# United States Patent

[11] 3,607,767



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[21] Appl. No. **865,525**
[22] Filed **Oct. 10, 1969**
[45] Patented **Sept. 21, 1971**



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**Continuation-in-part of application Ser. No. 661,807, Aug. 21, 1967, now abandoned.**


[54] **AZEOTHROPIC COMPOSITION OF 1,1,2-TRIFLUOROETHANE, METHYLENE CHLORIDE, AND CYCLOPENTANE**
**2 Claims, No Drawings**


[52] **U.S. Cl.** ..... **252/171,** 106/311, 252/66, 252/364
[51] **Int. Cl.** ..... **C09d 9/00,** C11d 7/50, C23g 5/02
[50] **Field of Search** ..... 252/171, 170, 172, 66–68, 305, 364; 62/112, 114; 260/652.5; 106/311



[56] **References Cited**

UNITED STATES PATENTS

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|---|---|---|---|
| 2,999,817 | 9/1961 | Bower | 252/172 |
| 3,249,546 | 5/1966 | Eiseman, Jr. | 252/171 |

OTHER REFERENCES

Rose, The Condensed Chem. Dict. Reinhold Publ. Co. (1961) Page 327



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**ABSTRACT:** It is not intended that the following abstract of the disclosure be construed as limiting the invention in any manner.

The disclosure relates to azeotropic mixtures of trichlorotrifluoroethane, methylene chloride and cyclopentane. The azeotropes are useful in solvent cleaning applications.

# AZEOTHROPIC COMPOSITION OF 1,1,2-TRIFLUOROETHANE, METHYLENE CHLORIDE, AND CYCLOPENTANE

This application is a continuation-in-part application of U.S. Pat. application, Ser. No. 661,807 filed Aug. 21, 1967 and now abandoned.

This invention relates to an azeotropic composition and particularly to the azeotropic mixture of trichlorotrifluoroethane, methylene chloride and cyclopentane.

Several of the chlorofluoroethanes have attained widespread use as specialty solvents in recent years, particularly trichlorotrifluoroethane. This compound is a relatively low boiling liquid ($CC1_2FCC1F_2$, 47.6° C.), which is nontoxic and nonflammable, and which has satisfactory solvent power for greases, oils, waxes and the like. It has therefore found widespread use for cleaning electric motors, compressors, oxygen storage tanks, photographic film, lithographic plates, typewriters, instruments, gauges, sound tape, and as noncorrosive brines.

For certain solvent purposes however, the chlorofluoroethanes alone have insufficient solvent power. This is particularly true in the electronic industry during the manufacture of "printed circuits." "Printed circuits" are well known in the electronics art; and consist of a circuit formed from a soft metal on a solid, nonconducting surface such as a reinforced phenolic resin. During manufacture, the solid surface is coated with the metal, the desired portion of metal is coated with an impervious coating and the excess metal is removed by etching with a suitable acid. After the excess metal has been removed, it is necessary to remove the impervious coating because solder joints must be made to the "-printed circuit" and these will not form if the coating is present. After the impervious coating is removed, the circuits are coated with a rosin flux to permit the joints to be soldered, after which the rosin flux must be removed. The chlorofluoroethane solvent does not have sufficient solvent power to clean "printed circuits"; that is, to effectively remove the rosin flux. Although mixtures of solvents may be used for this purpose they suffer the disadvantage that they boil over a range of temperatures and consequently undergo fractionation in vapor degreasing or ultrasonic applications which are open to the atmosphere. When employing either of these methods the solvent must also be substantially nontoxic and nonflammable for safety reasons. Accordingly, it is an object of this invention to provide a constant boiling or azeotropic solvent that will not fractionate and also has the foregoing advantages. Another object is to provide an azeotropic composition which is valuable as a solvent and particularly for cleaning printed circuits. A further object is to provide an azeotropic composition which is both relatively nontoxic and nonflammable both in the liquid phase and in the vapor phase and which at the same time is an excellent solvent for cleaning printed circuits.

The above object of this invention may be accomplished by the provision of a novel azeotropic composition consisting of trichlorotrifluoroethane (e.g., 1,1,2-trichloro-1,2,2-trifluoroethane), methylene chloride and cyclopentane.

It has been found that when 1,1,2-trichloro-1,2,2-trifluoroethane and methylene chloride and cyclopentane are admixed they form an azeotrope which distills at constant temperature, the liquid phase and the vapor phase in equilibrium therewith having the same composition. Such mixture is nonflammable and relatively nontoxic in both the liquid phase and the vapor phase. These mixtures are particularly useful as solvents for greases, oils, waxes, and the like and cleaning electric motors, compressors, photographic films, lithographic plates, typewriters, precision instruments, gauges, sound tapes, and the like and are particularly useful for cleaning printed circuits.

