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ANALYSIS OF MIL-L-23699C SYNTHETIC LUBRICANT  
CONTAMINATION OF 3-GP-26MA H (U) DEFENCE RESEARCH  
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J A HILTZ ET AL MAY 87 DREA-TM-87/207

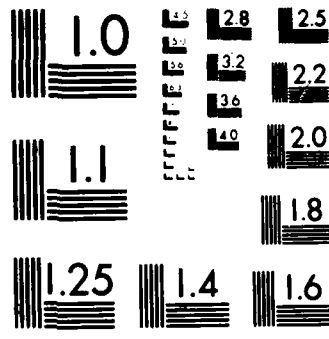
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TECHNICAL MEMORANDUM 87/207

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ANALYSIS OF MIL-L-23699C  
SYNTHETIC LUBRICANT CONTAMINATION  
OF 3-GP-26Ma HYDRAULIC FLUID BY GAS  
CHROMATOGRAPHY-MASS SPECTROMETRY

J.A. Hiltz - D.E. Veinot  
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ABSTRACT

A method is described for the determination of MIL-L-23699C synthetic lubricant contamination of 3-GP-26Ma hydraulic fluid. The method utilizes capillary gas chromatographic separation of the constituents of these fluids and quadrupole mass spectrometric detection of these constituents. The analysis takes 25 minutes and the lower limit of detection is 0.4 nanograms (ng) of MIL-L-23699C with a linear response range up to at least 4000 ng depending on the instrumental parameters used.

RÉSUMÉ

On décrit une méthode permettant de déterminer la contamination du fluide hydraulique 3-GP-26Ma par le lubrifiant synthétique MIL-L-23699C. Cette méthode utilise la chromatographie en phase gazeuse sur colonne capillaire pour séparer les constituants, et la spectrométrie de masse quadripolaire pour les détecter. L'analyse dure 25 minutes. Le seuil de détection du lubrifiant MIL-L-23699C est de 0,4 nanogramme (ng) et la réponse est linéaire jusqu'à au moins 4000 ng selon les paramètres instrumentaux utilisés.

TABLE OF CONTENTS

	<u>Page No.</u>
1.0 INTRODUCTION	1
2.0 EXPERIMENTAL	2
2.1 Equipment	2
2.2 Preparation of Standards	2
3.0 RESULTS AND DISCUSSION	3
3.1 Infrared Analysis of 3-GP-26Ma Hydraulic Fluid Contaminated with MIL-L-23699C Synthetic Lubricant	3
3.2 Gas Chromatographic Separation of MIL-L-23699C Synthetic Lubricant from 3-GP-26Ma Hydraulic Fluid	4
3.3 Detection of MIL-L-23699C Synthetic Lubricant	6
4.0 CONCLUSIONS	7
TABLES	8
FIGURES	11
REFERENCES	18

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### ABBREVIATIONS

amu	- atomic mass unit
°C	- degrees Celsius
CGSB	- Canadian General Standards Board
cm	- centimeter
cm <sup>-1</sup>	- reciprocal centimeter (1/cm)
GC	- gas chromatograph, gas chromatography
IR	- infrared
K <sub>D</sub>	- distribution coefficient
MID	- multiple ion detection
mm	- millimeter
MS	- mass spectrometer, mass spectrometry
m/z	- mass to charge ratio
ng	- nanogram (10 <sup>-9</sup> g)
r <sup>2</sup>	- correlation coefficient
μL	- microliter

## 1.0 INTRODUCTION

The analysis of lubricants and hydraulic fluids is an integral part of the service function provided to the Canadian Forces by DREA Dockyard Laboratory. Historically, this service function has involved the use of standard tests (1-8) to determine if the fluid meets the standard specified for the continued safe operation of a system.

The results derived from these standard tests are often augmented with information from chemical identification techniques, such as infrared (IR) (9) and ultraviolet (uv) spectroscopy, and gas chromatography (4) and high performance liquid chromatography. The information obtained from these techniques supplements that obtained from the physical tests and can be utilized to determine if the material meets specifications, or if it is contaminated with a fluid from another system. Results from chemical identification techniques are particularly important when the standard tests yield inconclusive or ambiguous results, and can become the basis for the recommendations regarding the operational status of the system in which the fluid is being utilized.

However, there are instances where the utilization of a particular technique does not afford the analyst the required analytical capabilities, i.e., the necessary selectivity or sensitivity, to perform an analysis. For instance, IR spectroscopy cannot easily differentiate between hydrocarbon based fluids containing a mixture of aromatic and aliphatic hydrocarbons or between fluids containing constituents with similar functional groups. This limitation in selectivity can be overcome through the use of a suitable separation technique. However, IR spectroscopy is also a relatively insensitive technique, especially for the detection of the presence of one compound in another.

A frequent inquiry at Dockyard Laboratory deals with the question of whether or not a petroleum-based hydraulic fluid (CGSB 3-GP-26Ma) is contaminated with a synthetic lubricant (MIL-L-23699C). This is of concern as the synthetic lubricant does not have the same compressibility factor or flow characteristics as the hydraulic fluid and could affect the operation of the system in which it is used. Further, there is concern that the synthetic lubricant may cause deterioration of the seals in the hydraulic system and its detection would prevent the possibility of a system failure.

To date infrared spectroscopy has been used in a limited manner to determine gross contamination of 3-GP-26Ma with MIL-L-23699C (levels above approximately 10%). However, a technique with the sensitivity to detect contamination at levels well below this is desirable. Capillary gas chromatography provides a technique that

can separate the components of complicated mixtures. Coupled with a quadrupole mass spectrometer, this hybrid technique is capable of separating, detecting and identifying the components of complex mixtures. Further, the quadrupole mass spectrometer has the sensitivity to detect the presence of most compounds at the part per million (ppm) level.

This paper describes the development of an analytical technique that allows the determination of MIL-L-23699C synthetic lubricating oil in 3-GP-26Ma hydraulic fluid. The technique involves the use of capillary gas chromatographic separation of the components of the hydraulic fluid-synthetic lubricant mixture with mass spectrometric detection of the components. Emphasis is placed on varying parameters that will decrease the time for an analysis. This is accomplished through changing the temperature program controlling the gas chromatographic oven and reducing the amount of time the components of the hydraulic fluid and the synthetic lubricant are retained on the column.

## 2.0 EXPERIMENTAL

### 2.1 Equipment

All infrared spectra were acquired on a Perkin Elmer Model 681 infrared spectrophotometer with a Perkin Elmer 3600 data station. The synthetic lubricant-hydraulic fluid mixtures were run as 0.025 mm films on sodium chloride plates.

