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STORAGE FLUID MAINTENANCE STUDY USING CALORIA HT-43® HYDROCARBON OIL

Final Report for September 1977—November 1978

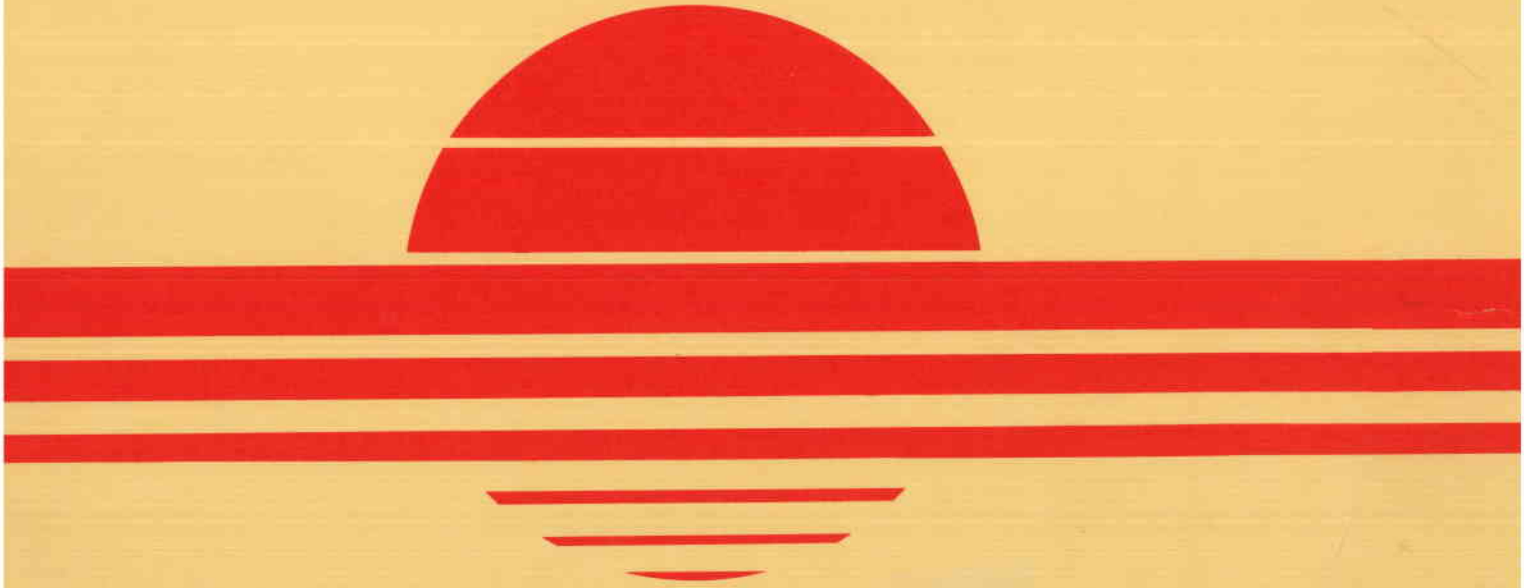
23.752

By
D. G. Beshore
J. E. Myers
W. R. Morgan
M. G. Barth

April 1979
Date Published

Work Performed Under Contract No. EY-76-C-03-1110

Martin Marietta Aerospace
Denver, Colorado



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MARTIN MARIETTA AEROSPACE
DENVER DIVISION

FOREWORD

This report, prepared by the Solar Thermal Systems group of Martin Marietta Aerospace, Denver Division, Denver, Colorado, summarizes the work performed under DOE Contract DE-AC03-76ET-20422 Oil Evaluation Subprogram to Central Receiver Solar Thermal Power System, Phase I. The DOE contract manager was Dr. S. Douglass Elliott. The Sandia Laboratories-Livermore technical manager was Dr. Lee Radosevich assisted by Mr. Victor Burolla. Initially, Mr. C. N. Bolton was the Martin Marietta Program Manager, with Mr. D. N. Gorman replacing him just prior to the program completion. Mr. T. Chevens of Howe-Baker Engineers, Inc. and Mr. J. McWhorter of McWhorter, Reeves, and Assoc. were responsible for the design and construction of the sidestream processor.

ABSTRACT

Thermal energy storage concepts using hydrocarbon oils are being considered by the Department of Energy in first generation solar thermal power feasibility studies and plant development efforts. Martin Marietta Aerospace, Denver Division, investigated the thermal degradation characteristics of Exxon's Caloria HT-43 oil under this program. The program has been accomplished under two phases of effort. Phase I has studied the thermophysical effects of oil degradation in a partially vented storage tank at 316^oC (600^oF) for a period of 1000 hours. Phase II has examined the effect of a sidestream processor (vacuum distillation column) on oil degradation in a vented storage tank. Test results indicate that the thermal degradation process is autocatalytic. Therefore, both tank venting and oil reprocessing can actually reduce the degradation rate. The experimentally determined degradation rates under the two phases of effort were in Phase I-2.4 percent by weight per week and in Phase II-0.1 percent by weight per week. **However, computed fluid replenishment rates were in Phase I and II, 1.1 and 0.8 percent respectively. Improved processor designs could substantially lower the 0.8 percent per week observed for the Phase II sidestream processor.**

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I. EXECUTIVE SUMMARY

The use of hydrocarbon based oils is being considered by the Department of Energy for use as a thermal energy storage medium in first generation solar thermal power plant development. The experimental study program described in this report evaluated the stability of Exxon's Caloria HT-43 oil at a storage temperature of 316°C (600°F). Two phases of tests were conducted to determine the storage fluid replenishment rate and oil degradation rate in (1) a partially vented storage tank and (2) a vented storage tank with a sidestream processor (vacuum distillation unit). The complete system consisting of sidestream processor and storage tank is shown in Figure I-1.

Results from this program indicate that the decomposition process for this oil is autocatalytic. That is, decomposition products which remain in the oil solution promote further degradation in addition to thermal cracking. Chemical analyses indicate thermal cracking, no oxidation, and no polymerization occur in the bulk oil in both Phase I, a partially vented system, and Phase II, a vented system with a sidestream processor. Decomposition products vented from the storage tank indicates the presence of olefinic (unsaturated hydrocarbon) groups. The volatiles had carbon numbers in the C_3 to C_{16} range.

The sidestream processor used in the Phase II tests performed continuously for 1000 hours and separated oil fractions as designed. However some polymerization products were formed during the reprocessing step which was likely caused by slight oxidation of the oil. These polymerization products were separated from the processor and therefore were never returned to the storage vessel. Simulation models were developed and used to assess the processor performance and determine oil degradation rates.

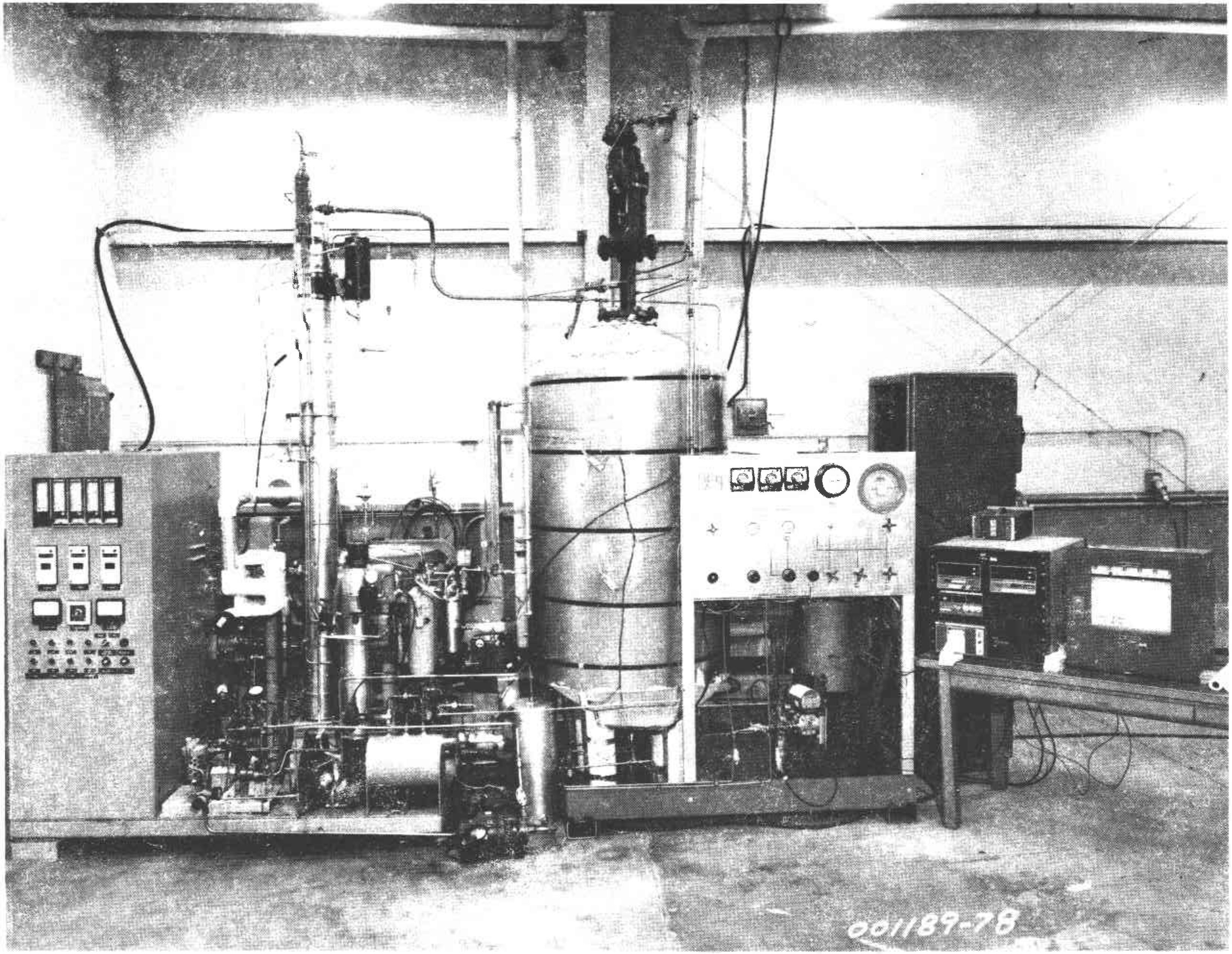

FIGURE I-1 Phase II System

Through the use of detailed mass flow accounting in both phases of the program and the results of the chemical analyses, fluid replenishment rates and oil degradation rates were calculated. The fluid replenishment rate is the rate at which oil must be added to the storage vessel to maintain a constant mass. The oil degradation rate, however, is defined as the amount of decomposition products formed in the oil or escape from solution below the initial boiling point and above the final boiling point of the original oil. The initial boiling point of a hydrocarbon mixture is the temperature at which the fluid first begins to boil and generally represents the boiling point of lowest molecular weight fraction in the oil. The final boiling point of a mixture is the highest temperature at which a portion of the fluid remains in liquid phase and therefore indicates the boiling point of the oil fraction with the highest molecular weight. The replenishment and degradation rates of the oil at 316°C (600°F) are summarized below:

	Phase I	Phase II
Degradation rate (wt %/week)	2.4	0.1
Replenishment rate (wt %/week)	1.1	0.8

In phase I the replenishment rate was less than the degradation rate resulting from some decomposition products remaining in solution. Conversely, the Phase II replenishment rate was greater than the degradation rate due to the processor extracting non degraded oil in addition to decomposition products.

The results of the program show that venting and reprocessing reduce the oil degradation rate. Reprocessing the oil results in a replenishment rate greater than the degradation rate. The unresolved question after this program is what contribution does either venting or processing singly have on reducing the degradation rate. The answer can only result from additional testing and analysis. A Phase III program has been proposed to answer these questions and will probably commence in early 1979.

II. INTRODUCTION

Thermal energy storage concepts using hydrocarbon oils are being considered by the Department of Energy in first generation solar thermal power feasibility studies and plant development efforts. Martin Marietta Corporation, Denver Division, is currently investigating the thermal degradation characteristics of Exxon's Caloria HT-43 oil under contract EY-76-C-03-1110. The program has been accomplished under two phases of effort. Phase I has studied the thermophysical effects of oil degradation in a partially vented storage tank at 316°C (600°F) for a period of 1000 hours. Phase II has examined the effect of a sidestream processor (vacuum distillation column) on oil degradation in a vented storage tank for a period of 1000 hours. Test results from this program indicate that the thermal degradation process is autocatalytic. Therefore, both tank venting and oil reprocessing can actually reduce the degradation rate.

A. BACKGROUND

Exxon's heat transfer oil Caloria HT-43 was chosen for evaluation in this program because of its potential application in the 10 MWe Barstow pilot plant. A literature search revealed decomposition rate data (Figure II-1) as a function of oil storage temperature (Ref. 1, 2, 3, 4). This data was obtained by placing a small quantity of oil in a sealed container and subjecting the oil and container to elevated temperatures for various lengths of time. The decomposition data (Figure II-1) indicates that at 589°K (600°F) a total decomposition rate of 2.5 percent by weight per week can be expected for Caloria HT-43 in sealed systems. However, this laboratory data does not coincide with actual industrial operating experience in systems which have used Caloria HT-43 for years. Unfortunately, most industrial records are scant and are inadequate

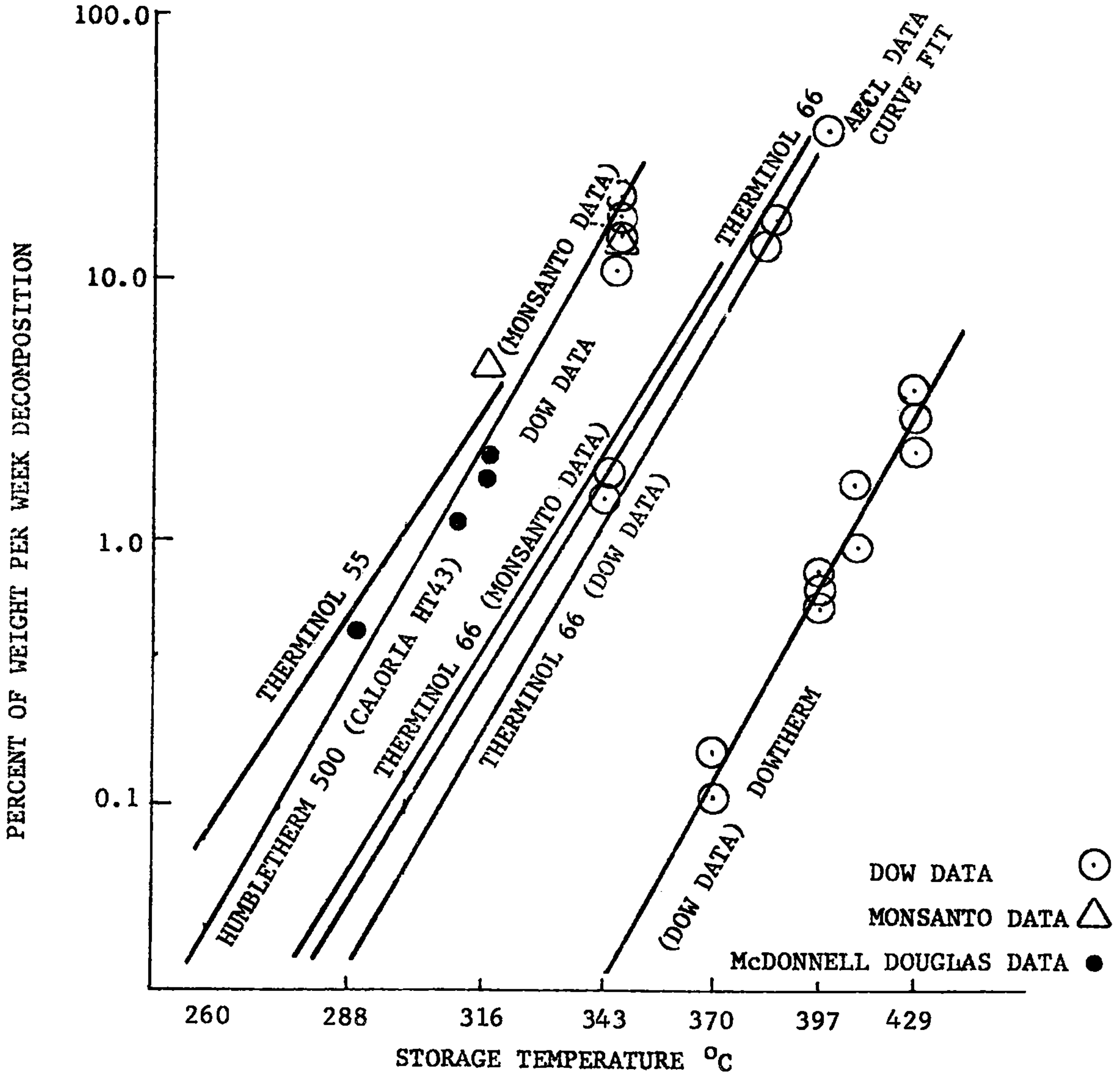

FIGURE II-1 Oil Decomposition Rate vs Temperature.

in determining accurate decomposition rates.

B. TEST OBJECTIVES

The test objectives in this program were to evaluate the thermal decomposition rate for two systems held at a constant temperature of 316°C (600°F): (1) oil in a 379 liter (100 gal.) storage tank with venting when the tank pressure reached 276 kPa (40 psig) and (2) oil in a 379 liter (100 gal.) vented storage tank in which a sidestream was drawn and processed through a vacuum distillation column to remove decomposition products.

C. APPROACH

As previously described, the program was accomplished in two phases of effort. In each program phase, weekly samples of bulk oil and processor products were taken to chemically evaluate their compositions. The chemical tests were conducted jointly by Martin Marietta and Sandia-Livermore to determine: (1) changes in the boiling range of the oil samples using gas chromatograph (GC) distillation, (2) engineering physical properties of viscosity and density, and (3) changes in oil composition using gel permeation chromatograph (GPC) and gas chromatography - mass spectrometry (GC-MS) for molecular weight distribution, IR and NMR spectra, iron, chloride, and acids concentrations.

During each program phase, detailed accounting of mass flows were recorded for accurate determination of oil replenishment rates. Combined with the chemical analysis, oil degradation rates were calculated.

III. TEST DESCRIPTION

The test objectives in this program were to evaluate the thermal decomposition rate for two systems held at a constant temperature of 316°C (600°F): (1) oil in a 379 liter (100 gal.) storage tank with venting when the tank pressure reached 276 kPa (40 psig) and (2) oil in a 379 liter (100 gal.) vented storage tank in which a sidestream was drawn and processed through a vacuum distillation column to remove decomposition products. Shown in Figure III-1 is a photograph of the actual test setup including both the vacuum distillation column on the left and the storage tank on the right. The complete system is shown in Figure III-2 as a simplified schematic indicating the oil stream flows and processor thermocouple locations of the Phase II test setup. The Phase I test apparatus consisted of only the tank without the sidestream processor. Complete test schematics are presented in Appendix A. "As run" test procedures for both phases of effort are included in Appendix B.

A. PHASE I

A photograph of the Phase I test apparatus is shown in Figure III-3. The storage tank had a 379 liter (100 gal.) volume and consisted of a 61 cm (24 in.) diameter cylinder 0.95 cm (3/8 in.) thick and 107 cm (42 in.) high. The tank was constructed from mild steel and was ASME certified for operation at 690 kPa (100 psig) at 427°C (800°F). The tank heating system consisted of six 500-watt band heaters mounted to the external surface of the tank plus two 500-watt heaters, one each on the tank nozzles. The power to these heaters was manually controlled from variable voltage power supplies. The tank was instrumented with eight thermocouples. Three on the cylindrical section of the tank fed into an over-temperature control system that removed all power from the system if actuated. An air-motor driven stirrer mounted

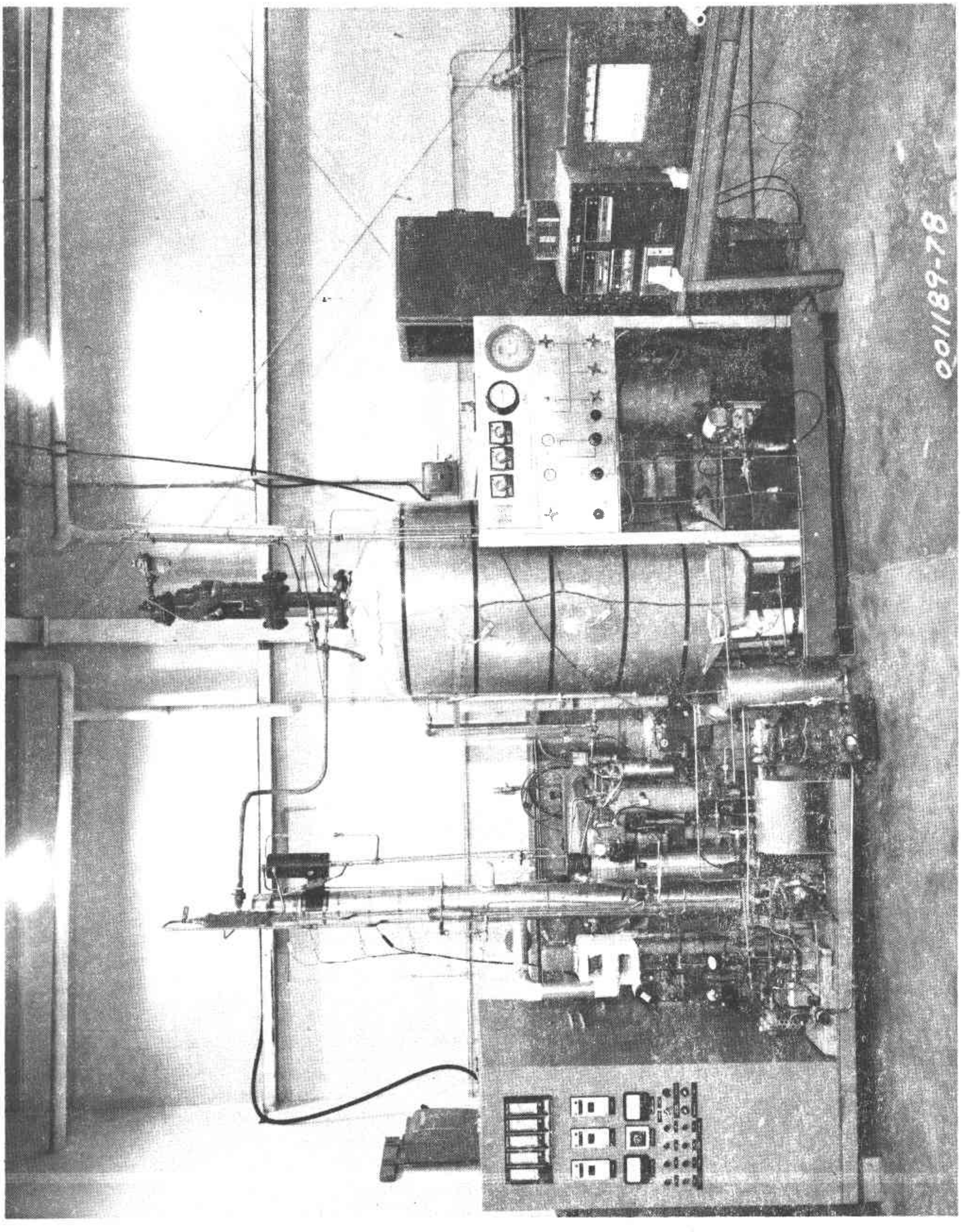

FIGURE III-1   PHASE II SYSTEM PHOTO

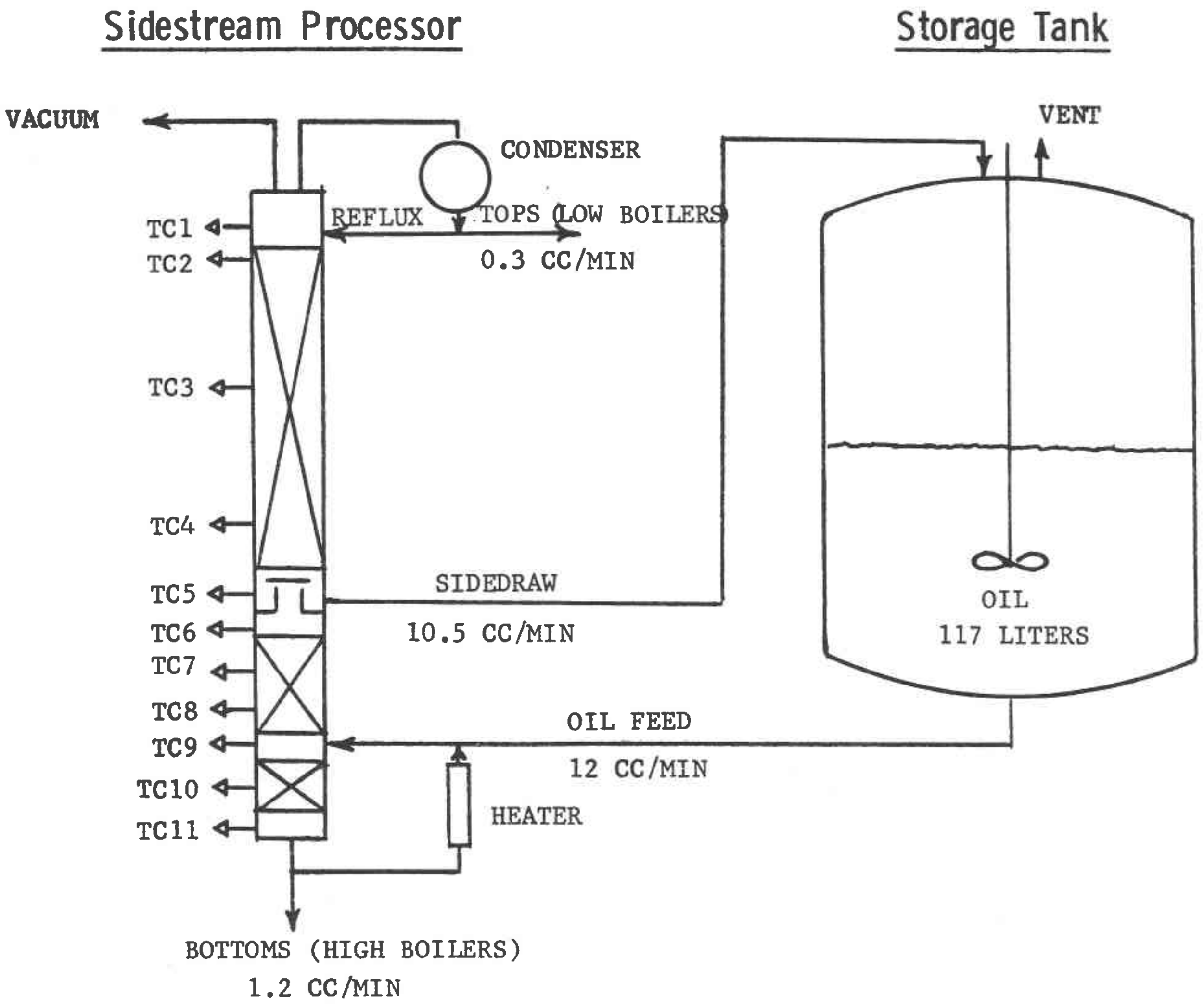

FIGURE III-2 PHASE II SYSTEM SCHEMATIC

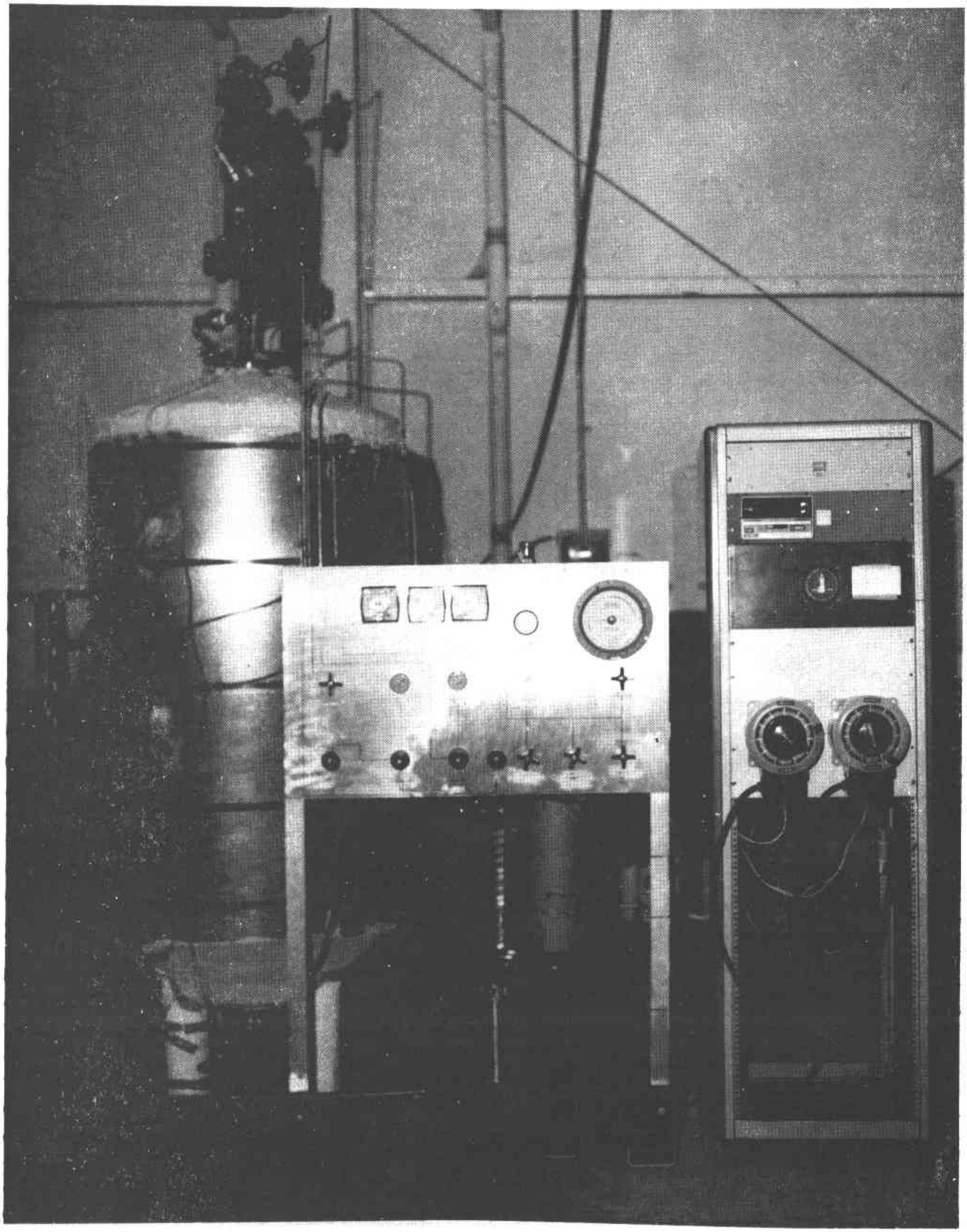

FIGURE III-3 Phase I Test Setup

on the top tank nozzle kept the oil sample isothermal and of homogeneous composition.

During Phase I, the tank was loaded with 143 Kg (316 lb) of oil and then evacuated to degasify the oil. The tank ullage was backfilled with GN_2 and the tank and oil temperatures were elevated to 316^oC (600^oF) and maintained at this level throughout the test. The tank was sealed throughout most of the 1000 hour test period except for 3 vents which occurred when the ullage pressure reached 276 kPa (40 psig). Shown in Figure III-4 is a history of the tank pressure during Phase I. The first reduction in pressure occurred at 550 hours into the test when a leak developed in the packing gland on the stirrer motor shaft, causing an uncontrolled pressure vent from the tank. Following this event the packing gland was adjusted and the pressure in the tank was vented in a controlled manner when the pressure reached 276 kPa (40 psig). During these controlled vents the ullage gases passed through a heat exchanger which collected all ambient temperature condensibles. Gas that was not condensed was metered. Weekly oil samples were extracted from the tank using an evacuated oil sampling device that allowed a hot oil sample to be taken without exposing the oil to the air. A series of chemical tests was conducted jointly by Martin Marietta and Sandia Laboratories-Livermore to determine: (1) changes in the boiling range of the oil samples using gas chromatograph distillation, (2) engineering physical properties of viscosity and density, and (3) changes in oil composition using gel permeation chromatography (GPC) for molecular weight distribution, IR and NMR spectra, iron, chloride, and acids concentrations.

During the six-week Phase I test period, a detailed accounting of mass flows from the tank during venting was performed. Tank residual gas pressure at the end of the test was vented through a gas meter to determine volume of gas vented.

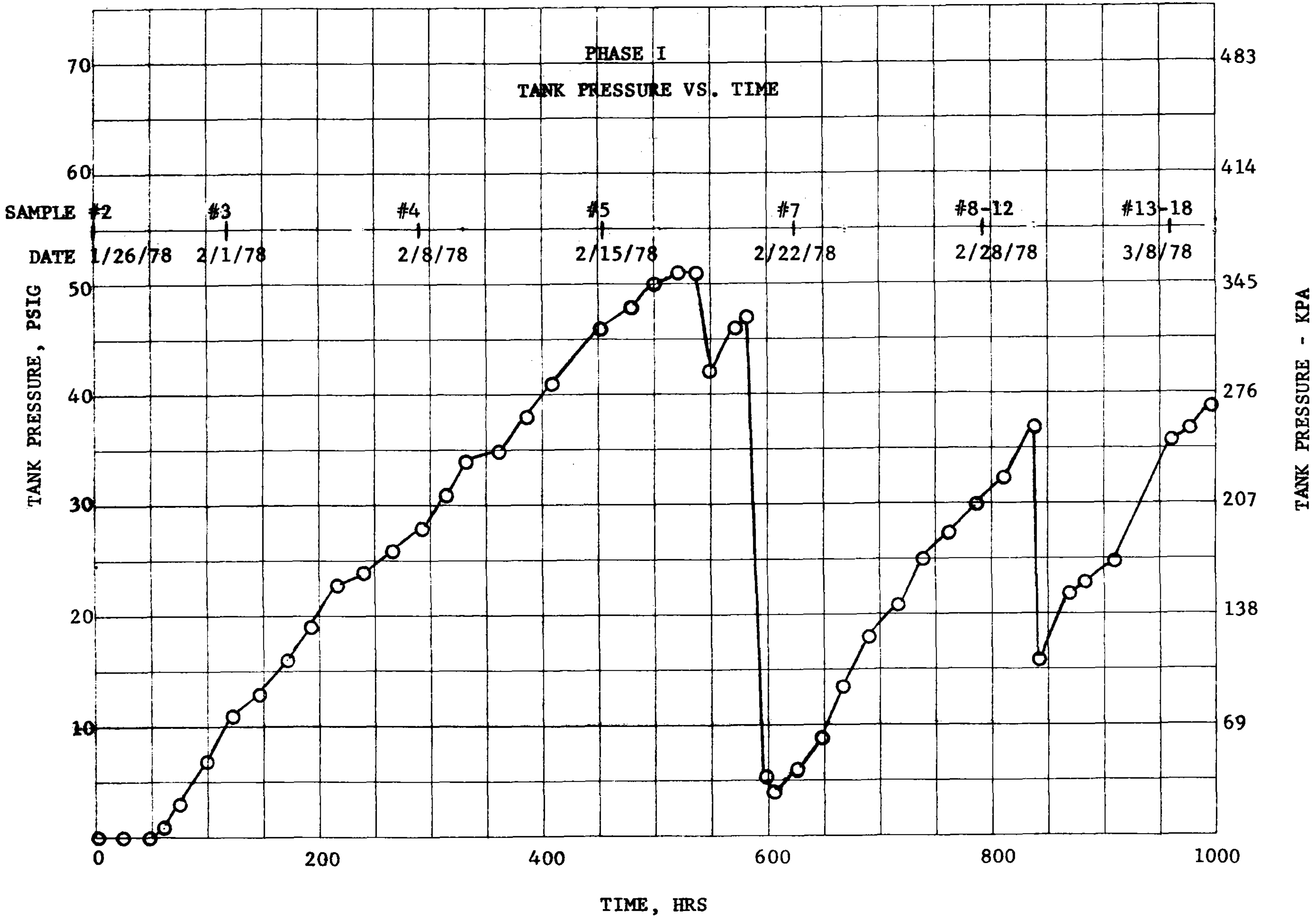

FIGURE III-4

TANK PRESSURE HISTORY

Condensibles were trapped in an ambient temperature heat exchanger and carefully weighed. Careful accounting of system mass allowed a determination of oil degradation rate and will be reported later in the test results section (see Section IV.A.2).

