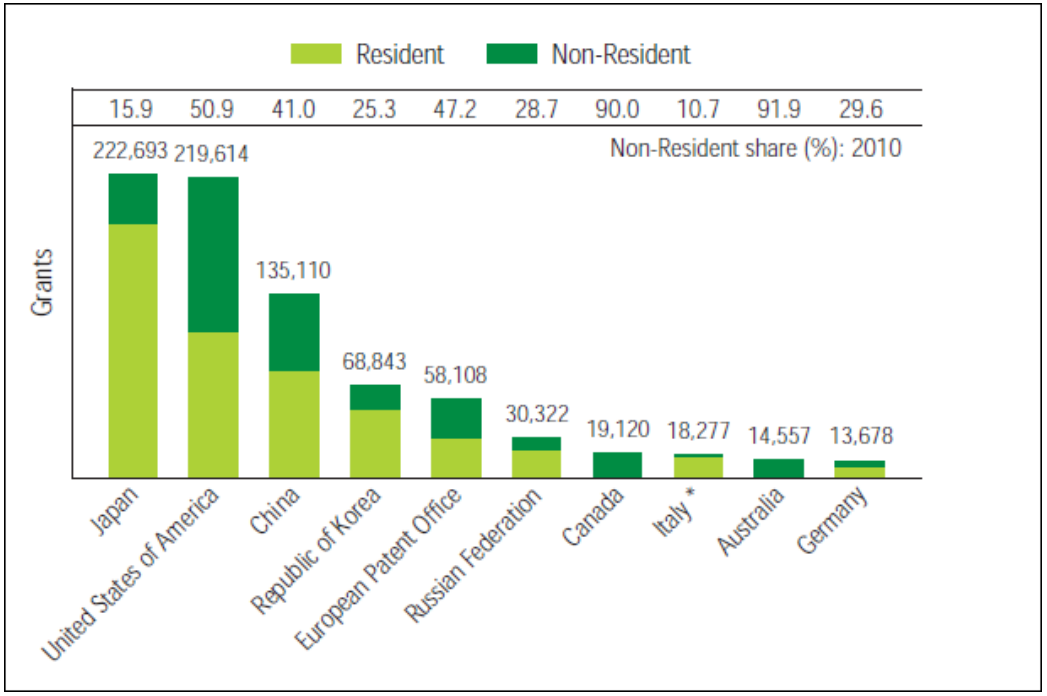
Why Search the Patent Literature?

- 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

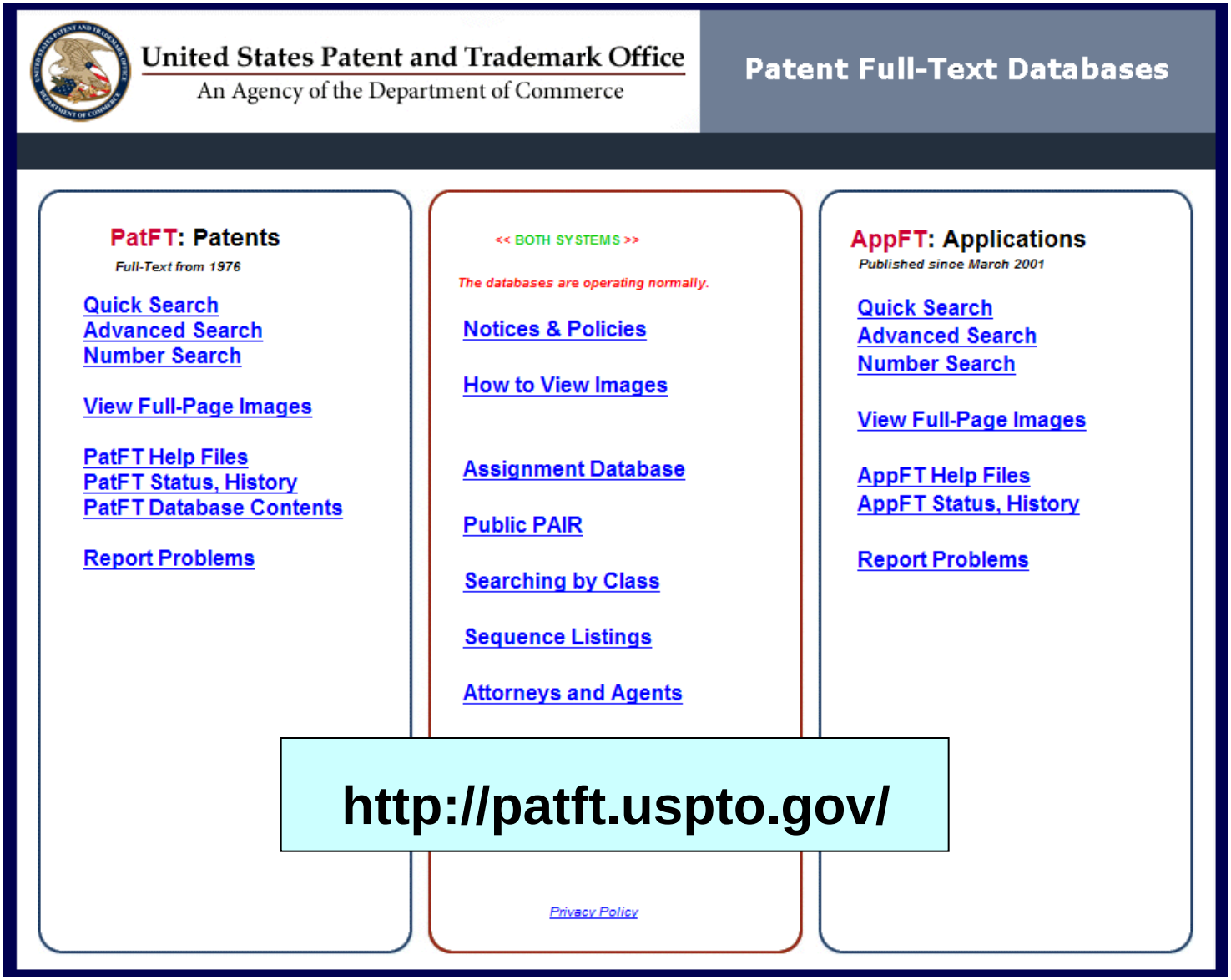
5. Search the Issued Patents and the Published Applications databases by "Current US Classification" and access full text patents and published applications.

Review and References

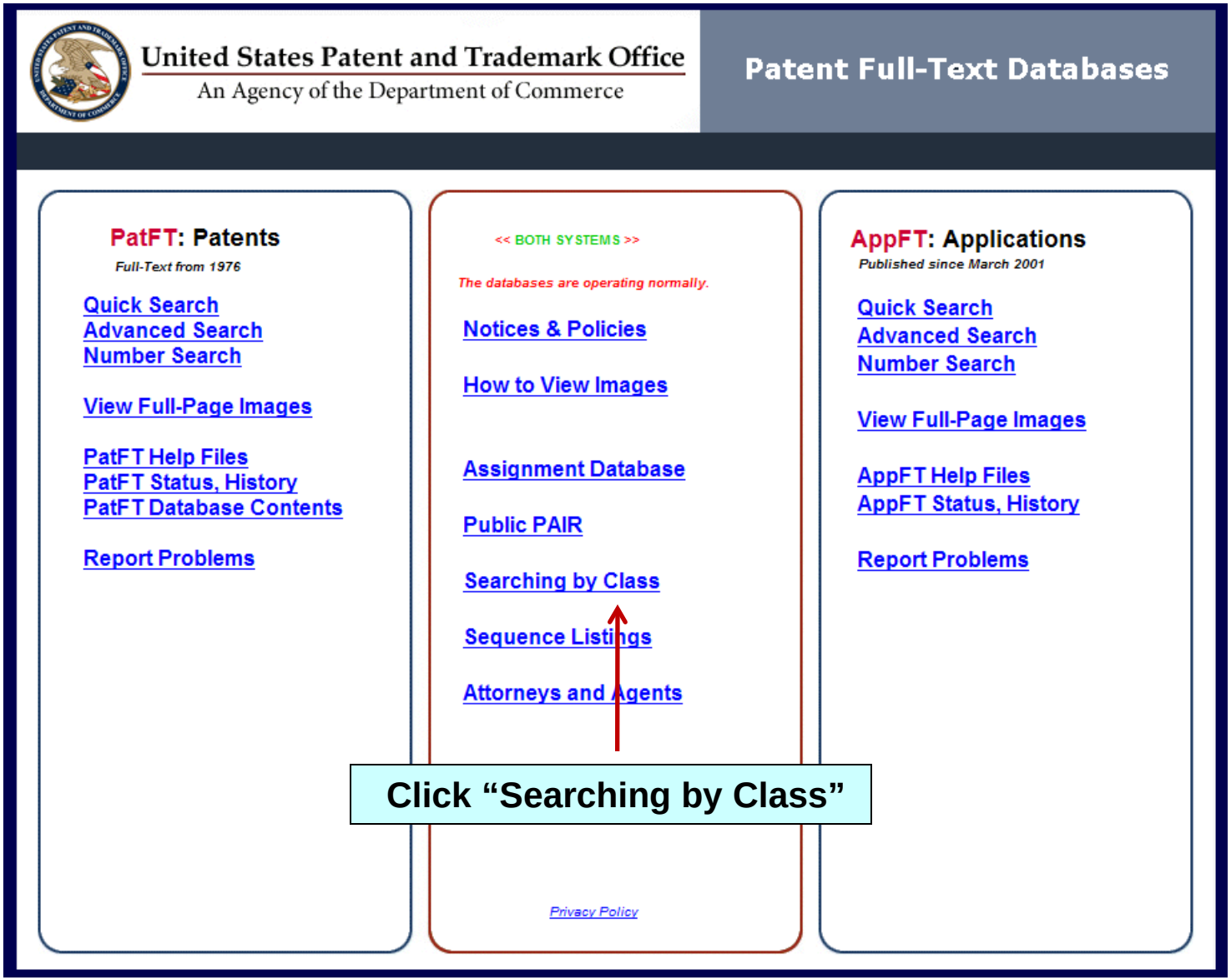
6. Review the claims, specifications and drawings of documents retrieved for relevancy.

