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IDENTIFICATION OF SYNTHETIC FLUIDS UTILIZING
PYROLYSIS GAS LIQUID CHROMATOGRAPHIC TECHNIQUES

by

Robert G. Jamison

September 1981

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Gas chromatography	Retention time											
Synthetic Materials	Military specifications											
Base stock ingredients												
Fluid formulations												
20. ABSTRACT (Continue on reverse side if necessary and identify by block number) Because of the increasing use of synthetic-based lubricants and functional fluids, a study was initiated to develop a rapid gas liquid phase chromatographic method that would enable qualitative identification of differing types of base stock ingredients. Results of this investigation revealed that by utilizing pyrolysis gas liquid-phase chromatographic techniques, identification of many different types of materials can be successfully accomplished in a relatively short period of time. Utilization of this technology will greatly enhance the resolution of field problems and permit a greater understanding of fluid and lubricant research.												

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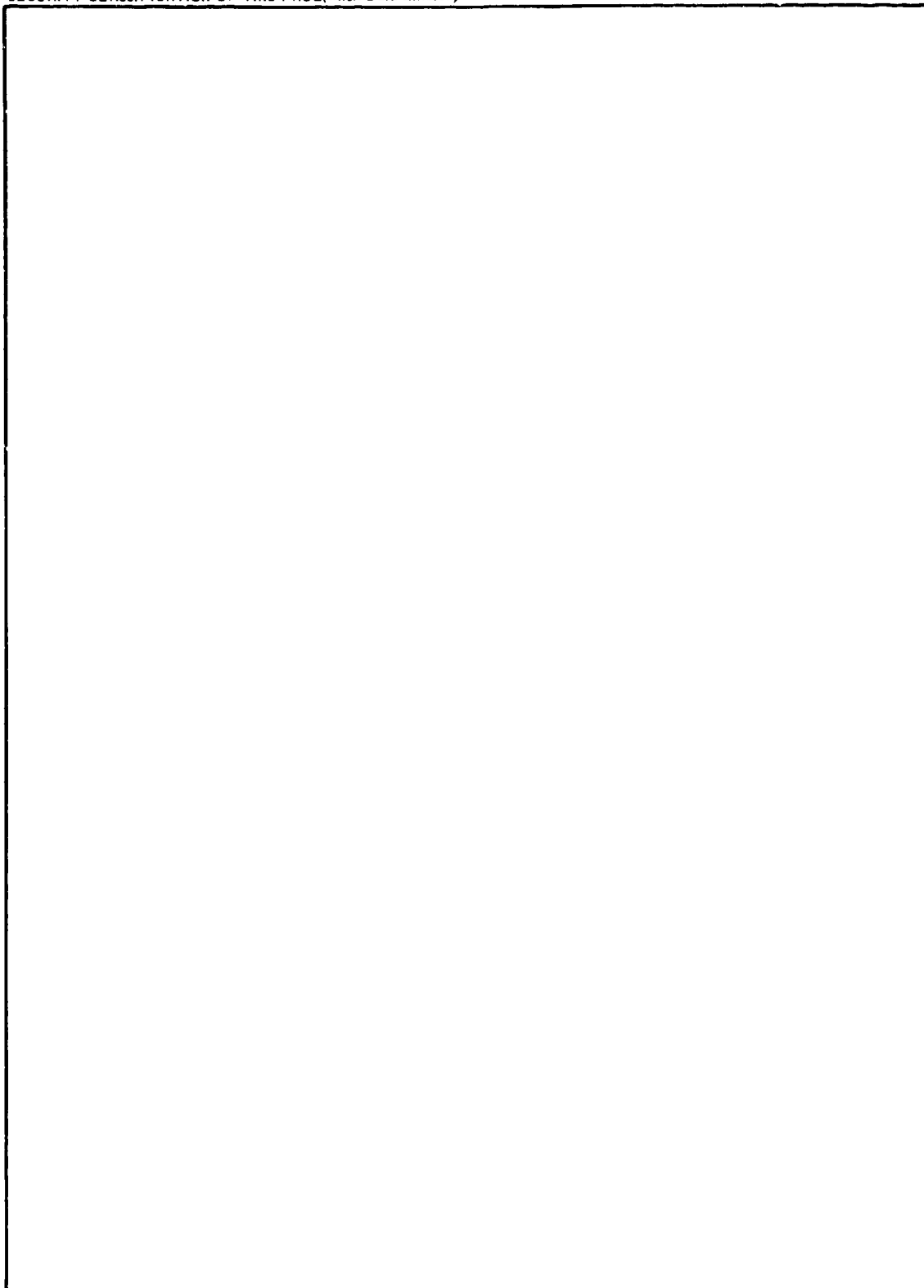
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PREFACE

Mr. James Conley of the Hydraulic Fluids and Corrosion Preventative Branch, Fuels and Lubricants Division, assisted in the application of special techniques for identifying different types of base stock ingredients of synthetic compounds.

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IDENTIFICATION OF SYNTHETIC FLUIDS UTILIZING PYROLYSIS

GAS LIQUID PHASE CHROMATOGRAPHIC TECHNIQUES

I. INTRODUCTION

Historically, lubricants and functional fluids intended for use in ground equipment have been formulated with petroleum basestock materials. These petroleum oils have been utilized because of their preferred use and economics which have maintained their dominance in this applications area. Beginning with the energy crises of 1973, which created a reduction in the import of foreign crude supplies, the emphasis has shifted toward the use of synthetically derived base materials.¹ These new synthetically manufactured materials are starting to lessen the demand for petroleum basestocks to a small degree. At the same time, these new materials are beginning to overcome many of the disadvantages inherent in petroleum oils and fluids such as refining cost, low availability, limited high-temperature applications, and enhanced viscosity temperature characteristics. For example, the synthesized hydrocarbons (commonly referred to as olefin oligomers) are compounds having predictable structures and properties that are not dependent upon the same crude or refinement technology to produce a similar product.

Currently, numerous specification products exist which either because of a compositional or a performance requirement, specify a synthetic base fluid/oil in lieu of the conventional petroleum basestock. To illustrate this point, Table 1 provides a listing of different specifications and the types of synthetic materials now being used. As noted, the four synthetic materials that are being used extensively are olefin oligomers, alkylated aromatics, di-basic acid esters, and polyol/polyesters. A sampling of these different basestocks is shown in Table 2. These synthetic materials offer high temperature stability, high viscosity index (i.e., little change in viscosity with temperature changes), improved antiwear properties, low volatility, enhanced fire retardancy, and excellent low temperature fluidity and are generally compatible with those products formulated with petroleum basestocks.

Synthetic lubricants have been used in limited applications for ground equipment over the past 10 to 15 years, but there has been no concentrated effort to identify the various base materials used. There have been many analytical methods developed^{2 3 4}

¹ Stavinoha, L. L.; Fodor, G. E.; Newman, F. M.; and Lestz, S. J.; Analytical Characterization of Synthetic Lubricants, American Society of Lubrication Engineers, No. 77 AM-4A-1, May 1977.

² Hamilton & Sewell, Introduction to High Performance Liquid Chromatography, Chapman and Hall, London, 1977.

³ Smith, A. Lee; Chemical Analysis; Vol 41; Wiley Interscience; 1974.

⁴ Hillman, D. E.; Paper presented at the 1977 International Symposium on Liquid Chromatographic Analysis of Polymers and Related Materials; Chicago; October 1977.

Table 1. Military Specifications Utilizing Synthetic Materials

Military Specification	Title	Base Stock Ingredients
MIL-L-7808H	Lubricating Oil, Aircraft Turbine Engine, Synthetic Base	Di-tridecyl Adipate
MIL-L-46167A	Lubricating Oil, Internal Combustion Engine, Arctic	Polyalkylated Benzene
MIL-L-46170A	Hydraulic Fluid, Rust Inhibited Fire-Resistant, Synthetic Hydrocarbon Base	Polyalpha Olefin
MIL-L-23699C	Lubricating Oil, Aircraft Turbine Engine, Synthetic Base	Triheptanoate
MIL-L-46152B	Lubricating Oil, Internal Combustion Engine, Administrative Service	Di-isodecyl Azelate
MIL-L-46000B	Lubricant, Semi-Fluid (Automatic weapons)	Di-2-ethylhexyl Sebacate
MIL-L-83282A	Hydraulic Fluid, Fire-Resistant Synthetic Hydrocarbon, Aircraft	Polyalpha Olefin
MIL-L-11734C	Lubricating Oil, Synthetic (For mechanical time fuses)	Di-2-ethylhexyl Sebacate
MIL-H-19457B	Hydraulic Fluid, Fire-Resistant Type 1, Type 2	Phosphate Ester
MIL-L-27502	Lubricating Oil, Aircraft Turbine Engine, Ester Base	Polyol Ester

Table 2. Synthetic Base Stock Fluids

Di-isooctyl Azelate
Di-2-Ethylhexyl Azelate
Di-isodecyl Azelate
Di-2-Ethylhexyl Adipate
Di-Tridecyl Azelate
Di-Tridecyl Adipate
Di-2-ethylhexyl Sebacate
Di-isodecyl Adipate
TMP Triheptanoate
Neopentyl dipelargonate
Polyalpha Olefin, 6cSt Synfluid
Polyalpha Olefin, 4cSt Synfluid

to characterize a particular type of material. In all these procedures, the techniques are considerably manpower intensive and, in some instances, require extremely advanced instrumented analysis techniques as shown in Figure 1. Because of the increasing products now being supplied to the Army and other DOD users, a need has surfaced to have a capability that would enable rapid determination and/or characterization of products in question. The problems associated with identification of synthetic basestock and conventional petroleum materials are usually related to inability of separating these high molecular weight polymers and extremely complex mixtures. With the success of pyrolytic gas liquid phase chromatography (PGC) for determining the types of elastomers found in wheel braking systems⁵ firmly established, this approach was considered for characterization and defining the types of synthetic basestocks.

Pyrolytic gas chromatography offers a convenient approach to the analysis of macromolecules/polymers without the necessity of chemical pretreatment. The synthetic materials are thermally decomposed using a heated filament and decomposition products are directed to the Gas Chromatograph (GC) where separation of species will occur by standard techniques. In the subsequent experimentation, the thermal decomposition products of synthetic materials give a definitive characterization using flame ionization detector (FID). The retentive time data and peak area percent were used to correlate peak similarities.

II. DETAILS OF TEST

All tests were conducted on a programmable Hewlett Packard 5830A Gas Chromatograph with a pyrolysis unit (Pyroprobe). The column selected for the purpose of this study consisted of 5.48-m (18-ft) by 3-mm (1/8-in.) stainless steel tubing packed with 20% silicone grease SE30 on Chromosorb W, High-Performance, 80-100 Mesh.

The operating conditions used were as follows:

Flame Ionization Detector	-	250° C
Temperature 1	-	100° C
Rate/min	-	8° C
Temperature 2	-	330° C
Time @ Temperature 2	-	14 min
Aux. Temperature	-	200° C
Chart Speed	-	0.5 in./min

⁵ Jamison, R. G. and Exposito, G. E.; Characterization of Elastomeric Materials used in Automotive Brake and Cooling Systems by Pyrolytic Gas Chromatography; CCT No. 309, Aberdeen Proving Ground; July 1972.

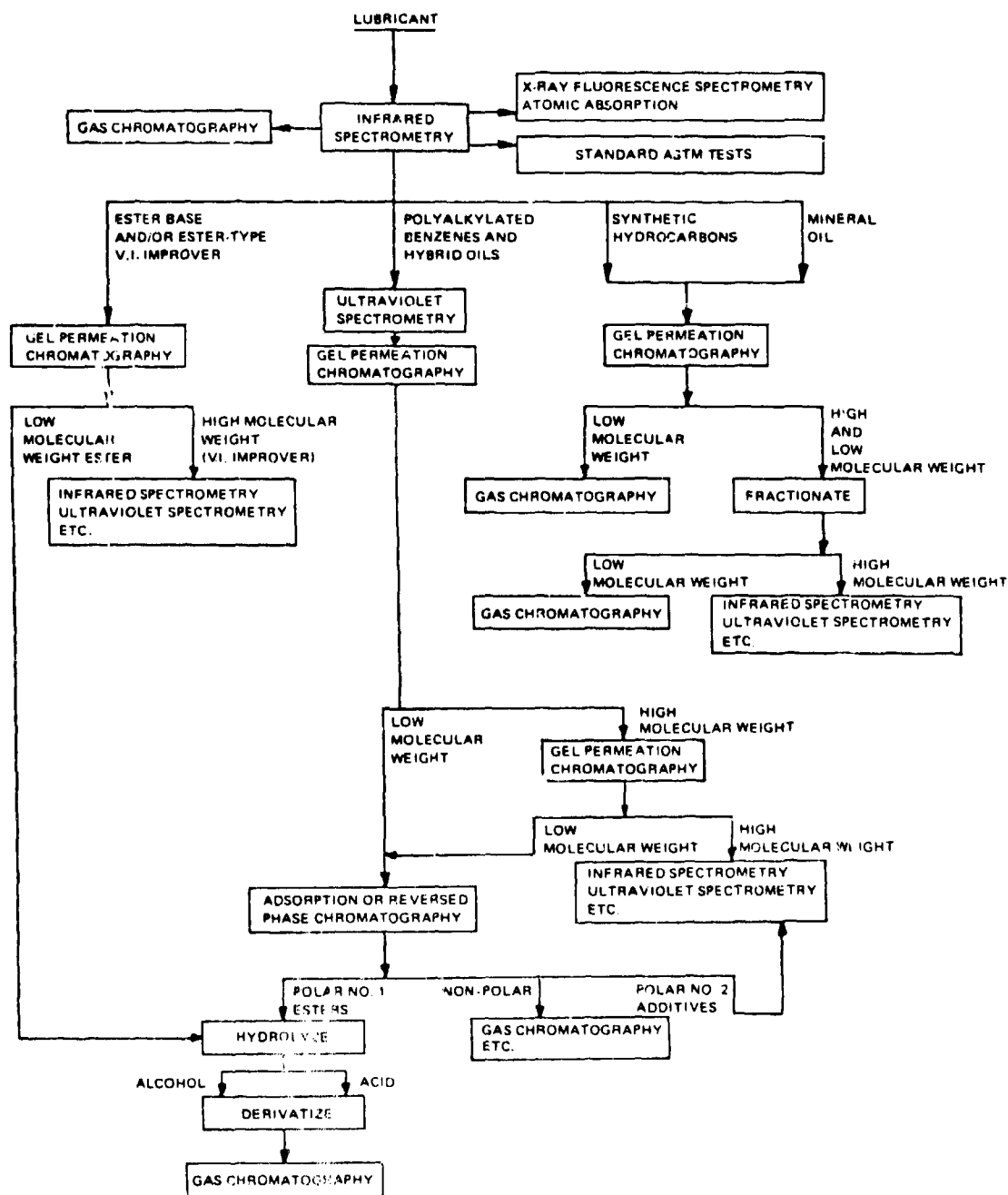


Figure 1. Analytical approach to the characterization of lubricants.