B. PHASE II

After Phase I testing, the storage vessel was cleaned with petroleum ether, purged with dry GN_2, and interconnected with the vacuum distillation system (Figure III-1). The vacuum distillation system was designed to operate at 10 torr and removed from the system compounds that either boil above 302°C (575°F) and 10 torr (high boilers) or boil below 143°C (290°F) at 10 torr (low boilers). The distillation column consisted of two 5.1 cm (2 in.) Schedule 40 pipe sections (insulated with 5.08 cm (2 in.) of fiberglass) for a total column height of 226 cm (89 in.). The column was filled with Hyperfill* packing material. High boilers that accumulated in the bottom of the column were heated with a forced circulation heater and mixed with fresh feed to preheat the feed stream. The feed stream entered the column at 50.8 cm (20 in.) above the bottom of the column. Heater power was adjusted frequently during Phase II testing to minimize the buildup of high boilers in the column bottom. A side draw stream located 66.0 cm (26 in.) above the feed point location was sent from the column back to the storage tank. The composition of this sidestream was similar to the fresh Caloria HT-43. Vapor exiting the column at the top was sent through a water cooled condenser and passed to a reflux accumulator. From the accumulator, part of the condensed oil was sent back to the top of the column as reflux and the rest went to a products accumulator. Two wrap-around heaters were installed around the upper and lower sections of the column. External mass flows, temperatures, and

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*Registered trade-mark

column pressures were automatically controlled during the entire test duration.

After an initial 9 week startup period the distillation system began reprocessing 99.3 Kg (219 lb) storage tank oil. This oil had been held at a 316^{o}C (600^{o}F) temperature for three weeks prior to the start of Phase II testing and therefore contained some decomposition products. During Phase II testing the oil in the tank was maintained at 316^{o}C (600^{o}F) and vented. A feed rate of approximately 0.61 Kg/hr (1.348 lb/hr) to the distillation system was maintained throughout the test duration. Weekly samples were taken of the bulk oil in the tank, high boilers, low boilers, and sidedraw return oil from the vacuum distillation system. The same chemical tests were performed on these samples as described under Phase I test description, with the exception of the iron, chloride, and acids concentrations. These analyses were not performed since negligible concentrations were detected in Phase I.

C. CHEMICAL ANALYSIS DESCRIPTION

Chemical analyses were performed on samples of Caloria HT-43 heat transfer oil which had been thermally exposed over a six week period in both Phase I and II tests. The analyses performed on the samples included density, viscosity, iron, chloride, total acid, mean molecular weight by gas chromatography/mass spectrometer and gel permeation chromatography (GPC), gas chromatograph distillation, infrared (IR) spectra, and nuclear magnetic resonance (NMR) spectra. The raw data from these analyses is included in Appendix D while the interpretation of the data is discussed in sections IV.A.2 and IV.B.2.

Iron was extracted from the oil with dilute hydrochloric acid and determined spectrophotometrically by formation of the thiocyanate complex. Chloride was extracted from the oil with hot water and determined by potentiometric titration with silver nitrate. Oil samples were dissolved in 50% V/V toluene/isopropanol and determined by potentiometric titration with alcoholic potassium hydroxide. Infrared spectra of oil were determined from neat thin films run between NaCl cells from 4000-6000 cm^{-1}. For NMR analysis the oil samples were dissolved 50% V/V in deuterochloroform with tetramethylsilane reference. Volatile oil sample fractions were chromatographed directly on a GC/MS and from the resulting mass chromatogram mean molecular weights were calculated.

Sandia Laboratories at Livermore, California was responsible for conducting GC distillation, GPC, and viscosity tests. The GC distillation tests were conducted by both Lawrence Livermore Laboratories and West Coast Technical Service, Inc. Both organizations conducted the distillation tests according to ASTM-2887. The GPC tests were conducted by Sandia Laboratories using Waters Associates Gel Permeation Chromatograph instrumentation, a differential refractometer, and a UV spectrometer as an absorbance detector. Viscosity tests were performed on a Cannon-Ubbelohde viscometer.

An independent analysis of Phase I samples was performed by EXXON Corporation and is included in Appendix D.

IV. TEST RESULTS AND ANALYSIS

Test results from this program and other literature sources indicate that in addition to high temperatures, high tank ullage pressures promote oil decomposition. However, both tank venting and reprocessing will have a beneficial effect in reducing oil degradation.

Oil decomposition rates were determined for both Phase I and II testing. These rate determinations have been based on the system mass balances and chemical analyses. At temperatures of 316°C (600°F) the tests conducted in Phase I under this program have indicated degradation rates lower than previously reported by DOW for sealed systems and higher degradation rates than those reported by Sandia Laboratories-Livermore for completely vented laboratory tests. Under this program in Phase II it has been shown that vented systems with reprocessing have lower degradation rates than unvented systems.

Results from chemical tests indicate thermal cracking, no oxidation and no polymerization of the bulk oil in both Phase I and Phase II. Bulk oil densities in both phases of the tests remained constant. No significant change in iron, chloride, or acids of the Phase I samples was detected. Nuclear magnetic residence (NMR) and infrared (IR) analysis of the vent gas from the tank indicates the presence of olefinic (unsaturated hydrocarbon) groups.

Included in this report section are the engineering and chemical analysis results from both phases of this program. By using computer simulation models, degradation rates were determined and processor performance was evaluated.

A. PHASE I

The purpose of the Phase I test was to generate decomposition data of oil in a test facility that was more representative of an actual storage size application than the laboratory tests previously reported in the literature. The decomposition data generated during the Phase I test served as a baseline for evaluation during the Phase II test when the products of decomposition were removed by a fluid maintenance processor.

1. Engineering Test Results

The Phase I test apparatus, previously described in Section III.A, provided data on oil decomposition characteristics on an engineering scale. Temperatures of the oil in the tank were maintained at a constant level of 316°C (600°F) throughout the test duration (Figure IV-1). The stirrer maintained homogeneous and isothermal conditions of the oil at no more than a ± 1°C (2°F) temperature difference at any location in the oil as measured by the internally mounted thermocouples (TC1, TC2, Appendix A, Figure A-1). Power to the externally mounted band heaters were manually adjusted to maintain a constant temperature. No problems were encountered in maintaining the constant bulk oil temperature as evidenced by the temperature data.

During Phase I tests, detailed accounting of mass flows from the tank was maintained. Shown in Table IV-1 is a tabulation of the initial oil weight loaded into the storage tank and the amount of oil extracted from the tank due to venting and sampling. Sample numbers 12, 15, 17, 20 represent the measured amounts of gases vented from the system during the test. An estimated 4.04 Kg (8.9 lbm) was released from the pressurized tank during an uncontrolled vent. Therefore the total amount of gases released during controlled and uncontrolled venting was 10.42 Kg (22.97 lbm) and represent vented decomposition products. The total test duration of Phase I was 1128 hours. Therefore the oil replenishment rate based on the mass of decomposition products vented was 1.08% per week (22.97 x 168/(316.5 x 1128)). This calculation does not consider the amount of decomposition products that remained in

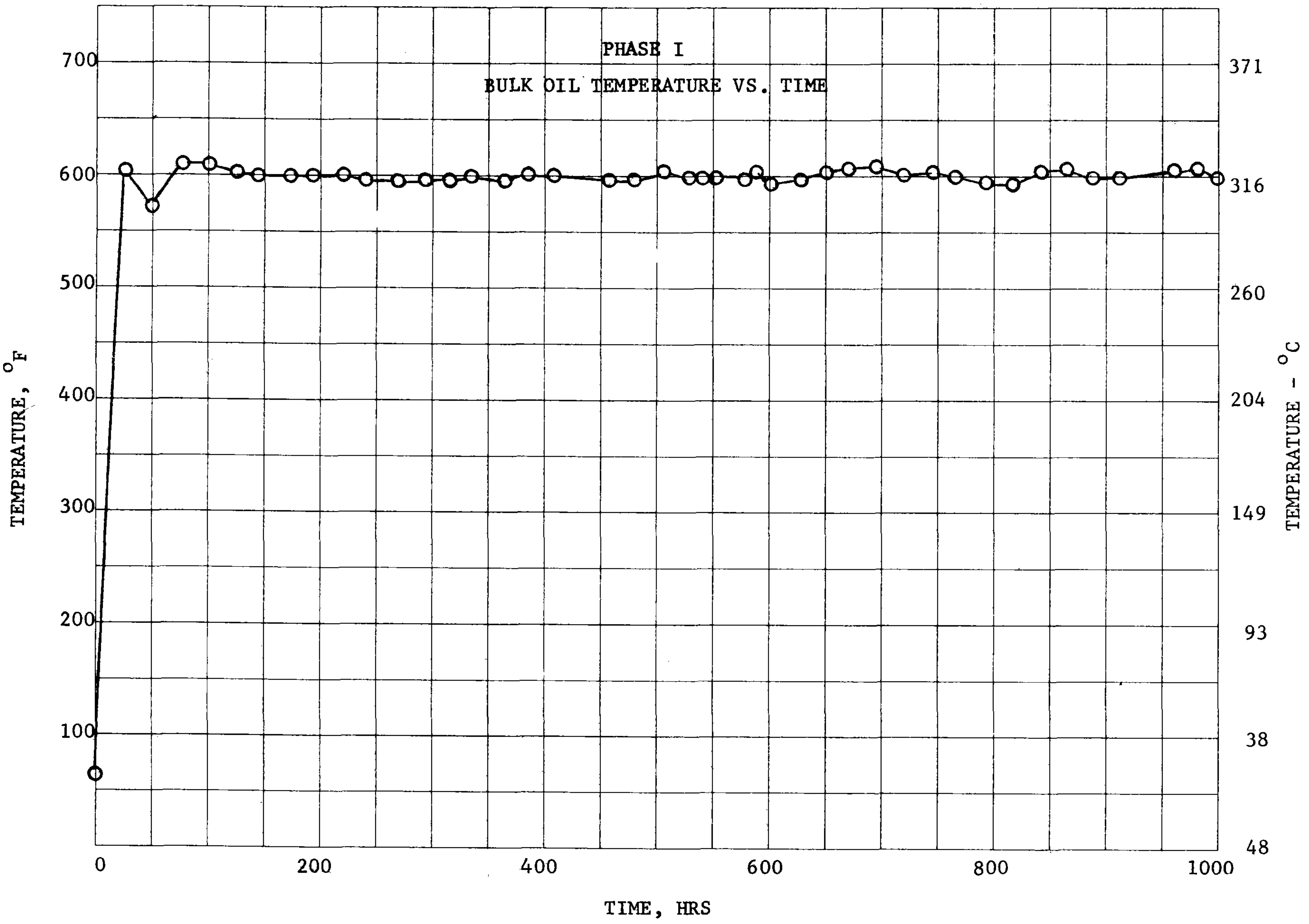

FIGURE IV-1

OIL TEMPERATURE HISTORY

TABLE IV-1

PHASE I OIL WEIGHT DATA

WEIGHT LOADED INTO TANK

Initial Tank Loading	(Kg)	(lbm)
Initial Barrel Wt	195.7	431.5
Final Barrel Wt	52.4	115.5
Total Wt Loaded	143.6	316.5

WEIGHT EXTRACTED FROM TANK

Sample No.	(Kg)		(lbm)		NOTES
1	.26		.58		
2	.28		.63		
3	.41		.90		
4	.83		1.82		
5	.49		1.07		
6	.26		.57		
7	.28		.63		
8	.26		.58		
9	-		-		(1)
10	-		-		(1)
11	.79		1.75		
12	1.16		2.55		(2)
13	.25		.56		
14	-		-		(1)
15	2.17		4.79		(2)
16	-		-		(1)
17	.72		1.58		(2)
18	.57		1.25		
19	.69		1.53		
20	2.34		5.15		(2)
Sample Total		12.01		26.53	
Drain					
Final Barrel Wt	188.9		416.4		
Initial Barrel Wt	61.4		135.3		
Total Wt Unloaded		127.5		281.1	
Uncontrolled Vent		4.1		8.9	
TOTAL		143.6		316.5	

NOTES:
(1) Vent Gas
(2) Vent Condensibles

bulk oil solution. After a calculation of the decomposition product concentration in oil solution, an overall degradation rate was calculated. (See Section IV.A.3).

2. Chemical Analysis

Oil samples were taken from the storage tank and condensate trap throughout the six week test period of Phase I. Chemical analyses of these samples were subsequently performed to determine changes in physical and chemical properties of the oil with time at the 316°C (600°F) operating temperature. Shown in Table IV-2 is a list of chemical analyses performed on the oil samples. Hand valve locations described as "HV-" correspond with those illustrated in Appendix A of this report. The GC distillation, viscosity and GPC tests were performed by Sandia-Livermore. Martin Marietta has been responsible for the other analyses shown in Table IV-2. The analyses for chloride and iron were not completed on all samples when only traces of these elements were detected in the first few samples. The unreduced data from these analyses can be found in Appendix D.

The chemical analysis results obtained in Phase I provided baseline data on thermal degradation of Caloria HT-43 in a closed system. The results of the wet chemical tests are summarized in Table IV-3. The results indicate no significant change in iron, chloride or acid during the time of the test above trace concentrations. Thus, chemical processes occurring in the bulk oil tank are not due to iron or chloride initiated reactions. The absence of acid buildup and lack of C-O absorptions in the IR Spectra during the test period indicate that oxidation processes have not occurred during the test. Figure IV-2 shows that the bulk oil density rose slightly during the first few test days, then leveled off to a constant value for the duration of the test.

TABLE IV-2

OIL EVALUATION PROGRAM SAMPLE ANALYSIS

SAMPLE NUMBER	SOURCE	DATE	TEST PROGRAM STATUS	ANALYSIS STATUS													REMARKS
				MOLECULAR WT. DISTRIBUTION BY GPC	VISCOSITY	GC DISTILLATION		CHLORIDE	IRON	ACIDS	IR	NMR	MOLECULAR WT. DISTRIBUTION BY GC-MS	DENSITY			
A-1	Barrel A	1/18/78				X					X	X					
B-1	Barrel B	1/18/78				X			X		X	X					
C-1	Barrel C	1/18/78		X	X	X		X	X	X	X	X		X			
A-2	Barrel A	1/26/78															Entire sample 5 gal. sent to Quest Int.
1	HV-15	1/25/78															Entire sample held for storage, insufficient for oil analysis. Retake of sample of oil at temperature.
2	HV-15	1/26/78		X				X	X	X	X	X		X			
3	HV-15	2/1/78		X	X			X	X	X	X	X		X			
4	HV-15	2/8/78		X	X			X	X	X	X	X	X	X			
5	HV-15	2/15/78		X	X	X			X	X	X	X	X	X			
6	HV-15	2/16/78				X											Sample taken for storage after pressure loss on test fixture.
7	HV-15	2/22/78		X	X	X			X	X	X	X	X	X			
8	HV-15	2/28/78		X	X	X			X	X	X	X		X			
9	HV-29	2/28/78															Gas sample from vent down tank at 28 psig.
10	HV-29	2/28/78											X				Gas sample from vent down tank at 16 psig.
11	HV-15	2/28/78		X	X	X				X	X	X	X	X			
12	HV-19	2/28/78		X						X	X	X	X	X			Ice water condensibles from vent down.
13	HV-15	3/8/78		X	X	X			X	X	X	X		X			
14	HV-29	3/8/78											X				Gas sample from vent down 38 psig.
15	HV-19	3/8/78		X						X	X	X	X	X			Ice water condensibles from vent down.
16	HV-29	3/8/78															Gas sample from vent down 6 psig.
17	HV-19	3/8/78		X						X	X	X	X	X			Ice water condensibles from vent down.
18	HV-15	3/8/78		X	X	X				X	X	X		X			
19	HV-15	3/13/78				X			X	X	X	X		X			Taken when oil cooled to 121°F-end of Ph. 1
20	HV-19	3/13/78								X	X	X	X	X			From an uncontrolled vent 3/8-13/78. Caught in condenser.

TABLE IV-3

ANALYSES OF CHLORINE, IRON, AND ACIDS

SAMPLE NUMBER	ANALYTE CONCENTRATION			
	CHLORINE	IRON	ACIDS	
	µg Cl/g Sample	µg Fe/g Sample	µg KOH/g Sample[a]	µg Myristic Acid/g Sample[b]
A-1	Test deleted	Test deleted	Test deleted	
B-1	Test deleted	2.3	Test deleted	
C-1	16.7	1.8	0.2	0.8
1	Sample held for storage			
2	17.7	2.3	1.0	3.2
3	11.6	2.0	<0.2	0.8
4	9.0	1.4	<0.5	1.6
5	Test deleted	1.7	<2.0	6.5
6	Sample held for storage			
7	Test deleted	2.3	4.2	13.7
8	Test deleted	2.6	<4.3	<14.2
11	Test deleted	Test deleted	<4.4	14.3
12	Test deleted	Test deleted	4.3	14.3
13	Test deleted	2.1	<4.4	14.6
15	Test deleted	Test deleted	<4.0	13.1
17	Test deleted	Test deleted	4.2	13.7
18	Test deleted	Test deleted	3.6	11.9
19	Test deleted	1.9	<4.3	<14.0
20	Test deleted	Test deleted	4.2	13.8

a. Converted from actual amount of titer (rounded off to range of experimental error).

b. Acids converted to µg of a representative acid.

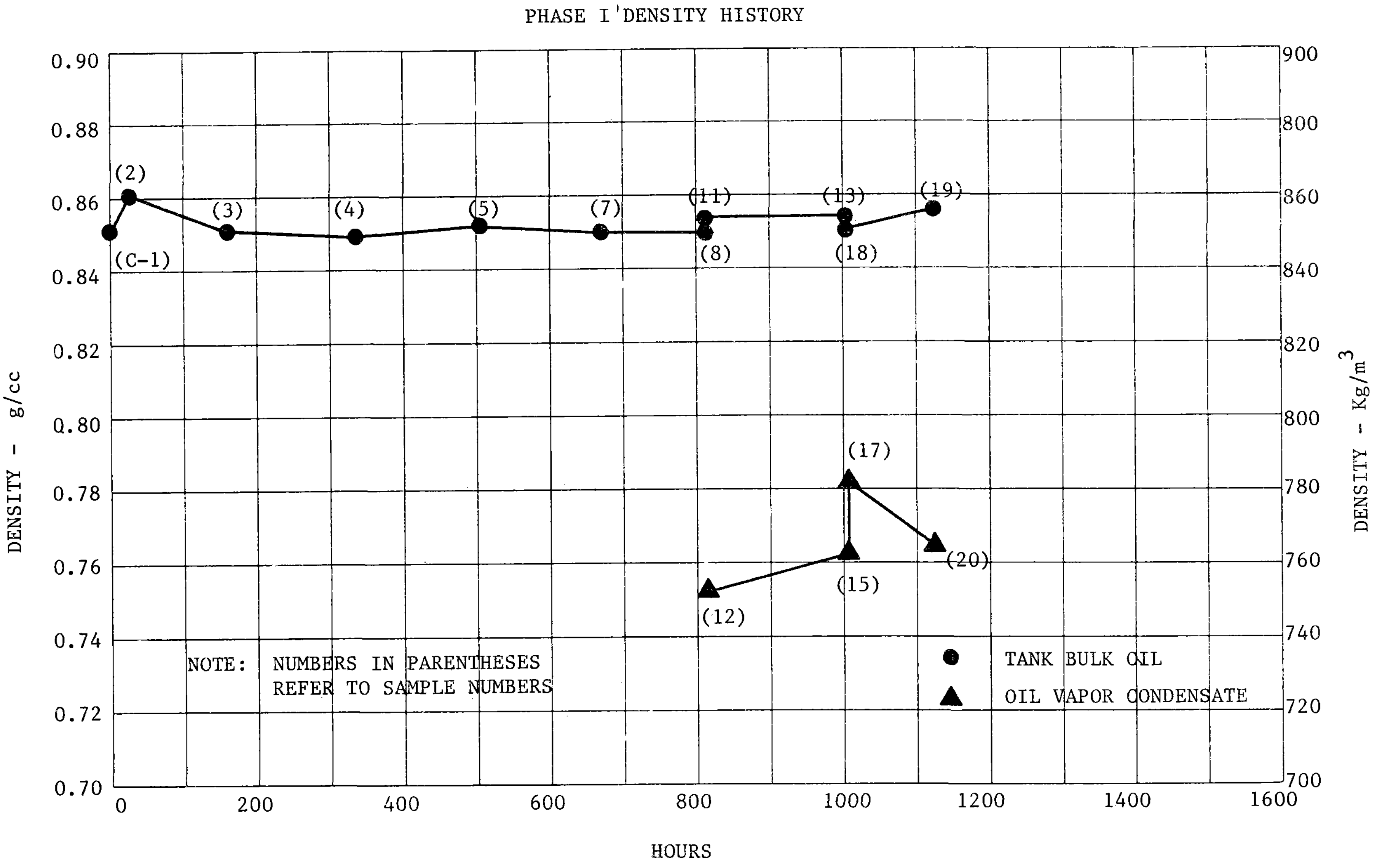

FIGURE IV-2 PHASE I DENSITY HISTORY

The density determinations made on the volatile ice-water condensate samples are characteristic of C_{12}-C_{16} range straight chain hydrocarbons. Undoubtedly, though, some of the condensate consists of aromatic and olefinic type compounds, since the samples exhibited high aromatic odor. The mean molecular weights determined by GC/MS are summarized in Table IV-4 . The mean molecular weights indicate that the vent gas fractions were C_3-C_4 range, the oil volatiles C_4-C_6 range, and the vented ice water condensates C_9-C_{12} range. The condensate molecular weight data and density data point to a fraction that is more complex than a straight chain saturated hydrocarbons mixture.

The NMR and IR spectral data are complementary in terms of ability to indicate formation and disappearance of chemical group types. Both methods are insensitive to structural changes on the order of a percent, or less. Consequently no direct changes are apparent in the bulk oil composition using these techniques. However, examination of the NMR and IR spectra of ice water condensate indicate the presence of olefinic groups as evidenced by NMR absorptions at 4.7 - 5.7δ* and IR absorptions at 1650, 950 and 870 cm^{-1}. Other IR absorptions for alkene type groups are obscured by more intense alkane methyl and methylene group stretching in the 1300 - 1500 cm^{-1} region. There exists some fluctuation in NMR absorptions around 1.8, 2.6 and 3.0δ which are due to formation and destruction of linear alkenes or ring compounds. There is a slight increase in intensity of an IR absorption at 1150 cm^{-1} that may be due to a C-O or cyclopropane stretching vibration. Generally changes in the intensity of the infrared absorption spectra must be viewed with caution, since changes in sample path length due to varying film thickness may account for the intensity changes.

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*δ is parts per million on the NMR spectra

TABLE IV-4

PHASE I

GC/MS MEAN MOLECULAR WEIGHT DETERMINATIONS:

Investigator: M. Barth (MMC)

SAMPLE NO.	TYPE	MEAN MOLECULAR WEIGHT (g/mole)
4	Oil Volatiles	53.8
5	Oil Volatiles	67.6
7	Oil Volatiles	46.5
8	Oil Volatiles	78.1
9	Vent Gas	59.3
10	Vent Gas	43.1
11	Oil Volatiles	69.5
12	Condensate	140.5
"	Condensate	155.6
13	Oil Volatiles	63.1
14	Vent Gas	43.4
15	Condensate	134.0
16	Vent Gas	51.4
17	Condensate	145.3
18	Oil Volatiles	Not Done
19	Oil Volatiles	Not Done
20	Condensate	163.5

Additional information regarding structural changes which occurred during the time of the test can be found by examining Figure IV-3. Figure IV-3 shows the time dependency of the methylene to methyl group ratio as measured by the ratio of the integrated peak areas due to straight chain methyl groups at 0.87δ and methylene groups at 1.20δ. The ratio for bulk oil samples steadily decreases for the first five weeks of the test indicating methylation of double bonds or ring opening followed by methylation, and cracking of long chain compounds. The abrupt increases in the ratio from 860 to 1030 hours reflect the composition of the bulk oil before and after vent of light ends. The ratio increases, because the light ends have been removed from the bulk oil. As expected, the ratio for the ice-water condensates is significantly lower than that for the bulk oil.

An examination of the viscosity of the bulk oil during Phase I (Figure IV-4) shows a gradual decrease in viscosity over the test duration. The lower viscosity indicates cracking of the hydrocarbon oil to lower molecular weight species. The shifts of increased viscosity in the bulk oil after venting show that lower molecular weight species left solution and therefore increased the average molecular weight and viscosity of the oil.

The gel permeation chromatography (GPC) tests illustrate similar trends in the average molecular weight and molecular weight distribution of the bulk oil. Depicted in Table IV-5 is a summary of the GPC results of the bulk oil contained in Appendix D. The samples show a consistent downward trend in carbon number which is indicative of lowering of the bulk oil molecular weight. The shape of the GPC curves for these samples in Appendix D, also show a general flattening with time indicating a increasingly wider molecular weight distribution.

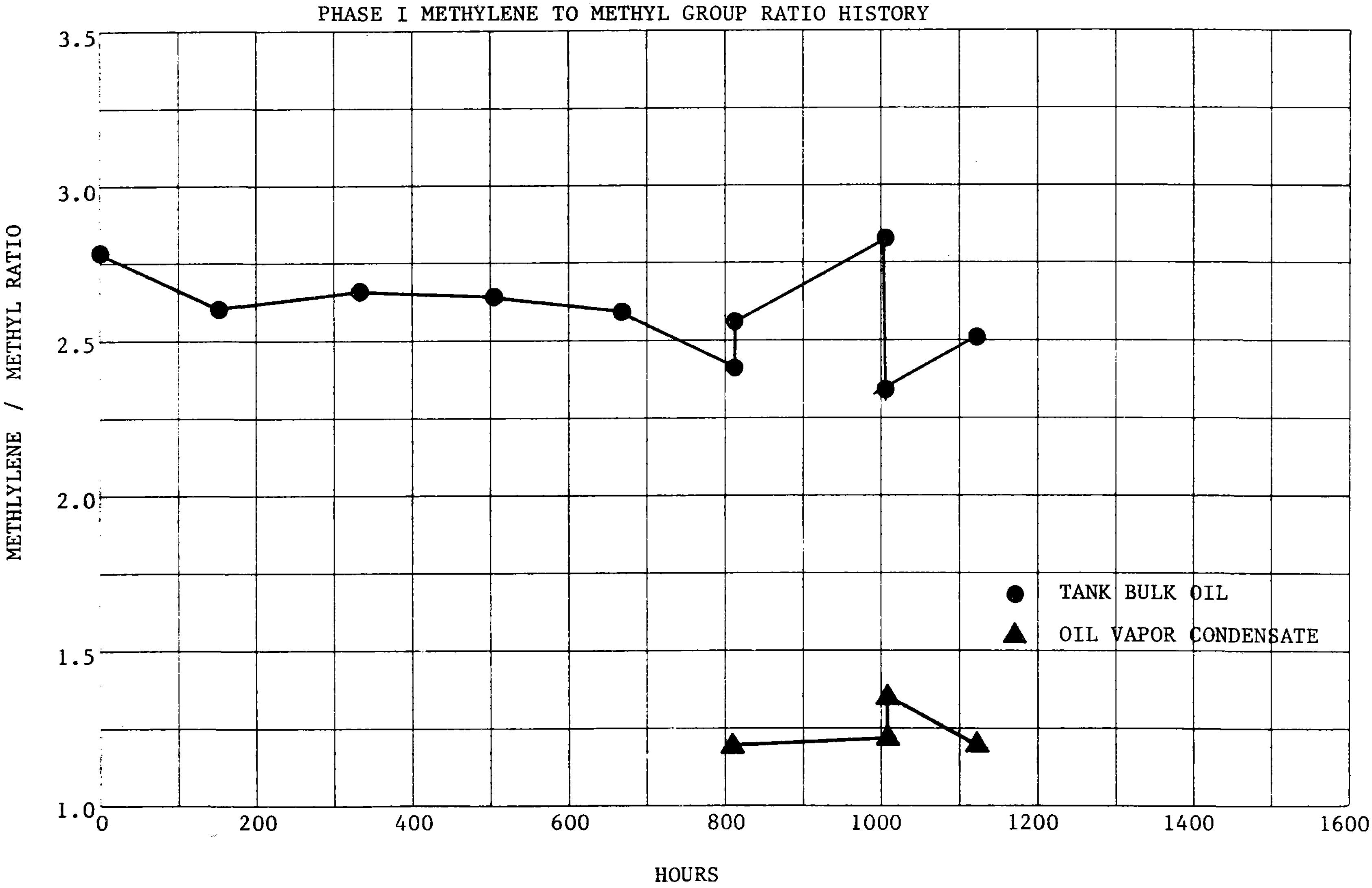


FIGURE IV-3 PHASE I METHYLENE TO METHYL GROUP RATIO HISTORY

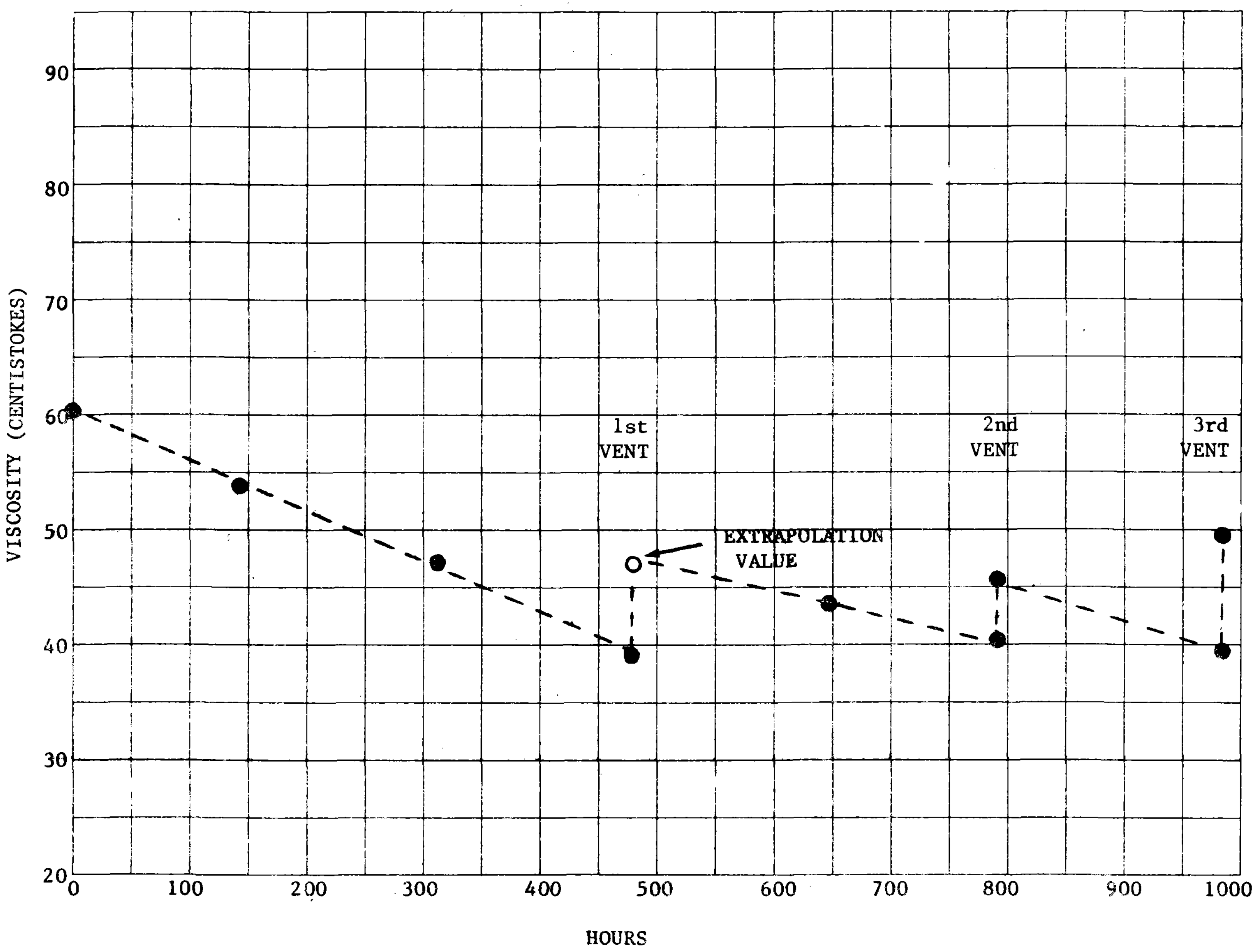


FIGURE IV-4 **PHASE I BULK OIL VISCOSITY HISTORY**

TABLE IV-5

PHASE I GEL PERMEATION CHROMATOGRAPHY RETENTION TIME

(See Table IV-2 for Sampling Times)

SAMPLE NO.	FIRST PEAK UV RETENTION TIME (Min)	CARBON NO.
2	7.76	24.0
3	7.82	23.0
4	7.79	23.5
5	7.82	23.0
7	7.82	23.0
8	7.84	22.5
11	7.82	23.0
13	7.84	22.5
18	7.88	22.0

One of the most informative of the chemical tests performed on the oil samples was the gas chromatograph (GC) distillation test. This test determines the entire normal boiling temperature range for a sample and can therefore be used to quantify the extent of thermal degradation or the separation performance of an oil processor. The Phase I distillation curves, shown in Figure IV-5 , illustrate the effects of venting on oil samples and thermal degradation. Prior to venting, the oil showed a significant increase in low boilers indicated by the curve shift downward from the original fresh oil curve (Figure IV-5) in the 0 to 50 volume percent distilled range. After the first vent, however, low boilers escaped from solution and were vented. Low boilers are still in significant concentration to keep the bulk oil distillation curve below the fresh oil curve even after the vent.

The distillation curves can also be used to evaluate the amount of degradation products in solution. By comparing the original fresh oil distillation curve with the bulk oil distillation curve at the end of the test program **(Figure IV-6), one can determine an additional 10.5% by volume of degraded products. Since** the original fresh oil initial boiling point was 233°C (451°F), then one can determine from Figure **IV-6** that approximately 10.5% by volume of the final bulk oil sample is below 233°C (451°F). One can also determine from comparison of the distillation curves that no higher boiling compounds were generated during Phase I. That is, the final boiling point of the last bulk oil sample is lower than the fresh oil sample indicating slight thermal cracking rather than polymerization of high boiling oil species.

