

MIL-STD- 1844
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MILITARY STANDARD
GAS CHROMATOGRAPHY METHOD FOR DETERMINATION OF
TRACE CHLORINATED SOLVENTS IN HYDRAULIC FLUID



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MIL-STD-1844

DEPARTMENT OF DEFENSE
Washington, DC 20360

Gas Chromatography Method for Determination of Trace Chlorinated
Solvents in Hydraulic Fluids.

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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 Energy Management, San Antonio
Air Logistics Center/SF, Kelly AFB TX 78241. ATTN: SFRM

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FOREWORD

1. 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.
2. Recent research efforts proved that analysis for trace amounts of chlorine by gas chromatography, X-ray diffraction and micro-coulometry produced uniquely dissimilar values which could not be used together in correlation studies.
3. A decision was made to select electron capture gas chromatography as the optimum choice of methodology.

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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

- μg - Abbreviation for microgram, a measure of weight 1000 μg = 1 mg
- μl - Abbreviation for microliter, a measure of volume. 1000 μl = 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 μl
- 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 and under the conditions described below.

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 baseline 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 10.0 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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4.3 Material and Supplies.

Balance	- capable of accurately weighing to nearest 0.1 milligram
Bottles (or vials)	- approx. 10 ml with tight-fitting, screw-cap closures that form a good seal
Vials	- approx. 2 ml with tight-fitting, screw-cap closures (auto-sampler vials with screw-cap lids and replaceable septa are excellent)
Syringes	- gas-tight, 100 microliters and 250 microliters or a positive displacement pipettor constructed of glass and teflon capable of accurately delivering 50 and 100 microliters
Volumetric flasks	- 10 ml, 100 ml
Chloroform	- reagent grade or better
1,1,2-trichlorotrifluoroethane (Freon 113)	- reagent grade or better
1,1,1-trichloroethane	- reagent grade or better
Tetrachloroethylene	- spectro grade or better
Hydraulic fluid, MIL-H-5606	- free of chlorine containing materials
Hexane	- distilled in glass, non-spectro

4.4 Preparation, Calibration, and Standardization.

WARNING: Chloroform is a suspected carcinogen. Appropriate safety measures should be followed to minimize exposure including wearing gloves and using it only in a hood. While the other solvents have not yet been found to be carcinogenic, they should be handled in the same manner. Prepare all standards and samples in a hood.

4.4.1 Preparation of operating range standard stock and working solutions.

(a) Stock solution. Accurately weigh approximately 200 milligrams (mg) of each chlorinated solvent (1,1,2-trichlorotrifluoroethane, 1,1,1-trichloroethane, and tetrachloroethylene) into a 10 milliliter (ml) volumetric flask. Dilute to volume with hexane, stopper, and mix well. Calculate the concentration of each chlorinated solvent using the following equation:

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$$C = \frac{W}{V}$$

where: W = weight of chlorinated solvent in mg

V = volume of volumetric flask in ml

C = concentration of chlorinated solvent in mg/ml

An alternative method may be used to prepare the standard. Using a clean, 250 μ l gas-tight syringe, accurately measure approximately 150 microliters (μ l) of each chlorinated solvent (1,1,2-trichlorotrifluoroethane, 1,1,1-trichloroethane, and tetrachloroethylene) into a 10 ml volumetric flask. Dilute to volume with hexane, stopper, and mix well. Calculate the concentration of each chlorinated solvent using the following equation:

$$C = \frac{D \times A}{V}$$

where: D = density of chlorinated solvent at 20°C in mg/ μ l (see Table 1)

A = amount of chlorinated solvent in μ l

V = volume of volumetric flask in ml

C = concentration of chlorinated solvent in mg/ml

Transfer the standard to a 10 ml screw-cap bottle or vial, cap tightly, and store in a refrigerator or freezer to minimize loss of solvents due to evaporation.

(b) Working solutions.