All gas chromatographic separations were performed on a Finnigan model 9611 capillary gas chromatograph with a Durabond DB-1 30 meter long by 0.25 mm inside diameter capillary column (100 % methyl silicone). The GC oven is temperature programmable over the range from ambient to 425°C with ramping rates between 0.1° and 30°C per minute in 0.1°C increments. The capillary column was coupled to a Finnigan MAT model 5100 quadrupole mass spectrometer with a SuperIncos<sup>TM</sup> data system. The data system contains the National Bureau of Standards reference library of 38,791 mass spectra.

The mass spectrometer was used in the full scan mode, i.e., the ion fragments with masses between 50 and 500 atomic mass units (amu) were tuned through the quadrupole during each two second scan and in the multiple ion detection (MID) mode. The basis of operation of a quadrupole mass spectrometer and full and MID scanning modes are explained more fully in reference 10. Typical values of parameters to control the quadrupole mass spectrometer are shown in Table 1.

### 2.2 Preparation of Standards

Samples of 3-GP-26Ma hydraulic fluid and MIL-L-23699C synthetic

lubricant were obtained from the stores system at CFB Halifax. Standard solutions of MIL-L-23699C in 3-GP-26Ma (1, 10, 20, and 90 % volume/volume) for the IR study were prepared by adding measured volumes of the synthetic lubricant to volumetric flasks and making up to volume with 3-GP-26Ma.

Standard solutions of MIL-L-23699C in 3-GP-26Ma for the gas chromatograph-mass spectrometer analyses were prepared by the addition of known volumes of MIL-L-23699C to 3-GP-26Ma. Solutions of MIL-L-23699C in hexane for the detection limit study were prepared by the serial dilution of a 1/250 (volume/volume) solution of MIL-L-23699C in hexane. One microliter (1 $\mu$ L) injections of the various synthetic lubricant-hydraulic oil mixtures (as dilutions in hexane) were introduced onto the column through a 'modified Grob type' injector in the modified splitless mode. In the modified splitless mode the split gas is turned off until approximately 3 minutes after the injection has been made.

### 3.0 RESULTS AND DISCUSSION

#### 3.1 Infrared Analysis of 3-GP-26Ma Hydraulic Fluid Contaminated with MIL-L-23699C Synthetic Lubricant

Infrared spectra of 3-GP-26Ma hydraulic fluid and MIL-L-23699C are shown in Figures 1a and 1b respectively. Although the hydraulic fluid is composed primarily of a mixture of hydrocarbons as evidenced by the absorbances between 3000 and 2800  $\text{cm}^{-1}$ , 1400  $\text{cm}^{-1}$ , and 1380  $\text{cm}^{-1}$ , the absorbances at 1700  $\text{cm}^{-1}$  and between 1300 and 1000  $\text{cm}^{-1}$  are due to the presence of tricresyl phosphate, an anti-wear additive.

These absorbances make the detection of small quantities of MIL-L-23699C, a synthetic ester based lubricant, in hydraulic fluid impossible using the major absorbance frequencies since the esters that make up the synthetic lubricant absorb infrared radiation over the same frequency ranges as the additive in the hydraulic fluid. However, there are minor absorbances that are present in the IR spectrum of MIL-L-23699C that are not found in the 3-GP-26Ma spectrum. Two of these are found at 1110  $\text{cm}^{-1}$  and 1020  $\text{cm}^{-1}$  respectively (see Figure 1b) and might be used to determine if a sample of 3-GP-26Ma was contaminated with MIL-L-23699C synthetic lubricating oil.

Figures 2a through 2d show the infrared spectra of 3-GP-26Ma hydraulic fluid containing 1, 10, 20, and 90% (volume/volume) MIL-L-23699C synthetic lubricant. There is little difference between the IR spectra of 3-GP-26Ma (Figure 1a) and that of 3-GP-26Ma contaminated with 1% MIL-L-23699C (Figure 2a). As the percentage of lubricating oil (MIL-L-23699C) in the sample of 3-GP-26Ma is increased to 10 per cent (Figure 2b), the absorbances at 1110 and 1020  $\text{cm}^{-1}$ , which are due to the synthetic lubricant, can be observed. These absorbances become stronger as the

concentration of the synthetic lubricant in the hydraulic fluid is increased from 10 to 20 and finally to 90% (Figures 2b through 2d).

Figure 3 shows plots of absorbance versus concentration for the infrared absorbances at 1110 and 1020  $\text{cm}^{-1}$ . The absorbances of these peaks was found to increase linearly with concentration of MIL-L-23699C for mixtures containing between 1% and 100% MIL-L-23699C in 3-GP-26Ma. However, it can be seen that it would be difficult to use the curves in Figure 3 to determine MIL-L-23699C concentrations below the 10% level with accuracy. Also these plots are for new fluids and degradation of the fluids during use has been found to make the use of a plot of absorbance versus concentration even more difficult at low levels (less than 10%) of contamination.

In order to determine levels of contamination below 10%, a more sensitive technique was required. The development of a technique that utilizes capillary gas chromatography/mass spectrometry is described in the next section.

### 3.2 Gas Chromatographic Separation of MIL-L-23699C Synthetic Lubricant from 3-GP-26Ma Hydraulic Fluid

The separation of components of a mixture on a gas chromatographic column is a result of the varying affinities of the components for the mobile and stationary phases. For a particular stationary phase and column temperature, each component in a mixture has a characteristic distribution coefficient  $K_D$ , which is the ratio of the amount of the compound in equal volumes of the stationary and mobile phases. By increasing the temperature of the GC column, the volatilities of the components of the mixture are increased. This in turn affects the distribution coefficients of the compounds, their affinities for the stationary and mobile phases, and results in a decrease in the length of time the compounds stay on the column. The decrease in retention time of the various compounds allows an analysis to be completed in a shorter amount of time. However, this can result in a decrease in the separation (resolution) of the components in a mixture and a compromise between speed of analysis and resolution must be made.

As a first approach to the separation of MIL-L-23699C from 3-GP-26Ma, the temperature program CH, listed in Table 2, was used to control the GC oven. This temperature program is commonly used because it allows good separation of unknown mixtures of aliphatic and aromatic hydrocarbons that have a broad range of boiling points. Temperature program CH had an initial oven temperature of 35°C. After five minutes the temperature was ramped to 200°C at a rate of 3° per minute, then ramped to 300°C at a rate of 10° per minute. It took 70 minutes for the oven to reach 300°C from the

starting temperature of 35°C.