The fraction of oil in the 110-230°C boiling range has a specific gravity of 0.731 and the average specific gravity of the bulk oil sample, determined from a mid-boiling point of 352°C (666°F) is 0.863. The weight fraction of degraded products is

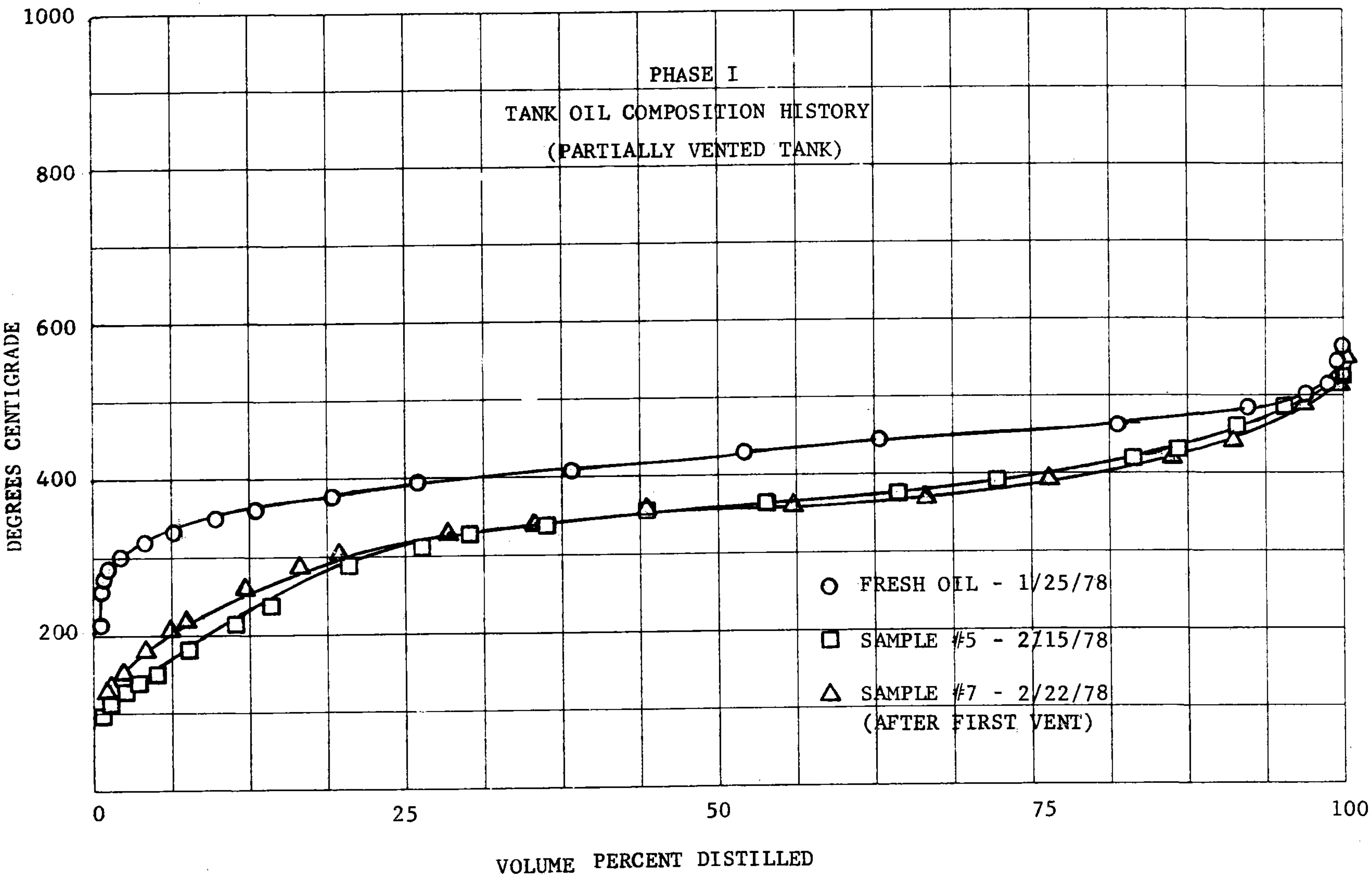


FIGURE IV-5 BULK OIL COMPOSITION HISTORY

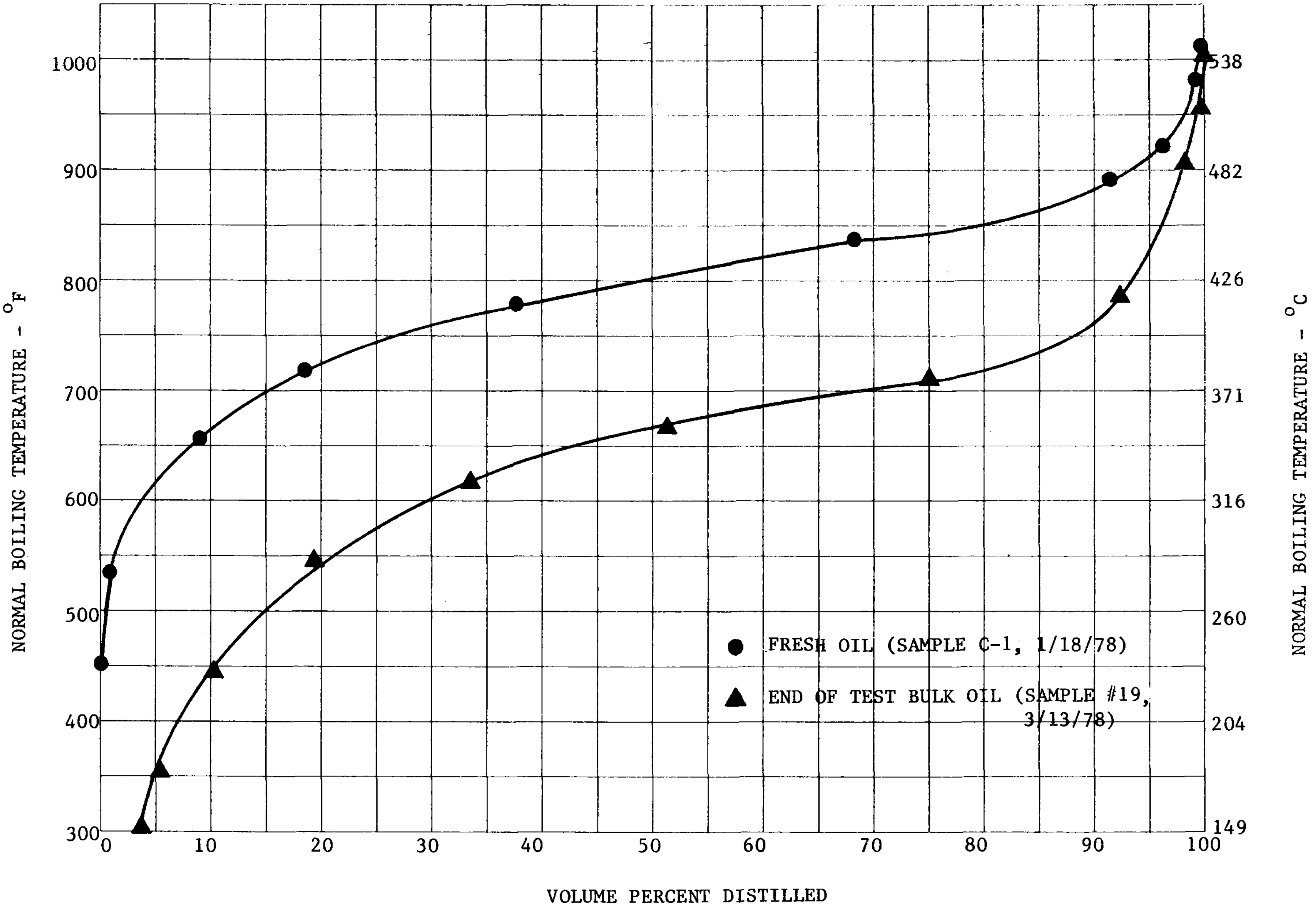


FIGURE IV-6 DISTILLATION CURVE COMPARISON equivalent to 8.89% (10.5 x 0.731/0.863). Averaged over the 1128 hour test duration of Phase I, the degradation rate due to decomposition products in solution amount to 1.32% per week (8.89 x 168/1128).

To summarize, the chemistry data have served to eliminate iron and chloride initiated reactions and oxygenation processes as significant contributors to Phase I oil degradation. Thermal degradation was observed as evidenced by the appearance of low boiling components which were isolated by ice water condensation and found to contain olefinic functional groups. Such changes were not readily discernable in the bulk oil by the analysis techniques employed, because they resulted in formation of decomposition products at fractional percent levels. Further evidence of decomposition was apparent in the visual appearance of the oil with time. During the course of the six week test the color of the oil changes from straw yellow to bluish gray. Some of the samples yielded a cloudy appearance and sediment formation with time, suggesting the formation of higher molecular weight components. Since 1.32% per week of the degraded products remain in solution and the fluid replenishment rate is 1.08% per week as determined from Phase I mass balance, the overall degradation rate of the oil is 2.4% by weight per week at a constant temperature of 316°C (600°F). Dow Chemical tests have shown a slightly higher degradation rate of 2.5% per week for completely closed systems.

3. Analysis of Decomposition Process

Before accurate large scale effects on oil degradation can be predicted, a understanding of the chemical decomposition processes must be determined. Oil test results obtained from Phase I indicate that a contributing factor in oil thermal degradation is autocatalysis. That is, a radical species produced from initial cracking of a hydrocarbon species promotes further degradation of the oil. These radicals have relatively high volatilities compared to the original fluid since they have lower molecular weights resulting from cracking of longer chain hydrocarbons. From phase equilibrium relationships existing between the oil vapor and liquid, one can show that with increasing total pressure above the liquid, the concentration of a highly volatile component decreases in the vapor phase and increases in the liquid phase. Assuming Raoults Law applies (good assumption for systems less than 4.14×10^2 KPa (60 psia)) as well as the assumptions of ideal-liquid solution and ideal gas phase, the phase equilibrium constant, K, can be defined as

$$K_k = \frac{Y_k}{X_k} = \frac{Pvp_k}{\pi} \tag{IV-1}$$

where

K_k = phase equilibrium constant for component k
Y_k = mole fraction of component k in the vapor phase
X_k = mole fraction of component k in the liquid phase
Pvp_k = pure component vapor pressure of component k
π = total system pressure

For a simple binary component system the effect of an increase in total pressure on component concentrations can be examined for the effect of pressure on component concentrations. For a system at constant temperature, assume component 1 has a vapor pressure of 3.45×10^1 KPa (5 psia) and component 2, 3.45×10^2 KPa (50 psia). For a total system pressure of 6.89×10^1 KPa (10 psia), this gives values of $K_1 = 0.5$ and $K_2 = 5$ according to Eq (IV-1). Using the relationship for molar component balances of:

$$X_1 + X_2 = 1 \qquad \text{(IV-2)}$$

$$Y_1 + Y_2 = 1 \qquad \text{(IV-3)}$$

leads to

$$X_1 = \frac{K_2 - 1}{K_2 - K_1} \qquad \text{(IV-4)}$$

Using values previously calculated for K_1 and K_2, results in:

$X_1 = 0.8889$
$X_2 = 0.1111$
$Y_1 = 0.4444$
$Y_2 = 0.5556$

If, however, the total system pressure is raised to 1.38×10^2 KPa (20 psia), then K_1 is 0.25 and K_2 equal to 2.5. The component concentrations become:

$X_1 = 0.6667$
$X_2 = 0.3333$
$Y_1 = 0.1667$
$Y_2 = 0.8333$

Since X_2 represents the concentration of the higher volatile component, then it has been shown that an increase in pressure causes an increase in the liquid phase concentration of the high volatile components.

This thermodynamic treatment can also be applied toward multicomponent hydrocarbon mixtures with the same conclusion regarding the effect of total pressure on component concentrations. Coupled with assumptions of the kinetic mechanism of oil degradation, one can correlate the effects of total pressure on the oil degration processes. Shown in Figure IV-7 is a comparison of the oil degradation rates from various investigations. This data shows that

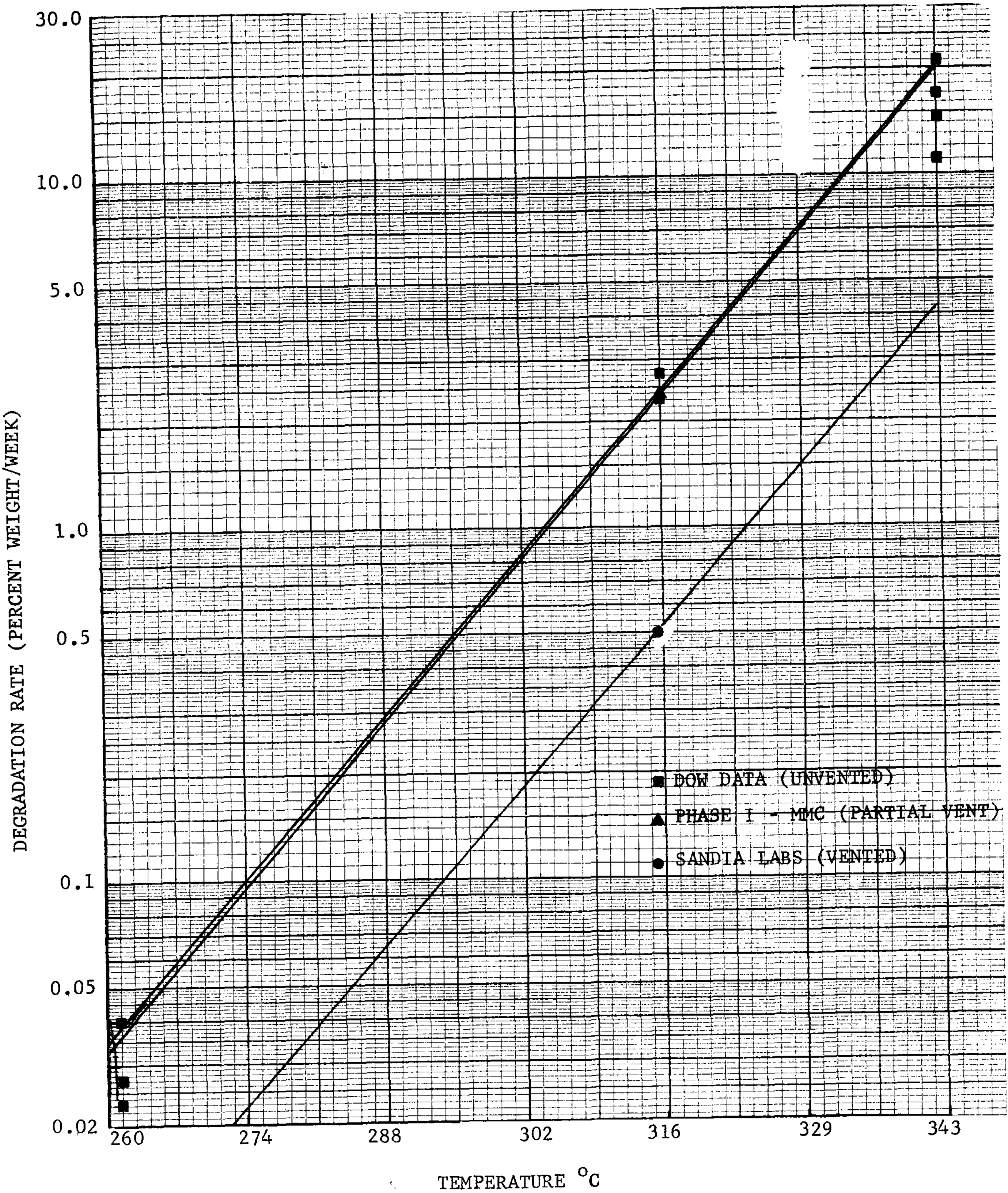


FIGURE IV-7 OIL DEGRADATION TEST RESULTS at a constant temperature of 316°C (600°F), a reduction in vessel pressure reduces the degradation rate. A closed system (Dow Data - closed bomb) exhibits a degradation rate of 2.5% per week or almost 5 times the rate of an open vented system of 0.5% per week. This effect of pressure on degradation rates indicates that the decomposition is brought about in part by autocatalysis. This phenomenon can best be explained by consideration of possible reaction mechanisms. If A is a reacting specie, R is a radical specie from an initial thermal cracking step, and S is a polymer formed from a radical specie and a specie B, then a series of simple reaction steps may be:

[I.] $A \rightleftharpoons R$ (Chain initiation or thermal cracking)

[II.] $A + R \rightleftharpoons R + R$ (Chain propogation or autocatalysis)

[III.] $R + B \rightleftharpoons S$ (Chain termination or polymerization)

The reaction shown by Step I is related to the splitting of the hydrocarbon molecule due to higher vibrational, rotational, and translational motion at high temperatures. The reaction rate of step I, can be expressed as:

$$-r_A = -\frac{dC_A}{dt} = K_I' \, [C_A] \qquad \text{(IV-5)}$$

The reaction rate constant K_I' is a function of temperature. Shown in Figure IV-8 are the rate constants of thermal cracking developed by one investigator for a variety of hydrocarbon compounds. Looking at the gas-oil-API 36 (Caloria HT-43 API = 33) and extrapolating the line to 316°C (600°F) one obtains a value for K_I' of 4.93×10^{-8} sec $^{-1}$. Assuming that 40% by volume of the oil is reactive material, then after integration of Eq (IV-5), one obtains a total oil degradation rate, expressed as moles oil degraded/moles original oil, of 0.64%/week. Note that this computed degradation

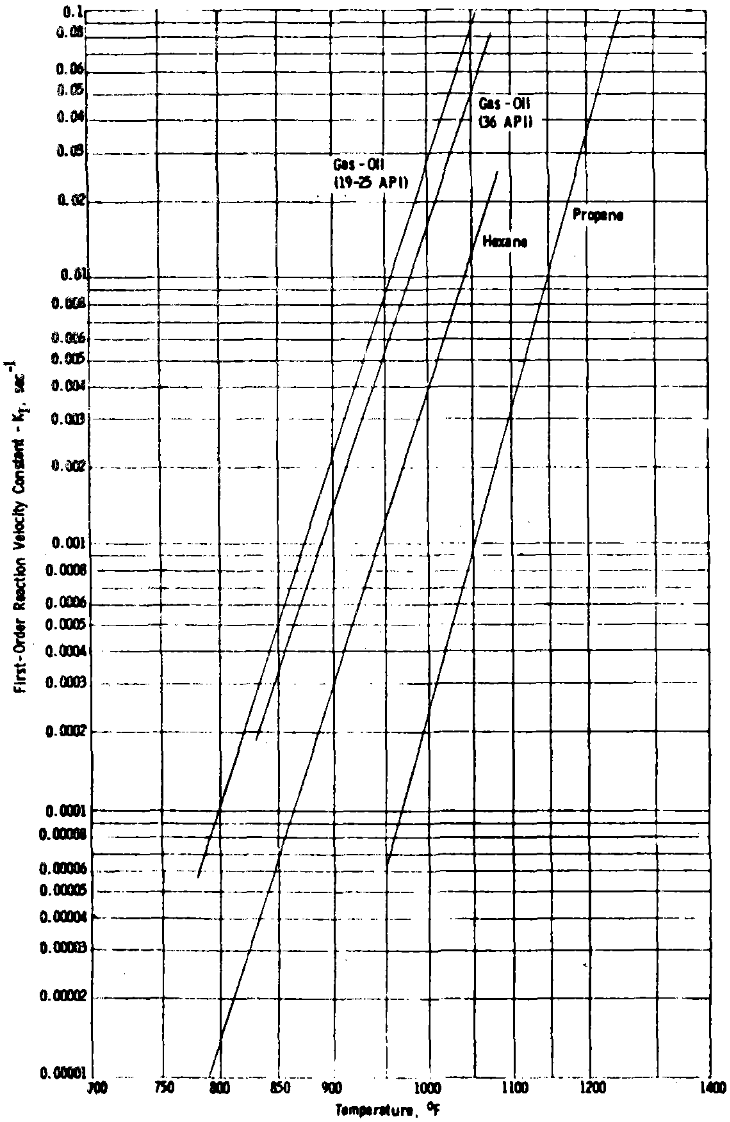


FIGURE IV-8

REACTION VELOCITY CONSTANTS FOR THE DECOMPOSITION OF HYDROCARBONS AND PETROLEUM FRACTION INTO VARIOUS PRODUCTS (REF. 5) is very close to the 0.5% determined experimentally for vented systems by Sandia Laboratories.

The computed rate of decomposition using Eq (IV-5) is dependent on the assumed amount of oil reactive for thermal cracking. A close examination of the GC distillation curves determined on Phase I oil tank samples indicates that only 40 to 50% of the oil (in the lower boiling range) has reacted. A comparison of samples, numbers 5 and 7 in Figure IV-9, show that these distillation curves are markedly different up to 40% distilled where the curves become identical. The curves indicate that 40% by volume of the oil is decomposing in the lower half of the boiling range.

The second reaction step is an autocatalytic step and is usually expressed as a second order reaction. The rate of disappearance of reactive species according to reaction step II can be written as:

$$-r_A = -\frac{dC_A}{dt} = K'_{II}\left[C_A\right]\left[C_R\right] \qquad (IV-6)$$

One should note that this reaction does not start until reaction step I has evolved some reactive radical species, R. Eq (IV-6) also states that this reaction will not be appreciable until the concentration of R, C_R, become significant. When this concentration increases, as it will for systems under higher pressure, the decomposition of A will increase because of the added rate of the autocatalytic step II. One other effect of the reaction step II indicates that the rate of disappearance of A will achieve a maximum value and then decline as the concentration of A diminishes.

The third reaction step, expressed as step III, is also a second order reaction. The rate of production of high molecular weight species S can be written as:

$$r_S = \frac{dC_S}{dt} = -\frac{dC_B}{dt} = K'_{III}\left[C_R\right]\left[C_B\right] \qquad (IV-7)$$

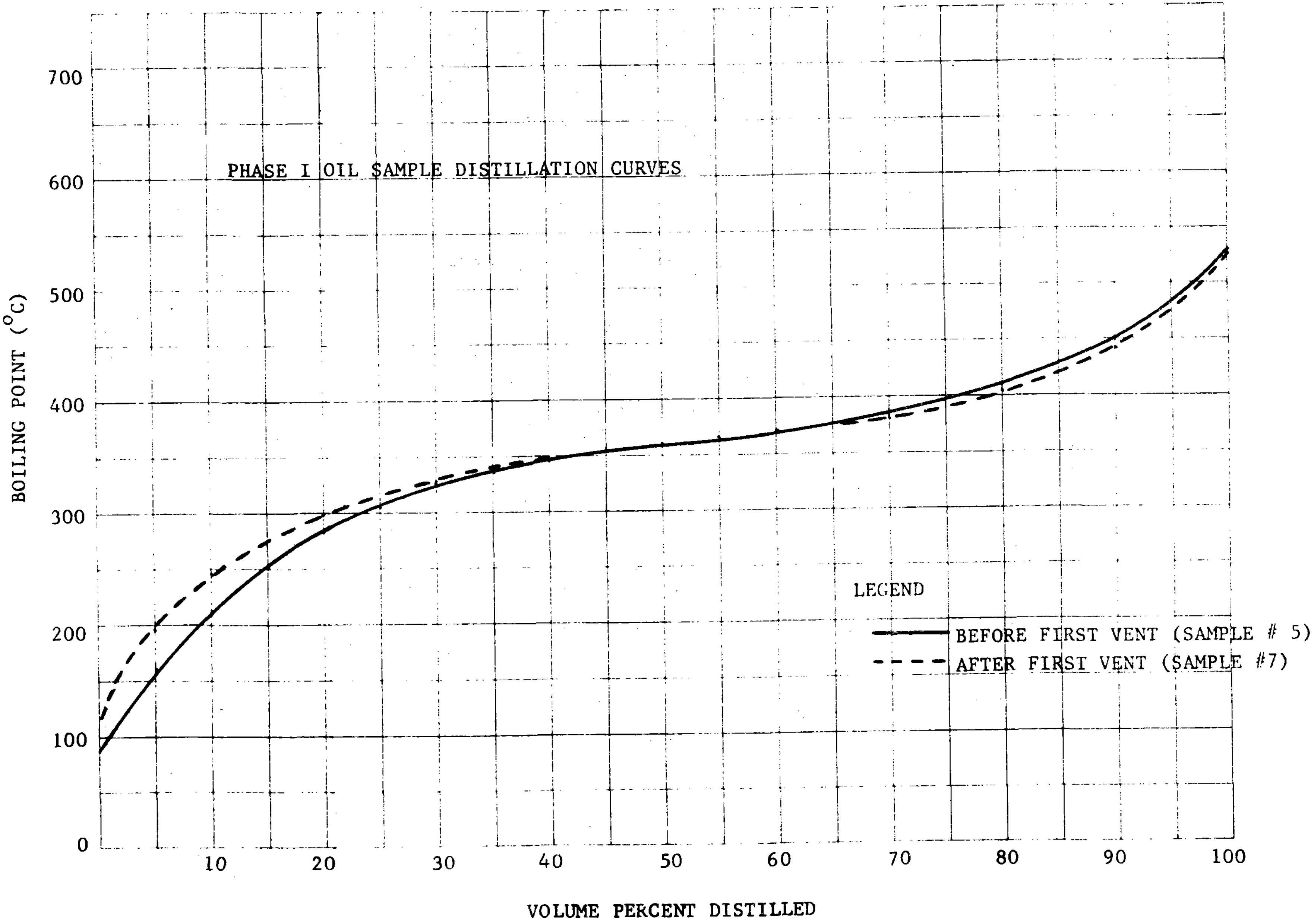


FIGURE IV-9

DISTILLATION CURVE SHIFT DUE TO VENTING

This step is a polymerization step which reduces the concentration of the radical reactive species, R, and hydrocarbon concentration B. This reaction forms high molecular weight species and increases the final boiling point of the oil mixture. Eq (IV-7) also states that the concentration of R must become significant to generate the polymer S. Again the concentration of R in the liquid oil solution will increase as total system pressure increases assuming R is a highly volatile species. To date, no high molecular weight species have been formed in the vented or partially vented tests conducted by Sandia Laboratories or Martin Marietta Corporation. These tests perhaps confirm that the reactive species are highly volatile and that the tests have not been conducted for a long enough period of time or under high enough system pressures to produce high molecular weight species.

If the reactive radical species formed in the liquid phase are impeded from escaping into the vapor phase then their concentrations in the liquid phase increase. These reactive species are most likely very short chain hydrocarbons (methyl, ethyl, or propyl groups). In this case, high tank ullage pressures would keep the liquid phase radical concentrations higher than if the oil were vented. Therefore the higher liquid phase concentrations would increase the degradation rate due to the autocatalytic step. If the assumption of autocatalysis is correct then the presence of rocks, metal, glass beads, etc., may impede these radicals from escaping the liquid oil surface and thus increase degradation. The tests performed by Martin Marietta described in this report and tests performed by Sandia Laboratories-Livermore have confirmed the increased degradation rate due to higher tank ullage pressures or the presence of rock and metal in oil (Figure IV-10). Other speculation of the increased degradation due to rock have been based on the catalytic effects of rock impurities.

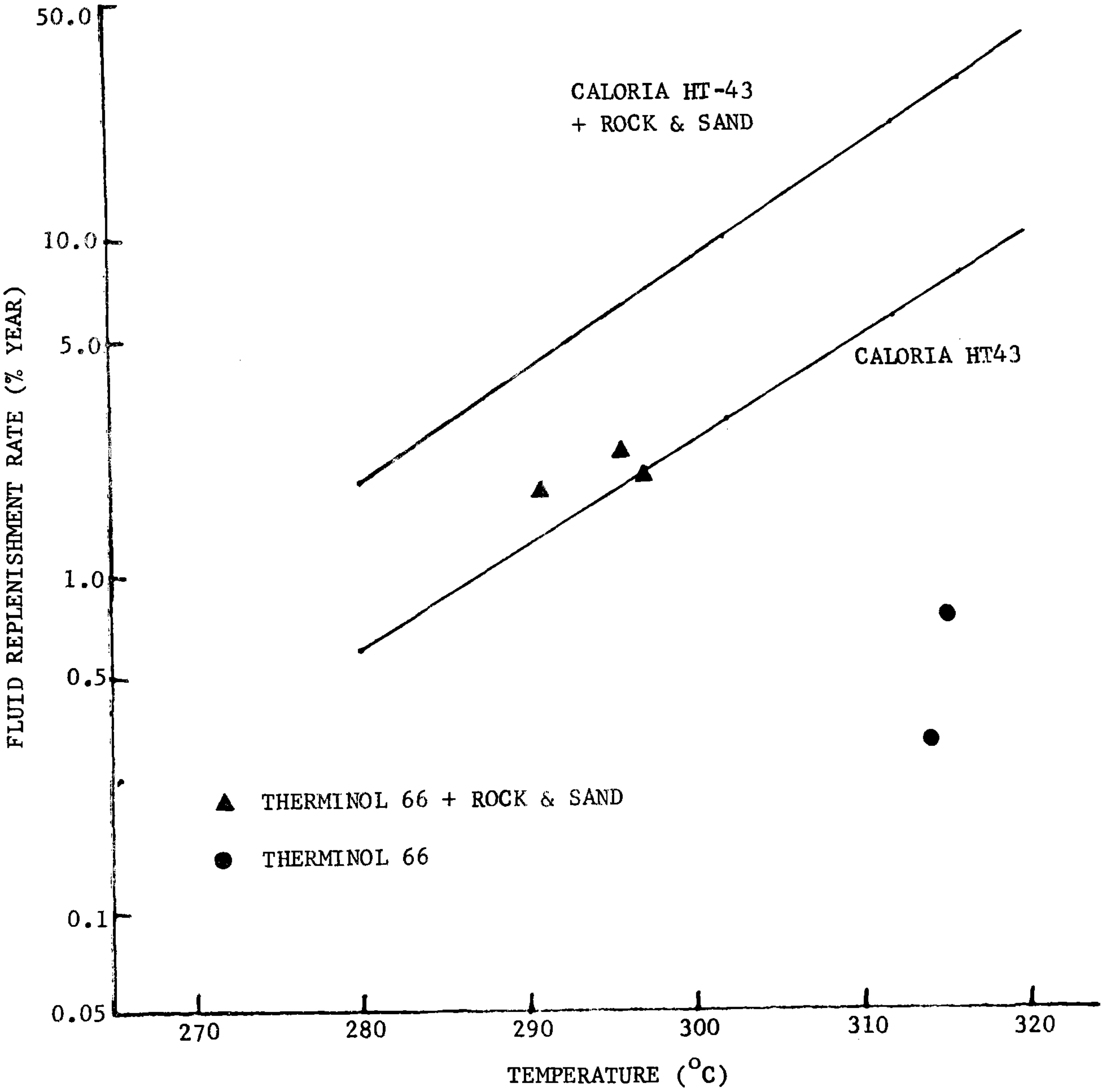


FIGURE IV-10

PREDICTIONS FOR BARSTOW PILOT PLANT YEARLY FLUID REPLENISHMENT RATES FOR SINGLE AND DUAL MEDIA STORAGE SYSTEMS (REF. 6)

Extending what has been learned from tests to date to large scale pilot plant and commercial systems, one may expect higher degradation rates in large systems than those determined for laboratory scale vented vessels. In large scale systems the hydrostatic head on oil in the bottom of storage tanks may keep reactive radicals in solution, thus increasing the oil degradation rate. Large thermocline systems will exhibit higher degradation rates due to either (1) lack of recirculation within thermocline strata (absolutely necessary for thermocline stability), or (2) rocks (dual media system) inhibiting reactive species from escaping the oil solution. However, thermocline movement during charge and discharge cycling will decrease degradation. A pressurized inert gas atmosphere above the solution would also increase oil degradation by not allowing radicals to "breathe" from the oil surface.

However, understanding that the decomposition process is brought about in part by autocatalysis, one can minimize thermal degradation through various techniques. Venting the oil and reprocessing the oil would have beneficial effects in reducing degradation. Stirring the oil, where possible, would promote oil recirculation, releasing radicals to the vapor phase and minimizing degradation.

B. PHASE II

The Phase II test was conducted to determine the effect of continuous reprocessing of the storage oil on degradation rate. A reduction in degradation rate with reprocessing would support the autocatalytic hypothesis of oil degradation described in the previous section. The test apparatus, described in detail in section III.B , consisted of a vacuum distillation column fed by a sidestream from the same storage tank used in Phase I.

1. Engineering Test Results

The Phase II test apparatus with the sidestream processor was more complex than the tank only system tested in Phase I. Some processor and tank temperature control problems were encountered during the test period due mainly to the changing mass and composition of oil. The engineering performance of each subsystem, the storage tank and the processor, will be discussed in this section. An assessment of the processor performance utilizing the chemical analyses and simulation results are presented in later discussion in Section IV.B.3.

a. Storage Tank Subsystem - A constant tank bulk oil temperature was more difficult to maintain in Phase II. Shown in Figure IV-10A is a plot of the oil temperatures measured over the Phase II test duration. At approximately 170, 290 and 360 hours into the test a high temperature kill on the heater controllers was actuated causing loss of power to the tank heaters. This loss of power was the cause of the bulk oil temperature drops shortly thereafter. In addition to these problems, a smaller initial mass of oil was in the tank in Phase II, 99.3 Kg (219.4 lb), as compared with Phase I, 143 Kg (316 lb). That is, Phase II bulk oil had a lower thermal capacitance which resulted in the requirement for much tighter thermal control. Also, as the Phase II test proceeded the bulk oil mass became lower due to the extraction of oil from the system by the sidestream processor. Nevertheless, once these

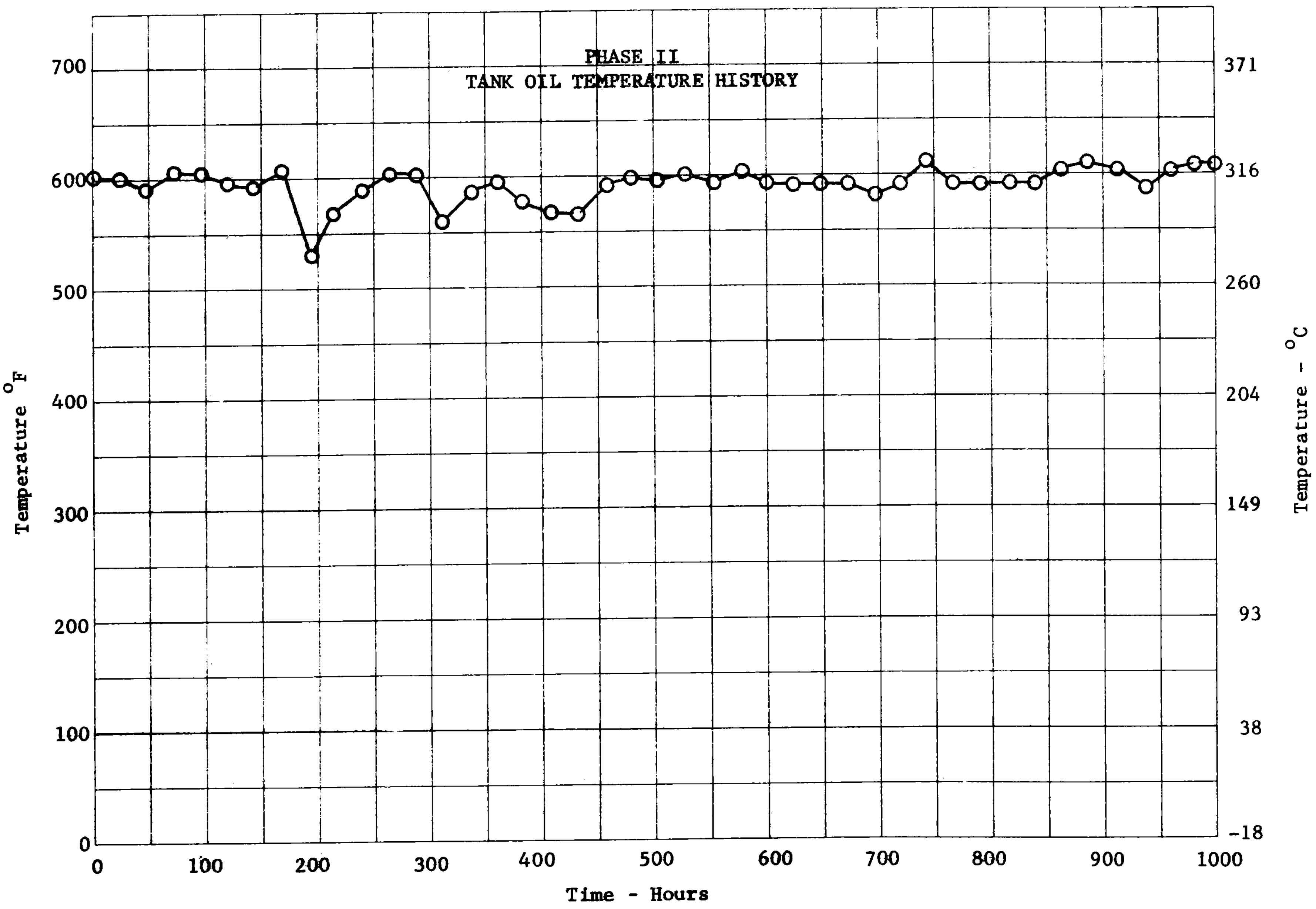


FIGURE IV-10A BULK OIL TEMPERATURE HISTORY problems were understood the bulk oil temperature was controlled to a constant level toward the latter part of the test period.