7. Check all references and note the "U.S. Cl." and "Field of Search" areas for additional class/subclasses to search.

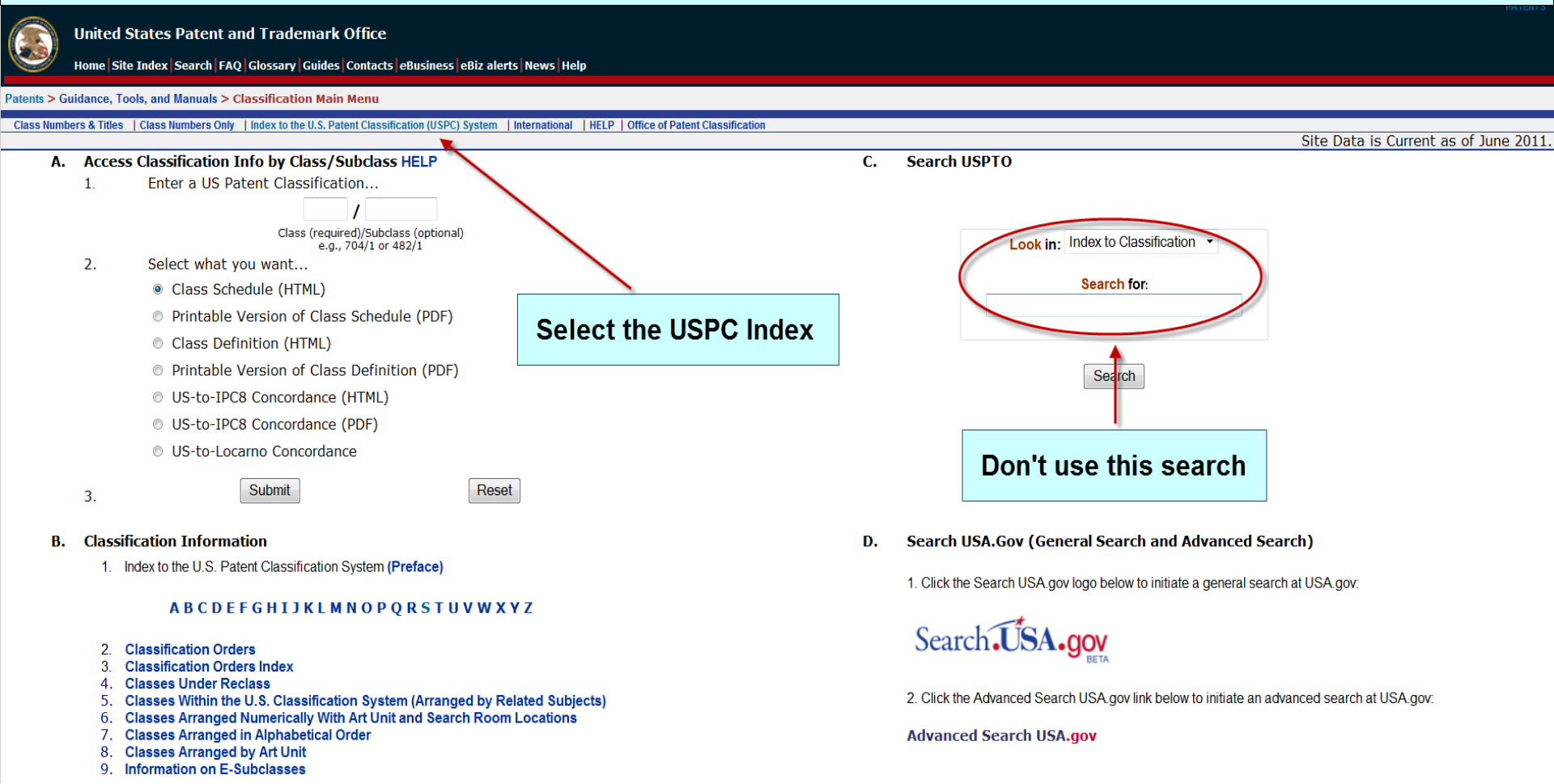
Accessing the USPTO Patent Full-Text and Image Database



Searching by the U.S. Patent Classification System



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



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Class Numbers & Titles | Class Numbers Only | USPC Index | International | HELP | Office of Patent Classification

Index to the USPC

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)

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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

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Separable	602 / 68
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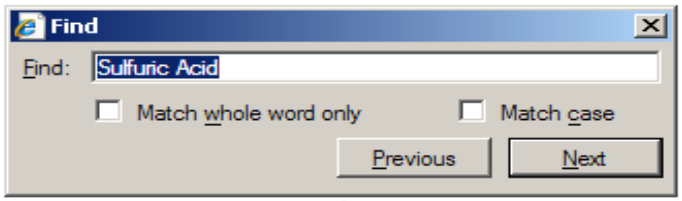

Use the Find function (Ctrl + F) to search page for "Sulfuric Acid"

Sulfur

Acids and acid anhydrides	423 / 512.1+
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Binding agents	44 / 589+
Combined with fuel	44 / 604
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Carboxylic acids	
Acyclic	562 / 512+
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Alicyclic esters	560 / 125
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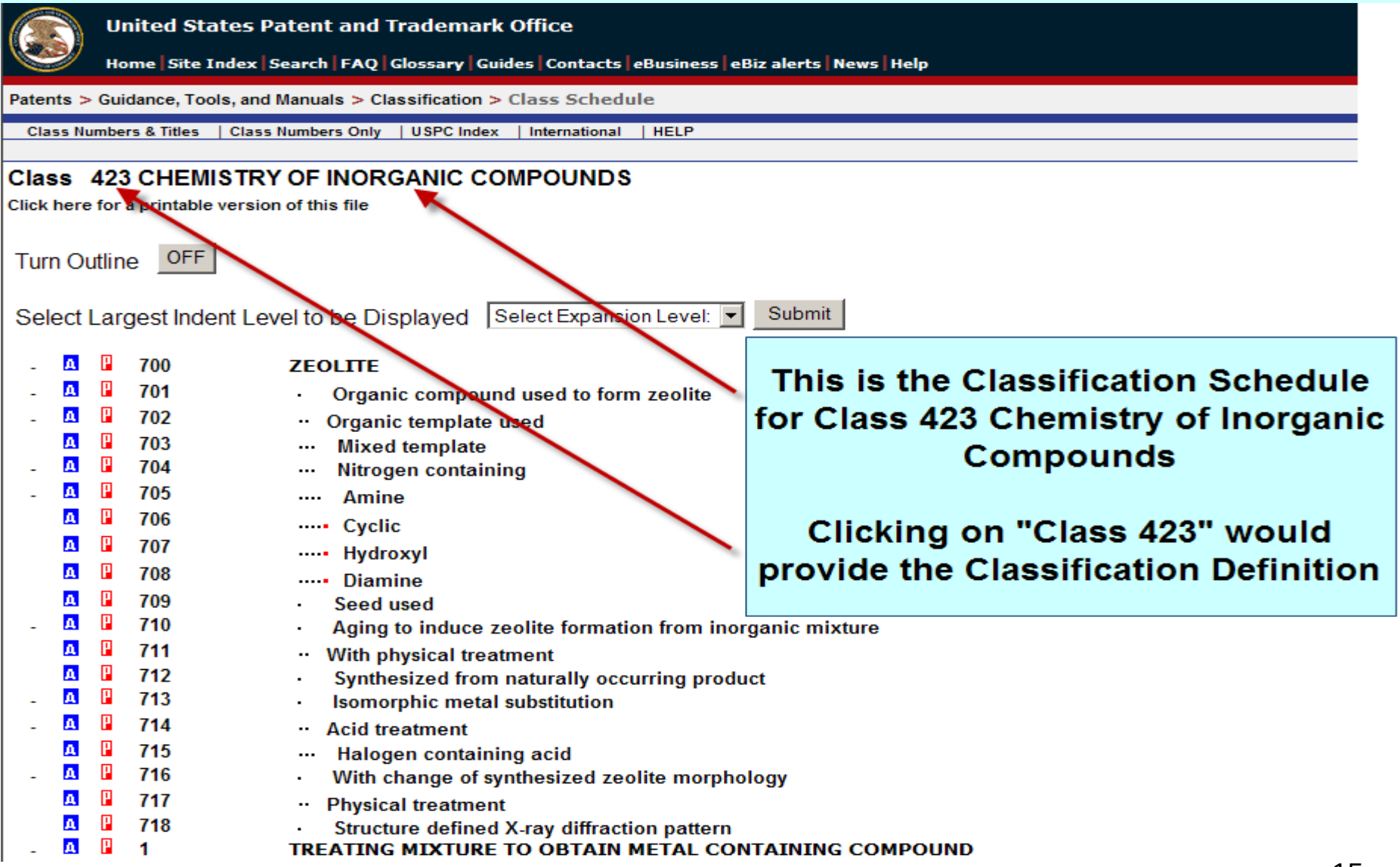
Trains	422 / 160
Electrolytic synthesis	205 / 554
Esters	558 / 20+
Mineral oil sludge source	208 / 13

