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AZEOTROPIC COMPOSITION

James Gordon Burt, Oxford, and Donovan Erb Kvalnes, West Chester, Pa., assignors to E. I. du Pont de Nemours and Company, Wilmington, Del., a corporation of Delaware

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The present invention is directed to novel compositions possessing unusually high solvent properties.

Solvent cleaning of complex electronic circuitry has become increasingly important. Printed circuits in television receivers, copying machines, and missile guidance systems are so intricate and compact that the only possible method of cleaning such circuits of grease and wax accumulations is to dip the circuits in a volatile, organic solvent bath until the accumulations are dissolved, thereafter removing the circuits from the bath and allowing the residual solvent to evaporate. In order to perform this unique service, the organic solvent must be low boiling, nonflammable, nontoxic, and possess a high solvent power for oily dirt, grease and wax.

It is, therefore, an object of this invention to provide a novel composition with an unusually high solvent power for oily dirt, grease and wax materials.

It is another object of this invention to provide new and valuable solvent compositions that possess azeotrope-like characteristics.

These and other objects will become apparent from the following description and claims.

More specifically, the present invention is directed to novel compositions having a boiling range of from 23.62° C. to 23.76° C. at 760 mm. and consisting essentially of from 60% to 90% by weight of trichlorofluoromethane and 40% to 10% by weight of 1,1-dichloro-2,2,2-trifluoroethane.

The present invention is also directed to an azeotrope consisting of about 78% by weight of trichlorofluoromethane and about 22% by weight of 1,1-dichloro-2,2,2-trifluoroethane.

The compositions of the present invention are ideal solvents in that they are relatively low boiling, nontoxic, nonflammable, and possess markedly greater solvent properties than either component.

One of the unique advantages of the compositions of the present invention is their azeotrope-like characteristics. Actually, within the defined composition of the present invention there exists a true azeotrope as defined above.

An azeotrope is a liquid mixture that exhibits a maximum or minimum boiling point. Characteristic of an azeotrope mixture is the fact that its distillate has the same composition as the original mixture and, hence, no change in properties of the mixture or the distillate occurs as a result of evaporation. It is well established that the existence of an azeotrope is entirely unpredictable.

The azeotropic character of mixtures of trichlorofluoromethane and 1,1-dichloro-2,2,2-trifluoroethane containing from 10% to 40% by weight of 1,1-dichloro-2,2,2-trifluoroethane is confirmed by the fact that the mixtures have essentially a constant boiling point, and the composition of the vapor phase or distillate is essentially identical with that of the liquid phase. The composition consisting of about 78% by weight of trichlorofluoromethane and

about 22% by weight of 1,1-dichloro-2,2,2-trifluoroethane has been identified by a careful fractional distillation procedure as a true binary azeotrope.

Another unusual, valuable property of the compositions of the present invention is their much increased solvent action for hydrocarbon wax and fats or greases compared to the individual components of the compositions. This solvent property, for example, adapts the compositions for use as constant boiling solvent mixtures for applying conditioning agents such as water repellents and fire retardants of the chlorinated paraffin type to textiles, and depositing dip coatings on metals. The unique properties of the solvent compositions of the present invention are also useful for scouring wool under conditions where high solvent power, rapid and complete evaporation, freedom from toxicity, fire and explosion hazards are required.

Compositions containing less than 10% 1,1-dichloro-2,2,2-trifluoroethane exhibit a sharply decreased solvent action while compositions containing more than 40% of this dichlorotrifluoroethane have a boiling point which is markedly increased over the essentially constant boiling point of mixtures containing from 10% to 40% of the dichlorotrifluoroethane. In addition, the high solvent power is lost in mixtures containing much over 40% of the dichlorotrifluoroethane.

Individually the components making up the present unique mixture have the following characteristics. Trichlorofluoromethane is a solvent boiling at 23.77° C. at 760 mm. Hg pressure. Trichlorofluoromethane is used as a cleaner for hermetic motor compressors, oxygen storage tanks, and the like. It is also useful as a cleaner for photographic film, lithographic plates, typewriters, and as a flash point retarder for flammable solvents. 1,1-dichloro-2,2,2-trifluoroethane, boiling at 27.60° C. at normal atmospheric pressure, is also used as a solvent and as a flash point retarder for hydrocarbons and other solvents.

The azeotrope like character and high solvent action of the solvent mixtures of the present invention work in combination to provide an unusually attractive solvent from an economic point of view. Naturally, after repeated use, it is necessary to rid the solvent mixtures of the dissolved wax and grease materials. Since the amount of dichlorotrifluoroethane in the mixture must be between 10% and 40% in order to possess the high solvent action, it is important that the process used to rid the solvent of the accumulated materials be such that the composition of the solvent after removal of the accumulation is the same as the original mixture. Since the compositions of the present invention possess azeotropic characteristics, ridding the solvent of accumulation is quite easily accomplished by simple distillation. Because of the solvent mixture's azeotropic nature, the condensed solvent will have the same composition and properties as the original solvent.

Representative examples illustrating the present invention follow. All parts are by weight unless specified otherwise.

