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AZEOTROPIC COMPOSITION OF TETRACHLORODIFLUOROETHANE ISOPROPANOL AND WATER

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ABSTRACT OF THE DISCLOSURE

The disclosure relates to azeotropic mixtures of tetrachlorodifluoroethane, isopropanol and water.

It is not intended that the preceding abstract of the disclosure be construed as limiting the invention in any manner.

This invention relates to an azeotropic composition and particularly to the azeotropic mixture of tetrachlorodifluorethane, isopropanol, and water.

Several of the chlorofluoroethanes have attained widespread use as specialty solvents in recent years, particularly tetrachlorodifluoroethane. This is a relatively high melting compound (CCl_2FCCl_2F, 24.5° C.) which is nontoxic and nonflammable, and which has satisfactory solvent power for greases, oils, waxes and the like under certain conditions. It has therefore found widespread use in cleaning electric motors, compressors, oxygen storage tanks, photographic film, lithographic plates, typewriters, instruments, gauges, sound tape, and as non-corrosive 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, non-conducting 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, the circuits are coated with a rosin flux to permit the joints to be soldered, after which the rosin flux must then 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 have 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.

Tetrachlorodifluoroethane is a relatively high boiling fluorocarbon and for this reason especially advantageous in vapor degreasing applications since at these temperatures the hot vapor has more of a tendency to dissolve high melting greases, or fluxes as well as oil residues and the like. When articles such as circuit boards are passed through a vapor degreaser, the solvent vapors tend to condense on the article until the articles are heated by the vapors from room temperature up to the temperature of the vapor. The condensation thus formed on the articles tends to drip back into the solvent reservoir taking with it some of the soil on the article. For this reason the ability of a cleaning solvent to condense on the surface is especially advantageous. Higher boiling solvents prolong this condensation effect in a continuous degreaser since it takes a greater amount of time to bring the article passing through the degreaser up to the vapor temperature of the solvent. Consequently higher boiling solvents generally have better cleaning power per unit of time in a continuous vapor degreaser than the lower boiling solvents.

Tetrachlorodifluoroethane also is a better solvent than materials such as trichlorotrifluoroethane however, it suffers the disadvantage that it is solidus at room temperature whereas the latter is liquidus. Accordingly tetrachlorodifluoroethane is more difficult to handle than liquid type cleaning solvents.

It is an object of this invention to provide a constant boiling or azeotropic solvent that is a liquid at room temperature, 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 especially by means of a continuous vapor degreasing machine.

The above object of this invention may be accomplished by a novel azeotropic composition of tetrachlorodifluoroethane (e.g. 1,1,2,2 - tetrachloro - 1,2 - difluoroethane) isopropanol, and water. Although 1,1,2,2-tetrachloro-1,2-difluoroethane is a preferred tetrachlorodifluoroethane, the isomer 1,1,1,2-tetrachloro-2,2-difluoroethane may be substituted therefor in whole or in part and especially in minor amounts or trace amounts.

The azeotrope of the present invention is further characterized in that it forms two liquid layers or phases which are designated herein as the "Top Phase" and "Bottom Phase." When at the boiling temperature the composition of both phases remains constant and equilibrium is established between the vapors which are coming off of the constant boiling mixture and the two liquid phases. This mixture forms an azeotrope which distills at a constant temperature, the liquid phase and the vapor phase in equilibrium therewith having the same composition. Such mixture is relatively nonflammable and nontoxic in both the liquid phase and the vapor phase. This mixture is particularly useful as a solvent 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.

Although the azeotropic mixture described herein is obtained at approximately 762 mm. Hg a variation in pressure and consequently a change in the compositions and boiling points are also intended to be within the broad scope of the invention. Thus the azeotropes may contain many different proportions of all of the aforementioned components provided a constant boiling mixture is obtained at the various pressures at which the compositions are used. Stated otherwise any pressure may be employed to obtain the azeotropes of this invention as long as a three component constant boiling mixture is obtained, and accordingly the ratio of components of the azeotrope of the invention will also vary. The variation of components is thus within the skill of the art and is easily determined once it is known that the halogenated hydrocarbons of this invention will form the aforementioned azeotrope. In a preferred embodiment the present invention relates to the aforementioned azeotropes that boil at 762 mm. Hg pressure ± about 25, especially ± about 15 mm. Hg and where the volume of the top and bottom phases

as well as the composition thereof will vary by ± about 10% or ± about 5%, especially ± about 2%.