(1) Using a clean, gas-tight 250 μ l syringe, accurately measure approximately 200 μ l of the operating range stock solution prepared in 4.4.1(a) into a 10 ml volumetric flask. Dilute to volume with hexane, stopper and mix well.

(2) Using a clean, gas-tight 250 μ l syringe, accurately measure approximately 100 μ l of the operating range stock solution into a 10 ml volumetric flask. Dilute to volume with hexane, stopper, and mix well.

(3) Using a clean, gas-tight 100 μ l syringe, accurately measure approximately 50 μ l of the operating range stock solution into a 10 ml volumetric flask. Dilute to volume with hexane, stopper, and mix well.

(4) Using a clean, gas-tight 100 μ l syringe, accurately measure approximately 35 μ l of the operating range stock solution into a 10 ml volumetric flask. Dilute to volume with hexane, stopper, and mix well.

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(5) Using a clean, gas-tight 250 μ l syringe, accurately measure approximately 200 μ l of the operating range stock solution into a 100 ml volumetric flask. Dilute to volume with hexane, stopper, and mix well.

(6) Using a clean, gas-tight 250 μ l syringe, accurately measure approximately 100 μ l of the operating range stock solution into a 100 ml volumetric flask. Dilute to volume with hexane, stopper, and mix well.

(7) Using a clean, gas-tight 100 μ l syringe, accurately measure approximately 50 μ l of the operating range stock solution into a 100 ml volumetric flask. Dilute to volume with hexane, stopper, and mix well.

Calculate the concentration of each chlorinated solvent for each working solution using the following equation:

$$U = \frac{C \times Y \times \frac{1 \text{ ml}}{1000 \mu\text{l}}}{Z} = \frac{C \times Y}{Z \times 1000}$$

where: C = concentration of chlorinated solvent in mg/ml of the operating range stock solution prepared in 4.4.1(a)

Y = amount of operating range stock solution in μ l

Z = volume of volumetric flask in ml

U = concentration of chlorinated solvent in mg/ml

Transfer the standards to 10 ml screw-cap bottles or vials, cap tightly, and store in a refrigerator or freezer when not in use to minimize loss of solvents due to evaporation.

4.4.2 Preparation of internal standard stock and working solutions.

(a) Stock solution. Accurately weigh approximately 200 mg of chloroform into a 10 ml volumetric flask. Dilute to volume with hexane, stopper, and mix well. Calculate the concentration of chloroform using the following equation:

$$B = \frac{E}{F}$$

where: E = weight of chloroform in mg

F = volume of volumetric flask in ml

B = concentration of chloroform in mg/ml

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An alternative method may be used to prepare the standard. Using a clean, gas-tight 250 μl syringe, accurately measure approximately 150 μl of chloroform into a 10 ml volumetric flask. Dilute to volume with hexane, stopper, and mix well. Calculate the concentration of chloroform using the following equation:

$$B = \frac{G \times H}{F}$$

where: G = density of chloroform at 20°C in mg/ μl (see Table 1)

H = amount of chloroform in μl

F = volume of volumetric flask in ml

B = concentration of chloroform in mg/ml

Transfer the standard to a 10 ml screw-cap bottle or vial, cap tightly, and store in a refrigerator or freezer when not in use to minimize loss of chloroform due to evaporation.

(b) Working solution. Using a clean, gas-tight 250 μl syringe, accurately measure approximately 100 μl of the internal standard stock solution prepared in 4.4.2(a) into a 10 ml volumetric flask. Dilute to volume with hexane, stopper, and mix well. Calculate the concentration of chloroform using the following equation:

$$I = \frac{B \times \frac{1000 \mu\text{g}}{1 \text{ mg}} \times \frac{1 \text{ ml}}{1000 \mu\text{l}} \times J}{K \times \frac{1000 \mu\text{l}}{1 \text{ ml}}} = \frac{B \times J}{K \times 1000}$$

where: B = concentration of chloroform in mg/ml in the internal standard stock solution prepared in 4.4.2(a)

J = amount of internal standard stock solution in μl

K = volume of volumetric flask in ml

I = concentration of chloroform in $\mu\text{g}/\mu\text{l}$

Transfer the standard to a 10 ml screw-cap bottle or vial, cap tightly, and store in a refrigerator or freezer when not in use to minimize loss of chloroform due to evaporation.

