Envirotemp® FR3® Fluid

Testing Guide

Section R900-20-12
Reference Document
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This guide neither claims to cover all testing variations nor provides for every possible testing contingency. It is the responsibility of the user to ensure that specific methods, processes, data interpretations, and treatments are suitable for Envirotemp FR3 fluid. Contact Cooper Power Systems - Dielectric Fluids for further information.

INTRODUCTION

Envirotemp® FR3® fluid is a dielectric coolant formulated for application in electrical equipment. It is suitable for use in new apparatus as well as retrofilling existing apparatus. It is a highly biodegradable, non-toxic fluid based on the natural ester chemistry of food-grade vegetable oil. It has been granted Environmental Technology Verification status by the US Environmental Protection Agency and Environmental Technology Certification by the California Environmental Protection Agency. It is also a "Nationally Recognized Testing Laboratory" listed less-flammable fluid. This guide discusses the application of standard dielectric fluid tests to new and in-service Envirotemp FR3 fluid, describes how the methods differ from those of conventional mineral oils, and compares their specifications.

DIFFERENCES IN FLUID PROPERTIES

Physical, chemical, and electrical properties are used to specify and evaluate new electrical insulating fluids and monitor in-service fluids [1]. Some traditionally acceptable indicators of mineral oil performance may not apply or may have different values for Envirotemp FR3 fluid.

The chemical composition of FR3 fluid is a mixture of relatively polar triglycerides (long-chain fatty acid ester molecules) having some unsaturation and readily form hydrogen bonds. Conventional mineral oil consists of cyclic naphthenes, branched alkanes, and aromatic molecules. These relatively low boiling point compounds are non-polar and hydrophobic. The difference in basic chemistry between vegetable oil and mineral oil accounts for disparate values in several tests assessing fluid characteristics. Table 1 shows the historical range of values (useful for transformer designers) for Envirotemp FR3 fluid and compares the ASTM specifications for mineral oil [2] and natural ester [3] dielectric fluids.

PERFORMANCE TESTS

Insulating fluids provide both electrical insulation and cooling capability. Two key properties that affect the function and performance of an insulating fluid are dielectric breakdown voltage and viscosity. The dielectric breakdown measures the effectiveness as

TABLE 1: Envirotemp FR3 fluid values and specification limits for natural ester fluid and mineral oil [2,3]

Test	ASTM Method	Historical Range for New Processed FR3 Fluid Prior to Packaging	Specification for New As-Received Fluid: Natural Ester ASTM D6871	Specification for New As-Received Fluid: Mineral Oil ASTM D3487
Dielectric Breakdown [kV]	D877	42 – 48	≥ 30	≥ 30
1mm gap	D1816	25 – 37	≥ 20	≥ 20
2mm gap		50 – 75	≥ 35	≥ 35
Dissipation Factor [%]	D924			
25°C		0.02 – 0.10	≤ 0.20	≤ 0.05
100°C		1.0 – 3.5	≤ 4.0	≤ 0.30
Volume Resistivity [Ω-cm]	D1169	$10 – 30 \times 10^{12}$	–	–
Kinematic Viscosity [mm^2/sec]	D445			
40°C		33 – 35	≤ 50	≤ 12.0
100°C		8.0 – 8.5	≤ 15	≤ 3.0
Water Content [mg/kg]	D1533	20 – 50	≤ 200	≤ 35
Pour Point [°C]	D97	-24 – -21	≤ -10	≤ -40
Acid Number [mg KOH/g]	D974	0.02 – 0.04	≤ 0.06	≤ 0.03
Gassing Tendency [µl/min]	D2300	-78 – -80	≤ 0	–
Interfacial Tension [mN/m]	D971	20 – 25	–	≥ 40
PCB Content [ppm (wt)]	D4059	not detectable	not detectable	not detectable
Flash Point [°C]	D92	310 – 330	≥ 275	≥ 145
Fire Point [°C]	D92	350 – 360	≥ 300	–

electrical insulation. The viscosity influences the cooling performance.

Dielectric Breakdown Voltage

ASTM D1816 [4]: The only modification to the D1816 test method is the stand time before test. The method calls for a stand time of 3-5 minutes. Because the viscosity of FR3 fluid is slightly higher than conventional mineral oil, a 30-minute stand time is recommended between pouring the room temperature equilibrated fluid sample and starting the test. This gives entrained air sufficient time to escape after pouring the sample.

ASTM D877 [5]: The stand time specified in this method is 2-3 minutes. As with D1816, a 30-minute stand time is recommended. (Although the ASTM D877 method for dielectric breakdown voltage works well for Envirotemp FR3 fluid, the D1816 method is preferred to D877 for all fluids. D877 is less sensitive to dissolved gas, water and particulate matter.)

