

QUASI-AZEOTROPIC MIXTURE BASED ON
1,1,1,3,3-PENTAFLUOROBUTANE, METHYLENE CHLORIDE AND
METHANOL FOR THE TREATMENT OF SOLID SURFACES

The present invention concerns the field of fluorinated hydrocarbons, and relates more particularly to a novel quasi-azeotropic mixture which can be used in various operations for treating solid surfaces, in particular for drying, cleaning, degreasing or dry-cleaning solid surfaces.

1,1,2-Trichloro-1,2,2-trifluoroethane (known in the trade by the name F113) has been used widely in industry for cleaning and degreasing a variety of solid surfaces (metal, glass, plastic and composite parts). Further to its application in electronics for cleaning soldering fluxes in order to remove the flux sticking to printed circuits, mention may be made of its applications for degreasing large metal parts and cleaning high-quality, high-precision mechanical parts such as, for example, gyroscopes and military, aerospace or medical equipment. In its various applications, F113 has most often been combined with other organic solvents (for example methanol), preferably in the form of azeotropic or quasi-azeotropic mixtures which do not demix and which, when used in reflux, have substantially the same composition in the vapour phase as in the liquid phase.

F113 has also been used in industry for drying or dewetting various solid substrates after they have been cleaned in an aqueous medium. In this application, intended to remove the water remaining on the surface of the substrates which have been cleaned, F113 was often supplemented by one or more surfactants (see for example Patents FR 2 353 625, FR 2 527 625, EP 90677 and 189 436, as well as the references cited in these patents).

Since F113 belongs to the chlorofluorocarbons (CFCs) suspected of attacking or degrading stratospheric ozone, it has been proposed to replace it in these

various applications by 1,1-dichloro-1-fluoroethane (known under the name F141b).

Although the ozone depletion potential (ODP) of F141b is much less than that of F113, it is nevertheless not zero, and the use of this substance has already been regulated.

In order to solve this problem, it has been proposed in US Patent 5 350 534 to replace F113 or F141b by an azeotropic mixture consisting, by weight, of from 10 30 to 69% of 1,1,1,3,3-pentafluorobutane (F365 mfc), from 30 to 60% of methylene chloride and from 1 to 10% of methanol. However, the high methylene chloride content of this mixture (30% minimum) makes it unusable for treating solid surfaces consisting entirely or partially of 15 fragile plastics, because its use causes crazing or cracks on these materials and/or makes them tacky.

It has now been found that this drawback can be overcome, and essentially all the advantages of the azeotropic mixture mentioned above can be retained by 20 using a mixture containing, by weight, no more than 15% of methylene chloride, the remainder consisting of from 75 to 95% purely of F365 mfc and from 1 to 10% of methanol, the minimum methylene chloride content being 1%.

25 This mixture allows unproblematic cleaning of sensitive materials such as acrylonitrile-butadiene-styrene copolymers (ABS), polycarbonates (PC) and polymethyl methacrylates (PMMA). Furthermore, this mixture does not exhibit a flashpoint under standard 30 determination conditions (ASTM Standard D 3828) and therefore makes it possible to work in full safety.

A more particularly preferred mixture according to the invention contains, by weight, from 85 to 90% of F365 mfc, from 5 to 10% of methylene chloride and from 2 35 to 5% of methanol.

As in the known compositions based on F113 or F141b, the mixtures according to the invention may, if so desired, be stabilized against the hydrolysis and/or the radical attacks which may take place during cleaning processes. To this end, they are supplemented by a customary stabilizer such as, for example, a nitroalkane, an acetyl or an epoxide, it being possible for the proportion of stabilizer to range from 0.01 to 5% relative to the total weight of the mixture.

10 The mixtures according to the invention can be used under the same conditions and according to the same techniques as the prior compositions based on F113 or F141b.

 The mixtures according to the invention
15 dissolve silicone products, in particular silicone greases. They can therefore be used to clean parts which have silicone derivatives on the surface or to deposit derivatives of this type on these parts, for example by soaking these parts in a solution of silicone in a
20 mixture according to the invention.

 The mixtures according to the invention are non-flammable and evaporate quickly. They can therefore be used, in full safety, in high-speed laser printers.

 The following examples illustrate the invention
25 without limiting it.

EXAMPLE 1

 150 g of a mixture containing, by weight, 89%
30 of F365 mfc, 3.5% of methanol, 7% of methylene chloride and 0.5% of nitromethane (stabilizer) were introduced into an ultrasonic cleaning vessel.

 After the system had been refluxed for one hour, an aliquot of the vapour phase was sampled.
35 Analysis of this, by gas chromatography (see table

below), showed that the composition of the mixture is virtually unchanged and that it is stabilized in the vapour phase.

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	Composition (% by weight)			
	F365 mfc	CH ₂ Cl ₂	Methanol	CH ₃ NO ₂
Initial mixture	89	7	3.5	0.5
Sampled fraction	88.8	6.9	4	0.3

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EXAMPLE 2

Five test circuits (IPC-B-25 standardized model) were coated with colophane-based flux (flux R8F from the company ALPHAMETAL) and stoved at 220°C for 30 15 seconds.

These circuits were cleaned using the quasi-azeotropic mixture in Example 1, in a small ultrasound machine for 3 minutes by immersion and 3 minutes in vapour phase.

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The cleaning was evaluated according to the IPC 2.3.26 standardized procedure with the aid of a precision conductimeter. The value obtained, 2.2 µg/cm² eq. NaCl, is less than the professionally tolerated ion impurity threshold (2.5 µg/cm² eq. NaCl).

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CLAIMS



1. Quasi-azeotropic mixture comprising, by weight, from 75 to 95 parts of 1,1,1,3,3-5 pentafluorobutane, from 1 to 15 parts of methylene chloride, and from 1 to 10 parts of methanol.
2. A mixture according to claim 1, consisting essentially of from 75 to 95% by weight of the pentafluorobutane, from 1 to 15% by weight of methylene 10 chloride and from 1 to 10% by weight of methanol.
3. A mixture according to claim 2 consisting substantially solely of the components recited therein.
4. A mixture according to any of Claims 1 to 3, containing from 85 to 90% of 1,1,1,3,3-15 pentafluorobutane, from 5 to 10% of methylene chloride and from 2 to 5% of methanol.
5. A mixture according to any preceding Claim, furthermore comprising at least one stabilizer.
6. A mixture according to Claim 5, in which 20 the proportion of stabilizer is from 0.01 to 5% relative to the total weight of the mixture.
7. Treatment of solid surfaces which comprises applying thereto a mixture according to any preceding claim.
- 25 8. A treatment according to claim 7, which comprises defluxing a printed circuit board and/or degreasing a mechanical part by applying thereto a mixture according to any of claims 1 to 6.
9. Drying or dewetting of solid surfaces 30 which comprises applying thereto a mixture according to any of claims 1 to 6.

ABSTRACT

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5 1,1,1,3,3-PENTAFLUOROBUTANE, METHYLENE CHLORIDE AND
METHANOL FOR THE TREATMENT OF SOLID SURFACES

In order to replace 1,1,2-trichloro-1,2,2-
trifluoroethane and 1,1-dichloro-1-fluoroethane in
10 compositions for treating solid surfaces (in particular
defluxing), the invention proposes the use of a quasi-
azeotropic mixture containing, by weight, from 75 to 95%
of 1,1,1,3,3-pentafluorobutane, from 1 to 15% of
methylene chloride and from 1 to 10% of methanol.

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