4.4.3 Preparation of calibration standard stock and working solutions.

(a) Stock solution. Accurately weigh approximately 100 mg of each chlorinated solvent (1,1,2-trichlorotrifluoroethane, 1,1,1-trichloroethane, and tetrachloroethylene)

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and chloroform into a 10 ml volumetric flask. Dilute to volume with hexane, stopper, and mix well. Calculate the concentration of each chlorinated solvent and chloroform using the following equation:

$$L = \frac{M}{N}$$

where: M = weight of chlorinated solvent or chloroform in mg

N = volume of volumetric flask in ml

L = concentration of chlorinated solvent or chloroform in mg/ml

An alternative method may be used to prepare the standard. Using a clean, gas-tight 100 μ l syringe, accurately measure approximately 75 μ l of each chlorinated solvent (1,1,2-trichlorotrifluoroethane, 1,1,1-trichloroethane, and tetrachloroethylene) and chloroform into a 10 ml volumetric flask. Dilute to volume with hexane, stopper, and mix well. Calculate the concentration of each chlorinated solvent and chloroform using the following equation:

$$L = \frac{O \times P}{N}$$

where: O = density of chlorinated solvent or chloroform at 20°C in mg/ μ l (see Table 1)

P = amount of chlorinated solvent or chloroform in μ l

N = volume of volumetric flask in ml

L = concentration of chlorinated solvent or chloroform in mg/ml

Transfer the standard to a 10 ml screw-cap bottle or vial, cap tightly, and store in a refrigerator or freezer when not in use to minimize loss of solvents or chloroform due to evaporation.

(b) Working solution. Using a clean, gas-tight 250 μ l syringe, accurately measure approximately 100 μ l of the calibration standard stock solution prepared in 4.4.3(a) into a 100 ml volumetric flask. Dilute to volume with hexane, stopper, and mix well. Calculate the concentration of each chlorinated solvent and chloroform using the following equation:

$$B = \frac{L \times \frac{1000 \mu\text{g}}{1 \text{ mg}} \times \frac{1 \text{ ml}}{1000 \mu\text{l}} \times S}{T} = \frac{L \times S}{T}$$

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where: L = concentration of chlorinated solvent or chloroform in mg/ml
in the calibration standard stock solution prepared in 4.4.3(a)
S = amount of calibration standard stock solution in μ l
T = volume of volumetric flask in mls
Q = concentration of chlorinated solvent or chloroform in μ g/ml

Transfer the standard to several 10 ml screw-cap bottles or vials, cap tightly, and store in a refrigerator or freezer when not in use to minimize loss of solvents or chloroform due to evaporation.

4.4.4 Standardization of gas chromatograph.

(a) Preparation of gas chromatograph. Set up the gas chromatograph in accordance with the manufacturer's operating instructions and the operating conditions outlined in 4.1.1 and 4.2.2.

(b) Analysis of working solutions. If the operating range working solutions prepared in 4.4.1(b) have been stored in a refrigerator or freezer, bring them up to room temperature (approx. 20°C) and mix well before proceeding. Inject 0.5 μ l of the first operating range working solution prepared in 4.4.1(b) into the gas chromatograph and run the oven temperature program specified in 4.2.1. Record the retention time and peak height or peak area for each chlorinated solvent. If the recommended column and operating conditions are used, the elution order for the chlorinated solvents is 1,1,2-trichlorotrifluoroethane, chloroform, 1,1,1-trichloroethane, and tetrachloroethylene (see Figure 1). Remember, there is no chloroform in the operating range working solutions. Repeat step (b) for the rest of the operating range working solutions.

