

National Aeronautics and Space Administration



NASA Electronic Parts and Packaging (NEPP) Program

JC-13 Fluorocarbon Tech Discussion

Prepared by: Dr. Carol Putman / NASA – MSFC
Benny Damron / NASA – MSFC

September 13, 2023

www.nasa.gov

Use of Perfluorocarbons Fluids on EEE parts Testing

Test Methods of interest:

- MIL-STD-883 - Gross Leak Test – Hermeticity Test - ICs
- MIL-STD-750 - Gross Leak Test – Hermeticity Test - Transistors/diodes
- MIL-STD-202 - Gross Leak Test – Hermeticity Test - passives (i.e relays)

Others Potential ones :

- High Voltage Testing
- Thermal Shock - MIL-STD-202-1 TM 107, MIL-STD-750-1 TM 1056 & MIL-STD-883-1 TM 1011

End Goal: To make aware JEDEC members of needed modifications /updates in standards that use fluorocarbon electrical fluids to address test sensitivity (discuss potential root cause of False/Passes) and change of content in standards when mentioning trademark names of electrical fluids to be phase out by 3M due to PFAs content.

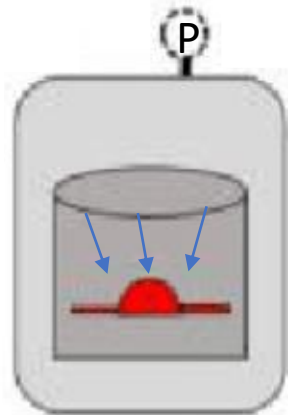
Perfluorocarbon Gross Leak Test

The gross leak test is an inspection test based on boiling points differential of two fluorocarbons. It requires a preconditioning in a **pressurized chamber filled with a low boiling point fluorocarbon fluid (detector) (Type I)**. Under the pressure, the Detector fluid may penetrate the device. After the preconditioning, the device is **submerged in a second fluorocarbon fluid (indicator) with a higher boiling point (Type II)**. When **exposed to the high temperature (125C)**, the detector fluid located within the cavity of the package boils, generating an elevated pressure inside the device causing a **bubble stream to emanate from the leak site**.

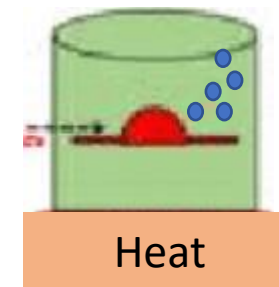
Fluid Characteristics

- Two fluorocarbon fluids with wide temperature range. (Boiling Point)
- Non-conductive fluid (High dielectric strength)
- Inert to surfaces (Fluorinated Fluid)
- Evaporates without leaving any residues (no solvents, low non-volatiles residue)
- Low surface tension

Step 1: Parts pressurization in Type I fluid (detector)
Parameters (Pressure, time)



Step 2: Transfer to Type II fluid (indicator) and heat up for gasification and release of Type I fluid (hermeticity fail)
Parameters (Temperature, time, height for immersion)



MIL-STD-883-1 Test method 1014 Seal Test Perfluorocarbon Fluid Requirements

2.3 Test condition C, Perfluorocarbon gross leak.

2.3.1 Apparatus. Apparatus for this test shall consist of:

- a. **A vacuum/pressure chamber for the evacuation and subsequent pressure bombing of devices up to 105 psia up to 23.5 hours. (enough preconditioning pressure for small packaging ?)**
- b. **A suitable observation container with provisions to maintain the indicator fluid at a temperature of 125C and a filtration system capable of removing particles greater than 1 micrometer in size from the fluid (condition C1 only).**
- c. A magnifier with a magnification in the range between 1.5X to 30X for observation of bubbles emanating from devices when immersed in the indicator fluid (condition C1 only).
- d. Sources of type I detector fluids, and type II indicator fluids as specified in Table II.
- e. A lighting source capable of producing at least 15-thousand-foot candles in air at a distance equal to that which the most distant device in the bath will be from the source. The lighting source shall not require calibration but the light level at the point of observation (i.e., where the device under test is located during observation for bubbles), shall be verified (condition C1 only).
- f. Suitable calibrated instruments to indicate that test temperatures, pressures, and times are as specified.
- g. Suitable fixtures to hold the device(s) in the indicator fluid (condition C1 only).
- h. **A perfluorocarbon vapor detection system capable of detecting vapor quantities equivalent to 0.167 or 1/6 microliter of type I fluid (condition C3 only). (Packaging sizes, going down to 0.01 and 0.001 cc, test sensitivity)**
- i. The vapor detector used for condition C3 shall be calibrated at least once each working shift using a type I fluid calibration source and following the manufacturer's instructions.