Pyroprobe conditions were as follows:

Interface Temperature	—	200° C
Interval	—	10 s
Final Temperature	—	850° C
Element Type	—	Ribbon

A 1- μ l sample of the fluid is placed on the ribbon of the Pyroprobe. The probe is inserted into the microoven (interface) injection part. The automated pyrolysis cycle and temperature programming mechanism are engaged and the chromatograms are developed.

III. RESULTS OF TEST

The 14 synthetic base materials that were analyzed by this method are shown in Table 2 and the response pattern for each material is presented in Figures 2 through 15. Examination of the 14 pyrograms clearly shows each one has a unique pattern. Comparison of the pyrograms of Di-2-ethylhexyl adipate (Figure 8), Di-2-ethylhexyl azelate (Figure 7), and Di-2-ethylhexyl sebacate (Figure 9) shows the adipate to have a distinguishing peak at 13 min, which does not appear in any of the others. The azelate and adipate polymers both show peaks in the 27- to 32-min elution time frame, but the peak height of the adipate is considerably greater than that of the azelate. The peak heights of the azelate is greater than the adipate in the 18- to 23-min time frame.

Comparison of the pyrograms of di-isodecyl adipate (Figure 5) and di-isodecyl azelate (Figure 6) show the azelate has a peak of 22 min and a large peak height at 22 to 30 min.

Di-Tridecyl adipate (Figure 11) and di-tridecyl azelate (Figure 10) differ considerably in the 15- to 23-minute time frame and are easily distinguishable.

The polyalpha olefin (Figure 4), polyalkylated benzene (Figure 5), and silicone brake fluids (Figures 25 and 26) are completely different over the entire pyrogram.

The di-ester (Figure 3) and polyol ester (Figure 2) differ in the 3- to 7-min and 18- to 20-min range.

Tables 3 and 4 show the various synthetic products that are currently being used in military product specification by the Army. An assessment of the pyrograms from the basestock samples as compared with those pyrograms produced from formulated Qualified Product Lists (QPL) (Figures 16 through 24) samples reveal striking similarities; for example, PGC was also applied to the analysis of two silicone brake fluids.

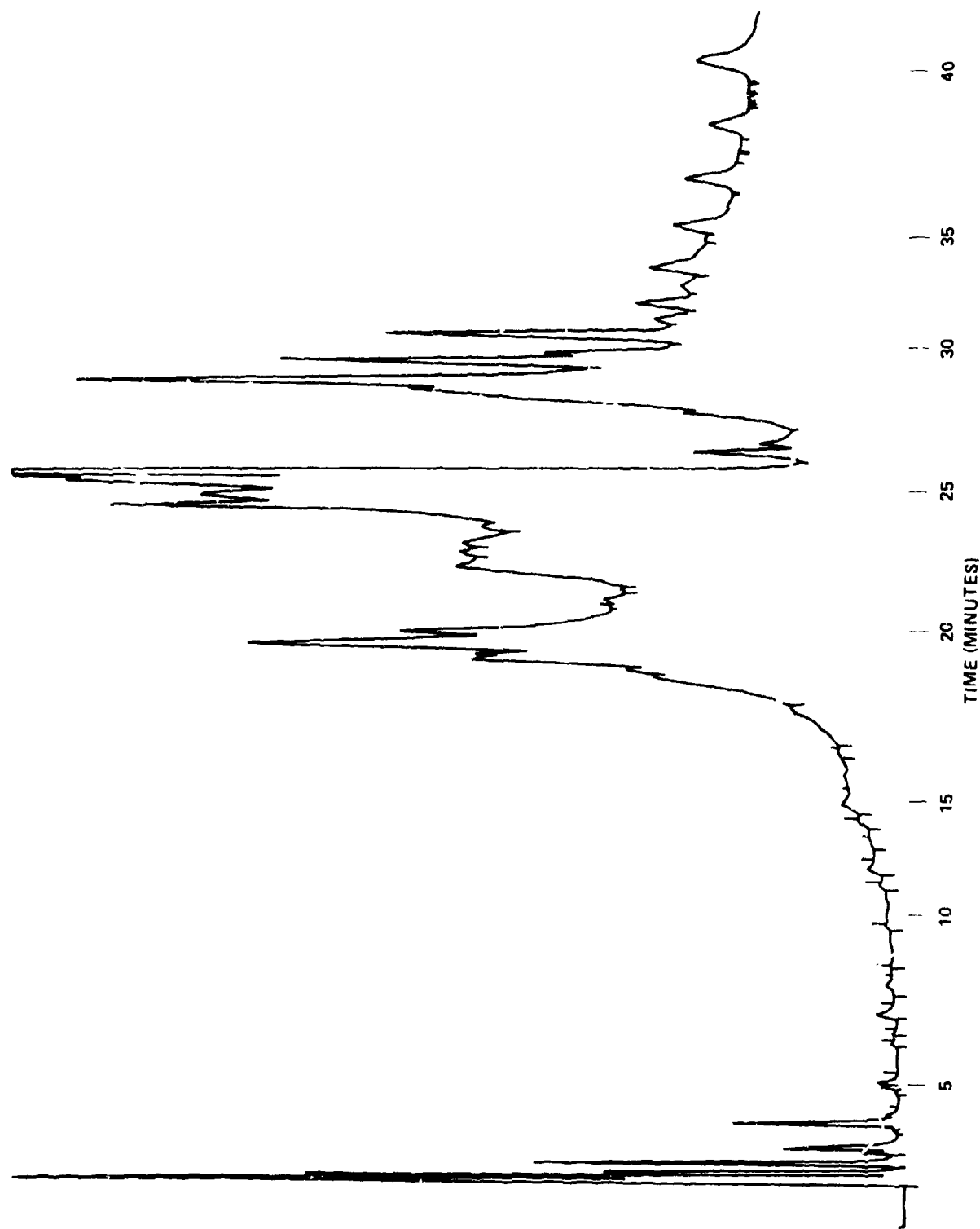


Figure 2. Synthetic base stock No. 7780 Polyol Ester.

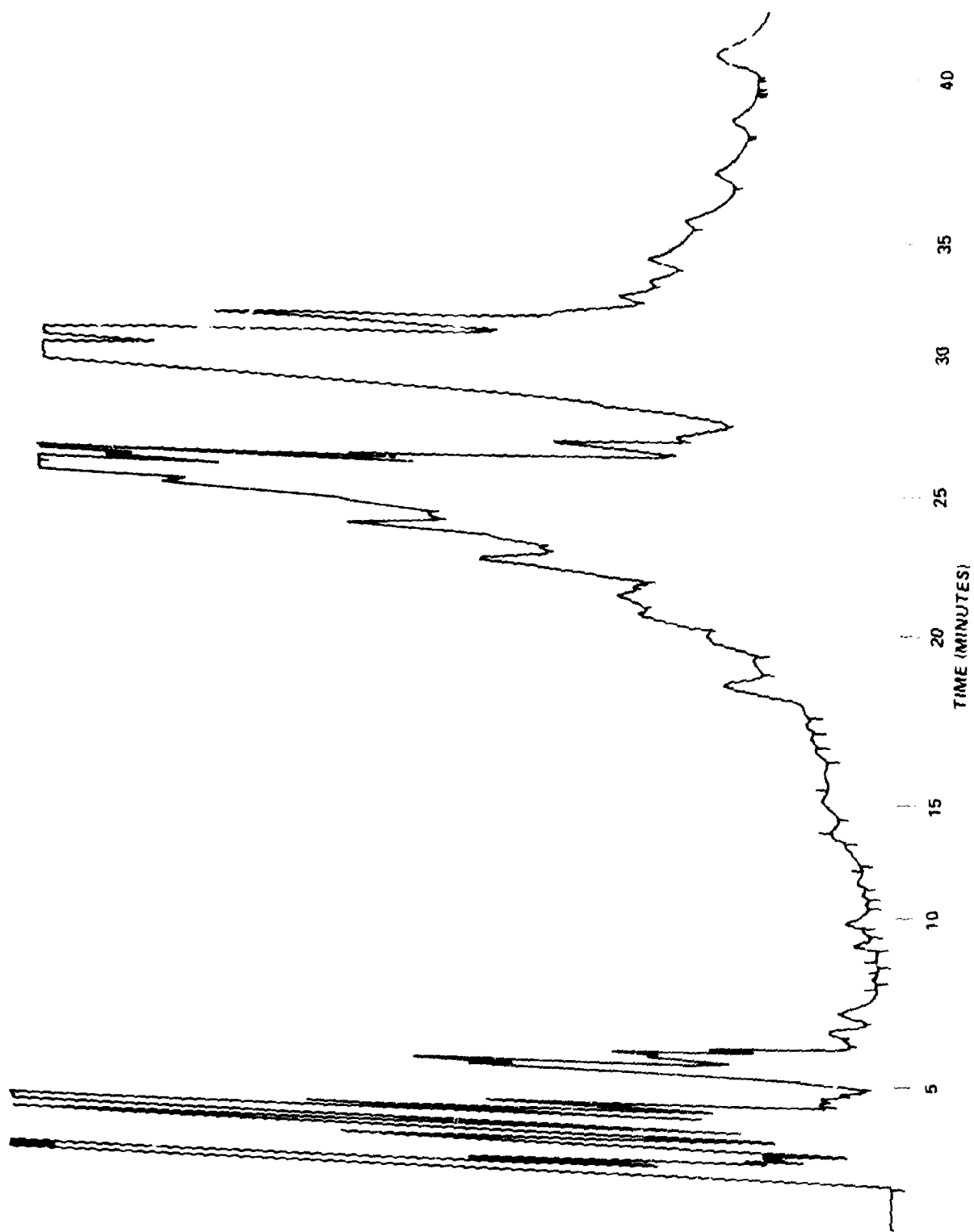


Figure 3. Synthetic base stock No. 7787 Diester.

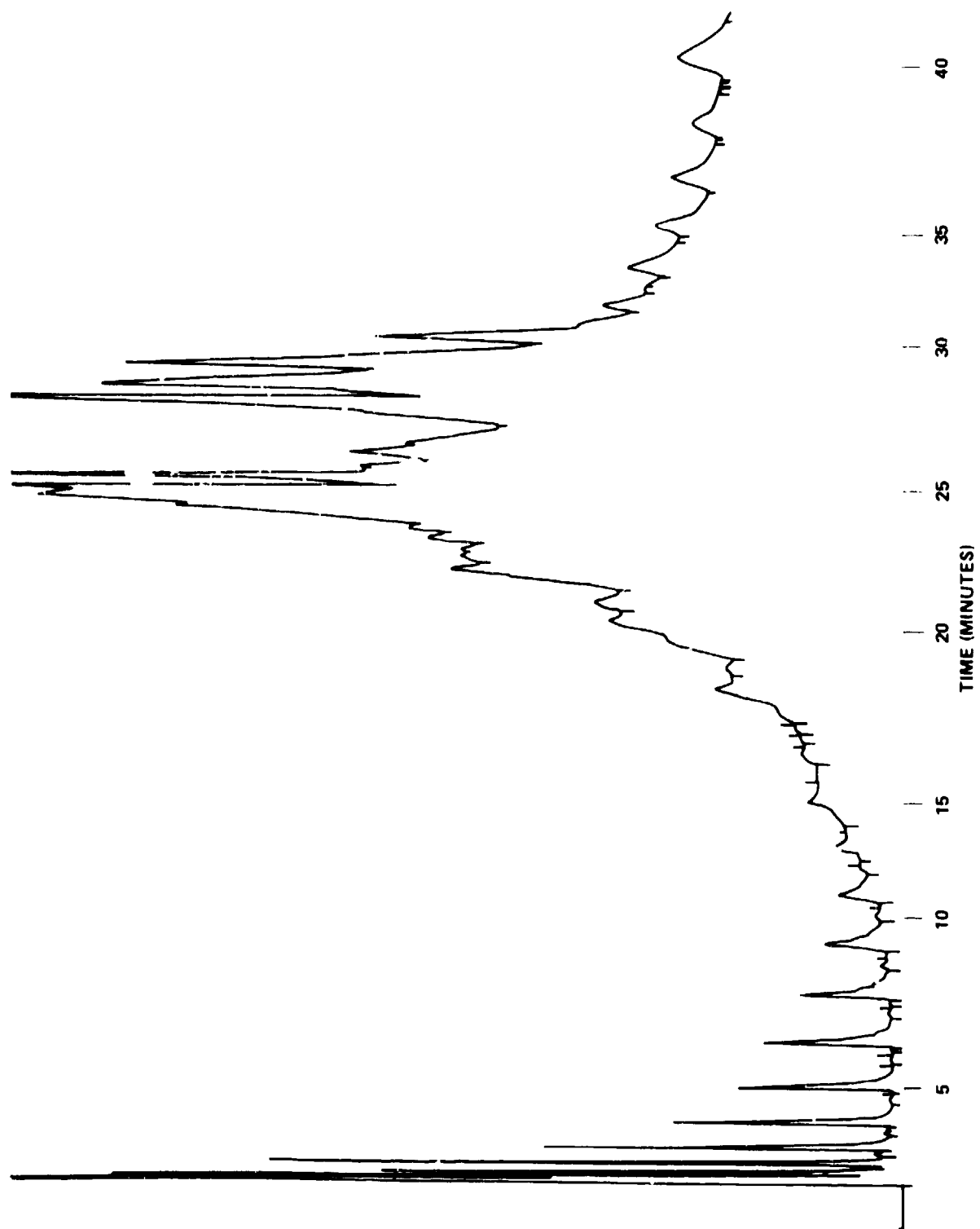


Figure 4. Synthetic base stock No. 7302 Polyalpha Olefin.