EFFECT OF WATER CONTENT: The breakdown strength of any dielectric fluid starts to decrease as the water content increases to about 40% relative saturation. At room temperature, 40% relative saturation in mineral oil occurs at an absolute water content of approximately 25 mg/kg (or ppm), and about 400 mg/kg in Envirotemp FR3 fluid. In order to get a meaningful comparison of water content in different types of dielectric fluids, we must use relative saturation rather than the absolute water content in mg/kg. Figure 1 compares the D1816 dielectric strength versus absolute and relative water content for Envirotemp FR3 fluid and mineral oil.

Viscosity

The kinematic viscosity of FR3 fluid is slightly higher than that of mineral oil. Use ASTM D445 [6] without modification.

Diagnostic Tests

The properties in this category do not directly affect transformer performance, but rather are used as indicators of changes in the fluid over time due to transformer operation. The trends are at least as useful as the values themselves. The quality of Envirotemp FR3 fluid is measured using the same standard test methods used for conventional mineral oil. However, due to the differences in their chemistry, the normal base line values will be different for certain properties.

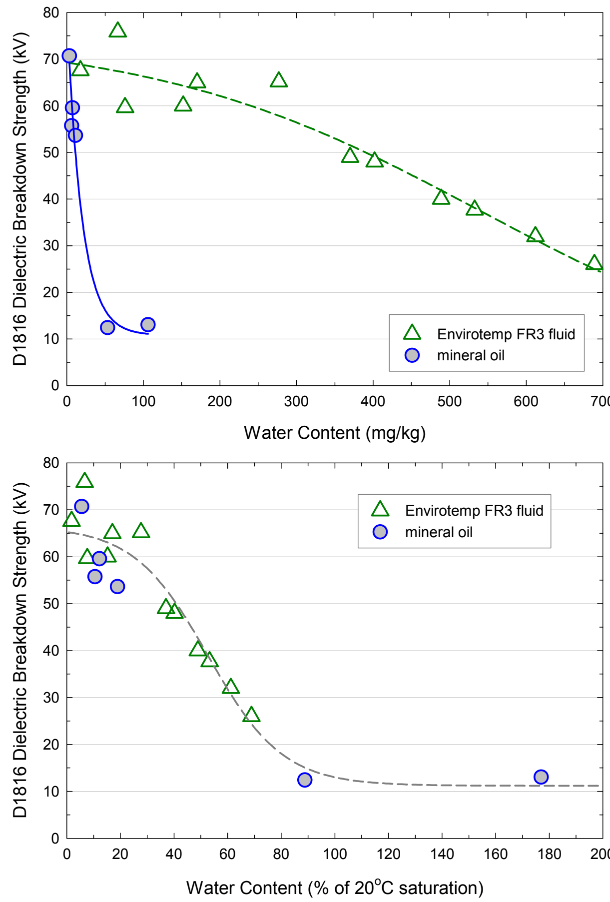

Figure 1. Dielectric breakdown strength *versus* water content for mineral oil and Envirotemp FR3 fluid. Top graph shows breakdown strength *versus* absolute water content; bottom shows breakdown strength *versus* relative water content.

The polarity and molecular structure of esters influence the values obtained from the standard ASTM tests. Water content, dissipation factor, pour point, and acid number are typically higher than those of conventional transformer oil. Interfacial tension, gassing tendency and resistivity are normally lower than mineral oil. Other tests, such as furanic compound content, require specific test methods to obtain reliable results.

Water Content

Use ASTM D1533 [7] without modification.

> NOTE: If erratic or unusual results are obtained, use the Karl Fischer reagents for aldehydes and ketones instead of those for mineral oil, as recommended in D1533.

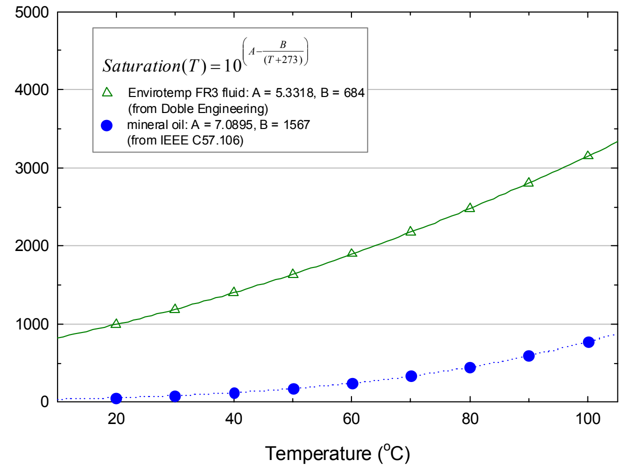

Figure 2. Water saturation versus temperature for mineral oil and Envirotemp FR3 fluid

Water saturation versus temperature is shown in Fig. 2. The room temperature water saturation of Envirotemp FR3 fluid is about 1000 mg/kg, and about 55 mg/kg for mineral oil. This high capacity for water is one of the important attributes of Envirotemp FR3 fluid, and is a major factor in the longer life of Kraft paper insulation compared to its life in mineral oil.