The GC/MS trace (reconstructed ion chromatogram or RIC) of a 1 $\mu$ L injection of a 1 in 250 dilution of 3-GP-26Ma in hexane using temperature P1 is shown in Figure 4a. It can be seen in Figure 4a that most of the constituents of this hydrocarbon based hydraulic fluid elute from the GC column between 16 and 40 minutes.

Similarly the RIC for a 1 $\mu$ L injection of a 1 in 250 dilution of MIL-L-23699C in hexane is shown in Figure 4b. The bulk of the constituents of this fluid elute from the column between 67 and 75 minutes. As the retention time envelopes of the constituents of these two fluids were significantly different, it appeared that a capillary GC/MS could be used to separate and detect the presence of synthetic lubricant MIL-L-23699C in 3-GP-26Ma hydraulic fluid.

Figure 5a shows the RIC of a 1 $\mu$ L injection of a 1 in 50 dilution of a 20% mixture of MIL-L-23699C in 3-GP-26Ma hydraulic fluid in hexane. An expansion of the RIC shown in Figure 5a between scans 1500 and 2400 is shown in Figure 5b. The synthetic lubricant can be easily detected in this sample (6 peaks marked in Figures 5a and 5b with retention times greater than 67 minutes). However, the time for the analysis is approximately 75 minutes. The time between the elution of the last of the components of the hydraulic fluid from the column and the first of the components of the synthetic lubricant is about 25 minutes and suggests that the separation could be carried out faster.

Several temperature programs were evaluated to determine the program that gave the desired separation and resulted in a reduced analysis time. The optimum temperature program H1, chosen for the analysis, is listed in Table 2.

In contrast to temperature program CH, program H1 held the temperature of the GC oven at 80°C for 2 minutes, then ramped the temperature at a rate of 20° per minute to 150°C, and finally ramped the temperature at a rate of 10° per minute to 300°C. It took 20.5 minutes for the oven to reach 300°C.

Figures 6a and 6b show the RICs for injections of 3-GP-26Ma and MIL-L-23699C using temperature program H1. The retention time envelopes for components of both 3-GP-26Ma and MIL-L-23699C move to shorter times as compared to those found with temperature program CH. The components of the two fluids have distinct retention time envelopes and elute from the column in less than 25 minutes. The utilization of temperature program H1 reduced the time of an analysis to under 25 minutes from 75 minutes required with program CH.

### 3.3 Detection of MIL-L-23699C Synthetic Lubricant

When a quadrupole mass spectrometer (MS) is utilized as a detector, the minimum detectable quantity of a compound or mixture of compounds is dependent on a number of factors. These include the tuning of the MS electronics, the selection of an electron multiplier voltage, and the scan sequence utilized.

The detection limit of MIL-L-23699C was determined using a full scan (50-500 amu) and a more specialized MID scan mode (see Table 3). The increase in sensitivity gained through the use of a MID is explained in reference 10. The detection limit for MIL-L-23699C using a full scan from 50 to 500 amu (AL in Table 3) and an electron multiplier voltage of 1800 volts was found to be 40 ng of MIL-L-23699C. Using the MID scan H2, shown in Table 3, and an electron multiplier voltage of 1800 volts, the detection limit was lowered to approximately 0.4 ng of MIL-L-23699C. In both instances the detection limit was taken as the amount of sample necessary to give a signal to noise ratio of 10 to 1.

Plots of concentration versus response (integrated area under the peaks in the RIC corresponding to the constituents MIL-L-23699C marked in Figure 6b) for solutions of MIL-L-23699C in hexane are plotted in Figure 7. Figure 7a utilizes data acquired in the full scan mode (50 to 500 amus), while Figure 7b utilizes data acquired with the MID shown in Table 3. The response is linear with concentration over the concentration range studied (40 to 4000 ng MIL-L-23699C in hexane for the full scan and 0.4 to 40 ng MIL-L-23699C in hexane for the MID) as indicated by the correlation coefficients,  $r^2 = 0.9999$  for the plot shown in Figure 7a and  $r^2 = 0.9985$  for the plot shown in Figure 7b.

When the compound or compounds of interest are present at low concentrations in a mixture, the material that is not of interest, i.e., the matrix, is also introduced into the GC/MS system. This material passes through the source and the detector electronics and can lead to a decrease in detector sensitivity and increase the frequency of cleaning and maintenance procedures. To minimize the amount of matrix introduced into the GC/MS system, the fluid to be analysed can be diluted in a suitable solvent, such as hexane. However, the solution cannot be diluted past the point where the compound of interest is not present in a high enough concentration to be detected.

As a first approach it was decided that a reasonable lower limit of MIL-L-23699C contamination 3-GP-26Ma was 0.1%. In order to detect MIL-L-23699C at the 0.1% level in 3-GP-26Ma (using a 1  $\mu$ L injection, a full scan from 50 to 500 amu, and an electron multiplier voltage of 1800), a 1 in 25 dilution in hexane was found to be the maximum permissible dilution to allow detection. This will give a solution containing 40 ng MIL-L-23699C. At such a level of dilution, a large amount of 3-GP-26Ma is introduced

into the electron ionization (EI) source of the quadrupole mass spectrometer. If a large number of analyses are required this level of sample concentration may not be desirable and may result in more frequent maintenance and more rapid deterioration of the MS system.

Through the use of a multiple ion detection (MID) sequence, the detection limit of MIL-L-23699C in 3-GP-26Ma can be reduced to less than 0.4 ng. For a solution containing 0.1% MIL-L-23699C in 3-GP-26Ma, a 1 in 2500 dilution in hexane will contain 0.4 ng of MIL-L-23699C and can easily be detected. Generally a dilution of 1 in 250 or 500 with hexane is sufficient to limit the amount of 3-GP-26Ma introduced into the GC/MS system.