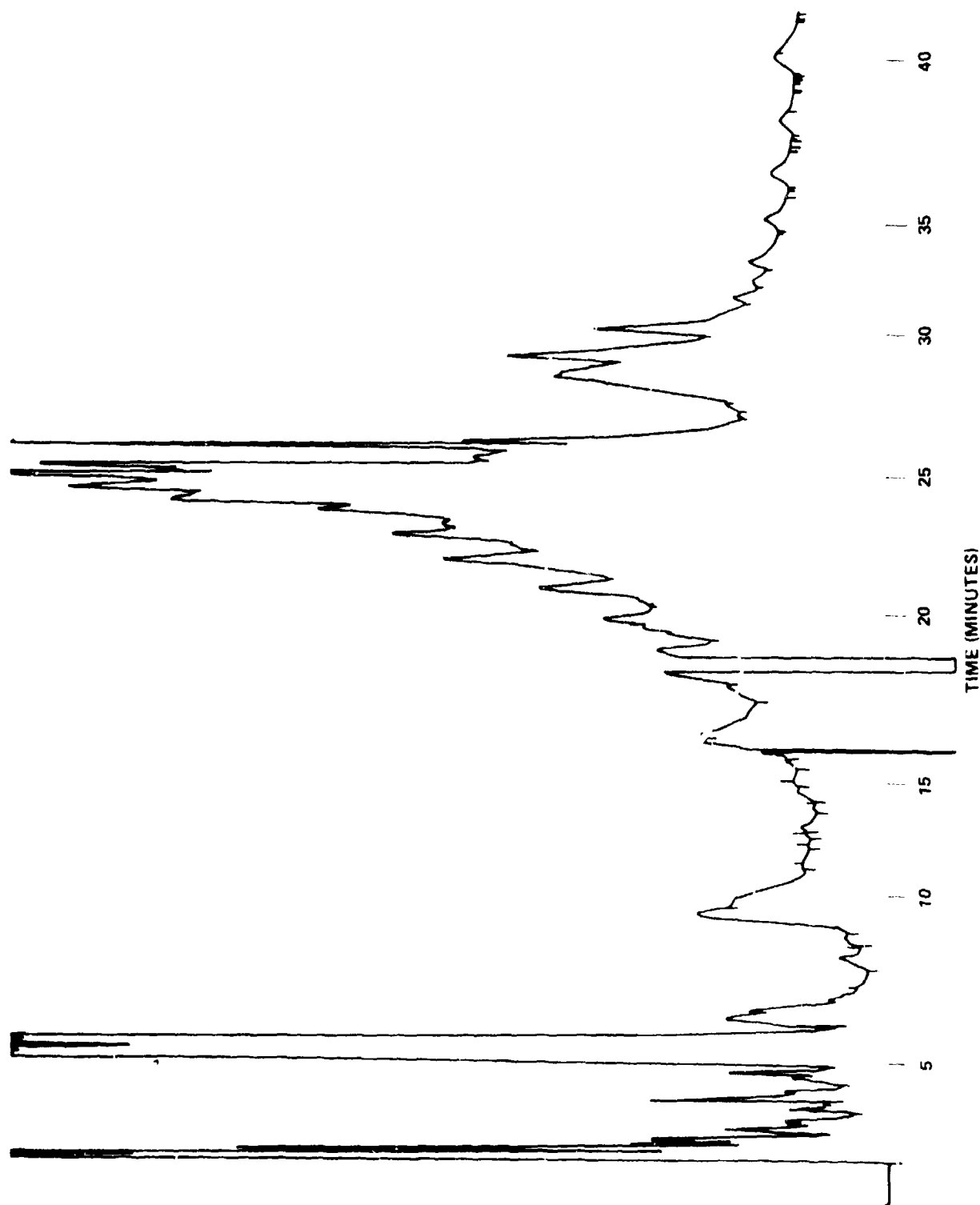


Figure 5. Synthetic base peak No. 6325 Di-Isodecyl Adipate.

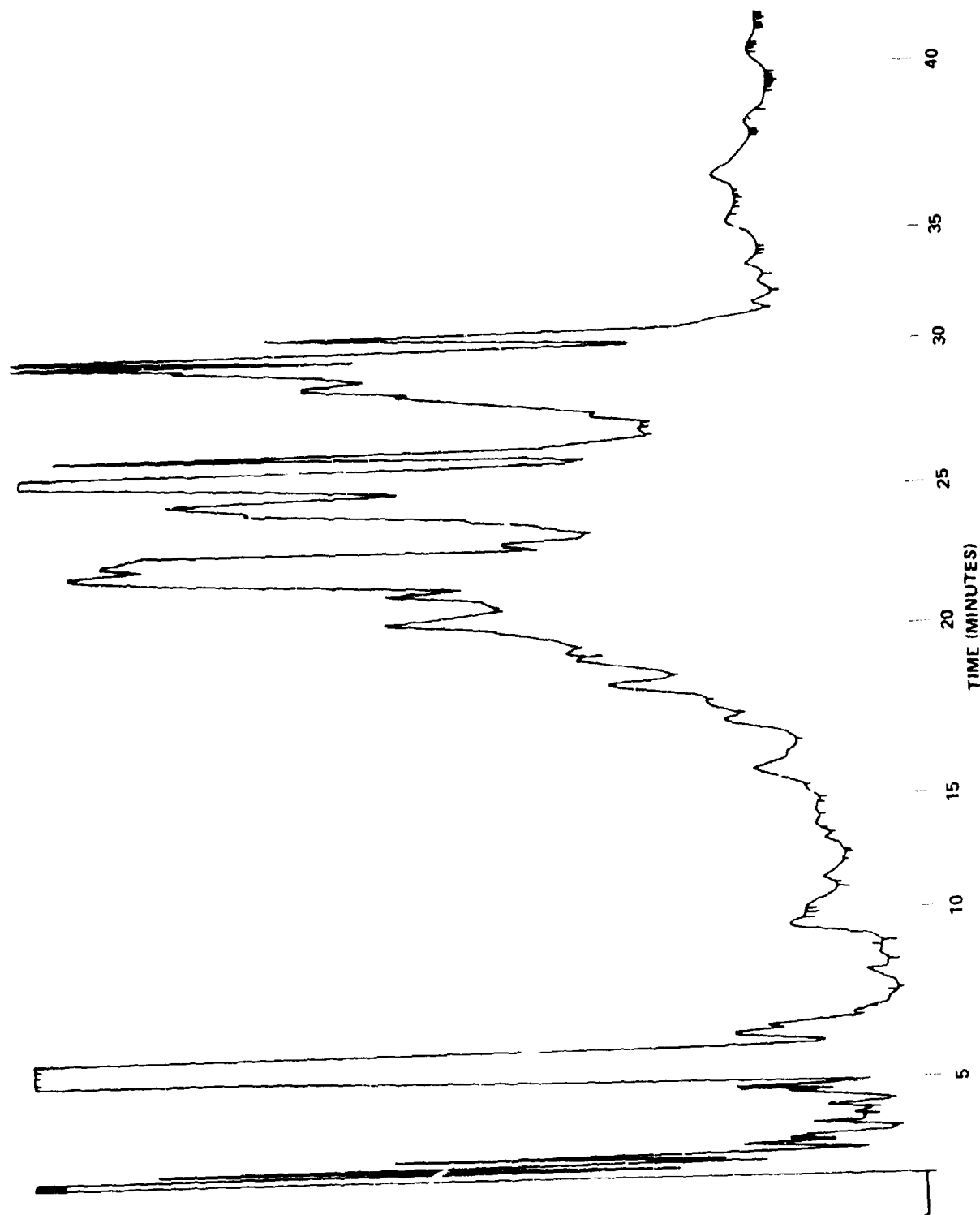


Figure 6. Synthetic base stock No. 6327 Di-Isodecyl Azelate.

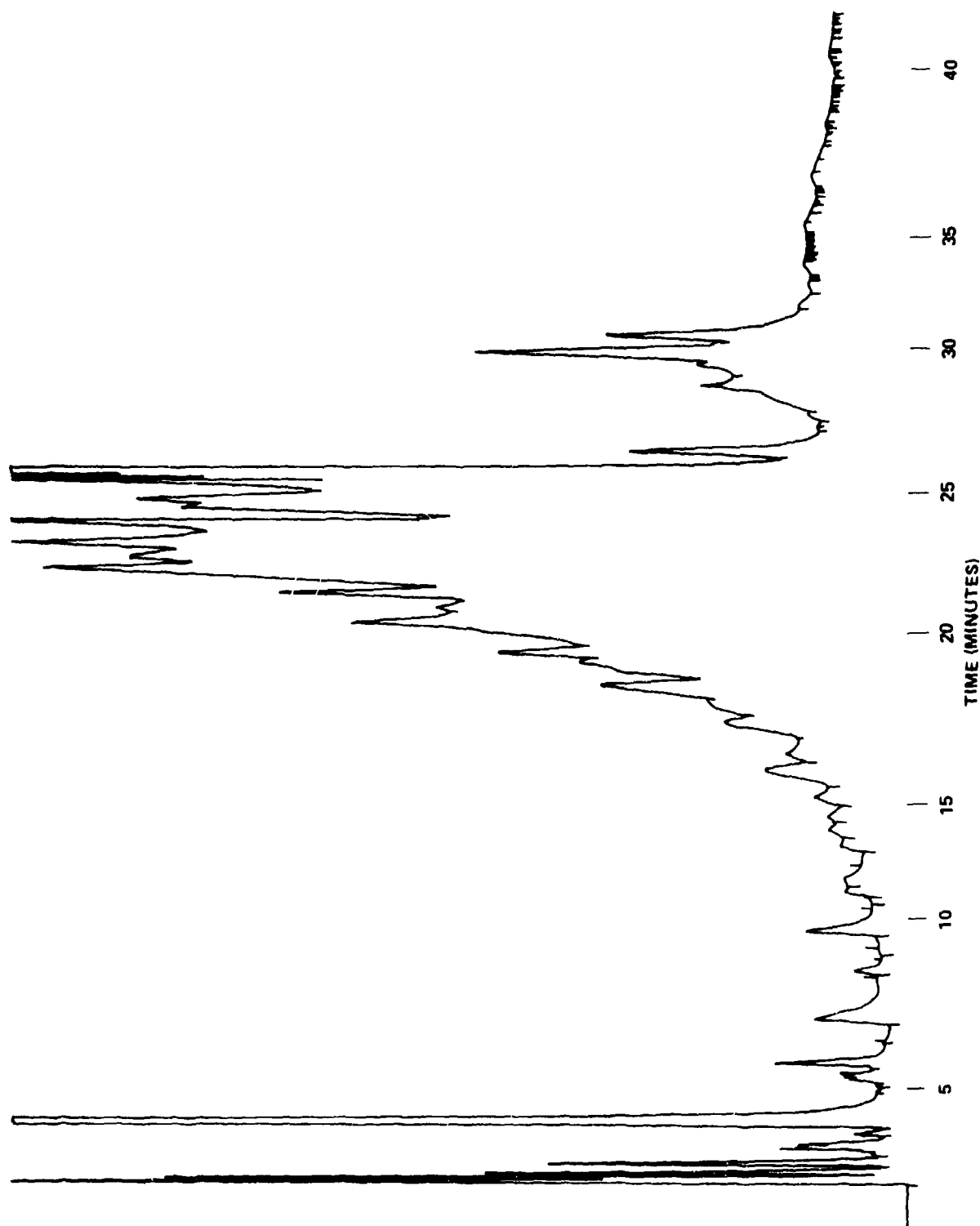


Figure 7. Synthetic base stock No. 6318 Di-2-Ethylhexyl Azelate.

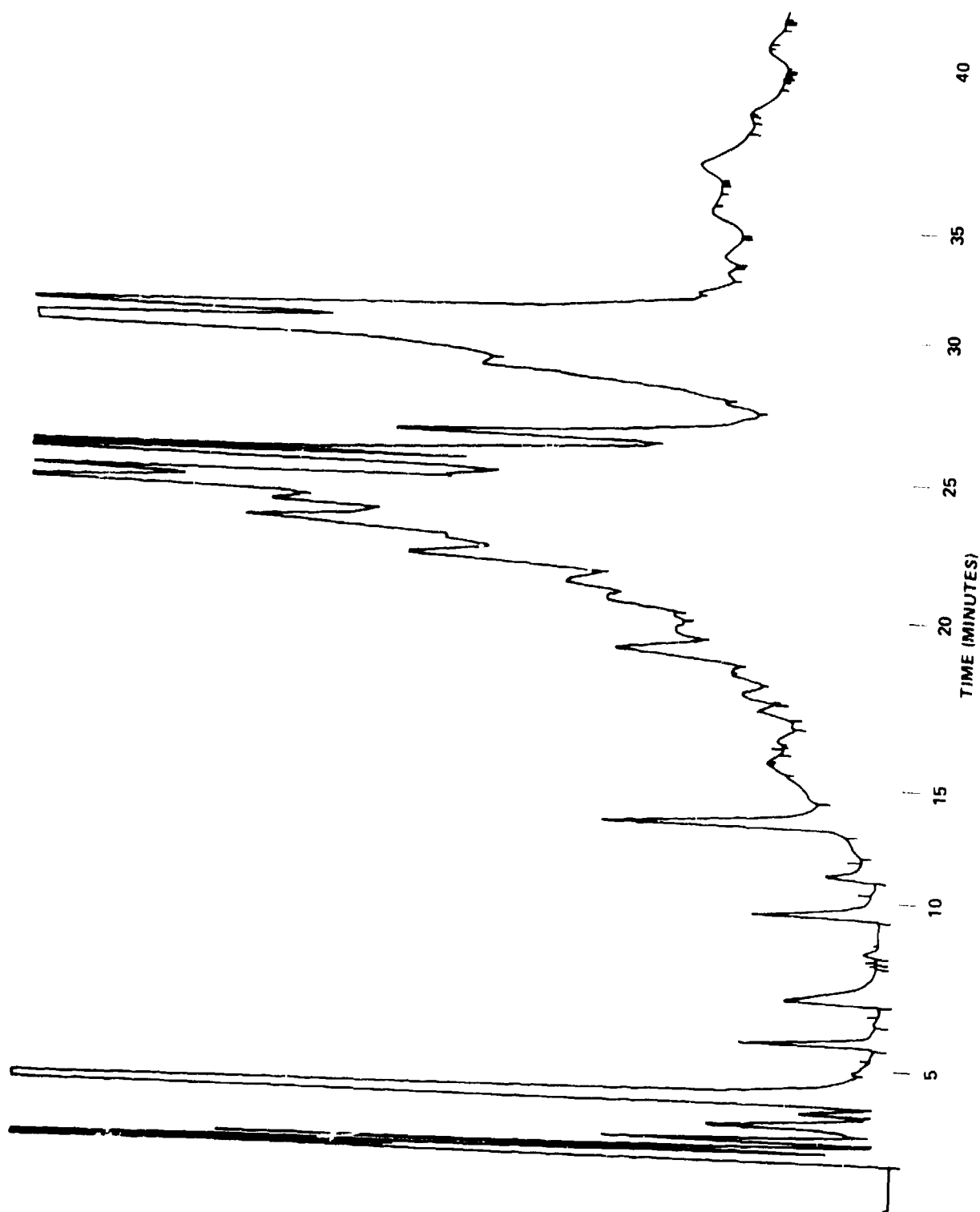


Figure 8. Synthetic base stock No. 6323 Di-2-Ethylhexyl Adipate.