The Phase II bulk oil initial mass was 99.3 Kg (219.4 lb) and finally 24.5 Kg (54.0 lb). However this bulk oil mass reduction does not represent a requirement for replenishment due to the manner in which the Phase II test was conducted. Shown in Figure IV-11 is the actual bulk oil mass history during the Phase II test. As will be shown in later discussion on processor performance, Section IV.B.3, the majority of the oil separated from the system was nondegraded oil. The high boiling fraction of the oil separated from the system, at the bottom of the column, contained a very high percentage of non-degraded oil (greater than 97.5% by volume throughout the test). This fraction should have been returned to the column. Unfortunately the chemical analysis of this fraction of oil was not performed until the test had been concluded. If this oil had been returned to the tank the reduction in tank mass would not have been as great.

As expected the bulk oil composition changed with time during the test due to both degradation and reprocessing. The compositions of oil as indicated by gas chromatograph (GC) distillation tests are illustrated in Figure IV-12. This composition comparison shows the effects of reprocessing by the upward boiling point shift in the lower boiling range and the downward shift in the upper boiling range. That is, the effect of reprocessing was to flatten the distillation curve of the bulk oil. The effect of reprocessing the oil can also be described by comparing the distillation curves of the fresh oil with the bulk oil at the end of the test. Shown in Figure IV-13 is such a comparison. The two curves show that the initial boiling point is the same for both the fresh and reprocessed bulk oil. The final boiling points are dissimilar because of the extraction of high molecular weight oil components by the processor. By comparing Figure IV-13 with the similar set of curves generated for Phase I in Figure IV-6, one can conclude that the processor achieved a final bulk oil composition closer to the characteristics of the fresh oil. If the high

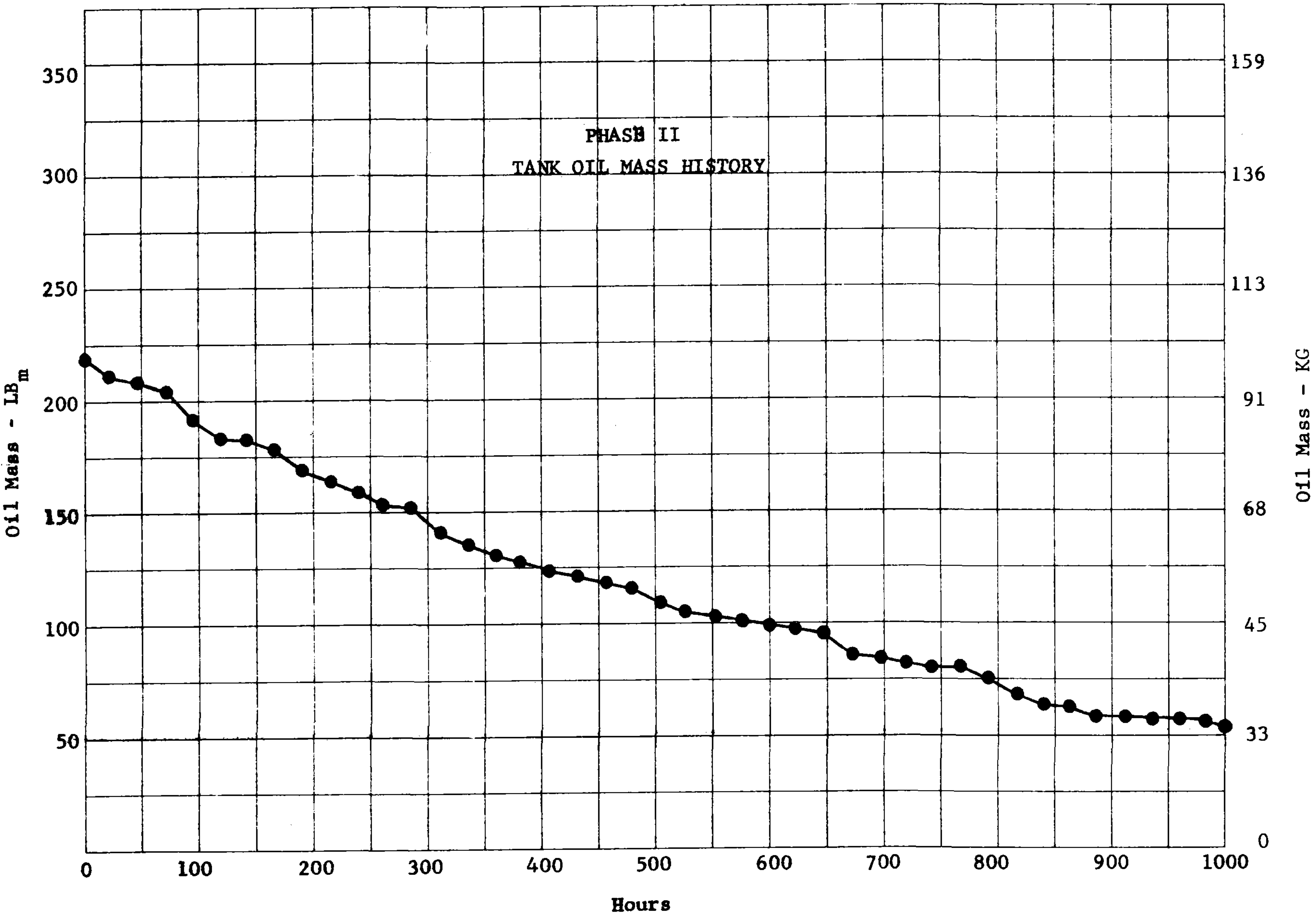


FIGURE IV-11 **BULK OIL MASS HISTORY**

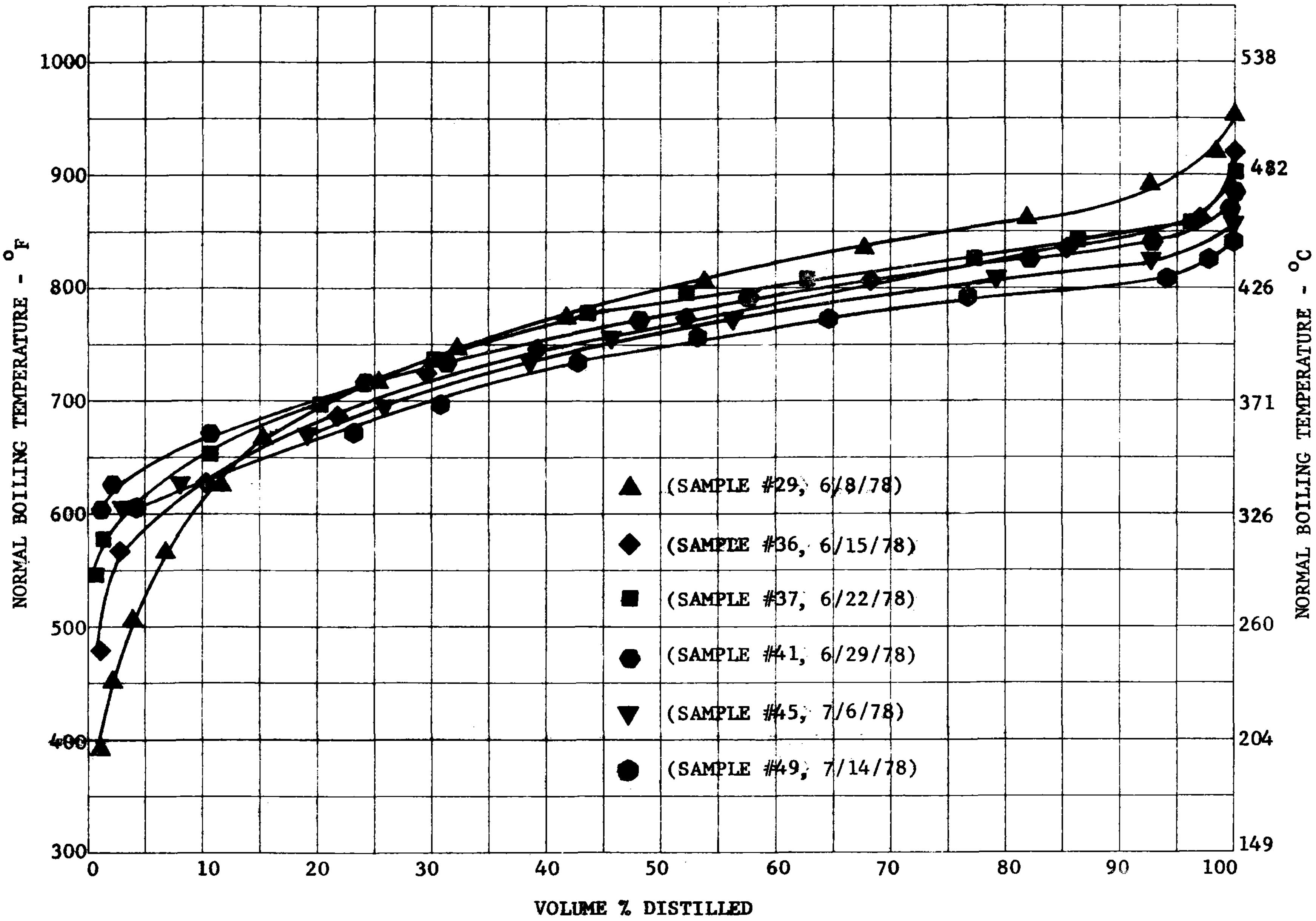


FIGURE IV-12 **PHASE II TANK COMPOSITION HISTORY**

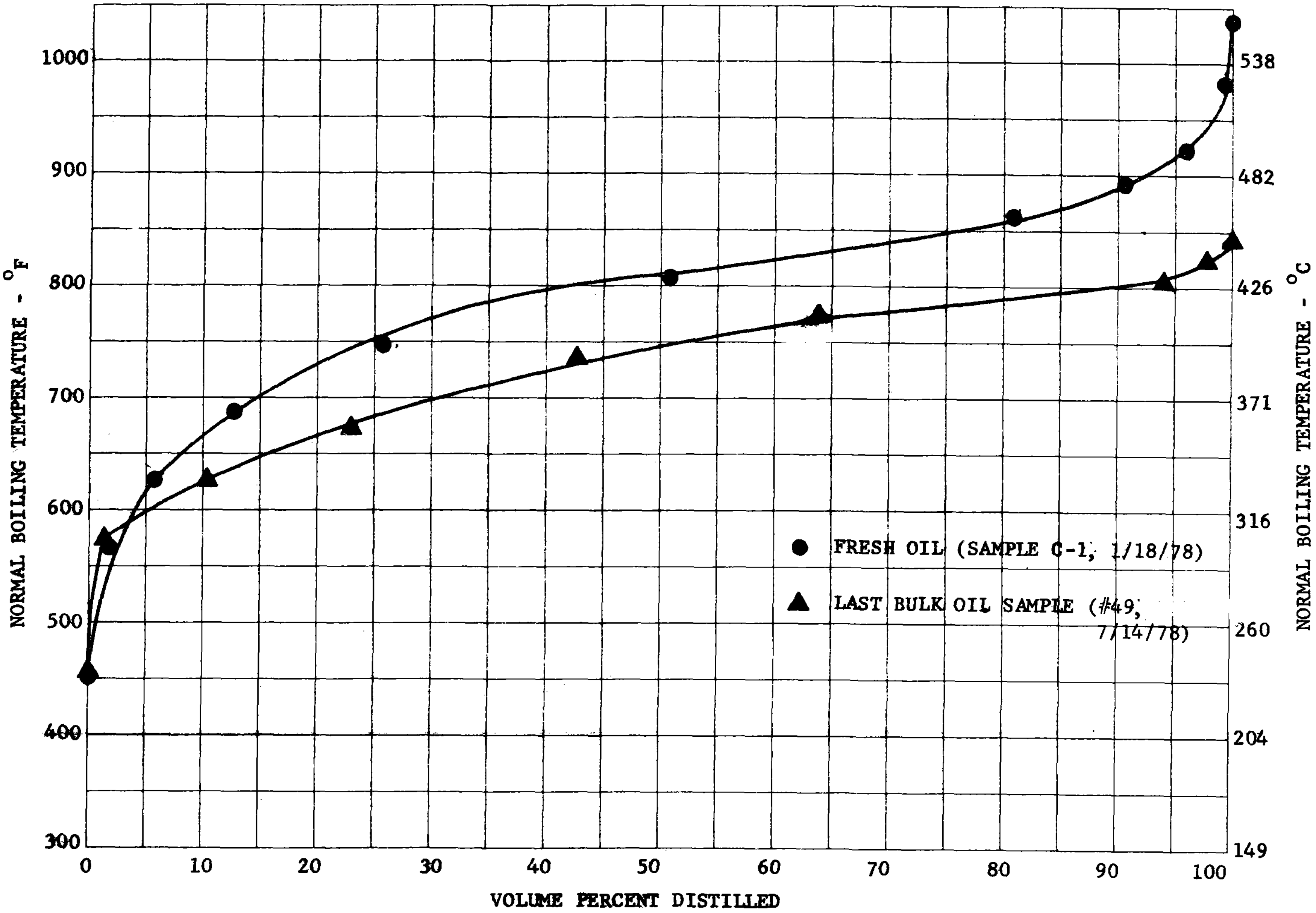


FIGURE IV-13 **FRESH OIL- BULK OIL COMPARISON** molecular weight products extracted by the processor had been returned to the tank, the bulk oil would have had a composition very similar to the fresh oil.

b. Sidestream Processor - The sidestream processor, as described in Section III.B, performed continuously over the six-week test duration, but required close surveillance and monitoring. This requirement was caused by both the changing oil composition fed to the processor and the processor design. A complete assessment of the processor operation is contained in Section IV.B.3 and recommended improvements are listed in Section IV.B.4.

The effect of the changing oil composition fed to the processor can be clarified by noting the change in the column temperature profile. Listed in Table IV-6 are the temperatures at various locations in the column. The feed point location as indicated by thermocouple #9, shows a consistent downward trend during the test and being the highest temperature in the column required to vaporize the feed, reflects the lowering of the final boiling point of the feed mixture. The upper section of the column showed some variability of temperatures which indicates a variation in the reflux flowrate from the tops accumulator. Variation in the reflux flowrate was expected since a gravity feed system without flow control was designed.

During the first and second week of operation, a "wax-like" residue was detected in the sidedraw product stream. This residue plugged a flow metering valve and differential pressure (DP) cell orifice in the processor sidedraw product return line to the storage tank. The orifice was removed from the cell for the remainder of the test since it was not critical to processor operation. The residue is believed to be a hydrocarbon isomer insoluble in the sidedraw oil mixture. The residue was not evident in the bottoms or tops fractions separated by the column. Some residue was noticed in the feed oil but not in the quantities detected in the sidedraw during the first two weeks of operation. After two weeks of operation, the quantity of residue diminished to negligible quantities. An analysis of this residue is included in Appendix D.

TABLE IV-6

COLUMN TEMPERATURE PROFILES

		TEMPERATURES - °C						
		HOURS INTO TEST						
THERMOCOUPLE NUMBER	NOTES	48	216	360	552	720	888	1056
1	Top of Column	135	134	141	148	146	134	129
2		134	139	142	144	149	140	136
3		163	159	158	159	158	153	151
4		168	169	172	172	170	166	168
5	Sidedraw	224	221	225	221	217	209	207
6		258	250	253	242	249	236	237
7		263	259	259	249	254	236	243
8		296	294	292	279	274	263	260
9	Feed Point	313	308	306	287	277	267	266
10		311	306	302	283	274	263	261
11	Bottom of Column	307	299	296	278	270	260	258
12	Pump Inlet	255	233	246	247	234	214	221

TABLE IV-6 (cont.)

COLUMN TEMPERATURE PROFILES

THERMOCOUPLE NUMBER	NOTES	TEMPERATURES - °F HOURS INTO TEST 48	216	360	552	720	888	1056
1	Top of Column	275	274	286	298	294	274	264
2		273	283	287	292	301	284	276
3		325	318	317	319	317	308	304
4		335	337	342	343	338	330	335
5	Sidedraw	435	429	437	430	423	408	405
6		497	482	488	467	481	456	459
7		506	499	499	480	489	456	469
8		565	562	558	535	525	505	500
9	Feed Point	595	586	582	548	530	513	510
10		592	583	575	541	526	506	502
11	Bottom of Column	585	571	565	533	518	500	496
12	Pump Inlet	491	452	475	477	453	418	429

2. Chemical Analysis

The chemical analysis results obtained in Phase II extended the information obtained from Phase I on thermal degradation of Caloria HT-43. In Phase II Martin Marietta chemical analyses were limited to IR and NMR spectrometry, density measurement, and mean molecular weight determination by GC/MS. The baseline data obtained in Phase I was sufficient for iron, chloride and acid analysis. Four sample types were generated in Phase II, namely,

1) hot oil feed from the bulk hot oil storage tank
2) low boilers removed by the distillation system
3) high boilers removed by the distillation system
4) sidedraw recycled into the bulk hot oil storage tank from the distillation system.

Shown in Table IV-7 is a summary of the chemical analyses performed on oil samples collected in Phase II. The raw data obtained from Phase II is compiled in Appendix D. This report will be limited to a discussion of the data obtained after 1 June 1978, which is when the distillation system became fully operational.

One indication of the distillation system's performance can be obtained by considering Figure IV-14. Figure IV-14 summarizes the density history of various samples obtained during Phase II. As in Phase I, the hot oil feed (corresponds to Phase I bulk tank hot oil) was maintained at nearly constant density over the test duration. The average density was 0.844 g/ml as opposed to 0.853 g/ml for the Phase I bulk oil. The method used to measure oil density was reproducible to ±0.002 g/ml. The mean density of the sidedraw is 0.841 g/ml. Removal of small amounts of high and low boilers did not appreciably alter the sidedraw density from that of the hot oil feed. As expected, the density of the high boilers, 0.858 g/ml, is elevated compared to the sidedraw and the density of the low boilers, 0.818 g/ml, is reduced. The

TABLE IV-7

PHASE II OIL EVALUATION PROGRAM SAMPLE ANALYSIS

SAMPLE NUMBER	SOURCE	DATE	TEST PROGRAM STATUS	ANALYSIS STATUS									REMARKS
				MOLECULAR WT. BY GPC	VISCOSITY	GC DISTILLATION	IR	NMR	MOLECULAR WT. DISTRIBUTION BY GC - MS	DENSITY			
21	HV-49	5/11/78					X	X		X			
22	HV100	5/11/78					X	X					
23	HV-15	5/11/78				X	X	X		X			
24	HV-100	5/11/78					X	X		X			
25	HV-15	5/18/78				X	X	X		X			
26	HV-15	5/26/78				X	X	X		X			
27	HV-15	6/1/78		X	X	X	X	X		X			
28	HV-100	6/1/78				X	X	X		X			
29	HV-15	6/8/78		X	X	X	X	X		X			
30	HV-49	6/8/78				X	X	X		X			
31	HV-103	6/8/78				X	X	X	X	X			
32	HV-100	6/8/78				X	X	X		X			
33	HV-103	6/15/78				X	X	X		X			
34	HV-100	6/15/78				X	X	X		X			
35	HV-49	6/15/78				X	X	X		X			
36	HV-15	6/15/78		X	X	X	X	X		X			
37	HV-15	6/22/78		X	X	X	X	X		X			
38	HV-49	6/22/78				X	X	X		X			
39	HV-100	6/22/78				X	X	X		X			
40	HV-103	6/22/78				X	X	X		X			
41	HV-15	6/29/78		X	X	X	X	X		X			
42	HV-49	6/29/78				X	X	X		X			
43	HV-100	6/29/78				X	X	X		X			
44	HV-103	6/29/78				X	X	X	X	X			
45	HV-15	7/6/78		X	X	X	X	X		X			
46	HV-49	7/6/78				X	X	X		X			
47	HV-100	7/6/78				X	X	X		X			
48	HV-103	7/6/78				X	X	X		X			
49	HV-15	7/14/78		X	X	X	X	X		X			
50	HV-49	7/14/78				X	X	X		X			
51	HV-100	7/14/78				X	X	X		X			
52	HV-103	7/14/78				X	X	X	X	X			

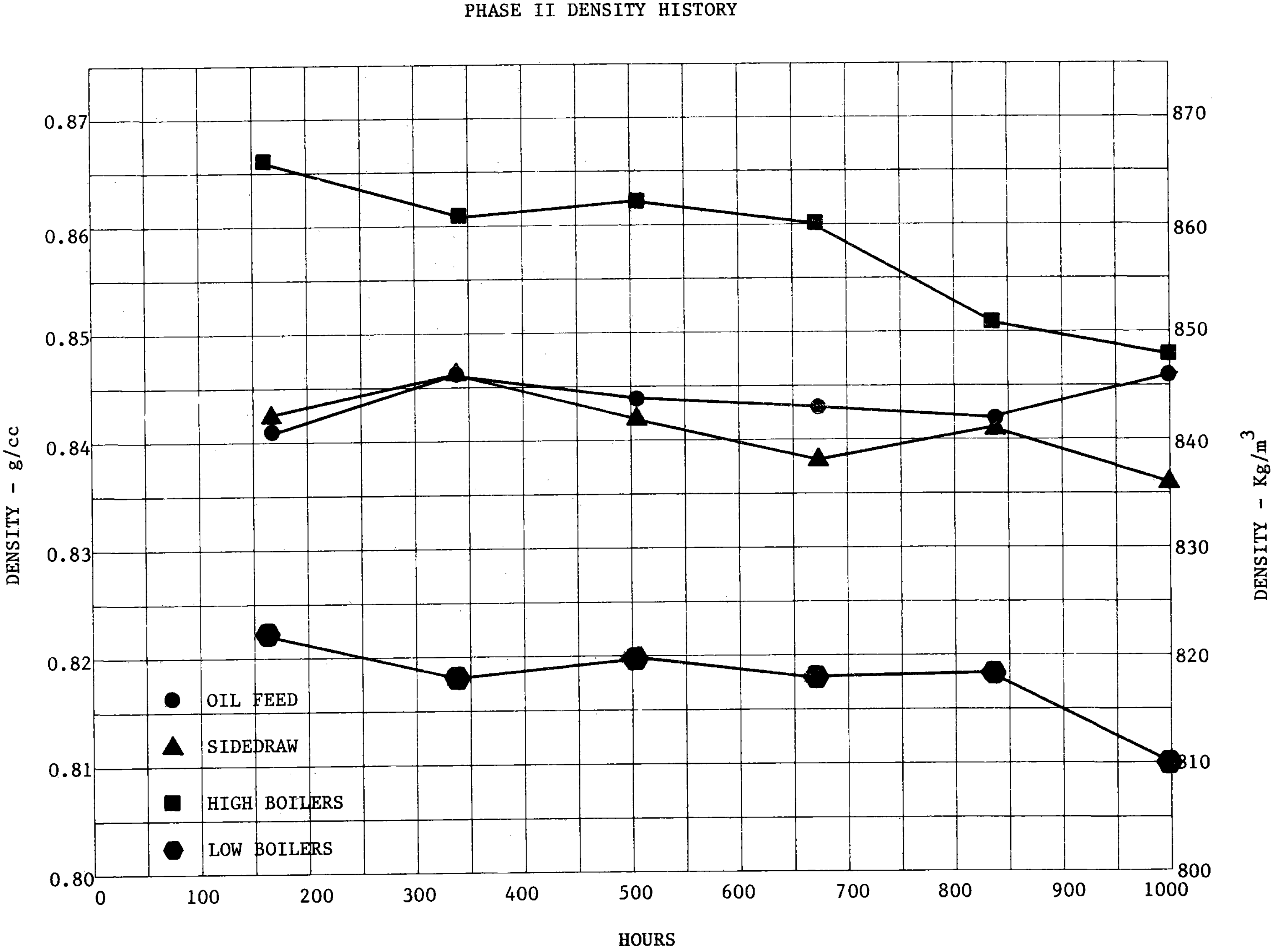


FIGURE IV-14 PHASE II DENSITY HISTORY

observed densities cannot be equated to equivalent saturated hydrocarbon densities, because the densities are reduced by the presence of unsaturated alkenes, cyclics and aromatics. The mean molecular weights of selected low boiler samples are included in Table IV-8. An average mean molecular weight of 202 g/mole indicates that the low boiler fraction was of the C_{14}-C_{15} range. Molecular weights were not determined on other sample types due to their high boiling point range.

NMR spectra of thermally exposed Caloria HT-43 were examined (1) to determine methylene to methyl group hydrogen (proton) ratios and (2) to monitor the appearance or disappearance of hydrogen due to other chemical environments. Table IV-9 summarizes the types of hydrogens found in samples from Phase I. All general types of hydrocarbon hydrogen environments are exhibited in these spectra, including saturated, unsaturated, and aromatic protons. Tables IV-10, IV-11, IV-12 and IV-13 summarize the distribution of various hydrogens in the sample types encountered, namely hot oil feed, high boilers, low boilers and sidedraw return samples.

Methylene to methyl group ratios are broadly indicative of chain breaking and formation processes. The methylene to methyl group ratios were calculated from the integration areas for the peaks having mean chemical shifts of 0.89δ and 1.26δ respectively. These shifts are predominantly due to saturated methyl (0.89δ) and saturated methylene (1.26δ) groups, and thus primarily represent saturated chain lengths. Figure IV-15 shows the time dependent trends in the methylene to methyl group ratios of each sample type. Variability as great as ±0.2 in the ratios was found due to changes in instrument performance between samples, and due to difficulty in interpreting integral areas. As a basis for comparison, the methylene to methyl group ratio for the hot oil feed is nearly constant at about 2.4. From this analysis it is evident that in the distillation process, low boilers (short chain

TABLE IV-8

LOW BOILER MEAN MOLECULAR WEIGHT DETERMINATIONS BY GC/MS

(See Table IV-7 for Sample Times)

SAMPLE NO.	SAMPLE TYPE	MEAN MOLECULAR WEIGHT (g/Mole)
31	Low Boilers	196
44	Low Boilers	220
52	Low Boilers	190

TABLE IV-9

CHEMICAL SHIFTS OBSERVED IN NMR SPECTRA OF THERMALLY EXPOSED CALORIA HT-43

Mean Chemical Shift (δ)	Possible Assignment(s)	
0.38 ± 0.02	CH_4, $-\underset{\vert}{\overset{\vert}{C}}-H$, $-CH_2-$ (methane, alicyclic methine or methylene)	
0.89 ± 0.01	$CH_3-\underset{\vert}{\overset{\vert}{C}}-$ (methyl, sat'd)	
1.26 ± 0.02	$-CH_2-$ (methylene, sat'd)	
1.60 ± 0.04	$-\underset{\vert}{\overset{\vert}{C}}-H$ (methine, sat'd), alicyclic methylene	
1.79 ± 0.02	$CH_3-\underset{\vert}{C}=C<$, alicyclic methylene	
2.18 ± 0.03	$CH_3-C=$; CH_3Ar, alicyclic methylene	
2.62 ± 0.05	Alicyclic methylene or methine $C_6H_5CH_2CH_3$	
3.05 ± 0.05	$H-C\equiv$ (conj.), $-CH_3$ near oxygenated group, alcohol or water $-OH$ alicyclic methylene	
4.73 ± 0.03	$C=CH_2$ (unconj.)	Substituted Ethylenes
5.15 ± 0.07	cis $-C=C$ (conj.)	
	trans $Ar-C=C$	
5.40 ± 0.07	cis $C=C$ (more than 3 conj.) $C=CH_2$ conj.	
7.03 ± 0.05	ArH (benzenoid or nonbenzenoid)	

Table IV-10

HYDROGEN DISTRIBUTION BY NMR - HOT OIL FEED
(See Table IV-7 for Sample Times)

CHEMICAL SHIFT (δ)	% OF HYDROGEN BY SAMPLE NUMBER									
	23	25	26	27	29	36	37	41	45	49
0.38	2.3	2.2	2.7	4.1	3.0	4.2	2.8	3.0	3.1	3.8
0.89	24.7	24.3	24.0	25.7	26.3	25.3	27.3	25.0	24.9	25.2
1.26	59.6	57.0	59.7	58.1	60.0	59.9	60.4	61.9	59.9	60.8
1.60	t	5.1	t	t	t	t	a	t	t	t
1.79	5.7	5.1	5.2	6.5	4.1	5.9	4.9	4.7	5.2	4.3
2.18	4.5	4.9	6.0	3.1	5.3	3.4	3.5	3.4	4.4	3.9
2.62	2.1	≤0.4	1.0	2.0	≤0.4	0.8	0.9	0.8	1.1	1.1
3.05	0.8	0.6	1.2	0.6	≤0.4	0.4	0.6	0.8	0.4	0.4
4.73	a	a	a	a	a	a	t	a	a	a
5.15	a	a	a	a	a	a	t	a	≤0.4	a
5.40	a	a	a	t	t	t	t	t	a	t
7.03	≤0.4	0.5	≤0.4	0.6	≤0.4	0.8	0.4	≤0.4	≤0.4	≤0.4

a. Peak Absent

t. Trace Peak Present

Table IV-11

HYDROGEN DISTRIBUTION BY NMR - HIGH BOILERS

(See Table IV-7 for Sample Times)

CHEMICAL SHIFT (δ)	% OF HYDROGENS BY SAMPLE NUMBER								
	22	24	28	32	34	39	43	47	51
0.38	1.9	2.4	4.0	5.7	6.5	5.7	3.1	3.5	2.9
0.89	22.9	23.1	26.5	29.8	29.8	32.6	24.0	25.5	25.1
1.26	63.9	61.1	58.4	57.0	57.2	54.6	59.6	61.3	61.2
1.60	t	t	t	t	1.1	a	a	a	a
1.79	5.0	5.1	5.3	3.4	0.7	3.7	5.0	4.3	4.5
2.18	4.3	5.2	3.5	2.3	1.9	1.6	4.6	3.6	4.1
2.62	0.9	1.5	1.1	0.4	≤0.5	≤0.4	1.5	1.0	1.1
3.05	0.7	0.8	0.4	0.7	≤0.5	0.5	1.0	0.4	0.7
4.73	a	a	a	a	a	a	a	a	a
5.15	a	a	a	a	a	a	a	a	a
5.40	a	a	a	a	a	a	a	a	a
7.03	0.4	0.7	0.7	0.8	≤0.5	1.0	1.0	≤0.4	≤0.4

a. Peak Absent

b. Trace Peak Present

Table IV-12

HYDROGEN DISTRIBUTION BY NMR - LOW BOILERS

(See Table IV-7 for Sample Times)

CHEMICAL SHIFT (δ)	% OF HYDROGENS BY SAMPLE NUMBER					
	31	33	40	44	48	52
0.38	5.9	6.9	5.6	2.8	2.9	2.6
0.89	33.6	29.3	36.2	28.5	28.8	27.0
1.26	42.9	50.0	43.3	44.4	53.5	54.7
1.60	5.7	4.6	5.6	11.7	4.9	5.2
1.79	4.0	2.4	3.1	t	t	t
2.18	3.9	3.4	2.6	6.7	5.8	5.9
2.62	≤0.4	≤0.4	≤0.5	2.5	0.8	≤0.4
3.05	≤0.4	0.9	1.0	1.0	0.8	1.6
4.73	≤0.4	≤0.6	≤0.5	0.5	0.4	0.5
5.15	≤0.4	t	≤0.5	≤0.4	0.6	0.6
5.40	0.7	≤0.6	≤0.5	≤0.4	≤0.4	0.6
7.03	1.7	1.5	0.6	1.1	0.6	1.0

a. Peak Absent

t. Trace Peak Present

Table IV-13

HYDROGEN DISTRIBUTION BY NMR - SIDE DRAW

(See Table IV-7 for Sample Times)

CHEMICAL SHIFT (δ)	% OF HYDROGENS BY SAMPLE NUMBER						
	21	30	35	38	42	46	50
0.38	4.5	3.7	3.9	5.1	2.9	3.1	3.4
0.89	29.0	32.0	27.0	30.4	25.3	26.9	24.8
1.26 57.1	57.1	56.9	58.8	56.5	58.7	59.8	56.1
1.60	t	a	t	a	t	a	a
1.79	3.6	3.6	4.7	5.1	5.6	5.1	5.6
2.18	2.1	2.5	3.2	1.7	4.3	3.7	3.2
2.62	≤0.5	≤0.4	1.3	≤0.4	2.0	≤0.4	0.8
3.05	≤0.5	≤0.4	0.5	0.5	0.8	0.5	0.6
4.73	a	t	a	t	a	a	a
5.15	a	a	a	a	a	a	a
5.40	a	t	t	t	t	a	t
7.03	≤0.5	≤0.4	0.8	≤0.4	<0.4	≤0.4	≤0.4

a. Peak Absent

t. Trace Peak Present

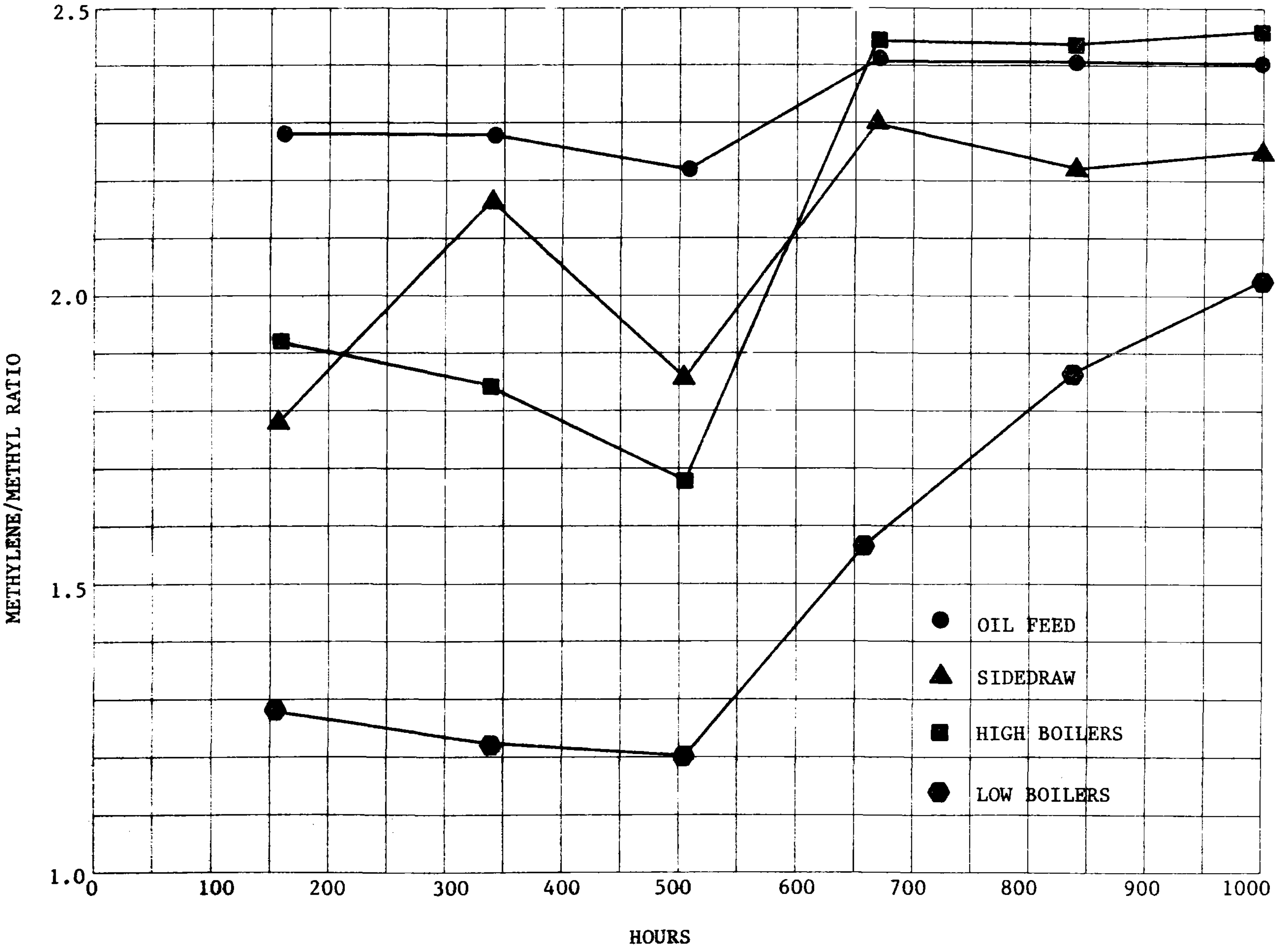


FIGURE IV-15 **METHYLENE to METHYL GROUP RATIO HISTORY** compounds and high boilers (long chain compounds) were removed, and a middle boiling range cut from the feed oil was returned to the bulk hot oil tank.