(c) Calculations. For each chlorinated solvent, plot its concentration (U) as determined in 4.4.1(b) versus its peak height or peak area as determined in 4.4.4(b) for all of the solutions (see Figure 2). Note the peak height or area where the curve is no longer linear. In Figure 2, this occurs at approximately 150, 200, and 200 peak area units respectively for 1,1,2-trichlorotrifluoroethane, 1,1,1-trichloroethane, and tetrachloroethylene. This point is the limit of the linear operating range for each solvent. It will vary from gas chromatograph to gas chromatograph, and

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can be affected by changing columns or cleaning the detector. Whenever a new gas chromatograph is used or columns changed or detectors cleaned or the gas chromatographic system changed in any other significant way, the limit of linear operating range for each chlorinated solvent should be determined again.

4.4.5 Analysis of calibration standard. This step should be done each time a set of samples is analyzed.

(a) Preparation of the gas chromatograph. Set up the gas chromatograph in accordance with the manufacturer's operating instructions and the operating conditions outlined in 4.2.1 and 4.2.2.

(b) Analysis of working solution. If the calibration standard working solution prepared in 4.4.3(b) has been stored in a refrigerator or freezer, bring it up to room temperature (approx. 20°C) and mix well before proceeding. Inject 0.2 - 0.5 µl of the calibration standard working solution into the gas chromatograph and run the oven temperature program specified in 4.2.1. Record the retention time and peak height or peak area for each chlorinated solvent and chloroform. If the recommended column and operating conditions are used, the elution order is 1,1,2- trichlorotrifluoroethane, chloroform, 1,1,1-trichloroethane, and tetrachloroethylene (see Figure 1).

(c) Calculations. Calculate the relative response factor for each chlorinated solvent using the equation:

$$f_1 = \frac{Q_1}{Q_S} \times \frac{A_S}{A_1}$$

where: Q_1 = concentration of chlorinated solvent in µg/ml in the calibration standard working solution prepared in 4.4.3(b)

Q_S = concentration of chloroform in µg/ml in the calibration standard working solution

A_1 = peak area or height of chlorinated solvent as determined in 4.4.5(b)

A_S = peak area or height of chloroform as determined in 4.4.5(b)

4.5 Analysis of Hydraulic Fluid Samples.

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4.5.1 Sample preparation. If the internal standard working solution prepared in 4.4.2(b) has been stored in a refrigerator or freezer, bring it up to room temperature (approx. 20°C) and mix well before proceeding. Shake the hydraulic fluid sample vigorously for approximately 30 seconds. Fill a 2 ml, screw-cap vial approximately half full with hexane. Using a clean, gas-tight 100 µl syringe or a positive displacement pipettor, accurately measure 50 µl of the internal standard working solution into the 2 ml screw-cap vial. Using a clean, gas-tight 250 µl syringe or a positive displacement pipettor, accurately measure 100 µl of the hydraulic fluid sample into the same 2 ml screw-cap vial. Fill the vial with hexane, cap tightly, and mix well. Repeat step 4.5.1 for each sample.

4.5.2 Blank preparation. Repeat step 4.5.1, using chlorine-free hydraulic fluid as the sample.

4.5.3 Analysis of samples. The calibration standard working solution should have already been analyzed as described in 4.4.5. Inject 0.2 - 0.5 µl of the sample prepared in 4.5.1 into the gas chromatograph and run the oven temperature program specified in 4.2.1. Record the retention time and peak height or peak area for each chlorinated solvent and chloroform. If the recommended column and operating conditions are used, the elution order is 1,1,2-trichlorotrifluoroethane, chloroform, 1,1,1 trichloroethane, and tetrachloroethylene (see Figure 1). If the peak area or height for any solvent exceeds the limit of linear operating range as determined in 4.4.4(c), dilute the hydraulic fluid sample by accurately measuring 1 ml of sample into a 10 ml volumetric flask and diluting to volume with hexane. Stopper, mix well, and repeat step 4.5.1. Remember to multiply results by 10. Repeat step 4.5.3 for each sample and the blank.