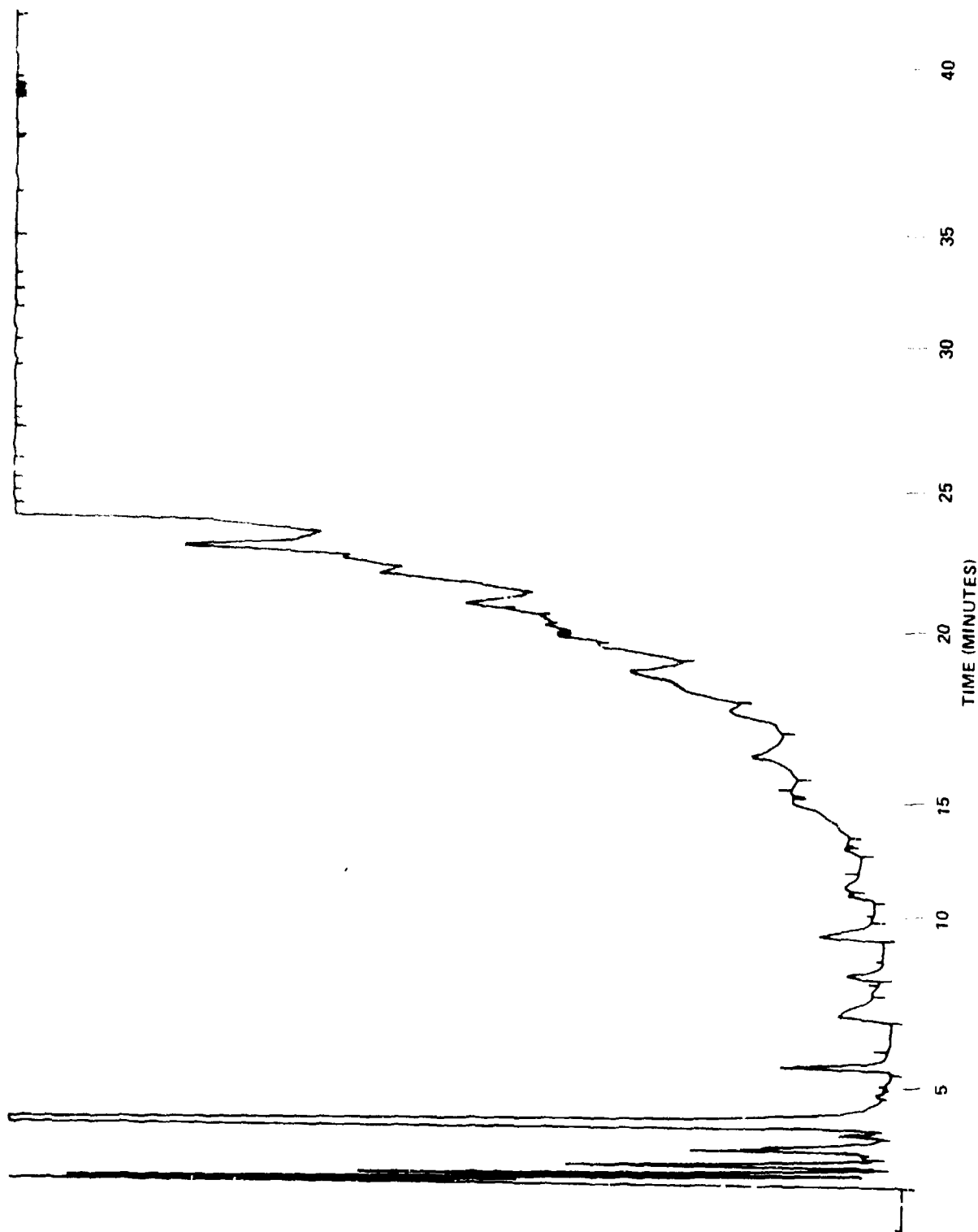


Figure 9. Synthetic base stock No. 6321 Di-2-Ethylhexyl Sebacate.

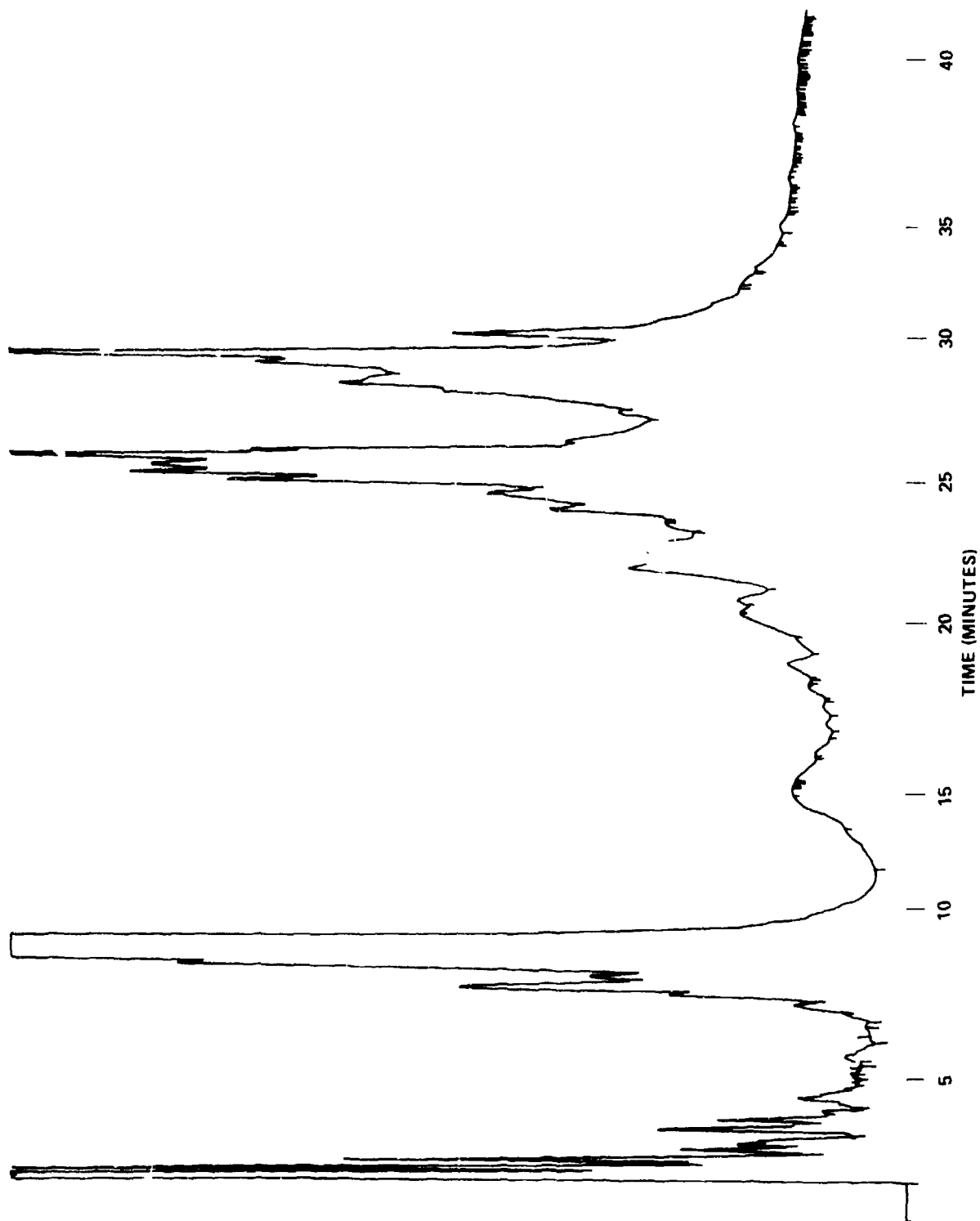


Figure 10. Synthetic base stock No. 6326 Di-Tridecyl Azelate.

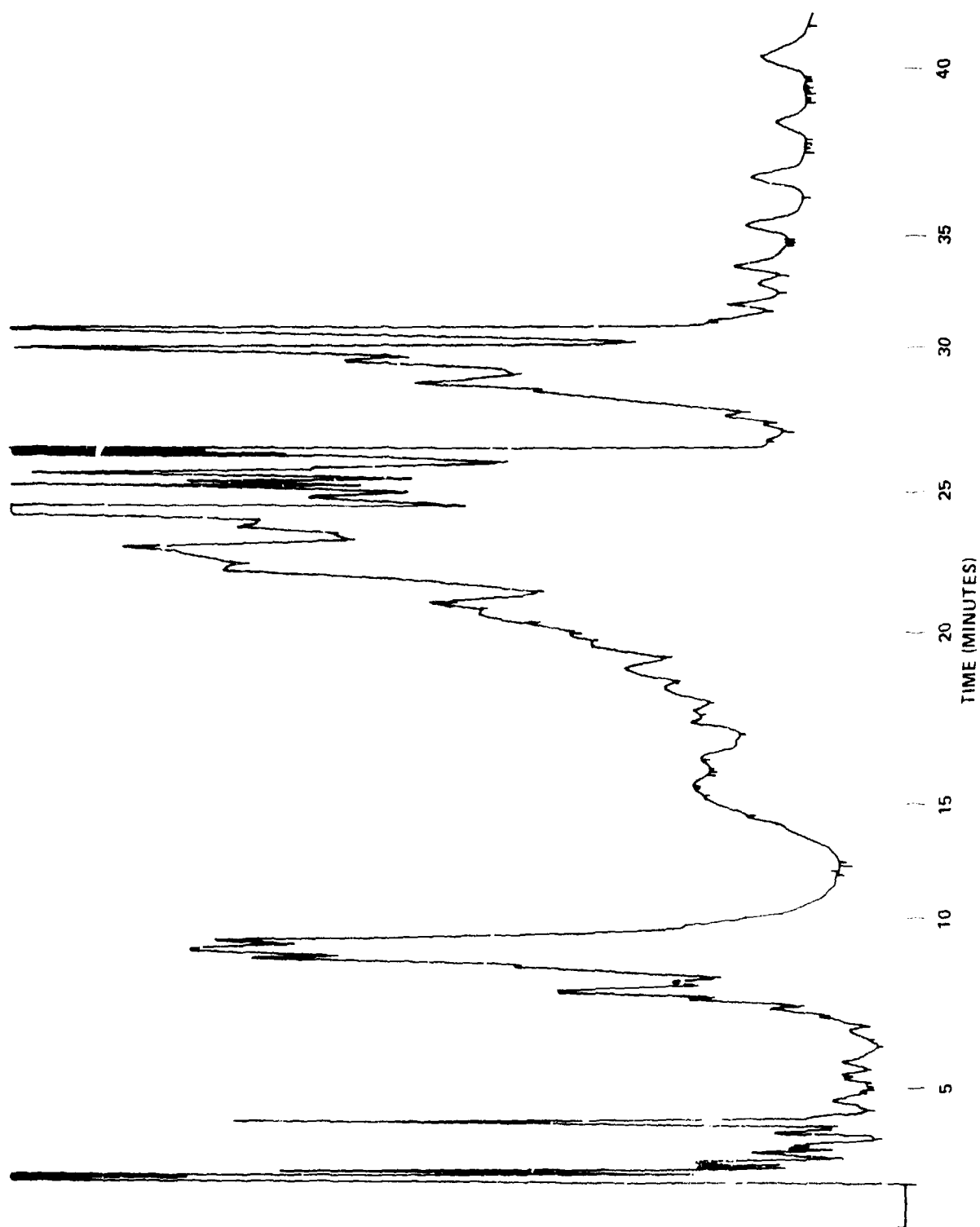


Figure 11. Synthetic base stock No. 6319 Di-Tridecyl Adipate.

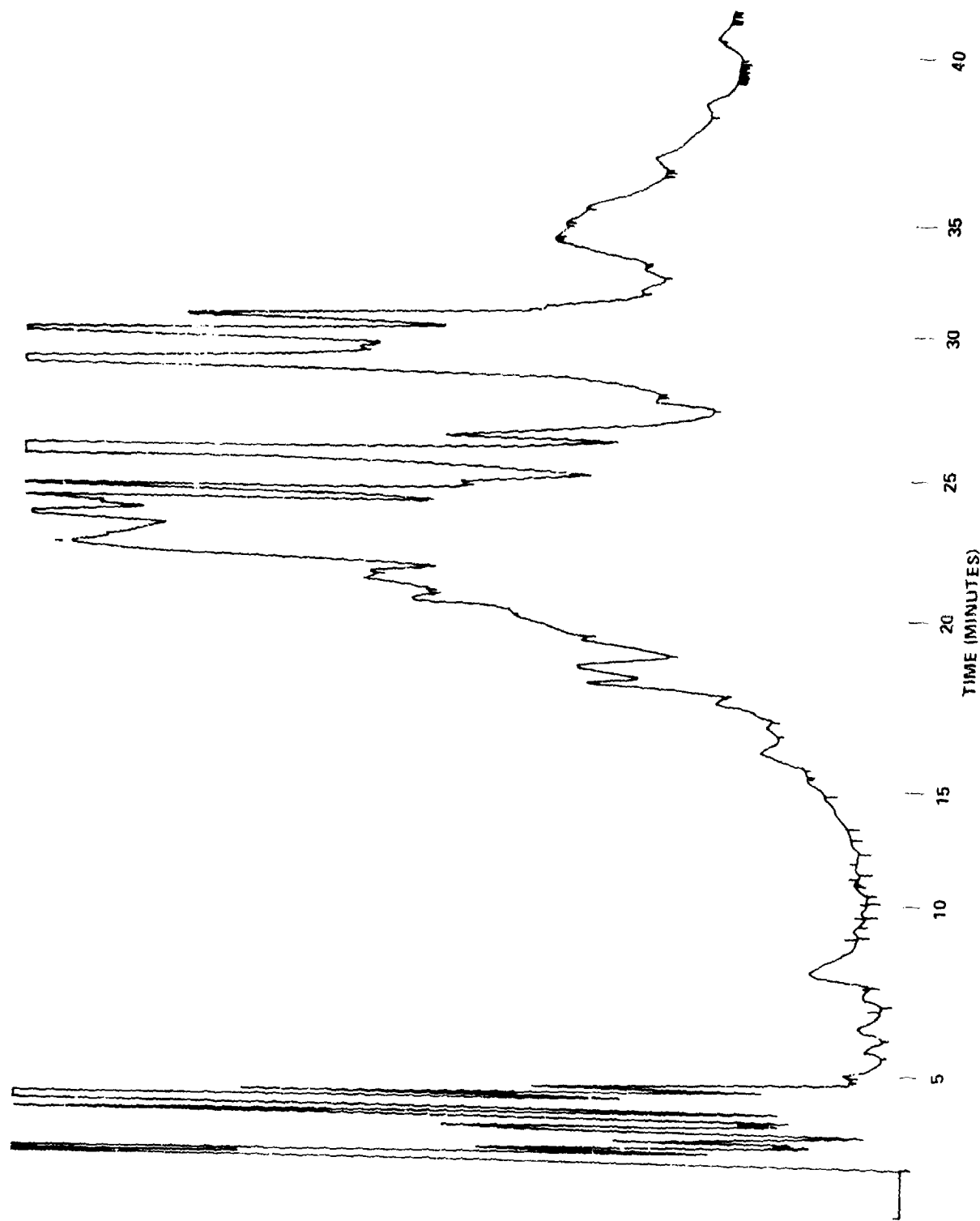


Figure 12. Synthetic base stock No. 6320 Di-Iso Octyl Azelate.