The low boiler methylene to methyl group ratio is consistently lower than that of the other fractions, because this fraction primarily contains compounds having lower molecular weights than the other fractions. Initial decrease in the ratio with time may be due to increase in thermal cracking of long chain components to form short chain compounds. Subsequent increase in the ratio may be due to loss of hydrogen or hydrogen and methyl groups (methane) to form olefins. The latter process may result in formation of a lower boiling compound while increasing the methylene to methyl group ratio. For example, consider the reaction(1) where the methylene to methyl group

2 - Methyloctane ⟶ 1 - Octene + Methane
b.p. 143°C b.p. 121°C

[IV.] $$CH_3\ (CH_2)_6CH(CH_3)_2 \longrightarrow CH_3(CH_2)_6CH = CH_2 + CH_4$$

Sat'd CH_2/CH_3 Ratio = 1.2 Sat'd CH_2/CH_3 Ratio = 4.0

ratio increases from 1.2 to 4.0 while the boiling point is reduced from 143 to 121°C. Such a process is also consistent with the appearance of ethylenic group protons in all of the low boiler NMR spectra. Also, it appears that the ratio was beginning to level off toward the end of the test as did the ratios for the other sample types.

The high boiler methylene to methyl group ratio decreased slightly in the first half of the test, rose abruptly between the third and fourth week and then leveled off at a ratio that was slightly higher than that for the hot oil feed. Also, there was no evidence for ethylenic hydrogens in the NMR spectra of the high boiler samples, which indicates that such groups are not a characteristic of the high boiling oil fraction. Chemical processes which may account for the initial decreasing ratio are ring opening followed by chain lengthening, methylation of double bonds to form saturated compounds having lower methylene to methyl group ratios, but higher boiling points due to longer chains, for example, reaction [IV] (pg 66). On the other hand, the observed ratios may be indicative of the distillation system's performance.

The sidedraw samples show a cycling in the methylene to methyl group ratio which parallels the trend observed in the other sample types. The sidedraw stream exhibits a ratio which is intermediate, between those of the high and low boilers, but is always lower than that of the hot oil feed. The sidedraw stream results from removal of about 10% by volume high boilers and 2.5% by volume low boilers from the hot oil feed stream, which account for the trend in the sidedraw methylene to methyl group ratio. The periodic rises in the sidedraw methylene to methyl group ratio trend are possibly due to incomplete removal of high boilers. There were increases observed in the density of the sidedraw samples exhibiting high ratios.

There was a slight indication of ethylenic protons in some of the sidedraw NMR spectra as had also been observed in some hot oil feed samples and in all low boiler samples. This observation indicates periodic buildup of ethylene groups to a detectable level ($\geq$0.5%) in the hot oil feed and incomplete removal of ethylenic type compounds from the sidedraw stream. The ethylene groups were present in the low boiler samples at about 2 percent.

As in Phase I, the Phase II infrared (IR) data were used to supplement the information provided by the other analyses, particularly the NMR results. Limited conclusions regarding changes in relative intensity of absorptions present in the IR spectrum of thermally exposed versus unexposed Caloria HT-43 could be made, because of variation of cell path length from sample to sample. However, there is strong evidence of ethylenic groups present by absorptions at 1500-1700 cm^{-1} in the IR spectra of low boilers samples 48 and 52. There is some fluctuation, both increases and decreases in intensity of absorptions at 760-780 cm^{-1} and 710 cm^{-1}. The absorptions cited reflect changes in extent and length of saturated chains. Some of this variation may be due to changes in ethylenic groups. Substituted benzenes which exhibit IR absorptions in 1400-1650 cm^{-1} and 700-900 cm^{-1} are present in the oil samples, but the intensity of the absorptions appear to be constant based on comparison of spectra resulting from about the same sample thickness. The above cited ethylenic absorptions were observed in spectra which had about the same intensity of hydrocarbon C-H stretching in the 2700-2900 cm^{-1} and 1300-1500 cm^{-1} regions as had been observed in the reference IR spectrum for unexposed Caloria HT-43.

Other sample characteristics noted were changes in visual appearance. The orange-brown hue of the bulk oil gradually changed to a light yellow more like the unexposed Caloria HT-43. The high boilers were generally thick and tended more toward the bluish gray characteristic of the thermally degraded oil from near the end of Phase I. The low boilers were consistently a pale, light yellow, aromatic smelling fraction. The sidedraw at times (early part of test) appeared cloudy and orangish. In the last few weeks of the test sidedraw samples were very clear and pale yellow. Upon standing at room temperatures, all the hot oil samples and the sidedraw samples from the latter half of the test exhibited formation of a residue. Brief examination of this residue indicated a waxy material, insolubility in hexanes and toluene, slight solubility in nitrobenzene. A solubility analysis is included in Appendix D. Melting point of the compound, determined from a hot plate microscope, was in the range of 120 to 160°C.

The IR of a pressed pellet of the residue showed only general hydrocarbon absorptions and an NMR spectra could not be obtained. The formation of residue was not observed in the high boiler samples. Although no residue formed in the low boiler samples, a reddish-brown oily component did form a separate phase in the low boiler samples from the last half of the test period.

The viscosity of the bulk oil showed a general downward trend over the Phase II test duration (Figure IV-16). The final bulk oil viscosity was much lower in Phase II (26.8 CSt.) than in Phase I (49.7 CSt.). The lower viscosity is indicative of the removal of the higher molecular weight, viscous hydrocarbons from the bulk oil. One processor bottoms sample (No. 43) had a viscosity measurement of 147.7 CSt. which exemplifies the processor's ability to remove highly viscous compounds from the bulk oil.

Gel Permeation Chromatography (GPC) tests exhibit a similar trend of a decrease in bulk oil molecular weight (Table IV-14). As evidenced by the carbon number, the bulk oil average molecular weight decreased substantially to light hydrocarbons.

Gas Chromatograph (GC) distillation tests were performed on all Phase II samples. A comparison of the processor feed and product stream (tops, bottoms, and sidedraw) illustrates how efficient the processor was in separating the hydrocarbon mixture (Figure IV-17). The processor removed both high and low boiling fractions from the oil. However one can also see upon examination of Figure IV-17 that the bottom fraction contains higher boiling fractions than the feed. This phenomena indicates that some polymerization is occurring in the processor. Polymerization may have been initiated by either high temperatures (as high as 332^{o}C, 630^{o}F) in the processor heating element or by oxidation. Since heater temperatures were only greater than 316^{o}C (600^{o}F) for the first week of operation and polymerization was evident in the bottoms samples throughout the Phase II test, oxidation

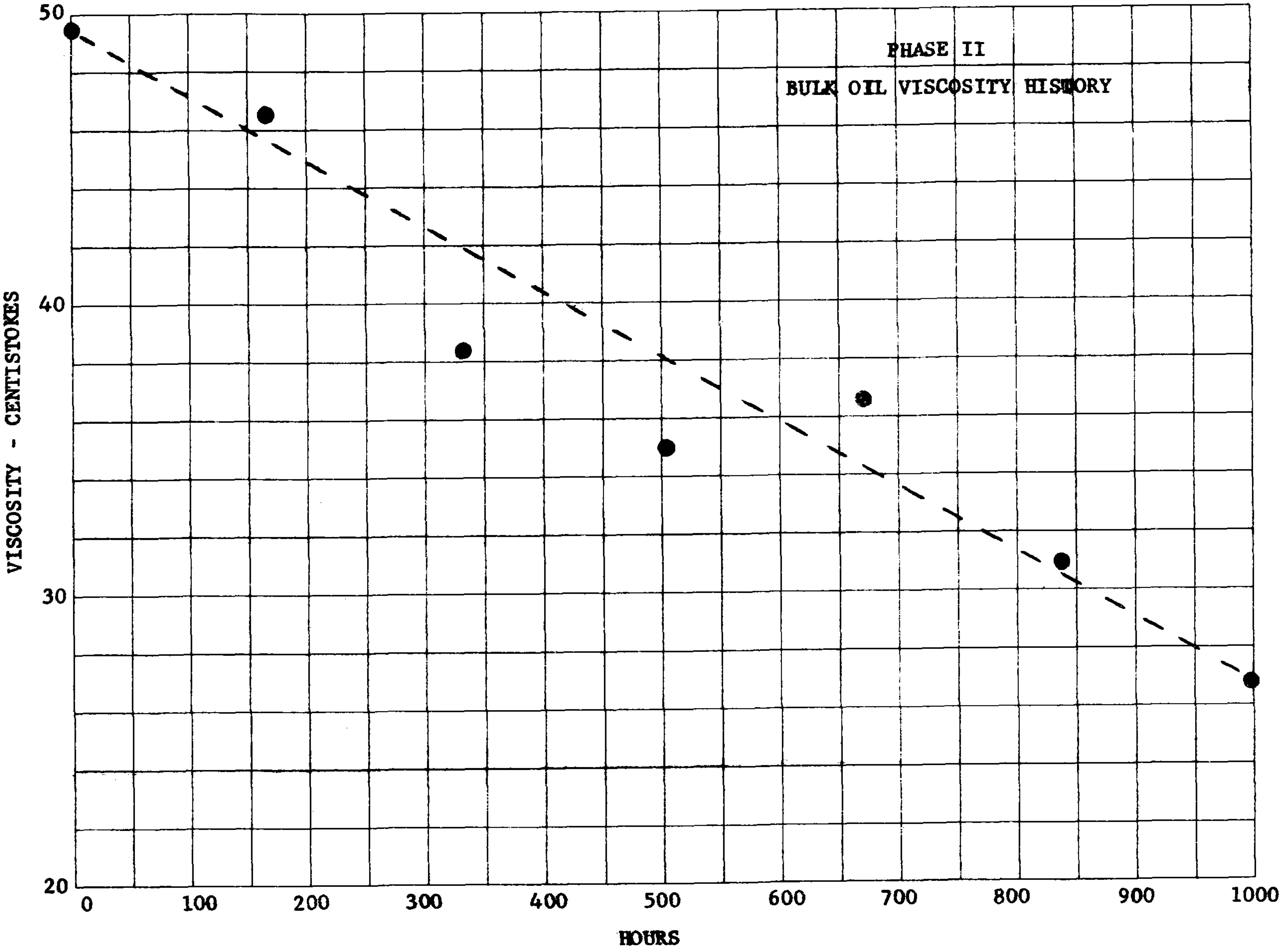


FIGURE IV-16 PHASE II BULK OIL VISCOSITY HISTORY

TABLE IV-14

PHASE II GEL PERMEATION CHROMATOGRAPHY RETENTION TIMES

(SEE TABLE IV-7 FOR SAMPLE TIMES)

SAMPLE NO.	FIRST PEAK UV RETENTION TIME (MIN)	CARBON NUMBER
27	7.68	25.2
29	8.00	20.5
36	8.16	18.7
37	8.64	14.5
41	9.44	10.0
45	9.92	7.5
49	10.63	4.0

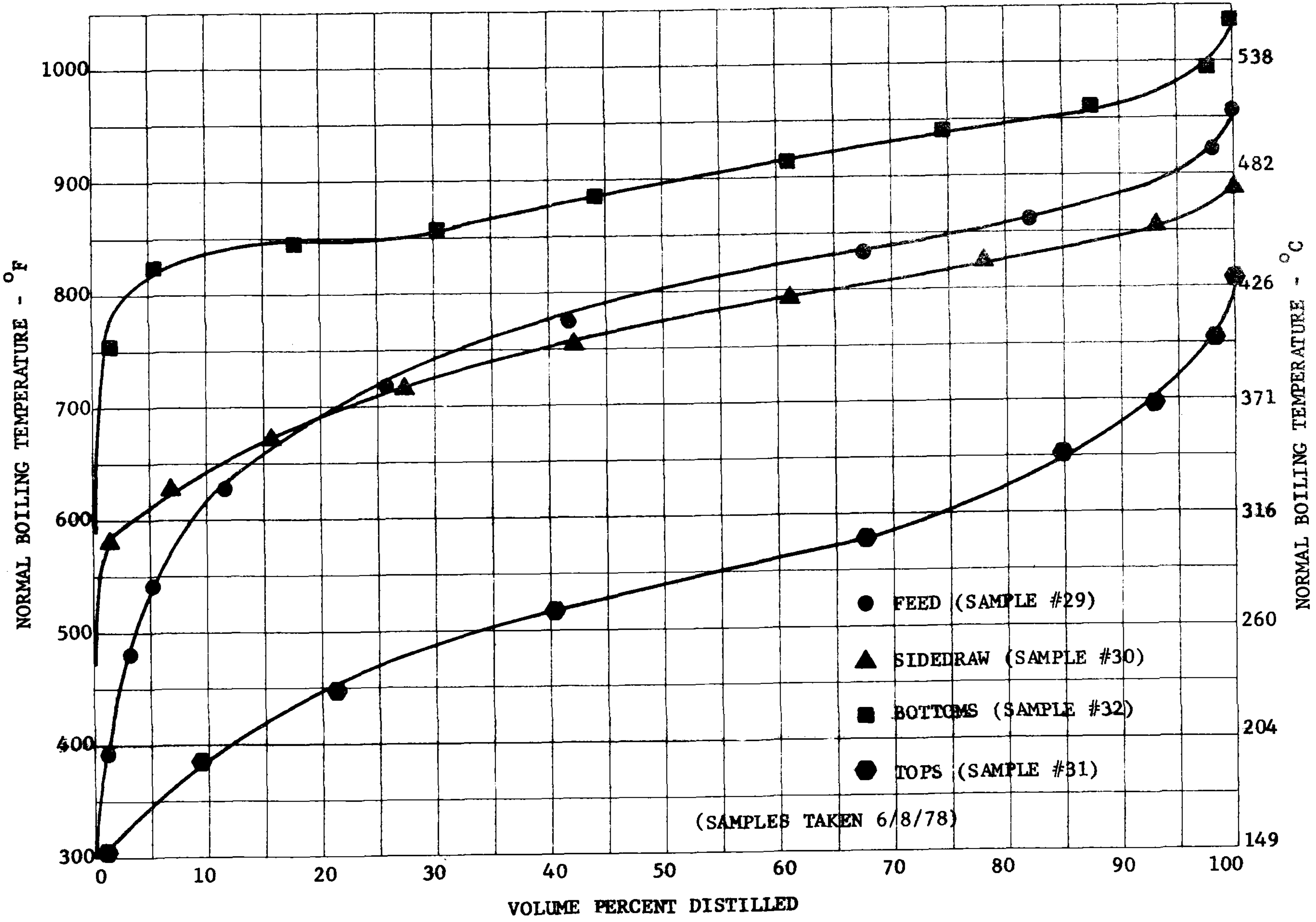


FIGURE IV-17 **FIRST WEEK ACTUAL PROCESSOR STEAM COMPOSITION** induced polymerization is suspected. Obviously, oxidation could only have been caused by a leak in the vacuum distillation system. Although many precautions were taken to ensure a sealed system, a small leak in the high vacuum system was probable and likely undetectable.

The chemical analysis results from Phase II have added to, and support the results from Phase I. The main conclusion that can be drawn is that during the thermal degradation process low boilers are formed. Visual observation indicates association of chromophoric groups with the high boiler fraction, but the instrumental methods employed have not detected their presence in this fraction. On the other hand, the NMR and IR spectra of the low boilers from Phase II and the ice-water condensates from Phase I indicate distinct changes relative to the unexposed Caloria HT-43. These changes are associated with formation of double bond systems. The ethylenic groups formed are not readily observed in the bulk oil spectra, because they are present at very low (<1%) concentrations. The distillation process employed in Phase II removed and concentrated the compounds containing these groups into the low boiling fraction. The relative consistency in the hot oil feed samples in the last three weeks of the test indicate that the distillation process was effectively maintaining a uniform bulk oil composition in the thermal storage tank.

3. Processor Performance Analysis

Numerical analyses and computer model simulation of the sidestream processor performance were undertaken to aid in quantifying the important process design and operating parameters on processor separation efficiency. Two separate computer models were developed to perform the calculations of time-dependent storage tank degradation products concentration and the prediction of steady state distillation performance of the sidestream processor. The first model, labeled TMASS, was used to determine the level of the degradation products concentration in storage tank oil for a given sidestream replenishment flowrate. A predecessor of this model was used early in this program to determine an adequate sidestream flow rate for the processor. The second model, called DISTILL, was developed using an existing computer model (Ref. 9) to calculate the distillation parameters of the "as tested" sidestream processor used in Phase II of this program. The model was also developed with the additional objective of providing an aid in the evaluation of future designs and operating modes of other conceptual processors. However, the evaluation of alternate designs was not within the scope of this program and was not performed.

a. TMASS Model Development and Simulation - The TMASS model was developed using conservation of mass equations for the tank to determine the tank degraded product concentration for the processor system shown in Figure IV-18. For this model the oil in the tank was assumed to consist of two global species--degraded and non-degraded oil. The concentration of degraded products, C_D, was determined by assuming that the degradation rate, R. and oil flowrate to a sidestream processor, $\dot{W}_{ss}$, were constant. Two cases were considered: (1) fresh oil is added to the storage vessel to replenish the oil extracted by the sidestream processor (constant mass), and (2) no fresh oil replenishment to the tank (changing mass). The first case would probably represent the operation of an actual commercial system. The second case was developed to simulate the operation of the system operated in Phase II.

An equation was developed for both the constant and changing mass systems by starting with the Conservation of Mass Law for decomposition products. That is:

$$R_I = R - R_R \qquad \text{(IV-8)}$$

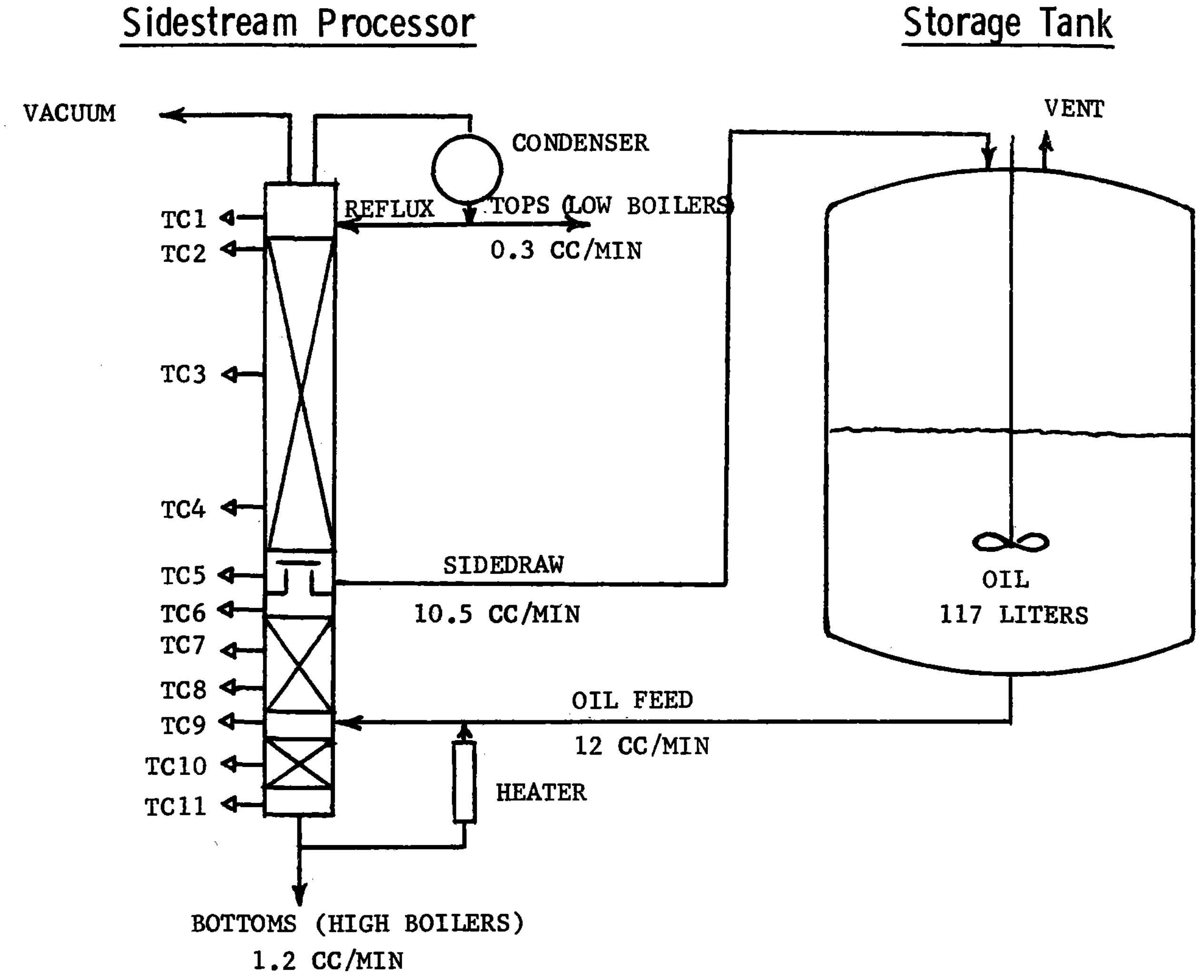


FIGURE IV-18 PHASE II SYSTEM SCHEMATIC

or the rate of degraded products increase in the tank, R_I, is equal to the rate at which those products are generated, R, minus the rate at which they are removed, R_R, using Eq (IV-8) it can be shown that the degraded oil concentration for a constant mass system is described by the differential equation:

$$\frac{dC_D}{d\theta} + C_D\left(R + \frac{\dot{W}_{ss}}{M_o}\right) - R = 0 \qquad \text{(IV-9)}$$

and for the changing mass system

$$\frac{dC_D}{d\theta} + C_D\left(R + \frac{\dot{W}_{ss}}{M_o}\right) - \dot{W}_{ss}\left(\frac{C_D}{M_o}\right)^2 - R = 0 \qquad \text{(IV-10)}$$

Eq (IV-9) can be solved in closed form and has the general solution:

$$C_D = Ee^{-D\theta} + \frac{R}{D}$$

where: $D = R + \frac{\dot{W}_{ss}}{M_o}$

E = a constant, θ = Time, M_o = Initial Tank Mass

Eq (IV-10) cannot be easily solved in closed form since it is nonlinear. The equation was solved however by developing a computer program using an improved Euler method for solution of differential equations. The equations describing each system also assume that the tanks are perfectly stirred and that the processor separates only the degraded products from the sidestream (i.e., 100% separation efficiency).

Both Eqs (IV-9) and (IV-10) were solved for an initial tank oil mass of 99.4 Kg (219.4 lb_m) and several assumed degradation rates in the range between 27% to 60% per year. The oil mass represents the approximate weight of oil in the tank at the beginning of Phase II and the degradation rates represent the range of expected rates for vented storage systems. Shown in Figure IV-19 are the results from solution of equations describing constant and

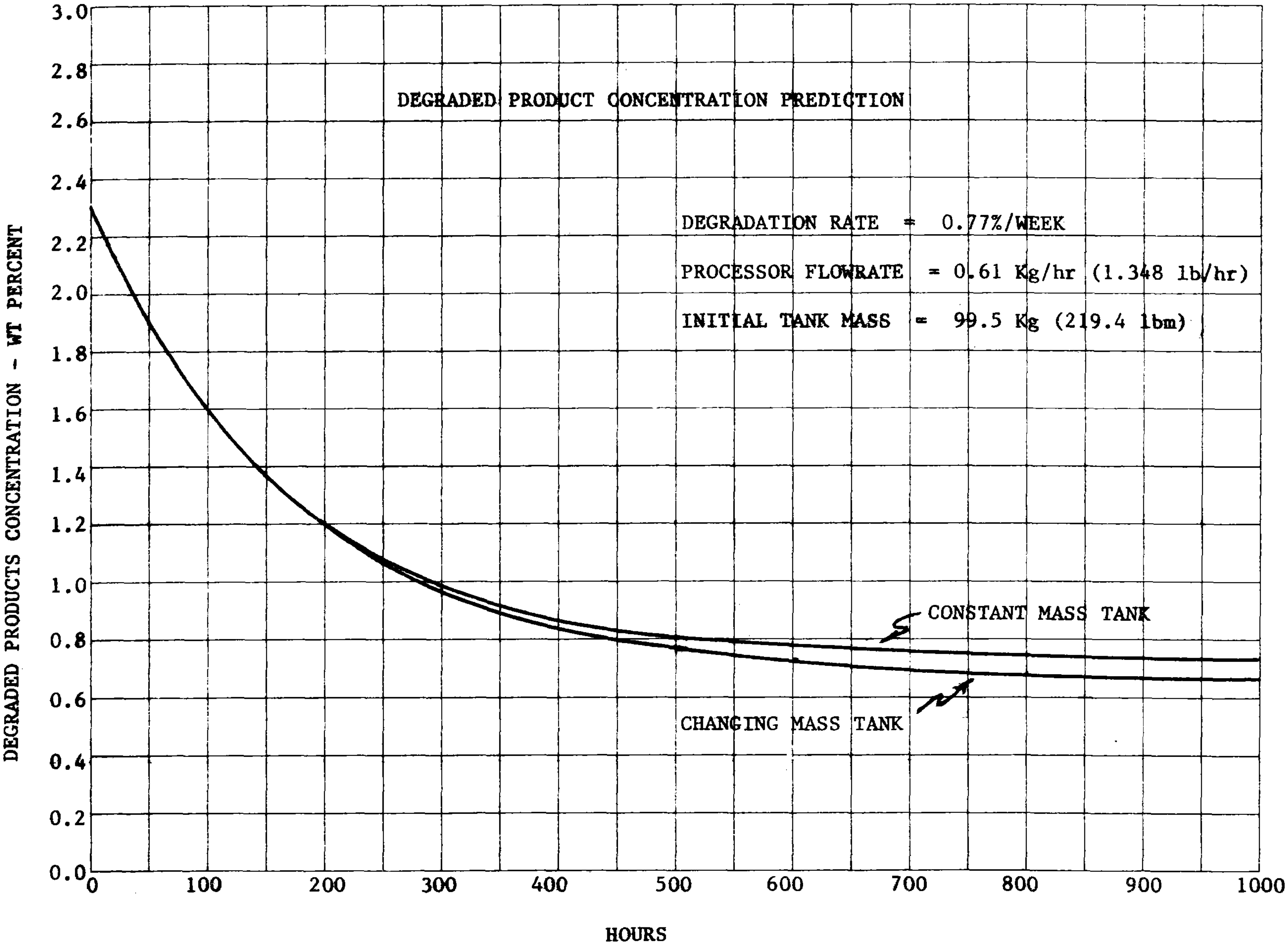


FIGURE IV-19 **MODEL COMPARISON**

changing mass systems. Initial degradation product concentrations represent three weeks of decomposition without a processor. Results shown in this figure indicate that in the constant mass system the degraded products concentration achieves a steady state concentration with time and for the changing mass system the concentration continually decreases.

In order to determine the actual Phase II oil degradation rate, the test data was examined to determine the processor separation efficiency. The TMASS model assumes that the processor is a perfect separator. That is, only degradation products are extracted from the system. Therefore, the actual Phase II mass balance data had to be "smoothed" to typify system mass extraction by a perfect separator. This method was accomplished by:

1) First determining what fraction of oil in the bottom and top product streams contained degradation products through examination of the weekly GC distillation curves of these streams.
2) Subtracting from the initial bulk oil mass only that amount which was degradation products over the 1000 hour test duration.

This method assumed that the good oil in the tops and bottoms product streams would have not been further degraded. Understandably, this simplification could result in a low prediction of degradation. However, Phase I data indicates that the high boiling fractions of the oil remain stable and are not as subject to thermal cracking as the lower boiling fraction.

Nevertheless, using the above described method of "smoothing" the mass balance data via the results of chemical analysis resulted in the calculation of actual degradation product fractions shown in Table IV-15. Using these weekly product fractions and an initial bulk oil mass of 99.5 Kg (219.4 lb) resulted in a

TABLE IV-15

PROCESSOR EFFICIENCY CALCULATIONS

(See Table IV-7 for Sample Times)

WEEK	SAMPLE	BOTTOMS VOLUME PERCENT ABOVE 538°C(1)	BOTTOMS VOLUME PERCENT BELOW 538°C	SAMPLE	TOPS VOLUME PERCENT BELOW 232°C(2)	TOPS VOLUME PERCENT ABOVE 232°C
1	#32	2.0	98.0	31	21.0	79.0
2	#34	1.5	98.5	33	30.0	70.0
3	#39	2.5	97.5	40	27.0	73.0
4	#43	0.0	100.0	44	23.0	77.0
5	#47	0.0	100.0	48	19.0	81.0
6	#51	0.0	100.0	52	18.0	82.0
	AVERAGE	1.0	99.0		13.0	77.0

(1) Bottoms degraded products

(2) Tops degraded products

calculated remaining mass in system shown in Figure IV-20. Along with this calculated data are the results of the TMASS model using an initial degraded products concentration of 0.932% by weight which was determined by comparing the distillation curves of the fresh oil and the bulk oil at the beginning of Phase II test. Three degradation rates were examined, 1%, 5%, and 10% per year. A comparison of the data with model results indicates that the degradation rate is close to 5% per year. If one assumes that the processor cannot achieve perfect separation of the low boiling degradation products and can only separate a top fraction that is on the average 13% by volume low boiler degradation products, the fluid replenishment rate is:

$$\begin{aligned}\text{Fluid Replenishment Rate} &= \text{Degradation rate} \div \text{weight percent degraded products} \\ &= \frac{\text{Degradation Rate}}{\text{Low boiler volume percent}} \times \left(\frac{\text{Density of tops}}{\text{Density of low boilers}}\right) \\ &= 0.05 \left(\frac{1}{0.13}\right)\left(\frac{0.795}{0.731}\right) \\ &= 0.42\end{aligned}$$

That is, the fluid replenishment rate with the use of the processor designed for Phase II resulted in a value of 42%/year. This value compares with the fluid replenishment rate of 56%/year under Phase I testing without a processor. However, with improved processor design one could achieve replenishment rates closer to 5%/year for a storage system held at a constant 316°C (600°F).

b. DISTILL Model Development and Simulation - An existing well documented computer program (Ref. 9) provided the basis for the development of a processor performance model. The computer

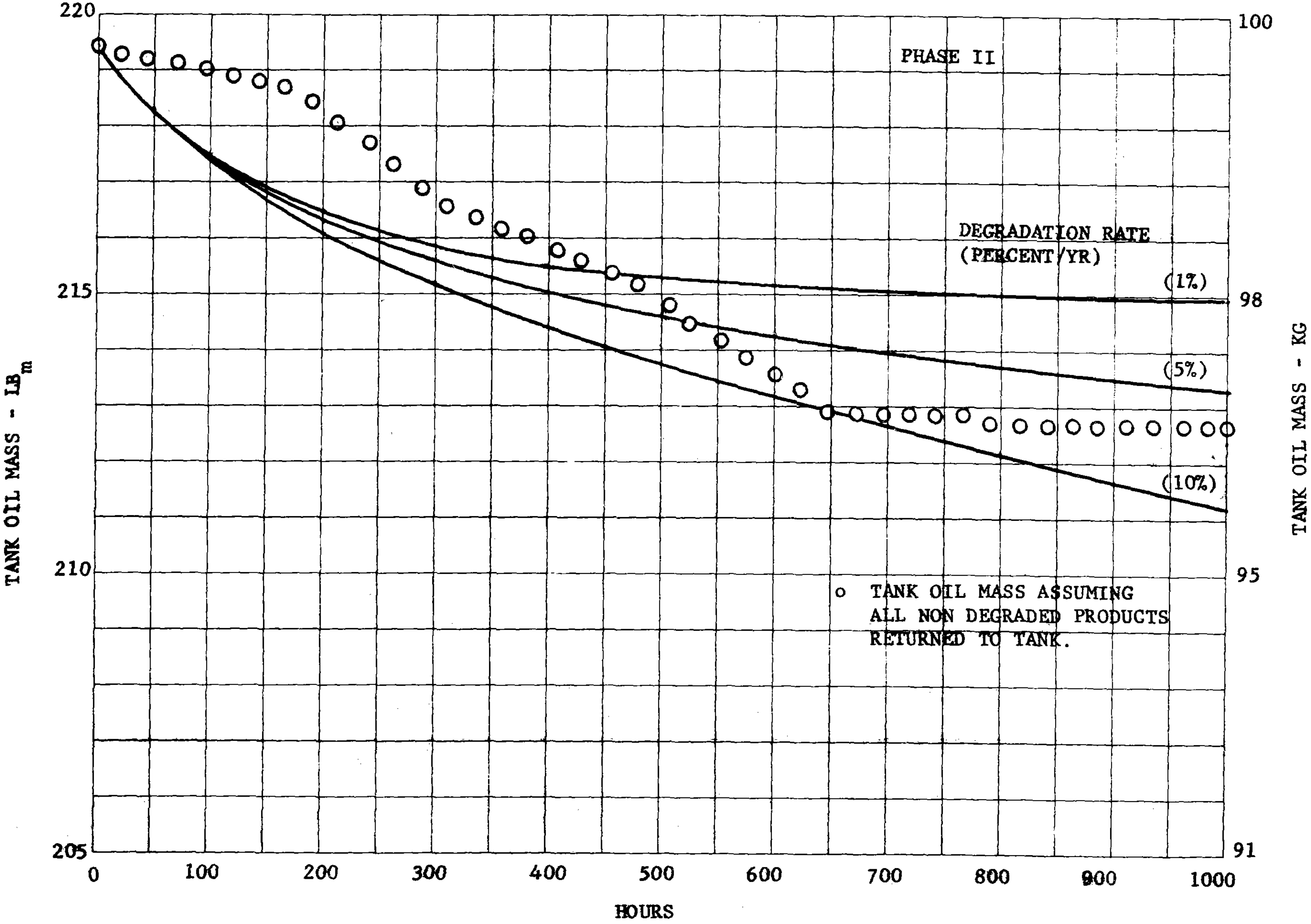
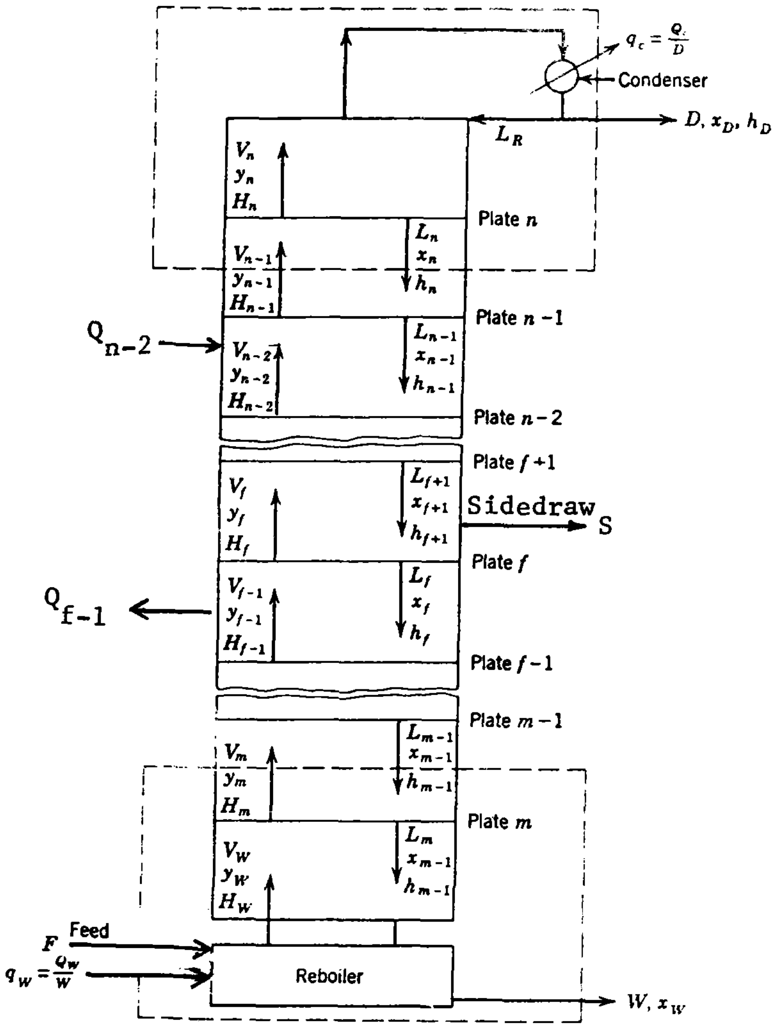


FIGURE IV-20 **PHASE II DEGRADATION RATE DETERMINATION** program named DISTILL was modified and updated to execute on Control Data Corporation (CDC) computers. The model is based on a method of solution which solves the mass and energy balances simultaneously and can be easily applied either to single columns or to very complex interlinked combinations of up to five distillation columns. The program can also handle multiple feedstreams, sidestreams, and heat losses from the column. Therefore the program is very versatile and can be used for current and future process designs.

