

Test Methods
Evaluation of Nonflammable, Chlorotrifluoroethylene
Base Hydraulic Fluid

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SAE AS4707 R vision A

1. SCOPE:

This SAE Aerospace Standard (AS) covers laboratory test methods required to evaluate nonflammable hydraulic fluids. The methods of testing and description of the equipment have been used typically for evaluating chlorotrifluoroethylene (CTFE) base hydraulic fluids.

1.1 Classification:

Test methods are described in detail as follows:

- a. METHOD 1 - Saturation Determination for Chlorotrifluoroethylene (CTFE) - Tests for Oxidizable Materials
- b. METHOD 2 - Capillary Gas Chromatography - CTFE Fluid Analysis
- c. METHOD 3 - Thermal Stability
- d. METHOD 4 - Corrosion Rate Evaluation - CTFE Fluid
- e. METHOD 5 - Bulk Modulus
- f. METHOD 6 - Performance of Hydraulic Fluid in a High Performance Hydraulic Pump

1.2 Safety - Hazardous Materials:

While the materials, methods, applications, and processes described or referenced in this document may involve the use of hazardous materials, this document does not address the hazards which may be involved in such use. It is the sole responsibility of the user to ensure familiarity with the safe and proper use of any hazardous materials and to take necessary precautionary measures to ensure the health and safety of all personnel involved.

2. REFERENCES:

2.1 Applicable Documents:

The following publications form a part of this specification to the extent specified herein. The latest issue of SAE publications shall apply. The applicable issue of other publications shall be the issue in effect on the date of the purchase order. In the event of conflict between the text of this specification and references cited herein, the text of this specification takes precedence. Nothing in this specification, however, supersedes applicable laws and regulations unless a specific exemption has been obtained.

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2.1.1 SAE Publications: Available from SAE, 400 Commonwealth Drive, Warrendale, PA 15096-0001.

AMS 5047	Steel, Sheet and Strip, 0.08-0.13C, Aluminum Killed, Deep Forming Grade
AMS 5567	Steel Tubing, Seamless or Welded, Corrosion Resistant, 19Cr - 10Ni (SAE 30304), Hydraulic, Annealed
AMS 5648	Steel Bars, Forgings, Tubing, and Rings, Corrosion and Heat Resistant, 17Cr - 12Ni - 2.5Mo (SAE 30316), Solution Heat Treated
AMS 5680	Steel Welding Wire, Corrosion and Heat Resistant, 18.5Cr - 11Ni - 0.40(Cb + Ta) (SAE 30347)
AMS 6440	Steel Bars, Forgings, and Tubing, 1.45Cr (0.98 - 1.10C), (SAE 52100), for Bearing Applications

2.1.1.1 Alloys Under the Unified Numbering System:

AISI 316	UNS S31600
AISI 304	UNS S30400
AISI 1010	UNS G10100

2.1.2 ASTM Publications: Available from ASTM, 1916 Race Street, Philadelphia, PA 19103-1187.

ASTM D 445	Kinematic Viscosity of Transparent and Opaque Liquids (and the Calculation of Dynamic Viscosity)
ASTM D 664	Neutralization Number by Potentiometric Titration
ASTM D 1748	Rust Protection by Metal Preservatives in the Humidity Cabinet

3. TECHNICAL REQUIREMENTS:

3.1 METHOD 1 - Saturation Determination for Chlorotrifluoroethylene (CTFE) - Tests for Oxidizable Materials:

3.1.1 Apparatus and Equipment:

3.1.1.1 Spectrophotometer Settings (UV Quartz Cuvettes Path Length 10 mm):

- Slit: 1.0 nm
- Absorbance scale for acetone (blank): 0 to 2.0
- Absorbance scale for samples and standard: 0 to 1.0
- Scan rate: 50 nm/min
- Wavelength scanned: 650 to 425 nm

3.1.1.2 Recorder Settings:

- 5 cm/min
- Full scale 100 mv

3.1.1.3 Constant temperature bath set at $25\text{ }^{\circ}\text{C} \pm 1\text{ }^{\circ}\text{C}$.

3.1.1.4 Balance capable of weighing to $\pm 0.0001\text{ g}$.

3.1.2 Materials:

- a. 10 mL pipet
- b. Test tubes: 25 mm x 200 mm, Pyrex
- c. Hamilton micro-syringe, type 710N or equivalent
- d. Small flask
- e. Reagent grade acetone, certified ACS (spectroanalyzed)
- f. Double distilled water
- g. Potassium permanganate, crystals, reagent grade, ACS
- h. Standard CTFE test fluid (see 4.3)

3.1.3 Equipment Operation: Shall be as follows:

3.1.3.1 Turn on spectrophotometer and constant temperature bath and allow to warm up.

3.1.3.2 Rinse test tubes with distilled water, follow with a rinse using reagent grade acetone, and dry in an oven. Clean micro-syringe with acetone and dry under nitrogen.