MIL-STD-883-1 Test method 1014

Seal Test perfluorocarbon Fluid Requirements

TABLE II. Physical property requirements of perfluorocarbon fluids. ^{1/}

Property	Type I	Type II	Type III	ASTM test method
Boiling point (°C)	50-95	140-200	50-110	D-1120
Surface tension (Dynes/cm) at 25°C		< 20		D-971 D-1331
Density at 25°C (gm/ml)	> 1.6	> 1.6	> 1.6	
Density at 125°C (gm/ml)		> 1.5		
Dielectric strength (volts/mil)	> 300	> 300	> 300	877
Residue (µgm/gm)	< 50	< 50	< 50	D-2109
Appearance	Clear colorless			NA

^{1/} Perfluorocarbons contain no chlorine or hydrogen.

Test Property	Min. Spec	Max. Spec	Unit	Result/Average
Density	1.845	1.895	g/ml	1.880
Appearance				Pass
Distillation Range	95		%	98
Residue		10	µg/ml	< 1
Pour Point		-50	°C	< -53
Viscosity	1.6	2.8	mm ² /s	2.5
Boiling Point	158.0	173.0	°C	160.7
Dielectric Strength 81.0.C	35		kV	58

Fluid Concern – “Material at Risk” due to 3M PFAs phase out ?

- Possibility of reclaimed fluids from one vendor
- Twain source from one vendor
- Residue quality spec is absence in at least one vendor. Residue value ensures that tested devices dry quickly, and no post-test cleaning is required.



Fluid Manufacturers List by DLA

- 1) 3M - Fluorinerts trademark (FC40, FC43, FC72), which are in the list of PFAs Phase out portfolio by 2025
- 2) Solvay – Galden trademark (Galden DET, Galden D02)
- 3) TMC Industries (Flutec PP1, TMC FC40)
- 4) Flourez Technologies (FL-50, FL-165)

MIL-STD-750-1, Test method 1071

Seal Test perfluorocarbon Fluid Requirements and FC cavity Limitations

6. Test condition C – liquid fluorocarbon gross leak. **Device packages with less than 0.05 cm³ internal free volume** shall be tested with extremely careful observation. The number of test samples, configuration, and all critical test criteria shall be included in the manufacturer's test procedure. **(Packaging size concern and test sensitivity) – False/passes**

6.1 Apparatus. The apparatus required for test condition C shall be as follows:

a. A vacuum/pressure chamber for the evacuation and subsequent pressure bombing of devices up to 90 psi (620 kPa) for a maximum of 24 hours. **(Do we need precondition at higher pressure for smaller packaging devices ?**

b. A suitable observation container with provisions to maintain the indicator fluid at a temperature of $+125^{\circ}\text{C} \pm 5^{\circ}\text{C}$ ($+100^{\circ}\text{C}$ for Germanium transistors with temperature rating of $+100^{\circ}\text{C}$ maximum) **and a filtration system capable of removing particles greater than one micrometer in size from the fluid.**

c. A magnifier capable of magnifying an object 1.5X to 30X its normal size (4 to 120 diopters) for observation of bubbles emanating from devices when immersed in the indicator fluid.

d. Sources of type I detector fluids and type II indicator fluids as specified in table 1071-II.

7. Test condition D- Bubble Test

Paragraph 7.1.a **Only devices with internal free volume greater than 0.05 can be tested by this method.** **(Packaging size concern and test sensitivity) – False/passes**

MIL-STD-750-1, Test method 1071

Seal Test perfluorocarbon Fluid Requirements

- TABLE 1071-II. Physical property requirements of perfluorocarbon fluids. 1/

Property	Type I	Type II	Type III	ASTM test method
Boiling point (°C)	50-95	140-200	50-110	D1120
Surface tension (dyness/cm) at +25°C		< 20		D971, D1331
Density at +25°C (gm/ml)	> 1.6	> 1.6	> 1.6	Not applicable
Density at +125°C (gm/ml)		> 1.5		Not applicable
Dielectric strength (volts/mil)	> 300	> 300	> 300	D877
Residue (Tgm/gm)	< 50	< 50	< 50	D2109
Appearance	Clear colorless			N/A

1/ Perfluorocarbons contain no chlorine or hydrogen.