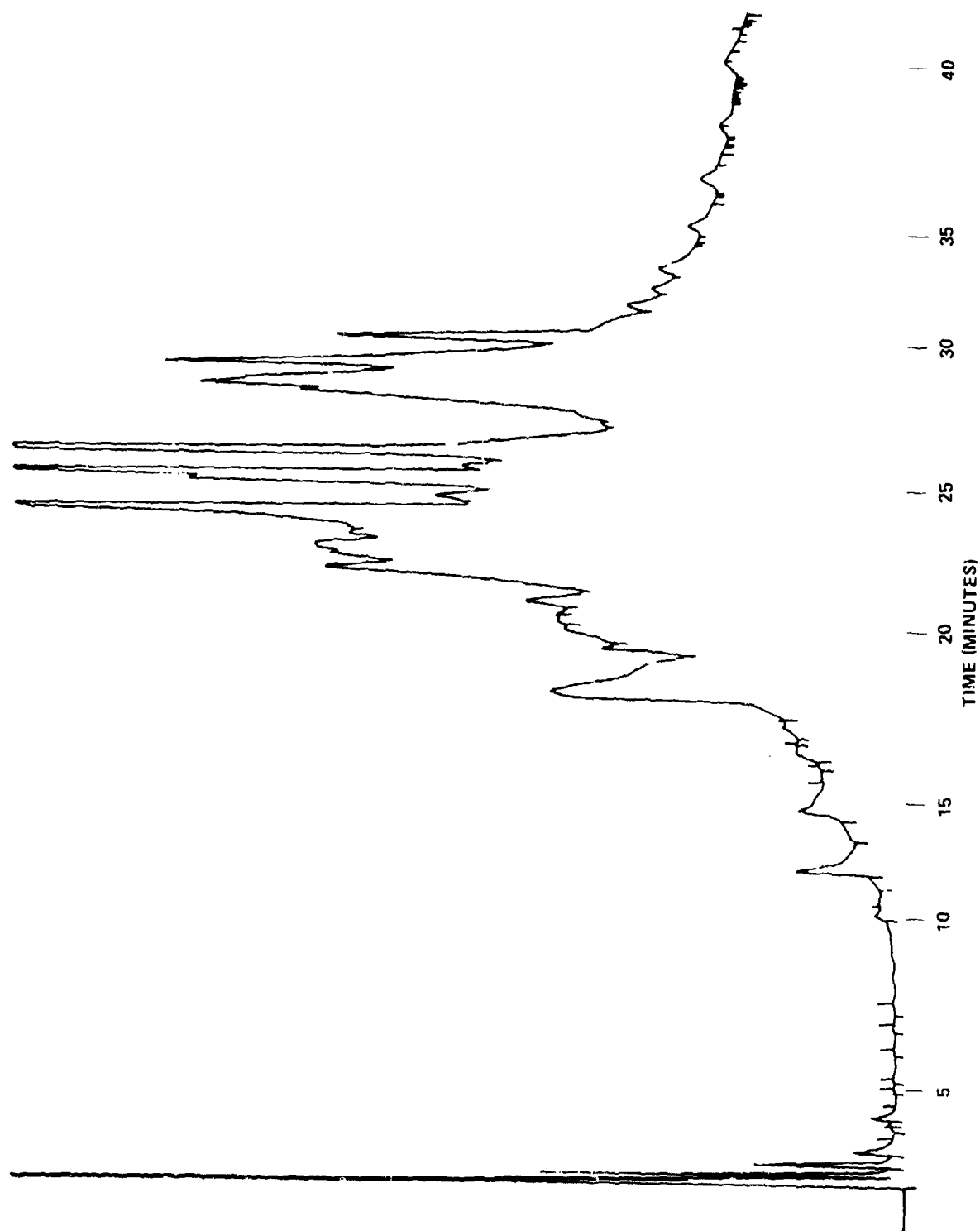


Figure 13. Synthetic base stock No. 6516 TMP Triheptanoate.

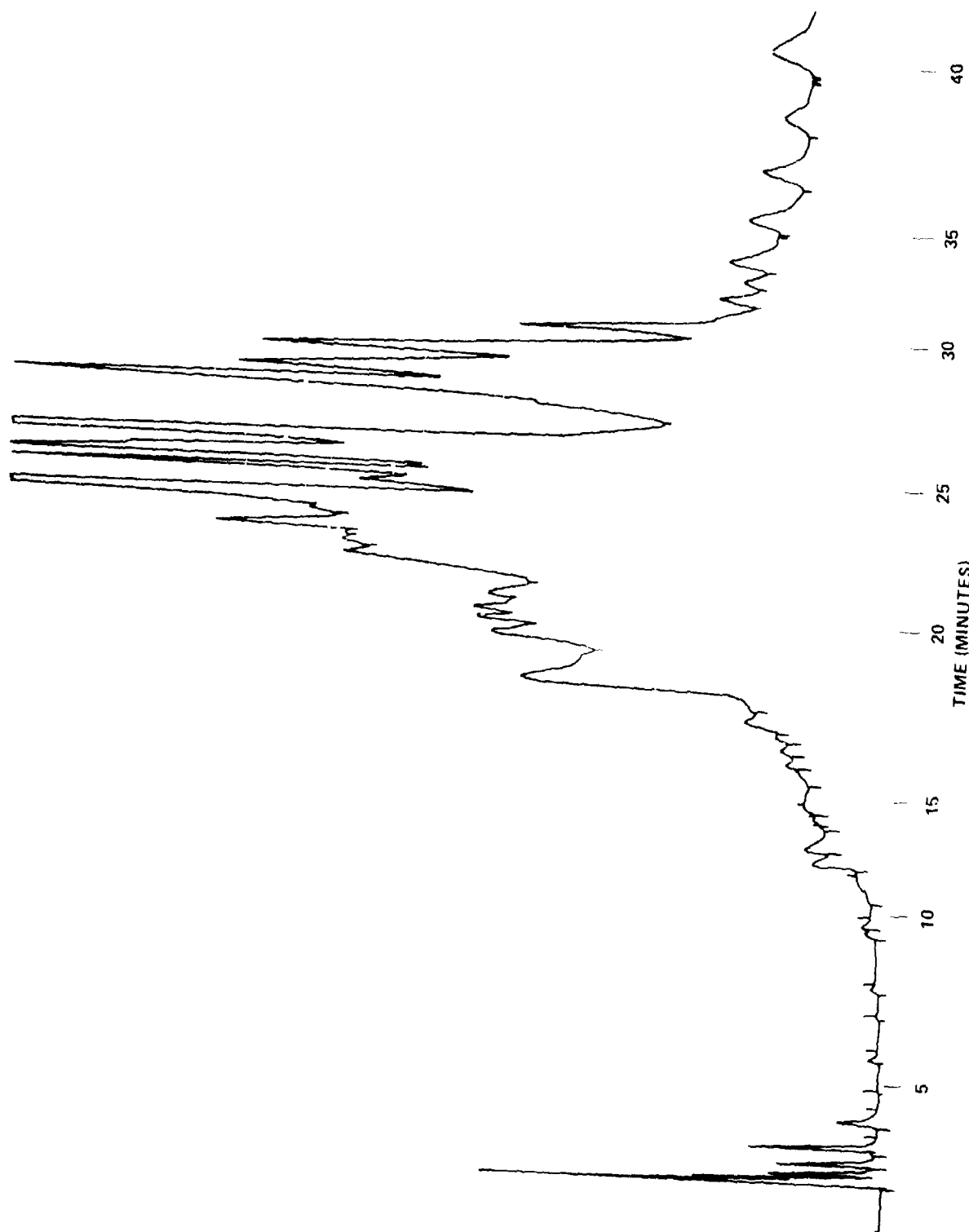


Figure 14. Synthetic base stock No. 6686 Neopentyl Dipelargante.

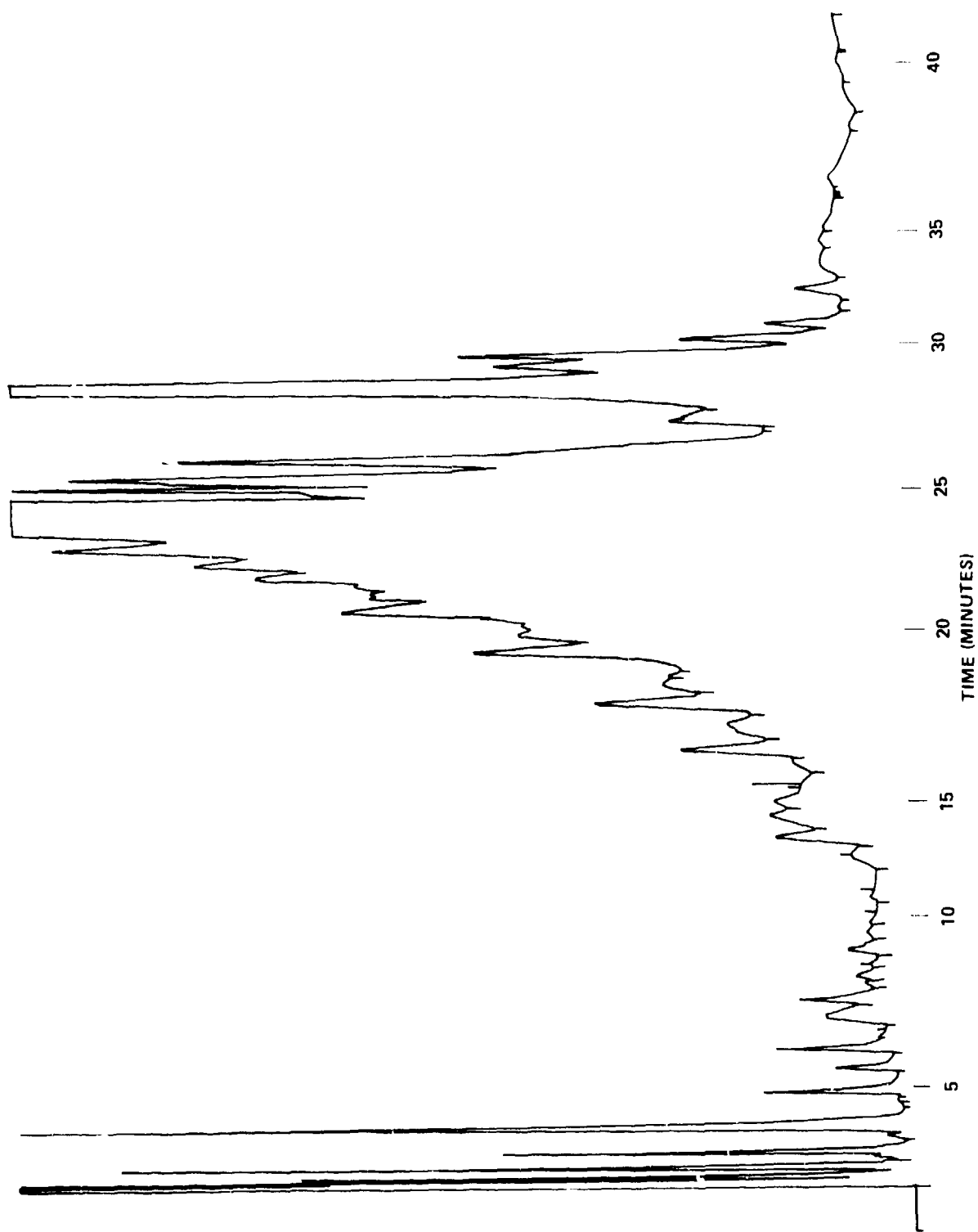


Figure 15. Synthetic base stock Polyalkylated Benzene.

