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CHARACTERIZATION OF HYDRAULIC FLUIDS, NADRAUL MS-5
AND MIL-H-5606

L. C. Fuller, et al

Naval Air Development Center
Warminster, Pennsylvania

1 April 1975

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) A currently used hydraulic fluid (MIL-H-5606), a new fire resistant hydraulic fluid (MS-5), and components of both fluids were characterized. The molecular structures of the fluid components were elucidated by infrared and nuclear magnetic resonance spectroscopy. The thermal stabilities of the fluids and their components were studied over the expected temperature range of service use by differential thermal analysis and thermogravimetric analysis.		

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S U M M A R Y

INTRODUCTION

A new hydraulic fluid developed by the Naval Air Development Center designated as NADRAUL MS-5 has been under study for some time and has been considered as a possible candidate to replace the present hydraulic fluid covered by the Military Specification MIL-H-5606. The NADRAUL MS-5 fluid is basically a silicone fluid (designated as DOW 560 chlorinated methyl phenyl silicone base fluid) to which additives, including 2,5-bis(t-dodecyldithio)-1,3,4 thiadiazole (AR-155) and dibutyl chlorendate (CCD), have been added. The MIL-H-5606 fluid consists of a petroleum base stock with additives to prevent wear and improve its viscosity over the working temperature ranges. The MS-5 fluid is fire resistant while the 5606 fluid is highly flammable.

This study has dealt largely with the MS-5 fluid and for the most part with the additives used to improve its properties as a hydraulic fluid. The work was conducted under AIRTASK A3200000/001A/4R02200000, Work Unit JP 202.

DISCUSSION

The information obtained in this investigation results from the first attempts to characterize these materials. The selection of infrared spectrophotometry (IR) and nuclear magnetic resonance spectroscopy (NMR), along with differential thermal analysis (DTA) thermogravimetric analysis (TGA), was made as the fundamental characterization techniques, these being basic tools for characterization analysis. The IR and NMR studies did help to elucidate the molecular structure of the components used in the hydraulic fluid formulations; however, in reviewing the data it can be readily seen that further work must be done because of the complex structure of some of these compounds. The component designated as AR-155 (antiwear additive), when analyzed by IR and NMR spectroscopy, raises some questions as to its true molecular configuration. Perhaps additional steps to purify the compound would resolve the assignment of a definite molecular structure to this component.

The IR and NMR studies indicated molecular structure of the component CCD (rubber swelling additive with antiwear qualities) was in good agreement with that reported by the supplier. However, it is believed that the purity of the compound could be increased and, therefore, better resolved spectra obtained.

The thermal work confirmed the fact that the fluids do break down at temperatures above 250°C, and the study shows that when MS-5 type fluid is contaminated with 5606 type fluid, the problem of the lower temperature range of 5606 is easily seen and indicates the necessity of limiting its use in combination with MS-5 and other hydraulic fluids.

CONCLUSION

This report summarizes the efforts of this laboratory in determining the molecular structure and characterizing the components used in MS-5 hydraulic fluid

and some of the components used in 5606 hydraulic fluid. No direct conclusion can be drawn. It is felt that there is much work to be done if the compounds are to be fully characterized. The IR and NMR studies helped to elucidate the molecular structure although some questions have been raised regarding the structure of the component AR-155 with respect to the methyl groups. The thermal study yielded information regarding the instability of the compounds when the MS-5 fluid is contaminated with 5606 type fluid, however, within the stated working temperature ranges of the uncontaminated formulated fluids all indications are that they are thermally stable.

FUTURE WORK

Since the AR-155 and CCD compounds are candidates for use in hydraulic fluid formulations of various base materials, an investigation utilizing liquid chromatography is currently in progress at this laboratory. This work is expected to further elucidate the chemical characteristics of these two components. Appropriate techniques could then be established to monitor the production of these materials with respect to their purity.

Chemical characterization should also proceed along the lines of examining fluids which have been subjected to pump tests and have recorded histories of good and bad performance. This has already been initiated with the NMR studies, and should continue with TGA and DTA studies.

To further define the molecular structure of all of these materials, techniques involving molecular weight determinations, mass spectrometry, and elemental analysis should be explored.

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INVESTIGATIVE PROCEDURES

DESCRIPTION OF INSTRUMENTAL TECHNIQUES

The infrared analyses of MS-5 and 5606 hydraulic fluids and their components were performed on a Beckman Model IR-9 infrared spectrophotometer. The spectra were obtained from thin films cast on sodium chloride plates. References (a), (b) and (c) were used in the assignment of spectral bands.

All nuclear magnetic resonance spectra reported herein were obtained on a 60 MHz Perkin-Elmer R-24 spectrometer. Samples were prepared in deuteriochloroform. When tetramethylsilane was used as an internal standard, its absorption appears at 0.00 ppm on each spectrum. Chemical shift values were calculated using the chemical shift correlation tables of references (d), (e) and (f). All chemical shift values in these spectra are subject to an error of ± 0.05 ppm due to magnetic drift of the instrument. Integration values are accurate to approximately 5%.

The equipment used for differential thermal analysis was the duPont 900, the basic thermal unit, with standard and intermediate temperature cells. The temperature range used was room temperature (RT) to 900°C for some and to 200°C or 500°C as required by the compound being studied. The specimens were heated at a rate of 15°C/min. Macro cells filled to a height of 2-3 mm were used.

The du Pont 950 TGA module for thermogravimetric analysis is a semi-micro balance designed to measure the weight of a material as a function of a linearly increasing temperature. Samples were heated to 500°C at a rate of 15°C/min in this study. The TGA can also be used to study the weight change of a material as a function of time while held at a specified temperature. The measurements on both thermal analysis instruments are reproducible within the experimental error limits when operated in the same manner each time.

INFRARED STUDIES

CCD Additive

The infrared curve of CCD (Figure 1) shows strong absorption between 2970 and 2840 cm^{-1} , corresponding to aliphatic C-H stretching vibrations. Bands at 1465 cm^{-1} and at 1390 cm^{-1} correspond to C-H deformations.

A strong carbonyl stretch occurs at 1750 cm^{-1} , and a broad C-O stretch is observed at 1200 cm^{-1} , which are characteristic of the ester group. The sharp band at 1605 cm^{-1} corresponds to the C=C stretch where the absorption frequency is decreased due to the attachment of chlorine atoms. The absence of an alkene C-H stretch is noted above 3000 cm^{-1} , confirming the structure of the disubstituted cycloalkene.

C-Cl absorption is normally observed in the region between 850 and 550 cm^{-1} . The spectrum of CCD shows several bands in this region as might be expected in a polychlorinated compound.

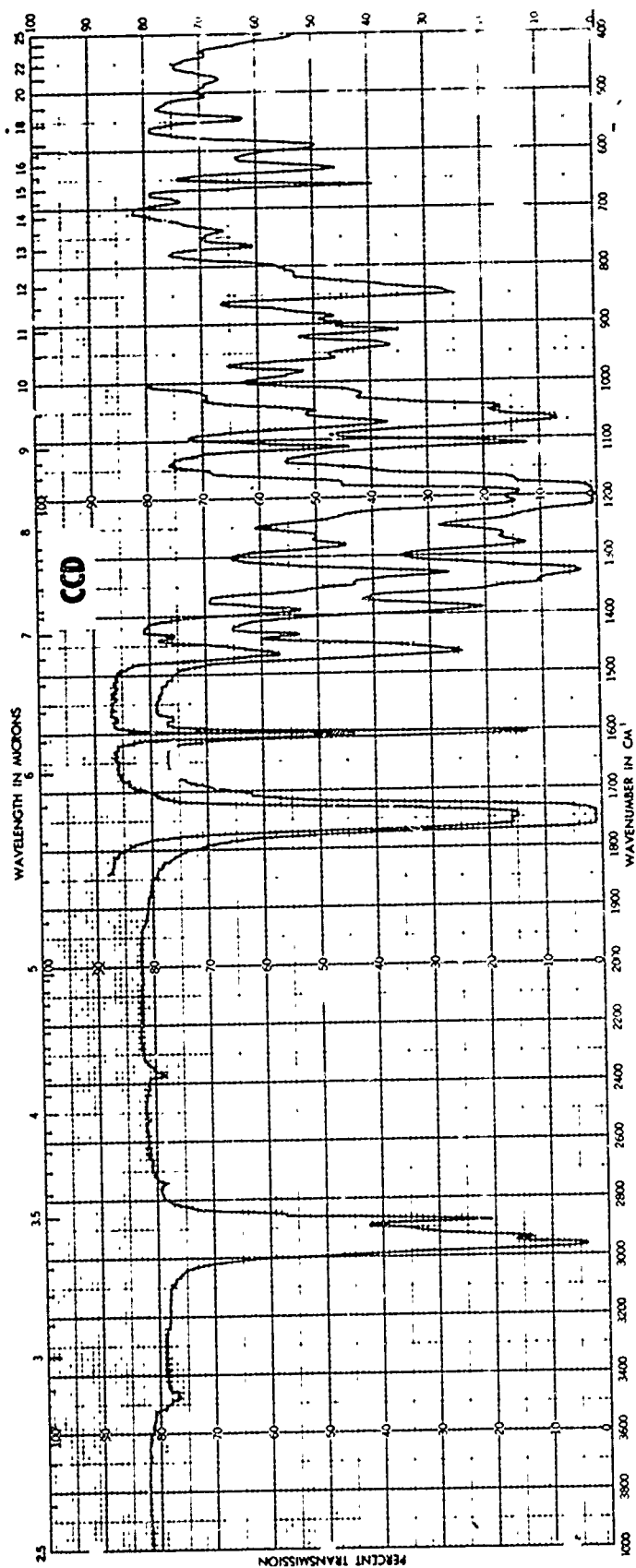


Figure 1 ~ IR SPECTRUM CCD

AR-155 Additives

The infrared curve of AR-155 (Figure 2) is rather featureless. Strong bands associated with C-H stretching appear between 2980 and 2840 cm^{-1} , and C-H deformations at 1465 and 1385 cm^{-1} . According to the structure, the aliphatic disulfide substituent should end in a t-butyl group. This is often characterized in IR by two bands in the region of 1395-1370 cm^{-1} . A single band appears in this region in the spectrum of AR-155. Straight chain aliphatic groups of 7 or more carbons generally show an absorption band at 720 cm^{-1} . The spectrum of AR-155 shows an absorption at higher frequency, 750 cm^{-1} , characteristic of a shorter chain.

Thiadiazoles often show absorptions which give rise to three bands in the regions 1570-1395, 1420-1260, and 1140-940 cm^{-1} . In the spectrum of AR-155, bands are found in each of these regions. At 1465 and 1385 cm^{-1} , however, these are more likely to result from the aliphatic C-H deformations described. A weak band possibly due to S-CH₂ wagging vibrations appears at 1250 cm^{-1} .