EXAMPLE 1

Solubility of paraffin

Various mixtures of trichlorofluoromethane and 1,1-dichloro-2,2,2-trifluoroethane were saturated at room temperature with flaked paraffin wax melting at 50° to 53° C.

The amount of dissolved wax was measured in each case by evaporating an aliquot of each solution. The specific mixtures taken together with the solubility of the wax in them and also in the separate components of the mixtures are given in Table I below.

TABLE I.—SOLUBILITY OF WAX IN SOLVENT MIXTURES

Composition of Mixture, Percent by wt.		Parts of Wax in Solution/100 Parts of Solvent
$CClF_3$	CF_3CHCl_2	
100	0	11
90	10	32
78	22	35
60	40	33
25	75	20
0	100	10

The criticality of the concentration range of 10% to 40% of the 1,1-dichloro-2,2,2-trifluoroethane in the mixture for high solvent action on paraffin is evident from Table I. The wax is seen to be at least three times more soluble in these mixtures than it is in either component alone, and the solubility plainly decreases in mixtures outside the 10% to 40% limits for the dichlorotrifluoroethane.

EXAMPLE 2

Mixtures of trichlorofluoromethane and 1,1-dichloro-2,2,2-trifluoroethane were distilled through a 100 cm. column packed with glass helices to provide a column equal to about 10 theoretical plates. The initial boiling point was recorded. After about 40% of a mixture had distilled, the distillate and residue were analyzed for the dichlorotrifluoroethane by vapor phase chromatography. Table II presents the boiling point data from these tests and Table III gives the results of the analysis of the distillates and residues.

TABLE II.—BOILING POINT OF MIXTURES OF CCl_3F AND CF_3CHCl_2

Mixture, Percent Composition by Weight		Boiling Point, ° C., at 760 mm.
CCl_3F	CF_3CHCl_2	
100	0	23.77
90	10	23.67
78	22	23.62
60	40	23.76
25	75	25.50
0	100	27.60

TABLE III.—CONSTANCY OF BOILING MIXTURES OF CCl_3F AND CF_3CHCl_2 CONTENT OF CF_3CHCl_2 IN MIXTURE, PERCENT BY WEIGHT

Initial Mixture	Distillate	Residue
10.0	10.8	9.5
19.4	19.5	19.4
38.0	37.1	38.6
67.8	52.9	77.7

The mixtures of trichlorofluoromethane and 1,1-dichloro-2,2,2-trifluoroethane having high solvent power for paraffin as shown in Example 1 are azeotropic in character as indicated in Table II by the maximum difference of only 0.14° C. between the boiling point of the mixture containing the 10% and 40% limits for 1,1-dichloro-2,2,2-trifluoroethane and the boiling point of the minimum boiling azeotrope containing about 78% by weight

of trichlorofluoromethane and about 22% by weight of 1,1-dichloro-2,2,2-trifluoroethane. Thus, the mixtures containing 1,1-dichloro-2,2,2-trifluoroethane within the range of 10% to 40% by weight of this component have a constancy of composition with distillation which is confirmed in Table III. Thus, the mixtures of the present invention possess the advantage of a single component solvent having a constancy of composition with boiling.

EXAMPLE 3

Solubility of lanolin

Mixtures of trichlorofluoromethane and 1,1-dichloro-2,2,2-trifluoroethane were saturated at room temperature with lanolin, and the amount of dissolved material was determined by evaporating a portion of each solution. The solubility of lanolin in these mixtures was compared with the solubility of lanolin in trichlorofluoromethane above. The results are shown in Table IV.

TABLE IV.—SOLUBILITY OF LANOLIN IN SOLVENT MIXTURES

Composition of Mixture, Percent by Wt.		Parts of Lanolin in Solution/100 Parts Solvent
$CClF_3$	CF_3CHCl_2	
100	0	36
90	10	59
60	40	58
0	100	41

The above significantly higher solubility of lanolin in mixtures of trichlorofluoromethane and 1,1-dichloro-2,2,2-trifluoroethane containing from 10% to 40% by weight of the latter adapts these compositions particularly well for the scouring of raw wool where a nonflammable, nontoxic, low-boiling, nonfractionating solvent having a high solubility for lanolin is required. The low boiling point of these azeotrope-like mixtures combined with their low heat of vaporization permits rapid, complete, and economic recovery of the solvent from the scouring solutions and from the cleaned wool.

As many apparently widely different embodiments of this invention may be made without departing from the spirit and scope thereof, it is understood that this invention is not limited to the specific embodiments thereof except as defined in the appended claims.

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

1. An azeotropic-like composition consisting essentially of from 60% to 90% by weight of trichlorofluoromethane and 40% to 10% by weight of 1,1-dichloro-2,2,2-trifluoroethane.

2. An azeotrope consisting of about 78% by weight of trichlorofluoromethane and about 22% by weight of 1,1-dichloro-2,2,2-trifluoroethane.

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LEON D. ROSDOL, *Primary Examiner.*

JOHN T. FEDIGAN, *Assistant Examiner.*