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



Class Schedule – Only Small Portions are Hierarchical

511	SULFUR OR COMPOUND THEREOF	
512.1	· Oxygen containing	
513	·· Persulfate	
514	·· Thiosulfate	
515	·· Dithionite	
516	··· Employing amalgam as reactant	
517	·· Metal and ammonium containing	
518	·· Plural metal containing	
519	·· Bisulfite	
519.2	·· Sulfite	
520	··· Bisulfate	
521	·· Ternary compound containing hydrogen	
522	··· Sulfuric acid	
523	···· Nitrogenous impurity or utilizing nitrogenous catalyst or reactant	
524	·····▪ Lead chamber process	
525	···· Starting material includes organic or carbonaceous impurity	
526	·····▪ Utilizing pressure or vacuum on mixture	
527	·····▪ Adding organic compound to mixture	
528	·····▪ Contacting mixture with gas, steam, or vapor	
529	···· Subjecting reactants to pressure, vacuum, or steam	
530	···· Utilizing metal sulfate	
531	···· Purifying acid or reactant	
532	·· Sulfur trioxide	
533	··· Utilizing catalyst in reaction	
534	···· Promoter or successive diverse catalysts	
535	···· Catalyst contains oxygen, vanadium, and another metal	
536	···· Platinum catalyst	
537	·····▪ With sulfate or asbestos carrier	
538	···· Oxygen containing catalyst	
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 pro… …ulfate or acid sulfate radical (SO_4^{2-}).

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 $H_2S_2O_7$ may be designated. However, this compound is actually H_2SO_4 with SO_3 dissolved therein and is classified in this subclass.

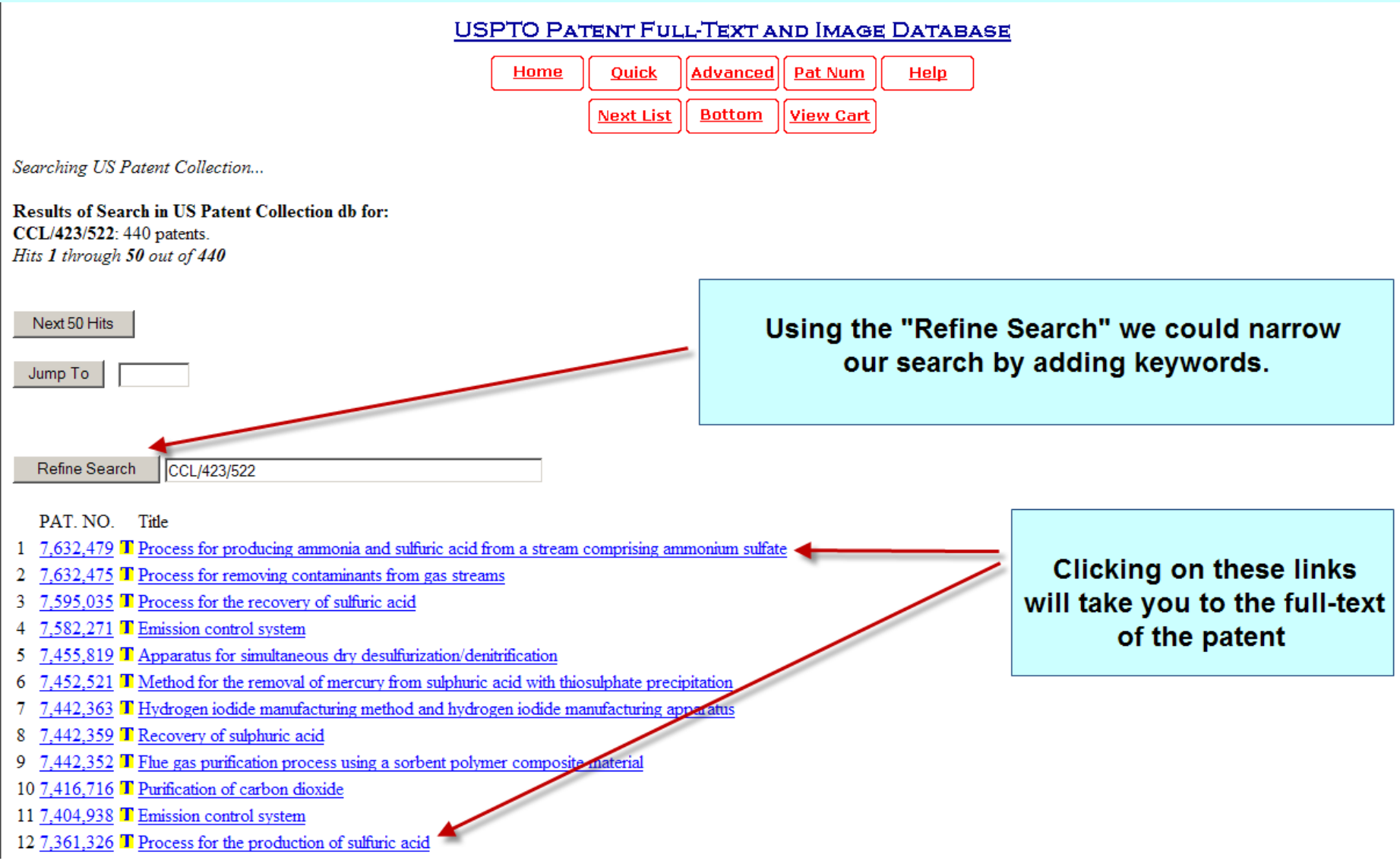
523 **Nitrogenous impurity or utilizing nitrogenous catalyst or reactant**
This subclass is indented under **subclass 522**. Processes in which during the manufacture of the sulfuric acid a compound of nitrogen is used as a catalyst or as a reactant, or is present as an impurity which requires removal.

524 …uric 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 …ans of nitrogen oxides (catalysts or reactants). The gases leaving the reaction chamber contain practically …t 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.

Definition for 423/522

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

Step 5 – Search Issued Patents and Access Full-Text



A Note about Accessing and Viewing Patents

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.

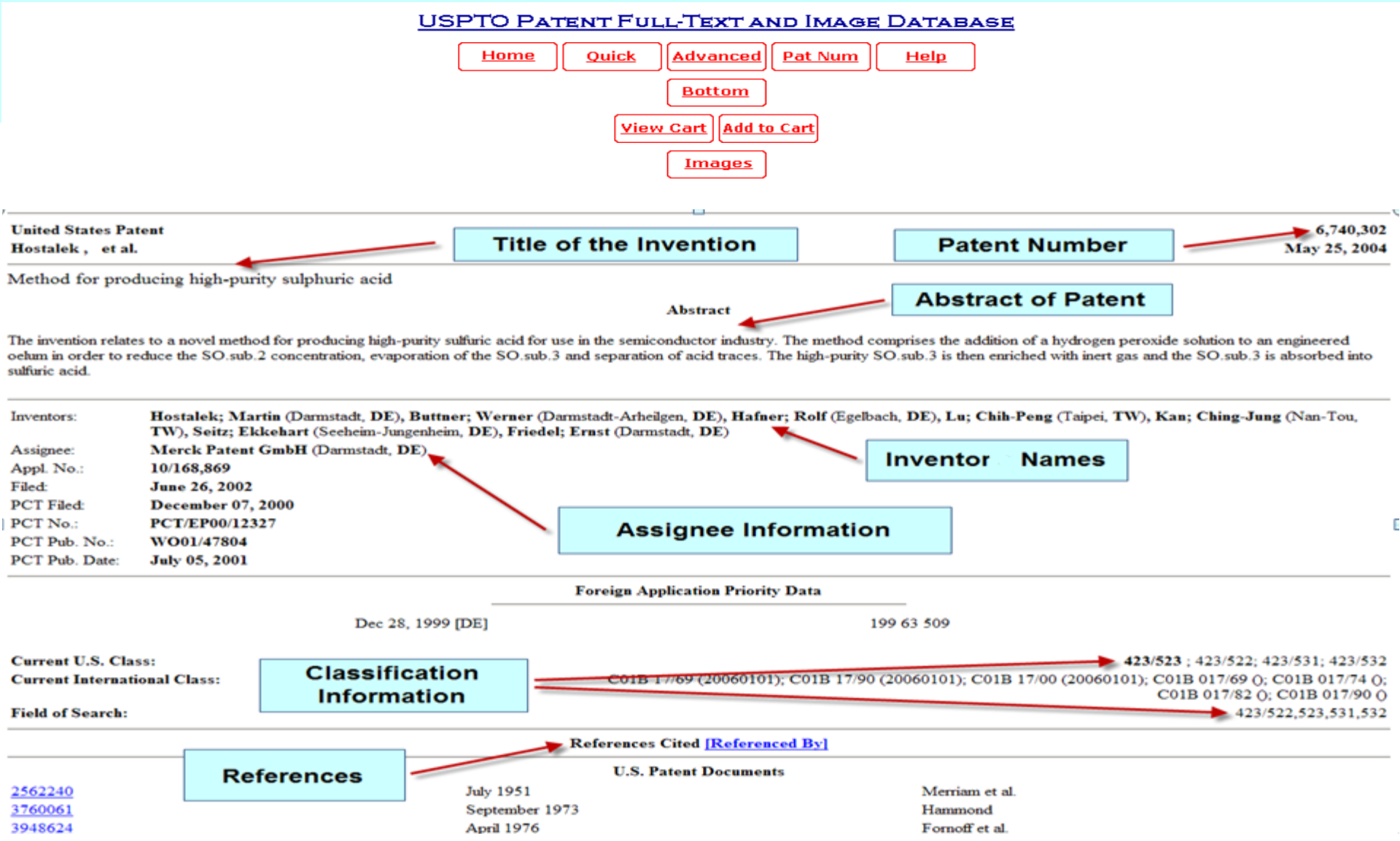
Results of Clicking the Class / Subclass Search

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



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 SO.sub.2 concentration to below 10 ppm, b) evaporating SO.sub.3 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 SO.sub.3 gas stream escaping from the evaporator by means of a demister, d) enriching the high purity SO.sub.3 with inert gas, and e) absorbing the SO.sub.3 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 .mu.m to 1 .mu.m are used.