New processed FR3 fluid typically contains 20-50 mg/kg of water. ASTM D6871 Standard Specification for Natural Ester Fluids Used in Electrical Apparatus allows a maximum of 200 mg/kg.

Dissipation Factor

Use ASTM D924 [8] without modification. When using a single test cell for both mineral oil and Envirotemp FR3 fluid dissipation measurements, the cell must be meticulously cleaned when changing from one type of fluid to another. This is especially true when measuring Envirotemp FR3 fluid after mineral oil. Artificially high values may be seen if the cell is not sufficiently clean.
The dissipation factor of new FR3 fluid is naturally higher than new mineral oil. Values of 0.05 to 0.10% at 25°C for new fluid are typical; values up to 0.2% are acceptable per ASTM D6871. The chemical makeup of ester fluid is relatively polar compared to mineral oil. This characteristic, along with the higher acid number, explains the higher dissipation factors.

Acid Number

Use ASTM D974 [9] without modification. New Envirotemp FR3 fluid naturally contains small amounts of free fatty acids that result in acid numbers higher than those typically seen in mineral oil. As the FR3 fluid ages, it reacts with water (hydrolysis), generating additional long-chain fatty acids. The long-chain fatty acids are mild and non-corrosive compared to the short chain organic acids found in mineral oil. Although the acid number method determines the amount of acidic components present, it does not tell us the type or reactivity of the acid.

Interfacial Tension

Use ASTM D971 [10] without modification. Interfacial tension should in theory be as useful for Envirotemp FR3 fluid as it is for mineral oil. However, more service history data is required to establish limits. New Envirotemp FR3 fluid has an inherently lower IFT value compared to mineral oil.

Resistivity

Use ASTM D1169 [11] without modification. For the same reasons that the dissipation factor of Envirotemp FR3 fluid is higher than mineral oil, the resistivity is lower.

Pour Point

The pour point of Envirotemp FR3 fluid is typically in the -24 – -21°C range, and is higher than that of mineral oil. Carefully follow ASTM D97 [12] to obtain accurate results. Heat fluid samples to 130°C and cool to room temperature before starting a pour point determination. Cooling ramp rates and pour point monitoring intervals need to conform to specification. Strict adherence to the D97 method is required to prevent inaccurate pour point values.

ASTM D5950 [13] can also be used to determine the pour point of Envirotemp FR3 fluid. Note that D5950 consistently gives a pour point about 3°C lower than D97. The main reason is the difference in optical sensitivity of the detector versus the human eye to the refracted light.

Gassing Tendency

The ASTM D2300 [14] gassing tendency for FR3 fluid is -79 µl/min, significantly lower than that of mineral oil. The high degree of polyunsaturation has a greater tendency and capacity to absorb hydrogen under partial discharge conditions.

TABLE 2. Gas chromatography details used by Cooper Power Systems for ASTM D4768 determination of oxidation inhibitor in Envirotemp FR3 fluid

Instrument	HP5890 Series II
Column	J&W DB-5ms, 15m x 0.32mm, 1.0µm film
Flow rate	4 ml/min helium @ 50°C
Split Ratio	10:1
Oven Temperature	50°C for 1.0 min, ramp 10°C/min to 300°C, hold 300°C for 20 min
Injector Temperature	300°C
Detector	FID at 335°C
Solution Concentration	0.5g/10ml heptane for all standards and samples

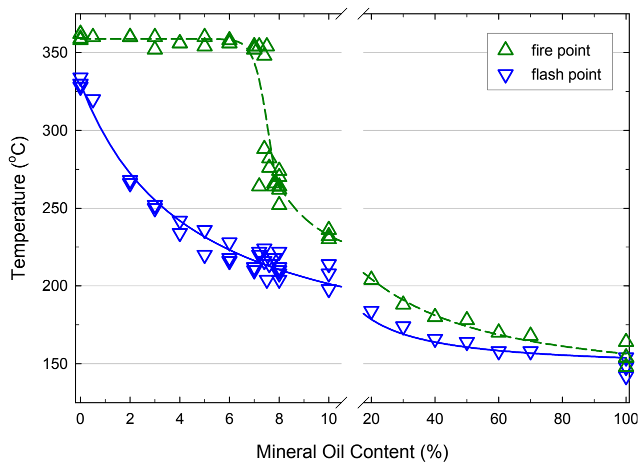

FIGURE 3. Flash and fire points versus mineral oil content.

