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AZEOTROPIC COMPOSITION

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This invention relates to an azeotropic composition and particularly to the azeotropic mixture of 1,1,2-trichloro-1,2,2-trifluoroethane and methylene chloride.

Several of the chlorofluoromethanes and chlorofluoroethanes have attained widespread use as specialty solvents in recent years, particularly trichlorofluoromethane and trichlorotrifluoroethane. Both of these compounds are relatively low boiling liquids (CCl_3F, 23.8° C.; CCl_2FCClF_2, 47.6° C.), which are nontoxic and nonflammable, and which have good solvent power for greases, oils, waxes and the like. They have 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 chlorofluoromethanes and chlorofluoroethanes have insufficient solvent power alone. 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 imprevious coating is removed, the circuits are coated with a rosin flux to permit the joints to be soldered, which rosin flux must then be removed. The chlorofluoromethane and chlorofluoroethane solvents do not have sufficient solvent power to clean "printed circuits"; that is, to effectively remove the rosin flux.

It is an object of this invention to provide a novel azeotropic composition which has new and unusual properties. 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 such a composition which is nonflammable and nontoxic, both in the liquid phase and in the vapor phase. Other objects are to provide new compositions of matter and to advance the art. Still other objects will appear hereinafter.

The above and other objects may be accomplished in accord with this invention which comprises an azeotropic composition consisting of about 52% by weight of 1,1,2-trichloro-1,2,2-trifluoroethane and about 48% by weight of methylene chloride and boiling at 37° C. at 760 mm. pressure.

It has been found that 1,1,2-trichloro-1,2,2-trifluoroethane and methylene chloride (CH_2Cl_2) when admixed in the above proportions form an azeotrope which distils at constant composition, the liquid phase and the vapor phase in equilibrium therewith having the same composition. Such mixture is nonflammable and nontoxic in both the liquid phase and the vapor phase. It is useful as a heat exchange medium and as a hydraulic fluid. It is particularly useful as a solvent for greases, oils, waxes, and the like, in cleaning electric motors, compressors, photographic film, oxygen storage tanks, lithographic, plates, typewriters, precision instruments, gauges, sound tape, components of engines which use liquid oxygen, and

printed circuits. The 1,1,2-trichloro-1,2,2-trifluoroethane alone has insufficient solvent power for many of such purposes, and the methylene chloride alone is objectionable for some of such purposes. It has been found that, in the azeotrope, the components modify the solvent properties of each other so that the azeotropic mixture is an excellent solvent for such purposes without having the disadvantages of either.

In the cleaning of components of engines which use liquid oxygen, it is particularly important that the solvent remove all oxidizable materials so that they will not contact the liquid oxygen and that the solvent leave no residue which will react with liquid oxygen. The azeotropic mixture of this invention has the desired properties which render it useful for this purpose.

The azeotropic mixture of 1,1,2-trichloro-1,2,2-trifluoroethane and methylene chloride is of special value for cleaning printed circuits. The 1,1,2-trichloro-1,2,2-trifluoroethane alone is not an effective solvent for cleaning printed circuits, and has no deleterious effect on the boards which form the bases of the printed circuits. Methylene chloride alone deleteriously affects such boards, particularly laminated boards impregnated with phenolic resins, causing softening, swelling and delamination of such boards and dissolving certain marking inks which are used to code various electronic components in the printed circuit assembly. The azeotropic mixture of this invention does not have these effects and is an excellent solvent for such purpose.

The azeotrope of this invention has several advantages over the solvents heretofore employed for cleaning printed circuits. Trichloroethylene, tetrachloroethylene, and methyl chloroform, are unstable and require stabilizers, whereas the azeotropic composition of this invention does not require a stabilizer, has a more favorable evaporation rate (is lower boiling), and is considerably less toxic than such solvents. The azeotropic composition of this invention is more effective as a solvent for such purposes than trichloroethylene and tetrachloroethylene. Inhibited methyl chloroform (containing dioxane as a stabilizer) is too strong a solvent and attacks the backing of the printed circuits. Isopropyl alcohol-toluene blends are flammable and irritating. The azeotropic mixture of 1,1,2-trichloro-1,2,2-trifluoroethane and methylene chloride does not suffer from any of these defects.

The azeotropic composition of this invention was established by the fractional distillation of a mixture containing 20% by weight of 1,1,2-trichloro-1,2,2-trifluoroethane and 80% by weight of methylene chloride. The first fraction collected, amounting to 40% of the original mixture, was the azeotrope containing 52% by weight of 1,1,2-trichloro-1,2,2-trifluoroethane and 48% by weight of methylene chloride having a boiling point of 37° C. at 760 mm. pressure, freezing point −95° C. to −92° C., $n_D^{26.5}$ 1.3860. Distillation of mixtures of different proportions, e.g. 60% CCl_2FCClF_2 and 40% CH_2Cl_2, always gave, as the first fraction, the azeotrope boiling at 37° C. The fractions boiling at 37° C. could be redistilled, giving material which boiled at 37° C. throughout with no separation into the components of the mixture.

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 then 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 operating at about 32 kilocycles per second and at about 25° C., where it remains for one minute. If 1,1,2-trichloro-1,2,2-trifluoroethane is used alone as the solvent in the bath, it has no effect on the rosin flux. If trichloroethylene is used as the solvent in the bath, about 5% to about 50% of the flux remains after the treatment. If the same cleaning operation is repeated on another circuit board using the azeotropic mixture of 1,1,2-trichloro-1,2,2-trifluoroethane and methylene chloride as the solvent, the rosin flux is completely removed. Neither the 1,1,2-trichloro-1,2,2-trifluoroethane nor the azeotropic mixture has any effect on the board which constitutes the backing of the printed circuit.

It will be understood that the preceding detailed description of the use of the azeotropic composition of this invention in cleaning printed circuits is given for illustrative purposes solely, that this invention is not restricted to such specific embodiment, and that other techniques may be employed. Also, the azeotropic composition can be used for other purposes as indicated in the general description.

From the preceding description, it will be apparent that this invention provides a novel azeotropic composition which has unusual properties and which is useful for a wide variety of purposes. Accordingly, it will be apparent that this invention constitutes a valuable contribution to and advance in the art.

The embodiments of the invention in which an exclusive property or privilege is claimed are defined as follows:

An azetropic composition consisting of about 52% by weight of 1,1,2-trichloro-1,2,2-trifluoroethane and about 48% by weight of methylene chloride and boiling at 37° C. and 760 mm. pressure.

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