Action item : Obtain specs from listed DLA manufacturers and compare reported quality parameters

MIL-STD-202-1, Test method 112

Seal Test perfluorocarbon Fluid Requirements

4.5. Test condition D.

4.5.1 Materials.

4.5.1.1 Fluorocarbon liquid. The fluid shall be D02, D02-TS, D03, **FC-40 1/ or FC-43 2/.** (Fluid modification)

4.5.2 Apparatus.

4.5.2.1 Heated fluid container. The container for the fluid shall be made of pyrex glass and shall be sufficient size to hold the test specimen in the fluid and to maintain a temperature of **125°C ±5°C (257°F ±9°F).**

1/ D02, D02-TS, and D03 are the registered trade names). Fix the missing letters

2/ **Minnesota Mining Co. (3M) registered trade name.** (Require change)

MIL-STD-202-1, Test method 112

Seal Test perfluorocarbon Fluid Requirements

4.6. Test condition E.

4.6.1 Materials.

4.6.1.1 Fluorocarbon liquid. **The fluids shall be D/80, FC-72 or PP-1 fluorocarbon detector fluids, and D02, D02-TS, D03, FC-40, FC-43, PP-7 or PP-9 fluorocarbon indicator fluids. (Fluid modification)**

4.6.2 Apparatus.

4.6.2.1 Heated fluid container. The container for the fluid shall be made of pyrex glass and shall be of sufficient size to hold the test specimen in the fluid and to maintain a temperature of **125°C ±5°C (257°F ±9°F)**.

4.6.2.2 Filter. A filter shall be used which is capable of removing particles greater than 1 micron in size from the fluid.

4.6.2.3 Magnifier. A magnifier with a magnification in the range between 3X to 30X for observation of bubbles emanating from devices when immersed in the indicator fluid.

4.6.2.4 Chamber. A vacuum/pressure chamber for the evacuation and subsequent pressure bombing of device up to **75 lbf/in² up to 10 hours. (Test sensitivity/ preconditioning for small packaging devices ?)**

MIL-STD-202-1, Test method 112

Seal Test perfluorocarbon Fluid Requirements

4.7. Test condition F.

4.7.1 Materials.

4.7.1.1 Fluorocarbon liquid. The fluid used shall be **FC-84 or D/80 fluorocarbon detector fluid.** (Fluid modification)

4.7.2 Apparatus.

4.7.2.1 Chamber. A vacuum/pressure chamber capable of the evacuation and subsequent pressure bombing of devices up to 75 psig and maintaining pressure up to 12.5 hours.

4.7.2.2 Filter. A filtration system capable of removing particles greater than 1 micron in size from the fluorocarbon fluid.

4.7.2.3 Detector. A fluorocarbon vapor detection system with a nominal sensitivity of 10^{-5} atm cm³/s. The sensitivity of the detector shall be better than 14 micrograms of **FC84 or D/80**. The detector shall have a linear response to 280 micrograms and a response time of better than 1 second.

4.7.2.4 Instruments. Suitable calibrated instruments to indicate that test, pressure times and temperatures are as specified. The detection system shall be calibrated using a calibration source of fluorocarbon at least once each shift to an accuracy of at least ± 15 percent.

4.7.3 Precautions. The following precautions shall be observed in conducting the fluorocarbon gross leak test.

a. **Fluorocarbons shall be filtered through a filter system capable of removing particles greater than 1 micrometer prior to use.** Bulk filtering and storage is permissible. Liquid that has accumulated observable quantities of particulate matter during use shall be discarded or reclaimed by filtration for re-use.

Precautions shall be taken to prevent contamination.

b. Precautions should be taken to prevent operator injury due to package rupture or violent evolution of bomb fluid when testing large packages.