Oxidation Inhibitor

Measure the oxidation inhibitor content using a gas chromatograph (GC) and the ASTM D4768 [15] method. This method is preferred over the infrared (IR) technique because esters and ester by-products absorb IR in the same region(s) as the inhibitor additives. The GC method is specific and accurate when using properly prepared standards. One practical set GC operating parameters is shown in Table 2. Consider replenishing the inhibitor if the inhibitor content falls below 0.12%.

Note that mineral oil oxidation stability tests are not suitable for use with natural ester fluids. Cooper Power Systems recommends using an oxidation induction time method employing a pressurized air differential scanning calorimeter to compare natural ester fluids and evaluate inhibitor additives. Contact Cooper Power Systems for method details.

Oxidation Stability

As of this writing, ASTM has not published an oxidation stability method applicable to natural ester fluids. Until such time as ASTM publishes a suitable method, Cooper Power Systems recommends using a pressure differential scanning calorimetric (PDSC) method [16] to determine the oxidation induction time. Contact Cooper Power Systems to obtain the detailed procedure.

PCB Content

New Envirotemp FR3 fluid contains no detectable polychlorinated biphenyls (PCB). PCB content is measured in accordance with ASTM D4059 [17] using a packed column. Accurate results can be obtained using the sulfuric acid treatment to remove interferences.

Adsorbent treatment to remove interferences is not recommended. PCB contents of spiked lab samples prepared using the adsorbent treatment were consistently low.

Flash and Fire Points

Use ASTM D92 [18] without modification. Contamination by more volatile fluids lowers the flash point, and may lower the fire point. Flash point values can be used to estimate the residual amount of mineral oil in a transformer retrofilled FR3 fluid. Figure 3 shows the flash and fire points as a function of mineral oil content in FR3 fluid.

Dissolved Gas Analysis

Samples of Envirotemp FR3 fluid for dissolved gas determinations are taken and analyzed using the same procedures and techniques as those used for mineral oil [19-21]. The data are interpreted in much the same way as for gases in mineral oil.

The combustible gases generated by faults in natural ester fluids are similar to those in mineral oil: high levels of hydrogen may be an indication that partial discharge is occurring; carbon oxides in certain ratios suggest overheated paper; hydrocarbon gases could result from a thermal fault in oil; acetylene points to arcing. Always, the first step is to determine if a fault exists using the amounts and generation rates of dissolved gases before trying to further interpret the gas data. The most useful approaches to dissolved gases in Envirotemp FR3 fluid use the gas generation rates combined with the IEEE Key Gases method or the IEC Duval method [22].

Corrosive Sulfur

The ASTM D1275 methods [23] for detecting corrosive sulfur can be applied without modification to Envirotemp FR3 fluid. Note that the method uses the intensity of discoloration observed on a copper strip to determine the presence of corrosive sulfur. The discoloration of a strip, especially at higher temperatures or longer aging times, can also be due to non-sulfur sources. Semi-quantitative elemental surface analysis using energy dispersive x-ray spectroscopy (EDS or EDX) provides the needed source discrimination.

Furanic Compounds

The ASTM D5837 method [24] for determining furanic content in mineral oils was applied to Envirotemp FR3 fluid although the method does not specifically include natural ester fluids in its scope. The technique works very well for both new mineral oil and new Envirotemp FR3 fluid. As the Envirotemp FR3 fluid becomes degraded, the method begins to suffer from interferences from other degradation products being extracted and concentrated along with the furans. These interferences raise the lower detection limit and increase the error of the method.

Solid phase micro-extraction (SPME) combined with gas chromatography/mass spectroscopy is now under investigation. Promising results have been obtained using new Envirotemp FR3 fluid, with very good responses for 4 of the 5 furanic compounds usually quantified by the method. A baseline for aged Envirotemp FR3 fluid has been set, but neither lower detection limits nor error levels for new or aged FR3 have been determined. Work is continuing on the solid phase micro-extraction method to optimize experimental parameters, establish lower detection limits, and generate calibration curves for the 5 furans.