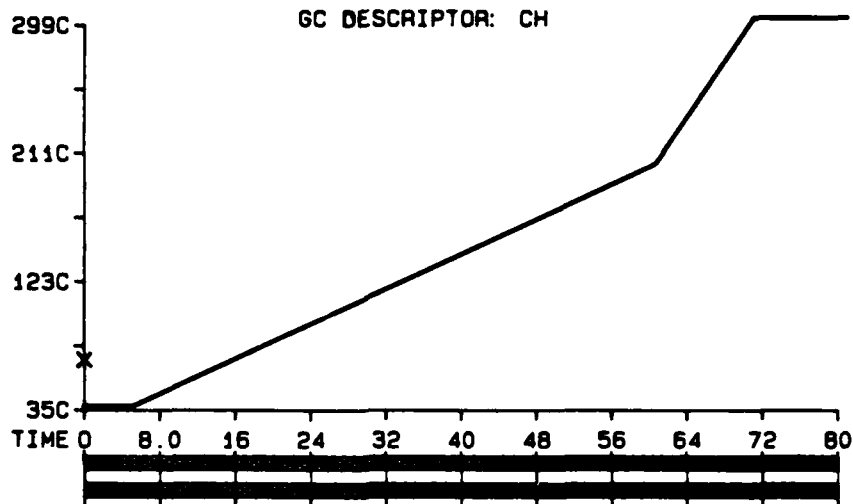
#### 4.0 CONCLUSIONS

A method has been described for the detection of MIL-L-23699C synthetic lubricant contamination of 3-GP-26Ma hydraulic fluid. This method utilizes capillary gas chromatographic separation with mass spectrometric detection and has the sensitivity to detect 0.4 ng of MIL-L-23699C. The response of quadrupole MS is found to be linear with concentration of MIL-L-23699C for solutions containing between 0.4 and 40 ng of (MID scan) and 40 and 4000 ng (full scan 50 to 500 amu) of MIL-L-23699C. In comparison to the previous method which utilized IR spectroscopy, this method yields greater than a 100 fold increase in sensitivity for the detection of MIL-L-23699C in 3-GP-26Ma.

Table 1

Typical parameters utilized to control the quadrupole mass spectrometer. The range of values that these parameters may assume are also listed.

Electron Impact Ionizing Voltage	-70 eV	
Electron Multiplier Voltage	1200 - 2000 V	0 - 2500 V
Collector	-30 V	
Extractor	0.50 V	0 - 10 V
Lens	60 - 80 V	0 - 120 V
Resolution (high)	134	0 - 250
Resolution (low)	134	0 - 250
Ion Energy	1.5 - 2.0 V	0 - 10 V
Ion Program	5.5 - 6.5 V	0 - 10 V
Scan Time	2.0 s	
Mass Range (full scan)	50 - 500 amu	

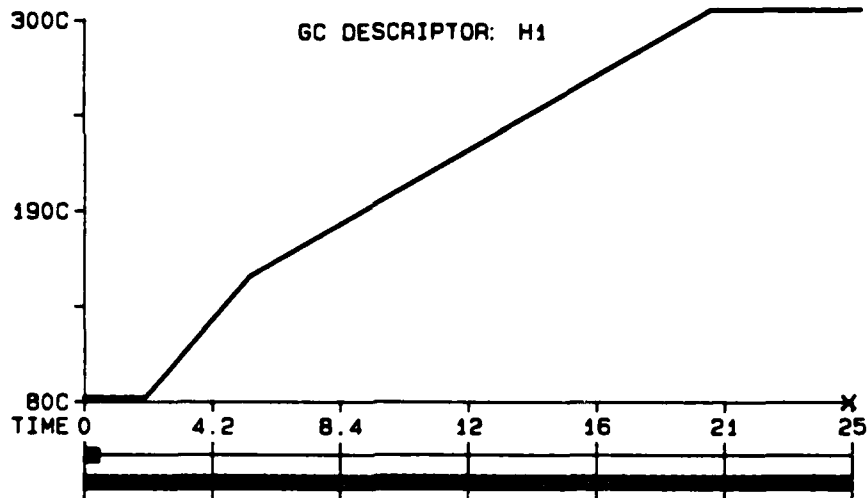


GC VALUES:

		CLOSE	OPEN
(CV) CAPILLARY		0.0	80.0
(DV) DIVERT		0.0	80.0

SEQ. #	FROM TEMP (C)	TO TEMP (C)	RATE (C/M)	TIME (MIN)	TOTAL TIME (MIN)
1	35	- 35	-	5.0	5.0
2	35	- 200	3.0	55.0	60.0
3	200	- 300	10.0	10.0	70.0
4	300	- 300	-	10.0	80.0



GC VALUES:

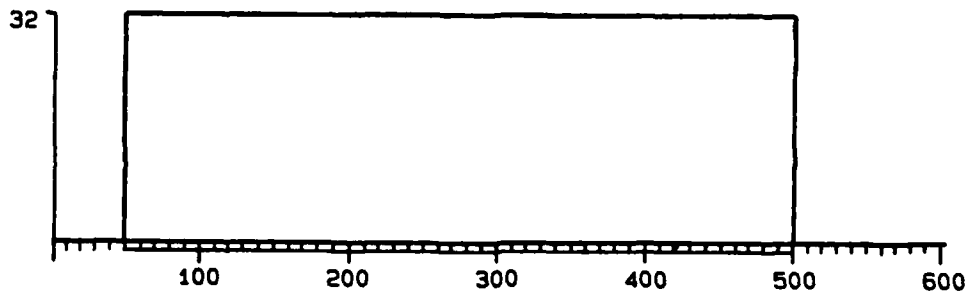
		CLOSE	OPEN
(CV) CAPILLARY		0.0	0.4
(DV) DIVERT		0.0	25.0

SEQ. #	FROM TEMP (C)	TO TEMP (C)	RATE (C/M)	TIME (MIN)	TOTAL TIME (MIN)
1	80	- 80	-	2.0	2.0
2	80	- 150	20.0	3.5	5.5
3	150	- 300	10.0	15.0	20.5
4	300	- 300	-	4.5	25.0

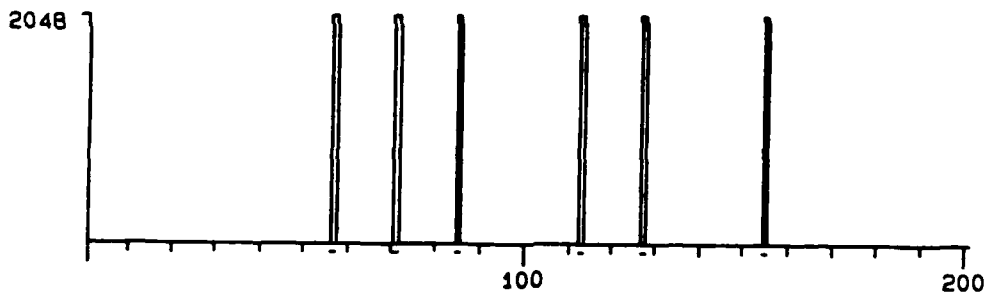
Table 2 - The temperature programs (CH and H1) utilized to control the GC oven.