The model selected for development in this program has been based upon the "rating" approach to distillation solution. That is, the column geometry and operating conditions are input and the separation capability is output. This type of distillation performance program is useful for evaluating existing or future designs. The method employed in this investigation has been to determine the number of contact stages in processor actually tested in Phase II and then determine if such parameters as column temperatures, component mass flows, and component concentrations can be predicted. Experimental data was used to verify the accuracy of predictions.

Oil is a mixture of hydrocarbons having various physical and thermodynamic properties. The oil used in this program is indeed a multicomponent mixture and poses a particular requirement for a computer method to simulate the distillation process. Shown in Figure IV-21 is a general flow diagram of the internal and external mass and energy flows occurring between contacting stages in a distillation column. This figure shows liquid and vapor flow counter-current to one another at each stage in the column (denoted by vertical arrows), the multiple feed locations into the column at each stage (horizontal arrow to left of the stage), and the multiple sidedraw locations at each stage for vapor and liquid streams (horizontal arrows to the right of the stage).



F = Feed Rate

W = Residue Rate

V = Vapor Rate

L = Liquid Rate

S = Sidedraw Rate

D = Distillate Rate

Q = Heat Added, Removed or Lost

H = Enthalpy of Vapor

h = Enthalpy of Liquid

q = Heat Added or Removed per Mole

x = Concentration in the Liquid

y = Concentration in the Vapor

Subscripts

C = Condenser

D = Distillate

R = Reflux

W = Residue

n,f,m - Plate Designation

FIGURE IV-21 DISTILLATION FLOW DIAGRAM

Energy flows can be added or extracted from each stage as depicted in Figure IV-21. The problem required for solution is thus: given the external mass and energy flows of the column, determine the internal mass and energy flows, the mixture composition at each stage, and output stream and the temperatures of each stage. The model selected for this evaluation performs those calculations by first using an assumed column temperature profile, then iterating on each stage of the column until component mass and energy balances are satisfied based upon component liquid-vapor phase equilibrium relationships. The number of contacting stages (n in Figure IV-21) represents the theoretical number of equilibrium stages of a column. The actual number of stages will be greater than the theoretical number of stages since a finite rate of the mass transfer exists between liquid and vapor phases.

For packed columns the height of packing equivalent to a theoretical plate needs to be determined. The height equivalent to a theoretical plate, H.E.T.P., has been empirically defined as (Ref. 8):

$$\underset{(\mathrm{cm})}{\mathrm{H.E.T.P.}} = 2.54\ K_1\ G^{K_2}\ d^{K_3}\ h^{1/3}\ \frac{\alpha\mu}{\rho} \qquad \text{(IV-11)}$$

G = vapor mass flow rate, lb/(hr)(ft^2)

d = tower diameter, inches

h = packed height, feet

α = relative viscosity of key components

μ = liquid viscosity, centipoise

ρ = liquid density, g/cm^3

K_1, K_2, K_3 = constants for specific packing

For the packing used in the distillation column of Phase II tests these constants are $K_1 = 0.076$, $K_2 = 0.5$, and $K_3 = 0.30$. For a vapor molar flow of 3.7 (this value was determined after several preliminary computations using the DISTILL computer program) times the feedrate, a relative volatility of key components of 4.0, and Eq (IV-11) the H.E.T.P. is equivalent to 25.6 cm (10.1 in.).

Since the column height from feed location to the top of the column is 175.3 cm (69 in.), the total number of theoretical stages is approximately 7 (175.3/25.6). Shown in Figure IV-21 is a column flow schematic depicting the operation of the column used in Phase II. Note that the feed entered the first stage of the column which is also the reboiler stage. The sidedraw stream was estimated to be located at the third theoretical stage based upon actual physical location and the H.E.T.P. of 25.6 cm (10.1 in.). The column consists of seven theoretical stages (previously calculated) plus the condenser for a total of eight theoretical stages. Based upon actual flows derived from the Phase II experimental phase, the volume split of the streams were estimated on a weekly basis. The energy flows shown in Figure IV-21 indicate the heat transfer is from the column to surroundings representing heat loss. Heat losses to the environment were estimated for the 5 cm (2 in.) diameter column with 5 cm (2 in.) insulation. Column temperatures from test data were used to determine the heat transfer at each stage. Shown in Table IV-16 are the calculated heat losses assuming that the heat transfer is in the column radial direction only.

TABLE IV-16

Heat Transfer to Environment

Stage	Btu/Mole Feed x 10^4	Kwatt-hr/Mole Feed
1	3.7281	10.919
2	3.2135	9.412
3	2.527	7.401
4	1.7553	5.141
5	1.4980	4.387
6	1.2406	3.633
7	1.0691	3.131
8 (Condenser)	--	

As will be seen in later discussion these heat losses have a significant impact on the performance of the distillation column. Due to the scale of the equipment, however, these losses were expected.

Once these heat loss and flowrate input parameters were calculated, the simulation of the distillation column performance with the computer model proceeded. Eight theoretical stages were input to the model along with the previously estimated heat losses, feed composition from the tank oil chemical data and product stream flows (sidedraw, tops, and bottoms). A condenser temperature of 21°C (70°F) was assumed. Reboiler heat load was increased to match the test data reboiler temperature during the first week of operation. Top product reflux flowrate back to the column was varied until the top temperature was approximately equal to the actual top of column temperature during the first week of Phase II testing. Once these temperatures were simulated with the computer model, the intermediate temperatures, between the reboiler and the condenser, were examined and compared with test data. Shown in Figure IV-22 are the results of the computer simulated column temperatures with actual test data collected on 2 June 1978, one day after the start of Phase II testing. Equilibrium stages were matched with the physical locations of the thermocouples. As can be seen there is very good agreement between predicted and actual column temperatures. From the computer output, true boiling temperatures of the product streams were determined and are shown in Figure IV-23. These results can be compared to the actual first week stream compositions. Again agreement between test derived product compositions and those simulated is good and indicates that (1) simulation of the process is accurate, and (2) the distillation column performed as designed.

The next step in the processor simulation was to understand the effect of column heat loss on processor performance. A case was run without heat loss using the identical feed composition from the previous simulation. Reboiler heat load was adjusted until the first stage temperature matched the actual first stage

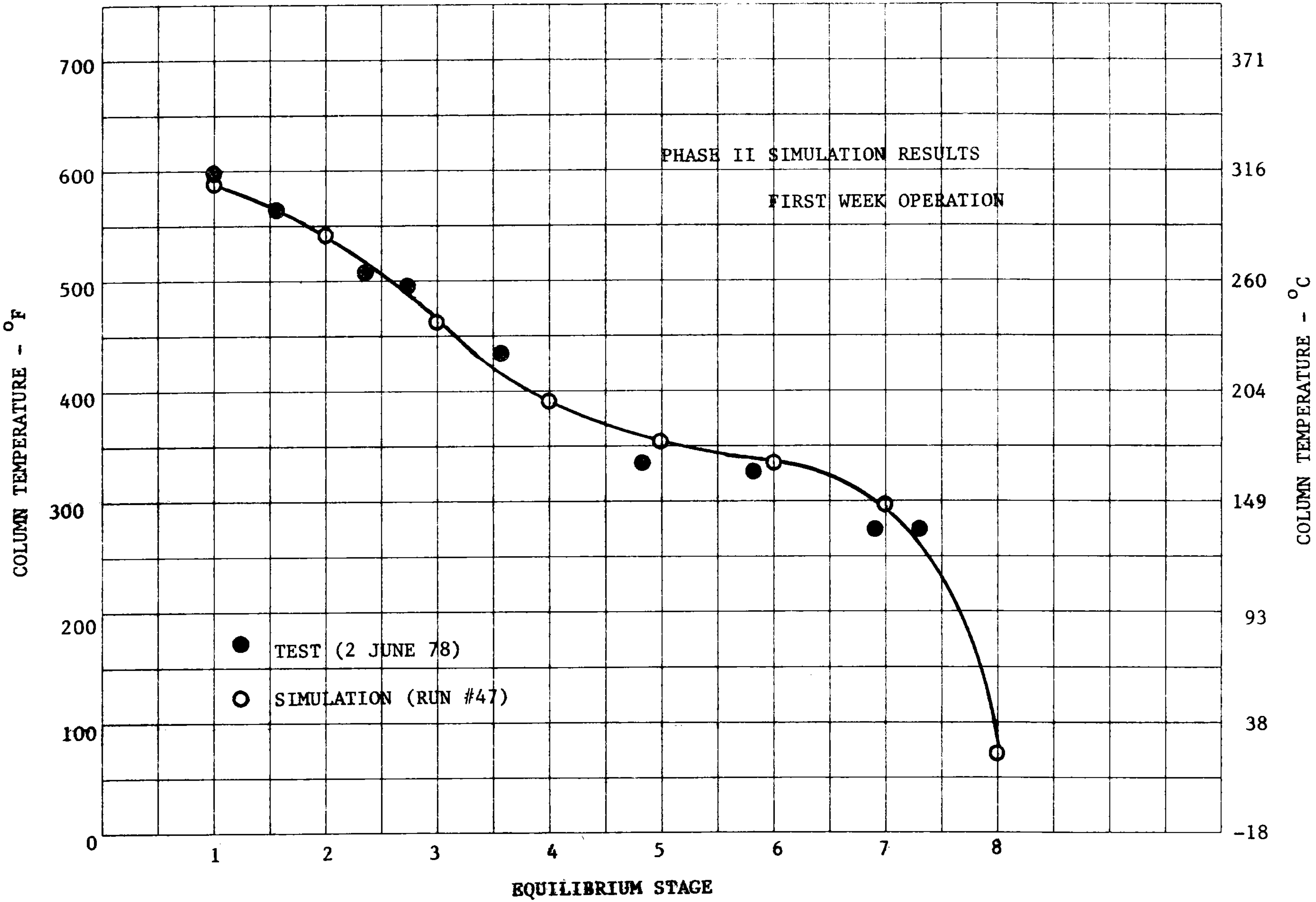


FIGURE IV-22 FIRST WEEK PROCESSOR TEMPERATURE SIMULATION

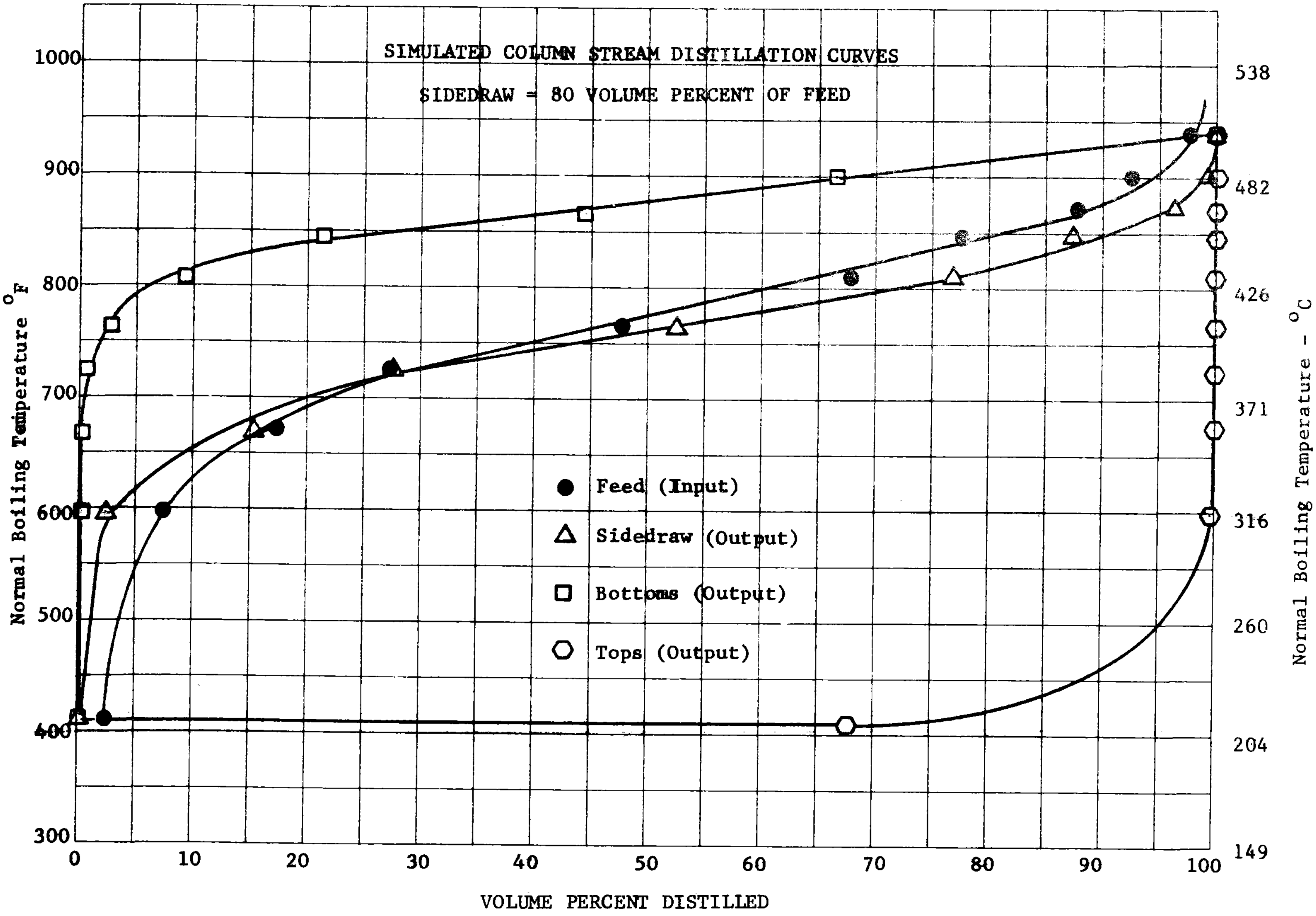


FIGURE IV-23 **FIRST WEEK PROCESSOR SIMULATION STREAM COMPOSITION** temperature. Reflux flowrate was held at the same rate as the previous case of 0.75 moles reflux/mole feed. Shown in Figure IV-24 is a comparison of the predicted column temperatures with heat loss to those temperatures without heat loss. Without column heat loss the reboiler vapor flowrate, which is an indication of energy required to "drive" the distillation-process, is reduced from 3.7 to 1.4 moles vapor/mole feed. The predicted reboiler and condenser energy requirements in both cases were:

	Reboiler Load (Kwatt-hr/mole feed)	Condenser Load (Kwatt-hr/mole feed)
With Column Heat Loss	48.324	14.425
Without Column Heat Loss	9.7920	20.251

As seen in the above figures the energy required to heat the bottom of the column is on the order of 5 times less for a column with no heat loss than the column tested in Phase II with heat loss. A higher condenser load is required on the column without heat loss since more and higher temperature vapors reach the top of the column and proceed through the condenser. For an actual commercial processor the energy requirements to operate the distillation processor (excluding pumping requirements) would approach those of a column without heat loss if sufficient insulation is used. The additional value of the computer model is that it can determine the energy requirements of various processor designs and the economic advantage of insulation to reduce energy consumption.

The next step in the simulation analysis was to predict the effect of variations in feed composition on processor performance. This analysis was important since a storage tank oil composition is known to change due to thermal cracking and, as in the case of Phase II, due to the addition of reprocessed oil. In Phase II a

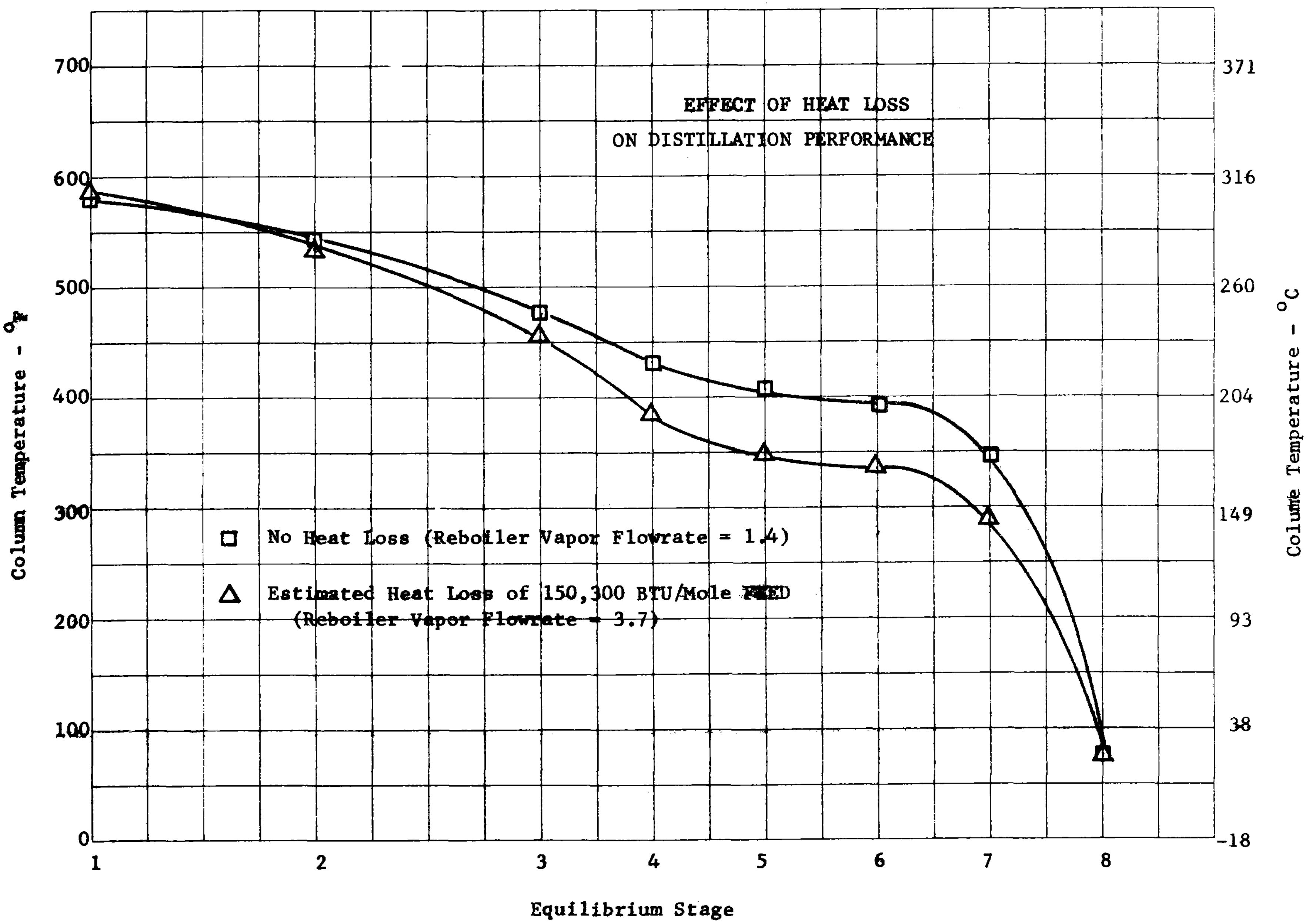


FIGURE IV-24 PROCESSOR HEAT LOSS EFFECTS feedstream flowrate from the storage tank to the processor of approximately 12 cc/min was maintained throughout the test duration. At an oil specific gravity of 0.85 and an average molecular weight of 385, the mass flowrate is 612 g/hr or 1.348 lb/hr and a molar flowrate of 1.590 g•mole/hr or 0.00350 lb•mole/hr. With an initial tank oil mass of 99.5 Kg (219.4 lb), the total volume of oil would be displaced in 162 hours or approximately one week with a flowrate of 12 cc/min. Since the reprocessed oil is being added back to the degraded oil in the tank, the oil in the tank should become compositionally more like the reprocessed oil as time proceeds. Since the space-time (tank volume/sidestream volumetric flowrate) in the tank is approximately one week, the composition in the tank should resemble reprocessed oil at some period in the time thereafter. However due to the thermal cracking process and mixing effects of degradation products with reprocessed oil the time for the tank oil to resemble the reprocessed oil would be lengthened. A sidestream processor simulation could indeed verify when this would occur by predicting the column temperature shift due to change in feed and thus storage tank concentration.

The approach taken in determining the effects of changing composition on processor performance was to take the processor computed sidedraw oil composition from one computer run and use that composition as feed in the next run. All column operating variables, such as reboiler vapor flowrate, reflux flowrate, and heat losses, were held constant from case to case so that only the change in feed composition would affect the column performance. Shown in Figures IV-25, IV-26, IV-27 and IV-28 are the column temperatures predicted from computer simulation compared with actual test temperatures. In each case the predicted sidedraw composition was used as feed for the next run. Note that the first stage predicted temperatures are very close with the actual test temperatures. After six weeks of operation the required reboiler

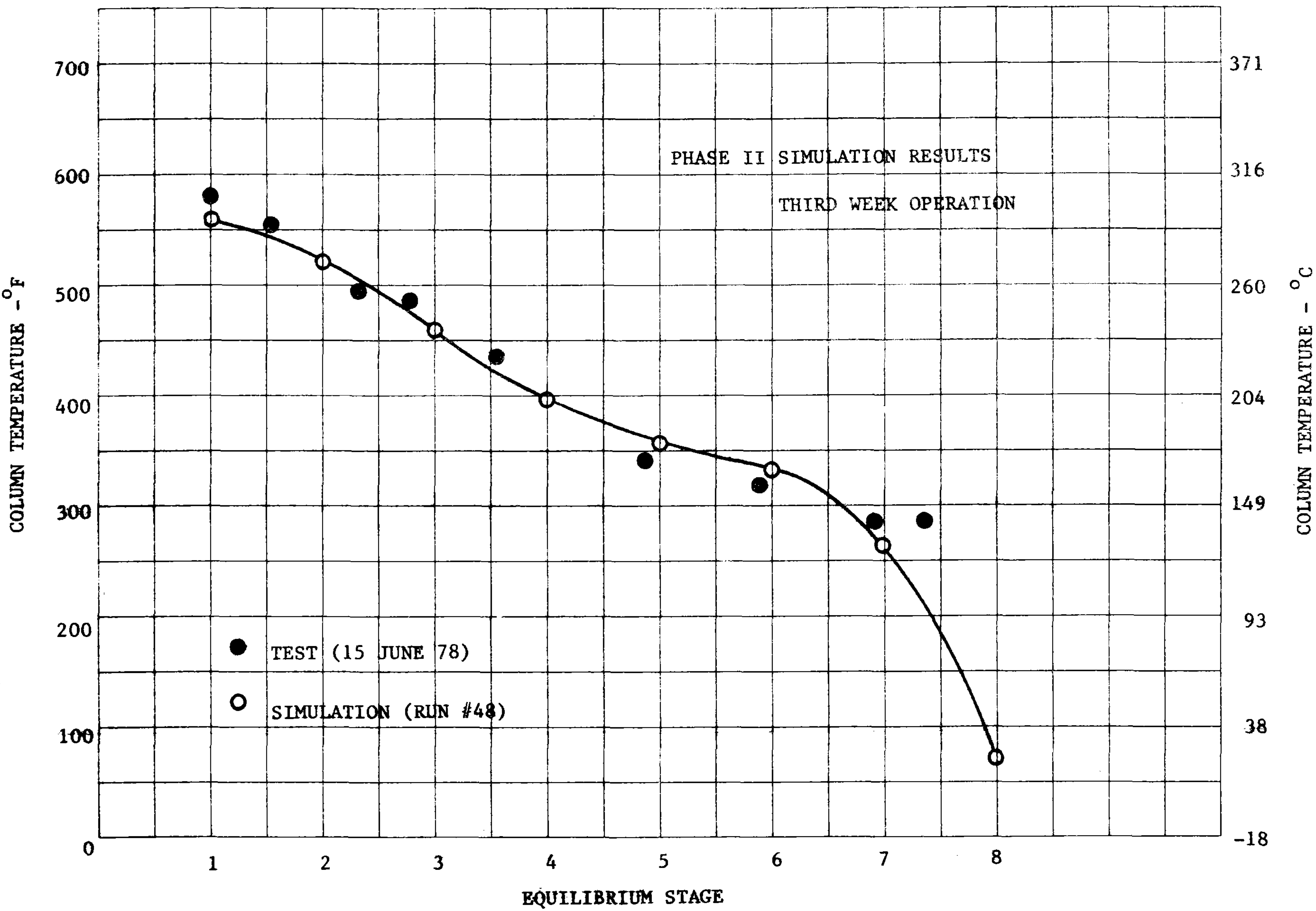


FIGURE IV-25 **THIRD WEEK SIMULATION RESULTS**

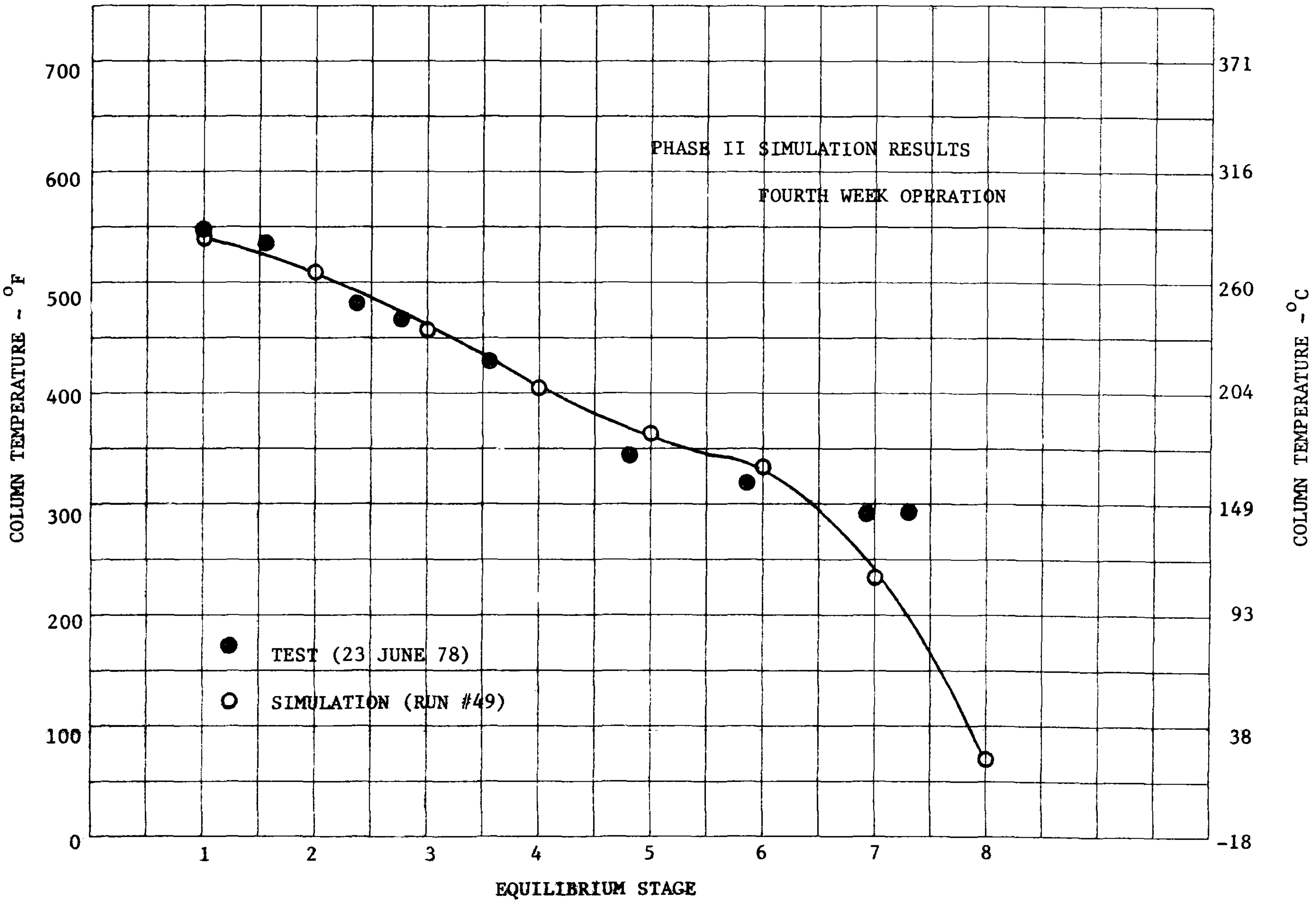


FIGURE IV-26 **FOURTH WEEK SIMULATION RESULTS**

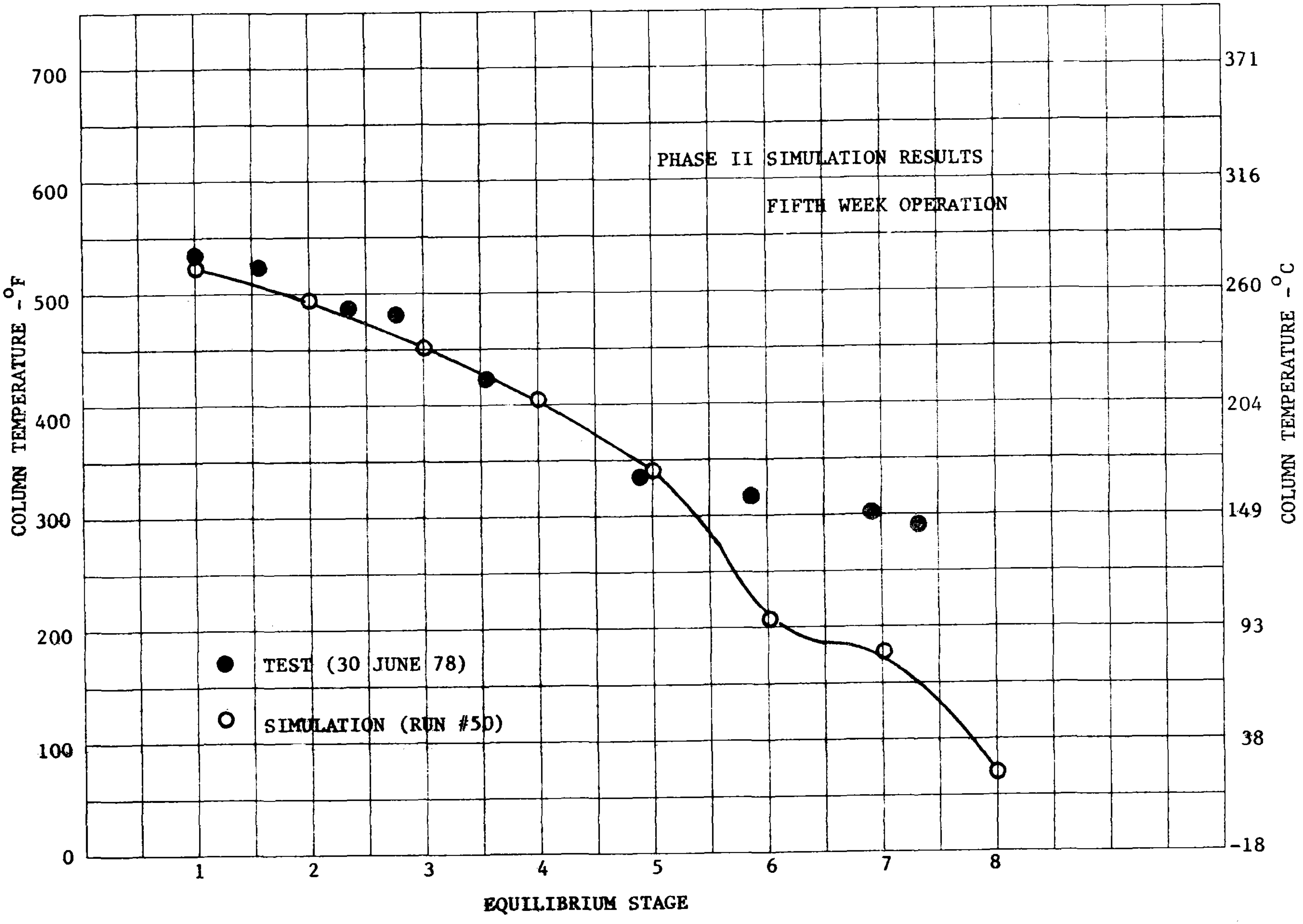


FIGURE IV-27 **FIFTH WEEK SIMULATION RESULTS**

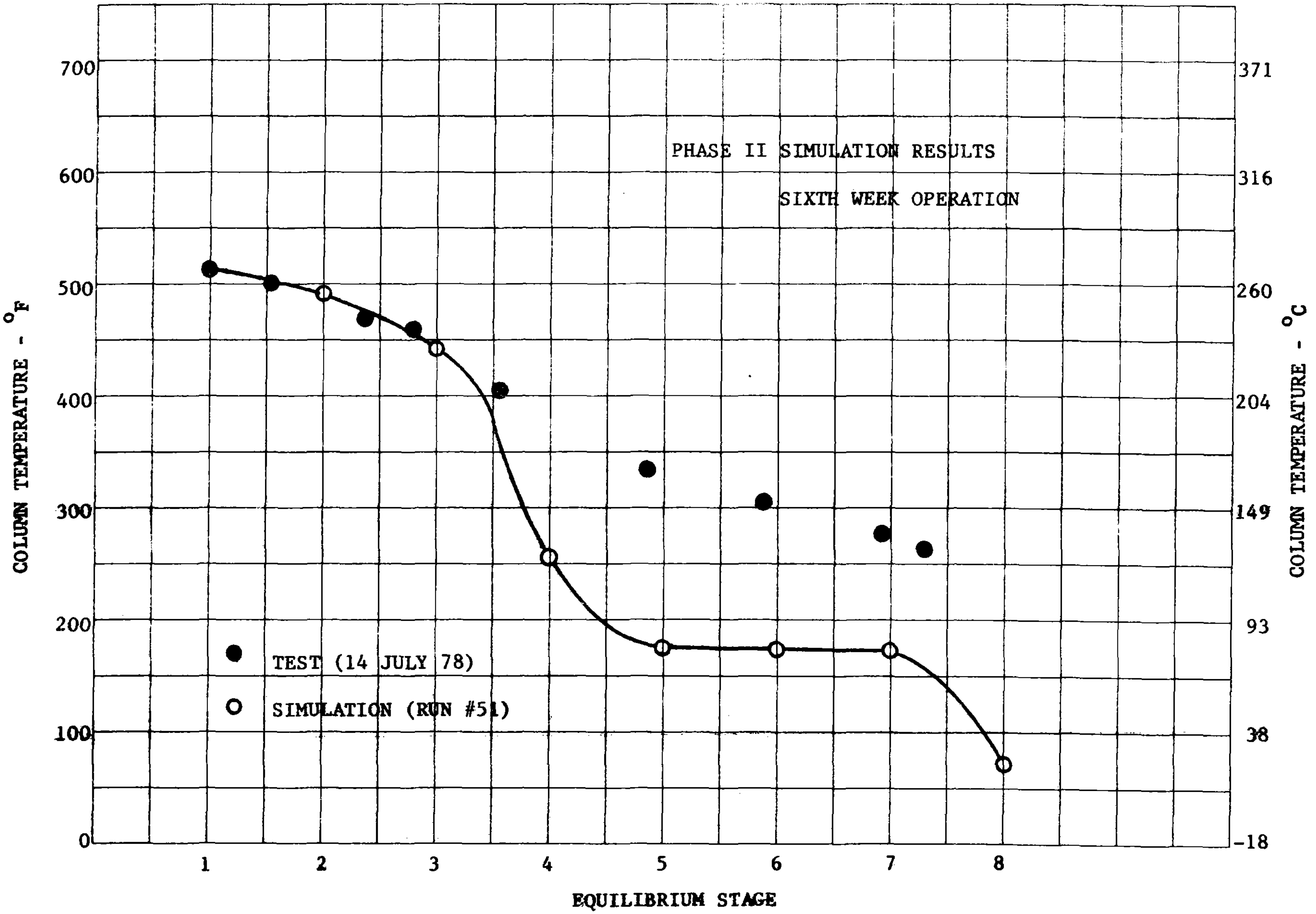


FIGURE IV-28 **SIXTH WEEK SIMULATION RESULTS** temperature (i.e., feed temperature) dropped from 310°C (590°F) to 266°C (511°F). The simulation predicted exactly the same trend. Due to the use of constant reflux flowrate back to the column, the predicted temperatures at the upper section of the column were lower than the actual test temperatures. This comparison indicates that the reflux flowrate actually became lower as the test proceeded. This was actually noticed during the test as the amount of tops (low boilers) collected in the accumulators became less as the test proceeded.