MIL-STD-202-1, Test method 107

Thermal Shock Perfluorocarbon Fluid Requirements

TABLE IV. Suggested thermal fluids. ^{1/} ^{2/}

Test condition	AA, AA-1, AA-2 fluids	BB, BB-1, BB-2 Fluids	CC, CC-1, CC-2 fluids	DD, DD-1, DD-2 fluids
Step 1	FC40 ^{4/} or Water ^{3/} D02 } D02-TS } ^{6/} D/80 }	FC77 ^{4/} D02 } D02-TS } ^{6/} D/80 }	FC77 ^{4/} D02 } D02-TS } ^{6/} D/80 }	FC77 ^{4/} D02 } D02-TS } ^{6/} D/80 }
Step 2	FC40 ^{4/} Water ^{3/} D02 } D02-TS } ^{6/} D03 }	FC70 FC40 } ^{4/} UCON-WS ^{5/} D02 } D02-TS } ^{6/} D03 }	FC70 FC40 } ^{4/} UCON-WS ^{5/} D02 } D02-TS } ^{6/} D03 }	FC70 ^{4/} UCON-WS } ^{5/} D05 } LS/230 } ^{6/} LS/215 }

^{1/} See 4.2.2.

^{2/} Ethylene glycol shall not be used as a thermal shock test fluid.

^{3/} Tap water is indicated as an acceptable fluid for this temperature range. Its suitability chemically shall be established prior to use. A mixture of water and alcohol may be used to prevent freezing at the low temperature extreme. The water shall not be allowed to boil at the upper temperature extreme.

^{4/} FC77, FC70, FC40 are the registered trademark of 3M.

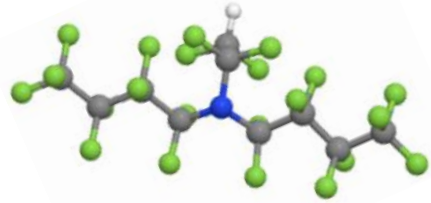
^{5/} UCON-WS process fluid is the registered trademark of Union Carbide Corporation.

^{6/} D02, D02-TS, D03, D05, D/80, LS/215 and LS/230 are the registered trademark of Ausimont (Division of Montedison).

Perfluorocarbons Electrical Fluids

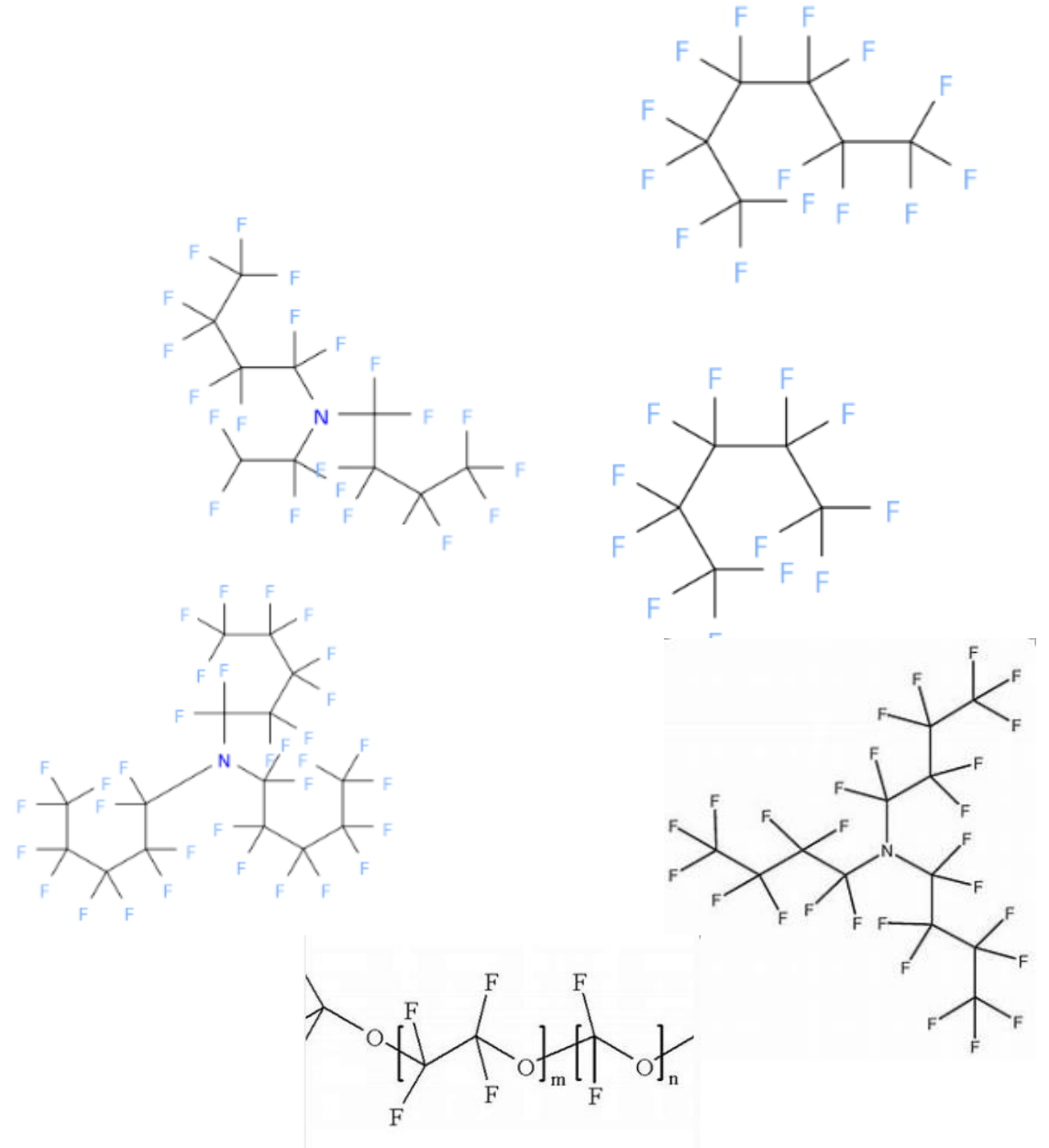
- What are they ?