MID            DESC: AL            MASS DEFECT AT 100 U            30 MMU  
 INST: 5100    CALI: JP30    MASTER RATE                    32  
 TOTAL ACQU TIME                1.874 S  
 TOTAL SCAN TIME                2.000 S  
 CENT SAMP INT                  0.200 MS  
 CALI MASS RANGE                69 TO 614 U



INT #	BEGIN MASS	END MASS	TIME REQUEST	(SECS) ACTUAL	MPW	MFW	MA	TH	BL	ION
1.	49.500	500.500	1.000	2.000	1	80	0	1	0	POS
1.	49.500	500.500	2.000	1.874	3	80	5	1	0	POS

MID            DESC: H2            MASS DEFECT AT 100 U            30 MMU  
 INST: 5100    CALI: JP30    MASTER RATE                    2048  
 TOTAL ACQU TIME                1.593 S  
 TOTAL SCAN TIME                2.000 S  
 CENT SAMP INT                  0.200 MS  
 CALI MASS RANGE                69 TO 614 U



INT #	BEGIN MASS	END MASS	TIME REQUEST	(SECS) ACTUAL	MPW	MFW	MA	TH	BL	ION
6.	56.517	155.546	1.000	2.000	1	80	0	1	0	POS
1.	56.517	57.517	0.200	0.265	3	80	5	1	0	POS
2.	70.521	71.521	0.200	0.265	3	80	5	1	0	POS
3.	84.525	85.525	0.200	0.265	3	80	5	1	0	POS
4.	112.534	113.534	0.200	0.265	3	80	5	1	0	POS
5.	126.538	127.538	0.200	0.265	3	80	5	1	0	POS
6.	154.546	155.546	0.200	0.265	3	80	5	1	0	POS

Table 3 - Diagrammatic representation of the full and multiple ion detection scans utilized to control the quadrupole mass spectrometer. AL is a full scan from 50 to 500 amu while H2 scans for ions of masses 57, 71, 84, 113, 127, and 155 respectively. Both complete a scan in 2 seconds.

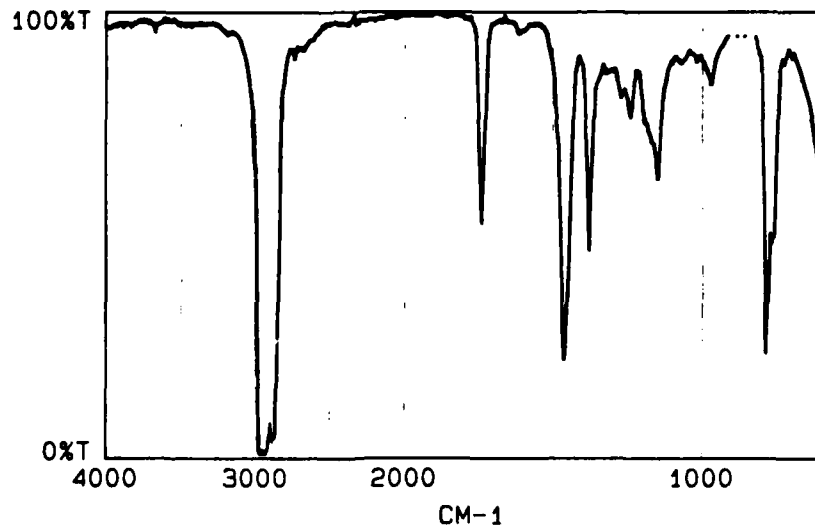


Figure 1a - Infrared spectrum of 3-GP-26Ma hydraulic fluid.

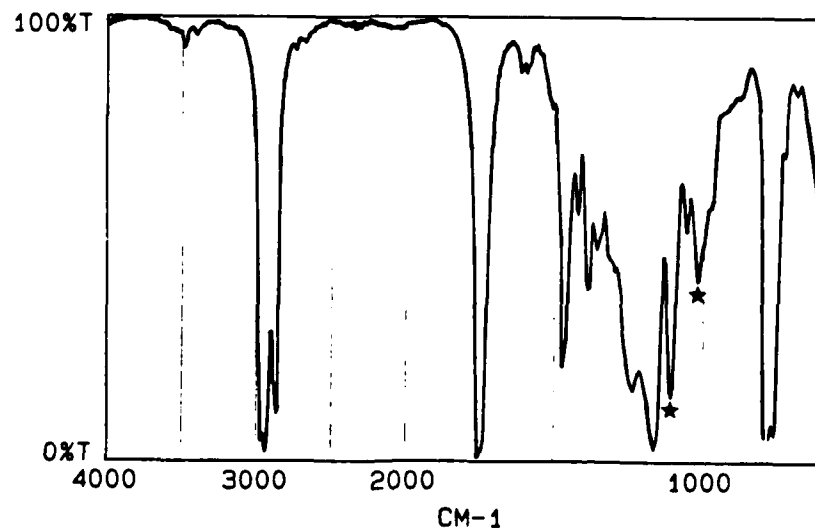
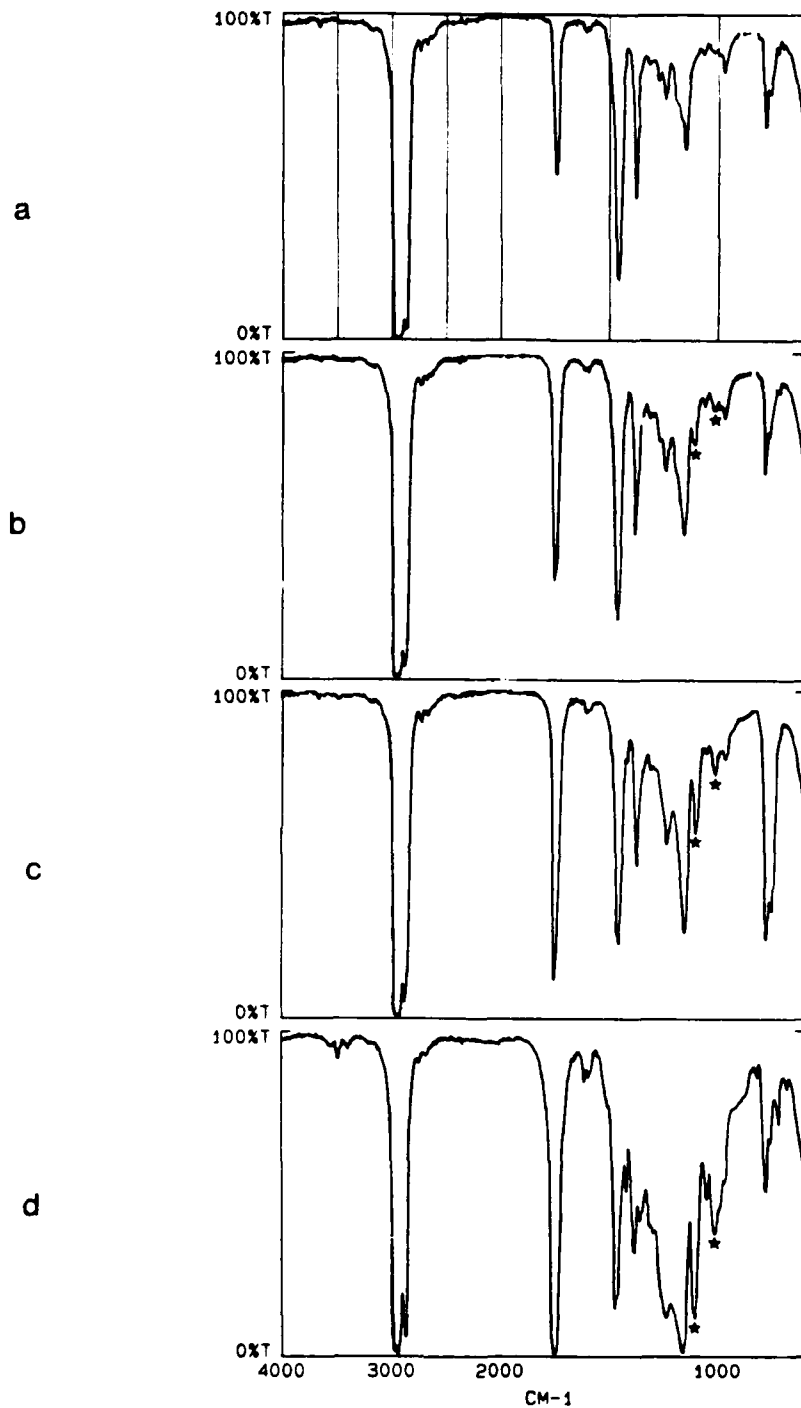


