

INCH-POUND

NOTICE OF  
CHANGEMIL-STD-1844  
NOTICE 1  
15 January 1991MILITARY STANDARD  
GAS CHROMATOGRAPHY METHOD FOR DETERMINATION OF  
TRACE CHLORINATED SOLVENTS IN HYDRAULIC FLUID

TO ALL HOLDERS OF MIL-STD-1844

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PAGES LISTED

| NEW PAGE | DATED           | SUPERSEDED PAGE | DATE                     |
|----------|-----------------|-----------------|--------------------------|
| 1        | 15 January 1991 | 1               | 30 March 1984            |
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2. RETAIN THIS NOTICE AND INSERT BEFORE TABLE OF CONTENTS

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INCH-POUND  
MIL-STD- 1844  
30 March 1984

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GAS CHROMATOGRAPHY METHOD FOR DETERMINATION OF  
TRACE CHLORINATED SOLVENTS IN HYDRAULIC FLUID



AMSC N/A

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MIL-STD-1844

FOREWARD

- 1 This Military Standard is approved for use by all Air Force activities
- 2 Recommended corrections, additions or deletions should be addressed to Directorate of Aerospace Fuels Management, SA-ALC/SFRT-1, Kelly AFB TX 78241-5000
- 3 In furtherance of the Defense Standardization Program, it is essential that a single, clear and concise procedure for determining the chlorine concentration in a petroleum base hydraulic fluid in the range of 0 to 500 ug/gm chlorine be made available
- 4 Initial research efforts proved that analysis for trace amounts of chlorine by several analytical procedures such as gas chromatography and microcoulometry produced uniquely dissimilar values which could not be used together in correlation studies
- 5 A decision was made to select electron capture gas chromatography as the optimum choice of methodology

Supersedes page 11 and 111 of 30 Mar 1984

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## 1. SCOPE

1.1 Principal Statement. This military standard describes a procedure for determining the chlorine concentration in a petroleum base hydraulic fluid in the range of 0 to 500  $\mu\text{g/gm}$  chlorine

## 2. REFERENCE DOCUMENT

MIL-H-5606

## 3. DEFINITIONS

$\mu\text{g}$  - Abbreviation for microgram, a measure of weight 1000  $\mu\text{g}$  = 1 mg  
 $\mu\text{l}$  - Abbreviation for microliter, a measure of volume 1000  $\mu\text{l}$  = 1 ml  
 $\mu\text{g/gm}$  - Abbreviation for microgram per gram, a measure of concentration  
gm - Abbreviation for gram, a measure of weight. 1 gm = 1000 mg  
mg - Abbreviation for milligram, a measure of weight. 1000 mg = 1 gm  
ml - Abbreviation for milliliter, a measure of volume 1 ml = 1000  $\mu\text{l}$   
baseline resolution - In gas chromatography, the condition where the separation between components is sufficient to cause the peak trace for one component to return to the baseline before the peak trace for the next component rises from the baseline  
carcinogenic - The tendency to produce cancer  
concentration - The amount of a given substance contained in a unit quantity of sample  
elute - In gas chromatography, to remove the components from the absorbent (packed column)  
elution order - The order in which components elute from a column  
internal standard - A material added to a sample in a known amount to serve as a reference for calculation  
linear operating range - The range of peak areas or heights where a plot of concentration versus peak area or height results in a straight line i.e. detector response is directly proportional to concentration  
relative response factor - Used to compensate for differences in detector response to different components. The concentration of the component divided by its peak area or height relative to the peak area or height of an internal standard and its concentration.

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## 4 GAS CHROMATOGRAPHY METHOD FOR DETERMINATION OF TRACE CHLORINATED SOLVENTS IN HYDRAULIC FLUID

4.1 Summary of Method A known amount of internal standard is added to a known amount of sample. The mixture is diluted, then injected into a gas chromatograph equipped with an electron capture detector. The concentrations of chlorinated solvents are determined by using response factors previously calculated from calibration standards and an internal standard method.

4.2 Gas Chromatograph Any gas chromatograph, commercially available or custom designed, may be used provided the system has an oven that may be temperature programmed and sufficient sensitivity to detect trace amounts of chlorinated solvents when operated in accordance with the manufacturer's instructions. (One commercially available unit is the Varian Gas Chromatograph, National Stock Number (NSN) 6630-01-268-1671. This unit or any other equivalent unit may be used.)

4.2.1 Operating Conditions

|                          |   |
|--------------------------|---|
| Oven temperature program | - 80°C for 5 min, then 25°C/min to 300°C and hold for 15 minutes  |
| Injector temperature     | - 300°C   |
| Detector temperature     | - 350°C   |
| Detector                 | - electron capture  |
| Carrier gas              | - nitrogen or 95% argon/5% methane at 30 ml/minute  |
| Column                   | - twelve feet of 1/8 inch O.D., stainless steel tubing packed with 10% SP2100 on 80/100 Supelcoport, or any equivalent packed column that provides base-line resolution for the chlorinated solvents. A capillary (WCOT) column is also acceptable, provided the analyst is experienced in its use and can achieve baseline resolution of the chlorinated solvents. |
| Sample size              | - 0.2 - 0.5 microliters   |

4.2.2 Data Collection A recording potentiometer with full scale deflection of 100 mV or less may be used. Or an electronic integrator that measures peak retention times and either peak heights or peak areas (preferable) is also acceptable. Or a data system that incorporates peak measurements and data reduction may also be used.

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