3.1.3.3 Prepare a 1% solution of potassium permanganate with double distilled water and cover with paraffin film or stopper.

3.1.3.4 Clean four test tubes in accordance with 3.1.3.2. Add $1.0\text{ g} \pm 0.05\text{ g}$ standard test fluid to two test tubes. Add $1.0\text{ g} \pm 0.05\text{ g}$ sample hydraulic fluid to be tested to the two remaining test tubes and cover with aluminum foil.

3.1.3.5 Prepare a blank (10 mL acetone in test tube).

3.1.3.6 Using the micro-syringe, add 0.06 mL of 1% potassium permanganate to the blank (3.1.3.5), shake and swirl, record time and quickly transfer test tube to constant temperature bath set at $25\text{ }^{\circ}\text{C} \pm 1\text{ }^{\circ}\text{C}$ ($77\text{ }^{\circ}\text{F} \pm 2\text{ }^{\circ}\text{F}$).

3.1.3.7 Maintain blank in bath for 15 min minimum; add 10 mL acetone to standard, shake, and swirl; add 0.06 mL of 1% potassium permanganate solution, swirl, record time, and quickly place standard in bath.

3.1.3.8 After 60 min minimum, remove the first sample (blank), observe color, quickly transfer into a clean cuvette and place in the spectrophotometer. Check settings. Start the spectrophotometer and zero the recorder. Scan 650 to 425 nm, making certain that the blank run is set on absorbance scale (0 to 2.0). Measure and record absorbance at 528 nm. Stop at 425 nm, remove sample, clean cuvette, and prepare for next run. Set absorbance scale (0 to 1.0).

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3.1.3.9 Remove the standard from water bath, observe color, quickly transfer to cuvette, and proceed as above (3.1.3.8). Record absorbance at 528 nm. Test other samples in the same manner.

3.1.3.10 Compare the duplicate standard's absorbance. Difference should not be greater than 0.04 absorbance units. If difference is greater than 0.04, repeat the test.

3.1.4 Acceptance Standard:

3.1.4.1 If the absorbance of the sample being tested is equal to or greater than the absorbance of the standard CTFE sample, the test fluid is acceptable.

3.1.4.2 If the absorbance of the sample being tested is lower than the standard CTFE sample, the sample is rejected.

3.2 METHOD 2 - Capillary Gas Chromatography Method (CTFE Fluid Analysis):

A candidate CTFE fluid and a standard CTFE trimer fluid are analyzed by a gas chromatographic system. The concentration of the peaks is calculated by taking the percent of the total area contributed by each peak. The total percent of the candidate sample peaks eluting earlier than the standard trimer are reported.

3.2.1 Apparatus and Equipment:

- a. Hewlett Packard gas chromatograph, Model 5710 or equivalent, adapted for use with capillary columns with a flame ionization detector or equivalent
- b. Fused silica capillary column, length 12 m, diameter 0.22 mm, methyl silicone, carbowax 20M deactivated stationary phase or equivalent
- c. Chlorotrifluoroethylene trimer standard (see 4.2)
- d. Syringe, 5 mL capacity
- e. Linear recorder, 0 to 1 mV
- f. Sample vials
- g. Hewlett Packard Autosampler model 7671A or equivalent (optional)
- h. Hewlett Packard 3354 lab automation system or equivalent for peak integration and calculation

3.2.2 Equipment Operation: Shall be as follows:

- a. Equipment designation: See 3.2.1
- b. Support: None
- c. Split ratio: 100 to 1
- d. Auxiliary gas: Helium, 40 mL/min
- e. Carrier gas: Helium
- f. Carrier gas flow rate: 1 mL/min
- g. Chart speed: 0.66 cm/min
- h. Detector: FID
- i. Attenuation: 10 x 16

3.2.2 (Continued):

j. Temperature

- (1) Injector: 250 °C (480 °F)
- (2) Detector: 250 °C (480 °F)
- (3) Column: 100 to 250 °C (212 to 480 °F)

k. Temperature program rate: 8 °C (15 °F) minimum
Initial hold - 8 min
Final hold - 8 min

3.2.3 Calculation of CTFE base fluid areas.

3.2.3.1 Sum all peak areas.

3.2.3.2 Express each peak as a percent of the total area.

3.2.3.3 Assume that the area percent is the same as the true composition.

3.2.3.4 Calculation results using Equation 1:

$$C_i = \frac{A_i \times 100}{n} \quad (\text{Eq. 1})$$

where:

C_i = Concentration of component i
 A_i = Area of component i in units of microvolt-seconds
 n = Total area for entire run, microvolt-seconds

3.2.3.4.1 Assumptions used in this calculation are:

The detector responds quantitatively in the same way to all sample components; all response factors are the same.

All components are eluted from the column.

All components are detected and are represented as peaks.

NOTE: The Hewlett Packard 3354 Automation System carries out this integration.