4.5.4 Calculations. Calculate the concentration of each chlorinated solvent in the sample using the following equation:

$$C_1 = \frac{f_1 \times A_1}{A_S} \times \frac{I \times 50 \mu\text{l}}{W_1}$$

where: f_1 = relative response factor for chlorinated solvent as
calculated in 4.4.5(c)

A_1 = peak area or height of chlorinated solvent peak from 4.5.3

A_S = peak area or height of chloroform peak from 4.5.3

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I = concentration of chloroform in $\mu\text{g}/\mu\text{l}$ in the internal standard working solution prepared in 4.4.2(b)

W_i = weight in gm of hydraulic fluid sample analyzed
(0.085 gm for 100 μl)

C_i = concentration of chlorinated solvent in $\mu\text{g}/\text{gm}$ in the hydraulic fluid sample

Repeat the calculation for each sample and the blank. If any chlorinated solvents are found in the blank, subtract the concentration of each chlorinated solvent in the blank from its concentration in each sample. Chlorinated solvents in the blank indicate contamination. Determine the source of contamination and eliminate it.

4.5.5 Final calculations. To calculate the concentration of chlorine in the hydraulic fluid sample, use the following equations:

$$K_i = \frac{C_i \times P_i}{100} \quad \text{and}$$

$$X = \sum K_i$$

where: C_i = concentration of chlorinated solvent in $\mu\text{g}/\text{gm}$ as determined in 4.5.4

P_i = % chlorine in chlorinated solvent (Table 1)

K_i = concentration of chlorine in $\mu\text{g}/\text{gm}$ as that chlorinated solvent in the sample

X = total concentration of chlorine in $\mu\text{g}/\text{gm}$ in the sample

Round results to the nearest $\mu\text{g}/\text{gm}$. Refer to Figure 3 for an example of calculations for a hypothetical sample.

Note: Most integrators can do internal standard calculations and it is possible to program most data systems normally provided with today's gas chromatographs to do all of these calculations. It is strongly recommended that analysts take advantage of these features.

4.6 Precision. Duplicate results by the same operator should be suspect if the value differs by greater than 5 percent.

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5. GENERAL INFORMATION

5.1 Isothermal Operation of Gas Chromatograph. If the capability to temperature program the gas chromatograph oven does not exist, the analyses may be run isothermally at 80°C. However, this will dramatically increase the rate at which the gas chromatography column deteriorates. In order to slow this down as much as possible, after each set of samples is run, raise the oven temperature to 300°C and hold it there for 30 minutes to elute the higher-boiling components of hydraulic fluid.

5.2 Loss of Resolution. As the gas chromatography column deteriorates (with either temperature-programmed or isothermal operation) it will no longer be possible to achieve baseline resolution of the chlorinated solvents. It may be possible to improve the resolution by lowering the initial oven temperature. If this does not help, it is time to replace the column.

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Table 1

	Density at 20°C (mg/μl)	% chlorine
1,1,2-trichlorotrifluoroethane	1.575	56.8
1,1,1-trichloroethane	1.338	79.8
tetrachloroethylene	1.623	85.5
chloroform	1.484	29.1