7. A process according to claim 1, wherein absorption of SO.sub.3 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 SO.sub.3.

9. A process according to claim 1, wherein said high purity SO.sub.3 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 SO.sub.3 into dilute sulfuric acid, by combining SO.sub.3 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 SO.sub.3 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 SO.sub.2 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 SO.sub.2, 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 SO.sub.2 concentration to below 10 ppm, b) the SO.sub.3 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 SO.sub.3 gas stream escaping from the evaporator by means of a demister, for example in the form of a candle filter, d) the high purity SO.sub.3 is enriched with inert gas, and e) the SO.sub.3 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.

Step 7 – Review References

- Patents which are cited by the inventor
- References that cite this patent
- Other related works (e.g., journal articles, etc.)

Note Classifications assigned and Field of Search

References Cited [Referenced By]

U.S. Patent Documents

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3948624	April 1976	Fornoff et al.
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6627172	September 2003	Wagner et al.

Foreign Patent Documents

36 32 623	Mar., 1988	DE
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460745	Dec., 1991	EP
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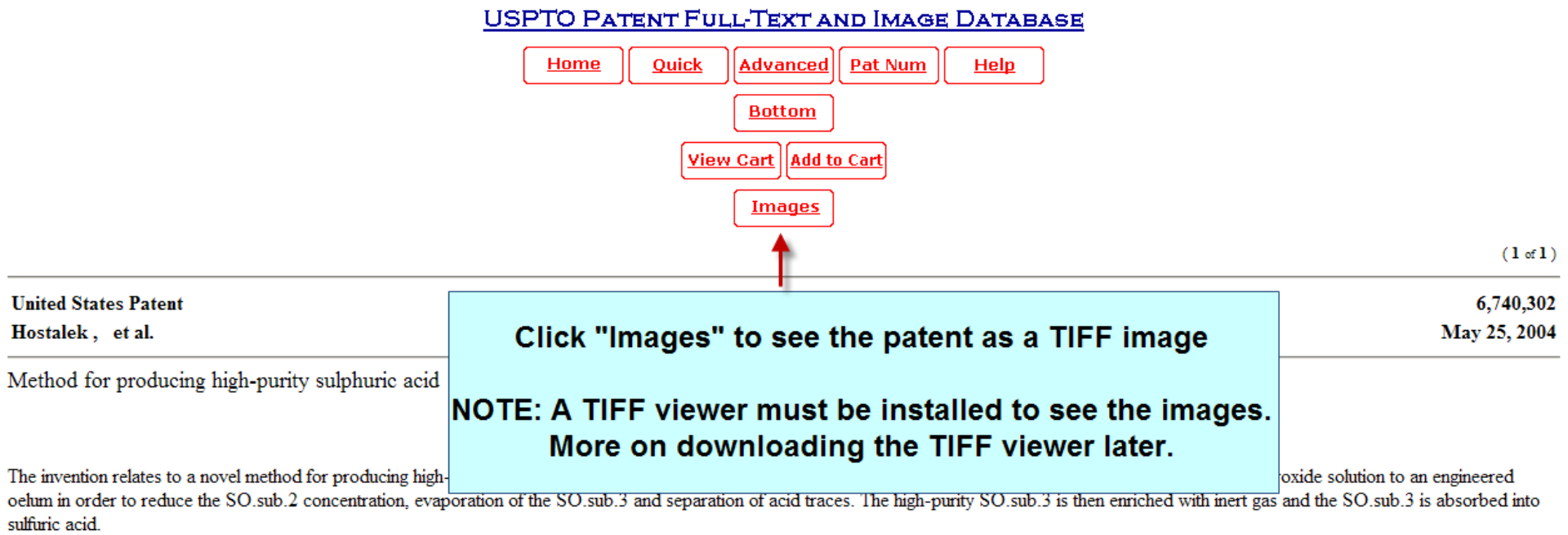
Other References

George Segeler Fuel Flue Gases pub. by the American Gas Association, U.S.A., pp. 113-114.* .
Gaspierk, I. et al., Databse Chemabs 'Onlline! Chemical Abstracts Service, "Manufacture of sulfuric acid useful in microelectronics," Database Accession No. 111:80844 CA XP002163475 Abstract for CS 259 353B (Oct. 14, 1988)..

Primary Examiner: Langel; Wayne A.
Attorney, Agent or Firm: Miller, White, Zelano, Branigan, P.C.

Viewing the Patent Images



TIFF Image of U.S. Patent 6,740,302

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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 Büttner**, Darmstadt-Arheilgen (DE); **Rolf Haftner**, Egelbach (DE); **Chih-Peng Lu**, Taipei (TW); **Ching-Jang Kan**, Nan-Tou (TW); **Ekkehart Seltz**, Seeheim-Jugenheim (DE); **Ernst Friedel**, Darmstadt (DE)

(73) Assignee: **Merck Patent GmbH**, Darmstadt (DE)

(*) 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.: **10/168,869**

(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.**[7] **C01B 17/69**; 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

(56) **References Cited**

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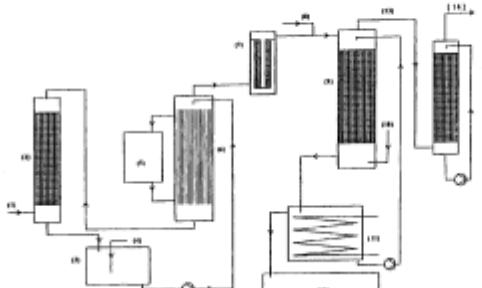
* cited by examiner

Primary Examiner—Wayne A. Langel
(74) *Attorney, Agent, or Firm*—Miller, White, Zelano, Branigan, P.C.

(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 oleum in order to reduce the SO_3 concentration, evaporation of the SO_3 and separation of acid traces. The high-purity SO_3 is then enriched with inert gas and the SO_3 is absorbed into sulfuric acid.

21 Claims, 3 Drawing Sheets



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Drawings

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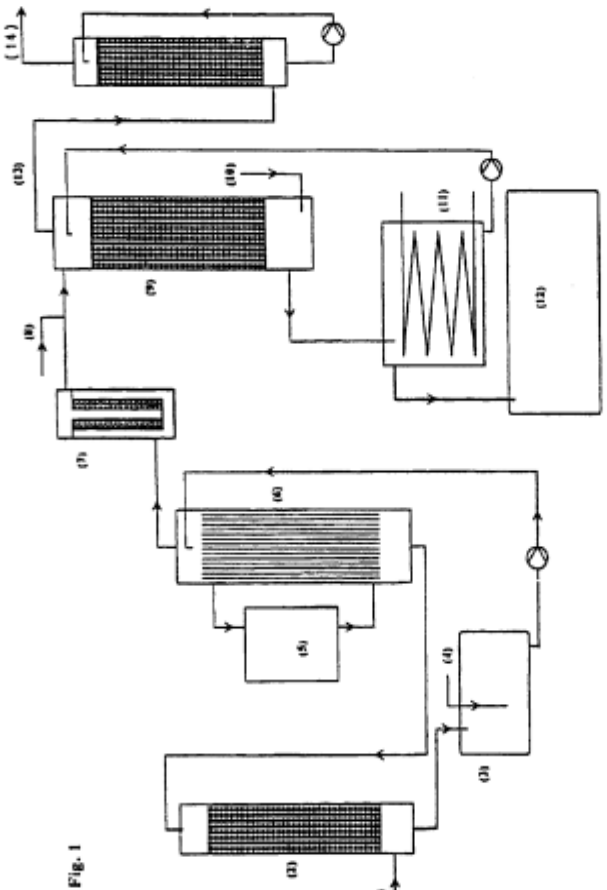

Fig. 1

Specifications

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 SO_3 into dilute sulfuric acid, by combining SO_3 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 SO_3 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 SO_2 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 SO_2, 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 SO_2 concentration to below 10 ppm,
b) the SO_3 in the oleum is evaporated at 90–130° C. in a falling film evaporator,
c) sulfuric acid and nitrosylsulfuric acid traces are removed from the SO_3 gas stream escaping from the evaporator by means of a demister, for example in the form of a candle filter,
d) the high purity SO_3 is enriched with inert gas, and
e) the SO_3 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 filters having a pore size of 0.1 μm to 1 μm.

The SO_3 is advantageously absorbed concurrently 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 SO_3 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 SO_2 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 SO_2 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 K8951-1995.

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 SO_2 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 SO_3 free of sulfuric acid is then obtained on evaporating SO_3 from the SO_2-free oleum under mild conditions in a falling film evaporator FIG. 1 (6) 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

The process of the invention is particularly useful in combination with a plant for synthesizing SO_3.

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

(1) SO_3 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) End 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) SO_3 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) SO_3 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:

a) adding hydrogen peroxide solution having a concentration of 1–70% to 24–70% technical grade oleum in a sufficient amount to lower the SO_3 concentration to below 10 ppm,

b) evaporating SO_3 in the oleum at 90–130° C. in a falling film evaporator,

c) removing traces of sulfuric acid and nitrosyl sulfuric acid from the resultant SO_3 gas stream escaping from the evaporator by means of a demister,

d) enriching the high purity SO_3 with inert gas, and

e) absorbing the SO_3 in sulfuric acid at a concentration of

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 SO_3 is performed concurrently 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 SO_3.

9. A process according to claim 1, wherein said high purity SO_3 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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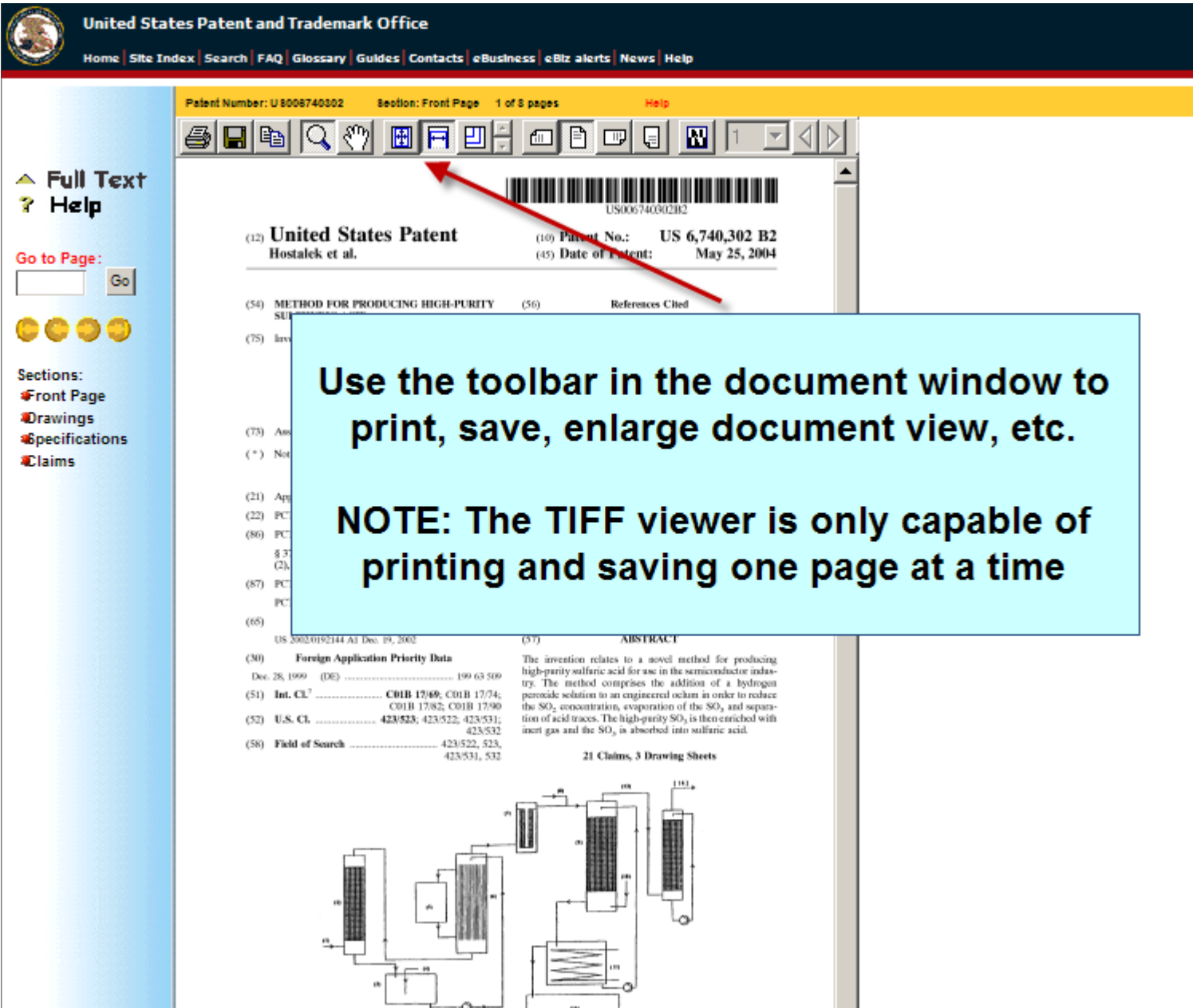

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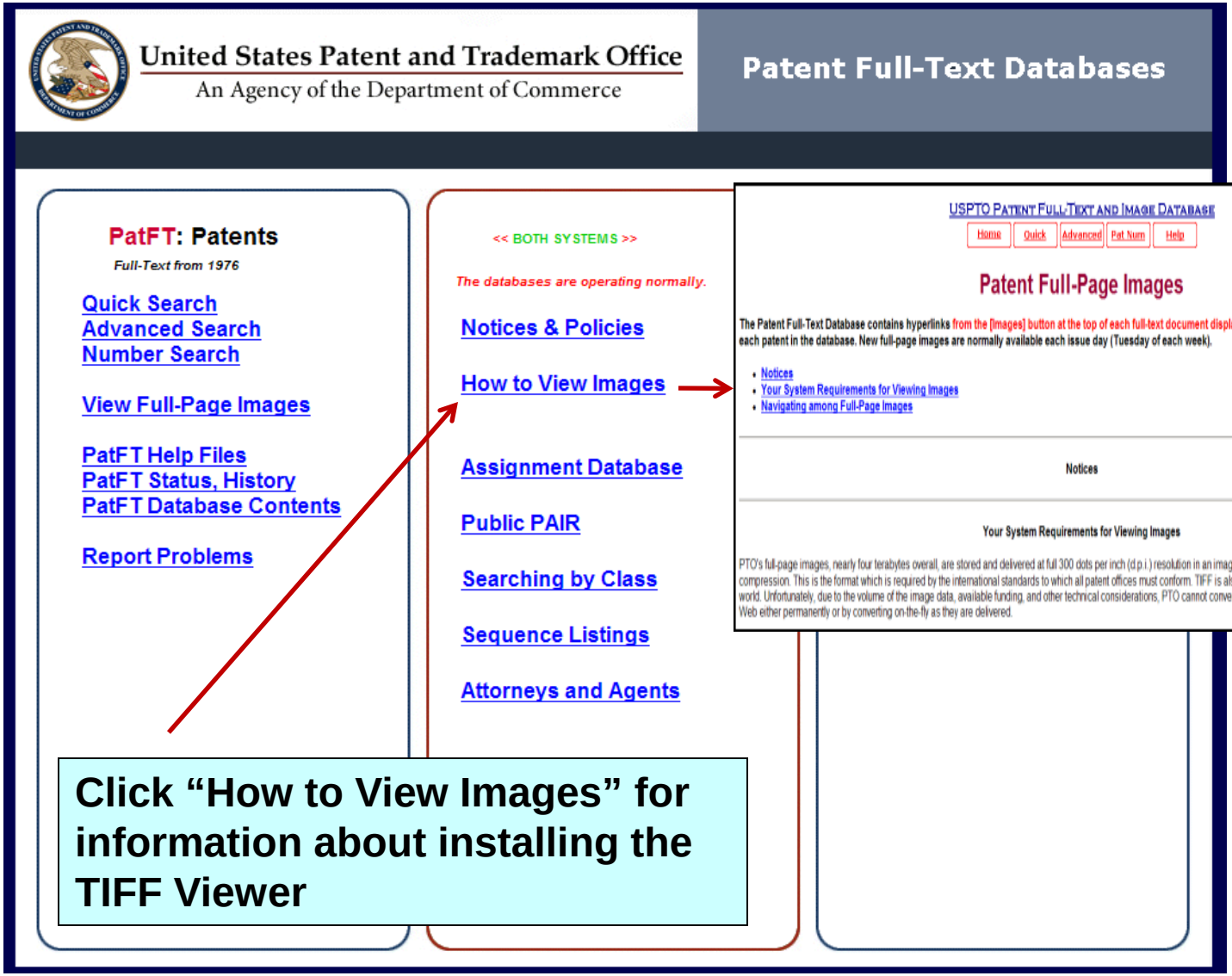