Shown in Figure IV-29 is a comparison of the GC distillation curves of the sidedraw sample taken on 8 June 1978, and the tank sample taken on 22 June. The oil compositions are nearly identical. The combination of simulation results and the test data indicate that the tank oil became much like the reprocessed oil in a period of two weeks. Therefore a volume equivalent to approximately two times the storage oil volume was required to fully replenish the degraded oil with reprocessed oil.

On 23 June 78 approximately 48 Kg (105 lbs) of oil remained in the tank. At a processor flow rate of 12 cc/min or 1.348 lb/hr, the entire volume of oil would be displaced in approximately 78 hours. The approximate time required to replace two volumes would be 156 hours or approximately 0.93 weeks. The predicted sidedraw composition of the third week should then represent the oil tank composition during the fourth week of processor operation. The sidedraw composition resulting from the second processor simulation run was therefore used to predict the processor performance after four weeks of operation and so on. Since the temperatures predicted using this method are so close to the selected sequence of test temperatures, one can also conclude from these results that approximately two volumes of oil are required to pass through the processor to fully reprocess the oil.

In summary, the sidestream processor simulation has determined quantitatively the effects of heat loss and feedstream

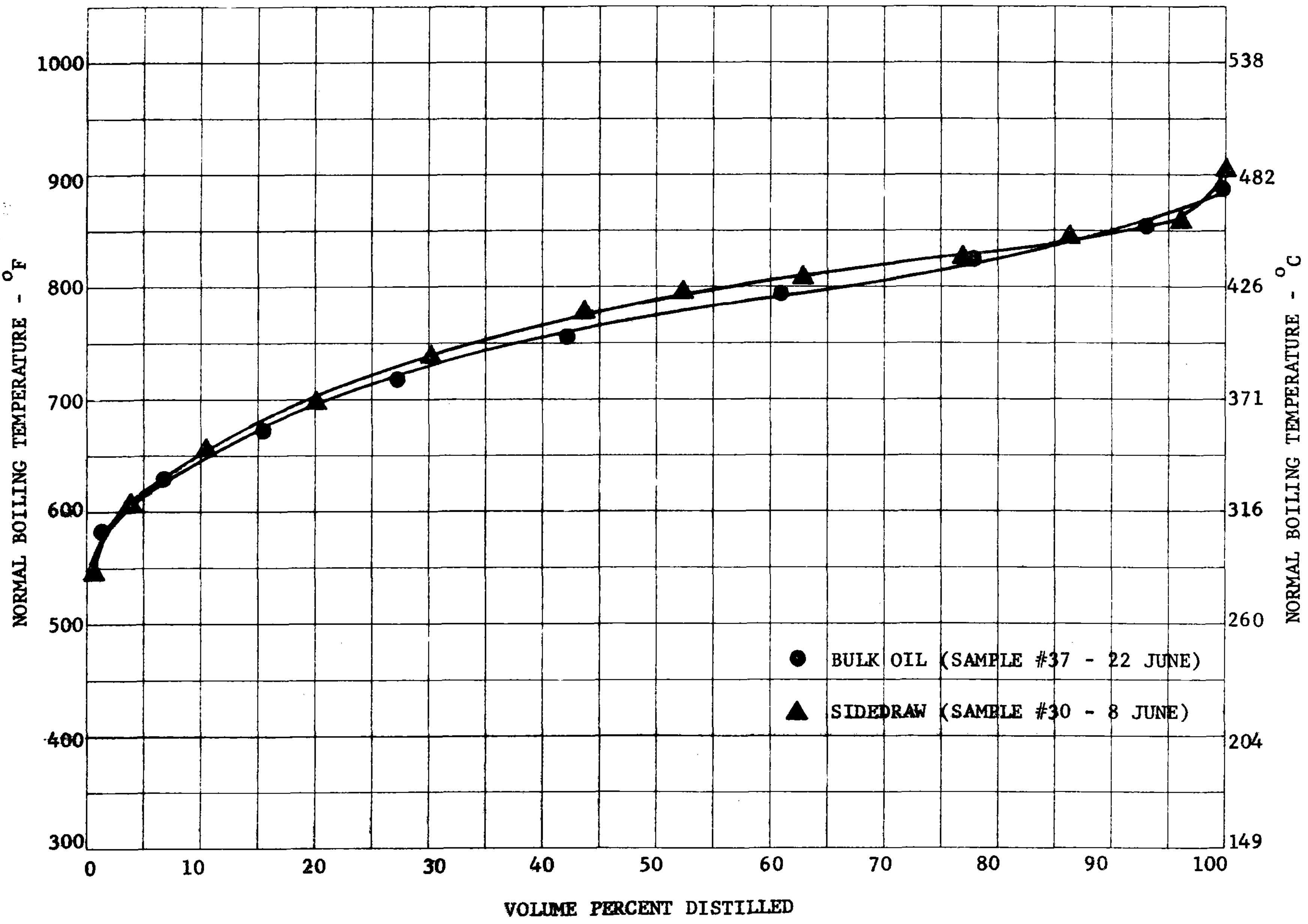


FIGURE IV-29 BULK OIL/SIDEDRAW COMPARISON composition on distillation performance. Through the use of the DISTILL simulation program it has been shown that approximately two oil volumes must be processed for the oil to obtain sidestream composition characteristics. Shown in Figure IV-30 is a composite of the simulated sidestream compositions. When compared with the actual bulk oil compositions in Figure IV-12, the simulated distillation curves agree very closely. The DISTILL model provides a valuable tool for future processor design concepts and assessment of energy requirements for its operation.

c. Processor Operation Assessment - Judging from the results of the simulation and comparison with experimental data, the sidestream processor performed as designed. High and low boiling fractions were separated from the feed oil and the column responded to changing feed composition in a predictable manner. However, there were some control problems as a result of this design that should be avoided in future design considerations. Some of these problems were chiefly related to the small size of the column and the location of the feedstream entering the column.

The small size of the column and the limited amount of insulation caused relatively large heat losses. The heat loss through the column shell required high reboiler energy loads to drive the column. A larger scale system would have much lower heat losses on a per unit feed basis with proper insulation so the energy requirements to drive the column would be much less. For a column with no heat loss, the reboiler load would be 9.7920 Kwatt-hr/mole feed (calculated previously) which would represent the minimum energy requirement for column operation.

The oil feed was mixed with reboiler oil at a ratio of approximately 30:1 before the mixture entered the column. The design was incorporated as a minimal cost method to heat the oil feed before it entered the column. Unfortunately this method

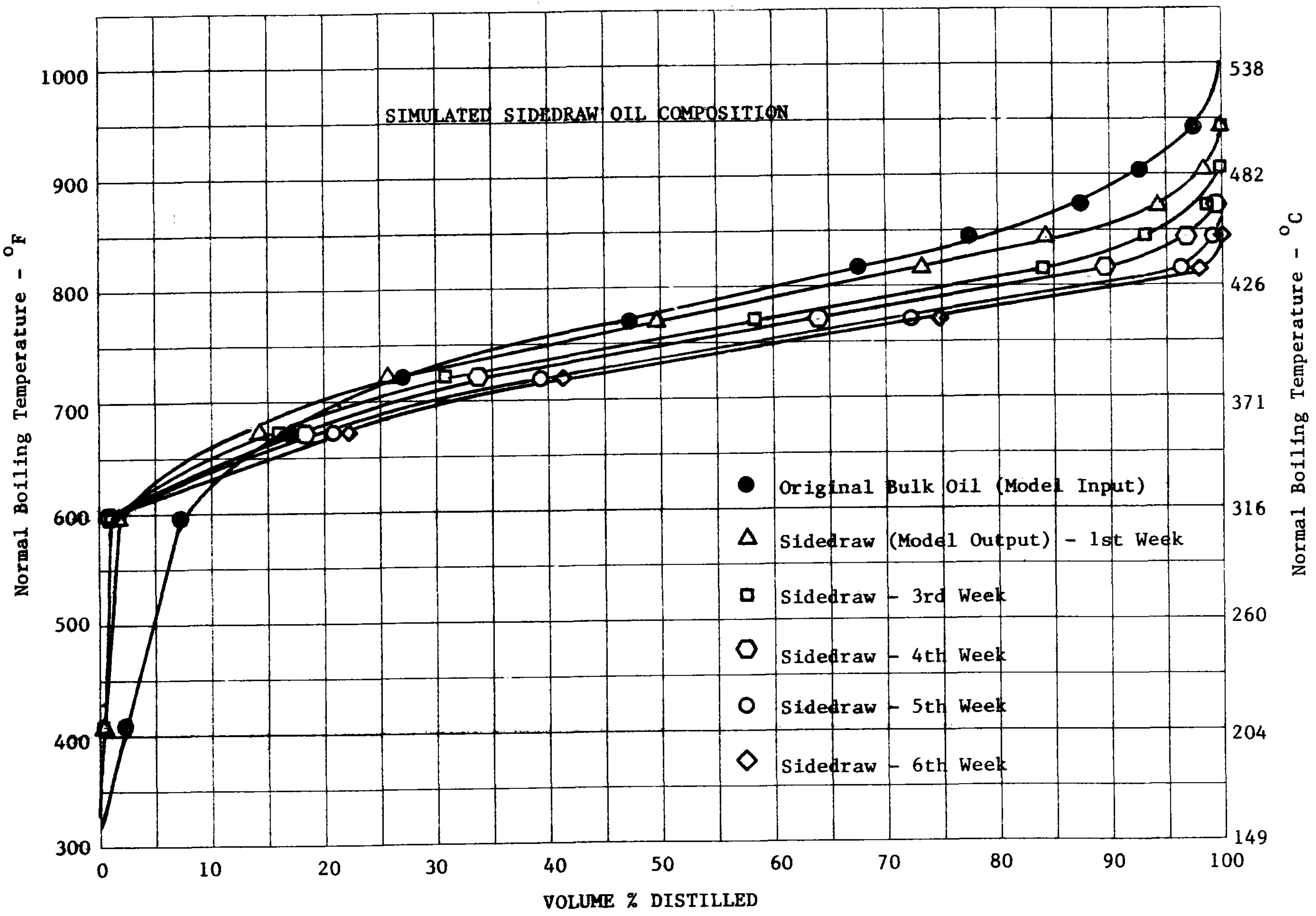


FIGURE IV-30 **SIMULATED SIDEDRAW COMPOSITION** had some drawbacks during testing in Phase II. The feed and recycled bottoms (or reboiler) oil entered 50.8 cm (20 in.) above the bottom of the column. This section between the column bottom and feed point was cooler than the feed temperatures throughout the test due to a heater burnout in this section. Therefore part of the feed was actually entrained to these lower portions with the recycled bottoms oil. Due to the small volume in the bottom of the column which served as a reservoir and the method of tight level control of this reservoir the purity of the high boiling fraction was diluted with fresh feed oil. The additional requirement to maintain the proper reservoir level and correct feed temperature with varying feed composition resulted in close operator control of the recycle heater.

Another problem which affected the operation of the process was the lack of adequate control on tops reflux flowrate back to the column. The reflux system tested used a gravity feed of reflux from an elevated reservoir. For good separation performance the reflux ratio, defined by the molar flowrate back to the column divided by the molar top product flowrate, should be relatively constant. Due to the changing feed composition and the low flowrates of this system, reflux flow control was impossible in Phase II tests. As shown in the test data the temperatures at the top of the column remained above $135^{o}C$ ($275^{o}F$) for the duration of the tests. From simulation runs it was shown that for a constant reflux flowrate the top of the column temperatures should become lower. This comparison indicates that the actual reflux flowrate in the test became smaller as the test progressed thus keeping the top of the column at temperatures above $135^{o}C$ ($275^{o}F$). A larger scale column with gravity reflux control would not have these control problems because of the larger flowrates.

Some concern about possible flooding in the column during Phase II tests occurred when the pressure drop across the entire column, as indicated by a differential pressure cell, fluctuated between 0 to 2.0 mm Hg. These fluctuations were not evident however on the absolute pressure gauge located at the bottom of the column. Flooding in a packed column is the phenomena occurring when the vapor velocity up the column is great enough to entrain the liquid flow down the column causing the liquid to hold-up and accumulate until either the vapor pushes the pocket of liquid up and out of the column or the gas bubbles through the liquid. Flooding causes an increase in pressure drop and results in unstable column operation. The phenomena also reduces the mass transfer between liquid and vapor phases and thus reduces separation efficiency.

Using the results of the DISTILL computer simulation for liquid and vapor flowrates in the column, calculations were performed in determining whether the column was actually operating in a flooding regime. Shown in Figure IV-31 is a correlation of flood points for packed columns. Using average gas and liquid flowrates calculated from DISTILL, the following values were determined for the correlation comparison:

G = 59.29 gas mass rate, gm/(hr-cm^2)
L = 52.66 liquid velocity, gm/(hr-cm^2)
a_p = 2.49 total area of packing, cm^2/cm^3
ϵ = 0.83 fractional voids in dry packing
ρ_g = 0.0011 gas density, gm/cc
ρ_1 = 0.8554 liquid density, gm/cc
μ_1 = 1.0 liquid viscosity, centipoise
g_c = 980.66 gravitational constant, cm/sec^2
U_t = 168.6 superficial gas velocity, cm/sec

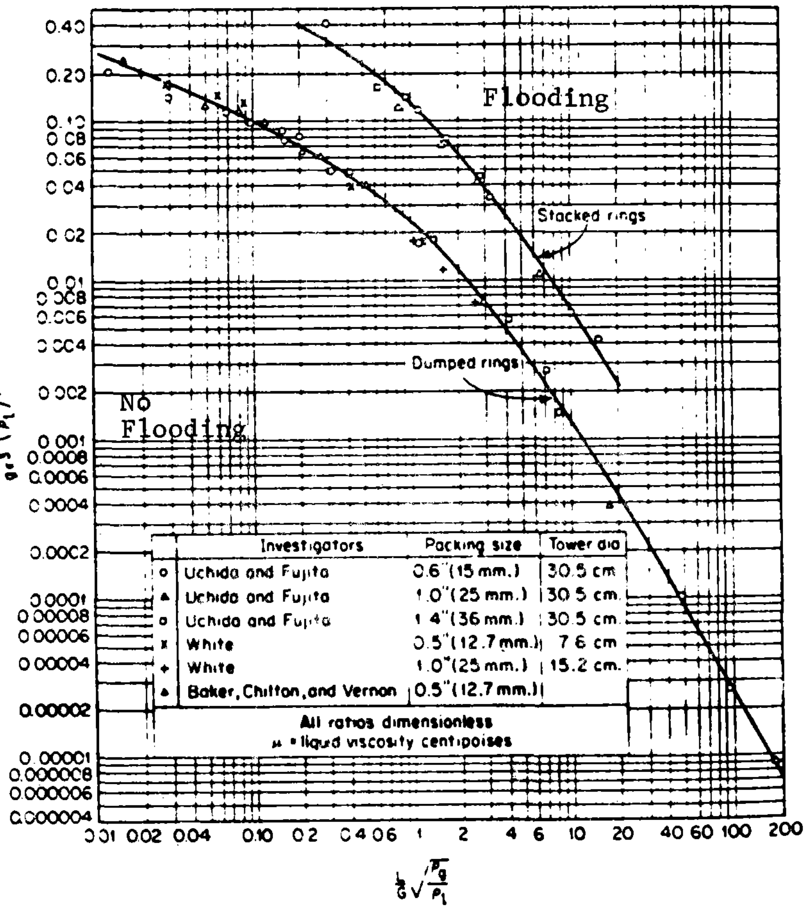


FIGURE IV-31

GENERALIZED CORRELATION OF FLOOD POINTS, PACKED COLUMNS (REF. 10)

Using these values for the calculation of the coordinates in Figure IV-31 results in 0.0091 for the abscissa and 0.0157 for the ordinate. These values show that the column was actually performing well below the flood point. A second calculation was performed to determine the pressure drop across the column using a form of the orifice equation:

$$\Delta P = C_1 \rho_g U_t^2 \qquad \text{(IV-12)}$$

ΔP = pressure drop, mm Hg/m packing
C_1 = constant for a given packing, mmHg (sec^2)/gm
ρ_g = 110 gas density, gm/m^3
U_t = 1.69 superficial gas velocity, m/sec

Using a value of $C_1 = 0.0021$ which represents the maximum value of this coefficient for any packing, the pressure drop is 0.619 mmHg/m packing. For 1.8 m (6 ft) of packing the total pressure drop is 0.606 in. H_2O or 1.13 mm Hg. The minimum pressure drop at which flooding occurs is dependent upon the type of packing. For most packings this minimum pressure drop is 15.32 mmHg/m packing (Ref. 10, Table 18-10). Therefore from pressure drop calculations, the column was also determined to be operating below the flooding point.

One can conclude from these calculations that the column was not operating in a flooding regime. Therefore the DP cell sporadic pressure fluctuations were probably caused by faulty instrumentation and did not represent unstable column operation.

4. Recommended Processor Improvements

The current processor design tested in Phase II has shown that it can indeed separate components within an oil mixture very adequately. There is also some evidence, which at this time is very preliminary, that the processor had an effect on the oil degradation rate. The reduction in degradation rate in Phase II as compared with tests conducted in Phase I is not currently known to be either the result of venting or the processor. Some modifications can be incorporated into the existing design to effect better process control. These are:

1) Determine the optimum feed location in the column (this location will be somewhere other than the reboiler stage used in Phase II).
2) If the feed needs to be preheated prior to entering the column use a two fluid heat exchanger with the recycle bottoms.
3) Use an elevated reflux reservoir and control the reflux and top product flowrates via gravity (this eliminates any parasitic pumping costs of other methods of flow control).
4) Provide an adequate vessel volume for a bottoms products reboiler and use a forced circulation heating system--the reboiler is the first stage of the column.
5) Use a metering pump in the feed line between the storage tank and the processor to insure a constant flowrate to the column.
6) Provide a condensate trap in the vacuum line between the processor and the vacuum pump.
7) Minimize processor heat loss with adequate insulation and thus reduce reboiler energy load requirements.

These modifications would improve the separation efficiency of the processor and reduce the requirement for operator surveillance.

C. SUMMARY

From the two phases of effort performed under this program, oil degradation rates for a mini-pilot plant scale system have been determined to be lower than previously reported in the literature. The differences can be attributed in large part to effects of tank ullage pressure on decomposition rate. The decomposition mechanism including an autocatalytic step can explain the effects of venting and/or processing on reducing decomposition rate. A sidestream processor was designed, constructed, and tested in this program to assess its effect on oil degradation rate. The tests performed in Phase II with a vacuum distillation sidestream processor indicate that degradation rates were lower than the Phase I system without a processor tested. However, it is not known at the present time whether the lower degradation rate is attributable to venting or the processor. A follow-on Phase III effort is recommended to resolve this question. This follow-on program would provide additional test results and evaluation regarding the characteristics of oil degradation for a mini-pilot scale storage tank in a vented mode. The recommended plan also includes the development of a thermal degradation model and economic analysis of the benefits of a sidestream processor.

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APPENDIX A

TEST SCHEMATICS

APPENDIX A: TEST SCHEMATICS

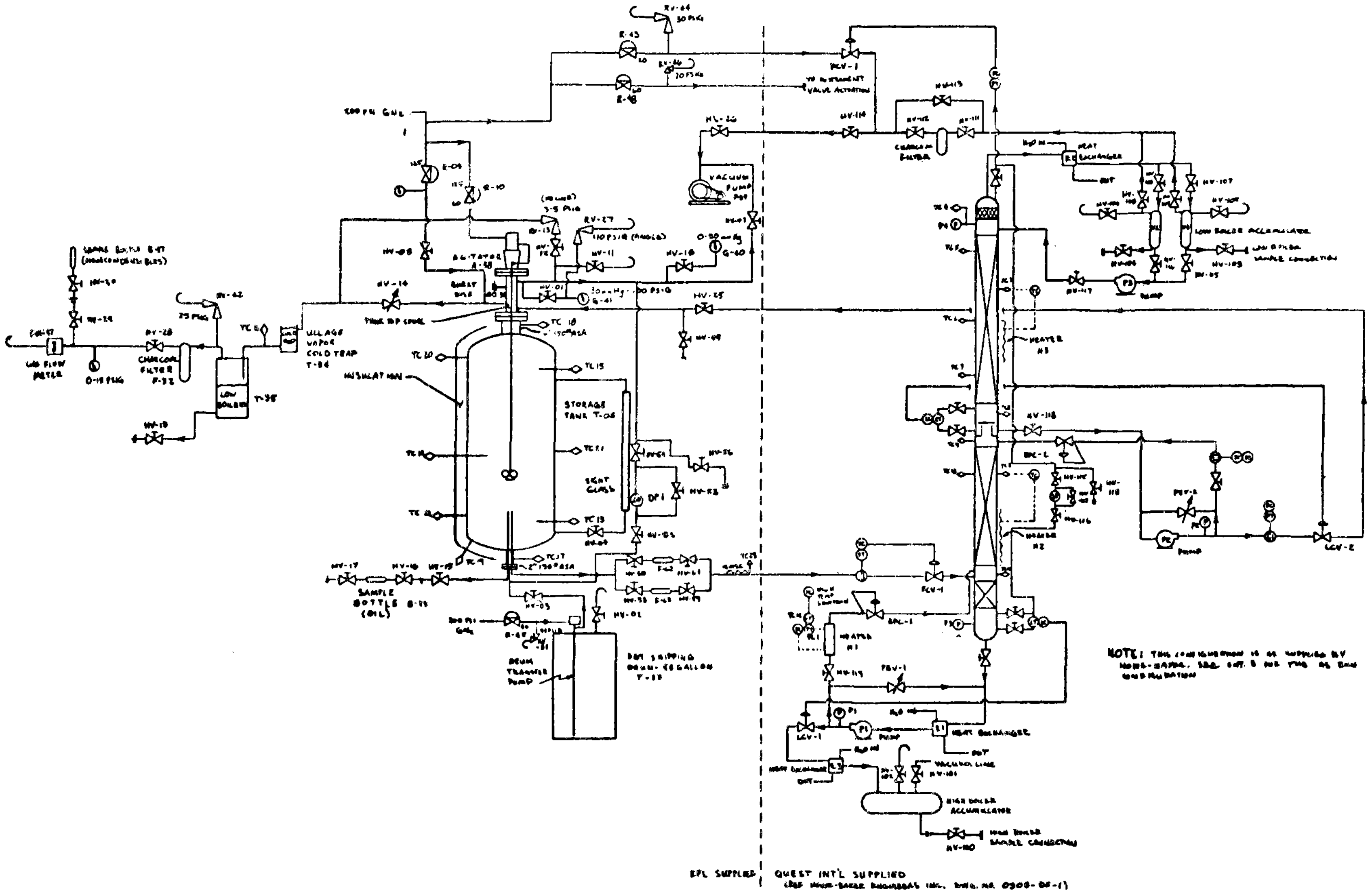
Included in this appendix are test schematics illustrating equipment units, instrumentation, valves, and process flow lines. The process flow diagrams included are:

(1) Phase II System Schematic

(2) Sidestream Processor Schematic

In schematic (1) is the Phase I system schematic which consisted on the storage tank subysstem only. Schematic (2) contains the final detailed flow schematic of the processor.

A		ADDED THIS SHEET.	12/6/77	
B		AS BUILT CHANGES, REPLACED SIGHT GLASS W/ ΔP CELL, ELIMINATED [illegible] ON CAMERAS, ADDED GAS FLOW METER, ADDED [illegible]	3/2/78	
C		ADDED SHEET 3		



-10 PHASE II SYSTEM SCHEMATIC

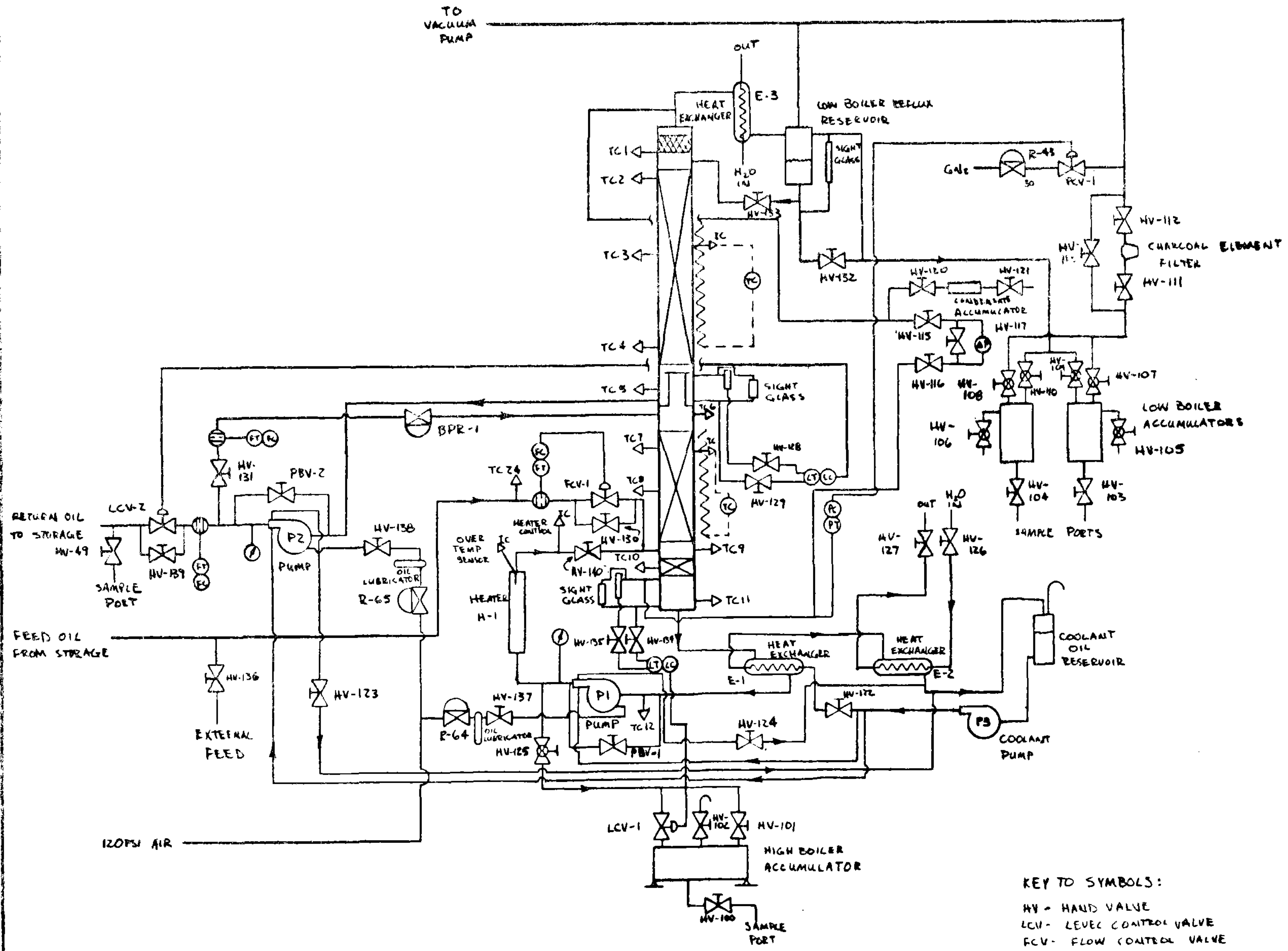
ADDITIONAL HARDWARE FOR PHASE II

	F-63	INLINE FILTER
	F-62	[illegible] LINE FILTER
-24	HV-61	FILTER ISO
-24	HV 60	FILTER ISO
24	HV-59	FILTER ISO
-24	HV-58	FILTER ISO
-5	SV 57	[illegible]
24	HV-56	DELTA P CLEAN
24	HV 55	DELTA P BYPASS
-24	HV 54	DELTA P ISOLATION
-24	HV-53	DELTA P ISOLATION
17	B-52	[illegible] SAMPLE BOTTLE
24	HV 51	SAMPLE BOTTLE ISO
-24	HV 50	SAMPLE BOTTLE ISO
-24	HV-49	[illegible] SAMPLE ISO.
7	R-48	[illegible] CONTROL [illegible] REG.
	FM 47	GAS FLOW METER
-15	SE-46	[illegible]
-15	P 45	[illegible] PUMP [illegible]
-13	RV 44	[illegible]
-12	R-43	[illegible]

EPL 6301677C

FIGURE A-1

SYM	ZONE	DESCRIPTION	DATE	APPROVED
C		ADDED THIS SHEET	5/3/78	



KEY TO SYMBOLS:

HV - HAND VALVE
LCV - LEVEL CONTROL VALVE
FCV - FLOW CONTROL VALVE
PCV - PRESSURE CONTROL VALVE
PBV - PUMP BYPASS VALVE
E - HEAT EXCHANGER
P - PUMP
R - REGULATOR
TC - THERMOCOUPLE
H - HEATER
PT - PRESSURE TRANSMITTER
PC - PRESSURE CONTROLLER
LT - LEVEL TRANSMITTER
LC - LEVEL CONTROLLER
FT - FLOW TRANSMITTER
FC - FLOW CONTROLLER
TC - TEMPERATURE CONTROLLER
ΔP - DIFFERENTIAL PRESSURE

AS OF 11 AUG 78

INTERPRET DWG PER MIL-STD-100
UNLESS OTHERWISE SPECIFIED
DIMENSIONS ARE IN INCHES AND ARE AFTER PLATING
TOLERANCES ON: FRACTIONS ± 1/32 DECIMALS ± .1 ± .06 ± .010 ANGLES ± 1/2°
MACHINED SURFACES ✓
MIL - STATUS
INTERCHANGEABLE
REPLACEABLE
UNCONTROLLED

DRAWN BY DEPT 0483 DATE 5/3/78
CHECKER
STRESS ENGR
WT ENGR
MATL ENGR
RELIABILITY
GD ENGR
PROJECT
PART PRIORITY

MARTIN MARIETTA CORPORATION
DENVER DIVISION POST OFFICE BOX 179, DENVER, COLORADO 80201

HOWE-BAKER SIDESTREAM
PROCESSOR MODIFIED
BY EPL

SIZE D CODE IDENT NO 04236 EPL6301677C
SCALE N/A T-77-93 SHEET 3 of 3

EPL6301677C

FIGURE A-2

APPENDIX B

"AS RUN" TEST PROCEDURES

APPENDIX B: "AS RUN" TEST PROCEDURES

Contained in this report section are the test procedures used in performing tests in Phase I and Phase II. These procedures were modified during and at the end of each test duration and therefore represent "as run" methods. Included in this appendix is:

PHASE I

TEST PROCEDURE

1.0 SCOPE

1.1 This procedure provided the steps necessary to perform the development testing of Exxon Caloria HT-43 Heat Transfer oil to determine a large sample decomposition rate.

1.2 Test Objective

The purpose of testing was to generate decomposition rates for a large sample, approximately 50 gallons, which will then be compared with literature data to see if the decomposition rates are different from the published data using small samples.

2.0 SUPPORT REQUIREMENTS

2.1 Test Fixtures

2.1.1	EPL 6301681 Rev. A	Storage Tank with ASME Code Stamp
2.1.2	EPL 6301688	Skid
2.1.3	EPL 6301698	Tank Top Spool
2.1.4	EPL 6301697	Panel Layout

2.2 Test Systems

2.2.1	EPL 6301677	System Schematic
2.2.2	EPL 6301682	Heater Installation and Control Circuitry
2.2.3	Quest International	Sidestream Distillation Column Schematic
2.2.4	EPL Instrumentation Sheets, T-77-93	

2.3 Test Methods and Controls, M-67-45

2.4 Safety Standards Manual, M-61-58

2.5 Standard Procedure 101.1

2.6 Electrical Requirements

2.6.1 110 VAC, 60 Hz

2.6.2 220 VAC, Single Phase

2.7 Mechanical Requirements

2.7.1 125 psi GN_2

2.7.2 Facility Water

2.8 Safety Equipment

2.8.1 Asbestos Gloves

2.8.2 Face Shields

2.8.3 Flame Retardent Coveralls

2.8.4 CO_2 Bottles

3.0 SPECIAL CONSIDERATIONS

3.1 Caution and Warning

The material written in a CAUTION or WARNING precedes the information it is intended to emphasize. A CAUTION is used to prevent personnel from damaging equipment. A WARNING is used to prevent personnel from endangering their safety and that of others.

3.2 Procedure Changes

Red line changes to be incorporated into the test procedure will require unanimous approval of the test conductor, project engineer and safety.

3.3 Cleanliness Control

System cleanliness will be controlled to insure accurate and representative sampling of test fluid. System will be verified clean prior to test start.

3.4 Protection of Assembly Structural Integrity

Careful handling of system components will be required during assembly, transporting and test activities to prevent damage to those components, particularly the storage vessel and sidestream processor.

3.5 Safety

All testing performed by this procedure will be performed in compliance with the requirements of Safety Manual M-61-58.

3.6 General Safety Requirements

3.6.1 When an unsafe condition exists, the Test Conductor shall take whatever immediate action is necessary to prevent injury to personnel and/or damage to equipment.

3.6.2 The Test Conductor and Safety are responsible for providing safe working conditions and insuring personnel compliance with all safety rules and regulations.

3.6.3 Personnel involved in testing will be responsible for reviewing the procedure prior to each test for specific safety hazards that may be encountered during the test.

3.6.4 Deviations from safety standards and regulations will be made only with the concurrence of safety.

3.6.5 The Test Conductor will insure that only properly trained personnel participate in the operation.

3.6.6 Safety documents listed in the reference section shall be adhered to.

3.6.7 Where safety clothing or equipment is to be used, the test will not start until such items are in the test area and ready for immediate use.

3.6.8 Operational areas shall be kept clean and orderly, free of trash and combustibles.

3.6.9 The Test Conductor shall maintain personnel control around the operating area.

3.7 Pressurization Safety Requirements

3.7.1 No repairs shall be made to pressurized systems or components.

3.7.2 Upon the direction of Safety, personnel working in the vicinity of pressurized systems shall wear safety approved eye protection.

3.8 Electrical Safety Requirements

3.8.1 Electrical connections shall not be connected or disconnected while energized.

3.8.2 All electrical connections shall be examined prior to mating for damage, corrosion, and foreign materials. Excessive forces, which may result in bent pins, shall not be applied during mating.

3.8.3 Wires/cables will be routed to avoid chafing, cuts, kinks, etc..

3.9 Test Sequence

The sequence of testing shown in this document is mandatory. Rearrangement of the sequence will be permitted only with the approval of EPL Engineering and the Program Manager.

4.0 OPERATIONS

4.1 Prerequisites

4.1.1 The test will be conducted in the F1 Building.

4.1.2 One working day prior to test start day, notify Safety (ext. 4444), that testing will commence the following day.

4.1.3 Insure the test system is built according to the system schematic and has been verified ready by test engineer (W. Morgan, ext. 3818) and instrumentation engineer (A. Anderson, ext. 3925).