Dow Corning 560 Chlorinated Methyl Phenyl Silicone Fluid

The infrared spectrum of DOW 560 silicone fluid (Figure 3) shows absorption associated with aliphatic C-H stretching in the region from 2980 to 2900 cm^{-1} . The strong bands at 1260 and 805 cm^{-1} are associated with Si-CH₃ and Si-(CH₃)₂ vibrations. C-Cl absorption may also be present at 760 cm^{-1} . Additional weaker bands at 760 and 850 cm^{-1} suggest some Si-(CH₃)₃ absorptions. There are several weak bands in the region from 1460 to 1360 cm^{-1} . Groups that absorb in this region include Si-CH₃, Si-CH₂, and Si-phenyl groups. Si-phenyl also absorbs about 1125, 730, and 700 cm^{-1} ; however, these regions are obscured by Si-CH₃ and Si-O-Si absorptions. The characteristic absorptions associated with aromatic substituents are not apparent in the spectrum. However, it must be considered that the phenyl group should be present in low concentration. The very strong doublet at 1100-1030 cm^{-1} is characteristic of the Si-O-Si stretching vibrations of a long siloxane polymer.

NADRAUL MS-5

Absorptions in the MS-5 fluid, (Figure 4) which do not appear in the base fluid, occur at 1750, 1600 and 1330 cm^{-1} , and as a shoulder at 1200 cm^{-1} . These absorptions correlate with the IR curve of CCD. The CCD additive also increases the intensity of the C-H stretch region in MS-5. Contribution of the AR-155 component is not apparent in the spectrum.

MIL-H-5606 Petroleum Base Stock

The only bands of significance in the spectrum of the 5606 petroleum base stock (Figure 5) appear to be due to the vibrations of aliphatic C-H: 2960-2840 cm^{-1} (corresponding to C-H stretching), 1460 and 1380 cm^{-1} (corresponding to C-H deformations).