3.2.4 Acceptance Standard:

3.2.4.1 New CTFE samples with less than 11% of the total concentration eluting before the trimer standard as measured (retention time 2.35 min for the above system) are acceptable.

3.2.4.2 Representative chromatograms are shown in Figures 1 and 2.

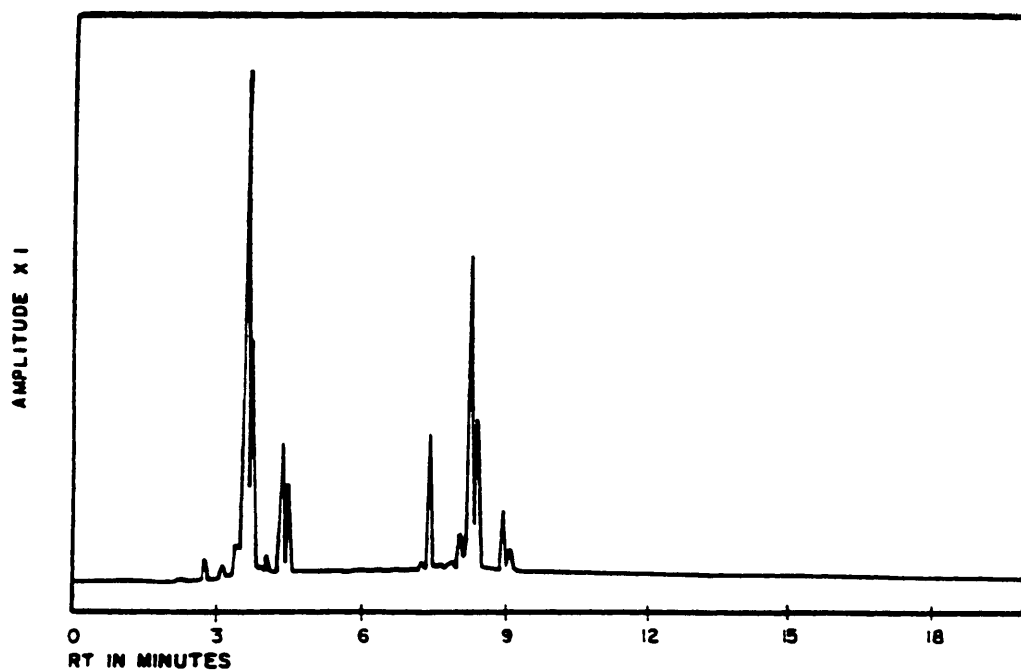


FIGURE 1 - Capillary GC of CTFE Base Fluid (Amplitude x 1)

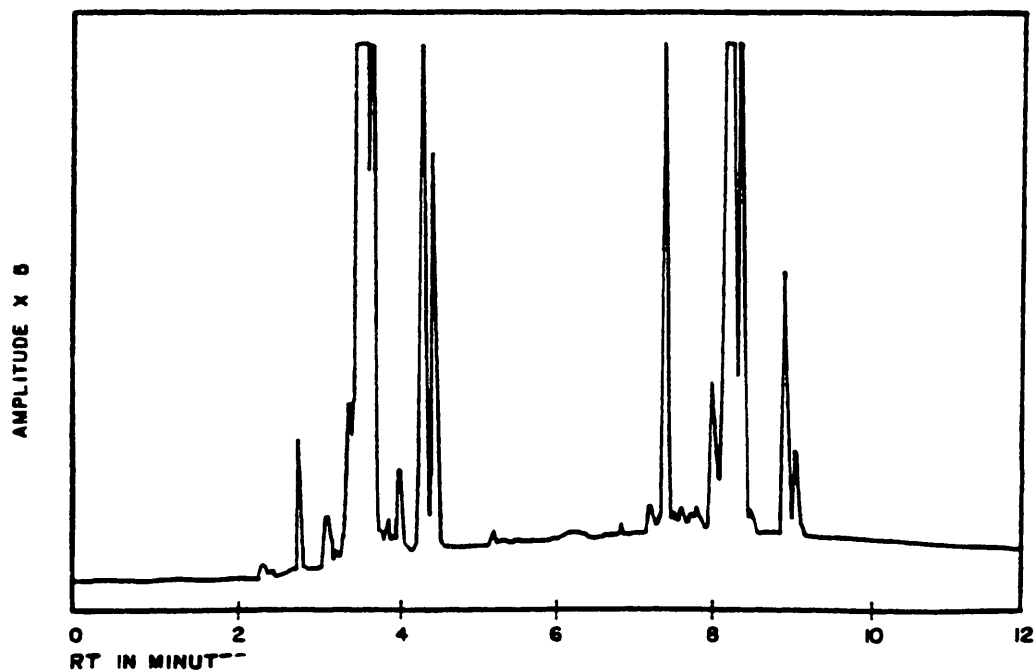


FIGURE 2 - Capillary GC of CTFE Base Fluid (Amplitude x 5)