Figure 1

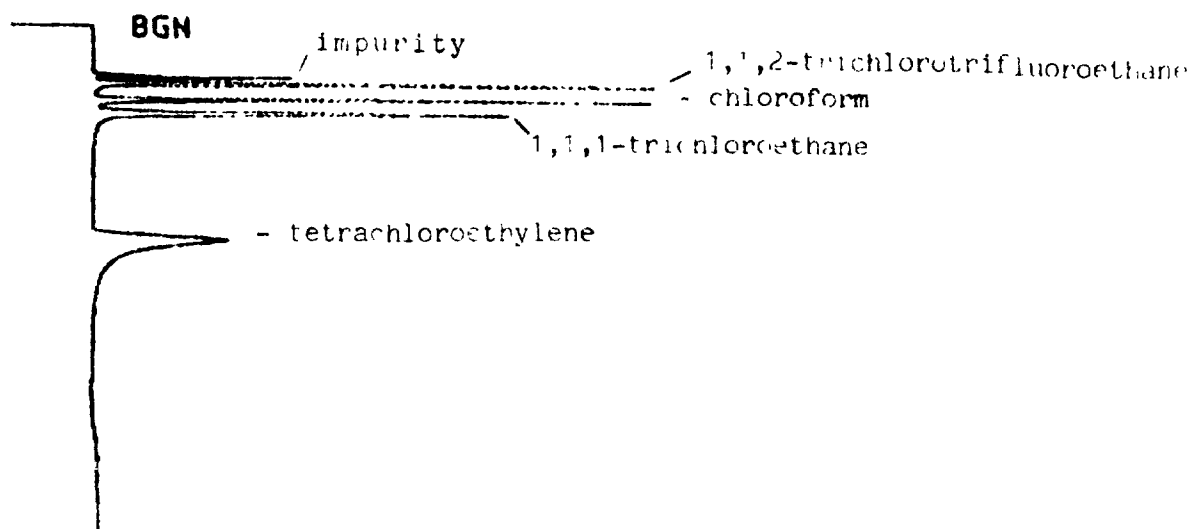
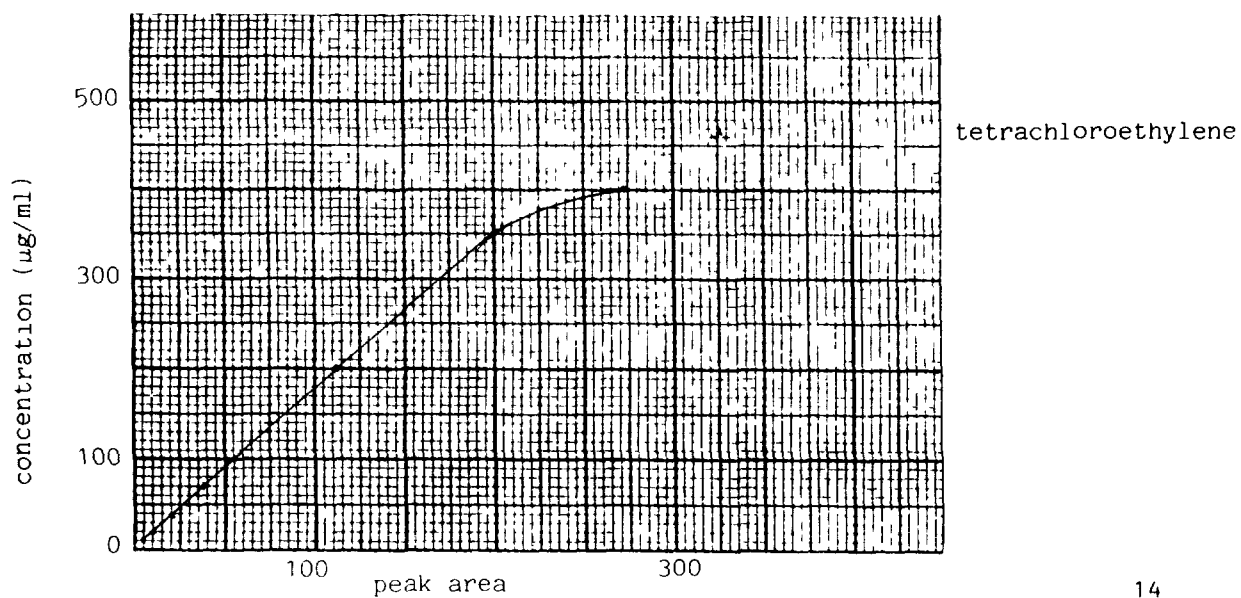