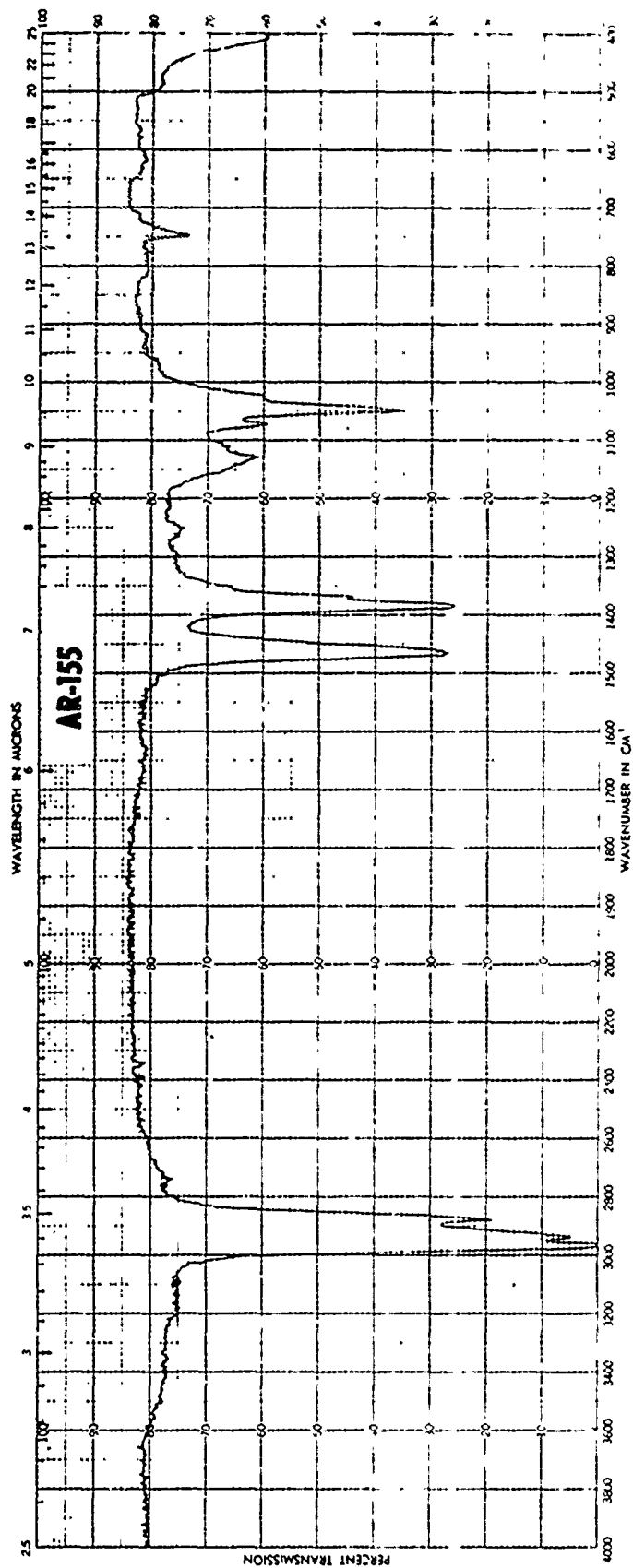


Figure 2 - IR SPECTRUM AR-155

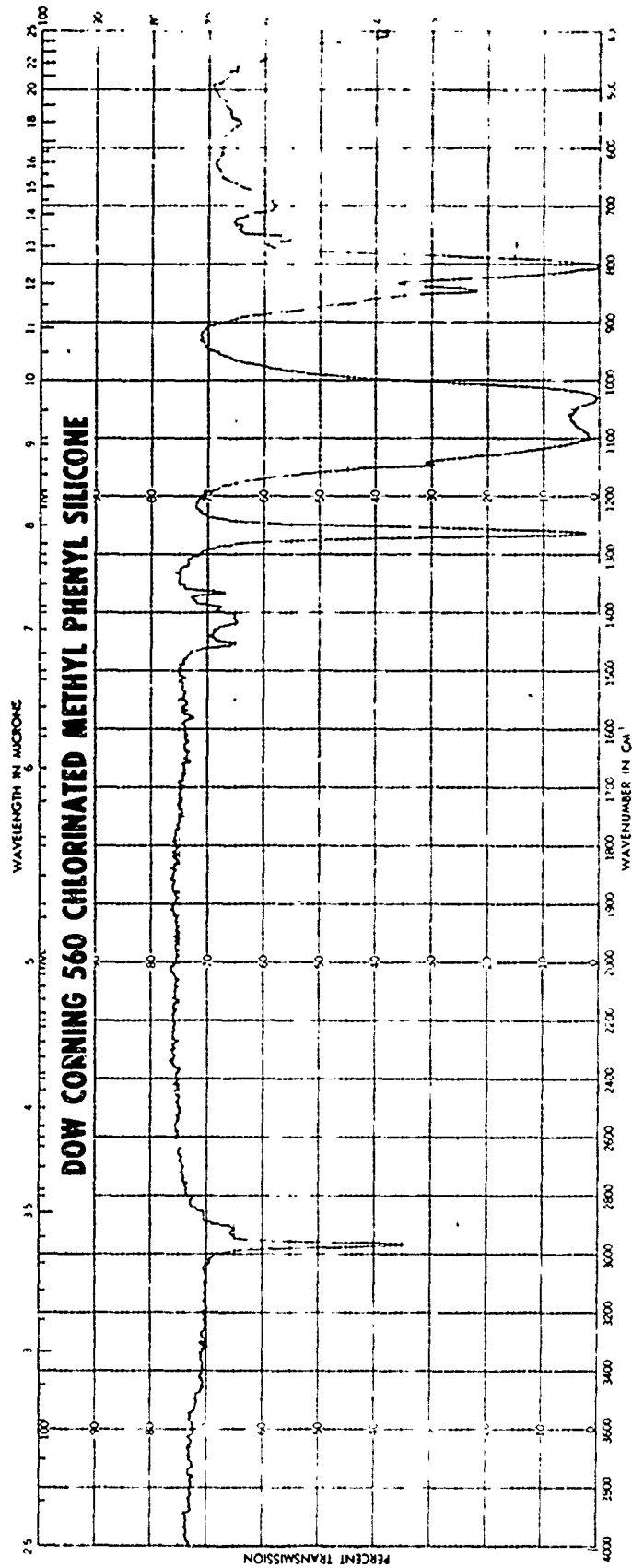


Figure 3 - IR SPECTRUM DOW CORNING 560 SILICONE FLUID

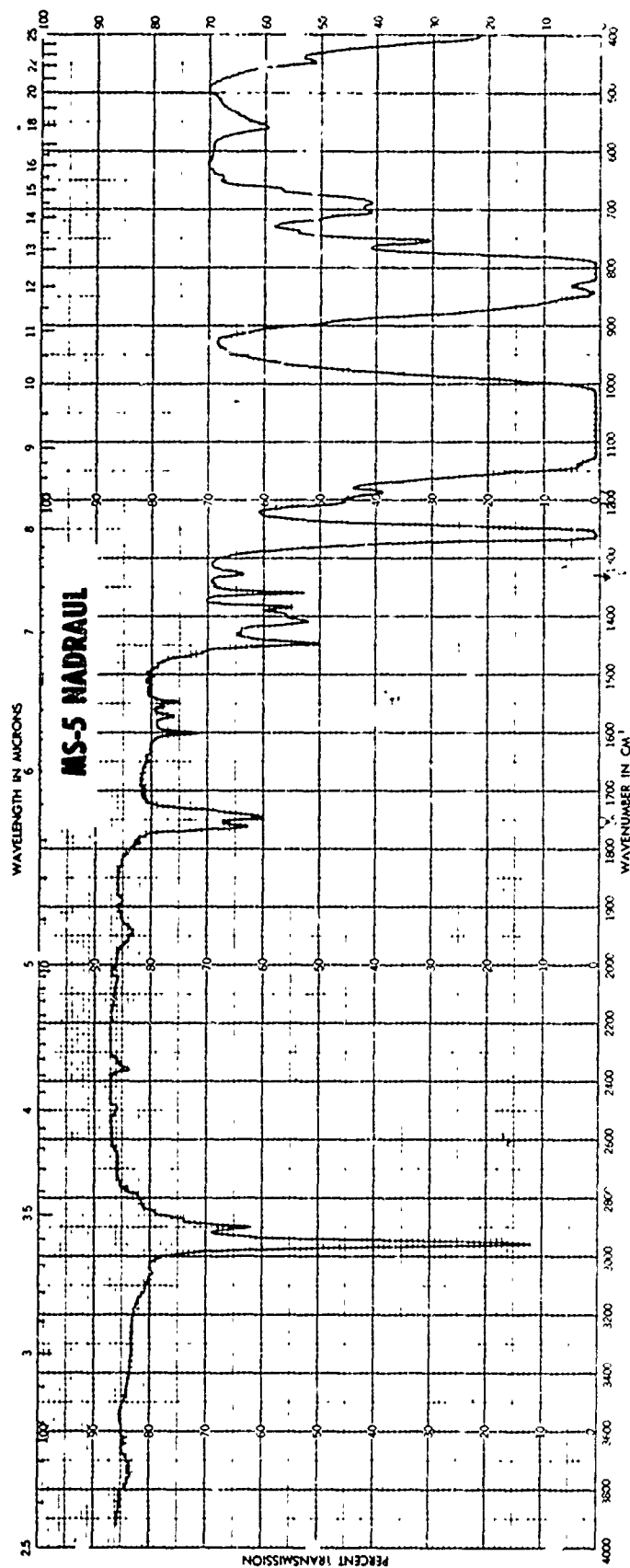


Figure 4 - IR SPECTRUM NADRAUL MS-5

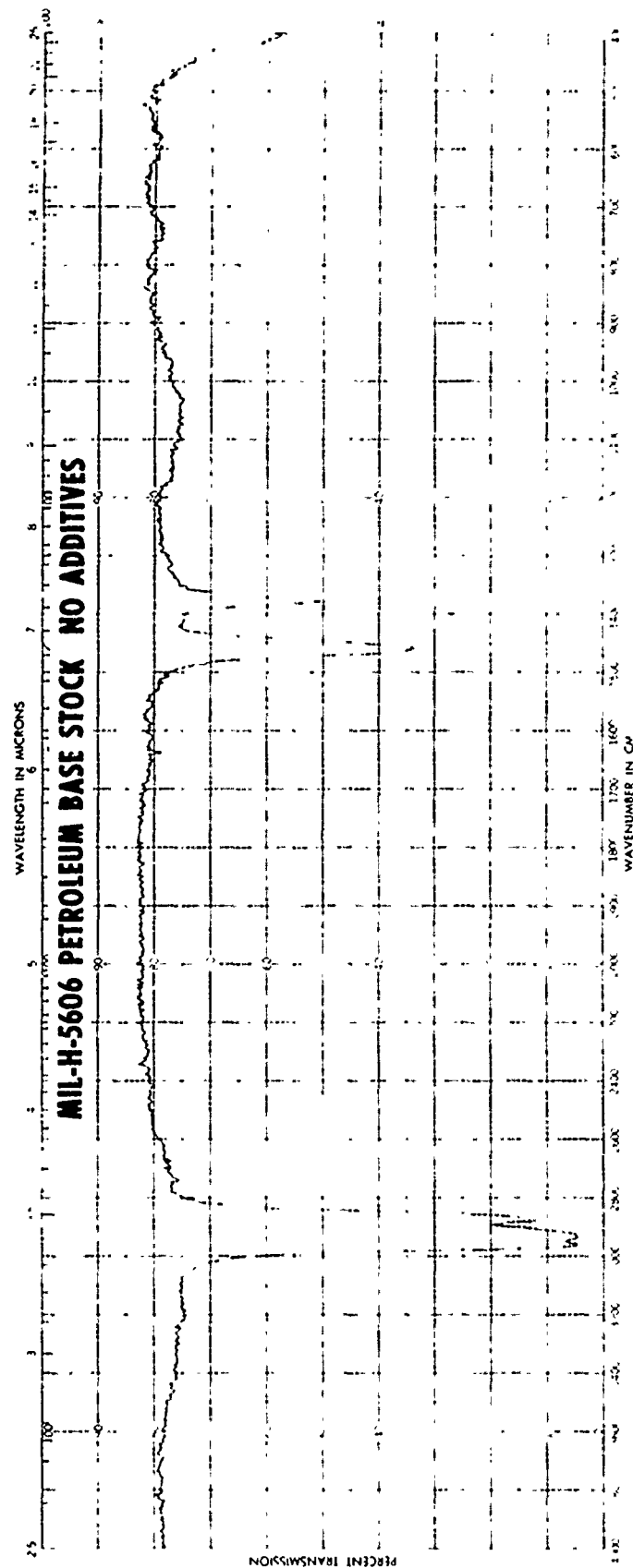


Figure 5 - IR SPECTRUM MIL-H-5606 PETROLEUM BASE STOCK

NUCLEAR MAGNETIC RESONANCE STUDIES

CCD Additives

Several batches of the CCD rubber swell additive to the MS-5 hydraulic fluid were studied, including a batch that had been evaluated as unacceptable and another which had been evaluated as good. No significant differences could be detected by NMR. In each spectrum the ratio of peak integrations was found to be approximately the same.

According to its structure, the NMR spectrum of CCD should show four types of proton absorptions. Spectra of CCD run in this laboratory (Figure 6) showed a distorted triplet centered at 0.9 ppm, which integrated for 3 protons (calculated value 0.9 ppm for the terminal methyl protons of the n-butyl group). A complex multiplet centered at 1.5 ppm integrated for 4 protons (calculated value 1.3 - 1.6 ppm corresponding to the protons of the two inner methylene groups).

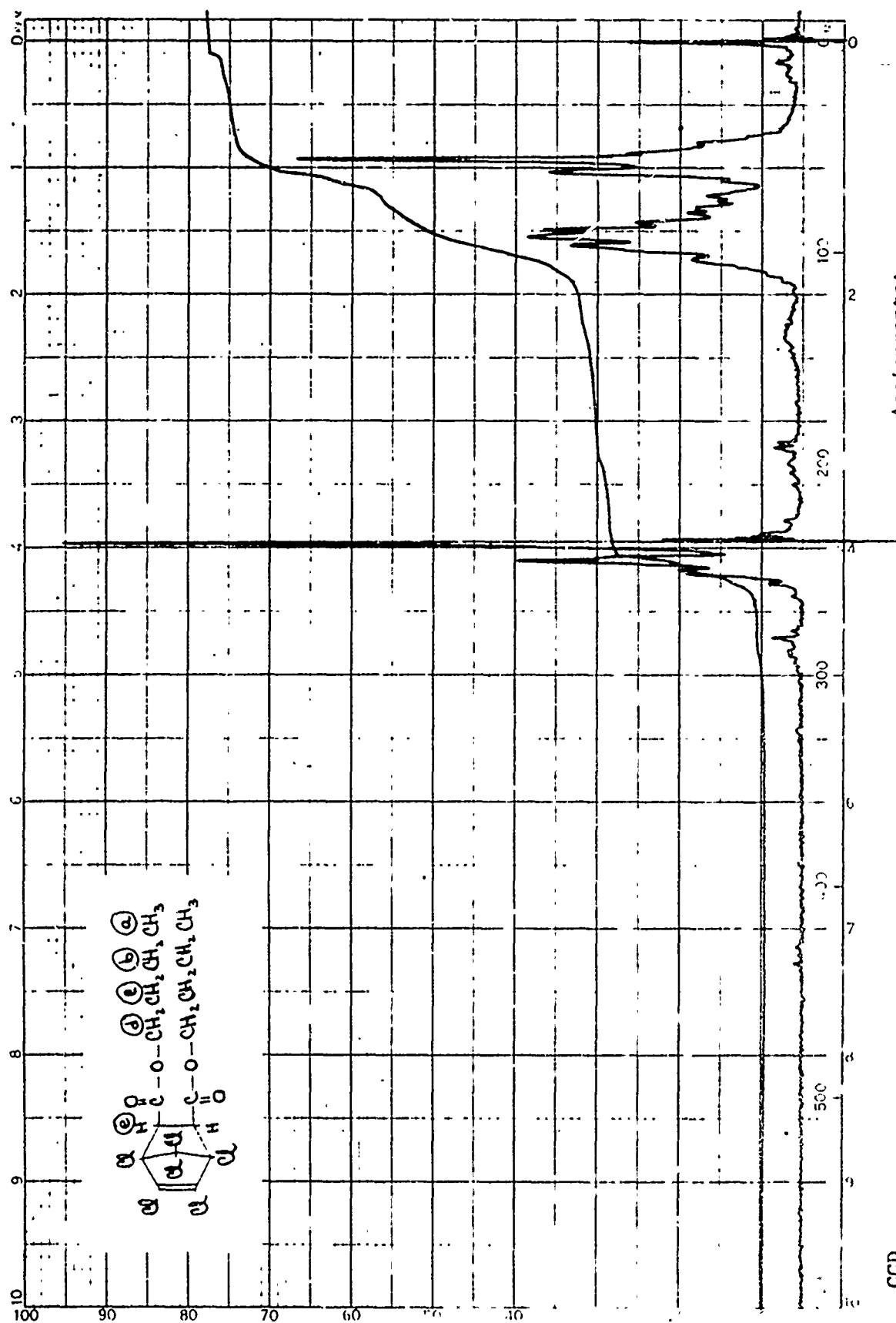
The spectrum showed a multiplet centered at 4.1 ppm which integrated for 3 protons. Values calculated from the correlation tables predict that the absorption for the methylene protons adjacent to the ester group should be shifted appreciably downfield to about 4.2 ppm, and should appear as a triplet. The theoretical position of the methine proton could not be predicted accurately. Whatever its chemical shift, it should appear as a sharp singlet. It would appear from comparison of theoretical information with the spectrum of CCD that the absorption peak of the methine proton overlaps with the right peak of the methylene triplet, since the entire multiplet integrates for 3 protons and no sharp singlet appears in any other part of the spectrum. Additional evidence is the presence of ringing below the baseline at 3.9 ppm which is characteristic of a sharp singlet.

AR-155 Additives

Analysis of AR-155 by NMR was not conclusive. Samples were run in various solvents. However, poor resolution resulted in all cases. Inflection points on the integration scans were poorly resolved, and so actual integration ratios are not reported here. Although the spectra are not conclusive, several things should be noted.

The principle absorptions are a distorted triplet at 0.9 ppm (indicating methyl protons), and a multiplet at 1.3 - 1.7 ppm (methylene protons) (Figure 7).

From theoretical predictions, the methyl protons of the t-butyl group should absorb as a singlet at 0.9 ppm. The presence of splitting in the methyl absorption is in disagreement with the structure given for AR-155. Most of the methylene protons should be centered about 1.3 ppm. Some would be deshielded slightly by the -SSR group. This is in agreement with the spectrum obtained. However, protons directly adjacent to the -SSR group might be expected to absorb at about 2.7 ppm. This absorption would be small in relation to the methyl and other methylene absorptions. Such an absorption is not apparent in the spectrum.