Particle Count

The ASTM D6786 method [25] for determining the number of particles in a fluid can be used if the sample is diluted. The viscosity of Envirotemp FR3 fluid may not allow the air bubbles entrained in the fluid to dissipate in the time allowed to make the measurement.

Dilute the Envirotemp FR3 fluid about 75% with pre-filtered heptane or hexane. Filter the solvent using a 0.2 μm membrane filter. Use the filtered solvent to obtain a blank value for particles. This works effectively unless the particle count in the fluid is low. In that case, less dilution and some trial and error are required. Note that the applied pressure should be limited to 80 psi in order to minimize the pressure drop across the cell.

A high particle count can be the result of crystallites in fluid recently below the cloud point temperature. If this is the case, warm the fluid to re-dissolve the crystallites.

LABORATORY NOTE

Remember to thoroughly clean the laboratory equipment after test completion. Thin films of natural ester fluids have a greater tendency, compared to mineral oil, to oxidize and eventually polymerize when exposed to air. Fresh natural ester films are easily cleaned, but polymerized films require some scrubbing.

REFERENCES

[1] ASTM D117 “Standard Guide for Sampling, Test Methods, and Specifications of Electrical Insulating Oils of Petroleum Origin”, ASTM International

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[3] ASTM D3487 “Standard Specification for Mineral Insulating Oil Used in Electrical Apparatus”, ASTM International

[4] ASTM D1816 “Standard Specification for Mineral Insulating Oil Used in Electrical Apparatus”, ASTM International

[5] ASTM D877 “Standard Test Method for Dielectric Breakdown Voltage of Insulating Liquids Using Disk Electrodes”, ASTM International

[6] ASTM D445 “Standard Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (the Calculation of Dynamic Viscosity)”, ASTM International

[7] ASTM D1533 “Standard Test Method for Water in Insulating Liquids by Coulometric Karl Fischer Titration”, ASTM International

[8] ASTM D924 “Standard Test Method for Dissipation Factor (or Power Factor) and Relative Permittivity (Dielectric Constant) of Electrical Insulating Liquids”, ASTM International

[9] ASTM D974 “Standard Test Method for Acid and Base Number by Color-Indicator Titration”, ASTM International

[10] ASTM D971 “Standard Test Method for Interfacial Tension of Oil Against Water by the Ring Method”, ASTM International

[11] ASTM D1169 “Standard Test Method for Specific Resistance (Resistivity) of Electrical Insulating Liquids”, ASTM International

[12] ASTM D97 “Standard Test Method for Pour Point of Petroleum Products”, ASTM International

[13] ASTM D5950 “Standard Test Method for Pour Point of Petroleum Products (Automatic Tilt Method)”, ASTM International

[14] ASTM D2300 “Standard Test Method for Gassing of Electrical Insulating Liquids Under Electrical Stress and Ionization (Modified Pirelli Method)”, ASTM International

[15] ASTM D4768 “Standard Test Method for Analysis of 2,6-Ditertiary-Butyl Para-Cresol and 2,6-Ditertiary-Butyl Phenol in Insulating Liquids by Gas Chromatography”, ASTM International

[16] M. Slovachek, “Oxidation Induction Time (OIT) Method Development”, ML 2005090.005, Thomas A. Edison Technical Center, Cooper Power Systems, Aug. 9, 2007

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[18] ASTM D92 “Standard Test Method for Flash and Fire Points by Cleveland Open Cup”, ASTM International

[19] ASTM D3612 “Standard Test Method for Analysis of Gases Dissolved in Electrical Insulating Oil by Gas Chromatography”, ASTM International

[20] “IEEE Guide for the Interpretation of Gases Generated in Oil-Immersed Transformers”, IEEE Std. C57.104-1991, Institute of Electrical and Electronics Engineers, New York, USA

[21] “Mineral oil-impregnated electrical equipment in service – Guide to the interpretation of dissolved and free gases analysis”, IEC Standard 60599, Edition 2.0, 1999-03, International Electrotechnical Commission, Geneva, Switzerland

[22] “Envirotemp FR3 Fluid Dissolved Gas Guide”, Section R900-20-19, Cooper Power Systems, August 2006

[23] ASTM D1275 “Standard Test Method for Corrosive Sulfur in Electrical Insulating Oils”, ASTM International

[24] ASTM D5837 “Standard Test Method for Furanic Compounds in electrical Insulating Liquid by High-Performance Liquid Chromatography (HPLC)”, ASTM International

[25] ASTM D6786 “Standard Test Method for Particle Count in Mineral Insulating Oil Using Automatic Optical Particle Counters”, ASTM International