Table 3. Military Specifications and Base Stocks Utilized

Specification No.	Manufacturer's Sample	OPL No.	Synthetic Base Stock
MIL-L-7808H	Hatco Mobile No. 1	None None	Di-Tridecyl Adipate Di-2-Ethylhexyl Azelate
MIL-L-46167A	EMERY	ME-5	Di-isodecyl Azelate
MIL-H-46170A	BRAY	MF-2	Polyalpha Olefin
MIL-H-46170A	Royal	MF-4	Polyalpha Olefin
MIL-H-46170A	GULF	MF-7	Polyalpha Olefin
MIL-H-46170A	HANOVER	MF-5	Polyalpha Olefin
MIL-L-46000B MIL-L-11734C	BRAY RUBBER Swelling Agent	None	Di-e-ethylhexyl Sebacate
MIL-L-23699C	MOBIL	None	Triheptanoate
MIL-L-46152B	CONOCO	ML55	Polyalkylated Benzene

Table 4. Blends of Synthetic Products

Known Composition	Pyrolysis Identification of Synthetic Base
Blend A 100% Diester (Isodecanol, Azelaic)	Di-isodecyl Azelate
Blend B 40% Diester (2-ethylhexanol, Azelaic) 60% Polyalkylated Benzenes	Polyalkylated Benzene Derivative
Blend C 12% Midcontinent 86% Polyalkylated Benzenes 2% Diester (2-ethylhexanol, Azelate)	Polyalkylated Benzene derivative
Blend D 100% Diester (isotriclecanol, Adipic)	Di-Tridecyl Adipate
Blend E 34% Polyol Ester 65% Polyalpha Olefin 1% Diester	Polyalpha Olefin

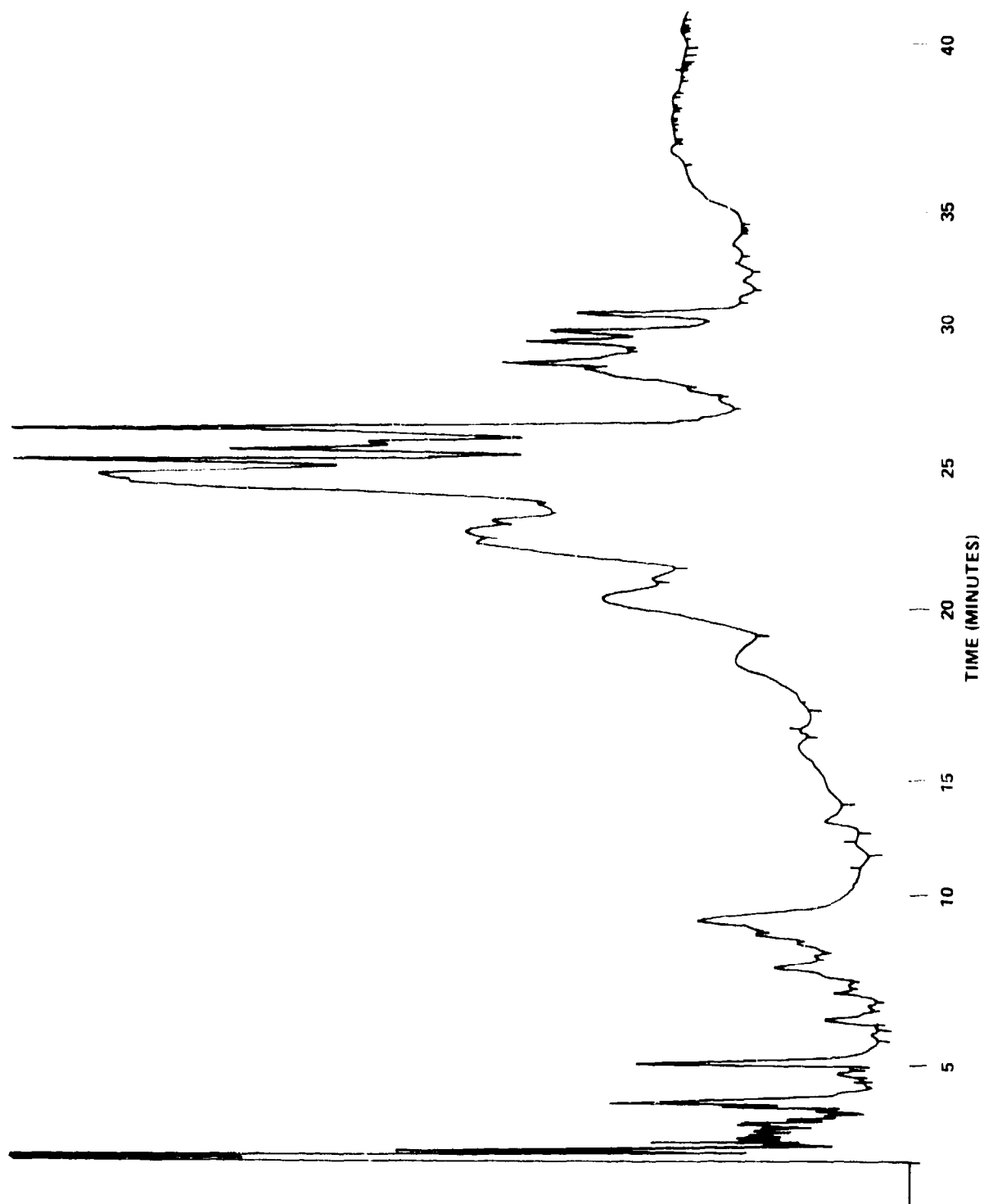


Figure 16. MIL-L-46152A, Lubricating oil, I-C Engine, administrative service.

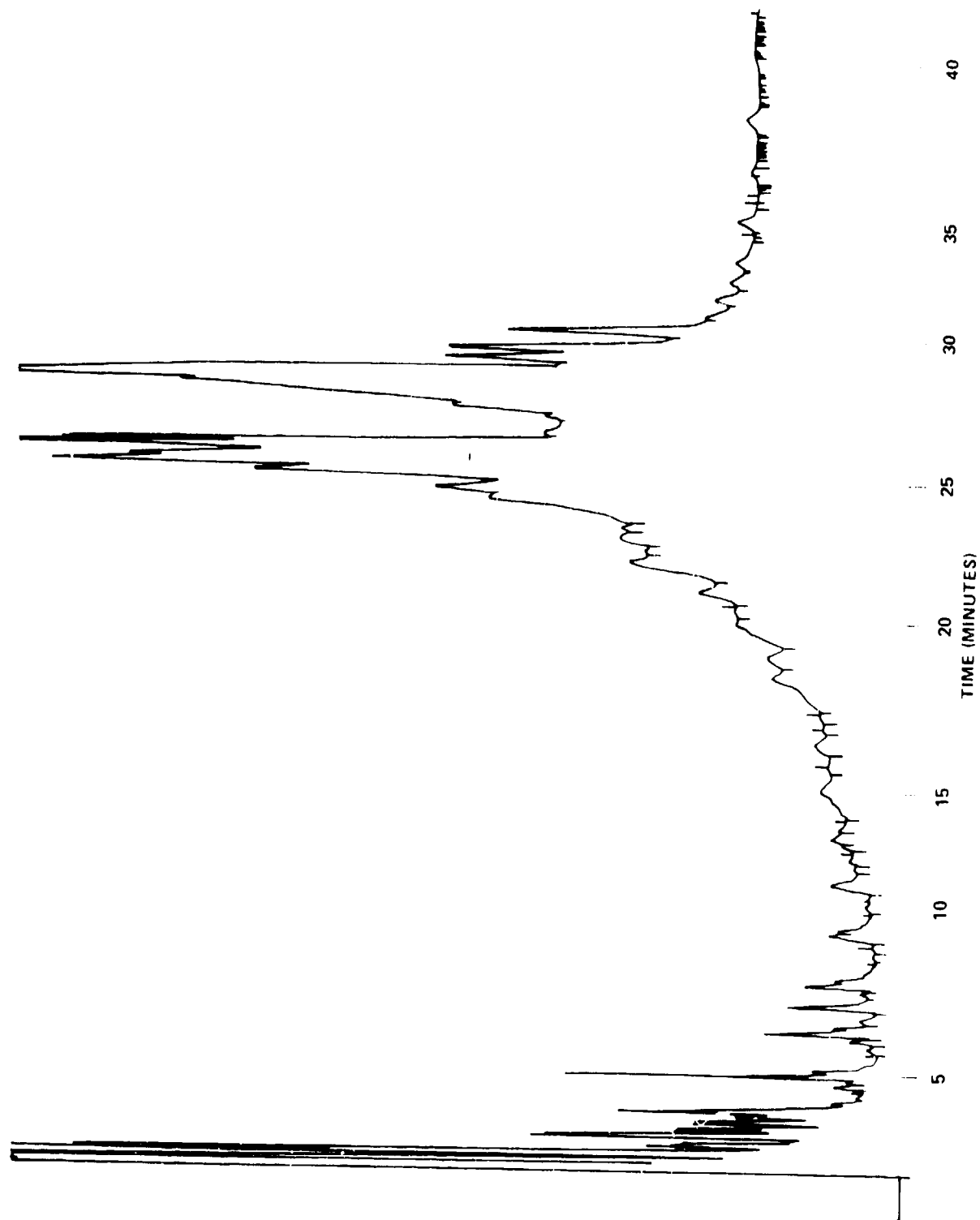


Figure 17. Lubricating oil, internal combustion engine, Arctic.

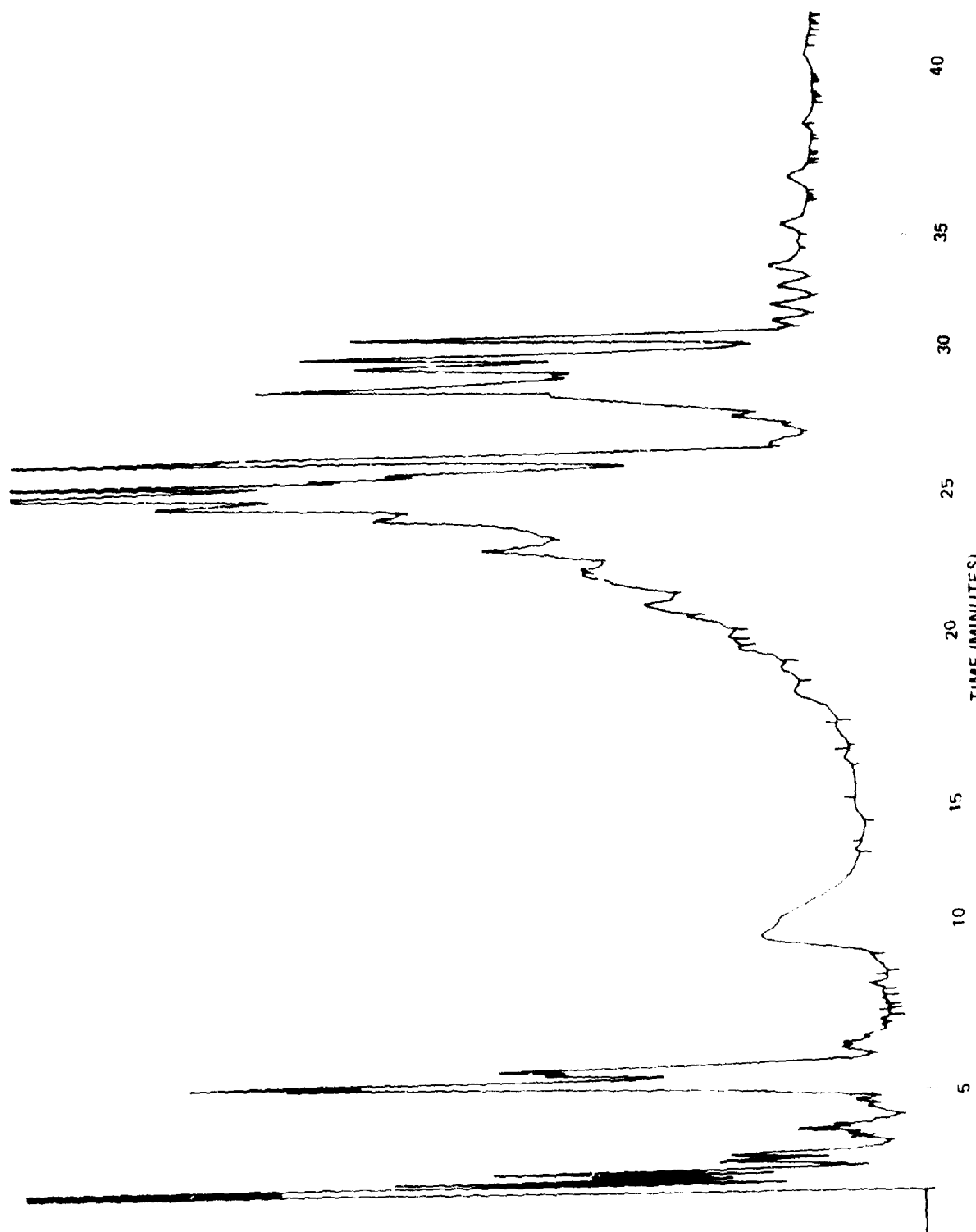


Figure 18. MIL-L-46167A, Lubricating oil, I-C Engine, Arctic grade.

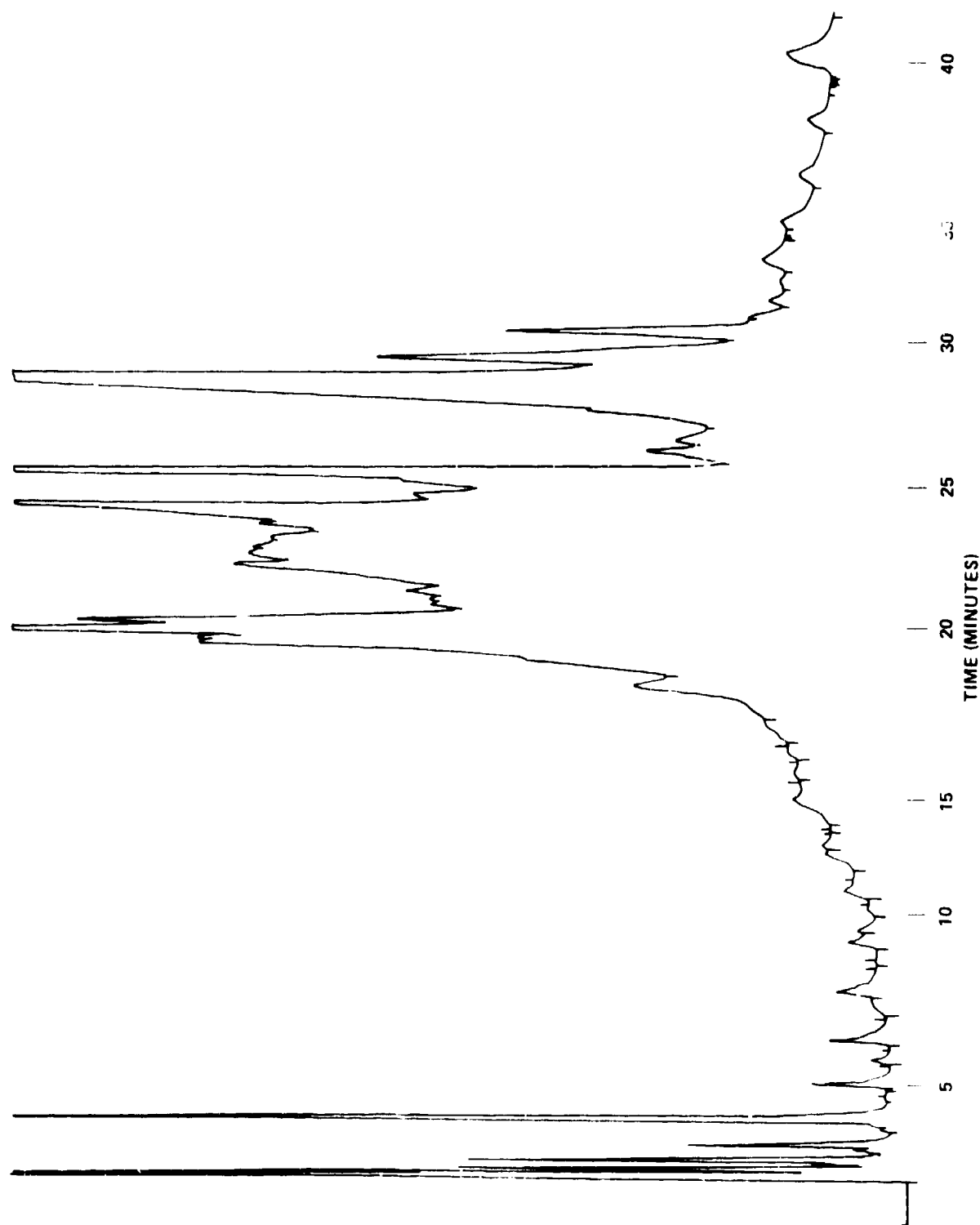


Figure 19. MIL-H-46170A Hydraulic fluid, rust inhibited, fire-resistant, synthetic hydrocarbon base, Hanover.