Assignments:

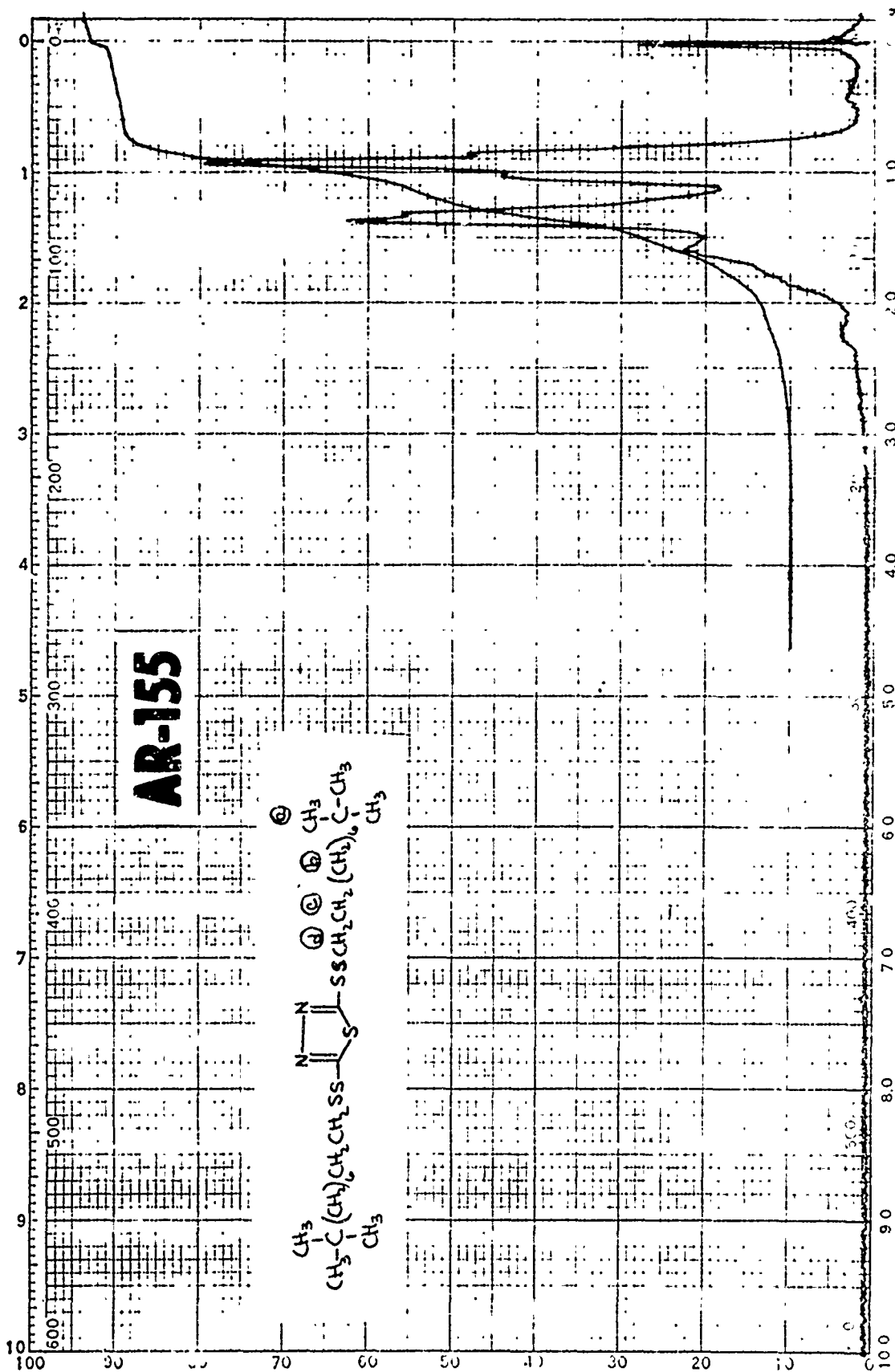
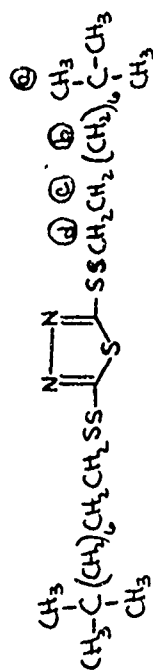
CCD

(hydraulic fluid additive)

Solvent: CDCl_3

Reference: TMS

Figure 6 - NMR SPECTRUM CCD

AR-155

Assignments:

- a. 0.9 ppm
- b. ca 1.4 ppm
- c. ca 1.6 ppm
- d. ca 2.5 ppm

AR-155

(hydraulic fluid additive)

Solvent: CDCl₃

Reference: TMS

Figure 7 - NMR SPECTRUM AR-155

Dow 560 Chlorinated Methyl Phenyl Silicone Fluid and NADRAUL MS-5

Spectra of the Dow 560 silicone fluid (Figure 8) and the complete MS-5 hydraulic fluid show approximately the same absorption patterns in the regions of 0.1 ppm and 7.2 - 7.7 ppm. The absorption at 0.1 ppm appears as a singlet. Its high field position and the absence of splitting indicate that this absorption corresponds to methyl protons adjacent to silicon. Although silicon has a strong shielding effect upon alkyl protons, it does not strongly shield phenyl protons. The absorption appears in the spectrum at 7.2 - 7.7 ppm. Sidebands and the low concentration CHCl_3 absorption in the CDCl_3 solvent interfere with the integration; however, the ratio of the two areas of absorption appear to be on the order of 20:1 (methyl to phenyl groups).

Comparison of the spectra shows that the complete fluid has additional low concentration absorptions, visible on the high amplitude spectrum of MS-5 (Figure 9) at 1.6 ppm and at 4.0 ppm, due probably to the absorption of the CCD and AR-155 components of the MS-5 fluid.

Tricresyl Phosphate

Examination of a spectrum of tricresyl phosphate (Figure 10) (additive to the 5606 hydraulic fluid) by NMR showed a singlet at 2.2 ppm which integrates for 3 protons, and a multiplet centered at 7.2 ppm, integrating for 4 protons. Theoretically the spectrum should show two areas of absorption: a singlet at 2.3 ppm (corresponding to the methyl protons), and a complex multiplet centered at about 7.2 ppm (corresponding to the protons of the ortho-disubstituted phenyl group). The spectrum obtained is, therefore, in agreement with that predicted for tricresyl phosphate.

5606 Hydraulic Fluid, Petroleum Base Stock

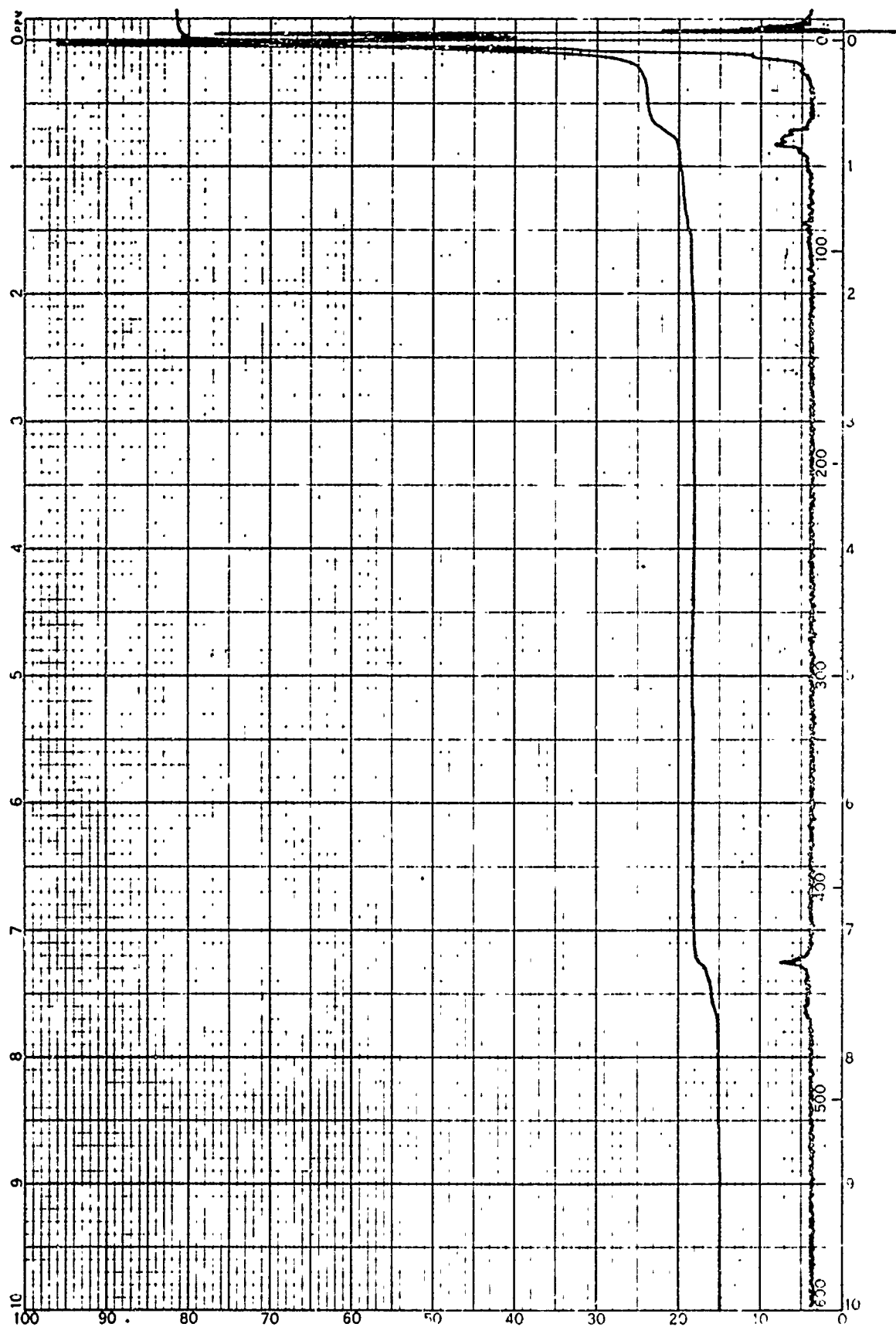
Comparison of the complete 5606 fluid and the 5606 petroleum base stock (Figures 11 and 12) shows that the principle absorptions occur in the same areas: 0.9 ppm (characteristic of methyl protons), and from 1.2 - 1.8 ppm (characteristic of methylene protons), and in the same ratio (approximately 1:1.6). The complete 5606 fluid shows a low concentration absorption at about 4.0 ppm which is not observed in the base stock.

Comparison of Various Formulations of 5% 5606 in MS-5

Comparison spectra were run of several formulations of 5% 5606 in MS-5:

- I pump tested, filtered, showing bad wear qualities
- II pump tested, filtered, showing good wear qualities
- III pump tested, unfiltered
- IV prepared in laboratory, 5% (w/w) 5606 in MS-5

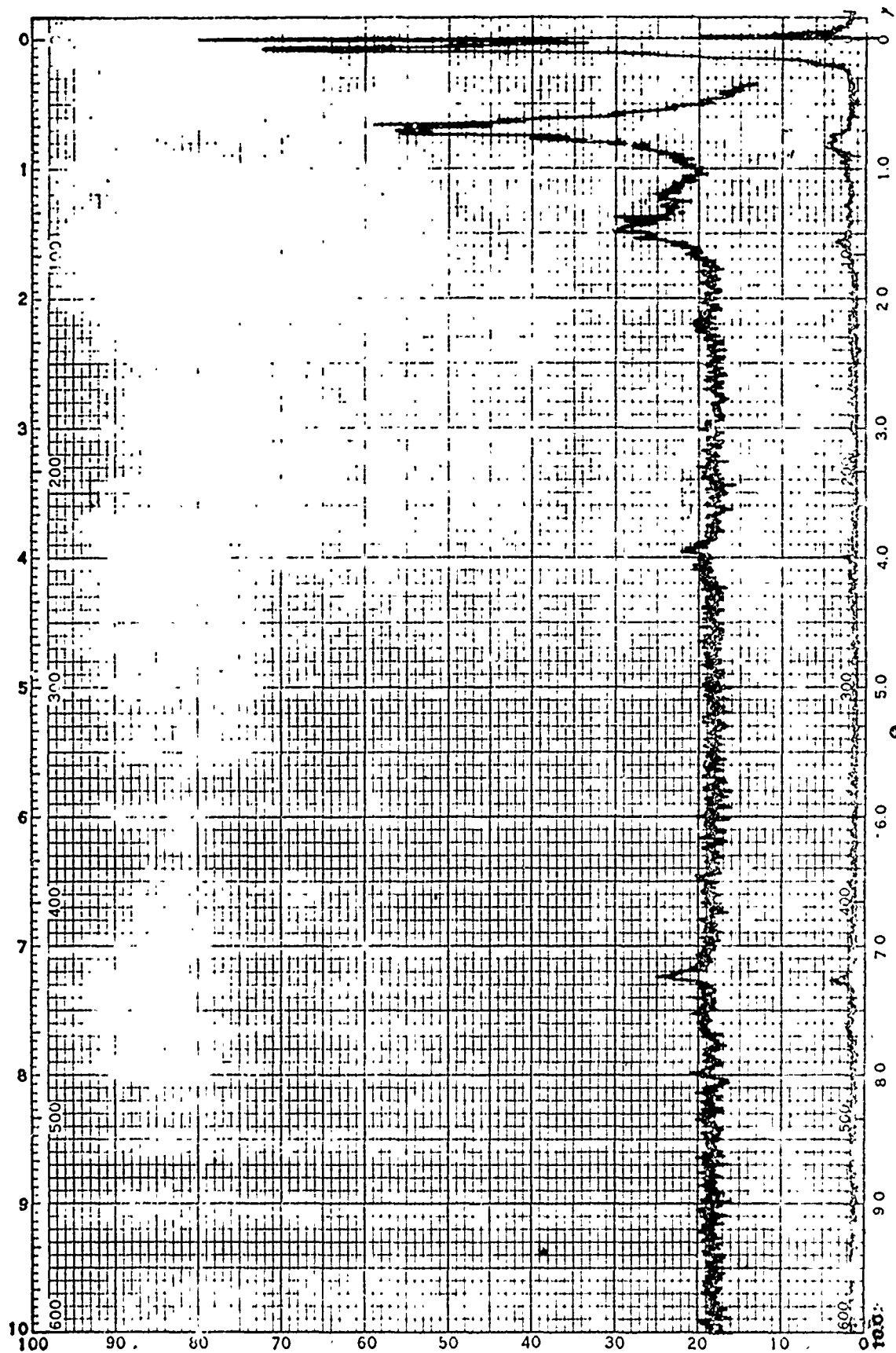
An external standard was used in these samples because of the effect a TMS internal standard would have on the integration of the methyl absorption of the silicone base polymer.



Solvent: CDCl_3

Reference: TMS

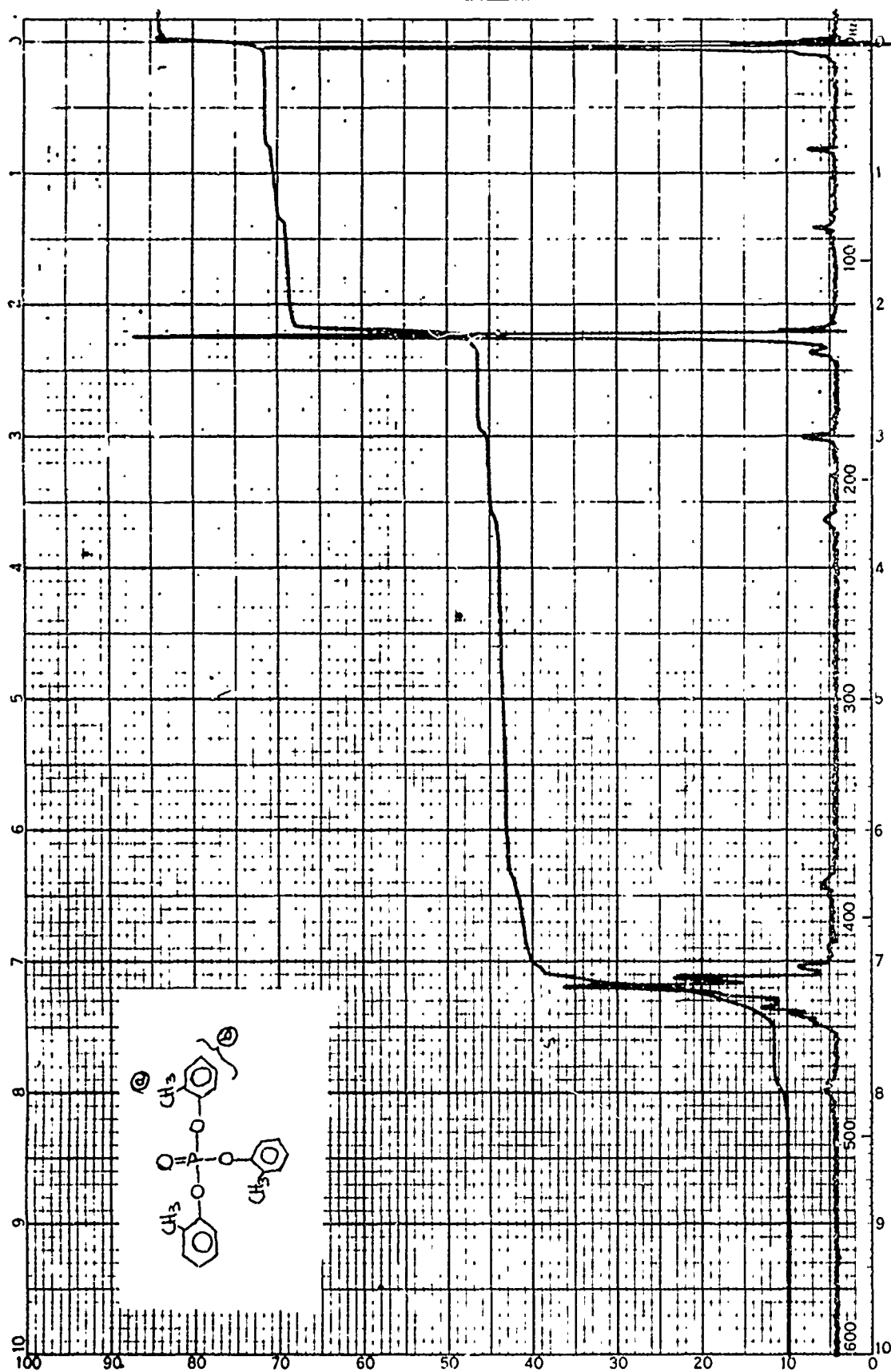
Figure 8 - NMR SPECTRUM DOW 560 SILICONE FLUID



Solvent: CDCl_3

Reference: TMS

Figure 9 - NMR SPECTRUM NADRAUL MS-5
(Normal and High Amplitude Scans)



TRI-o-CRESYL PHOSPHATE
(phosphoric acid, tri(2-tolyl)ester
--5%06 additive)

Solvent: CDCl₃

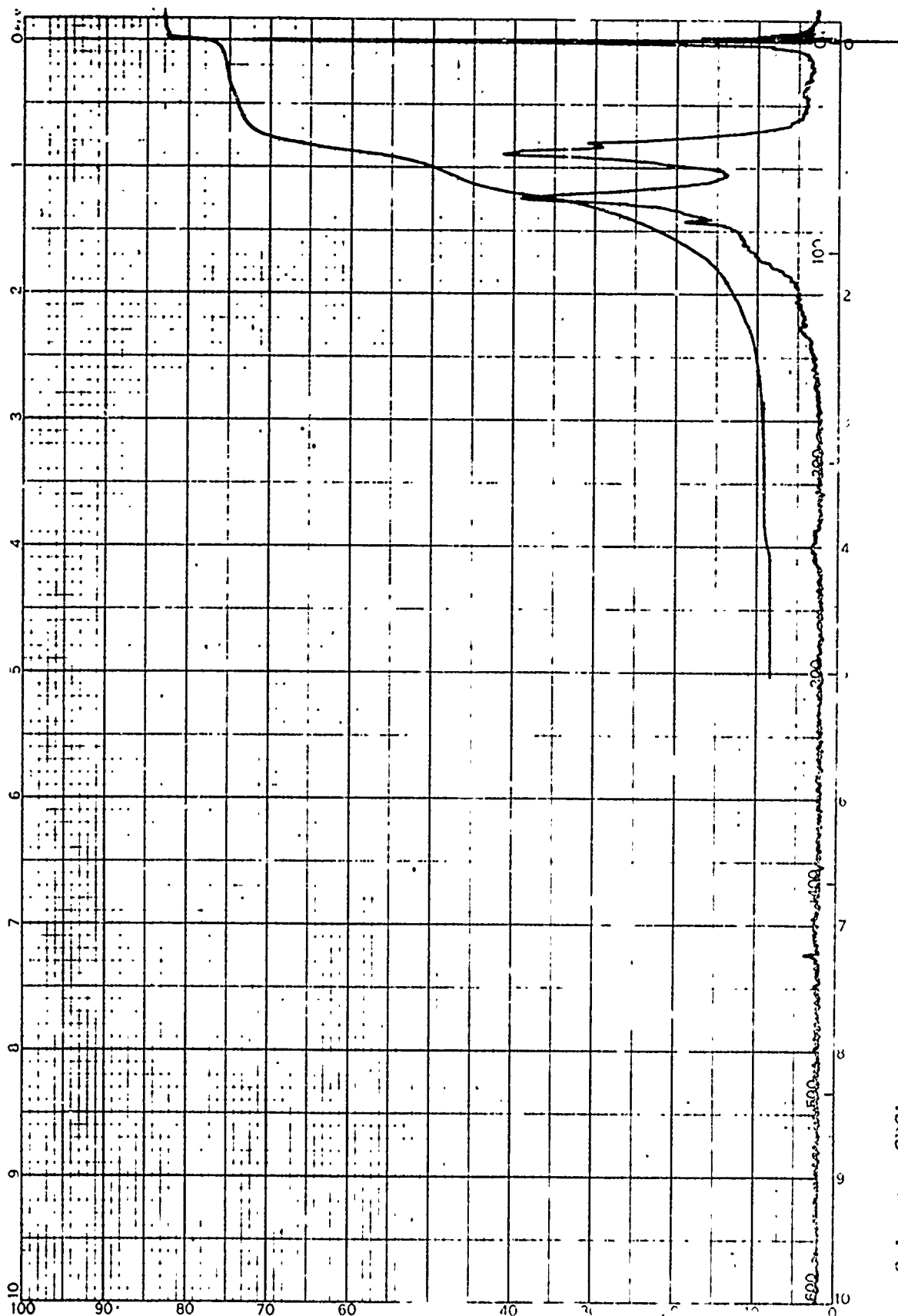
Reference: TMS

Assignments:

a. 2.2 ppm

b. 7.2 ppm

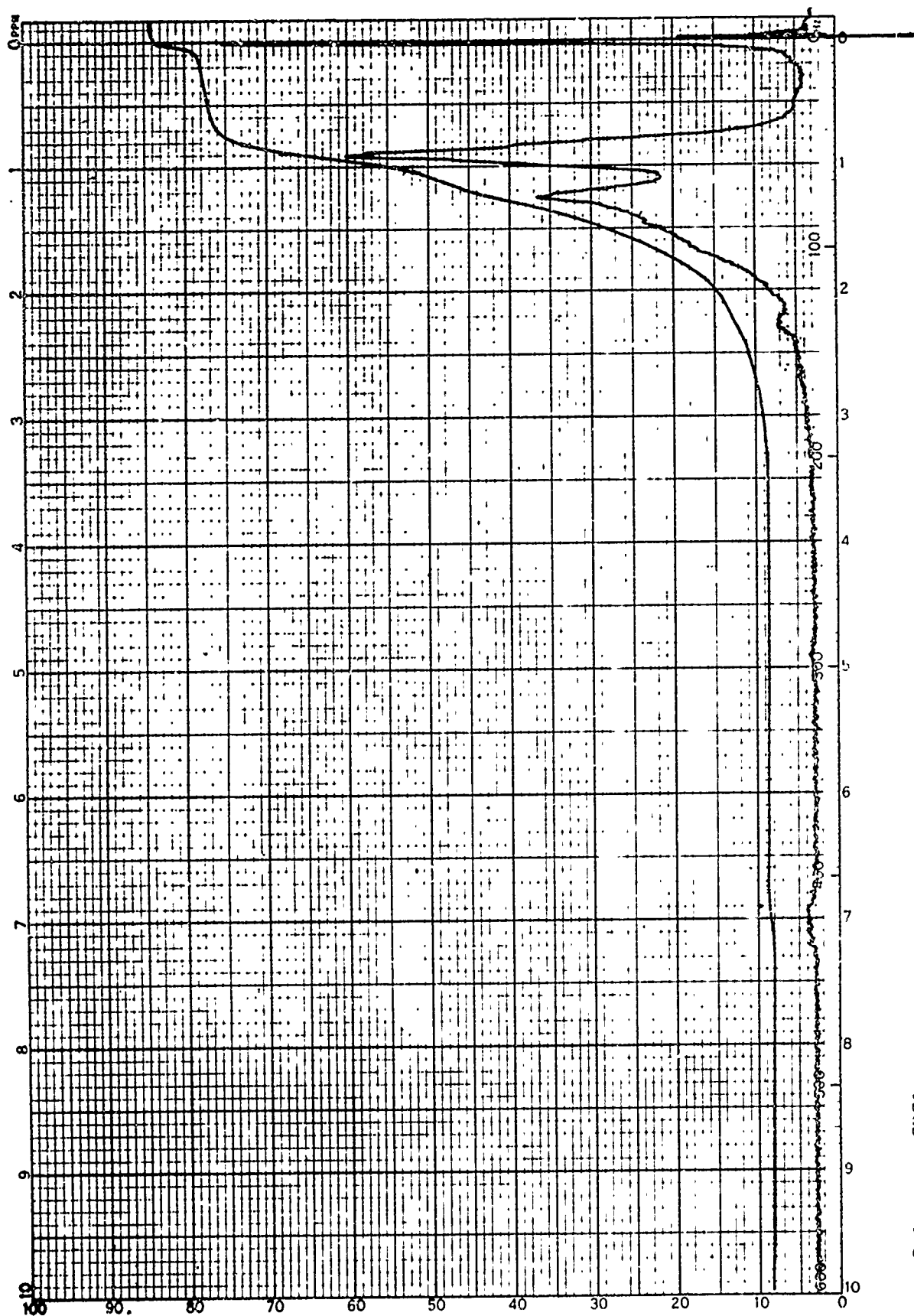
Figure 10 - NMR SPECTRUM TRI-o-CRESYL PHOSPHATE



Solvent: CDCl_3

Reference: TMS

Figure 11 - NMR SPECTRUM MIL-H-5606 HYDRAULIC FLUID



Solvent: CDCl₃

Reference: TMS

Figure 12 - NMR SPECTRUM MIL-H-5606 PETROLEUM BASE STOCK

The positions of proton resonance were the same in all spectra. Several samples were prepared of each of the four formulations. Normal and high amplitude spectra were run on each (Figures 13 and 14). The integration values were calculated for all spectra, and no significant differences could be found between any of the formulations (see Table I).

TABLE I

INTEGRATION VALUES

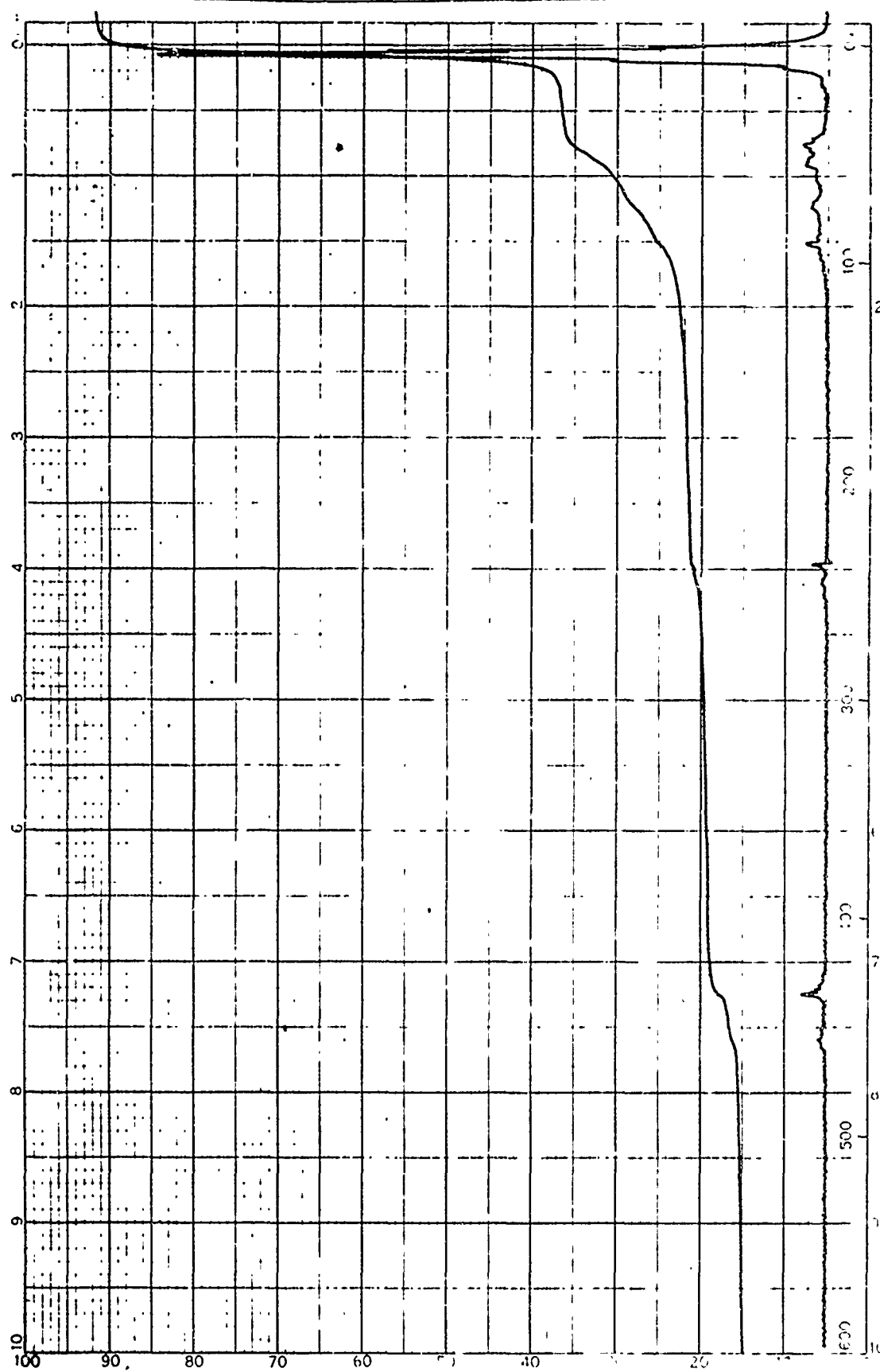
FORMULATIONS OF 5% 5606 IN MS-5

	CHEMICAL SHIFT (PPM)			
	7.2 - 7.7	4.0	0.9 - 1.6	0.1
<u>Normal Amplitude</u>				
I	4.4	1.4	18.7	75.5
II	4.0	1.6	20.4	74.0
III	4.0	1.5	18.8	75.8
IV	4.0	1.5	19.2	75.3
<u>High Amplitude</u>				
I	18.9	6.8	74.2	
II	15.4	6.2	78.5	
III	17.6	6.2	76.2	
IV	16.1	7.8	76.0	

Integration values were calculated for each chemical shift region as its percent value of the total integration for the scan. The table lists the average values determined for several samples of each formulation.

THERMAL ANALYSIS

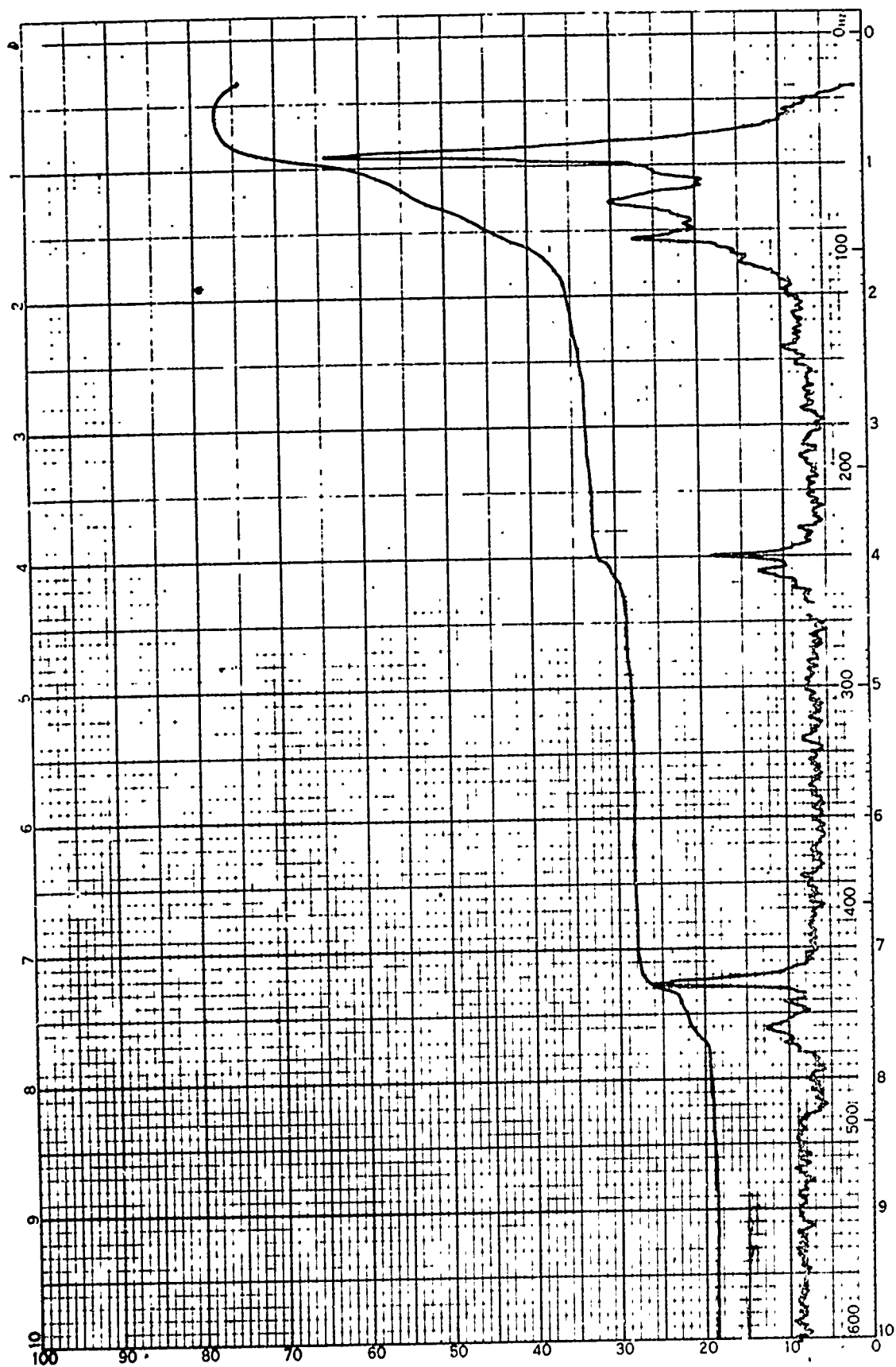
Samples of each component of the MS-5 hydraulic fluid and of the formulation MS-5 + 5% 5606 were analyzed using DTA and TGA. The results are as follows: DTA of MS-5 + 5% 5606 when heated to 200°C at 15°C/min., cooled to room temperature and repeated 5 times, gave no indication of breakdown. Figure 15 shows this graphically. MS-5 + 5% 5606 begins to give off vapor at 300°C. After 350°C it decomposes rapidly (Figure 16). This agrees with the TGA results on the same material. This is shown by a decrease in weight between 200°C and 275°C, indicating a loss of low boiling hydrocarbon material. The step from 275° to 475° indicates a complete breakdown of the compound, only a carbonaceous residue remains in the sample tube. Figures 17 and 18 illustrate the relationship between the various components of MS-5 and the breakdown temperature. Figure 19 shows the thermal properties of 5606, the component which degrades at the lowest temperature. Curve 2 of Figure 17 shows the thermal characteristics of thiadiazole (AR-155), dense fumes are given off from 225°C to 500°C, a bright yellow condensate forms on the glass dome of the cell.



Solvent: CDCl_3

(no internal standard)

Figure 13 - NMR SPECTRUM MS-5 + 5% 5606 - NORMAL AMPLITUDE



Solvent: CDCl₃

(no internal standard)

Figure 14 - NMR SPECTRUM MS-5 + 5% 5606 - HIGH AMPLITUDE

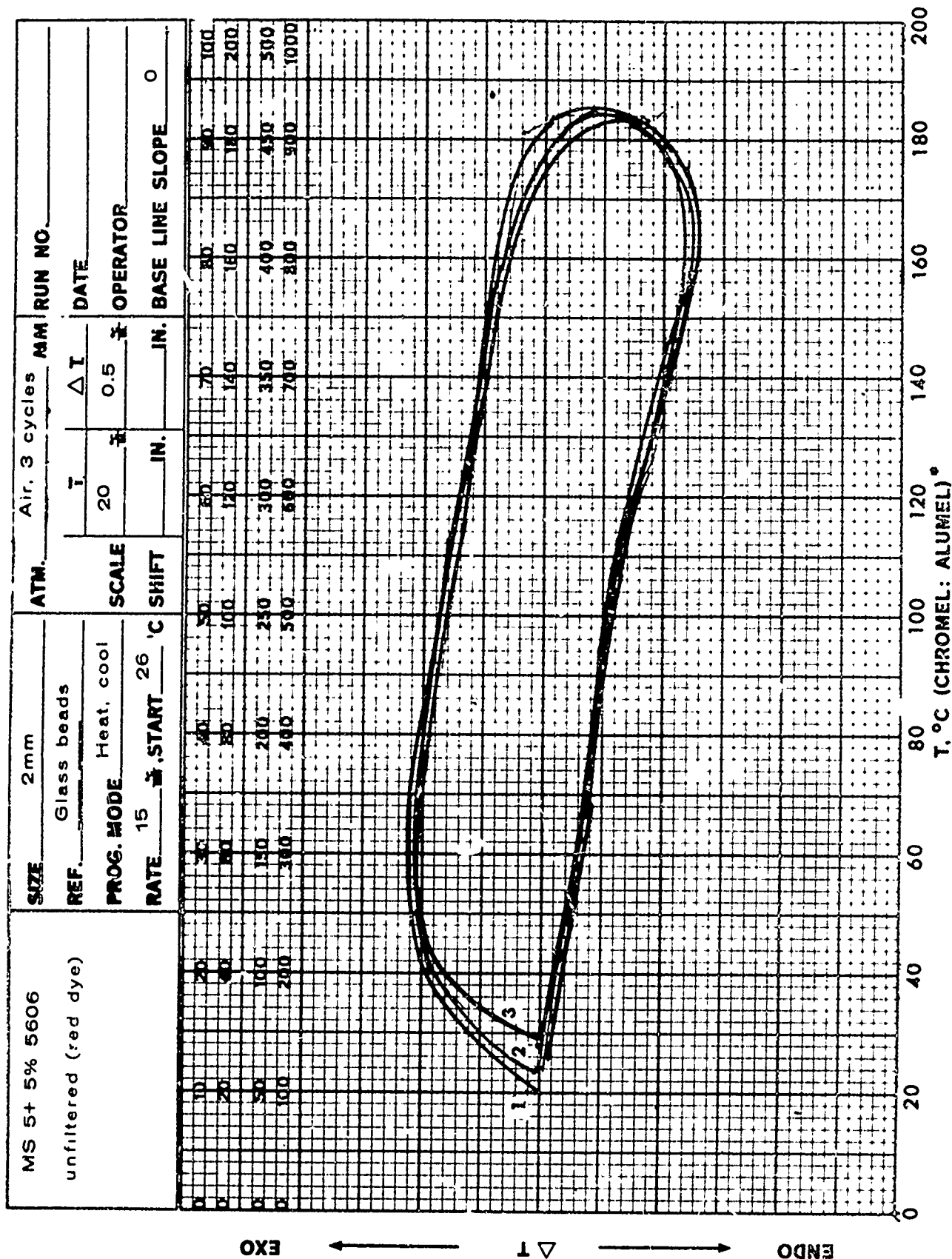


Figure 15 - DTA MS-5 + 5% 5606 UNFILTERED

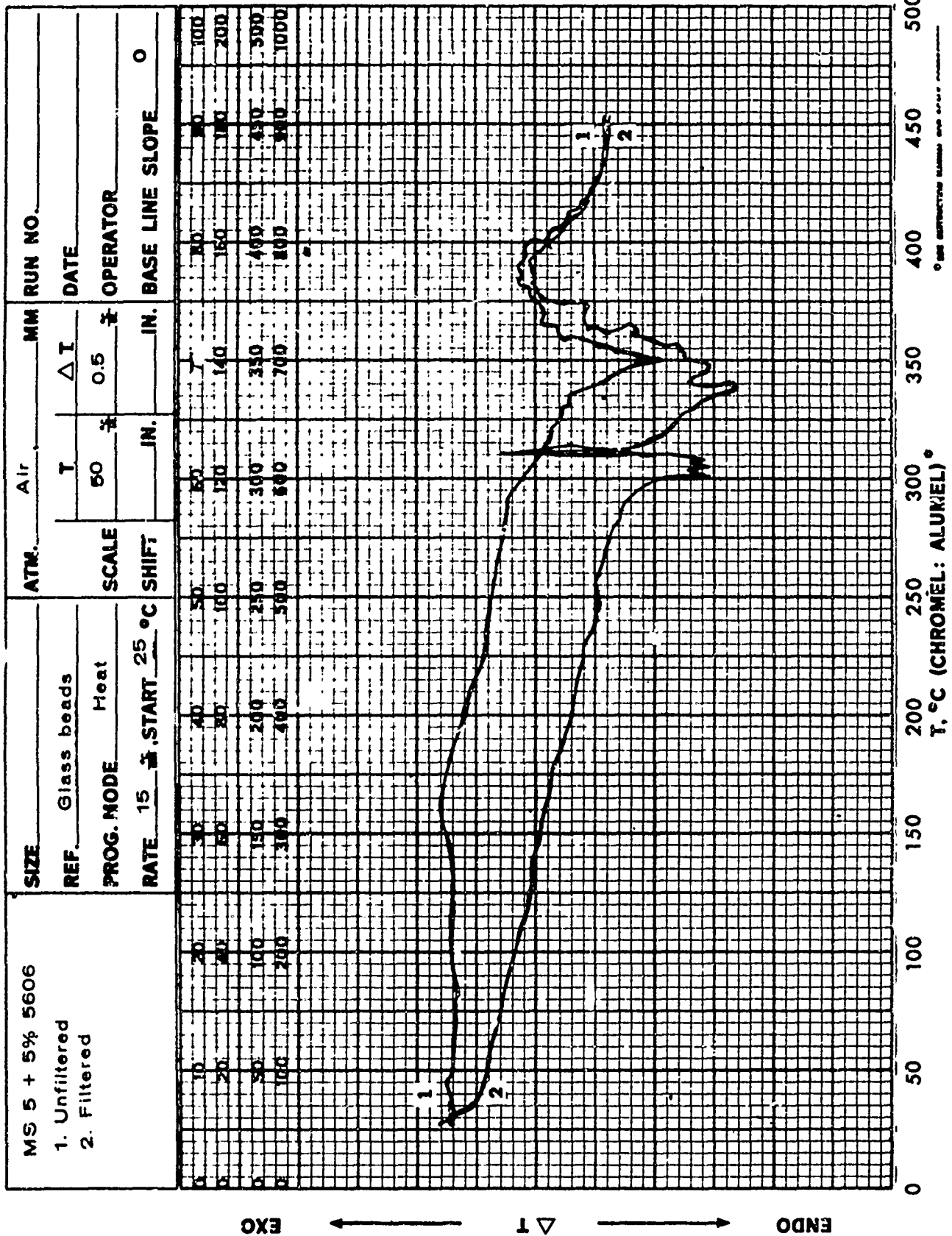


Figure 16 - DTA MS-5 + 5% 5606 UNFILTERED & FILTERED

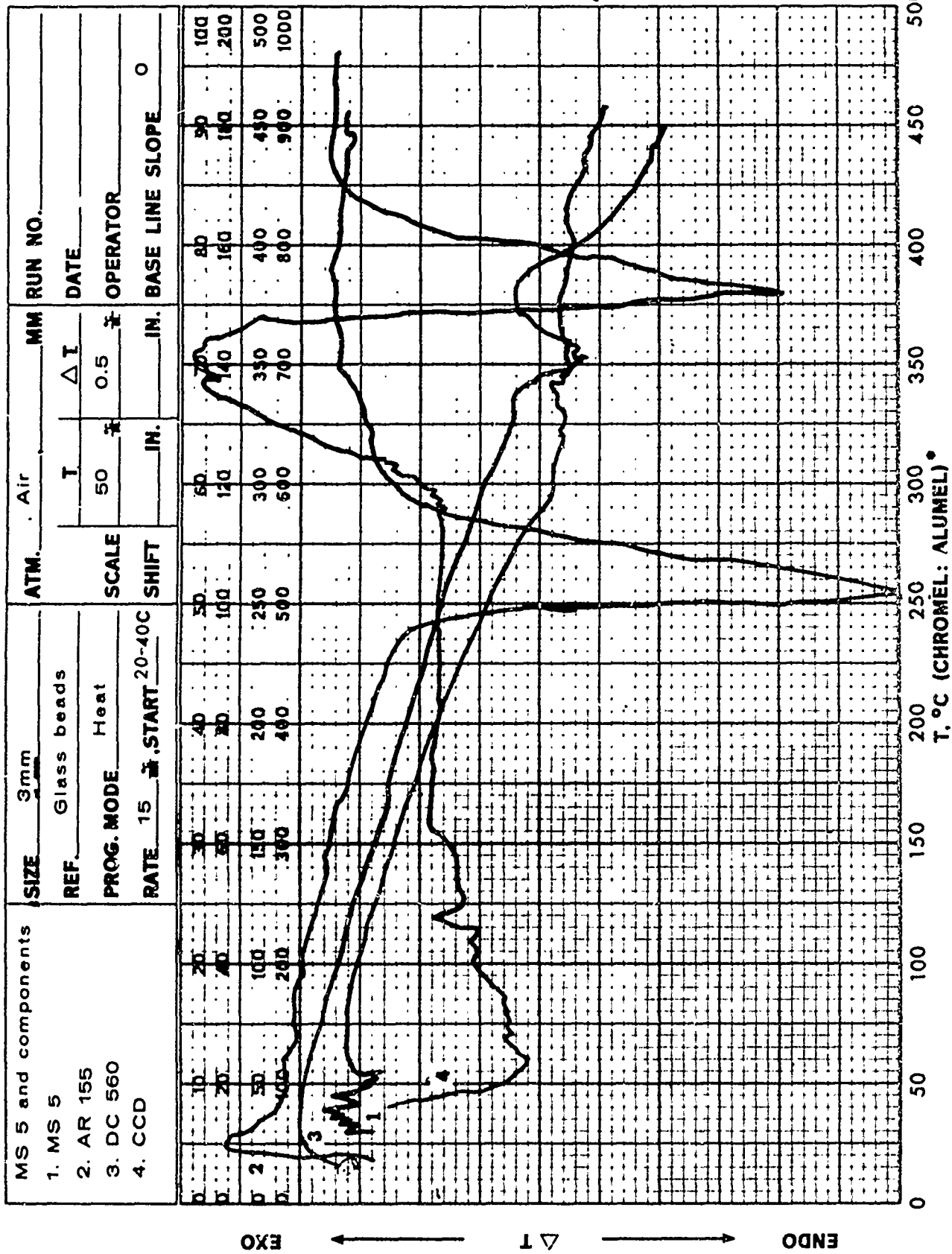


Figure 17 - DTA MS-5 AND COMPONENTS

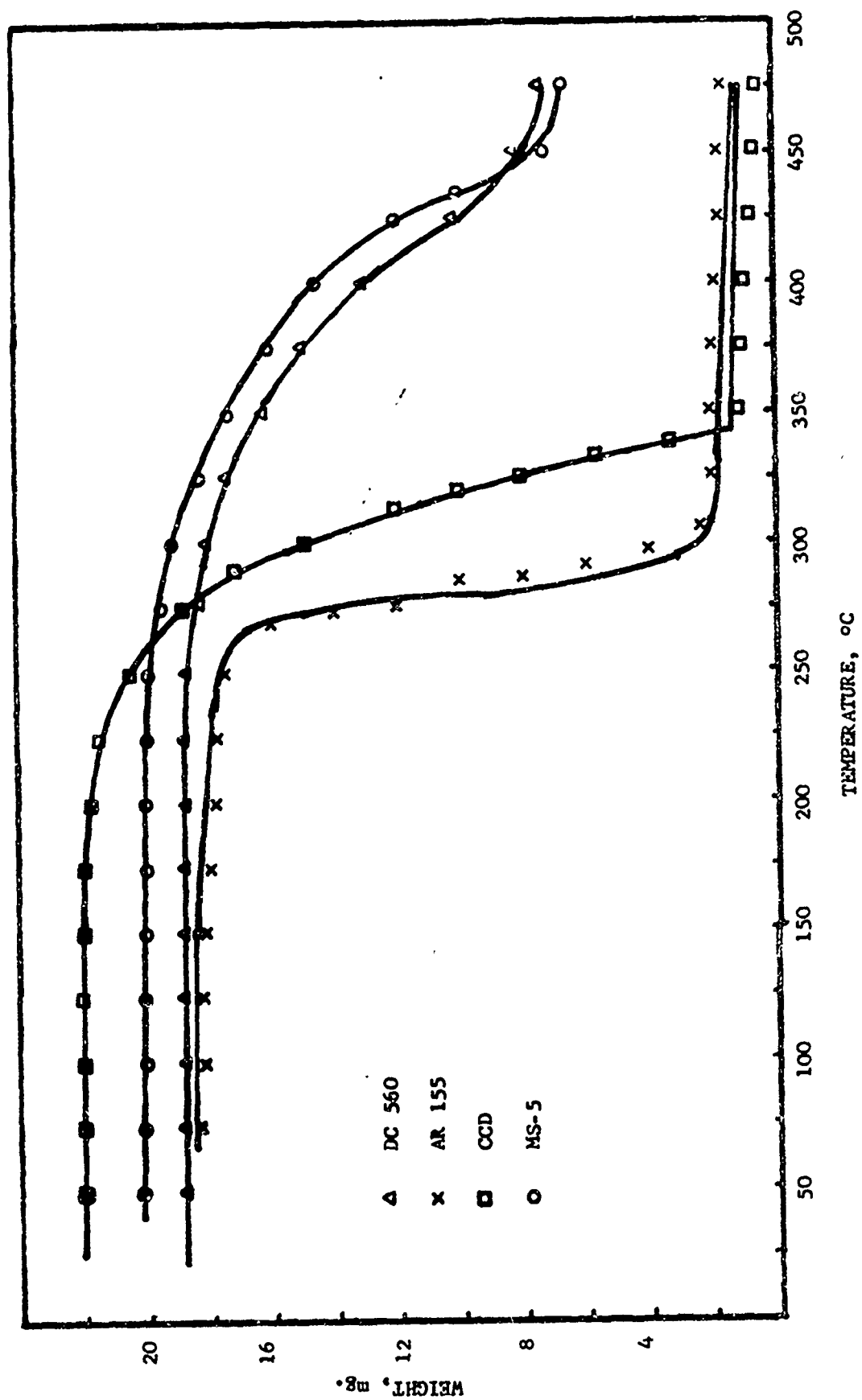


Figure 18 - TGA MS-5 AND COMPONENTS

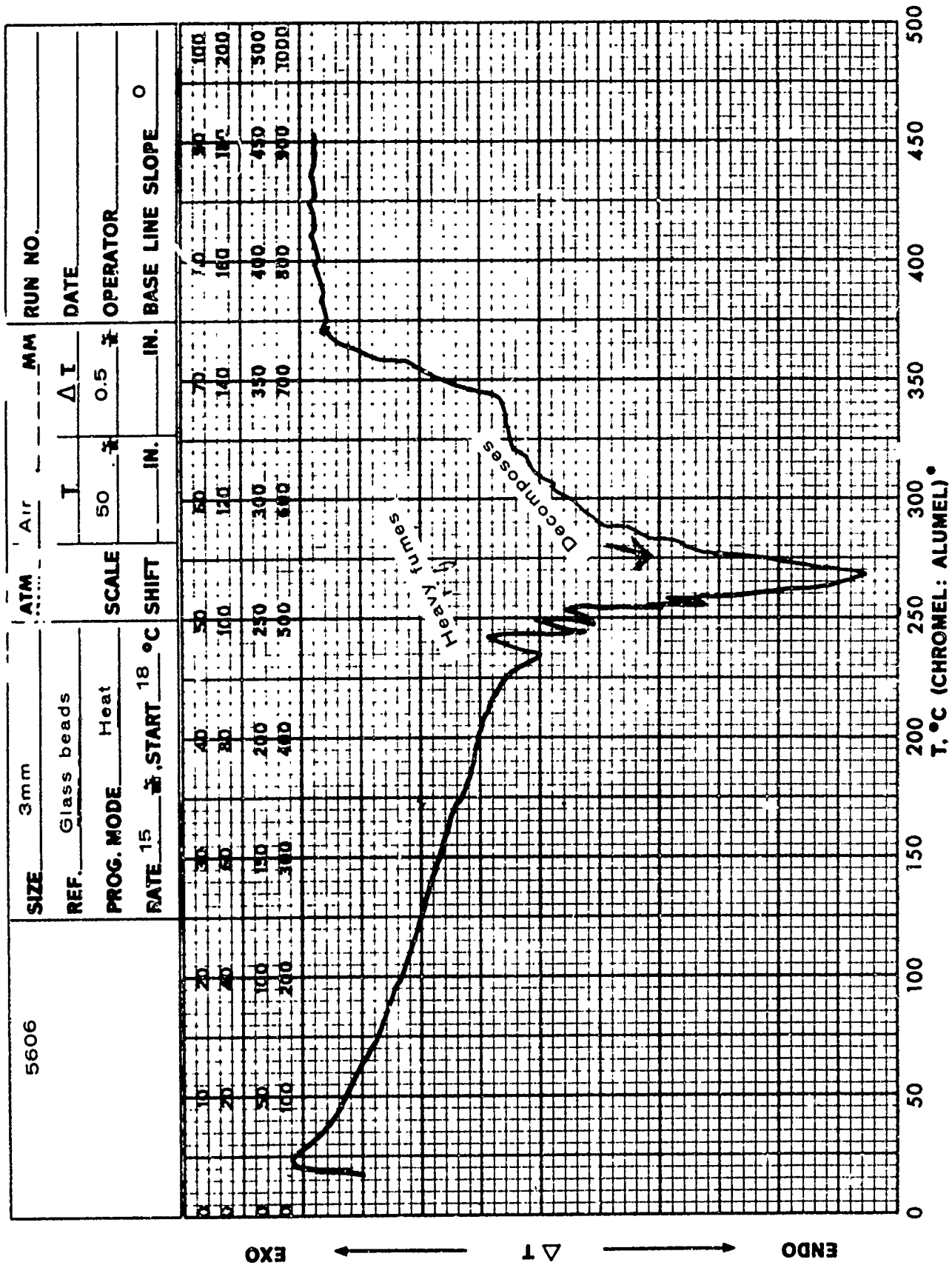


Figure 19 - DTA MIL-H-5606

Table II shows the TGA results in terms of percent weight loss over the temperature range used for each component and MS-5 + 5% 5606, filtered and unfiltered.

T A B L E I I
HYDRAULIC FLUID THERMOGRAVIMETRIC ANALYSIS

Item	TGA Percent Weight Loss over 100-500°C Range											
	100	120	140	160	180	200	250	300	350	400	450	500
5606	2	8	15.5	30	52.6	70	100					
DC 560						5		13.6		31.7		38
AR-155						2	4	88				95
CCD						1	7	33	94			95.5
MS-5						0	1	5	26	28		68
MS-5 + 5% 5606 unfiltered						2	4	9	18.6	33.7	53.3	53.5
MS-5 + 5% 5606 Filtered						2.5	2.75	10	18.75	38.75	48.75	50.00

A C K N O W L E D G E M E N T

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