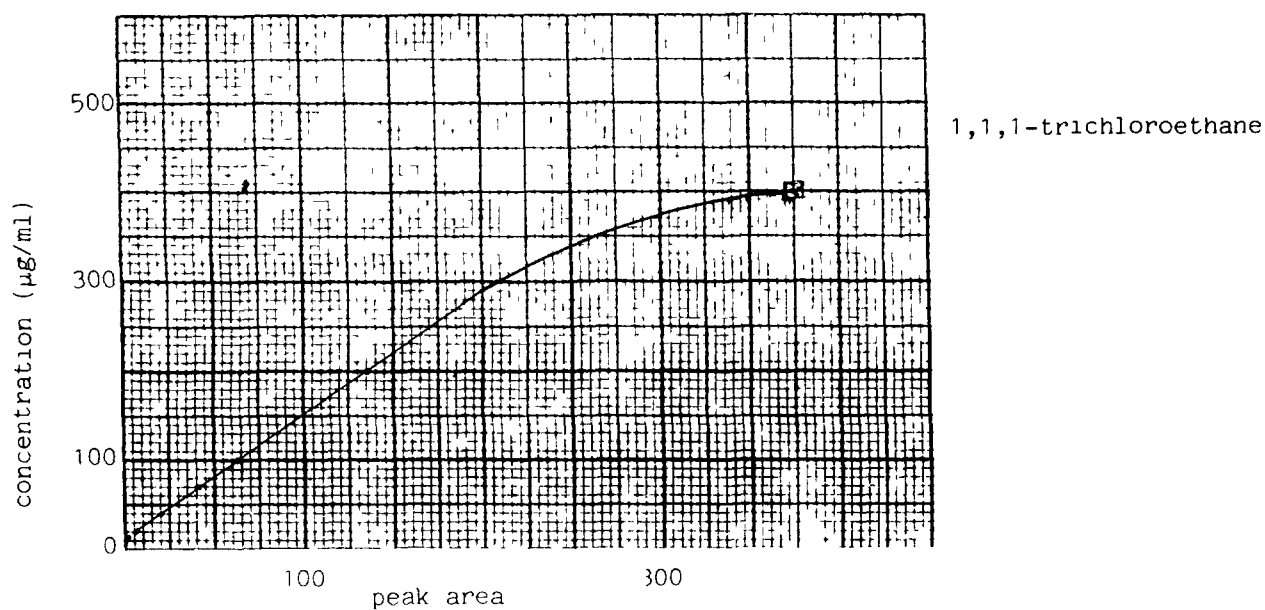
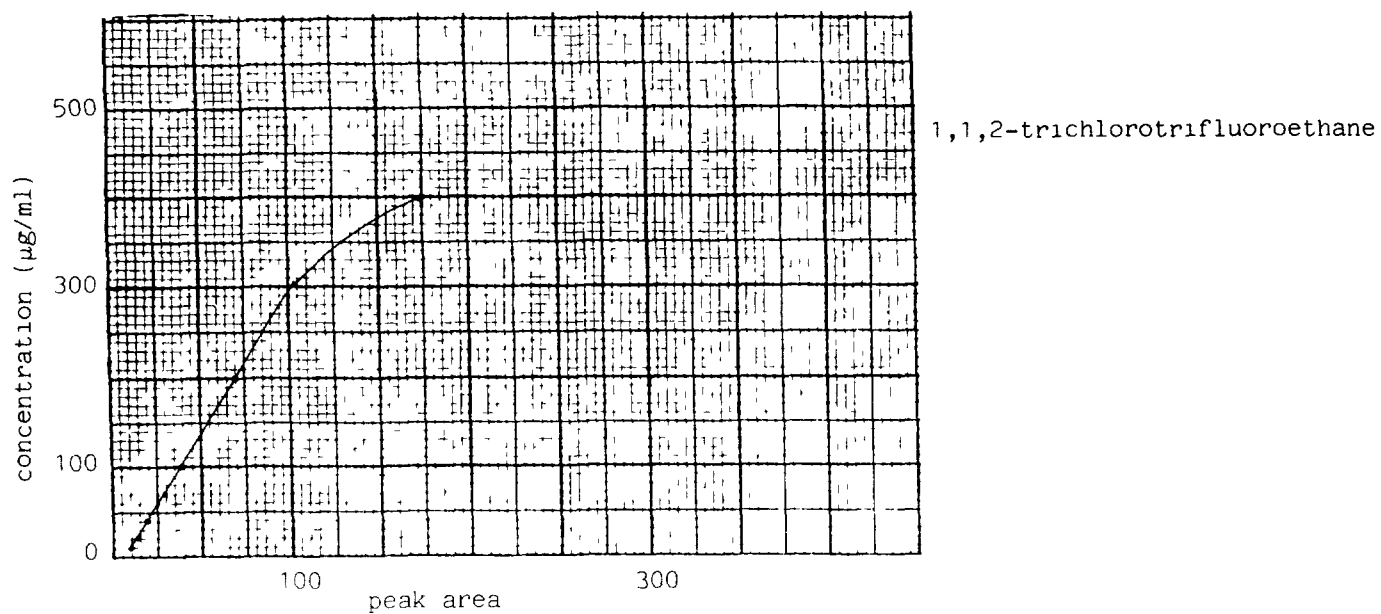