Figure 1b - Infrared spectrum of MIL-L-23699C synthetic lubricant.



Figures 2a-2d - Infrared spectra of mixtures of 3-GP-26Ma hydraulic fluid and MIL-L-23699C synthetic lubricant. Figure 2a - 99% 3-GP-26Ma / 1% MIL-L-23699C, Figure 2b - 90% 3-GP-26Ma / 10% MIL-L-23699C, Figure 2c - 80% 3-GP-26Ma / 20% MIL-L-23699C, Figure 2d - 10% 3-GP-26Ma / 90% MIL-L-23699C.

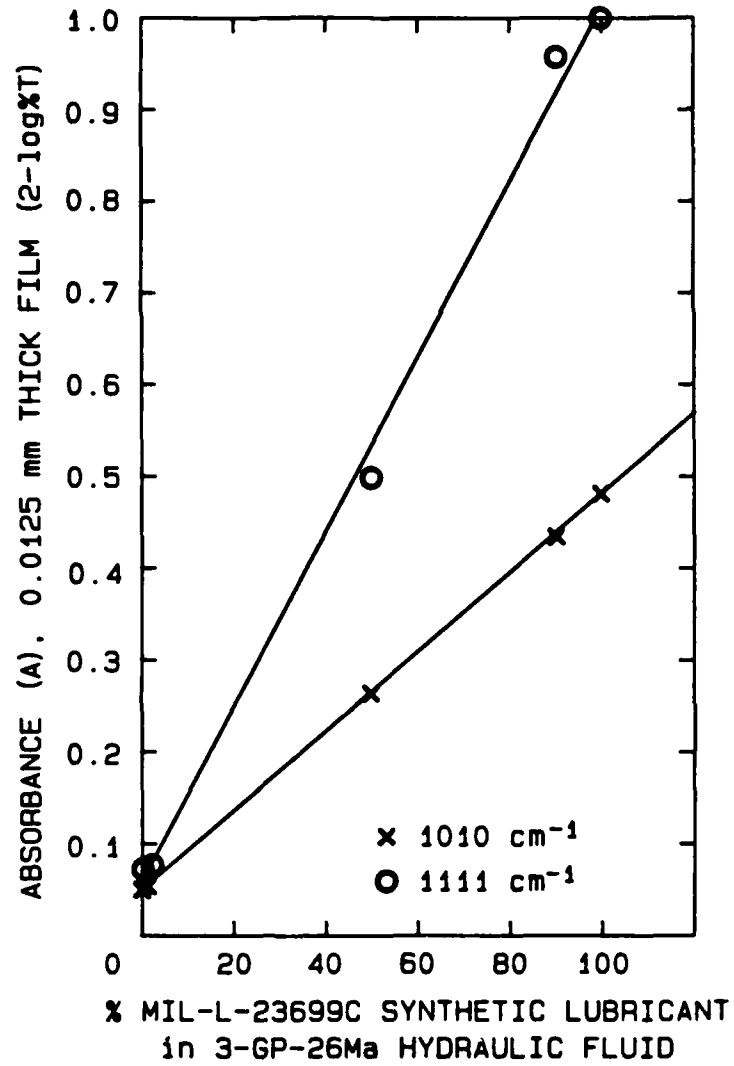


Figure 3 - Plots of the absorbance of the infrared peaks of MIL-L-23699C synthetic lubricant at  $1111\text{ cm}^{-1}$  and  $1010\text{ cm}^{-1}$  against concentration of MIL-L-23699C in solution with 3-GP-26Ma hydraulic fluid.

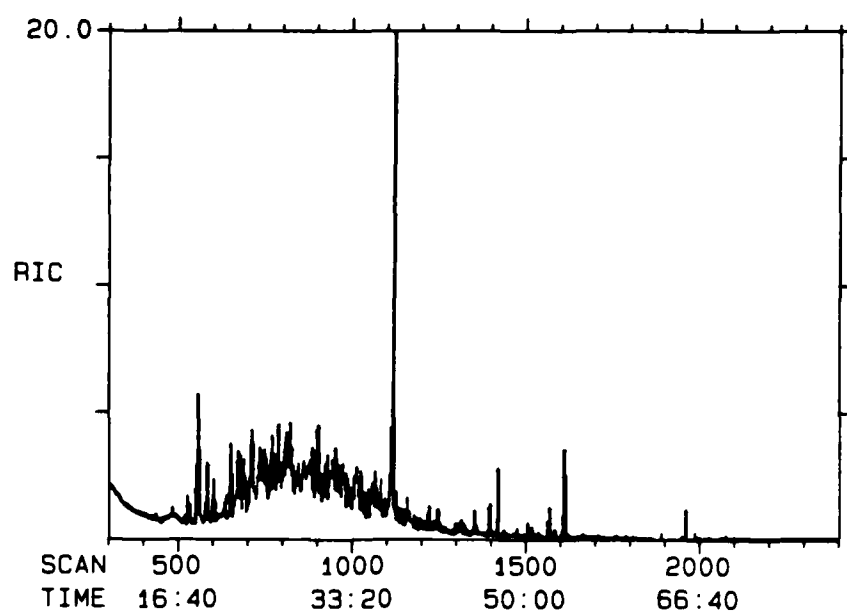


Figure 4a - Reconstructed ion chromatogram (RIC) of 3-GP-26Ma hydraulic fluid.

