

[54] AZEOTROPIC COMPOSITION  
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[57] ABSTRACT  
This invention relates to a binary azeotrope of 1,1,2-trichloro-1,2,2-trifluoroethane and tertiary butyl alcohol useful as a cleaning solvent.

4 Claims, No Drawings

## AZEOTROPIC COMPOSITION

This invention relates to binary azeotropic mixture of trichlorotrifluoroethane and tertiary butanol.

Several of the chlorofluoroethanes have attained widespread use as specialty solvents in recent years, particularly trichlorotrifluoroethane. This is a relatively high boiling compound ( $\text{Cl}_2\text{FC}-\text{CF}_2\text{Cl}$ ,  $47.7^\circ\text{C}$ ), 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 use in cleaning electric motors, compressors, lithographic plates, typewriters, instruments, gauges, 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, 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 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 cleaning or degreasing methods the solvent must also be both relatively nontoxic and nonflammable for safety reasons.

Trichlorotrifluoroethane is a relatively high boiling fluorocarbon and for this reason is 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.

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 fore-

going 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 binary azeotropic composition of 1,1,2-trichloro-1,2,2-trifluoroethane and tertiary butanol.

The preferred azeotropic composition of this invention is composed of from about 95 percent to about 98 percent 1,1,2-trichloro-1,2,2-trifluoroethane and from about 2 percent to about 5 percent tertiary butanol by weight. These mixtures form an azeotrope which distills at a constant temperature of about  $46^\circ\text{C}$ , the liquid phase and the vapor phase in equilibrium therewith having the same composition. These mixtures are relatively nonflammable and 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, lithographic plates, typewriters, precision instruments, gauges, and the like and are particularly useful for cleaning printed circuits.

Although the azeotropic mixtures are obtained at approximately 760 mm Hg a variation in pressure and consequently a change in the composition and boiling point are also intended to be within the broad scope of the invention. Thus the azeotrope may contain many different proportions of its components provided a constant boiling mixture is obtained at the various pressures at which the composition is used. Stated otherwise any pressure may be employed to obtain the azeotrope of this invention as long as a constant boiling mixture is obtained, and accordingly the ratio of components of the azeotrope of the invention may also vary. The variation of components is thus within the skill of the art and is easily determined once it is known that the trichlorotrifluoroethane and butanol will form an azeotrope.

## EXAMPLE I

An azeotropic composition was obtained in the course of distillation of a mixture of 1,1,2-trichloro-1,2,2-trifluoroethane and tertiary butanol. 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. Aliquot portions of the constant boiling mixture were removed and analyzed and found to consist essentially of the following composition:

| Components                            | Composition Percent by Weight | Boiling Point, $^\circ\text{C}$ . 768 mm Hg |
|---------------------------------------|-------------------------------|---------------------------------------------|
| 1,1,2-trichloro-1,2,2-trifluoroethane | 95-98                         |                                             |
| tertiary butanol                      | 2-5                           | 46                                          |

These results were confirmed by preparing a series of mixtures ranging from 0.5 to 6.0 percent tertiary butanol and from 99.5 to 94 percent 1,1,2-trichloro-1,2,2-

trifluoroethane. The samples were placed in a Podbielniak distillation column and distilled at high reflux. All were found to yield constant boiling mixtures having the same temperature at the top and bottom of the column and varying not more than 0.3°F. from each other at constant pressure.

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). 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 10°-20°F. below the boiling point of the azeotrope for about one minute.

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 compositions be limited thereby but that certain modifications are intended to be included within the spirit and broad scope of the following claims.

I claim:

1. A constant boiling azeotropic composition consisting essentially of 1,1,2-trichloro-1,2,2-trifluoroethane and tertiary butanol and boiling at 46°C at about 760 mm. Hg.

2. A constant boiling composition consisting essentially of from about 95 to about 98 parts by weight of 1,1,2-trichloro-1,2,2-trifluoroethane and from about 2 to about 5 parts by weight of tertiary butanol and boiling at 46°C at about 760 mm. Hg.

3. A method for cleaning a solid surface comprising contacting said surface with an azeotropic composition according to claim 1.

4. A method in accordance with claim 3 wherein said composition is used in vapor form.

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