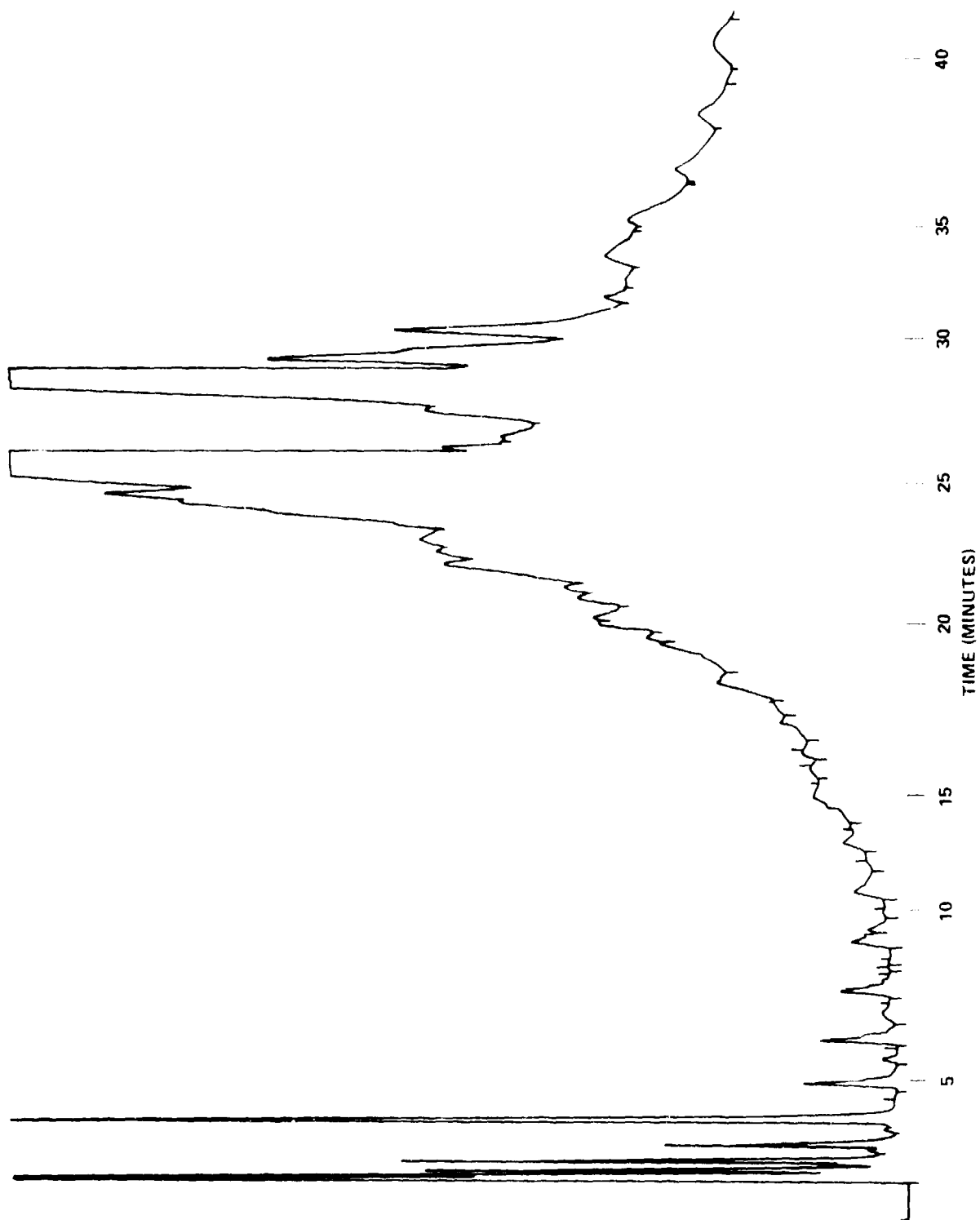


Figure 20. MIL-H-46170A, Hydraulic fluid, rust inhibited, fire resistant, synthetic hydrocarbon base, Gulf.

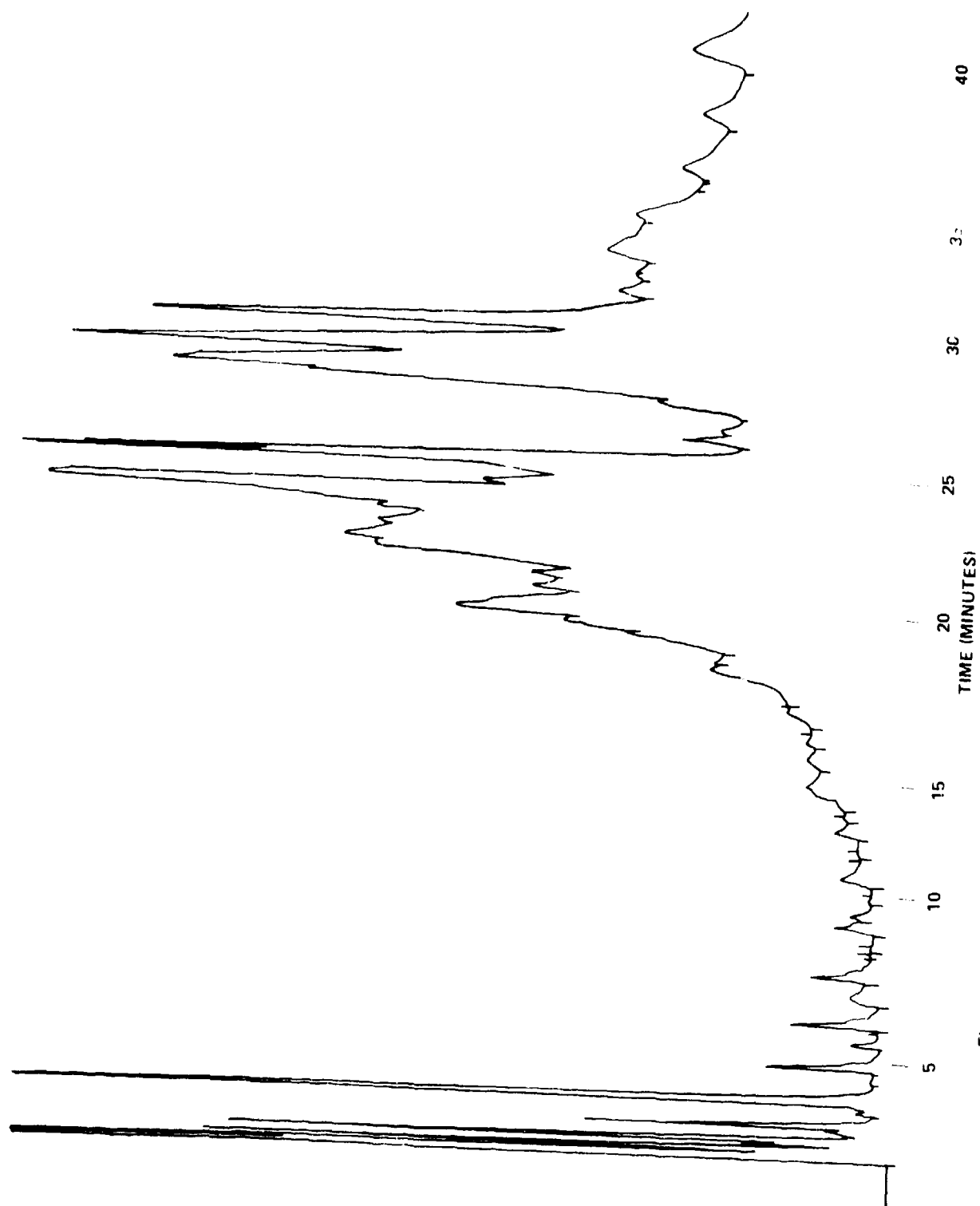


Figure 21. MIL-H-46170A, Hydraulic fluid, rust inhibited, fire resistant, synthetic hydrocarbon base, 50:1.

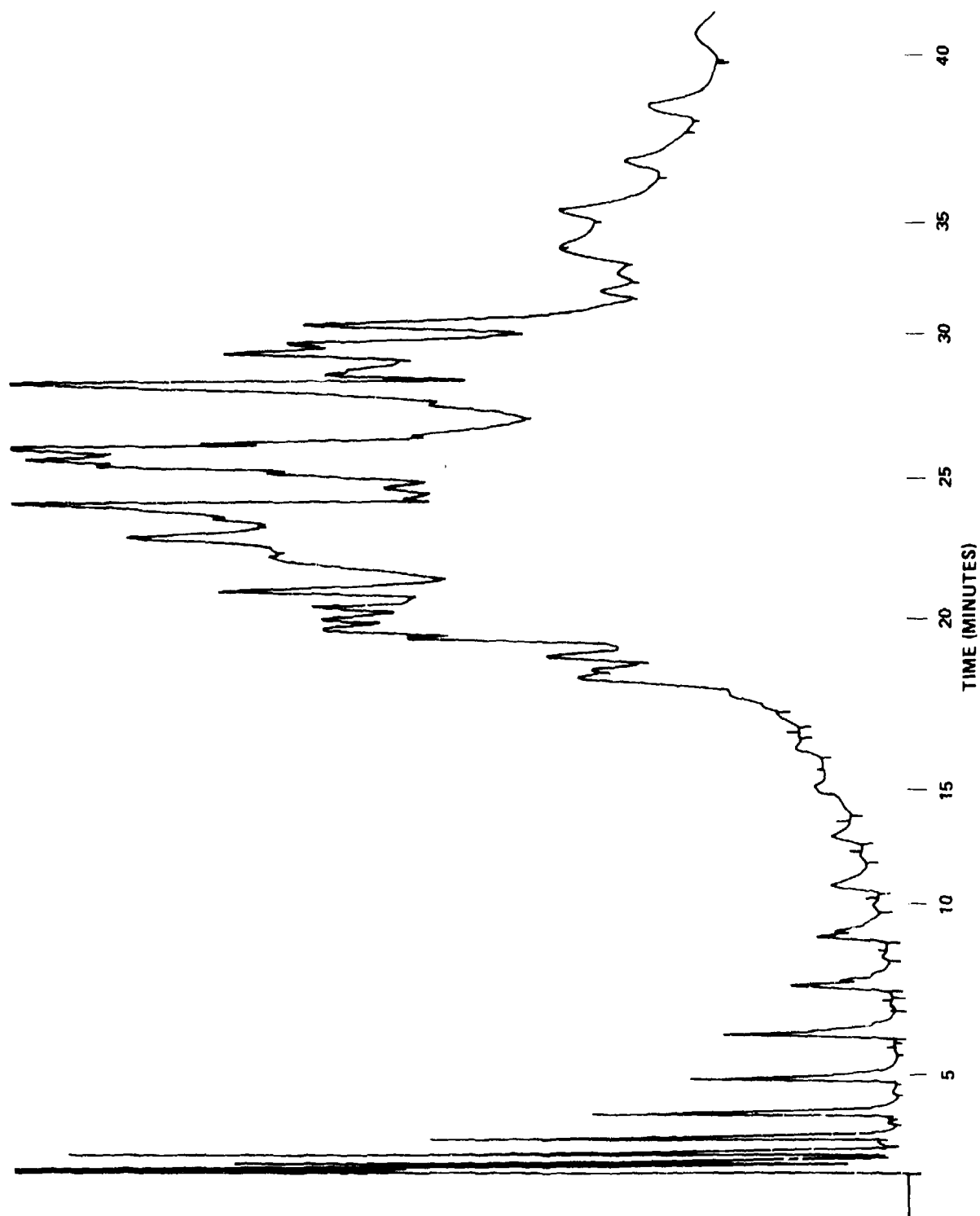


Figure 22. MIL-H-46170A, Hydraulic fluid, rust inhibited, fire resistant, synthetic hydrocarbon base, Bray.

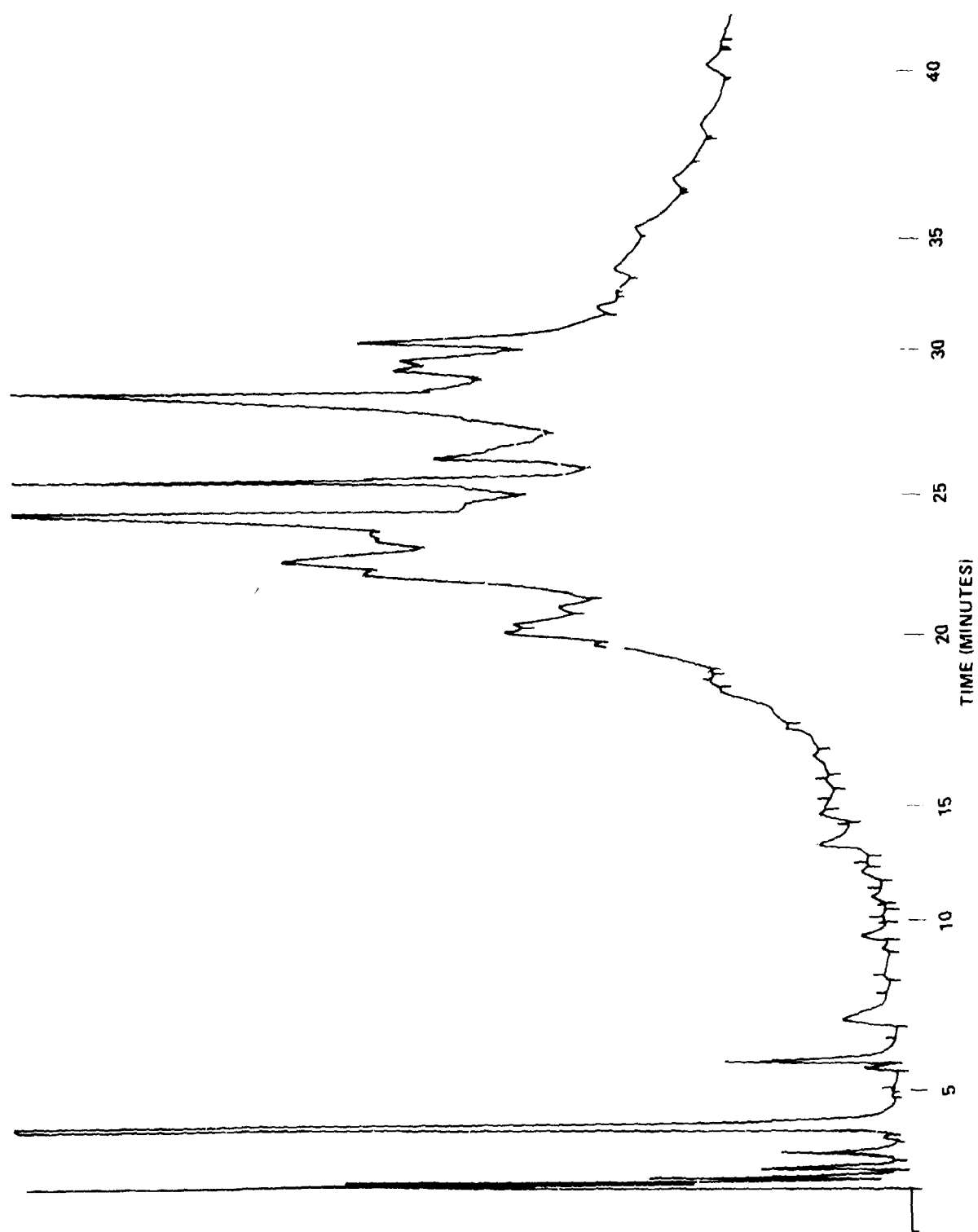


Figure 23. MIL-L-7808H, Lubricating oil, aircraft turbine engine, synthetic base.

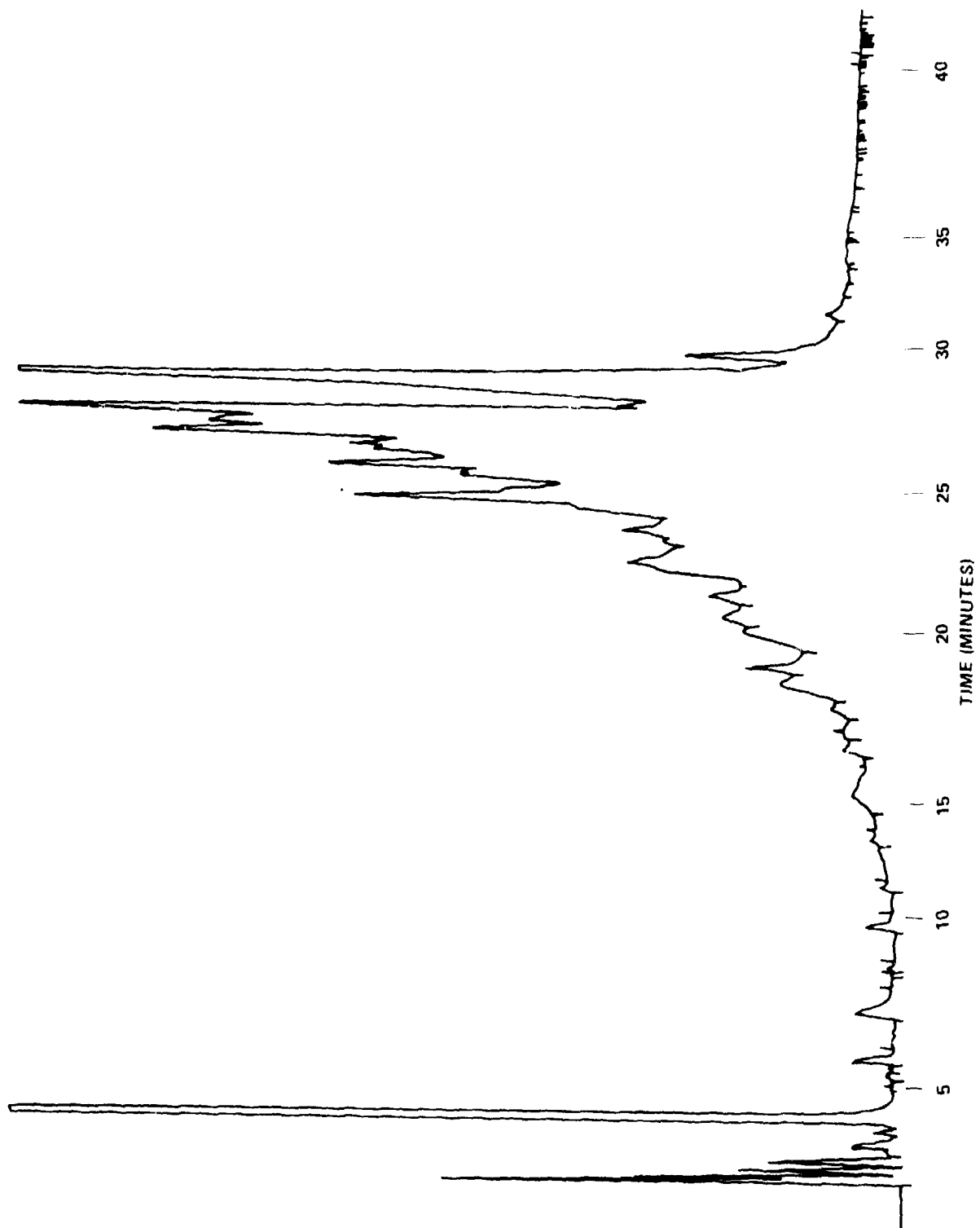


Figure 24. Rubber swell agent.

It was found that the two silicone fluids (Figures 25 and 26) were completely different presenti. no problems in identifying other marketed materials containing the same base fluids. The same principle, however, did not apply to the petroleum products because of the extreme peak distortions due to additive package and chemical structure decomposition.

IV. CONCLUSIONS

As the carbon number of synthetic materials increase, all pyrograms differ in elution time, peak configuration, and peak height. Silicone brake fluids produce a unique pattern of eluted peaks.

The PGC method provides a capability for an accurate fingerprint of extremely complex mixtures. Characterization of these high molecular weight materials, which usually require many hours of extensive wet chemical analysis can now be completed in relatively short time periods by using pyrolysis gas liquid-phase chromatography.

The data generated on the synthetic materials was treated in a Hewlett Packard 9830A computer for simple comparison purposes. Because of the peak pattern distortion in fully formulated products, it was virtually impossible to enter the data in the computer and positively identify via computer reduction techniques the base material. Therefore, the comparisons were made using visual techniques.

The use of this method will allow resolution of field problems in a very short time frame.

Future efforts will be made to develop a computer program that will allow rapid analysis of finished products and identification of the individual components.

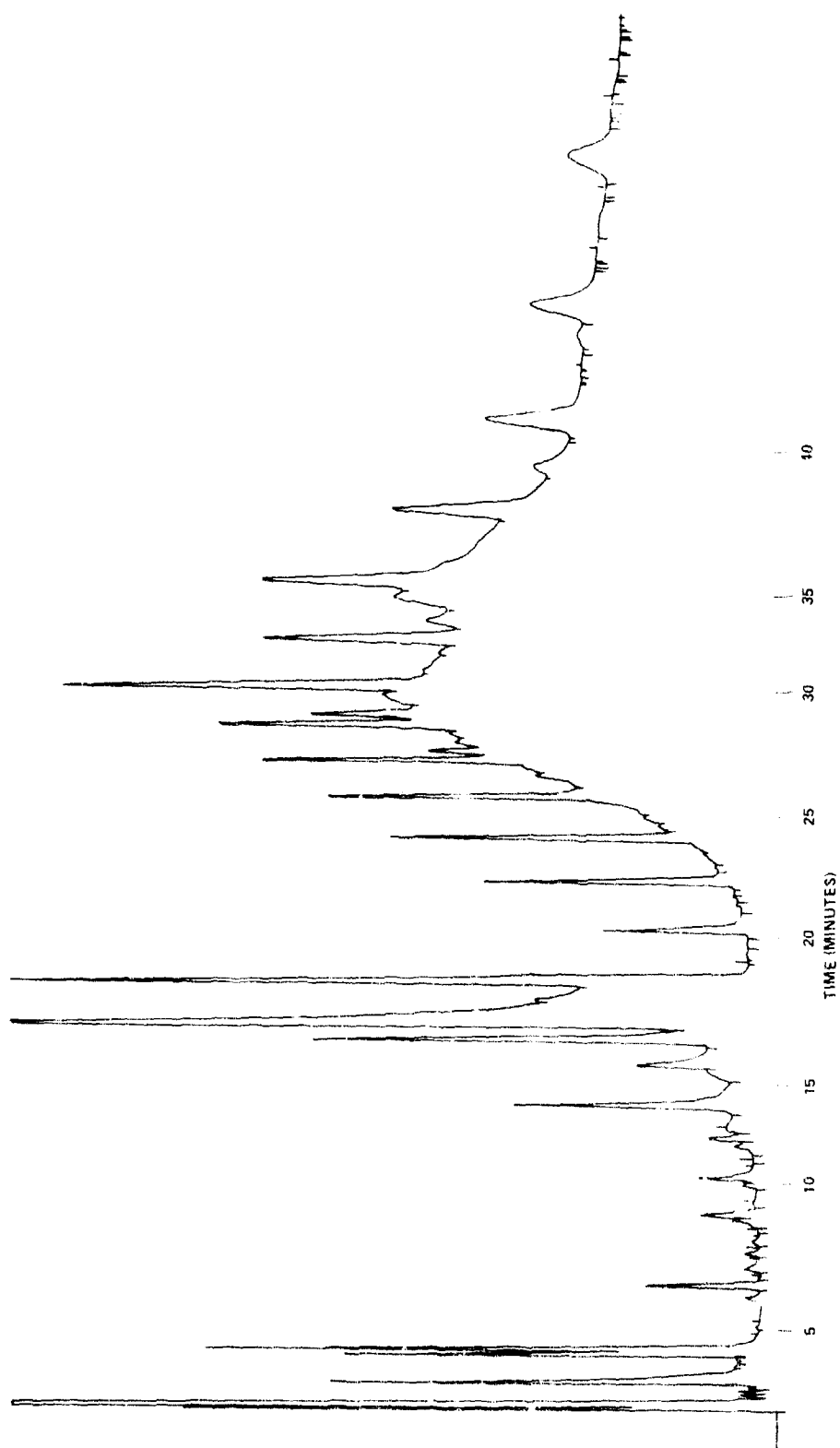


Figure 25. MIL-B-45176, Brake fluid, silicone, automotive, all weather, operational and preservative, Dow.

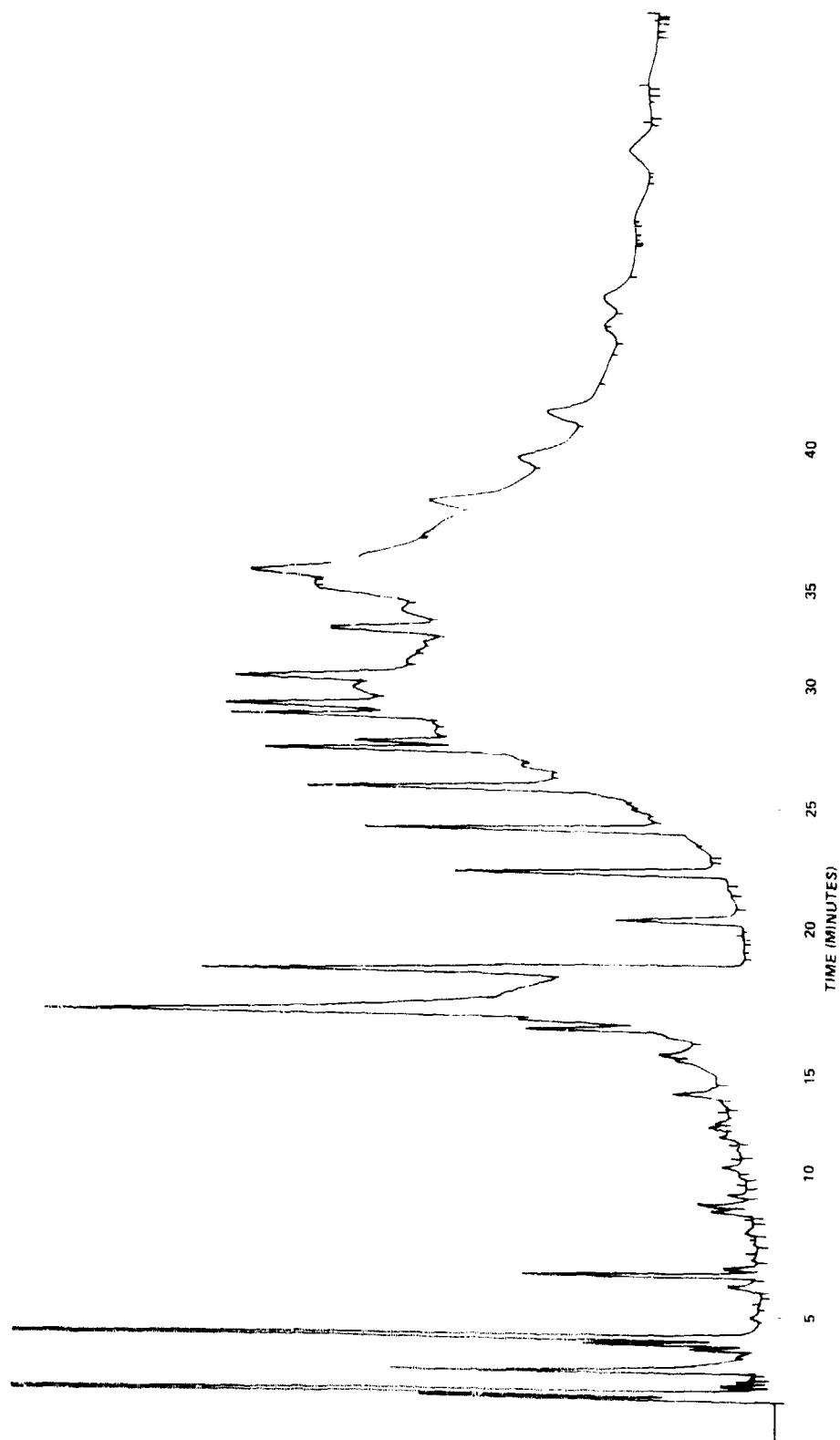


Figure 26. MIL-B-46176, Brake fluid, silicone, automotive, all weather, operational and preservative, General Electric.

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