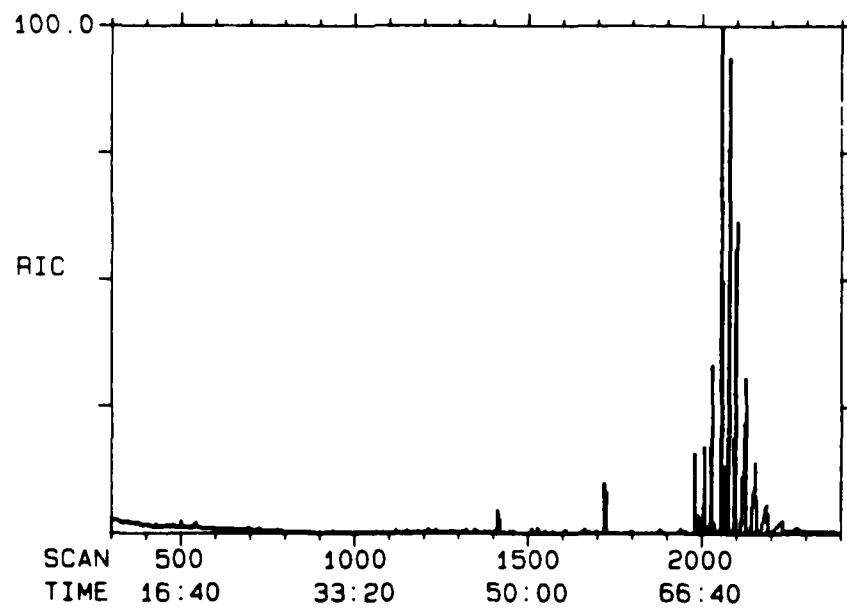


Figure 4b - RIC of MIL-L-23699C synthetic lubricant. Both RICs were acquired using a full scan from 50 to 500 atomic mass units (amus) and the GC oven was controlled with temperature program CH shown in Table 2.

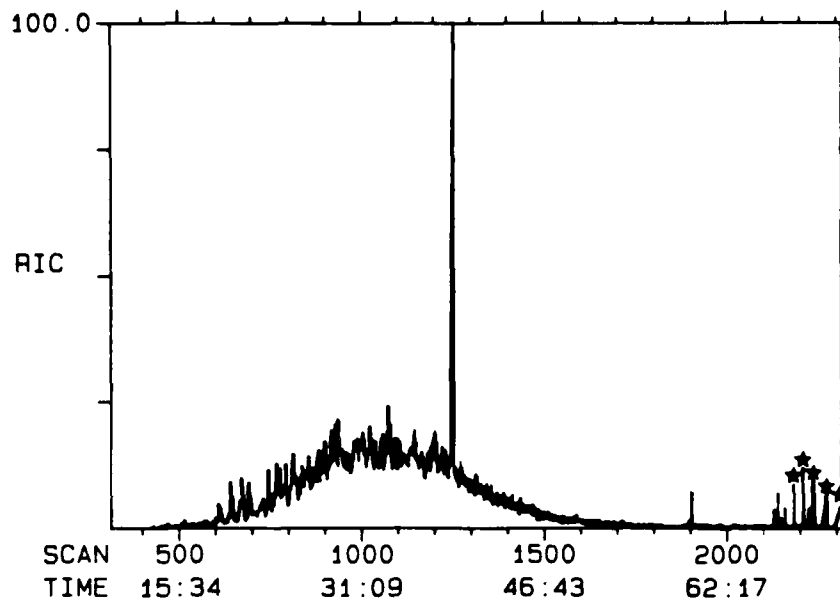


Figure 5a - Reconstructed ion chromatogram (RIC) of a 20% solution of MIL-L-23699C synthetic lubricant in 3-GP-26Ma hydraulic fluid.

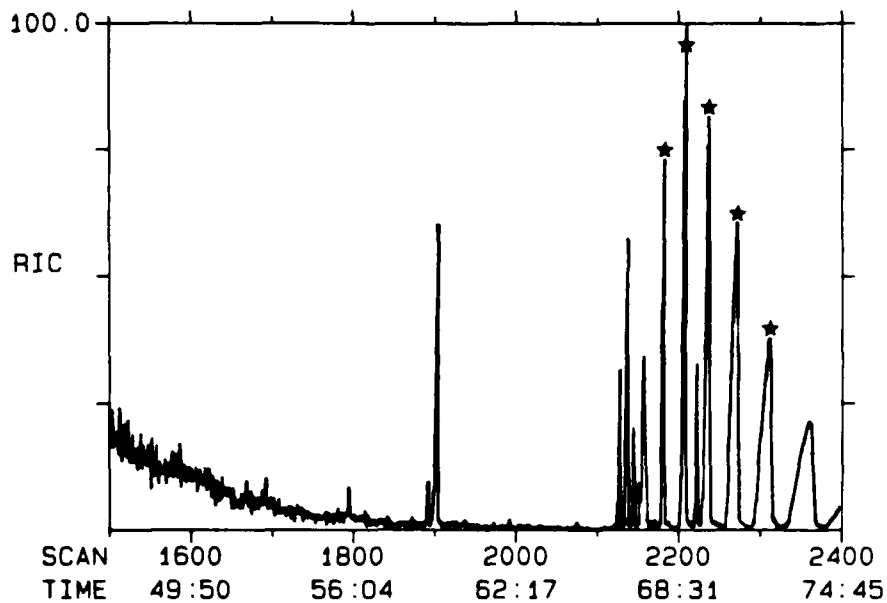


Figure 5b - Expansion of the RIC shown in Figure 5a between scans 1500 and 2400 showing the peaks due to MIL-L-23699C synthetic lubricant contamination of the sample.