The azeotropic composition of this invention was obtained in the course of a fractional distillation of a mixture of 1,1,2-trichloro-1,2,2-trifluoroethane, methylene chloride and cyclopentane. The mixture was charged to a laboratory distillation flask provided with an overhead condenser for the



removal of distillate. The mixture was heated to its atmospheric boiling point and distilled at high reflux. An azeotropic mixture was removed consisting of about 39.1 percent by weight 1,1,2-trichloro-1,2,2-trifluoroethane, about 51.6 weight percent methylene chloride, and 9.3 weight percent cyclopentane having a boiling point of about 97.0° F. at substantially 760 mm. Hg pressure.

Although the azeotropes are obtained at approximately 760 mm. Hg pressure a variation in pressure and consequently a change in the composition and boiling point is also intended to be included within the broad scope of this invention. Thus, the azeotropes may contain any proportion of the three components provided that a constant boiling mixture is obtained at the various pressures at which the compositions are used. Stated otherwise any pressures may be used to obtain the azeotropes of this invention so long as a three component system comprising trichlorotrifluoroethane, methylene chloride and cyclopentane is obtained and accordingly the ratios of these components will vary. The variation of the proportions of components is thus within the skill of the art once it is known that the components will form azeotropes. In a preferred embodiment however, the present invention relates to the azeotropic mixture of 1,1,2-trichloro-1,2,2-trifluoroethane, methylene chloride and cyclopentane at atmospheric pressure ± about 5 to about 25 especially ± about 5 to about 15 mm. Hg. The cyclopentane may be pure or contain up to about 5 or about 10 percent by weight of about 4 to about 7 carbon atom homologues or analogues and substantially the same results obtained.

Additionally 1,1,2-trichloro-1,1,2-trifluoroethane may be substituted in part or completely by 1,1,1-trichloro-2,2,2-trifluoroethane.

Printed circuit boards are usually prepared by impregnating glass cloth, nylon, or paper laminates with a phenol-formaldehyde resin or an epoxy resin. Printed circuits are prepared by a variety of methods. In a typical procedure, the board consists originally of a phenolic resin impregnated base to which is bonded a sheet of copper, 2- to 4-mils thick, covering one surface of the board. The desired circuit is drawn on the copper with an asphalt based ink, using the silk screen method. The excess copper is then removed by etching with a ferric chloride-hydrochloric acid bath, sometimes containing ammonium chloride, leaving on the board the copper that is covered by the ink. After washing off the etch solution, the asphalt ink is removed by cleaning with the azeotropic composition of this invention in an ultrasonic bath (some mechanical scrubbing is often used). The entire surface of the board is coated with a rosin flux and dried. The electronic components (resistors, capacitors, etc.) are then added at the proper places for soldering to the circuit. The board is then passed over a molten solder bath, contacting the desired joints with the molten metal, whereby the soldering is effected. AFter cooling, the excess rosin flux remaining on the board must be removed since, if present in the final assembly, it will lead to corrosion, poor electrical resistance and other deleterious properties.

The board is cleaned by placing it in an ultrasonic bath of any of the aforementioned azeotropes and operating at about 32 kilocycles per second at about 25° C., where it remains for about 1 minute.

When the board is cleaned with 1,1,2-trichloro-1,2,2-trifluoroethane alone a considerable amount of rosin flux remains after treatment. On the other hand, if the board is cleaned with methylene chloride alone, even though the rosin flux is substantially removed, the board itself will be considerably damaged. However, if the board is treated with the azeotropic compositions of this invention substantially all of the rosin flux is removed without any detrimental effect on the board which constitutes the backing of the printed circuit.

The variations in pressure noted previously result in the production of an azeotropic composition wherein each of the components vary between about ±10 percent especially ±5 percent of the weight of each of the individual components

noted in the specific embodiment noted previously. Thus the azeotropic mixture compromises from about 43.01 to about 35.19 parts by weight, especially from about 41.06 to about 37.15 parts by weight 1,1,2-trichloro-1,2,2-trifluoroethane; from about 56.76 to about 46.44 parts by weight, especially from about 54.18 to about 49.02 parts by weight of methylene chloride and from about 10.23 to about 8.37 parts by weight, especially from about 9.77 to about 8.84 parts by weight of cyclopentane.

Although the invention has been described by reference to some preferred embodiment it is not intended that the broad scope of the novel azeotropic compositions be limited thereby, but that certain modifications are intended to be included within the spirit and broad scope of the following claims.

What is claimed is:

1. The azeotropic mixture consisting essentially of about 39.1 parts 1,1,2-trichloro-1,2,2-trifluoroethane, about 51.6 parts methylene chloride and about 9.3 parts cyclopentane on a weight basis at a boiling temperature of about 97.0° F. at about 760 millimeters mercury pressure.

2. A method of cleaning a surface by contacting said surface with an azeotropic mixture consisting essentially of about 39.1 parts 1,1,2-trichloro-1,2,2-trifluoroethane, about 51.6 parts methylene chloride and about 9.3 parts cyclopentane on a weight basis at a boiling temperature of about 97.0° F. at about 760 millimeters mercury pressure.