Fluorinated (C-F bonds), non-conductive, thermally and chemically stable fluid.



Other names

- Per-fluorinated liquids
- Fluorinated electrical fluids
- PFAs fluid type: Chain of C atoms surrounded by F atoms, chemically stable, hydrophobic, with O, H, S, N atoms to create a polar end



Perfluorocarbons Electrical Fluids - Options

Electrical Fluorinated Fluids					
Manufacturer	Trade name	Grade			
			DLA list		DLA list
3M	Fluorinert	FC72	yes	FC40	yes
Solvay	Galden	Galden HT55		Galden HT170	
		Galden DET	yes	Galden HT200	
				Galden D02 / D02TS	yes
Chemours	Opteon	SF10			
TMC Industries		Flutec PP1	yes	TMC FC-40	yes
Fluorez Technology		FL-50	yes	FL-165	yes



Type I
Low boiling point
Detector



Type II
High boiling point
Indicator

Additional Tables

MIL-STD-750-1, Test method 1056

Thermal Shock perfluorocarbon Fluid Requirements

TABLE 1056-I. Physical property requirements of perfluorocarbon fluids. ^{1/}

Test condition		C	D	ASTM test method
Step 1	Boiling point, °C	>125	>150	D1120
	Density at 25°C gm/ml		>1.6	Not applicable
	Dielectric strength volts/mil		>300	D877
	Residue, microgram/gram		<50	D2109
	Appearance	Clear, colorless liquid		Not applicable
Step 2	Density at 25°C gm/ml		>1.6	Not applicable
	Dielectric strength volts/mil		>300	D877
	Residue, microgram/gram		<50	D2109
	Appearance	Clear, colorless liquid		Not applicable

^{1/} The perfluorocarbon used shall have a viscosity less than or equal to the thermal shock equipment manufacturer's recommended viscosity at the minimum temperature.

MIL-STD-883-1, Test method 1011

Thermal Shock perfluorocarbon Fluid Requirements

TABLE II. Physical property requirements of perfluorocarbon fluids. ^{1/}

Test condition		B	C	ASTM test method
Step 1	Boiling point, °C	>125	>150	D1120
	Density at 25°C gm/ml Dielectric strength volts/mil Residue, microgram/gram Appearance	>1.6 >300 <50 Clear, colorless liquid		D877 D2109 Not applicable
Step 2	Density at 25°C gm/ml Dielectric strength volts/mil Residue, micrograms/gram Appearance	>1.6 >300 <50 Clear, colorless liquid		D877 D2109 Not applicable

^{1/} The perfluorocarbon used shall have a viscosity less than or equal to the thermal shock equipment manufacturer's recommended viscosity at the minimum temperature.