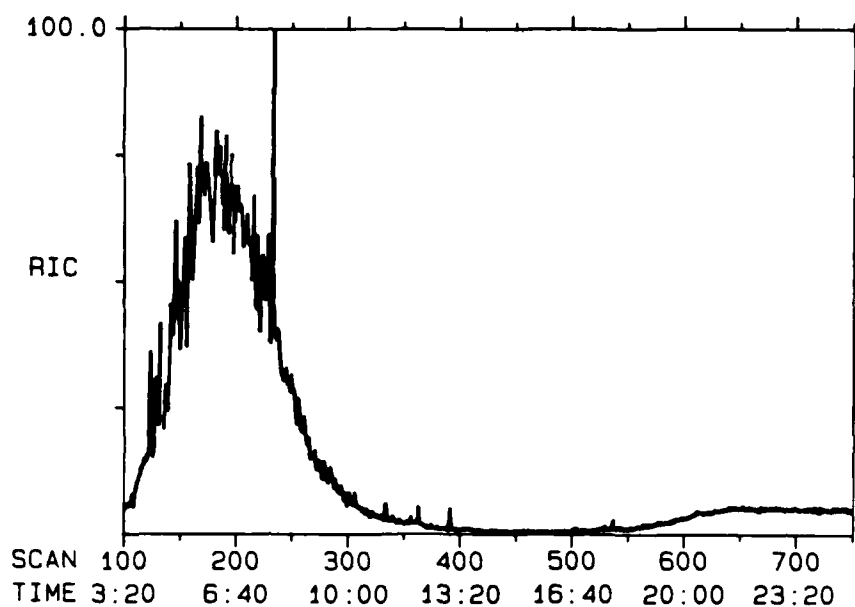


Figure 6a - Reconstructed ion chromatogram (RIC) of 3-GP-26Ma hydraulic fluid acquired using multiple ion detection (MID) scan H2 shown in Table 3.

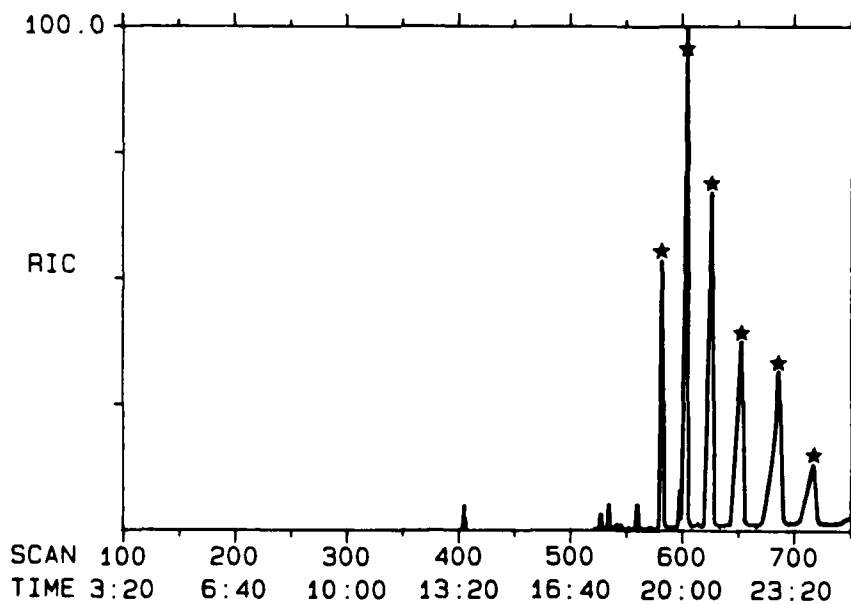


Figure 6b - RIC of MIL-L-23699C synthetic lubricant acquired using MID scan H2 shown in Table 3. For the RICs in Figures 6a and 6b, the GC oven was controlled with temperature program H1 shown in Table 2.

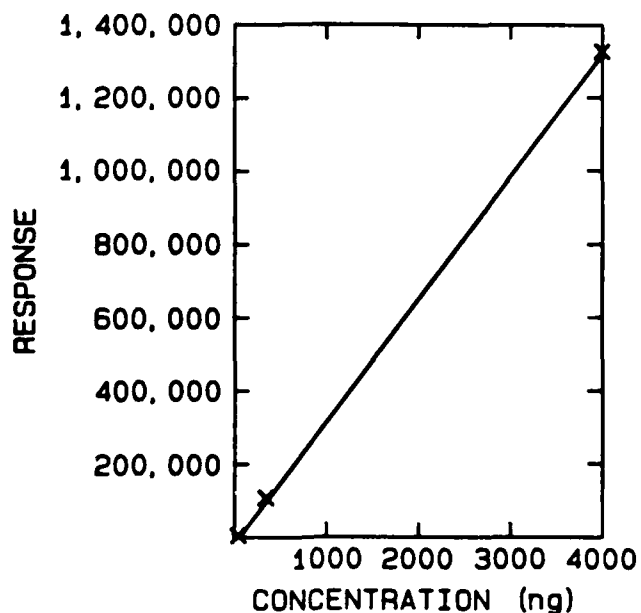


Figure 7a - Plot of the response of MIL-L-23699C synthetic lubricant (measured as the area under the peaks marked in Figure 6b) against concentration of MIL-L-23699C in solution. The RIC was acquired using a full scan from 50 to 500 amus.

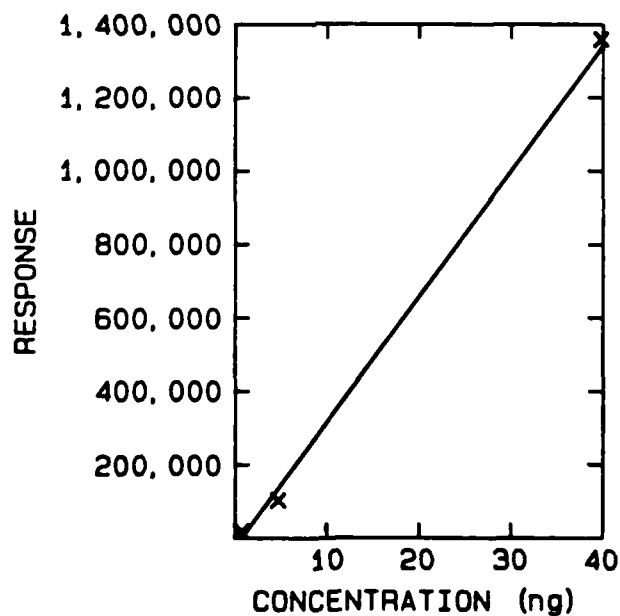


Figure 7b - Plot of the response of MIL-L-23699C synthetic lubricant against concentration. The RIC was acquired using the MID scan H1 shown in Table 3.

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## KEY WORDS

3-GP-26Ma Hydraulic Fluid  
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