4.1.4 Verify the heat transfer oil is on hand and the drum is properly plumbed for transfer to storage tank.

4.1.5 Draw a 1.5 liter oil sample from 55 gallon drum for analysis. See Section 4.9.

4.2 Pretest Leak Check and System Checkout

4.2.1 Verify the following subsystems ready for operation:

X 4.2.1.1 Instrumentation System

X 4.2.1.2 Heater Power Supply

X 4.2.1.3 Individual Tank Heaters

X 4.2.1.4 Stirrer Air Motor

X 4.2.1.5 Vacuum Supply

X 4.2.1.6 Valves all Closed, relief valves properly set and tagged

X 4.2.1.7 Nitrogen Supply

X 4.2.1.8 Fire CO_2 Bottles

4.2.2 System Leak Check

4.2.2.1 Announce: "F1 Building is now in an AMBER condition for leak check." Place area in AMBER.

X 4.2.2.2 Open HV-04 and HV-05, Sight Glass Isolation

X 4.2.2.3 Load R-09 Tank GN_2 Supply Regulator to 25 psig and open HV-01, Pressure Gage Isolation

X 4.2.2.4 Slowly open HV-08 Tank Pressure Isolation and allow pressure to stabilize on G-41 Tank Pressure Gage.

X 4.2.2.5 Check for leaks on all fittings connected to tank.

NOTE

Any time a leak requires more than tightening a B-nut to stop, close HV-08, vent pressure through HV-11, close HV-11, repair leak source and start step 4.2.2.2 again.

X 4.2.2.6 Load R-09 to 75 psig and allow pressure to stabilize.

X 4.2.2.7 Check for leaks.

X 4.2.2.8 Load R-09 to 100 psig and allow pressure to stabilize. Hold for 10 minutes while monitoring tank pressure.

X 4.2.2.9 Check for leaks. Check carefully sight glass.

___ 4.2.2.10

X 4.2.2.11 Slowly open HV-11 Tank Vent.

X 4.2.2.12 Unload R-09 to 5 psig and allow pressure to stabilize.

X 4.2.2.13 Close HV-11.

X 4.2.2.14 Open HV-28 N.C. Sample Supply. Sample bottle will not be attached.

X 4.2.2.15 Open HV-12 Low Pressure Relief Isolation and verify RV-13 low pressure Relief is discharging gas through HV-28.

NOTE

If RV-13 does not relieve, slowly increase pressure through R-09 and note cracking pressure. Do not pressurize more than 25 psig. Notify test engineer for determination of cracking pressure acceptability.

X 4.2.2.16 Open Drain Plug on HV-04 Sight Glass Isolation.

X 4.2.2.17 Open HV-14, Ullage Sample Supply, and allow gas to flow through ullage condenser and catch tank for 2 minutes.

X 4.2.2.18 Close HV-12.

X 4.2.2.19 Close HV-28.

X 4.2.2.20 Close HV-14

X 4.2.2.21 Install gauge in drain plug on HV-04.

X 4.2.2.22 Open HV-24 Column Supply and purge for 1 minute.

X 4.2.2.23 Close HV-24.

X 4.2.2.24 Open HV-15 oil bottle supply and purge for 1 minute.

X 4.2.2.25 Close HV-15.

X 4.2.2.26 Unload R-09 to zero psig.

X 4.2.2.27 Open HV-11 and vent tank to 0 psig, and close HV-11 and HV-08.

X 4.2.2.28 Announce "F1 is now in a GREEN condition, leak check complete." Return area to GREEN.

NOTE

After all systems have been determined to be performing within the specified limits, the tank will be charged with Caloria HT-43 Heat Transfer Oil, a product of Exxon Corporation.

4.3 Charging Tank with Oil

NOTE

The storage tank will be charged with 50 gallons oil. The oil must be de-oxygenated and protected from exposure to air. Exxon states that as received, HT-43 may contain 5 volumes dissolved oxygen, or nearly 1% by weight oxygen. This gas must be removed initially to prevent oxidation during heating.

X 4.3.1 Turn on vacuum pump.

X 4.3.2 Close HV-01 Tank Pressure Gauge Isolation.

X 4.3.3 Open HV-07 Tank Vacuum Isolation.

X 4.3.4 Attach tank fill line to 55 gallon drum line if not previously done.

X 4.3.5 Open HV-02 Oil Drum Pressure Vent.

____ 4.3.6 Open HV-03 one turn and allow oil to flow from drum to storage tank.

X 4.3.7 Examine vacuum sight tube on vacuum pump to ascertain whether the oil has foamed sufficiently to reach vacuum exit port.

NOTE

If oil is observed in vacuum pump sight tube, or tank sight glass, reduce flow of oil until oil is no longer observed.

X 4.3.8 When oil level reaches 50 gallons on tank sight glass, close HV-03.

X 4.3.9 Close HV-02, HV-05, and HV-04.

X 4.3.10 Open HV-18 vacuum Gage Isolation.

X 4.3.11 Start air motor on tank agitator, by loading R-10 Agitator GN_2 Supply Regulator to approx. 40 psig.

X 4.3.12 Allow oil to stir under vacuum for 24 hours. Maintain vacuum at 7 mm Hg max. as read on G-40.

4.4 Initial Heating of Oil

NOTE

The oil is now ready to be brought to temperature.

X 4.4.1 Actuate instrumentation system for monitoring TC1-TC10.

X 4.4.2 Check G-40 vacuum gage to be 10 mm Hg or less.

NOTE

If tank pressure is above 10 mm Hg, evacuation and stirring should be continued until pressure is below 10 mm Hg.

4.4.3 Oil Tank Warmup Procedure

X 4.4.3.1 Check Power Inputs to Variacs and Verify Proper Connection

Variac 1 ________ Variac 2 ________ Variac 3 ________
Variac 4 ________ Variac 5 ________

X 4.4.3.2 Check Heater Connections to Variacs and Verify Proper Connection

Heater 1 ________ Heater 2 ________ Heater 3 ________
Heater 4 ________ Heater 5 ________ Heater 6 ________
Heater 7 ________ Heater 8 ________

X 4.4.3.3 Check Thermocouple to readout connections and verify proper connections.

TC1 ______ TC2 ______ TC3 ______ TC4 ______
TC5 ______ TC6 ______ TC7 ______ TC8 ______
TC9 ______ TC10 ______

X 4.4.3.4 Check and verify that thermocouple readouts are operating properly. Enter initial tank temperatures in log.

X 4.4.3.5 Set overtemperature kill at 655°F.

OT1 ______ OT2 ______ OT3 ______

X 4.4.3.6 Check oil level in tank and record level in log.

X 4.4.3.7 Turn on variacs to 240 V. output.

X 4.4.3.8 Record beginning warmup date and time here and in log.

Date 24 Jan Time 0805

X 4.4.3.9 Record all thermocouple temperatures on Data Sheet 4 times every

X 4.4.3.10 Reduce power on Variac 3 to 107 V. when TC 10 reaches 602°F. Record date and time when power is reduced.

Date 25 Jan Time 0030

X 4.4.3.11 Reduce power on variac 2 to 107 V. when TC9 reaches 604°F. Record date and time power is reduced.

Date 25 Jan Time 0030

X 4.4.3.12 Reduce power on variac 1 to 107 V. when TC8 reaches 630°F. Record date and time power is reduced.

Date 24 Jan Time 2005

X 4.4.3.13 Reduce power on variac 4 to 35% when TC6 reaches 600°F. Record date and time when power is reduced.

Date 24 Jan Time 1757

X 4.4.3.14 Reduce power on variac 5 to 19% when TC5 reaches 600°F. Record date and time when power is reduced.

Date 25 Jan Time 0005

X 4.4.3.15 As oil temperature begins to stabilize vary power level on variacs as needed to maintain an oil temperature of 600°F ±5°F. Record date, time, variac number, and amount of change in log whenever a change is made.

X 4.4.3.16 Open HV-12 Low Pressure Relief Isolation.

X 4.4.4 Monitor Pressure on G-40.

X 4.4.5 Monitor TC1, TC2, TC3.

X 4.4.6 When temperature reaches 200°F, shut off the vacuum pump for 30 minutes and observe pressure.

X 4.4.7 Turn off heaters 1-8.

X 4.4.8 Close HV-07.

X 4.4.9 Close HV-18.

X 4.4.10 Open HV-01, pressure gauge isolation.

X 4.4.11 Load R-09 to 5 psig.

X 4.4.12 Open HV-08 and monitor pressure on G-41.

X 4.4.13 When pressure reads 0-2 psig, close HV-08.

X 4.4.14 Remove burst disc plug.

X 4.4.15 Crack HV-08 ½ turn open.

X 4.4.16 Install burst disc and overboard vent line.

X 4.4.17 C Connect discharge lines from RV-27 and HV-17 to overboard vent line.

X 4.4.18 Close HV-08.

X 4.4.19 Load R-09 to 25 psig.

X 4.4.20 Open HV-08 and allow pressure to stabilize.

_X_ 4.4.21 Leak check burst disc face.

_X_ 4.4.22 Load R-09 to 75 psig and check for leaks.

_X_ 4.4.23 Load R-09 to 90 psig. Hold for 10 minutes and check for leaks. Check for leaks on stirrer and adjust packing if necessary.

_X_ 4.4.24 Vent pressure through HV-11.

_X_ 4.4.25 Close HV-11.

_X_ 4.4.26 Open HV-12.

_X_ 4.4.27 Open HV-28.

_X_ 4.4.28 Turn on heaters to full power.

_X_ 4.4.29 Turn off HV-26.

_X_ 4.4.30 Close HV-18.

_X_ 4.4.31 Close HV-07.

_X_ 4.4.32 When oil temperature stabilizes at $600^{\circ}F$, open HV-01 Tank Pressure Gauge Isolation. Remove the handle on HV-01. (This valve must remain open.)

_X_ 4.4.33 Close HV-12 low pressure relief isolation.

_X_ 4.4.34 Note oil level in data log.

X 4.5 Oil Sampling Procedure

X 4.5.1 Carefully open HV-15 and drain dead volume of oil from sample line into a beaker.

X 4.5.2 Shut valve HV-15.

X 4.5.3 Attach clean, evacuated and labelled sample bottle to coupling downstream from HV-15.

X 4.5.4 Open HV-15.

X 4.5.5 Open HV-16, and vacuum load sample.

X 4.5.6 Close HV-16

X 4.5.7 Close HV-15.

X 4.5.8 Allow sample bottle to cool to a low enough temperature to handle.

X 4.5.9 Disconnect sample bottle.

X 4.5.10 Transport sample bottle and dead volume oil to lab for disposition and analysis.

X 4.5.11 At the lab, weigh the sampled oil and dead volume oil.

X 4.5.12 Transfer sampled oil to bottles for shipment, storage and laboratory analysis.

X 4.5.13 Before sealing the sample bottles, blanket any dead space with nitrogen.

X 4.5.14 Record sampling data in the sample log and transmit the weight of oil removed to test log.

4.6 Long Duration Temperature Hold

NOTE

The oil will be held and stirred at temperature for the test duration (6 weeks) with weekday monitoring.

X 4.6.1 Monitor tank temperatures and pressure on a five day per week basis. Enter in data log.

X 4.6.1.1 Instructions for off duty hour monitoring by security.

X 4.6.1.2 Check G-41 Tank Pressure Gage on panel. Pressure should be less than 100 psig.

X 4.6.1.3 A horn will indicate an over temperature problem.

X 4.6.1.4 Check for oil leaks.

X 4.6.1.5 If pressure exceeds 100 psig or the horn is on call W. Morgan (781-3666), J. Myers (798-2895), A. Anderson (789-3660), or Safety (ext. 4444).

OIL TANK VENT PROCEDURE

X 4.6.2 When tank pressure reaches 37 psig start step 4.6.2.1.

X 4.6.2.1 Take data reading.

X 4.6.2.2 Adjust packing so motor stops at 70 psi. Set R-10 to 90 psi.

X 4.6.3 Load ullage vapor cold trap with ice water.

X 4.6.4 Attach sample bottle to tee in vent line. Assure bottle is evacuated.

X 4.6.5 Open H.V. 28, N.C.

X 4.6.6 Record gas meter reading, ambient temperature, TC 4, G 41 pressure, vent line pressure, number of turns on HV-14 and time on pressure vent data sheet.

X 4.6.8 Open HV-14 Monitor tank pressure, TC-4 and gas meter on data sheet.

X 4.6.9 Monitor G-41 tank pressure and adjust HV-14 to a pressure decay rate of 1 - 2 psig per minute.

X 4.6.9.1 Record all data on data sheet, as needed.

X 4.6.10 Monitor TC4. If temperature rises above 100°F slow decay rate with HV-14.

X 4.6.10.1 After flow through vent is initialized and steady, open sample valve on tee, take gas sample and record the time.

X 4.6.10.2 After 5 minutes, close sample valve and record the time.

X 4.6.11 When tank pressure reaches 15 psig, take gas sample as per above.

X 4.6.12 Close HV-14.

X 4.6.14 Close HV-28.

X 4.6.15 Place sample bottle underneath HV-19.

X 4.6.16 Open HV-19 and drain condensate into bottle.

X 4.6.17 When condensate has drained, close HV-19.

X 4.6.18 Take oil sample from HV-15 as per regular procedure.

X 4.6.19 Weigh the effluent and record in data log.

X 4.6.20 Remove low boiler condensing tank and catch tank and plumbing between HV-14, RV-42 and HV-19 for cleaning between vents.

X 4.6.21 Return above hardware system before next vent.

4.7 Test Termination Procedure

____ 4.7.1 Check position of all valves.

____ 4.7.2 Take oil sample from HV-15 as per regular procedure.

____ 4.7.2.1 Take data reading.

____ 4.7.2.2 Adjust packing so motor stops at 70 psi. Set R-10 to 90 psi.

____ 4.7.3 Load ullage vapor cold trap with ice water.

____ 4.7.4 Attach ullage gas sample bottle to tee in vent line. Assure bottle is evacuated.

____ 4.7.5 Open H.V. 28, N.C.

____ 4.7.6 Record gas meter reading, ambient temperature, TC 4, G 41 pressure, vent line pressure, number of turns on HV-14 and time on pressure vent data sheet.

____ 4.7.8 Open HV-14 Monitor tank pressure, TC-4 and gas meter on data sheet.

____ 4.7.9 Monitor G-41 tank pressure and adjust HV-14 to a pressure decay rate of 1 - 2 psig per minute.

____ 4.7.9.1 Record all data on data sheet, as needed.

____ 4.7.10 Monitor TC4. If temperature rises above 100°F slow decay rate with HV-14.

____ 4.7.10.1 After flow through vent is initialized and steady, open sample valve on tee, take gas sample and record the time.

____ 4.7.10.2 After 3 minutes, close sample valve and record the time.

____ 4.7.11 When tank pressure reaches 3 psig, take gas sample as per above.

____ 4.7.12 Close HV-14.

____ 4.7.14 Close HV-28.

____ 4.7.15 Place sample bottle underneath HV-19.

____ 4.7.16 Open HV-19 and drain condensate into bottle.

____ 4.7.17 When condensate has drained, close HV-19.

____ 4.7.18 Take oil sample from HV-15 as per regular procedure.

____ 4.7.19 Weigh the effluent and record in data log.

____ 4.7.20 Open HV-08.

____ 4.7.21 Set R-09 to 15 psig and allow GN$_2$ to backfill into tank.

____ 4.7.22 When G-41 reads 15 psig, close R-09.

____ 4.7.23 Close HV-08.

____ 4.7.24 Turn off all variacs and verify.

____V1 ____V2 ____V3 ____V4 ____V5

____ 4.7.25 Monitor and record oil temperature and tank pressure as needed.

____ 4.7.26 When oil temperature reaches 200°F take oil sample from HV-15 as per regular procedure.

____ 4.7.27 If tank pressure is less than 5 psig, open HV-08 and adjust R-09 until G-41 reads 5 psig.

____ 4.7.28 Weigh a clean, empty 55 gallon drum and record weight in log.

____ 4.7.29 Attach oil drain line to drum and HV-03.

____ 4.7.30 Open HV-02.

____ 4.7.31 Open HV-03 and allow oil to drain into drum.

____ 4.7.32 Ensure oil tank pressure is a constant 5 psig by adjusting R-09 as necessary.

____ 4.7.33 After all oil has drained into drum close HV-03 and HV-02.

____ 4.7.34 Weigh the drum and record weight in log.

____ 4.7.35 After waiting 30 minutes, open HV-03 and HV-02 to allow any residual oil to drain into drum.

____ 4.7.36 After all residual oil has drained into drum close HV-03 and HV-02.

____ 4.7.37 Weigh drum and record weight in log.

____ 4.7.38 After 30 minutes, repeat above procedure and keep repeating until no significant mass gain in drum is noted.

____ 4.7.39 Clean system as per cleanout procedure.

4.8 Oil Transfer from Storage Tank to Drum.

____4.8.1 Weigh empty 55 gallon drum and record weight in data log.

____4.8.2 Connect drum with associated plumbing for oil transfer.

____4.8.3 Load R-09 Tank Pressure Regulator to 5 psig.

____4.8.4 Open HV-08 Tank Pressure Isolation and allow pressure to stabilize.

____4.8.5 Open HV-02 Drum Pressure Vent.

____4.8.6 Slowly open HV-03 and allow oil to flow from the storage tank to the drum.

____4.8.7 After liquid level drops below the sight glass, wait 10 minutes and close HV-08.

____4.8.8 Unload R-09 to zero psig.

____4.8.9 Open HV-11 Tank Vent.

____4.8.10 Close HV-03.

____4.8.11 Close HV-02.

____4.8.12 Disconnect plumbing from drum.

____4.8.13 Weight drum to determine tare weight. Record in data log.

____4.8.14 Tag drum for future reference and place in storage.

____4.8.15 Remove cartridge from filter, being careful not to contaminate filter.

____4.8.16 Prepare cartridge for shipment to Sandia Laboratories.

4.9 Data Analysis

NOTE

The initial oil, the aged oil and the samples obtained during the test will be analized. A mass balance between the input materials and output materials will provide a baseline comparison between published data and test results.

4.9.1 The initial and final oil samples will be subjected to the following chemical analysis.

____4.9.1.1 Infrared spectra 4000-650 cm.$^{-1}$ This will provide data on oil chemical structural changes.

____4.9.1.2 NMR spectra. This will support conclusions derived from the infrared analysis.

____4.9.1.3 Iron Content.

____4.9.1.4 Chloride Content. Combined with the iron content, these data will provide understanding of possible catalytic effect.

____4.9.1.5 Titratable Aeidity. This will provide data relating to the amount of oxidation occurring in the oil.

____4.9.1.6 Molecular weight distribution to be performed by Sandia Livermore.

4.9.2 The initial weight of the oil should be equal to the sum of the final weight plus the samples taken plus the total weight collected in catch basins.

4.9.3 Data Log

NOTE

All data collected will be compliled in a final data book. This will include at least the follwwing:

4.9.3.1 Initial weight of oil.

4.9.3.2 Weight of oil collected in catch basins.

4.9.3.3 Sample weight.

4.9.3.4 Time vs. Pressure Plots.

4.9.3.5 Temperature history.

4.9.3.6 Chemical analysis of original oil at ambient.

4.9.3.7 Chemical analysis of original oil at 600°F.

4.9.3.8 Chemical analysis of final oil.

4.9.3.9 Chemical analysis of noncondensable, low boiler, and fluid samples during test.

4.9.3.10 Chemical analysis of final vent cold trap sample.

4.9.3.11 Mass balance during test.

4.10 Phase I Emergency Procedure

NOTE

For this procedure an emergency will be defined as any situation occurring to or by the test system that if left unchecked could

1) cause potential or real harm to personnel or equipment

2) compromise the accuracy of data collection.

4.10.1 General Emergency Procedure

4.10.1.1 Call Safety (ext. 4444) and notify the extent and type of problem.

4.10.1.2 Tell any unneeded personnel to leave the area.

4.10.1.3 Don any appropriate Safety Equipment.

4.10.1.4 If the problem is electrical, switch main breaker OFF.

4.10.1.5 If a subsystem is leaking, shutoff the nearest upstream source of the leak.

4.10.1.6 Note time and nature of emergency in log book.

4.10.2 Fire Caused by Oil Leak

____4.10.2.1 Shut off main breaker to cut all power to test equipment.

____4.10.2.2 Open HV-11 Tank Pressure Vent and dump tank pressure.

____4.10.2.3 If insulation is smoking or burning, remove the lagging surrounding the area affected.

____4.10.2.4 Cut out the insulation affected by the leak.

____4.10.2.5 If required douse any flames with CO_2.

____4.10.2.6 Ascertain the location of the leak source for determination of the best fix.

4.10.3 Fire Caused by Electrical Short

____4.10.3.1 Shut off main breaker to cut all power to test equipment.

____4.10.3.2 Douse any fire with CO_2 fire extinguisher.

4.10.4 High Temperature Shutdown

____4.10.4.1 Check to make sure all power to heaters has been cut off.

____4.10.4.2 Note time of shutdown and enter in data log.

____4.10.4.3 Note temperatures TC1, TC2, TC3, TC5, TC6, TC8, TC9, TC10 in data log.

____4.10.4.4 Do not restart heaters until cause of the shutdown has been determined.

____4.11 Interphase Cleaning

NOTE

Upon the completion of Phase I and after the oil is back in its shipping drum and stored, the tank and associated hardware and plumbing will be cleaned in preparation for Phase II. The tank will be washed with an additive free gasoline cut petroleum solvent (petroleum ether).

____4.11.1 Verify closed all hand valves.

____4.11.2 Remove ullage cold trap and low boiler tank and lines between H-14, HV-19 and RV-42.

____4.11.3 Verify petroleum solvent tank is on hand and filled with solvent.

____4.11.4 Attach solvent outlet to the inlet of HV-25, Dist. Column Return Isolation.

____4.11.5 Disconnect line between R-09 and HV-08.

____4.11.6 Run flexline from R-09 outlet to solvent tank pressure inlet.

____4.11.7 Load R-09 to 5 psig.

____4.11.8 Open HV-04, lower sight glass isolation.

____4.11.9 Place solvent cath basin under HV-24.

____4.11.10 Open HV-25 and wait one minute to allow pressure to stabilize.

____4.11.11 Close HV-25.

____4.11.12 Slowly open HV-24 and allow approx. one gallon of solvent to flow into catch basin.

____4.11.13 Close HV-24.

____4.11.14 Move catch basin under HV-15.

____4.11.15 Open HV-15 and allow approx. one gallon of solvent to flow into catch basin.

____4.11.16 Move catch basin under HV-03.

____4.11.17 Close HV-15.

____4.11.18 Open HV-03 and allow approx. one gallon of solvent to flow into catch basin.

____4.11.19 Close HV-03.

____4.11.20 Open HV-11 Tank Vent and zero the tank pressure.

____4.11.21 Close HV-11.

____4.11.22 Repeat steps 4.11.9-4.11.21 a total of 4 times, then continue with step 4.2.23.

____4.11.23 Open HV-03 and allow any remaining solvent to flow into catch basin.

____4.11.24 Close HV-03.

____4.11.25 Replumb Cold Trap, Low Boiler Tank and Hardware into system.

____4.11.26 Unload R-09 to zero psig.

____4.11.27 Disconnect solvent tank from HV-25.

____4.11.28 Replumb R-09 to HV-08.

____4.11.29 Open HV-04 drain a plug.

____4.11.30 Load R-09 to 100 psig.

____4.11.31 Open HV-15.

____4.11.32 Open HV-14.

____4.11.33 Open HV-25.

____4.11.34 Open HV-03.

____4.11.35 Open HV-25.

____4.11.36 Open HV-24.

____4.11.37 Open HV-28.

____4.11.38 Open HV-19.

____4.11.39 Open HV-08 Tank Pressure Reg. Isolation.

____4.11.40 Purge system with GN_2 for 8 hours.

____4.11.41 Close HV-14.

____4.11.42 Close HV-19.

____4.11.43 Close HV-28.

____4.11.44 Close HV-24.

____4.11.45 Close HV-25.

____4.11.46 Close HV-03.

____4.11.47 Close HV-15.

____4.11.48 Close HV-08.

____4.11.49 Open HV-11 and vent pressure.

____4.11.50 Close HV-11

____4.11.51 Replace HV-04 and drain plug.

____4.11.52 Unload R-09 to zero psig.

NOTE

System is now ready for Phase II Testing.

PHASE II

TEST PROCEDURES

1.0 SCOPE

1.1 This procedure provided the steps necessary to perform the development testing of Exxon Caloria HT-43 Heat Transfer oil to determine the effects of a side stream processor on decomposition rate.

1.2 Test Objective

The purpose of this test was to determine the effects of a side stream processor on oil decomposition rates for a large sample of approximately 30 gallons. Chemical analyses were performed to determine the performance of the side stream processor in reprocessing degraded oil.

2.0 SUPPORT REQUIREMENTS

2.1 Test Fixtures

2.1.1	EPL 6301681 Rev. B	Storage Tank with ASME Code Stamp
2.1.2	EPL 6301688	Skid
2.1.3	EPL 6301698	Tank Top Spool
2.1.4	EPL 6301697	Panel Layout

2.2 Test Systems

2.2.1	EPL 6301677 Rev. C	System Schematic (Tank - Processor)
2.2.2	EPL 6301682	Heater Installation and Control Circuitry

2.2.3 EPL Instrumentation Sheets, T-77-93

2.3 Test Methods and Controls, M-67-45

2.4 Safety Standards Manual, M-61-58

2.5 Standard Procedure 101.1

2.6 Electrical Requirements

2.6.1 110 VAC, 60 Hz

2.6.2 220 VAC, Single Phase

2.6.3 460 VAC, 3 Ø

2.7 Mechanical Requirements

2.7.1 125 psi GN_2

2.7.2 Facility Water

2.7.3 120 psig Compressed Air

2.8 Safety Equipment

2.8.1 Asbestos Gloves

2.8.2 Face Shields

2.8.3 Flame Retardent Coveralls

2.8.4 CO_2 Bottles

3.0 SPECIAL CONSIDERATIONS

3.1 Caution and Warning

The material written in a CAUTION or WARNING precedes the information it is intended to emphasize. A CAUTION is used to prevent personnel from damaging equipment. A WARNING is used to prevent personnel from endangering their safety and that of others.

3.2 Procedure Changes

Red line changes to be incorporated into the test procedure will require unanimous approval of the test conductor, project engineer and safety.

3.3 Cleanliness Control

System cleanliness will be controlled to insure accurate and representative sampling of test fluid. System will be verified clean prior to test start.

3.4 Protection of Assembly Structural Integrity

Careful handling of system components will be required during assembly, transporting and test activities to prevent damage to those components, particularly the storage vessel and sidestream processor.

3.5 Safety

All testing performed by this procedure will be performed in compliance with the requirements of Safety Manual M-61-58.

3.6 General Safety Requirements

3.6.1 When an unsafe condition exists, the Test Conductor shall take whatever immediate action is necessary to prevent injury to personnel and/or damage to equipment.

3.6.2 The Test Conductor and Safety are responsible for providing safe working conditions and insuring personnel compliance with all safety rules and regulations.

3.6.3 Personnel involved in testing will be responsible for reviewing the procedure prior to each test for specific safety hazards that may be encountered during the test.

3.6.4 Deviations from safety standards and regulations will be made only with the concurrence of safety.

3.6.5 The Test Conductor will insure that only properly trained personnel participate in the operation.

3.6.6 Safety documents listed in the reference section shall be adhered to.

3.6.7 Where safety clothing or equipment is to be used, the test will not start until such items are in the test area and ready for immediate use.

3.6.8 Operational areas shall be kept clean and orderly, free of trash and combustibles.

3.6.9 The Test Conductor shall maintain personnel control around the operating area.

3.7 Pressurization Safety Requirements

3.7.1 No repairs shall be made to pressurized systems or components.

3.7.2 Upon the direction of Safety, personnel working in the vicinity of pressurized systems shall wear safety approved eye protection.

3.8 Electrical Safety Requirements

3.8.1 Electrical connections shall not be connected or disconnected while energized.

3.8.2 All electrical connections shall be examined prior to mating for damage, corrosion, and foreign materials. Excessive forces, which may result in bent pins, shall not be applied during mating.

3.8.3 Wires/cables will be routed to avoid chafing, cuts, kinks, etc..

3.9 Test Sequence

The sequence of testing shown in this document is mandatory. Rearrangement of the sequence will be permitted only with the approval of EPL Engineering and the Program Manager.

4.0 OPERATIONS

The following sections represent the "as run" test procedure conducted for Phase II testing. The sections are described in the following summary reference list:

4.1 Prerequisites

4.2 Pretest Leak Check and System Checkout

4.3 Charging Tank with Oil

4.4 Initial Heating of Tank Oil

4.5 Long Duration Tank Oil Temperature Hold

4.6 Distillation Column Start-up Procedure

4.7 Distillation Column Operation

4.8 Oil Sampling Procedure

4.9 Test Termination Procedure

4.10 Chemical and Data Analysis

4.11 Emergency Procedures

4.12 System Cleanup

4.1 Prerequisites

4.1.1 The test will be conducted in the Fl Building.

4.1.2 One working day prior to test start day, notify Safety (ext. 4444), that testing will commence the following day.

4.1.3 Insure the test system is built according to the system schematic and has been verified ready by test engineer (A. Anderson, ext. 3925).

4.1.4 Verify the heat transfer oil is on hand and the drum is properly plumbed for transfer to storage tank.

4.1.5 Draw a 1.5 liter oil sample from 55 gallon drum for analysis. See Section 4.10.

4.2 Pretest Leak Check and System Checkout

4.2.1 Verify the following subsystems ready for operation:

X 4.2.1.1 Instrumentation System (Tank and Processor)
X 4.2.1.2 Heater Power Supply (Tank and Processor)
X 4.2.1.3 Individual Tank Heaters
X 4.2.1.4 Tank Stirrer Air Motor
X 4.2.1.5 Vacuum Supply
X 4.2.1.6 Valves all Closed, relief valves properly set and tagged
X 4.2.1.7 Nitrogen Supply
X 4.2.1.8 Fire Extinguisher CO_2 Bottles
X 4.2.1.9 Compressed Air Supply
X 4.2.1.10 Individual Processor Heaters

4.2.2 Tank System Leak Check

NOTE

The following steps in this section were not performed in Phase II of the test program since they were performed in Phase I. However, this procedure should be followed if the system has been in idle storage for a long period of time.

X 4.2.2.1 Announce: "F1 Building is now in an AMBER condition for leak check." Place area in AMBER.

X 4.2.2.2 Open HV-04 and HV-05, Sight Glass Isolation

X 4.2.2.3 Load R-09 Tank GN_2 Supply Regulator to 25 psig and open HV-01, Pressure Gage Isolation

X 4.2.2.4 Slowly open HV-08 Tank Pressure Isolation and allow pressure to stabilize on G-41 Tank Pressure Gage.

X 4.2.2.5 Check for leaks on all fittings connected to tank.

NOTE

Any time a leak requires more than tightening a B-nut to stop, close HV-08, vent pressure through HV-11, close HV-11, repair leak source and start step 4.2.2.2 again.

X 4.2.2.6 Load R-09 to 75 psig and allow pressure to stabilize.

X 4.2.2.7 Check for leaks.

X 4.2.2.8 Load R-09 to 100 psig and allow pressure to stabilize. Hold for 10 minutes while monitoring tank pressure.

X 4.2.2.9 Check for leaks. Check carefully sight glass.

___ 4.2.2.10

X 4.2.2.11 Slowly open HV-11 Tank Vent.

X 4.2.2.12 Unload R-09 to 5 psig and allow pressure to stabilize.

X 4.2.2.13 Close HV-11.

X 4.2.2.14 Open HV-28 N.C. Sample Supply. Sample bottle will not be attached.

X 4.2.2.15 Open HV-12 Low Pressure Relief Isolation and verify RV-13 low pressure Relief is discharging gas through HV-28.

NOTE

If RV-13 does not relieve, slowly increase pressure through R-09 and note cracking pressure. Do not pressurize more than 25 psig. Notify test engineer for determination of cracking pressure acceptability.

X 4.2.2.16 Open Drain Plug on HV-04 Sight Glass Isolation.

X 4.2.2.17 Open HV-14, Ullage Sample Supply, and allow gas to flow through ullage condenser and catch tank for 2 minutes.

X 4.2.2.18 Close HV-12.

X 4.2.2.19 Close HV-28.

X 4.2.2.20 Close HV-14

X 4.2.2.21 Install gauge in drain plug on HV-04.

X 4.2.2.22 Open HV-24 Column Supply and purge for 1 minute.

X 4.2.2.23 Close HV-24.

X 4.2.2.24 Open HV-15 oil bottle supply and purge for 1 minute.

X 4.2.2.25 Close HV-15.

X 4.2.2.26 Unload R-09 to zero psig.

X 4.2.2.27 Open HV-11 and vent tank to 0 psig, and close HV-11 and HV-08.

4.2.3 Processor System Leak Check

NOTE

Both positive and vacuum pressure leak checks will be performed on the processor system components. The leak check will be performed with no oil in either the column or the DP cell legs.

_X_ 4.2.3.1 Announce: "Fl Building is now in an AMBER condition for leak check". Place area in AMBER

_X_ 4.2.3.2 Check to insure adequate oil level in coolant oil reservoir.

_X_ 4.2.3.3 Verify closed the following valves:

_X_ 1) Hv-100

_X_ 2) Hv-101

_X_ 3) Hv-102

_X_ 4) Hv-103

_X_ 5) Hv-104

_X_ 6) Hv-105

_X_ 7) Hv-106

_X_ 8) Hv-109

_X_ 9) Hv-110

_X_ 10) Hv-113

_X_ 11) Hv-117

_X_ 12) Hv-121

_X_ 13) Hv-130

_X_ 14) Hv-131

_X_ 15) Hv-132

_X_ 16) Hv-139

_X_ 4.2.3.4 Open the following valves:

_X_ 1) Hv-107

_X_ 2) Hv-108

X 3) Hv-110

X 4) Hv-111

X 5) Hv-112

X 6) Hv-115

X 7) Hv-116

X 8) Hv-120

X 9) Hv-122

X 10) Hv-123

X 11) Hv-124

X 12) Hv-125

X 13) Hv-128

X 14) Hv-129

X 15) Hv-133

X 16) Hv-132

X 17) Hv-135

X 18) Hv-140

X 19) PBV-1

X 20) PBV-2

X 4.2.3.5 Fill vacuum pump with oil to proper level and turn vacuum pump on.

X 4.2.3.6 Open Hv-26 slowly to full open.

X 4.2.3.6.1 Load R-48 to 30 psig.

X 4.2.3.7 Set heater control temperatures for H-1, H-2, and H-3 at 000.

X 4.2.3.8 Turn SHUTDOWN BYPASS switch to "BYPASS".

X 4.2.3.9 Switch TOWER FEED, TOWER PRESSURE, BOTTOM LEVEL, and SIDEDRAW LEVEL controllers to "MANUAL".

X 4.2.3.10 Switch MAIN POWER to "ON".

X 4.2.3.11 Manually close all control valves by reducing output signal to "0".

X 4.2.3.12 Check indicated tower pressure on control panel. If pressure is less than 10 mm Hg then the column is virtually leak free; proceed to start up procedures. If indicated tower pressure is greater than 10 mm Hg a leak exists in the system and the next steps should be performed.

X 4.2.3.13 Isolate various processor subsystems where possible by closing valves and checking the effect on tower pressure.

X 4.2.3.14 If isolation of subsystems has no effect on tower vacuum pressure perform a positive leak check on the system with nitrogen or Helium.

- X 1) Turn off vacuum pump.
- X 2) Close valve, Hv-26.
- X 3) Close and open valves described in Sections 4.2.3.3 and 4.2.3.4.
- X 4) Connect nitrogen or helium supply to tower by connecting gas line at fitting near valve Hv-130.
- X 5) Pressurize column to approximately 20 psig. Do not exceed 30 psig. (Burst disk will rupture at 40 psig).
- X 6) If nitrogen is in column apply soap solution around all areas suspected of leakage.
- X 7) If helium is in column use helium gas detector around all areas suspected of leakage.
- X 8) After positive leak check has been performed disconnect gas line and reconnect fitting at valve Hv-130.

X 4.2.4 Announce "Fl Building is now in GREEN condition, leak check complete." Return area to GREEN.

4.3 Charging Tank with Oil

NOTE

The storage tank will be charged with 50 gallons oil. The oil must be de-oxygenated and protected from exposure to air. