what every CHEMIST should know about PATENTS

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Foreword

The American Inventors Protection Act (AIPA) of 1999 introduced major changes in U.S. patent laws. Among the most significant changes:

- A U.S. patent application is no longer kept in secrecy by the U.S. Patent and Trademark Office (PTO), but is usually published 18 months after the application filing date.
- The term of protection granted for a patent is now adjusted to compensate for delays caused by the PTO during the examination of the application from which the patent was granted.

In addition to the changes enacted by the U.S. Congress in the AIPA, administrative initiatives taken by the PTO at the end of 2000 are also greatly changing the practice of patent law by providing for online filing of a patent application and making available for online review the PTO's internal data on the history and status of a patent application.

In light of all these changes, the Subcommittee on Education of the ACS Joint Board–Council Committee on Patents and Related Matters prepared this third edition of *What Every Chemist Should Know About Patents* to update the 1997 edition.

Review of the draft by the ACS Younger Chemists Committee is gratefully acknowledged, as well as support from the ACS Corporation Associates for the first print of this pamphlet.

Disclaimer

The purpose of this pamphlet is to provide you with a brief overview of patents. This booklet is for information only and is not meant to replace legal advice; we recommend that you direct legal questions to a patent attorney or patent agent on the register of the U.S. Patent and Trademark Office.

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Introduction

A chemical scientist or engineer may encounter patents in various ways:

- as citations in the chemical literature;
- when preparing a patent application, as an inventor or in some other capacity;
- when involved in the negotiations for licensing a patent; or
- in the case of a patent that is the subject of litigation, when
  - helping to assert the patent against an accused infringer, for example, by analyzing a product to help establish infringement;
  - helping to defend against a charge of infringement of the patent;
  - serving as an expert witness; or
  - helping to attack or defend the validity of the patent.

A patent is a form of property known as intellectual property. It can be licensed, sold outright, exchanged, or even given away. Some patents prove to be commercially valuable; others may have little or no commercial value.

Statistics on the number of U.S. patents granted every year are available from the PTO, in print and on the PTO's website at www.uspto.gov. The data are presented in various formats. For example, a ranked listing of all organizations granted 30 or more patents in a year is available, as well as a breakdown of U.S. patents by country of origin.

Patenting nowadays is very much an international phenomenon. In the following discussion, the main focus is on U.S. patents, with brief comments on patenting procedures in other countries.

The basis for U.S. patent rights

U.S. patent rights are based on Article I of the U.S. Constitution, which states that the Congress "shall have power ... to promote the progress of science and useful arts, by securing for limited times to … inventors the exclusive right to their … discoveries."

The patent laws enacted pursuant to this clause of the U.S. Constitution provide that "whoever invents or discovers any new and useful process, machine, manufacture, or composition of matter, or any new and useful improvement thereof, may obtain a patent therefor, subject to the conditions and requirements of this title" (35 U.S.C. § 101).

Patent rights are commonly viewed as a necessary incentive for private investment in scientific and technical research and development. The justification for the patent monopoly is an issue that is revisited periodically, particularly in developing countries faced with major public health crises where patents may be viewed as unwarranted obstacles in the efforts to address those crises. Nevertheless, patenting is becoming increasingly accepted in the global economy.

In return for the grant of U.S. patent rights, an inventor is required to describe the invention in a patent in a manner sufficient for others to practice the invention. After the term of protection granted with the patent has expired, anyone can practice the invention freely, which may benefit society at large. Disclosing the invention in a patent, as opposed to keeping the invention secret, may also spur others to improve on the patented technology and produce yet another invention that may also benefit society.

Subject matter patentable in the United States

As indicated in the language of the patent statute cited above (35 U.S.C. § 101), the following subject matter may be patented in the United States: a process, a machine, a manufacture (i.e., a manufactured product), or a composition of matter.

In the chemical and allied fields, a patent may protect any of the following subject matter that falls into at least one of the categories cited above:

- a compound, which may be defined as a class of compounds or as a specific compound or group of specific compounds;
- a composition containing the compound;
- an article made from the compound or containing the compound;
- a process for preparing the compound; or
- a method of using the compound for a specific purpose (e.g., for curing a polymer system or for treating a medical condition).

Subject matter not patentable in the United States

The following subject matter cannot be patented in the United States because of specific prohibitions in the patent statutes or as a result of court decisions interpreting and applying those patent statutes:

- inventions useful solely in the utilization of special nuclear materials or atomic energy for atomic weapons; or

- laws of nature, physical phenomena or scientific principles, abstract ideas, purely mental processes, and mathematical algorithms.

Conditions for patentability in the United States

As set forth in 35 U.S.C. § 101, to be patentable, an invention must be new or *novel* and be useful or have *utility.* In addition, 35 U.S.C. § 103 requires that the invention be *non-obvious.* Together, the three requirements of utility, novelty, and non-obviousness are commonly known as the three statutory requirements for patentability in the United States.

One-year grace period in the United States

An application for a U.S. patent must be filed no later than 1 year after the earliest date on which

- the invention was disclosed in writing anywhere in the world to the public (such as in a paper delivered at a scientific conference or an article published in a journal); or
- the invention was offered for sale in the United States, such as by providing a nonconfidential sampling to another party; or
- the invention was actually sold in the United States.

If the inventor fails to file a U.S. application within that 1-year period (known as the grace period), the public disclosure, the offer for sale, or the sale of the invention prevents a valid patent from being issued to the inventor for that invention.

The one-year grace period is also available for a U.S. patent application which claims priority based on an earlier filed foreign patent application, in which case the foreign application must be filed within the one-year grace period. However, there is no similar grace period available for patent applications in most other countries. (See “First-to-invent patent system” on page 13).

Description requirements for U.S. patent application

An invention that meets the three statutory requirements of utility, novelty, and non-obviousness nevertheless cannot be patented unless the patent application for the invention satisfies the following requirements of 35 U.S.C. § 112:

- the application must contain a written description of the invention;
- the description must be sufficient to enable any person skilled in the art to which it pertains to make and use the same; and
- the description must set forth the best mode contemplated by the inventor of carrying out his or her invention.

These description requirements are meant to ensure that the patent applicant delivers on his or her end of the bargain, by which a patent monopoly is granted in return for the public disclosure of the invention.

U.S. patent rights are rights of exclusion

A U.S. patent grants to an inventor the right to exclude others from making, using, importing, or selling the patented invention for a specified period of time (i.e., the term of the patent) in the United States. *A patent does not give the patentee the right to practice the invention.* In some cases, the ability to practice the patented invention may be circumscribed by factors such as the existence of patents for the same technology that are owned by other parties, or the existence of restrictions imposed by laws other than patent laws.

Three types of U.S. patents

Three types of patents are granted in the United States:

- utility patents, which constitute the greatest portion of U.S. patents and generally provide the strongest form of patent protection;
- plant patents, which could be considered a subgroup of utility patents granted exclusively for asexually reproduced new plant varieties (i.e., plants reproduced by methods other than seeds [*1*]); and
- design patents, which provide protection for a shorter term than utility patents for the ornamental designs of articles having a practical utility.

Who is an inventor?

Under U.S. laws, an inventor is defined as the person who originated the mental conception of an invention and completed the reduction to practice of the invention.

The mental conception must be a complete conceptualization of how to proceed for completing the invention, as opposed to being a mere idea without a specific plan for realizing the idea. The reduction to practice is the actual making of the invention.

The mental conception must be the inventor's own. In contrast, the physical steps necessary to complete the reduction to practice of the invention may be performed either by the inventor or by another person under the direction of the inventor. U.S. laws also consider the filing of a patent application to be a form of reduction to practice of the invention, that is, the filing of the application constitutes constructive reduction to practice of the invention.

Overview of the U.S. patenting process: An example

For illustration, the process for obtaining a U.S. patent is described in this section for an invention made by inventor A in organization X. This example shows a typical path for illustration. In practice, the patenting process before the PTO may vary greatly from case to case.

- Inventor A conceives of the idea (i.e., thinks of the idea) for an improved method of making a compound in a greater yield than was previously possible. In this case, inventor A writes a proposal that describes the steps of the synthesis, and the proposal is reviewed and approved by a supervisor. (Note: The inventor's proposal is evidence of the conception of the invention by inventor A.)
- Inventor A carries out the synthesis of the compound with the help of a laboratory technician who conducts the experiments as instructed by inventor A. The experiments are recorded in a laboratory notebook kept by the technician. The notebook entries are dated and witnessed daily by a co-worker of inventor A who is not working on the synthesis project. (Note: The performance of the synthesis by the technician under the direction of inventor A constitutes reduction to practice of the invention.)
- Inventor A prepares an invention disclosure by following the guidelines and using the form provided by the patent administrator of organization X. The invention disclosure identifies the pages of the laboratory notebook where the experimental results are recorded.
- A committee of organization X, of which the patent administrator is a member, reviews all the invention disclosures received in the last calendar quarter for inventions made in organization X. The committee selects the improved synthesis developed by inventor A for patenting.

The patent administrator has a patent application prepared by a patent attorney or patent agent, who may work directly with inventor A in addition to interacting with the patent administrator in preparing the patent application.

The text of the patent application must have at least the following parts:

- a description of the invention, known as the specification, that is sufficient to illustrate the invention and to show a person in the same technical field how to make and use the invention defined in the claims of the application. The description may include a discussion of the technical/scientific background and some experiments illustrating the invention;
- at least one claim, worded in such a precise and clear manner that the subject matter protected by the patent can be readily ascertained from the claim; and
- an abstract of the invention described in the application.

Some applications may also contain figures of line drawings or photographs, amino acid sequences, or computer program text, as appropriate.

Once a patent application has been filed with the PTO, it cannot be modified unless the modification is considered to add no "new matter" to the application. Only revisions of an editorial nature and those supported by the original application (i.e., revisions that are inherent in or evident from the original text) are permitted. The text of the patent application eventually becomes the text of the patent that issues from the application, with the incorporation of any changes made during the PTO's examination of the application.

Although an inventor may file and prosecute a patent application (*2*), experience has shown that it is important for the application to be prepared by a patent professional to minimize defects in the application at the onset. The patent professional may be a patent agent or patent attorney. A person with sufficient appropriate experience, although not recognized and registered by the PTO as an agent or attorney, can help with the application preparation but cannot represent the inventor before the PTO.

Although the assistance of a patent professional is recommended for the preparation of the patent application, the input of the inventor is of utmost importance. With knowledgeable participation by the inventor, it is possible to write a patent application strategically to protect those aspects of the invention that are potentially most important, and to make it difficult for the competition to circumvent the patent.

- A U.S. patent application for inventor A's invention is filed by submitting to the PTO the application papers that include at a minimum:
  - the text of the application, including the specification, claims, abstract, and any drawings, photographs, sequence listing, or computer program text, as appropriate;

- an oath or declaration for patent application; and
- the application filing fee.

The oath or declaration is a statement signed and dated by inventor A declaring that inventor A believes he or she is the sole inventor of the invention described and claimed in the application. The declaration also acknowledges the inventor's duty to disclose to the PTO any information known to the inventor that may affect "materially" the patentability of the invention and should be considered by the patent examiner during the examination of the application. Filing of the declaration may be deferred until receipt of a notice from the PTO requiring submission of the declaration together with a surcharge for the late submission.

The payment of the application filing fee may also be deferred until receipt of a notice from the PTO requiring payment to complete the application filing. Information on the application filing fee and other PTO fees is available from the PTO's website (www.uspto.gov).

In this illustration, because inventor A's employment contract with organization X specifies that all inventions made by A in the course of A's work for organization X belong to the organization, an assignment form is signed and dated by inventor A, assigning (i.e., transferring) A's rights in the invention to organization X. The assignment is submitted to the PTO, which records the transfer. This assignment may be submitted either at the same time as the original application papers or at a later date. The assignment is kept in separate records at the PTO and is not placed in the PTO's file for the application. The public can search the PTO's assignment records to determine the ownership of a particular patent.

- The PTO assigns the application a filing date, a serial number, a confirmation number, and a projected publication date. That information is printed on a filing receipt, which is sent by the PTO to the correspondence address given in the application. Ordinarily, the filing receipt also indicates that a foreign filing license is granted, which authorizes the applicant to file a patent application for the same invention in foreign countries.
- All foreign patent applications for the same invention must be filed within a year from the filing date of the U.S. application, to benefit from the filing date of the U.S. application under the terms of the Paris Convention. The patent attorney (or patent agent) and the patent administrator enter into their patent calendar dockets the deadline for foreign filing, that is, for filing patent applications for the same invention in other countries. A selection is made well in advance of that deadline of the countries and/or regional patent systems (such as the European Patent Office [EPO]) in which corresponding applications will be filed. Instructions are sent to patent agents registered in those countries (or in the regional patent organizations serving those countries) to prepare and file applications based on the U.S. application.
- In this illustration, the U.S. patent application was filed on or after November 29, 2000, which means that the application will be published as provided by the AIPA.
- The U.S. application is assigned by the PTO to a patent examiner. Patent examiners have backgrounds in science or engineering and receive training in patent examination from the PTO. Some examiners may also be part-time law students or attorneys. The examiner reviews the application with particular emphasis on the claims and conducts a search of the prior art, that is, the body of published information existing at the time of the filing date of the application. This prior art search nowadays is conducted electronically, although experienced examiners often have developed their own collection of paper copies of prior art relevant to their field of examination. The examiner selects a number of references (i.e., publications) considered to be particularly relevant to the invention as defined in the claims of the application, and cites those references on a notice of references cited.

  As in a typical case, the examiner issues a first office action, in which all or some of the claims of the application may be rejected as being unpatentable over one or more of the references listed on the notice of references cited. This type of rejection is known as a prior art rejection. Such prior art rejection may be on the grounds of anticipation, meaning that the examiner considers a single cited reference to describe all the features cited in the rejected claim. The rejection may also be on the grounds of obviousness, when the examiner considers that the subject matter of a claim would have been obvious from the teachings of one or more cited references, none of which taken individually shows completely all the features cited in the rejected claim.

  In addition to rejections based on prior art, the examiner may reject the claims for failing to define the claimed invention in sufficiently definite terms. The claims may also be rejected because the specification fails to provide a sufficient description of the invention being claimed or the best mode for practicing the invention. The examiner may also reject the claims on the grounds that the *utility* of the claimed subject matter has not been demonstrated convincingly, or the claimed subject matter is not the type of subject matter that can be patented under U.S. laws.

  In summary, the application examination process determines whether the claimed invention meets the statutory requirements for patentability (novelty, non-obviousness, and utility), and

whether the application meets the subject matter and description requirements mentioned above.

- The patent attorney/agent reviews this first office action and discusses the rejection with inventor A and the patent administrator for organization X. An agreement is reached among them on the approach to be followed for responding to the office action.

  The patent attorney/agent prepares a response (often referred to as an amendment) that is submitted to the PTO within the 3-month deadline set in the office action. The response presents arguments to rebut the assertion made by the examiner that the claims are not patentable over the references cited in support of the rejection of the claims. The response may also request that the claims be amended, in those cases where the patent attorney/agent and the applicant (i.e., the inventor and the inventor's employer) believe that the subject matter described in the prior art reference(s) cited by the examiner is fairly close to the subject matter of the rejected claims.

  The amendments and the arguments must be based on information already present in the originally filed application or on information known in the technical field of the invention at the time of the filing of the patent application. In addition, new information (usually in the form of experimental data) can be submitted to demonstrate the properties and/or operation of the invention. This new information has to be in a special format called a declaration or affidavit (*3*) signed by the person who obtained the experimental data or supervised the work of obtaining the experimental data. The declaration is not incorporated into the text of the patent issuing from the application, but is placed in the PTO's examination file for the patent (commonly known as the file wrapper or prosecution history for the patent).
- Within approximately 18 months of the U.S. application filing date, the PTO publishes the application. The publication takes place by way of posting on the PTO's website, without a hard copy publication. The published application enters into the body of prior art that is applied against other applications filed in the PTO after the filing date of inventor A's application or after the date of publication of inventor A's application.
- After reviewing the response filed by the attorney/agent in inventor A's application, the examiner issues a second office action, in which the examiner maintains the rejection of some of the claims but allows the remaining claims. This rejection is made final, which means that the applicant now has the option of appealing the rejection to the PTO's Board of Patent Appeals and Interferences. However, in practice few applicants appeal immediately, choosing instead to submit for the examiner's consideration a response to the final office action.

  In this illustrative example, only some of the claims are rejected and the remaining claims are allowed. The patent attorney/agent prepares a response, which specifically addresses and rebuts the comments made by the examiner in the final office action about the arguments previously presented by the attorney/agent.

  Note: If this second response fails to convince the examiner to allow the application, the applicant may proceed to the appeal stage before the Board of Patent Appeals and Interferences, or may choose to avoid the appeal stage by filing a request for continued examination (RCE) of the application or by filing a continuation application. By paying the fee for the RCE or the continuation application (which are essentially the same as the filing fee for the original application), the applicant in effect can "buy" a second round of examination for the application (*4*). Another option in this case is to cancel the rejected claims and let the application issue as a patent with the allowed claims.

  In summary, the give-and-take process between the applicant and the examiner often helps to narrow the issues and results in a meeting of the minds as to which aspects of the invention are patentable. This process is commonly referred to as the prosecution of the application.
- Upon consideration of the response to the final office action, the examiner decides in this case to allow all the claims of the application and issues a notice of allowance that sets a 3-month period for payment of the issue fee and payment of the application publication fee (*5*).
- If the patent attorney/agent pays the issue and publication fees in a timely manner, then the patent is issued a few months later. In this illustration, since there had been some delays in the PTO's handling of the application, a patent term adjustment (PTA) of 87 days is granted for this patent under the provisions of the AIPA. The PTA compensates the applicant for delays caused by the PTO in the examination of the application, from which are subtracted any delays caused by the applicant. The PTA is indicated on the cover page of a patent, but without any details on its calculation, which may be rather complicated. The applicant's representative may double-check that calculation by accessing online the PTO's application database, using the Patent Application Information Retrieval (PAIR) software provided by the PTO and a digital certificate that may be obtained from the PTO by the attorney/agent.
- The patent is added by organization X to its account with an annuity payment service (operated by a commercial entity). Before the first maintenance fee is due for payment (3½ years after the issuance of the patent), the annuity payment service sends reminders to organization X, which instructs the service to pay the first maintenance fee. The value of the patent to organization X is

reassessed each time a patent maintenance fee is due. A decision may be reached at some point to not pay a maintenance fee and allow the patent to lapse because the value of the patent is outweighed by the increasing cost of the maintenance fee (the second maintenance fee is higher than the first maintenance fee, and the third maintenance fee is even higher).

What does a U.S. patent look like?

Under the AIPA, publication of a patent application precedes its issuance as a patent. However, chemists and engineers are more familiar with patents than patent application publications. For that reason, an issued patent is described first in the following section, before an application publication is described.

The original patent grant, sometimes referred to as letters patent, is a ribboned official document issued by the PTO bearing the seal of the United States. The PTO issues patents every Tuesday. The official letters patent is sent to the patentee, and the PTO places copies into public circulation. A copy of a U.S. patent, now expired, is reproduced in its entirety in Appendix 1. This patent was selected because its text is relatively short. It serves as an example of a U.S. application that claims priority based on a foreign application (as indicated on the cover page of the patent); its claims illustrate a variety of chemical subject matter to which a patent claim may be directed.

The format of a U.S. utility patent, as prescribed by the PTO, includes the following consecutive parts, which are all derived from the patent application:

- A cover page, showing at a minimum the patent number and issue date, the title of the invention, the names and residences of the inventors, the application number and filing date, the classification assigned to the patented technology, the references cited in the examination of the application, the name of the examiner for the patent, and an abstract describing the invention. The cover page may also cite any prior applications having an earlier filing date, the benefit of which is claimed in the patent. The assignee and the attorney for the patent may also be listed on the cover page.
- Drawings, photographs, sequence listings, or computer programs (not found in all patents).
- A specification, that is, a body of text describing the invention and corresponding to the specification originally filed for the patent application.
- At least one claim that defines the subject matter covered by the right of exclusion granted by the patent. The claims are the most legalistic aspect of a patent because they define the property held by the patentee. The invention claimed in the claims may be of the same scope as, or narrower than, the invention disclosed in the specification. Subject matter disclosed but not claimed is not protected by the patent, and the patent owner cannot prevent others from using it (*6*). However, the publication of that subject matter is prior art against subsequently filed patent applications.

  The patent illustrated in Appendix 1 has claims directed to
  - a phosphite compound, defined generically (claim 1);
  - a stabilizer combination (i.e., a composition) containing the phosphite compound (claims 2 and 3);
  - a process of using the phosphite compound to stabilize polymers (claim 4 and claim 5, which depends on claim 4); and
  - a plastic molding composition (claim 6) containing the phosphite as a stabilizer.

What does a published U.S. patent application look like?

All U.S. patent applications filed on or after November 29, 2000, are published by the PTO approximately 18 months after the application filing date, except for applications by independent inventors who have requested an exemption from publication. Patent applications are published every Thursday. The very first patent application published by the PTO (on March 15, 2001) is shown in Appendix 2. In appearance, a published application is very much like an issued patent. After an application is published, a copy of the papers in the application file kept by the PTO may be obtained for a fee from the PTO, ending the secrecy under which an application was previously kept.

How to obtain information on patents and published patent applications

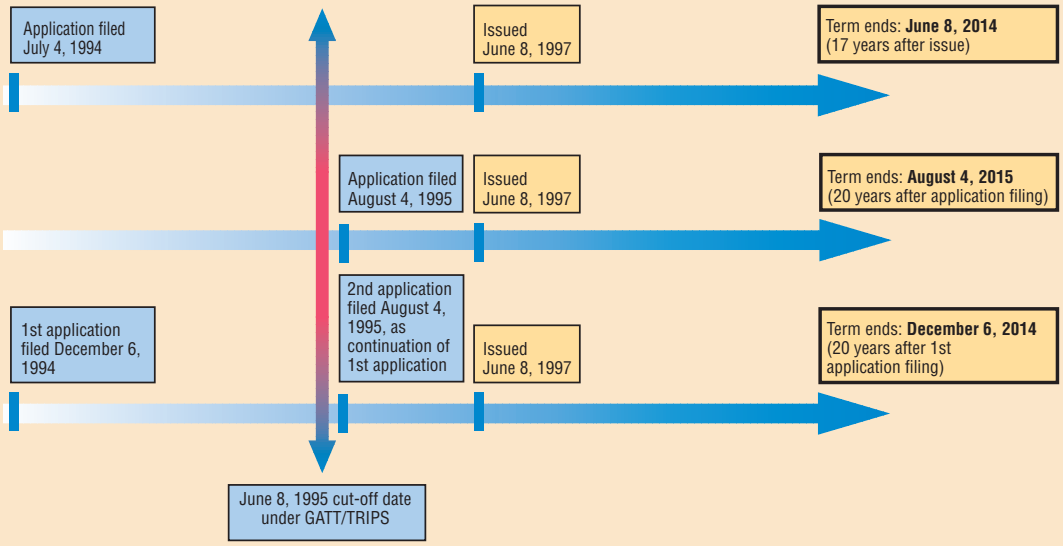
The text of U.S.-issued patents and published patent applications is available online from the PTO's website (www.uspto.gov), both in searchable full-text format or as images of the actual patent documents. The same information is also available from commercial database operators. Copies of the patents and published applications can be purchased directly from the PTO or from commercial database operators and other vendors.

Collections of U.S. patents are available in every state of the United States from a number of university libraries and public libraries known as patent and trademark depository libraries (PTDLs) affiliated with the PTO. For information on those libraries, visit the PTO website or consult the ACS book, *Understanding Chemical Patents* (see the Resources section at the end of this booklet). The website of a PTDL that offers a tutorial for searching patent databases is also listed in the Resources section (see University of Texas at Austin).

U.S. patent term calculation

Transition from the 17-year patent term to the 20-year patent term

The following examples illustrate how three patents issued on the same date may have three entirely different patent terms as a result of the changes brought by the GATT/TRIPS legislation. The length of the patent term depends on the *filing date* of the application from which a patent issued and on whether the patent issued from an application that is a continuation or division of an earlier application.

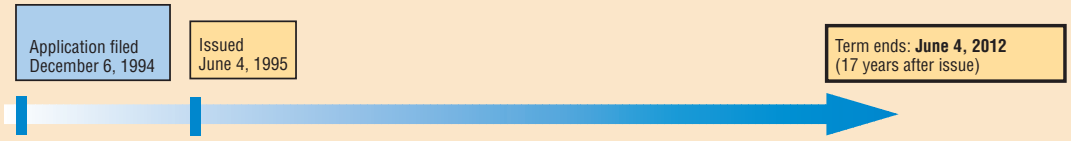


In the above scenarios, the 17-year patent term applies to applications filed before June 8, 1995. The 20-year method for calculating the patent term applies to applications filed on or after June 8, 1995.

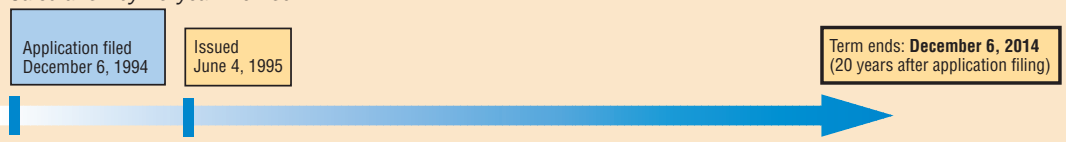
For patents in force on June 8, 1995

For patents issued before June 8, 1995, that are still in force on that date, the patent term is the longer of the term calculated by the 17-year method and the term calculated by the 20-year method, as shown in the following example.

Calculation by 17-year method:



Calculation by 20-year method:



As seen in the diagrams, the 20-year method of calculating the patent term results in a longer term than is obtained by calculation according to the 17-year term. Therefore, the term of the patent ends 20 years after the application filing date. This patent in effect received an extension of more than two years (from June 4, 2012, to December 6, 2014) of its patent term as a result of the enactment of the GATT/TRIPS legislation.

Patent term adjustment (for delays in application examination)

The GATT/TRIPS legislation made available a limited PTA, which the AIPA in effect broadened. A patent for which a PTA has been granted shows on its cover page the exact number of days of PTA. For example, a patent may have received a PTA in the amount of 23 days. In calculating the term for that patent, the 20-year term is first calculated. Then 23 days are added to that patent term to obtain the extended patent term.

Patent term extension for pre-marketing clearance

In addition to the PTA described, which compensates for delays caused by the PTO during the examination of the application, a patent term extension is available to compensate for postponement in the marketing of a patented product until regulatory pre-marketing clearance is received, such as in the case of pharmaceuticals undergoing review by the Food and Drug Administration. The PTO does not automatically calculate and grant such a patent term extension; it can be obtained only by submitting an application accompanied by supporting documents. In contrast, the PTA, which compensates for delays in examination of an application by the PTO, is automatically calculated by the PTO and printed on the face of the patent.

The PTO website provides a wealth of other information useful to a newcomer to patent law as well the experienced patent professional.

How to calculate the term of a U.S. patent

Until enactment of the GATT/TRIPS (General Agreement on Tariffs and Trade–The Agreement on Trade-Related Aspects of Intellectual Property Rights, Including Trade in Counterfeit Goods) legislation in 1994, the term of a utility patent or a plant patent *began on the date of issue* (grant) by the PTO and *ended 17 years after the issue date.* That simple-to-calculate patent term changed, however, with the enactment of the GATT/TRIPS legislation.

For a patent issuing from an application filed on or after June 8, 1995, the patent term still begins on the date of issue but ends 20 years from the effective filing date of the patent application from which the patent issued.

For (a) a patent issued from an application filed before June 8, 1995, or (b) a patent already issued that is still in force on June 8, 1995 (i.e., has not lapsed for failure to pay a maintenance fee), the patent term is the longer of (a) the term as calculated under the old rule (i.e., 17 years from the issue date), and (b) the term as calculated under the new method (i.e., 20 years from the filing date).

The AIPA made the calculation of a patent term even more complex, by providing a PTA to compensate for delays incurred during the PTO's examination of a patent application filed on or after November 29, 2000. (A more limited version of a PTA was already provided by the GATT/TRIPS legislation.) The PTA granted under the AIPA in effect guarantees a 17-year patent term as measured from the date of patent issue, to be reduced by any delays during the examination that are attributed to the applicant.

In summary, the term of protection granted now varies from patent to patent, depending on the following factors, among others:

- how much time was taken up in the PTO's examination of the application;
- how much of the delay during examination is attributed to the applicant;
- whether the application is a continuation or a division for which the effective filing date is the filing date of a prior application; and
- whether the application is the U.S. national stage of an international patent application, in which case the 20-year period begins with the international filing date (see Extending Patent Protection to Other Countries for a description of the PCT system and timeline).

A drawback of all these recent changes in the law governing patent term is that it is no longer possible for an uninitiated person to look at the face of a patent and determine whether its term has expired. Such determination now requires thorough consideration of rather complex rules. The PTO will print on the face of a patent the length of any PTA. However, the PTO will not print the expiration date on the face of the patent, apparently to prevent unwarranted reliance on that information in case a patent has lapsed before the expiration of its full term because a required maintenance fee was not paid.

The maintenance fees for a U.S. patent are due at three successive intervals after the issue date of the patent and increase incrementally with each interval. A patentee who no longer has an interest in maintaining a patent can allow the patent to lapse by failing to pay a maintenance fee. The PTO publishes a notice of that lapse in its weekly Official Gazette, which can be purchased in hard copy or may be viewed on the PTO website. See sidebar on page 9 for examples that illustrate the calculation of patent terms.

The option of electronic application filing

In 2001, the PTO made electronic patent application filing through the Internet available to all applicants, following completion of a test pilot program. For electronic filing, the text of an application must be in XML tagged format (prepared with word processing software available from the PTO). All drawings including formulas must be converted to TIFF documents. The declaration for patent application is also converted to a TIFF document. The application documents are sent in encrypted form to the PTO using software provided by the PTO.

The PTO expects that 80% of all patent applications will be filed electronically by 2006, and is expected to take actions as necessary to meet that target.

The option of filing a provisional application

The legislation implementing the GATT/TRIPS provisions introduced a new form of U.S. patent application called a provisional application. Unlike a regular U.S. patent application, the provisional application need not contain any claims. The government fee required for the provisional application is small compared with the fee for a regular application. The provisional application is not examined for patentability by the PTO and does not issue as a patent.

The intent behind the provisional application is to make it possible for an inventor to lock in an

application filing date at a lower cost and with less effort than would be required in the preparation and filing of a regular U.S. patent application. (In particular, a provisional application could be filed on short notice before the invention was disclosed to the public, e.g., before publication in a journal.)

Within 1 year of filing a provisional application, the inventor must file in all countries in which patent protection is desired, including the United States, a regular application for the same invention as described in the provisional application. The provisional application automatically expires 1 year after its filing.

A benefit to the applicant in filing a provisional application is that the 20-year period for calculating the patent term (for a U.S. patent issuing from the application) begins not with the filing date of the provisional application but with the filing date of the regular U.S. application that is based on the provisional application. At the same time, for the purpose of avoiding prior art references, the applicant benefits from the earlier filing date of the provisional application, yet without having the 20-year period for calculating the U.S. patent term be counted from the filing date of the provisional application. Another advantage is that any U.S. patent issuing from a regular application based on a provisional application is effective as prior art as of the filing date of the provisional application (as opposed to the filing date of the regular application) and thus may be more useful in preventing other parties from patenting similar inventions.

The provisional application can also serve as the basis for the filing of an application in a foreign country or the filing of an international application within 1 year of filing the provisional application (*7*).

A drawback of the provisional application procedure is that it may encourage applicants and their attorneys to prepare and file provisional applications with a lesser degree of care than would be given to a "regular" patent application, a fact that may be regretted later when the "regular" U.S. application and the foreign and/or or international patent applications are filed. It may be realized at that time that the provisional application may be lacking a fully adequate description of the invention to support the claim to priority (in those subsequent applications) for the earlier filing date of the provisional application.

Application fees

An application filing fee must be paid to the PTO when the patent application is filed. The basic filing fee is increased by a surcharge when the application contains more than 3 independent claims and/or more than 20 claims in total and also when the application contains 1 or more multiply dependent claims (i.e., a claim that depends on 2 or more other claims).

Additional fees may become due during the examination of the application, for example, fees for an extension of a response period when the applicant's required response to an action of the PTO is filed after the original deadline set by the PTO.

An issue fee is due after the application is allowed, and maintenance fees are due at successive intervals after the patent issues. The fee for the publication of the application under the provisions of the AIPA is also due after the application is allowed.

Most PTO fees are subject to a reduction of 50% for an application that qualifies for small entity status, that is, when

- the application is assigned to a company having no more than 500 employees or to a nonprofit organization, or
- the application is by one or more inventors who are under no obligation to assign the application to an organization that does not qualify for small entity status.

Proof of priority of invention (first to invent)

A patent application contains no information on the actual date of invention of the subject matter claimed in the application. In most cases, there is no need for the applicant to offer any information about the date of invention. However, in some instances the applicant may have to prove that the invention was made before the effective date of a reference cited by the examiner in support of the rejection of the claims of the application. This proof of being first to invent is also necessary in an interference proceeding when two or more inventors (or groups of inventors) are attempting to patent the same invention.

For the purpose of proving the date of invention, it is essential that the history of the development of the invention be recorded appropriately in laboratory notebooks. (See the ACS *Recordkeeping Fact Sheet* cited in the Resources section.)

Until recently, the proof of invention was limited to activities in the United States. For proving priority of invention based on activities outside the United States, only the act of filing a patent application in a foreign country was accepted as proof, and only when the U.S. application claims the benefit under the Paris Convention of the filing date of that foreign application. The legislation implementing GATT/ TRIPS in the United States has changed U.S. patent laws greatly by permitting a patent applicant to rely on records of activities outside the United States, in addition to relying on the filing of a foreign patent application, to prove priority of invention.

Disclosure document program of the PTO

The PTO provides a service for inventors by accepting and preserving for two years a *disclosure document* to serve as evidence of the date of conception of an invention. A minimal fee ($10) is charged for this service. If the disclosure document is not followed by the filing of a patent application that refers to the disclosure document, the PTO destroys the disclosure document two years after its receipt. The disclosure document program does not preserve an inventor's rights to an invention but merely provides a mechanism by which an inventor can prove a date of conception of an invention.

Statutory invention registration

In 1984, the U.S. Congress enacted legislation calling for the PTO to publish as a statutory invention registration (SIR) the text of the specification (and any drawings) of a regularly filed application for a patent under the conditions that the application meets the description requirements of 35 U.S.C. § 112 (discussed on page 4) and that the applicant waives the right to a patent and pays the required publication fee.

The SIR is published without examination, that is, without examination for patentability over the prior art. The SIR procedure is intended mainly for use by federal agencies to publish their research results and prevent another party from patenting similar research results and asserting the patent rights against the federal agencies. However, this form of defensive publication is also open to inventors outside of federal agencies.

The recent advent of patent application publication under the AIPA is expected to lessen reliance on the SIR publication, since the application publication not only achieves the same results as a SIR publication but also presents additional benefits. First, the right to a patent is not waived in the case of an application publication. Second, the fee for the application publication is not due until the application is allowed. No fee is ever paid for the application publication if the application is not allowed and the application is abandoned.

Enforcing a U.S. patent

The rights granted with a patent begin at noon on the day of issuance of the patent, and the patent owner can begin enforcement of the patent on that day.

Enforcement of a patent does not always require the filing of a lawsuit. When a patent owner believes that another party is infringing the patent, the patent owner usually has a study conducted to verify the validity of the patent and to confirm that the activities of the other party constitute infringement of the patent. Such a study is in the form of a legal opinion prepared by a patent attorney (*8*). After completion and review of the opinion, a warning letter may be sent to the alleged infringing party. The recipient of the letter may respond with an explanation of why there is no infringement. In other cases, there may be no response, at which point the patent owner must decide whether to further attempt to engage the other party in a dialogue. Such a dialogue may result in an agreement to settle the dispute and avoid litigation, which may be costly to both parties. In some instances, where the circumstances warrant it, a lawsuit is eventually filed.

Under U.S. laws, a patent is infringed when

- the technology practiced by the alleged infringer meets exactly all the terms of at least one claim of the patent (a case of literal infringement), or
- the technology practiced by the alleged infringer comes so close to the invention defined in at least one claim of the patent that the practice is considered to be infringement under the doctrine of equivalents. The determination of infringement under the doctrine of equivalents is difficult, because the court decisions on the subject do not provide clear-cut guidelines, and interpretation by the courts of law in this area is constantly evolving.

The new provisional patent rights

Under AIPA provisions, the publication of a U.S. patent application (or the publication of an international patent application that designates the United States and is published in English, as described in the section on "Extending patent protection to other countries") marks the beginning of the provisional rights that a patentee may enforce after a U.S. patent issues from the published application. The patentee may obtain reasonable royalties from a person who used the patented invention between the time of publication of the application and the issuance of the patent. These provisional rights are subject to a number of limitations, the most significant being that the invention claimed in the patent must be substantially identical to the invention as claimed in the published application. For this reason, upon request by the applicant accompanied by payment of a publication fee, the PTO will republish an application in its amended form so that the published claims are substantially identical to the claims in the patent that eventually issues from the published application.

The new first inventor defense for methods of doing business

The AIPA created a new defense, known as the first inventor defense, to protect a first inventor from a charge of infringement of a patent granted to another inventor for a business method. For this defense the first inventor must prove that he or she in good faith had reduced to practice in the United States the invention for that method of doing business, and had used that invention commercially in the United States before the filing date of the application by the other inventor that issued as the patent in question. Since the first inventor defense is limited to patents for methods of doing business, the immediate application of the first inventor defense to the work product of chemical scientists is considered at this time to be limited.

Post-issuance processes before the PTO: Reissue, reexamination

After a U.S. patent issues, the following two procedures provide for a renewed examination of the patent: a reissue application and a request for reexamination.

A reissue application is undertaken to correct a significant error in the application, usually in the claims, and can be filed only by the inventor or patent owner.

A reexamination request can be filed by anyone, including a third party, such as a competitor. The U.S. Congress enacted legislation in 1984 to permit the reexamination of the claims of a patent in light of prior art references that are relevant but were not considered in the original examination. The AIPA of 1999 expanded reexamination by providing for an optional *inter partes* reexamination in which a third party requester of a reexamination can participate throughout the reexamination process. Prior to the AIPA, the participation of a third party requester ended with the filing of a request for reexamination.

In any of these post-issuance reconsideration procedures (reissue, reexamination, or *inter partes* reexamination), the term of the original patent remains unchanged if the PTO concludes at the end of the reissue or reexamination procedure that the claimed subject matter is patentable.

First-to-invent patent system is unique to the United States: No grace period in other countries

The U.S. patent system is known as a first-to-invent system. It awards a patent to the party who is first to invent the subject matter claimed in an application (as opposed to the party who is first to file an application for the invention) as long as the application is filed within the year grace period discussed in the section "One-year grace period in the United States." In contrast, other countries have a first-to-file patent system that awards the patent to the party who is the first to file an application, regardless of whether that party was first-to-invent.

The most important thing an inventor planning to file patent applications in foreign countries should know is that there is no grace period in those countries. Most foreign countries will grant the benefit of the earlier filing date of the U.S. application to a foreign patent application that is filed within 1 year of the filing of the U.S. application. However, the patent laws of those countries require so-called absolute novelty for the invention at the time of the filing of the first application (i.e., the U.S. application in this case). That absolute novelty may be destroyed if there has been any public disclosure or sale of the invention before the filing of the U.S. application. Therefore, applicants must file their applications in the United States prior to any public disclosure or sale of the invention if they plan to seek patent protection outside the United States.

There are also practical reasons for filing a patent application as soon as reasonably possible. The sooner the application is filed, the sooner it can be used as prior art against patent applications filed later by others. Also, an earlier filing can be beneficial if the application becomes involved in an interference. In such a proceeding, the PTO is asked to (or may on its own initiative) declare an interference because similar subject matter is claimed in two applications, A and B, pending in the PTO at the same time, or an application A and a patent issued from an application B that was co-pending in the PTO at some point in time with application A.

The PTO determines in the interference proceeding which of the respective inventors (or groups of inventors) was first to invent the subject matter claimed in the applications or patent. The interference rules place the burden of proving prior inventorship on the party with the later-filed application. There is, therefore, a substantial advantage in being the first to have filed a patent application for the invention.

Finally, another reason for filing a patent application as soon as possible is that in the following two situations, a patent applicant must prove that due diligence was exercised (i.e., that there was no unreasonable delay) in completing and filing a patent application:

- when the application is involved in an interference proceeding, and the applicant is first in conception of the invention but last in filing a patent application; and
- when the claims in the application are rejected by the PTO examiner over a prior art reference, and the applicant had conception of the invention before the publication date of the prior art reference, but filed a patent application after the publication date of the reference.

Extending patent protection to other countries

A U.S. patent protects an invention in the United States only. To obtain worldwide protection, patent applications must be filed in the countries where patent protection is desired. Most patent-granting countries will give an applicant the benefit of the earlier application filing date in the United States. However, as discussed above, most countries also require absolute novelty as a condition for patentability.

Starting from a patent application filed in the United States, an applicant may choose from two scenarios (shown in the figure) for filing corresponding applications in foreign countries.

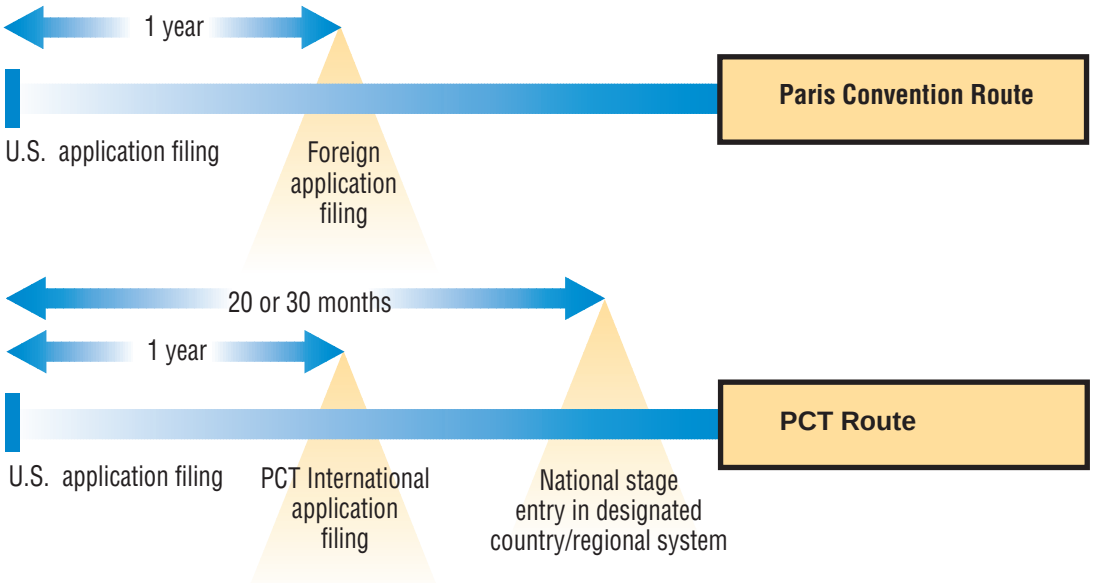
In the first scenario, known as the Paris Convention route, an application is filed within 1 year of the initial U.S. application filing in each of the selected foreign countries. In accordance with the Paris Convention for the Protection of Industrial Property, those countries grant to the applicant the benefit of the earlier U.S. filing date (*9*).

The second scenario, known as the PCT route, involves filing an international application under the Patent Cooperation Treaty (PCT) in one of the "receiving" offices of the World Intellectual Property Organization (WIPO; *10*). Applicants in the United States and other countries are increasingly using this PCT route. As shown in the figure (for a U.S. applicant), an international application is filed in the PTO, which serves as a U.S. receiving office. This international application is filed within 1 year of the initial U.S. application filing and claims priority under the Paris Convention based on the U.S. application. The international application designates the countries (or regional patent organizations such as the EPO [*11*]), in which the applicant wishes to obtain patents.

The international application is published by WIPO approximately 18 months after the international filing date (or the priority date if priority is claimed), the publication being in the language in which the application is filed. The published application shows the countries designated by the applicant, which informs other parties of the applicant's intention to pursue a patent in those countries.

Within 30 months (12) from the initial U.S. filing date, known as the priority date, the applicant must complete the formalities for entering into the "national stage" of the international application for each of the designated countries. Those formalities include paying the official filing fees and submitting translations (if necessary) of the international application into the languages accepted by the designated countries. Each of the countries in the PCT system accords to the "national stage" application the benefit of the earlier international application filing date, and in the case where the international application claims the benefit under the Paris Convention of an earlier domestic application (which is the U.S application in the second scenario shown in the figure) each country in the PCT system accords to the national stage application the benefit of that earlier priority date.

Two routes for filing applications in other countries



The entry into the national stages described above is according to the provisions of Chapter I of the PCT. The deadline under Chapter I was previously 20 months after the priority date. However, it was recently changed to 30 months for international applications for which the period of 20 months expires on April 1, 2002. As permitted by the PCT, certain PCT member countries (e.g., the U.K. and Japan) are not adopting immediately the new deadline because it conflicts with their national law.

For such countries, the deadline for national stage entry is still 20 months from the priority date until further notice. In such countries, a PCT applicant must file a Demand for International Preliminary Examination under Chapter II of the PCT if the applicant wishes to delay entry into the national stages. The deadline for such entry into the national stages under Chapter II is 30 months from the priority date.

A benefit of choosing the PCT route shown in the figure is that it permits an applicant to postpone the costs associated with filing applications in foreign countries and even postpone the final selection of those countries. For a relatively small "designation fee," an applicant in effect can preserve for a time period after the filing of the international application (8 months under current rules and 18 months under the revised rules effective April 1, 2002) the option of filing applications in all the countries belonging to the PCT system, of which there are currently about 115. Under current PCT rules, a further postponement up to 30 months from the priority date may be obtained by filing a demand (accompanied by a fee) for an international preliminary examination. After April 1, 2002, such demand is not even necessary for obtaining a 30-month period for entry into the national stages.

Patent laws differ from country to country, so it may be easier to obtain patents in some countries than in others. Unfortunately, whether effective enforcement of patent rights can be obtained also varies from country to country. Since patenting worldwide is very expensive, applicants usually weigh the prospects for successful enforcement when deciding where to seek patent protection outside the United States.

National security considerations: Secrecy order, foreign filing license

In its initial review of patent applications, the PTO may identify some applications as potentially affecting U.S. national security. Such applications are subject to further review, which in some instances may result in the imposition of a secrecy order for a period of at most 1 year (subject to extension by renewal of the secrecy order). During that period, the invention must be kept secret, and the grant of a patent on the invention is withheld.

A related security requirement is that no patent application may be filed in a foreign country on an invention made in the United States unless a patent application for the invention has been first filed in the United States and the applicant waits for at least six months before filing the foreign application or obtains a foreign filing license.

Ordinarily, the official filing receipt issued by the PTO for the application contains a notice to the applicant that a foreign filing license has been granted. Even if no official filing receipt has been received yet, the applicant is free to file an application in the foreign country at the end of the six-month period following the U.S. filing date.

Closing remarks

It is impossible to provide in this introductory booklet all of the information that may interest chemical professionals. A Resource section is provided for researching specific areas and general reading. The list is just a sampling of the many sources of information available that are constantly increasing. As with any information on the Internet, the reader must be alert and exercise good judgment.

In the AIPA, the U.S. Congress directed the PTO to serve as watchdog over invention promotion companies, some of which have been known to prey on independent inventors who come to them for help in promoting their inventions. The section of the PTO's website for independent inventors should be consulted for information on complaints lodged against invention promotion companies, as well as for general information on patents.

Text notes

(1) Plants reproduced by seeds may be protected by a Certificate of Plant Variety Protection issued by the U.S. Department of Agriculture. Plants created by biotechnology may be protected by regular utility patents (as opposed to plant patents).

(2) The word *prosecution* as used in patent prosecution has an entirely different meaning than it does in a criminal context. Patent prosecution refers to the interactive process that takes place between the PTO and the applicant during the examination of an application. In this context, prosecution in effect means solicitation.

(3) This declaration or affidavit, known as a declaration or affidavit under Rule 132, is different from the declaration or oath for patent application signed by the inventors to complete the filing of a patent application.

(4) The practice of filing a continuation application or obtaining another round of examination by other mechanisms provided by the PTO previously had no effect on the term of a patent issuing from the application. However, as a result of the enactment of the GATT/TRIPS legislation in

1994, such practice may shorten the patent term by prolonging the prosecution of the application.

(5) The AIPA permits the PTO to collect from an applicant a fee for the application publication only after the application has been allowed.

(6) This issue is currently under review *en banc* (i.e., by the entire court as opposed to a panel of three judges from the court) by the Court of Appeals for the Federal Circuit.

(7) There was some initial doubt about whether a U.S. provisional application could serve as the basis for a foreign or international application. An exchange of written communications between the U.S. Commissioner of Patents and Trademarks and the head of the World Intellectual Property Organization has confirmed that priority can be claimed in an international application based on a U.S. provisional application. However, some concerns still remain about whether the European patent court will grant the benefit of priority to a European application based on a U.S. provisional application.

(8) Patent agents are registered by the PTO and are authorized to represent a patent applicant in proceedings before the PTO but are not qualified to render opinions on the validity or the infringement of a patent.

(9) A similar filing may be effected in a country such as Taiwan, which does not adhere to the Paris Convention but grants priority based on U.S. patent applications.

(10) The World Intellectual Property Organization, an agency of the United Nations, is headquartered in Geneva, Switzerland, and administers the PCT system. See www.wipo.int/pct for an introduction to the PCT system.

(11) In a regional patent organization the application is initially processed and reviewed, with subsequent transfer of the proceedings to the national patent-granting organizations of individual countries that belong to the regional patent organization.

(12) In a recently adopted change effective April 1, 2002, this PCT deadline for entry into the national stages will be 30 months instead of 20 months.

Glossary of abbreviations and terms

AIPA	American Inventors Protection Act of 1999
CAFC	Court of Appeals for the Federal Circuit
EPO	European Patent Office
GATT-TRIPS	General Agreement on Tariffs and Trade–The Agreement on Trade-Related Aspects of Intellectual Property Rights, Including Trade in Counterfeit Goods
IP	Intellectual property
PCT	Patent Cooperation Treaty
PAIR	Patent Application Information Retrieval (software)
PTA	Patent term adjustment
PTDL	Patent and trademark depository library
PTO	U.S. Patent and Trademark Office
RCE	Request for Continued Examination
SIR	Statutory invention registration
USC	U.S. Code
WIPO	World Intellectual Property Organization

Abstract. The section of a patent that provides a brief summary of the invention described in a patent.

Allowance (of application). Decision by patent examiner at the PTO to allow the claims of a patent application.

Amendment (of part of application). A modification of a portion of an application, which may be in the specification, the claims, or the abstract.

Amendment or response. A written response to an office action of the PTO rejecting the claims of a patent application. The response may request that amendments be made to the application.

Annuity. A fee required to be paid annually to the patent-granting organization of a country to maintain a patent application or a patent. See **Maintenance fee** (for U.S. patents).

Anticipation. The basis for rejecting a patent claim over a prior art reference that shows all the features cited in the claim. See **Novelty.**

Application. A document submitted to the patent office of a country to describe an invention for which a patent is sought.

Application publication. The publication by the patent office of a country or by WIPO of an

application submitted by an applicant for patent. The publication ordinarily occurs 18 months after the filing date of the application, or 18 months after the earliest claimed priority date.

Assignment (of an invention). A document signed by the owner of an invention (the assignor) to transfer ownership of the invention to the assignee. See also **License (of patent).**

Best mode disclosure. A requirement in U.S. patent law that the inventor disclose in the patent application the best mode for practicing an invention.

Claim. A statement that defines the invention protected by a patent.

> **An independent** claim defines an invention without referring to another claim, for example, "Claim 1. A compound represented by the following formula (I)...."
>
> **A dependent claim** further defines the invention recited in another claim to which the dependent claim refers, for example, "Claim 2. A compound according to claim 1, wherein X is hydroxyl."
>
> **A multiply dependent claim** depends on more than one claim, for example, "Claim 3. A compound according to any one of claim 1 or 2, wherein Y is a heterocyclic aromatic five-membered ring structure."

Conception. The mental concept of an invention and how to make the invention.

Continuing application (or continuation application). An application that contains the same description of the invention as a prior filed application and claims the benefit of the filing date of the prior application. See also **Request for Continued Examination.**

Court of Appeals for the Federal Circuit. A U.S. federal court of appeals established in 1982 that reviews all appeals for patent cases from federal district courts.

Declaration (or affidavit) for presenting additional information. A formal statement, signed by the person making the statement who is not necessarily an inventor, that is optionally submitted to the PTO on behalf of a patent applicant during the examination of an application. The statement introduces additional information, for example, experimental data, to support the argument that the invention defined in the claims is patentable.

Declaration (or oath) for patent application. A formal statement required to be submitted to complete a patent application in the PTO, signed by the inventors to declare that they invented the invention described and claimed in the application, and that they acknowledge the duty to disclose to the PTO any information that may be material to patentability of the application.

Design patent. A type of patent protecting the ornamental designs of an article having a practical utility for a term of 14 years from the date of issue of the patent.

Disclosure. Description of the invention in a patent application or a patent. **Disclosure** is often used interchangeably with **Specification** in referring to a patent or patent application. See also **Duty of disclosure** and **Invention disclosure.**

Divisional application. An application that contains the same description of the invention (or inventions) as a prior filed application and claims the benefit of the filing date of the earlier application, but contains claims that are related to only a portion of the original disclosure.

Drawings (or figures of drawings). Line drawings or photographs submitted as part of a patent application to help describe the invention.

Duty of disclosure. A legal obligation on the part of an inventor and other individuals involved directly or indirectly in the prosecution of a patent application before the PTO to disclose to the PTO any information of which the individual is aware, or should be aware, that may be material to patentability. See **Information material to patentability.**

Enablement; enabling disclosure. The requirement that a patent application describe the invention sufficiently to enable a person of ordinary skill in the field of the patent to practice the invention claimed in the patent.

File wrapper; also prosecution history. The physical file at the PTO that contains the official record of the examination of the application.

Filing receipt. A document issued by the PTO formally acknowledging the filing of a patent application and informing the applicant of the filing date and serial number of the application. Since 2001, the filing receipt also indicates the confirmation number that serves as a cross-check for identifying the application.

Final office action (or final rejection). A formal written communication from the PTO that maintains on a "final" basis a rejection stated in a prior office action. After the issuance of a final office action, the applicant has the right to appeal the examiner's decision immediately to the PTO's Board of Patent Appeals and Interferences, and the option of submitting another written response to the rejection.

First inventor defense. A defense available to a person who was first to invent in good faith and to use commercially in the United States a method of doing business but did not patent it, and another inventor subsequently files a patent application that issues as a patent for the same method. The first inventor is protected against a charge of patent infringement of the patent.

Foreign filing license. A written statement from the PTO granting the applicant of a patent application filed in the PTO permission to file corresponding patent applications in other countries. The foreign filing license is ordinarily a statement that is incorporated into an official filing receipt issued by the PTO for the application.

Grant or issue (of patent). Publication of the formal grant of a patent, on which date the patentee can begin enforcing the patent.

Independent inventor. An inventor status given certain special considerations under U.S. patent laws, such as a 50% discount for most PTO fees, and the option of avoiding publication of a patent application if the independent inventor declares in writing that no corresponding foreign applications will be filed for the same invention.

Information material to patentability. Information that compels a conclusion that a claim of a patent application is unpatentable.

Infringement. The trespass on the rights of the owner of a patent by another party.

Intellectual property. A generic description encompassing patents, trademarks, copyrights, and other available forms of protection for the products of mental work. In some countries, industrial property is used interchangeably with intellectual property.

Interference. A proceeding in the PTO for determining which inventor or group of inventors was first to invent an invention claimed in an application X under examination, which is also claimed in another application Y or in a patent Z.

Invention disclosure. A form provided by organizations for use by their inventors to report their invention to management for consideration for possible patenting.

Issue fee. A fee to be paid to the PTO after a patent application has been allowed, to prompt the PTO to issue (grant) the patent by publishing it. An allowed application becomes abandoned if the issue fee is not paid in a timely manner.

Issue (of patent). See **Grant or issue (of patent).**

License (of patent). An agreement, usually in writing, in which the owner of a patent grants to another party the right to practice the patented invention without giving up ownership of the patent. A license may be granted to the party on an exclusive or non-exclusive basis.

Maintenance fee. Fee required to be paid periodically to the PTO to maintain an issued patent.

National stage (of international application filed in PCT system). The procedure of completing the formalities for a patent application in a country X after the filing in the PCT system of an international patent application in which X is designated as a country in which a patent is sought. The fee for designating a country in an international application is small compared with the cost of entering the national phase in each designated country. Therefore, the national stage eventually may not be entered for all the designated countries. The text of the international application is used as the text for the national stage, with translation into the language of country X as necessary. (Note: An application may also be filed directly in country X without relying on the PCT system.)

Non-obviousness. A basic requirement for a claimed invention to be patentable under U.S. laws. The claimed invention must not be obvious from previously known technology.

Novelty. A basic requirement for a claimed invention to be patentable. See **Anticipation.** In the United States, a claim lacks novelty if it is anticipated by a reference.

Oath. See **Declaration (or oath)** for patent application.

Obviousness. A basis for rejecting a claim in a patent application because the subject matter claimed is considered by the PTO examiner to be obvious from the technology described in a reference (or references) cited by the examiner.

Office action. A formal written communication from a PTO examiner, usually containing a rejection of the claims of a patent application.

Paris Convention. The Paris Convention for the Protection of Industrial Property is a treaty requiring each adhering country to accord to a patent applicant the benefit of the filing date of an application for the same invention filed not more than 1 year before in another country that adheres to the Paris Convention.

Patent. A grant by a government to a patentee, as evidenced by an official document, of exclusive rights to the subject matter or invention claimed in the patent.

Plant patent. A type of U.S. patent protecting plant varieties that are reproduced asexually, for a

term that is the same as for utility patents, without the requirement that maintenance fees be paid throughout the patent term.

Prior art; prior art reference. A document or other evidence of previously known technology against which the patentability of an invention is assessed.

Priority (claim to priority). The claim in a patent application to the benefit of the filing date of an earlier filed patent application for the same invention. Priority may be claimed under the Paris Convention based on the filing date of an earlier foreign application. Priority may also be claimed domestically in the United States based on a prior filed U.S. application.

Prosecution. The history of the examination of a patent application in the PTO.

Prosecution history. See **File wrapper.**

Provisional application. A relatively new form of U.S. patent application requiring a much smaller filing fee and less stringent formalities than for a regular patent application. A provisional application automatically expires 1 year after its filing date and must be followed by the filing in the PTO, and in other appropriate patent-granting organizations as desired, of a regular patent application that claims priority based on the provisional application.

Provisional rights. A new form of patent rights created under the AIPA to grant limited protection to a patentee for the period between the publication of a patent application and the date of issue of a patent.

Reduction to practice. The making of an invention. (The filing of a patent application is considered to be a form of reduction to practice, known as constructive reduction to practice.)

Rejection (of claim). A statement by an examiner that a claim in a patent application is not patentable for a reason specified by the examiner.

Request for Continued Examination. A new procedure available under the AIPA for obtaining a new round of examination for a patent application by making a request accompanied by a fee after a final office action or a notice of allowance has been issued by the PTO.

Response (to office action). See **Amendment.**

Small entity. A status granted under U.S. laws to independent inventors, nonprofit organizations, and small businesses with the benefit of a 50% discount in most PTO fees.

Specification. The portion of a patent application that describes in writing the invention, including the background of the invention.

Statute and interpretation by a court. A statute is a provision of law enacted by the U.S. Congress and signed by the President. A court interprets the statute in applying it to a case before the court.

Statutory invention registration. A publication by the PTO at the request of an applicant of the description portions of a patent application (i.e., without any claims). The applicant waives all rights to a U.S. patent in return for the publication, also known as defensive publication, which will prevent other parties from patenting the same technology.

Term (of patent). Time period for which patent protection is granted.

Utility. A basic requirement for an invention to be patentable under U.S. law. All applications, whether for utility, plant, or design patents, must describe an invention that has utility for it to be patentable.

Utility patent. The most common type of U.S. patent, which generally conveys the most protection compared with the other two types of U.S. patent (design and plant patents).

Resources

Internet sites of governmental patent offices

U.S. Patent and Trademark Office
www.uspto.gov
U.S. Patent and Trademark Office
Washington, DC 20231
800-786-9199 or 703-308-4357; fax 703-305-7786; TDD 703-305-7785
(Mailing address is in Washington, DC, even though PTO is in nearby Virginia.)

The PTO website offers a large amount of information, including the full text of the following publications:

Basic Facts about Patents. General information on patents, also available as a pamphlet at no charge from the PTO. Includes a list of publications available from the U.S. Government Printing Office.

General Information Concerning Patents. Greatly detailed information intended for inventors and prospective patent applicants. Also available as a publication for purchase ($2.25, document 003-004-00661-7) from the U.S. Government Printing Office, P.O. Box 371954, Pittsburgh, PA 15250-7954. For information, telephone 202-512-1800 (Washington, DC).

The full text of U.S. patents issued since 1976 can be searched and full-page images of the patents can be viewed on the PTO website. The list of attorneys and agents registered to practice before the PTO can be searched on the PTO site. Links to other sources of patent information, including the European Patent Office and the World Intellectual Property Organization (listed below), can also be found at the PTO site.

World Intellectual Property Organization
www.wipo.org
The WIPO website has a section under "Activities and Services" that is dedicated to the Patent Cooperation Treaty (PCT) system. See in particular "Basic Facts about the PCT."

European Patent Office
www.european-patent-office.org
The EPO website provides a link to its very extensive patent database Esp@cenet, which can be accessed directly at www.espacenet.com. The full text of patent applications published by the EPO and the full text of patents granted by the EPO can be searched on this site. The full text of international patent applications filed in the PCT system and published by WIPO (as "WO" patent documents) can also be searched.

The Esp@cenet database also provides English abstracts and titles for published Japanese patent applications and granted Japanese patents, as well as for patent documents from other countries.

Full-page images of published patent applications or granted patents from the United States, the PCT system, the EPO, and the Japanese Patent Office can also be viewed in the Esp@acenet database.

Academic Internet sites

Franklin Pierce Law Center
www.ipmall.fplc.edu
The Intellectual Property Mall website is hosted by the Franklin Pierce Law Center, a law school specializing in intellectual property (IP) law. The site offers "A Comprehensive Protocol for Mining Chemical IP Information" (81 pages) and other documents related to chemical patent information.

University of Texas at Austin–Engineering Library
www.lib.utexas.edu
The Engineering Library at this campus is one of the patent and trademark depository libraries affiliated with the PTO. The library's website offers a patent searching exploratory tutorial. The PTO website refers to this tutorial as a resource for independent inventors without endorsing the tutorial.

Professional societies' sites

American Intellectual Property Law Association
www.aipla.org
2001 Jefferson Davis Highway, Suite 203
Arlington, VA 22202
703-415-0780; fax 703-415-0786

Most of the information in this website is for attorneys practicing in intellectual property. However, the document "An Overview of Intellectual Property–What is a Patent, a Trademark, and a Copyright?" provides a brief overview for the nonspecialist.

Association of University Technology Managers
www.autm.net
This site offers samples of forms and other documents provided by member institutions, such as invention disclosure forms, intellectual property policies of the institutions, agreements between the institutions and researchers, licensing agreements, and others.

Internet sources charging a fee

Delphion
www.delphion.com
Delphion was spun off from IBM in 2000 to build an "online marketplace for buyers and sellers of intellectual property" and operates the database formerly known as the IBM Intellectual Property Network. Free access to the database was recently limited to a search of the bibliographic information for issued U.S. patents, copies of which may be ordered for a fee for online delivery. Otherwise, full access to the database and other services offered by Delphion requires a monthly subscription fee. The database includes U.S. patents and U.S.-published applications, European patent documents from the databases of the European Patent Office, PCT-published patent applications from the databases of the World Intellectual Property Organization, and information from "Patent Abstracts of Japan."

Nerac, Inc.
www.nerac.com
Nerac provides customized information retrieval services to subscribers, including monitoring for industrial customers of patenting activities of companies worldwide. Customers may obtain patent documents from Nerac's patent databases, which cover in essence the same U.S., European, PCT, and Japanese patent documents as Delphion.

Lexis-Nexis
www.lexisnexis.com
Lexis-Nexis, an information service provider for the legal profession, offers research and delivery of U.S. or foreign patent documents to subscribers.

Chemical Patents Plus
http://casweb.cas.org
The Chemical Patents Plus service of Chemical Abstracts Service offers the full text and patent page images of U.S.-issued patents, with CAS Registry Numbers and subject indexing for all chemical patents covered in *Chemical Abstracts.* When available, the 2-D or 3-D structures for the CAS Registry Numbers for substances identified in the patent claims may be viewed.

Print publications

Electronic Recordkeeping for Patent Purposes–Cautions and Pitfalls; American Chemical Society: Washington, DC, 1990.

Kanare, Howard M. *Writing the Laboratory Notebook;* American Chemical Society: Washington, DC, 1985.

Maynard, John T.; Peters, Howard M. *Understanding Chemical Patents*, 2nd ed.; American Chemical Society: Washington, DC, 1991 (3rd edition in progress, Peters, Howard M., Ed.).

Recordkeeping Fact Sheet; American Chemical Society: Washington, DC, 1988.

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Appendix 1. Example of a U.S. patent

United States Patent [19]
Häberlein et al.

[11] **4,129,553**
[45] **Dec. 12, 1978**

[54] **ORGANIC PHOSPHITES AND THEIR USE AS STABILIZERS**

[75] Inventors: **Harald Häberlein; Herbert Nies; Franz Scheidl,** all of Gersthofen, Fed. Rep. of Germany

[73] Assignee: **Hoechst Aktiengesellschaft,** Frankfurt am Main, Fed. Rep. of Germany

[21] Appl. No.: **799,277**

[22] Filed: **May 23, 1977**

[30] **Foreign Application Priority Data**

May 28, 1976 [DE] Fed. Rep. of Germany 2623985

[51] **Int. Cl.**[2] **C07F 9/02; C08K 5/36; C08K 5/10; C08K 5/06**

[52] **U.S. Cl.** ..... **260/45.85 R;** 260/45.8 A; 260/45.95 L; 260/403; 260/948; 260/949; 260/950; 260/951; 260/952; 260/953

[58] **Field of Search** ..... 260/399, 403, 951, 952, 260/950, 949, 948, 45.85 R, 953, 45.95 G, 45.7 PH, 45.7 PS, 45.95 L, 45.8 A

[56] **References Cited**
U.S. PATENT DOCUMENTS

2,711,401	6/1955	Lally	260/45.75 R
3,055,861	9/1962	Hersh et al.	260/953
3,396,130	8/1968	Leistner et al.	260/952
3,933,733	5/1974	Kimura et al.	260/950

Primary Examiner—V. P. Hoke
Attorney, Agent, or Firm—Connolly and Hutz

[57] **ABSTRACT**

The present invention is related to novel phosphites, their use as stabilizers for organic polymers, furthermore to stabilizer compositions containing these novel phosphites as well as the organic polymers being stabilized therewith.

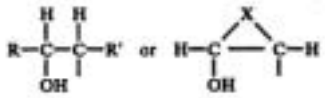
The novel phosphites have a good stabilization effect, especially in combination with known stabilizers, and they are substantially stable against hydrolytical influence. Their volatility and tendency to exudation are minimal.

6 Claims, No Drawings

ORGANIC PHOSPHITES AND THEIR USE AS STABILIZERS

Our copending application Ser. No. 689.811 which is incorporated herewith by reference, describes novel phosphites of the formula

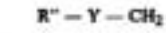
wherein A, B and C are identical or different organic radicals, at least one of which has the structure



I II

wherein R and R', which may be the same or different, each represents a hydrogen atom, an aryl or cycloalkyl group or an alkyl radical having from 1 to 60 carbon atoms, and wherein the sum of the carbon atoms included in R and R' does not exceed 60, and wherein X represents a straight-chain saturated or unsaturated alkylene radical having from 3 to 10 carbon atoms, while optionally remaining radicals B and C are aryl or cycloalkyl groups or alkyl groups having from 1 to 60 carbon atoms, and wherein the total number of carbon atoms included in the radicals A, B and C is at least 10. Furthermore, there is indicated that the novel phosphites may be used as stabilizers for organic polymers.

There has now been found that organic phosphites of the above formula, wherein the substituent R' in the radical (I) standing for A, B or C is hydrogen and the substituent R has the structure



wherein Y stands for —O—, —S— or

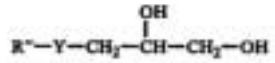
and R'' is a phenyl or naphthyl radical optionally substituted by alkyl or isoalkyl groups or by halogen, or a cycloalkyl group having from 5 to 12 ring carbon atoms, or an alkyl chain having from 1 to 60 carbon atoms, optionally interrupted by ether, thioether, carboxylic acid ester groups and/or —C=C double bonds, and optionally substituted by an (alkyl)substituted phenyl or naphthyl radical, are excellent stabilizers for plastic compositions.

The present invention provides therefore the above, hitherto unknown, organic phosphites, their use as stabilizers for organic polymers, stabilizer combinations containing these phosphites, and organic polymers so stabilized.

The novel phosphites are far superior to the known organic phosphites with respect to their stabilizing effect. A further advantage resides in the fact that those representatives of the novel phosphites which, at room temperature, are present in the form of solid substances impart an improved thermostability to the plastic

shaped articles processed with their aid as compared to the thermostability attained with the use of known liquid products. Also, the use of phosphites of the present invention reduces substantially tarnishing of the processing machines and the formation of grooves on the shaped articles being produced. Additional useful properties of phosphites of the present invention are the general lack of smell, the practical absence of volatility and the lack of tendency to exudation.

The phosphites of the present invention may be obtained according to known methods by transesterification of tri-lower alkyl-phosphites or of triphenyl phosphites with di-hydroxy compounds of the structure

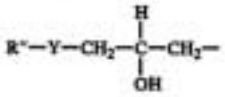


in which ester interchange the primary, more reactive, hydroxyl group reacts practically exclusively. The dihydroxy compounds may be of industrial-grade quality; thus, for example, industrial-grade glycerol monostearate or mono-oleate may be used. When mixtures of dihydroxy and high-boiling monohydroxy compounds, for example lauryl alcohol, stearyl alcohol or nonylphenol, are used for the ester interchange, a maximum 2 mols only of monohydroxy compound per mol of tri-lower alkylphosphite should be used, since at least one of the radicals A, B and C of the general formula must have the structure of the formula III according to the invention.

The novel phosphites are generally and preferably solid white products some of which have a wax-like character. Some are still liquid at room temperature. Most interesting are the first ones, having flow/drop-points of from 35° to 100° C., since in addition to their stabilization effect, they influence favorably the properties of the products made of polymer molding compositions containing such wax-like phosphites.

In the phosphites of the formula

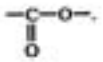
A, B and C are identical or different organic radicals; one at least of these radicals must have a structure of the formula



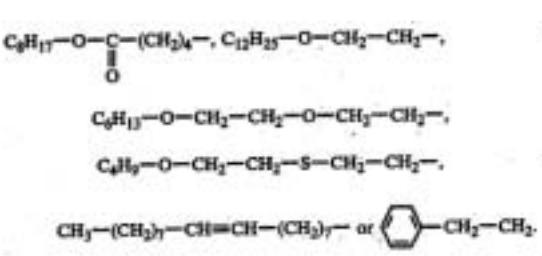
III

In the case where the radical A alone has one of these structures, B and C may be alkyl groups having from 1 to 60, preferably 1 to 30, carbon atoms, and/or aryl groups having from 6 to 12 carbon atoms and/or cycloalkyl groups having from 5 to 12 carbon atoms, preferably 5 or 6 carbon atoms. The aryl group may optionally be substituted by alkyl or alkoxyl radicals having preferably 1 to 6 carbon atoms. In the case where A and B have the structure III, C is one of the radicals just cited.

The symbol Y used in the formula III stands for —O—, —S—, or



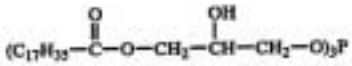
R'' represents a phenyl or naphthyl group optionally substituted by 1 to 3 alkyl or isoalkyl groups having from 1 to 12, preferably 1 to 9, carbon atoms, or by up to 5 halogen atoms, preferably chlorine atoms, or an unsubstituted or substituted cycloalkyl group having from 5 to 12, preferably 5 or 6, ring carbon atoms. Examples are the phenyl, tolyl, xylyl, tert.-butyl-phenyl, chlorophenyl, nonylphenyl, naphthyl, cyclopentyl, cyclohexyl, cycloheptyl, cyclooctyl or cyclododecyl group. R''' may also stand for a linear or branched alkyl radical having from 1 to 60, preferably 8 to 40, carbon atoms; this alkyl radical optionally containing ether, thioether, carboxylic acid ester groups and/or —C=C double bonds, and/or phenyl or naphthyl radicals optionally substituted for their part by 1 to 3 alkyl or isoalkyl groups having from 1 to 12, preferably 1 to 9, carbon atoms. Examples are the octyl, decyl, dodecyl, tetradecyl, hexadecyl, octadecyl, eicosyl, docosyl, tetracosyl, hexacosyl, octacosyl, triacontyl, dotriacontyl, tetratriacontyl, hexatriacontyl, octatriacontyl, tetracontyl, dotetracontyl, behenyl or mantanyl radicals and radicals of the following structures:



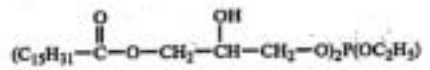
Preferred radicals R''' of the formula III are linear, saturated or unsaturated alkyl radicals having from 6 to 58, preferably 9 to 40, and especially 11 to 36, carbon atoms. The phosphites are furthermore characterized in that the total number of all carbon atoms contained in the radicals A, B and C is at least 10, preferably at least 16.

Some especially typical representatives of the novel phosphites are listed below; the invention, however, not being limited to the cited substances:

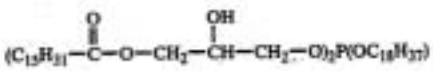
Tris-(3-stearoyl-2-hydroxy-propyl)phosphite



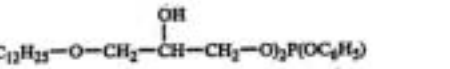
Ethyl-bis-(3-palmitoyl-2-hydroxy-propyl)phosphite



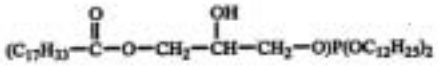
Stearyl-bis-(3-palmitoyl-2-hydroxy-propyl)phosphite



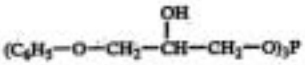
Phenyl-bis-(3-dodecyloxy-2-hydroxy-propyl)phosphite



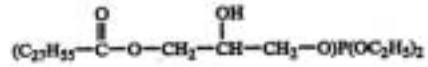
Dilauryl-(3-oleoyl-2-hydroxy-propyl)phosphite



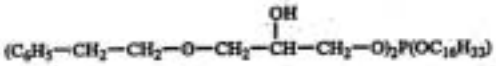
Tris-(3-phenoxy-2-hydroxy-propyl)phosphite



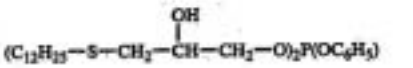
Diethyl-(3-montanoyl-2-hydroxy-propyl)phosphite



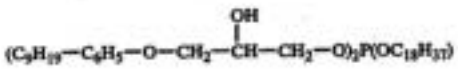
Palmityl-bis-(3-phenylethoxy-2-hydroxy-propyl)-phosphite



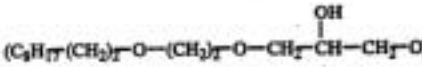
Phenyl-bis-(3-dodecylthio-2-hydroxy-propyl)phosphite



Stearyl-bis-(3-nonylphenoxy-2-hydroxy-propyl)-phosphite



Butyl-bis-(3-octyldiglycoxy-2-hydroxy-propyl)phosphite



The novel phosphites may be used also in mixture with each other, optionally together with other generally known stabilizers. There may be added to the compositions to be stabilized further stabilizing auxiliaries, antioxidants and UV stabilizing compounds. The amounts of phosphite of the invention to be used is from 0.01 to 10, preferably 0.05 to 3, parts by weight, relative to 100 parts by weight of polymer to be stabilized.

When chlorinated polymers, such, for example, as chloropolyethylene, hard and soft polyvinyl chloride, polyvinylidene chloride, polyvinyl chloroacetate and vinyl chloride-α-olefin-copolymers are processed, a substantially improved stability to heat and to light is achieved by adding the novel phosphites, in the presence of metal compounds known as stabilizers, indoles substituted in the 2-position, preferably 2-phenylindole, epoxide stabilizers and/or possibly polyhydric alcohols.

Suitable metal compounds known as stabilizers are, for example, Ca, Ba, Sr, Zn, Cd, Mg, Al and Pb soaps of

aliphatic carboxylic acids or oxycarboxylic acids having from 8 to 32 carbon atoms, preferably from 8 to 24 carbon atoms, salts of these metals with aromatic carboxylic acids of preferably from 7 to 12 carbon atoms, e.g. benzoates, salicylates as well as (alkyl)phenolates of these metals, the alkyl radical having from 1 to 12, preferably from 1 to 6, carbon atoms. This range of compounds also includes organo-tin compounds, e.g. dialkyltin-thioglycolates and carboxylates as well as — optionally — neutral and basic lead salts of inorganic acids such, for example, as sulfuric acid and phosphorous acid.

Known epoxide stabilizers are, for example, higher epoxidized fatty acids such, for example, as epoxidized soybean oil, tall oil or linseed oil, epoxidized butyl oleate and higher epoxyalkanes.

Polyhydric alcohols are, for example, pentaerythritol, trimethylol propane, sorbitol or mannitol, i.e., preferably alcohols having from 5 to 6 carbon atoms and from 3 to 6 OH groups.

Stabilizers of this kind, e.g. metal compounds, epoxides and polyhydric alcohols are described, for example, in J. Voigt "Stabilisierung der Kunststoffe gegen Licht und Wärme", Springer-Verlag, Berlin-Heidelberg-New York (1966).

A very efficient stabilizer composition for processing halogenated polymer molding compositions consists, for example, of from 0.01 to 10 parts by weight of a phosphite of the invention, 0.1 to 10 parts by weight of a metal compound known as stabilizer, 0.1 to 10 parts by weight of a known epoxide stabilizer and 0 to 1 part by weight of a polyhydric alcohol.

The novel phosphites display also an excellent efficiency for stabilizing polymers or copolymers of olefins free from halogen. The stability of, for example, polypropylene to heat and to light is considerably improved by the addition of the novel phosphites, especially in admixture with phenolic and/or sulfidic stabilizers.

By phenolic and sulfidic stabilizers there are to be understood the generally known stabilizers against heat and light which are used in the processing of plastics, for example 3,5-ditertiarybutyl-4-hydroxyphenyl-propionic acid ester, 2,6-ditertiarybutyl-p-cresol, alkylidene-bis-alkyl-phenols, esters of bis-(4'-hydroxy-3'-tertiary-butylphenyl)-butyric acid, thiodipropionic acid ester of fatty alcohols as well as dioctydecyl sulfide or dioctadecyl disulfide; (cf. J. Voigt, "Stabilisierung der Kunststoffe gegen Licht und Warme", Springer-Verlag, Berlin-Heidelberg-New York (1966)).

A synergistically efficient stabilizer composition for polymers or copolymers of olefins free from halogen consists, for example, of from 0.05 to 3 parts by weight of a phosphite according to the invention, from 0.05 to 3 parts by weight of a known phenolic stabilizer and/or of from 0.1 to 3 parts by of a known sulfidic stabilizer. Special stabilizers against ultra-violet rays may also be added to the stabilizer composition in an amount of from 0.1 to 3 parts by weight, if necessary. Known ultra-violet absorbers are, for example, alkoxy-hydroxybenzophenones, hydroxyphenylbenzotriazoles, salicilic acid phenolic ester, benzoic acid hydroxyphenolic ester, benzylidene malonic acid mononitrile ester as well as so-called "quenchers" such, for example, as nickel chelates, hexamethyl-phosphoric acid triamide or — as recently made known — piperidine stabilizers.

Stabilizer compositions of the phosphites according to the invention and known stabilizers not only improve the stability of polyolefins, chloropolyolefins and chlorinated vinylpolymers, but impart also an improved stability to polyesters, polyamides, polyacrylonitrile, polycarbonates, polysiloxanes, polyethers, polyurethanes and others.

The following Examples illustrate the invention and show the advantages of the novel phosphites.

EXAMPLE 1

A 1 liter-four-necked flask, equipped with an agitating device, an internal thermometer, gas inlet and descending cooler, was rinsed with nitrogen and subsequently charged with 516 g (1.5 mol) of octadecoxy-propanediol-1,2 and 124,5 g (0.5 mol) of freshly distilled triethyl phosphite. The contents of the flask were heated to 115°–120° C. in the course of 20–30 minutes in a weak nitrogen current, while stirring, ethanol then starting to separate at an internal temperature of about 110° C. Within a further 3–5 hours, while stirring was continued, the temperature of the reaction mixture was slowly increased from 120° C. to a final temperature of 160° C. 66 g of ethanol were distilled off during this period of time. Agitation was then continued for another hour in a water jet vacuum of 10–20 mm at the unaltered temperature of 160° C., in order to remove possible volatile components. After this period, 3.4 g of a liquid consisting of 2.0 g of triethyl phosphite and a further 1.4 g of ethanol were found in a cooling trap inserted between the described apparatus and the water jet pump.

Thus a total quantity of 67.4 g (97.6% of the theoretical yield) of ethanol was separated.

After cooling of the limpid, slightly yellowish melt 566 g (99% of the theoretical yield) of ethyl-bis(3-octadecoxy-2-hydroxy-propyl)phosphite were obtained in the form of a wax having a flow/drop point of 62°–63° C. (determined according to DGF M III 3 (57)), containing 3.9% of phosphorus, and having a molecular weight of 746. The calculated values for a compound of formula $C_{44}H_{91}O_7P$ are 4.1% P and a molecular weight of 762.

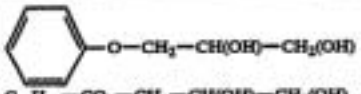
EXAMPLES 2 to 10

According to the method described in Example 1, further representatives of the novel phosphites (Examples 2 to 9) and, for a comparison, ethyl-bis(octadecyl) phosphite (Example 10) were synthetized. The following Table shows the special preparation characteristics and the analytical characterization of the products obtained.

In Example 9, a mixture of 1 mol of stearyl alcohol and 1 mol of 3-dodecoxy-propanediol-1,2 was reacted.

Example No.	starting materials			
	formula of the diol	Amount in mols and grams	Phosphite used	Amount in mols and grams
2	$C_{12}H_{25}-S-CH_2-CH(OH)-CH_2(OH)$	1.5 / 414	TEP[1)]	0.75 / 124.5

-continued

Example No.	starting materials: formula of the diol	Amount in mols and grams	Phosphite used	Amount in mols and grams
3		1.5 / 252	TEP	0.75 / 124.5
4	$C_{17}H_{35}-CO_2-CH_2-CH(OH)-CH_2(OH)$	1.5 / 537	TEP	1.5 / 249
5	$C_{17}H_{35}-CO_2-CH_2-CH(OH)-CH_2(OH)$	1.5 / 537	TEP	0.75 / 124.5
6	$C_{17}H_{35}-CO_2-CH_2-CH(OH)-CH_2(OH)$	1.5 / 537	TEP	0.5 / 83
7	$C_{17}H_{35}-CO_2-CH_2-CH(OH)-CH_2(OH)$	1.5 / 537	TPP[3]	0.75 / 232.5
8	ind.-grade GMS[1]	[4] / 510	TEP	0.75 / 124.5
9	$C_{12}H_{25}-O-CH_2-CH(OH)-CH_2(OH)$	1 / 260	TEP	1 / 166
	$C_{18}H_{37}OH$	1 / 270		
(Comp.) 10	$C_{18}H_{37}OH$	1 / 270	TEP	0.5 / 83

[1]GMS = industrial-grade glycerol monostearate composed of about 55% glycerol monostearate, 39% glycerol distearate and 10% glycerol tristearate of the hydroxy number 242
[2]TEP = triethyl phosphite
[3]TPP = triphenyl phosphite,
[4]2.2 equivalent OH groups

Example No.	Products of the process: Phosphite obtained	Analysis: P % b.w. calc.	found	mol.wt. calc.	found	flow/drop point ° C
2	Ethyl-bis-(3-dodecylthio-2-hydroxy-propyl) phosphite	4.9	4.6	626	595	liquid
3	Ethyl-bis-(3-phenoxy-2-hydroxy-propyl) phosphite	7.6	7.7	410	423	30/31
4	Diethyl-(3-stearoyl-2-hydroxy-propyl) phosphite	6.5	6.1	478	452	48/49
5	Ethyl-bis-(3-stearoyl-2-hydroxy-propyl) phosphite	3.9	4.0	790	756	59/60
6	Tris-(3-stearoyl-2-hydroxy-propyl) phosphite	2.8	2.6	1102	1049	62/63
7	Phenyl-bis-(3-stearoyl-2-hydroxy-propyl) phosphite	3.7	3.4	838	851	60/61
8	—	4.1	4.0	—	—	45/45.5
9	Ethyl-octadecyl-(3-dodecyloxy-2-hydroxy-propyl) phosphite	5.1	5.0	604	577	43/44
(Comp.) 10	Ethyl-bis-(octadecyl) phosphite	4.9	4.8	638	609	40.5/41.5

EXAMPLES 11 to 38

These Examples illustrate the stabilizing effect of phosphites of the present invention on the processing of polyvinyl chloride. The dynamic thermostability (Examples 11 to 24) and the static thermostability (Examples 25 to 38) were determined. The specified parts are parts by weight.

For each of a number of phosphites of the present invention, 100 parts of a mass-polyvinyl chloride having a K-value of 60 were mixed thoroughly with 0.2 parts of 2-phenylindole, 3 parts of epoxidized soybean oil, 0.25 parts of a complex calcium/zinc stabilizer consisting of 42 weight % of calcium stearate, 30 weight % of zinc stearate, 22 weight % of pentaerythrite and 6 weight % of 2,6-di-t-butyl-4-methylphenol, 0.2 part of a montanic acid ester (acid number 18, saponification number 154), 0.3 part of stearyl stearate, 0.5 part of glycerol monostearate, and 0.5 part of the phosphite.

In order to determine the dynamic thermostability each mixture was applied on to a laboratory-scale twin-roller device heated to 180° C., and rolled-out to a rough sheet within one minute at 20 rpm. At intervals of 10 minutes, spot samples were picked of these sheets, and their color shades compared with an internal color chart. The various tests were run until the rough sheet had taken up a dark-brown to black shade.

In order to determine the static thermostability, a rough sheet was first prepared from each mixture according to the description given above, and this sheet was rolled out on the twin-roller device at 180° C. for another 10 minutes' period. The sheet was then peeled off the roller and little plates of about 0.5 mm thickness and a diameter of 30 mm blanked therefrom. These specimens were wrapped in an aluminium sheet and tempered at 180° C. in a heating cabinet with internal air circulation. One specimen was selected every 10 minutes and its color shade compared with the color chart. The figures employed in the color chart have the following meaning:

1 = clear as water
2 = slightly yellowish
3 = distinctly yellow tint
4 = dark yellow-brown shade
5 = dark brown to black

As demonstrated by the following Tables, as far as dynamic and static thermostability are concerned, the polyvinyl chloride stabilized by organic phosphites of the present invention is clearly superior in comparison to polyvinyl chloride stabilized with known phosphites and with mixtures free from phosphites.

Dynamic thermostability

Example No.	Phosphite acc. to Example	Discoloration of rough sheet at a laminating time of 10'	20'	30'	40'	50'	60'	70'	80'
		to color number							
11	1	1	2	2-3	2-3	3	5	—	—
12	2	1	2	2-3	3	3	3-4	5	—
13	3	1	2	2-3	2-3	3	3	5	—
14	4	1	1-2	2	3	3	3	5	—
15	5	1	1-2	2	2-3	3	3	5	—
16	6	1	1-2	2	2-3	3	3	3-4	5
17	7	1	1-2	2	2	3	3	5	—
18	8	1	1-2	2	2-3	3	3	5	—
19	9	1	2	2	2	3	3	5	—
20 (comp.)	10	1	1-2	2	3	5	—	—	—
21 (comp.)	Triphenyl phosphite	1	2	2-3	5	—	—	—	—
22 (comp.)	Trisnonyl-phenyl phosphite	1	2	2-3	5	—	—	—	—
23 (comp.)	Diphenyl-isooctyl phosphite	1	2-3	3	5	—	—	—	—
24 (comp.)	none	2	2-3	3-4	5	—	—	—	—

Static thermostability

Example No.	Phosphite acc. to Example	Discoloration of rough sheet in drying cabinet at a tempering time of 0'	20'	30'	40'	50'	60'	70'	80'	0'
		to color number								
25	1	1	2	2	2-3	3	3	3	5	—
26	2	1	2	2	2-3	3	3	5	—	—
27	3	1	2	2	2	3	3	3	5	—
28	4	1	2	2	2-3	3	3	3-4	5	—
29	5	1	1-2	2	2-3	3	3	3-4	5	—
30	6	1	1-2	2	2-3	3	3	3	3-4	5
31	7	1	1-2	2	2-3	3	3	3-4	5	—
32	8	1	2	2-3	2-3	3	3	3-4	5	—
33	9	1	2	2-3	2-3	3	3	3-4	5	—
34 (comp.)	10	1	2	2	2-3	3	3-4	5	—	—
35 (comp.)	Triphenyl phosphite	1	1-2	2	2-3	3	5	—	—	—
36 (comp.)	Trisnonyl-phenyl phosphite	1	2	2-3	3	5	—	—	—	—
37 (comp.)	Diphenyl-isooctyl phosphite	1	2	2	2	3	3-4	5	—	—
38 (comp.)	none	1	2-3	2-3	2-3	3-4	5	—	—	—

EXAMPLES 39 to 42

These Examples show that the addition of the phosphites of the present invention to polypropylene improves considerably its stability to light and against alterations due to heat.

Powdery mixtures consisting each of
100 parts by weight of unstabilized polypropylene

[(spun at 230° C.)about fi]

0.15 part by weight of octadecyl-3-(3',5'-di-tert.-butyl-4'-hydroxyphenyl)-propionate
and
0.10 part by weight of the phosphites prepared according to Examples 1, 6 and 9 were injection-molded on an injection molding machine to yield test plates measuring 60 × 60 × 1 mm. Test specimens were blanked from these plates.

The stability to light was determined by means of the Xeno-test device, type 150, produced by Messrs. Hanau Quarzlampen GmbH with the filter combination 6 IR + 1 UV as per DIN 53 387. (DIN = German Industrial Standard). The time of exposure to light, i.e. the period of time after which the absolute elongation at break had decreased to 10% was measured in hours.

The resistance to alteration under heat of injection molded test samples was measured approximately to the procedure described by DIN 53 383 by storing the specimens at an air temperature of 140° C. until total embrittlement.

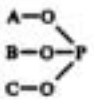
The following Table demonstrates the good light an heat stabilizing effect of the phosphites in polypropyene.

Stability to light and alteration due to heat

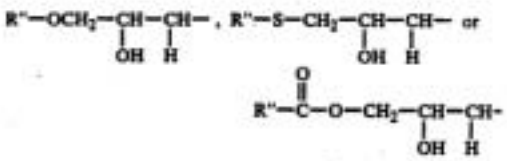
Example No.	Phosphite acc. to Example	Time of exposure in hours	Stability to alteration due to heat in days
39	1	710	41
40	6	685	44
41	9	725	39
42 (comp.)	without phosphite	530	21

What is claimed is:

1. Phosphites of the formula



wherein A, B and C are identical or different radical and at least one is a member of the structure



wherein R''' is a phenyl- or naphthyl group or a cycloalkyl group having from 5 to 12 ring carbon atoms or an alkyl chain having from 1 to 60 carbon atoms, and any radicals of A, B and C remaining are aryl groups having from 6 to 12 carbon atoms or cycloalkyl groups having from 5 to 12 carbon atoms or alkyl groups having from 1 to 60 carbon atoms, the total sum of carbon atoms contained in the radicals A, B and C being at least 10.

2. Stabilizer combination for chlorinated polyolefins and chlorine containing vinyl homo- and copolymers, consisting of 0.01 to 10 parts by weight of a phosphite as claimed in claim 1, 0.1 to 10 parts by weight of metal compounds known as stabilizers, 0 to 10 parts by weight of a known epoxide stabilizer, and 0 to 1 part by weight of a known polyol.

3. Stabilizer combination for homopolymers or copolymers of halogen-free olefins, consisting of 0.05 to 5 parts by weight of a phosphite as claimed in claim 1, 0.05 to 3 parts by weight of a known phenolic stabilizer and/or 0.1 to 3 parts by weight of a known sulfidic stabilizer, and 0 to 3 parts by weight of a known ultraviolet stabilizer.

4. A process for stabilizing chlorinated polyolefins, chlorine-containing vinyl homo- and copolymers or polymers or copolymers of halogen-free olefins, which comprises adding to the polymer a phosphite of claim 1.

5. The process of claim 4 wherein there is added to the polymer the phosphite of claim 1 in combination with at least one further additive selected from the group consisting of a metal compound stabilizer, an epoxide stabilizer, a phenolic stabilizer, a sulfidic stabilizer, a polyol stabilizer and an ultraviolet stabilizer.

6. Plastic molding compositions containing a phosphite as claimed in claim 1 as stabilizer.

* * * * *

Appendix 2. First U.S. patent application publication

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(12) **Patent Application Publication** (10) **Pub. No.: US 2001/0000001 A1**

Clark et al. (43) **Pub. Date: Mar. 15, 2001**

(54) **SOLVENT MIXTURE FOR USE IN A VAPOR DEGREASER AND METHOD OF CLEANING AN ARTICLE IN A VAPOR DEGREASER UTILIZING SAID SOLVENT**

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(22) Filed: **Nov. 29, 2000**

Related U.S. Application Data

(63) Continuation of application No. 09/375,495, filed on Aug. 17, 1999, which is a continuation of application No. 08/894,495, filed on Nov. 10, 1997, Pat. No. 5,938,859, which is a 371 of international application No. PCT/US97/05183, filed on Mar. 28, 1997, which is a continuation-in-part of application No. 08/580,840, filed on Dec. 29, 1995, Pat. No. 5,616,549.

Publication Classification

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(52) **U.S. Cl.** **134/31**

(57) **ABSTRACT**

The invention provides a solvent mixture comprising n-propyl bromide, a mixture of low boiling solvents and, preferably, a defluxing and/or ionics removing additive and/or at least one saturated terpene. The invention also provides a method of cleaning an article (e.g., an electrical, plastic, or metal part) in a vapor degreaser using the solvent mixture. The solvent mixture of the invention is non-flammable, non-corrosive, and non-hazardous. In addition, it has a high solvency and a very low ozone depletion potential. Thus, using the solvent mixture of the invention, oil, grease, rosin flux, and other organic material can be readily removed from the article of interest in an environmentally safe manner.

SOLVENT MIXTURE FOR USE IN A VAPOR DEGREASER AND METHOD OF CLEANING AN ARTICLE IN A VAPOR DEGREASER UTILIZING SAID SOLVENT

CROSS-REFERENCE TO RELATED APPLICATION

[0001] This is a continuation-in-part of U.S. patent application Ser. No. 08/580,840 filed Dec. 29, 1995, now U.S. Pat. No. 5,616,549 issued Apr. 1, 1997.

FIELD OF THE INVENTION

[0002] The invention relates generally to molecular level cleaning of parts by vapor degreasing. More particularly, the invention relates to a solvent mixture comprising n-propyl bromide, a mixture of low boiling solvents and, preferably, a defluxing and/or ionics removing agent and/or at least one saturated terpene, as well as to a method of cleaning an article in a vapor degreaser using the solvent mixture. The solvent mixture of the invention is non-flammable, non-corrosive, non-hazardous, and has a low ozone depletion potential.

BACKGROUND OF THE INVENTION

[0003] Molecular level cleaning by vapor degreasing has found wide acceptance in industry. In fact, molecular level cleaning by vapor degreasing is a preferred method of cleaning precision parts, such as electronics, machined metallic parts, etc., since vapor cleaning leaves virtually no residue on the parts. Generally, vapor degreasing involves the heating of a solvent to its boiling point to generate a vapor layer into which the object to be cleaned is placed. The vapor condenses on the object and subjects the surface to a solvent-flushing action as it flows downward. The solvent-flushing action dissolves the hydrocarbon contaminants and removes them from the object, thereby cleaning it. The liquid drops are then collected in a reservoir and are revaporized, typically through the use of steam-heating coils. Thus, the surface of the object is continually rinsed with fresh solvent.

[0004] There are four general types of vapor phase degreasers. The simplest form of a vapor phase degreaser is the straight vapor cycle degreaser which utilizes only the vapor for cleaning. As the parts are lowered into the hot vapor, the vapor condenses on the cold parts and dissolves the surface oils and greases. The oily condensate drops back into the liquid solvent at the base of the tank. The solvent is evaporated continuously to form a vapor blanket. Since the oils are not vaporized, they remain in the bottom of the tank in the form of a sludge. The scrubbing action of the condensing vapor continues until the temperature of the part reaches the temperature of the vapor whereupon condensation ceases, the part appears dry, and it is removed from the degreaser. The time required to reach this point depends on the particular solvent employed, the temperature of the vapor, the weight of the part, its specific heat and the type of contamination material to be removed. This particular vapor phase degreaser does an excellent job of drying parts after aqueous cleaning and before plating and, thus, it is frequently used for this purpose in the jewelry industry. Unfortunately, however, it is not as effective on small, light weight parts because such parts frequently reach the temperature of the vapor before the condensing action has fully cleaned the parts.

[0005] A second type of vapor phase degreaser, i.e., the vapor-spray cycle degreaser, is frequently used to solve the problems associated with the straight vapor cycle degreaser. In this vapor-spray cycle degreaser, the part to be cleansed is first placed in the vapor zone as is done in the straight vapor cycle degreaser. A portion of the vapor is condensed by cooling coils and fills a liquid solvent reservoir. This warm liquid solvent is pumped to a spray nozzle that can be used to direct the solvent on the part, washing off surface oils and cooling the part, thereby cleaning by vapor condensation.

[0006] The third type of vapor phase degreaser is a liquid-vapor cycle degreaser which has one compartment with warm solvent and another compartment with a vapor zone. This degreaser is particularly useful for heavily soiled parts or for cleaning a basket of small parts that nest together. The fourth type of vapor phase degreaser is the ultrasonic degreaser. Such degreasers are useful for cleaning critical parts. An ultrasonic degreaser has a transducer mounted at the base of the tank which operates in the range of 20 kHz to 40 kHz. The transducer alternately compresses and expands the solvent forming small bubbles which, in turn, cavitate or collapse on the surface of the part. This cavitation phenomenon disrupts the adhering soils, thereby cleaning the part.

[0007] Conventional solvents used with the foregoing vapor phase degreasers include trichloroethylene, perchloroethylene, methyl chloroform, methylene chloride, CFC 113, dibromomethane, bromochloromethane, trichlorotrifluoroethane and various hydrochlorofluorocarbons, such as "Genesolve" (manufactured by Allied Chemical). Vapor degreasing techniques employing the foregoing solvents or equivalents thereof are taught in U.S. Pat. No. 3,881,949 which issued on May 6, 1975 to Carl Martin Brock. Unfortunately, however, such solvents are typically on the Clean Air Act list of high ozone depleting chemicals and, thus, they are being phased out of production and/or banned from use in the United States. Thus, there exists a need for a solvent which can be used in place of these banned ozone depleting chemicals in vapor phase degreasers.

[0008] U.S. Pat. No. 4,056,403, which issued to Robert J. Cramer, et al. on Nov. 1, 1977, describes a method in which a number of non-regulated ozone depleting chemicals, including n-propyl bromide, are used in cleaning polyurethane foam generating equipment. Cramer, et al. teach a method wherein a solvent composition described therein is used for cleaning a polyurethane foam generating apparatus or a segment thereof. The solvents taught may be periodically injected under pressure through the mixer portion of the foaming apparatus in order to purge it of residual unreacted or partially foam forming materials. Unfortunately, the method described in this patent would be totally ineffective because its composition does not include the appropriate stabilizers necessary to prevent the n-propyl bromide from becoming acid and thereby attacking the metal surfaces which might be placed into the vapor layer.

[0009] The use of hot saturated vapors of a liquid halogenated hydrocarbon, including bromochloromethane, is taught in U.S. Pat. No. 4,193,838 which issued to Robert J. Kelly, et al. on Mar. 18, 1990. More particularly, this patent teaches the generation of a pool of hot saturated vapors of a halogenated acyclic hydrocarbon. Pieces of paper stock

which have been coated with "hot melt" coatings, such as are used on consumer items and milk cartons, etc., are then placed in the vapor pool and, thereafter, they are agitated. Again, it is noted that this method would be ineffective at cleaning flux and other articles because of the acidic nature of the non-stabilized compound utilized therein which would tend to destroy the object rather than just clean it.

[0010] U.S. Pat. No. 5,403,507, which issued to Richard G. Henry on Apr. 4, 1995, discloses a solvent mixture for use in vapor cleaning degreasing. Dibromomethane is used as the principal component. The dibromomethane is mixed with other solvents which are intended to stabilize the dibromomethane and to prevent the solvent mixture from becoming acidic on the release of bromine into the atmosphere. Although the solvent mixture disclosed therein is more stable than either of the solvent mixtures taught in U.S. Pat. Nos. 4,056,403 and 4,193,838, there are still a number of disadvantages associated with the use of dibromomethane which make it unsuitable for use as a solvent in vapor phase degreasers. In fact, the Clean Air Act now lists dibromomethane as an ozone depleting chemical which is banned from use in vapor degreasers or any other cleaning process which results in atmospheric release.

[0011] In view of the foregoing, it is readily apparent that there remains a need in the art for a solvent mixture which is suitable for molecular level cleaning of parts without the use of any of the high ozone depleting chemicals that are identified as Class I or Class II materials in the U.S. Federal Register, Vol. 58, No. 236, Friday, Dec. 10, 1993, Rules and bromochloromethane as a potential ozone depleter and possible banning in the U.S. Federal Register 40 CFR Part 82, Vol. 60, No. 145, Pages 38729-38734, Jul. 28, 1995.

SUMMARY OF THE INVENTION

[0012] It is an object of the invention to overcome at least one of the problems described above.

[0013] Accordingly, the invention provides a solvent mixture which can be used in vapor phase degreasers in place of traditional solvents.

[0014] More particularly, the invention provides a solvent mixture for use in a vapor degreasing system, the solvent mixture comprising effective amounts of n-propyl bromide and at least one low boiling solvent or mixture thereof and, preferably, a defluxing and/or ionics removing additive and/or at least one saturated terpene.

[0015] In another aspect, the invention provides a method for cleaning an article in a vapor degreaser, the method comprising: (a) providing a vapor degreaser system; (b) adding to the solvent reservoir of the vapor degreaser system the inventive solvent mixture; (c) boiling the solvent mixture to form a vapor layer; (d) introducing into the vapor layer an article to be cleaned; and, (e) removing the article from the vapor layer.

[0016] In this method, the vapor layer condenses on the article, thereby subjecting the surface of the article to a solvent-flushing action as it flows downward. The solvent-flushing action dissolves the hydrocarbon contaminants and removes them from the object, thereby cleaning it. As such, using the method of the present invention, oil, grease, rosin flux and other organic material can be readily removed from the article of interest.

[0017] Other features, objects, and advantages of the invention and its preferred embodiments will become apparent from a review of the detailed description which follows.

DETAILED DESCRIPTION OF THE INVENTION AND PREFERRED EMBODIMENTS

[0018] The solvent mixture of the invention is non-flammable, non-corrosive, and non-hazardous. Moreover, it has a high solvency and a low ozone depletion potential typically between 0.001 and 0.04 and a global warming potential typically between 0.0001 and 0.0003. As such, the solvent mixture of the present invention can effectively be used to remove oil, grease, rosin, flux, and other organic contaminants from the surfaces of numerous articles, e.g., electrical, plastic and metallic parts.

[0019] Preferably, the invention provides a solvent mixture for use in a vapor degreasing system, the solvent mixture comprising: (a) about 85 wt. % to less than about 96.5 wt. % n-propyl bromide; (b) 0 wt. % to about 6.5 wt. % of one or a mixture of saturated terpenes, the terpene mixture preferably comprising about 35 wt. % to about 50 wt. % cis-pinane and about 35 wt. % to about 50 wt. % trans-pinane; (c) an effective amount, preferably about 3.5 wt. % to about 5 wt. % of a mixture of low boiling solvents, the low boiling solvent mixture preferably comprising about 0.5 wt. % to about 1 wt. % nitromethane, about 0.5 wt. % to about 1 wt. % 1,2-butylene oxide, and about 2.5 wt. % to about 3 wt. % 1,3-dioxolane; and, (d) 0 wt. % up to about 5 wt. % of at least one defluxing and/or ionics removing additive selected from the group consisting of sec-butanol, ethanol, and methanol.

[0020] In a presently preferred embodiment, the terpene mixture of cis-pinane and trans-pinane includes terpenes. Suitable terpenes include, but are not limited to, one or more of the following: endo-isocamphene, α-pinene, cis-para-menthane and trans-para-menthane. In another preferred embodiment, the terpene mixture further includes endo-isocamphene, α-pinene, cis-para-menthane and trans-para-menthane. If present, these terpenes can, individually, make up to 100% of the terpene content of the solvent mixture and, preferably, about 0.01 wt. % to about 6.5 wt. % of the solvent mixture, and highly preferably about 0.05 wt. % to about 5 wt. % of the solvent mixture.

[0021] One of functions of the low boiling solvent or low boiling solvent mixture is to neutralize any free acid that might result from oxidation of the mixture in the presence of air, from hydrolysis of the mixture in the presence of water, and from pyrolysis of the mixture under the influence of high temperatures. Moreover, the low boiling solvent or solvent mixture serves to prevent pitting or corrosion of metal articles which are placed in the vapor layer.

[0022] It has been discovered that the solvent mixture of the invention meets the desired characteristics for the proper cleaning of electrical parts, metals, plastics, elastomers, circuit boards, etc. More particularly, the solvent mixture of the invention has the following characteristics: (1) it is properly stabilized against any free acid that might result from oxidation of the mixture in the presence of air, from hydrolysis of the mixture in the presence of water, and from pyrolysis of the mixture under the influence of high temperatures; (2) it is non-flammable and non-corrosive; (3) the

various components of the solvent mixture are not regulated by the U.S. Clean Air Act; and (4) none of the various components of the solvent mixture are known cancer causing agents (i.e., the various components are not listed by N.T.I., I.A.R.C. and California Proposition 65, nor are they regulated by OSHA). Moreover, the solvent mixture of the invention has a high solvency with a kauri-butanol value above 120 and, more preferably, above 125. In addition, the solvent mixture of the invention has an evaporation rate of at less 0.96 where that of 1,1,1-trichloroethane=1. Upon evaporation, the solvent mixture of the invention leaves a non-volatile residue (NVR) of less than 2.5 mg (or less than about 500 ppm) and, more preferably, no residue. Further, the solvent mixture of the invention has a latent heat of vaporization of about 58.8 cal/g which, in turn, facilitates condensation of the solvent mixture on the chiller side of a standard vapor degreasing system.

[0023] In addition, the use of n-propyl bromide in the solvent mixture of the invention has significant advantages over the use of dibromomethane and bromochloromethane. In contrast to n-propyl bromide, dibromomethane is listed by the Clean Air Act as an ozone depleting chemical which is banned from use in vapor degreasing or other cleaning processes involving atmospheric release and bromochloromethane which is suspect of having a ODP of greater>0.1 where the ODP of the banned 1,1,1-trichloroethane=0.1 and bromochloromethane will not obtain SNAP approval and may be banned. Moreover, in contrast to n-propyl bromide which has an atmospheric life of about 7 to 14 days and an ODP of 0.001-0.04, dibromomethane has an atmospheric life of about three years and bromochloromethane of three to four months and a ODP of 0.08-1.2. Dibromomethane is more toxic than n-propyl bromide and, in contrast to n-propyl bromide, dibromomethane undergoes bioaccumulation (e.g., in fish and aquatic life). In addition, while both dibromomethane and n-propyl bromide react with strong bases, strong oxidizing agents, aluminum, calcium, zinc, magnesium, alloys, etc., the compounds formed with dibromomethane are typically shock sensitive and, thus, potentially explosive, whereas those formed with n-propyl bromide are not. Further, the chemical and physical properties of the n-propyl bromide-based solvent mixtures of the invention make them more energy efficient than the bromochloromethane or dibromomethane solvent mixture of the prior art or the banned solvents 1,1,1- trichloroethylene, trichloroethylene, or methylene chloride. As a result of its boiling point, specific heat and latent heat of vaporization, the n-propyl bromide-based solvent mixtures of the invention require about the same or less energy to cause the mixture to boil and create a denser vapor zone for cleaning.

[0024] The sec-butanol, ethanol, and/or methanol present in the solvent mixture function(s) as an aid in defluxing and/or in removing ionic species in vapor degreasing and cold batch cleaning operations.

[0025] The presence of sec-butanol enhances flux removal for type R, type RMA, type RA, and synthetic fluxes. Sec-butanol also enhances cleaning of polar and non-polar soils including hand oils, solder oils, greases, silicones, and similar soils.

[0026] Methanol is effective in enhancing removal of type A and type RMA fluxes. Ethanol, when present, forms an azeotropic mixture with n-propyl bromide, and is especially useful in defluxing soldered metal parts.

[0027] The defluxing and/or ionics removing agent or mixture of agents is typically used in an effective amount of up to about 5 wt. %, preferably 3 wt. % or less, and highly preferably about 1 wt. %, based on the total solvent composition.

[0028] As a result of the foregoing properties, the solvent mixture of the invention can be advantageously used in vapor phase degreasers in place of traditional solvents including, for example, trichloroethylene, perchloroethylene, methyl chloroform, methylene chloride, trichlorotrifluoroethane, dibromomethane, CFC-113, etc. Moreover, the solvent mixture of the invention can be effectively used in the four major types of vapor phase degreasers, i.e., the straight vapor cycle degreaser, the vapor-spray cycle degreaser, and the liquid-vapor degreaser and the ultrasonic degreaser. In addition, emissions from a vapor phase degreaser operated with the solvent mixture of the invention are so low that local exhaust ventilation is not required, although in some instances, such a system may still be desirable.

[0029] The solvent composition of the invention is simply prepared by combining and mixing together the n-propyl bromide, the terpene mixture (if present), the low boiling solvent mixture, and the defluxing and/or ionics removing agent (if present) in the desired or specified proportions. The solvent mixture is then ready to use as the solvent in a vapor phase degreaser system. n-propyl bromide (C_3H_7Br, i.e., $CH_3CH_2CH_2Br$) is commercially available from Dead Sea Bromine LTD Israel. The terpenes used to make up the terpene mixture are commercially available from SCM Glidco Jacksonville Fla. Nitromethane (CH_3NO_2), 1,3-dioxolane, and 1,2-butylene oxide (also known as 1,2-epoxybutane) are commercially available from Aldrich Chemical Co. (Milwaukee, Wis.). Sec-butanol is also available from Aldrich Chemical Co. In addition to purchasing the foregoing compounds from commercial sources, it will be apparent to those of skill in the art that such compounds can be readily synthesized using known synthetic procedures. For instance, n-propyl bromide can be prepared, for example, when alcohols react with either inorganic acid halides or with hydrogen halides. (see, e.g., Carl R. Noller, *Textbook of Organic Chemistry*. Ch 6:81 (1956), the teachings of which are incorporated herein by reference for all purposes).

[0030] In another aspect, the invention provides a method of cleaning articles in a vapor degreaser using the solvent mixture of the invention. In this method, the solvent mixture of the invention is added to a vapor degreaser, such as Baron-Blakeslee or Branson models. The thermostat on the vapor degreaser is typically set to a temperature of about 156° F. to about 160° F. (Ultrasonic degreasers can operate effectively at temperatures as low as 70° F.) In this temperature range, the n-propyl bromide present in the solvent mixture will boil. When the solvent mixture reaches a temperature of about 156° F. to about 160° F., a vapor layer will appear above the solvent as a mist. This vapor mist constitutes the principal feature of cleaning by the vapor method. When the vapor mist appears, the object to be cleaned is placed into the vapor layer. The vapor condenses on the object and subjects the surface of the object to a solvent-flushing action as it flows downward. The solvent-flushing action dissolves the hydrocarbon contaminants and removes them from the object, thereby cleaning it. The

liquid drops are then collected in a reservoir and are revaporized, typically through the use of steam-heating coils. Thus, the surface of the object is continually rinsed with fresh solvent. As such, using the method of the invention, oil, grease, rosin flux and other organic material can be readily removed from the object of interest. Moreover, the vapors from the solvent will not contain any of the removed contaminants and, thus, the vapors can be used to clean additional objects.

[0031] The invention will be described in greater detail by way of specific examples. The following examples are offered for illustrative purposes, and are intended neither to limit or define the invention in any manner.

EXAMPLE I

[0032] A solvent mixture in accordance with the invention was blended and added together to a standard vapor degreaser, the solvent mixture comprising: (I) about 90.0 wt. % n-propyl bromide; (ii) about 6 wt. % of a mixture of saturated terpenes, the terpene mixture comprising about 45 wt. % cis-pinane, about 45 wt. % trans-pinane, about 2 wt. % endo-isocamphene, about 2 wt. % α-pinene, about 2 wt. % cis-para-menthane and about 2 wt. % trans-para-menthane; and (iii) about 4 wt. % of a mixture of low boiling solvents, the low boiling solvent mixture comprising about 0.5 wt. % nitromethane, about 0.5 wt. % 1,2-butylene oxide and about 3 wt. % 1,3-dioxolane. The thermostat on the vapor degreaser was adjusted to a temperature of about 160° F., and the system was allowed to equilibrate. After the mixture inside the solvent reservoir reached a temperature of about 160° F., the mixture began to boil. Upon inspection, a vapor layer several inches thick was observed inside the vapor degreaser unit. Enough vapor was being evolved to condense and be circulated from the chilled side of the vapor degreaser to the boiling side of the vapor degreaser.

EXAMPLE II

[0033] Fifteen gallons of the solvent mixture described in Example I were added to a vapor phase degreaser. The thermostat on the vapor degreaser was adjusted to a temperature of about 160° F., and the system was allowed to equilibrate. After the mixture inside the solvent reservoir reached a temperature of about 160° F., the mixture began to boil. A basket of steel parts covered with lithium-based grease was placed in the vapor layer. After a period of about 30 seconds, the basket of steel parts was removed from the vapor layer. All of the lithium-based grease had been removed and the steel parts were completely clean. Using a similar procedure as that just described, pieces of sheet metal containing light mineral oils, silicone oils, lithium greases, and other types of industrial release fluids were placed in the vapor layer to be cleaned. After a period of about a minute, the pieces of sheet metal were removed from the vapor layer. All of the contaminants, i.e., the light mineral oils, silicone oils, lithium greases, and other types of industrial release fluids, had been removed.

EXAMPLE III

[0034] Fifteen gallons of the solvent mixture described in Example I were added to a ultrasonic degreaser, which will operate effectively with the inventive solvent mixture at temperatures in the range of 70° F. to about 160° F. The ultrasonic degreaser had a transducer mounted at the base of the tank which operates in the range of 20 kHZ to 40 kHz. The thermostat on the ultrasonic degreaser was adjusted to a temperature of about 160° F., and the system was allowed to equilibrate. The ultrasonic degreaser also employed water chilled coils to control the solvent vapors and to eliminate the need for a local exhaust ventilation system. Several steel parts coated with lithium grease were immersed in the solvent for about one minute. The transducer alternately compressed and expanded the solvent thereby forming small bubbles which, in turn, cavitated at the surface of the lithium grease coated steel parts. The cavitation phenomenon disrupted the adhering soils and cleaned the parts. Using a similar procedure as that just described, pieces of sheet metal containing light mineral oils, silicone oils, lithium greases, and other types of industrial release fluids were immersed in the ultrasonic degreaser. After a period of about a minute, the pieces of sheet metal were removed from the vapor layer. All of the contaminants, i.e., the light mineral oils, silicone oils, lithium greases, and other types of industrial release fluids, had been removed.

EXAMPLE IV

[0035] Five gallons of the solvent mixture described in Example I were added to an emulsion soak tank. A steel part coated with lithium grease was immersed for one minute into an emulsion soak tank containing the solvent mixture at room temperature. While some cleaning occurred, the resulting cleaning was not at the molecular level. Similarly, five gallons of the solvent mixture described in Example I were added to a heated power washer emulsion degreaser. The thermostat on this degreaser was adjusted to 156° F., just below the boiling point of n-propyl bromide, and the system was allowed to equilibrate. Thereafter, the solvent mixture was sprayed on steel parts which were coated with lithium grease. Upon inspection, it was observed that the resulting cleaning was at the molecular level.

EXAMPLE V

[0036] Standard corrosion tests, similar to those performed by Dow Chemical Company, were performed using the solvent mixture of the invention, as oxidation is a potential problem with all solvent cleaners. In addition, methodology similar to that used by Dow Chemical Company was used to show equilivences to existing Clean Air Act banned solvents. In performing these test, strips of copper and steel measuring 1" wide by 6" long and of 20 mil thickness were buffed on a belt sander to remove any oxide films. Fifty milliliters of the solvent mixture described in Example I were placed in a cylindrical Pyrex glass container and strips were placed in so that 75% of the surface was immersed in the solvent. A sample container filled with tap water was used as a control for the test to insure that there were no alloys present in the metal strips which would have been prevented oxidation. The openings of the sample containers were all sealed with cork stoppers to reduce evaporation. After an eight hour incubation period and a 24 hour incubations period, the strips were removed and it was determined that the solvent mixture of the present invention was non-corrosive.

[0037] It is to be understood that the above description is intended to be illustrative and not restrictive. Many embodiments may be apparent to those of skill in the art upon

reading the above description. The scope of the invention should, therefore, be determined not with reference to the foregoing description, but should instead be determined with reference to the appended claims, along with the full scope of equivalents to which such claims are entitled. The disclosures of all articles and references, including patent applications and publications, are incorporated herein by reference for all purposes.

What is claimed is:

1. A solvent mixture for use in a vapor degreasing system, said solvent mixture comprising:

(a) about 85 wt. % to less than about 96.5 wt. % n-propyl bromide;

(b) about 3.5 wt. % to less than about 15 wt. % of a mixture of low boiling solvents, said solvent mixture comprising about 0.5 wt. % to about 1 wt. % nitromethane, about 0.5 wt. % to about 1 wt. % 1,2-butylene oxide, and about 2.5 wt. % to about 3 wt. % 1,3-dioxolane; and

(c) an effective amount of up to about 5 wt. % of at least one additive selected from the group consisting of sec-butanol, ethanol, and methanol.

2. The solvent mixture of claim **1** further comprising an effective amount of at least one saturated terpene.

3. The solvent mixture of claim **2** comprising up to about 6.5 wt. % of a terpene mixture of cis-pinane and trans-pinane.

4. The solvent mixture of claim **3** wherein said terpene mixture comprises about 35 wt. % to about 50 wt. % cis-pinane and about 35 wt. % to about 50 wt. % trans-pinane.

5. The solvent mixture of claim **2** wherein said terpene mixture further comprises at least one terpene selected from the group consisting of endo-isocamphene, α-pinene, cis-para-menthane, and trans-para-menthane.

6. The solvent mixture of claim **2** wherein said terpene mixture further comprises endo-isocamphene, α-pinene, cis-para-menthane, and trans-para-menthane.

7. The solvent mixture of claim **1** wherein said additive consists essentially of up to about 3 wt. % of sec-butanol.

8. The solvent mixture of claim **7** wherein said sec-butanol comprises about 1 wt. % of said solvent mixture.

9. A method for cleaning an article in a vapor degreaser, said method comprising:

(a) providing a vapor degreaser system;

(b) adding to the solvent reservoir of said vapor degreaser system a solvent mixture, said solvent mixture comprising:

i) about 85 wt. % to less than about 96.5 wt. % n-propyl bromide;

ii) about 3.5 wt. % to less than about 15 wt. % of a mixture of low boiling solvents, said solvent mixture comprising about 0.5 wt. % to about 1 wt. % nitromethane, about 0.5 wt. % to about 1 wt. % 1,2-butylene oxide and about 2.5 wt. % to about 3 wt. % 1,3-dioxolane; and

iii) an effective amount of up to about 5 wt. % of at least one additive selected from the group consisting of sec-butanol, ethanol, and methanol;

(c) boiling said solvent mixture to form a vapor layer;

(d) introducing into said vapor layer said article to be cleaned, said vapor layer condensing on said article, thereby subjecting the surface of said article to a solvent-flushing action; and

(e) removing said article from said vapor layer.

10. The method of claim **9** further comprising an effective amount of at least one saturated terpene.

11. The method of claim **10** comprising up to about 6.5 wt. % of a terpene mixture of cis-pinane and trans-pinane.

12. The method of claim **11** wherein said terpene mixture comprises about 35 wt. % to about 50 wt. % cis-pinane and about 35 wt. % to about 50 wt. % trans-pinane.

13. The method of claim **10** wherein said terpene mixture further comprises a terpene selected from the group consisting of endo-isocamphene, α-pinene, cis-para-menthane and trans-para-menthane.

14. The method of claim **10** wherein said terpene mixture further comprises endo-isocamphene, α-pinene, cis-para-menthane and trans-para-menthane.

15. The method of claim **9** wherein said defluxing additive consists essentially of up to about 3 wt. % of sec-butanol.

16. The method of claim **15** wherein said sec-butanol comprises about 1 wt. % of said solvent mixture.

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