Figure 2



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Figure 3

Example Calculations for a Hypothetical Sample

Given the following:

<u>Calibration Standard Working Solution</u>	<u>Q</u> (μg/ml)	<u>A</u> (peak area)
1,1,2-trichlorotrifluoroethane	10	13
1,1,1-trichloroethane	10	15
tetrachloroethylene	10	26
chloroform	10	10
<u>Internal Standard Working Solution</u>	<u>I</u> (μg/μl)	
chloroform	0.2	
<u>Hypothetical Sample</u>	<u>A</u> (peak area)	
1,1,2-trichlorotrifluoroethane	15	
chloroform	10	
1,1,1-trichloroethane	10	
tetrachloroethylene	5	

Calculate the response factor for each chlorinated solvent.

$$\begin{aligned}
 f(1,1,2\text{-trichlorotrifluoroethane}) &= \frac{Q_1}{Q_S} \times \frac{A_S}{A_1} = \frac{10}{10} \times \frac{10}{13} = 0.77 \\
 f(1,1,1\text{-trichloroethane}) &= \frac{10}{10} \times \frac{10}{15} = 0.67 \\
 f(\text{tetrachloroethylene}) &= \frac{10}{10} \times \frac{10}{26} = 0.38
 \end{aligned}$$

Calculate the concentration of each chlorinated solvent in the hypothetical sample.

$$\begin{aligned}
 C(1,1,2\text{-trichlorotrifluoroethane}) &= \frac{f_1 A_1}{A_S} \times \frac{I \times 50}{W_1} = \frac{0.77 \times 15}{10} \times \frac{0.2 \times 50}{0.085} = 136 \mu\text{g/gm} \\
 C(1,1,1\text{-trichloroethane}) &= \frac{0.67 \times 10}{10} \times \frac{0.2 \times 50}{0.085} = 79 \mu\text{g/gm} \\
 C(\text{tetrachloroethylene}) &= \frac{0.38 \times 5}{10} \times \frac{0.2 \times 50}{0.085} = 22 \mu\text{g/gm}
 \end{aligned}$$

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Calculate the total concentration of chlorine in the sample.

$$K \text{ (1,1,2-trichlorotrifluoroethane)} = \frac{C_1 P_1}{100} = \frac{136 \times 56.8}{100} = 77 \text{ } \mu\text{g/gm}$$

$$K \text{ (1,1,1-trichloroethane)} = \frac{79 \times 79.8}{100} = 63 \text{ } \mu\text{g/gm}$$

$$K \text{ (tetrachloroethylene)} = \frac{22 \times 85.5}{100} = 19 \text{ } \mu\text{g/gm}$$

$$X = \sum K_1 = 77 + 63 + 19 = 159 \text{ } \mu\text{g/gm}$$

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