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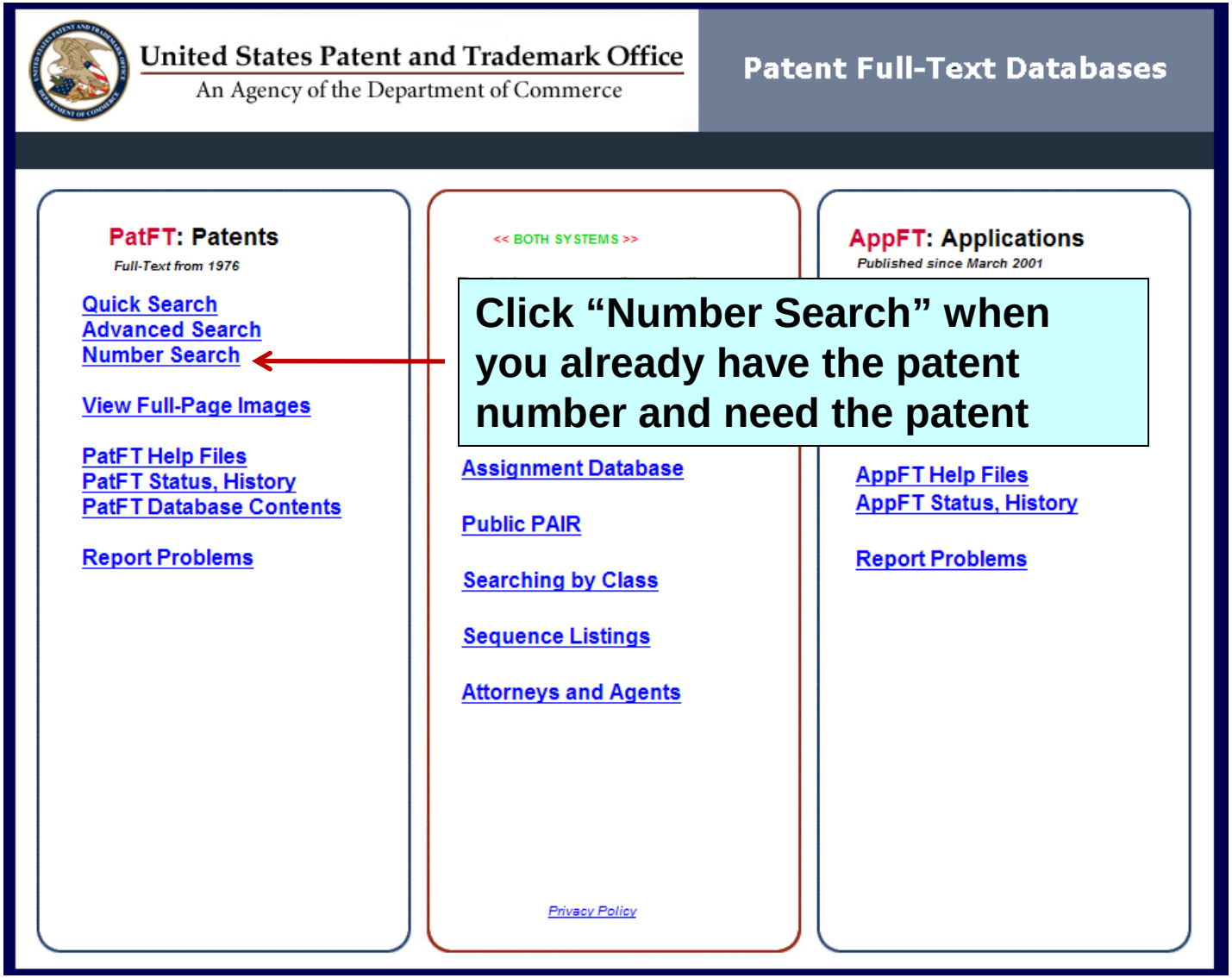

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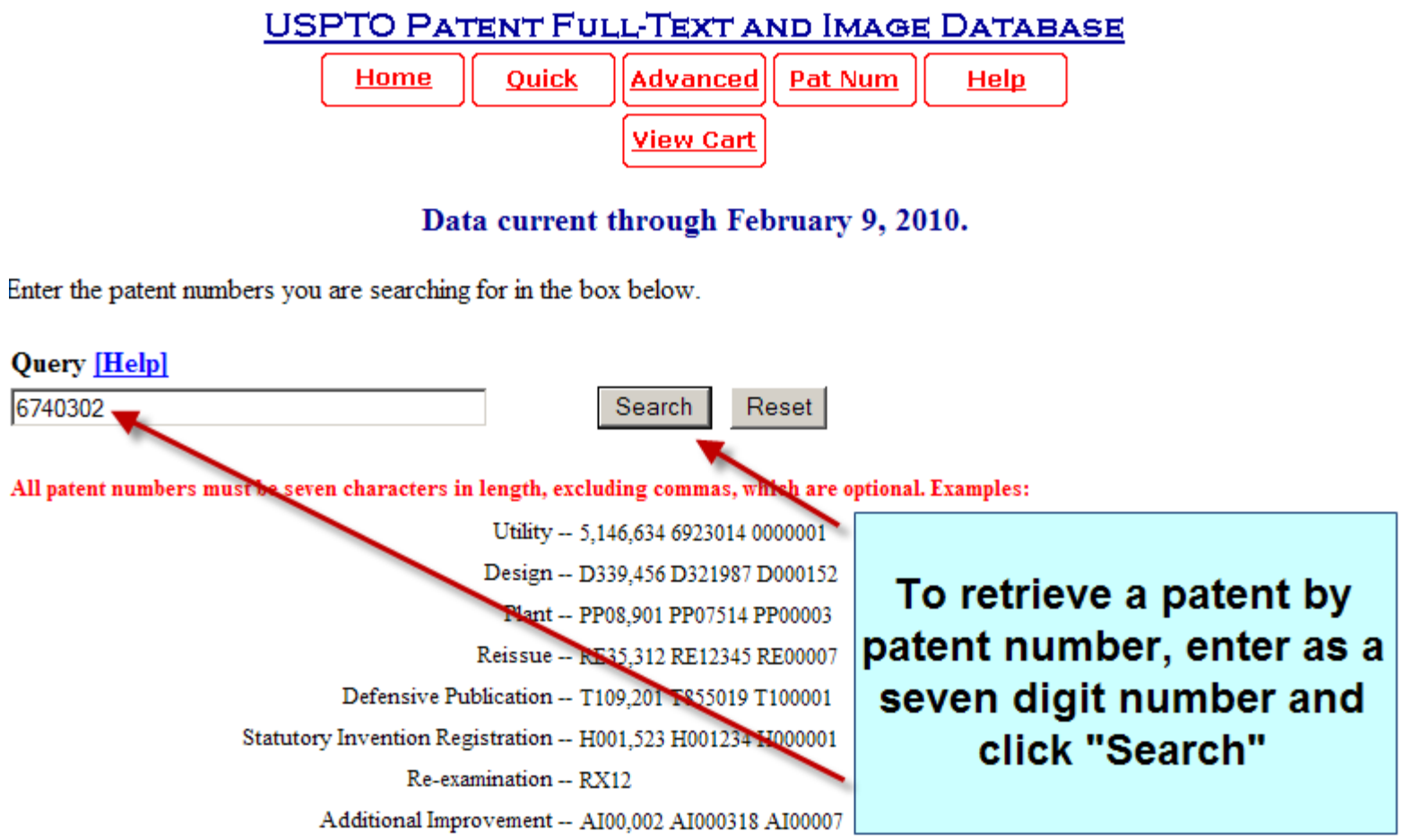

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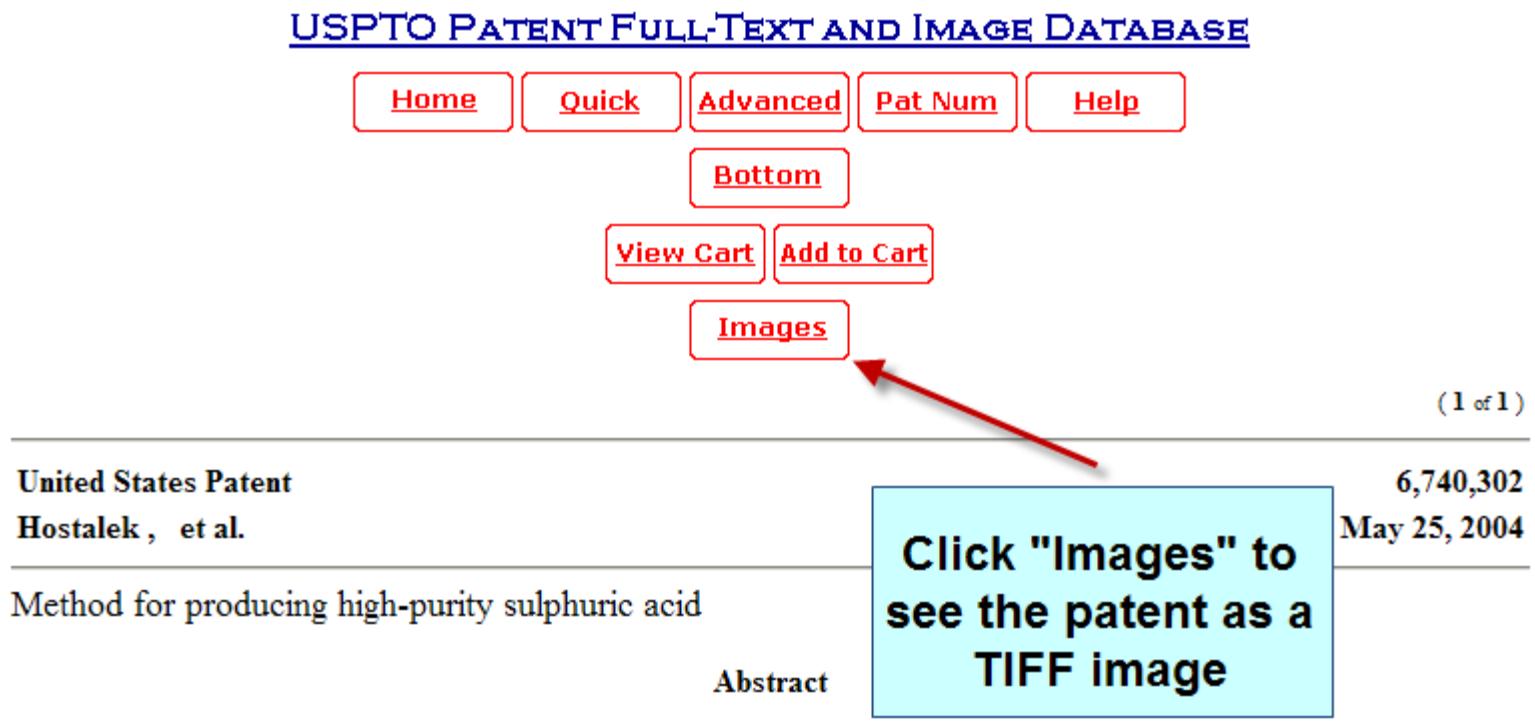

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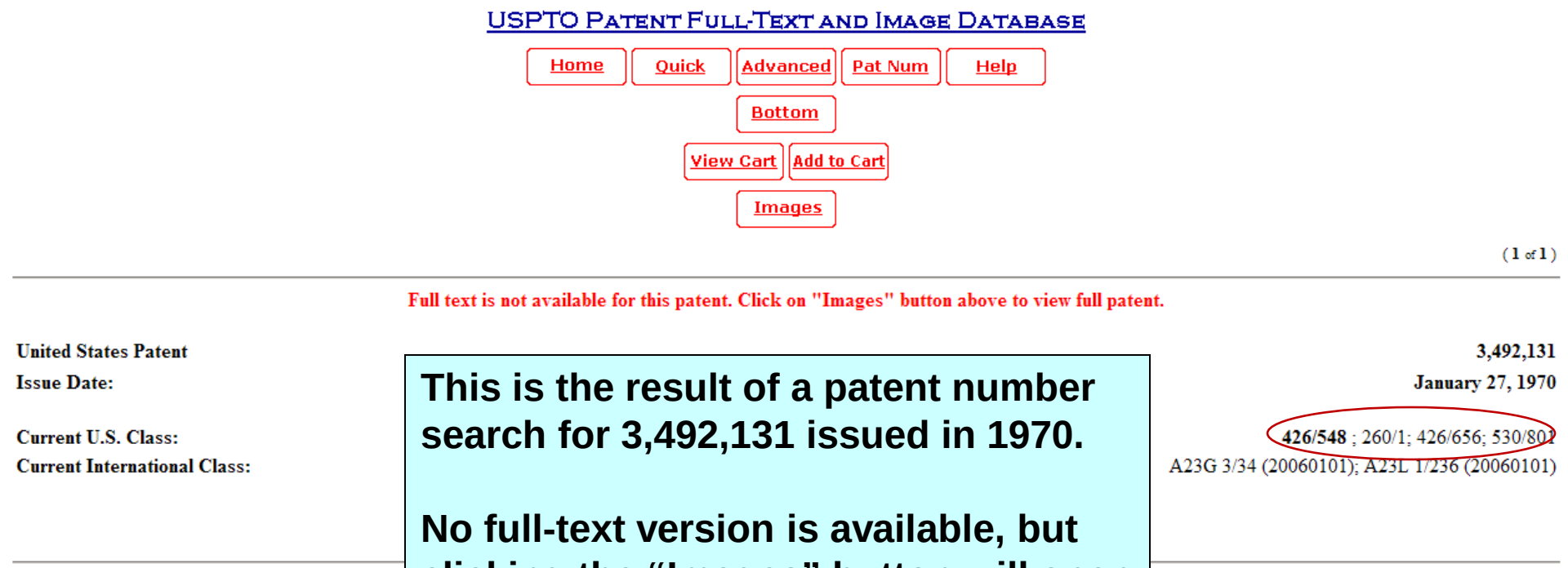

This is the result of a patent number search for 3,492,131 issued in 1970.

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Note that the Current U.S. Patent Classification. The TIFF image from 1970 shows an older classification.

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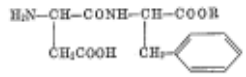
United States Patent Office 3,492,131 Patented Jan. 27, 1970

1 2

3,492,131
PEPTIDE SWEETENING AGENTS
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. A23l 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



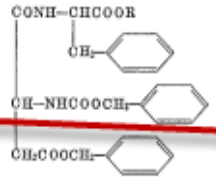
wherein R is a lower alkyl radical.

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 dipetides 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

group occurs to afford the protected dipeptide of the following formula



wherein R is a lower alkyl radical as exemplified above. Removal of the N-benzyloxycarbonyl 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-benzyloxycarbonyl - 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 catayst 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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Note: U.S. CL. 99-141 is an example of an older classification

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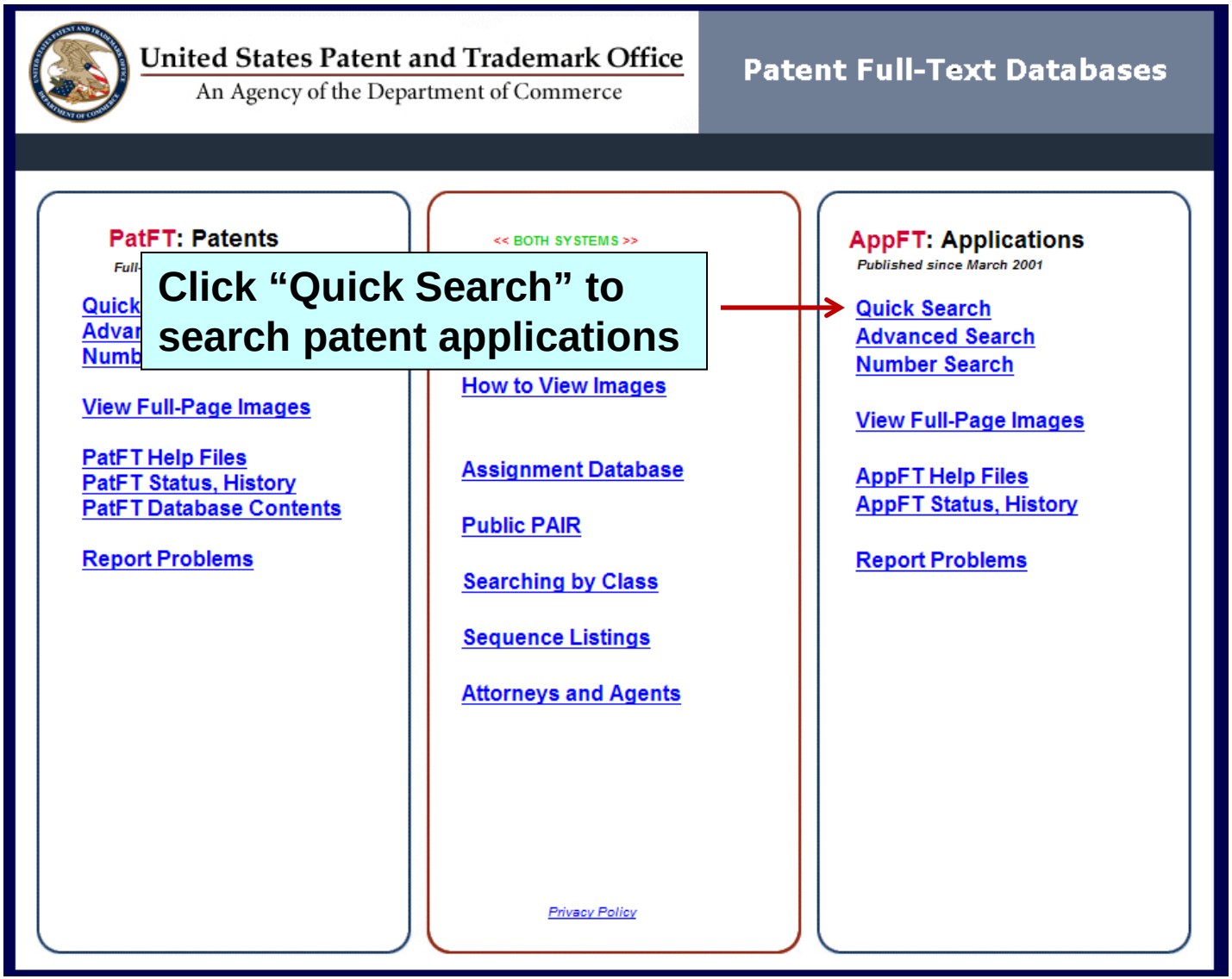

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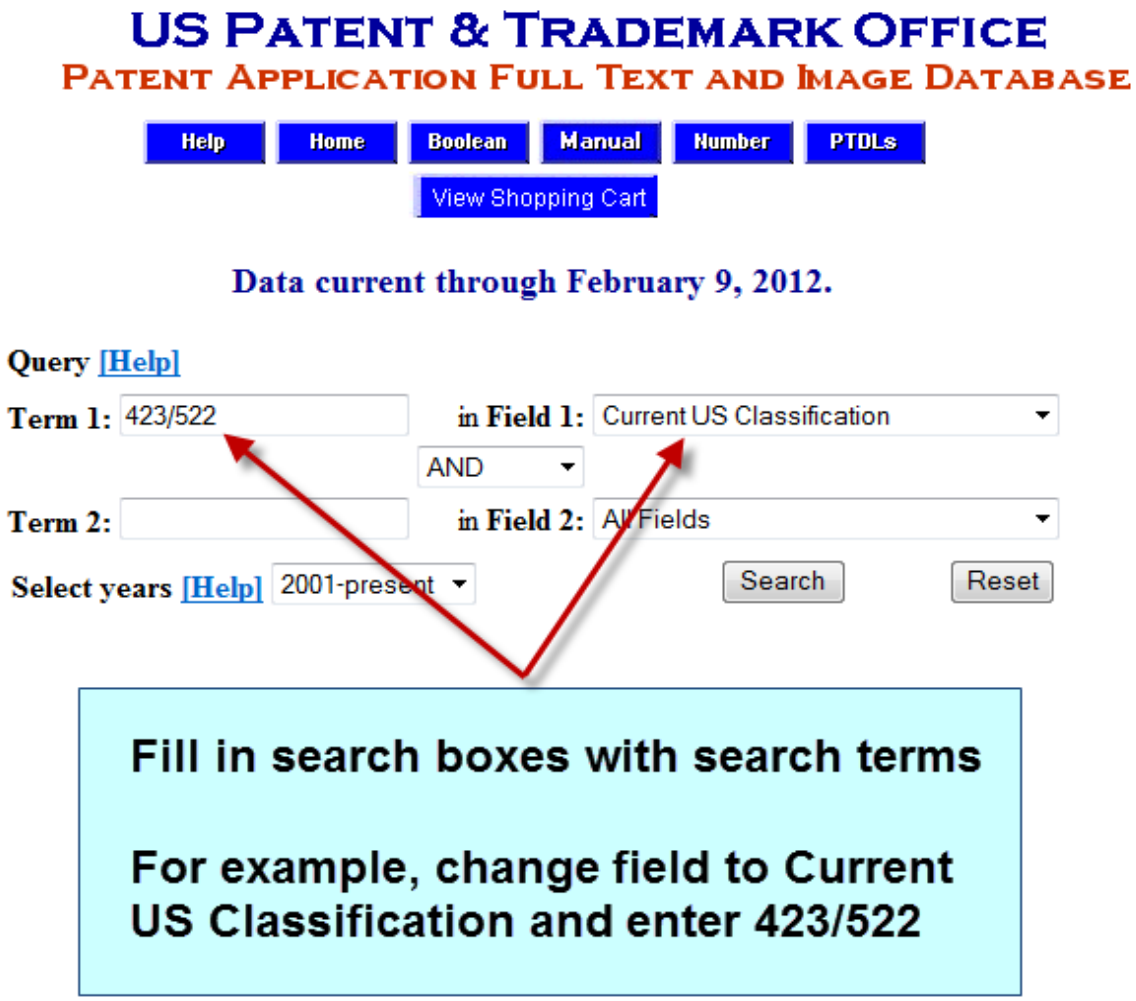

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2	20090123360	Inorganic hydrogen compounds
3	20080253957	Process and Plant for Producing Sulfuric Acid
4	20080226540	Ruthenium Oxide Catalysts for Conversion of Sulfur Dioxide to Sulfur Trioxide
5	20080044342	Fail-safe, on-demand sulfurous acid generator
6	20080044341	Sulfurous acid mist and sulfur dioxide gas recovery system
7	20070110663	Process for the production of sulfuric acid
8	20060245997	Process and plant for the manufacture of sulphuric acid from gases rich in sulphur dioxide
9	20060002845	Sulfuric acid process
10	20050255022	Emission control system

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US 20090178512A1

(19) **United States**

(12) **Patent Application Publication**
Partelpoeg

(10) **Pub. No.: US 2009/0178512 A1**

(43) **Pub. Date: Jul. 16, 2009**

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

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

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

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.

Publication Classification

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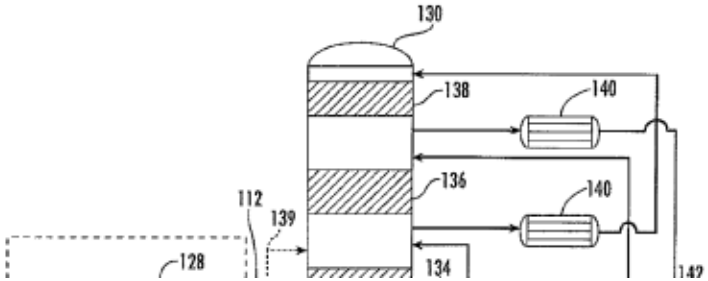
C01B 17/69 (2006.01)
B01J 19/00 (2006.01)

(52) **U.S. Cl.** **75/711**; 423/522; 422/161

(57) **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



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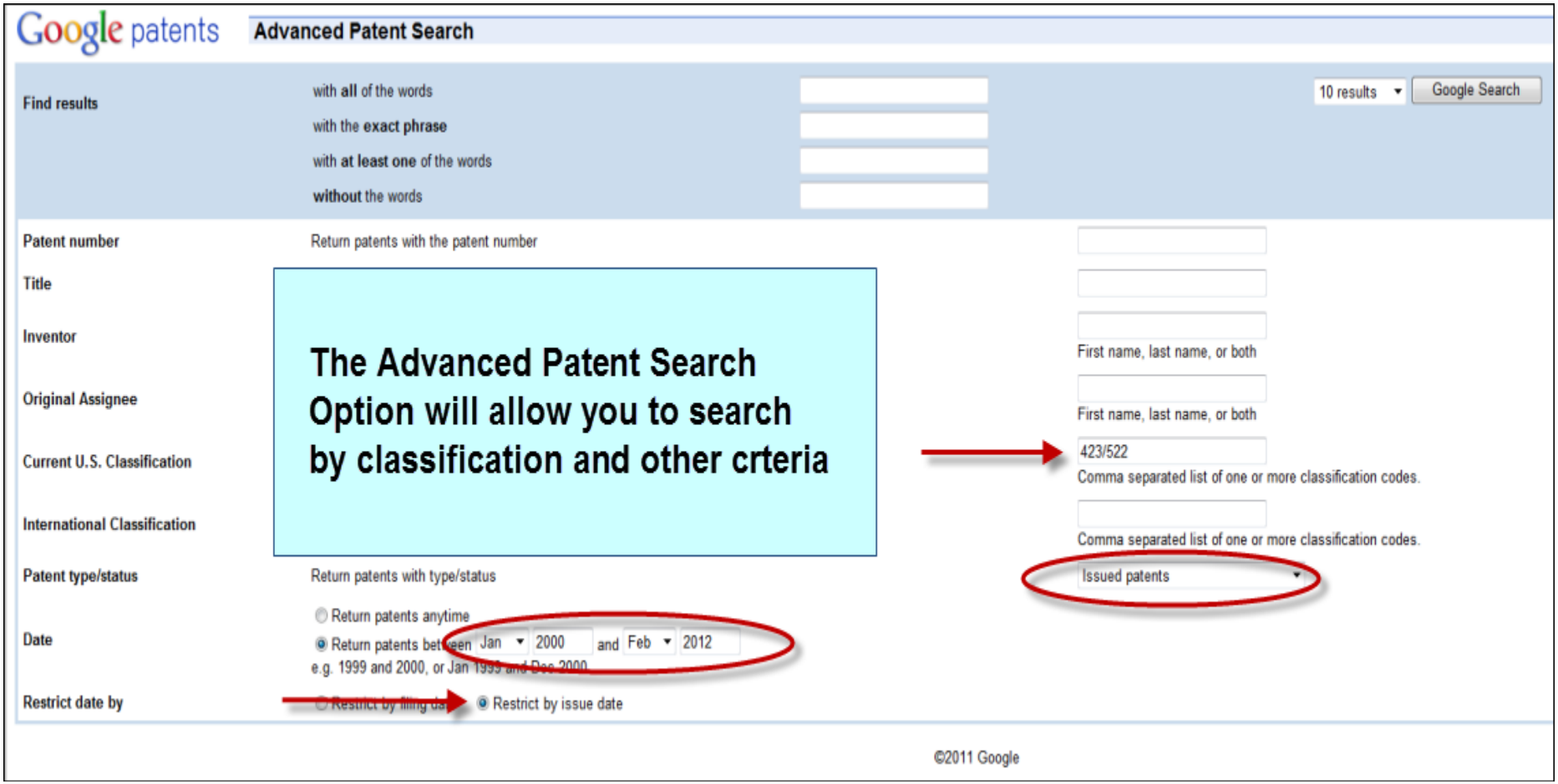

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  - Kirk-Othmer Encyclopedia of Chemical Technology
  - Ullmann's Encyclopedia of Industrial Chemistry
    - Ullmann's is little more Eurocentric, but these two sources are complementary.

Resources

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- What's the difference between these two numbers and why does it matter?
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