EXAMPLE I

An azeotropic composition is obtained in the course of distillation of a mixture of 1,1,2,2-tetrachloro-1,2-difluoroethane, isopropanol and water. The mixture is charged to a laboratory distillation flask provided with an overhead condenser for the removal of distillate. The mixture is heated to its atmospheric boiling point and distilled at high reflux. An azeotropic mixture is removed having two liquid layers consisting essentially of the following composition:

	Top phase 40% by volume, parts by weight	Bottom phase 60% by volume, parts by weight	Combined bottom and top phases, parts by weight
1,1,2,2-tetrachloro-1,2-difluoroethane	Trace	~85.7	~58.5
Isopropanol	~28.4	~13.1	~18.8
Water	~71.7	~1.2	~22.7

The above azeotrope has a boiling point of about 158.2° F. (about 70° C.) when measured at about 762 mm. Hg pressure. The trace amount of 1,1,2,2-tetrachloro-1,2-difluoroethane in the top phase of the azeotrope has been reported as such since it is present in such miniscule quantities that it is not possible to report its presence in specific numerical terms within the experimental error of the analytical methods and devices commonly used in azeotropic studies of halogenated hydrocarbons. In view of the trace amount of 1,1,2,2-tetrachloro-1,2-difluoroethane present in the top layer, azeotropic or substantially azeotropic compositions can be obtained according to the present invention if the top layer as noted above is free or substantially free of tetrachlorodifluoroethane as defined herein.

Because as mentioned previously the azeotropes exist at pressures other than ambient pressures, the components of the mixtures may vary as noted above. Thus, for example, where the lower phase of the azeotrope contains 85.7 parts by weight of 1,1,2,2-tetrachloro-1,2-difluoroethane ± about 2% as noted above, it is intended that this component may vary from about 87.4 to about 84.0 parts by weight and so forth for the rest of the ranges of all the other above components and their equivalents when the pressure at which the azeotrope is formed varies from 762 mm. Hg.

Printed circuit boards are usually prepared by impregnating glass cloth, nylon, or paper laminates with a phenolformaldehyde 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), by immersion in the liquid azeotrope of this invention or by vapor degreasing with the azeotrope of this invention. 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 the aforementioned azeotrope and operating at about 32 kilocycles per second at about 60° C., where it remains for about one minute. The board is alternately cleaned by immersion in the azeotrope of this invention or by vapor degreasing with the azeotrope of this invention.

When the board is cleaned with the azeotropic mixtures 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.

Although the invention has been described by reference to some preferred embodiments it is not intended that the broad scope of the novel azeotropic composition 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. A composition of matter consisting essentially of an azeotrope having two liquid phases, and at a boiling temperature of about 70° C. at about 762 mm. mercury pressure, the top phase amounts to about 40% by volume and is composed of a trace amount of 1,1,2,2-tetrachloro-1,2-difluoroethane, about 28.4 parts by weight isopropanol and about 71.7 parts by weight of water; the bottom phase amounts to about 60% by volume and is composed of about 85.7 parts by weight of 1,1,2,2-tetrachloro-1,2-difluoroethane, about 13.1 parts by weight of isopropanol and about 1.2 parts by weight of water; the combined top and bottom phases containing about 58.5 parts by weight of 1,1,2,2-tetrachloro-1,2-difluoroethane, about 18.8 parts by weight isopropanol and about 22.7 parts by weight of water based on 100 parts by weight of the total composition.

2. A method of cleaning a surface by contacting said surface with the azeotrope of claim 1.

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UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 3,686,131 Dated August 22, 1972

Inventor(s) John Allan Schofield

It is certified that error appears in the above-identified patent and that said Letters Patent are hereby corrected as shown below:

Column 1, after "John Allan Schofield, 9 Buxton Lane, Riverside, Conn. 06878", insert "Assignee: Union Carbide Corporation, New York, N. Y."

Signed and sealed this 17th day of April 1973.

(SEAL)
Attest:

EDWARD M.FLETCHER,JR.
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ROBERT GOTTSCHALK
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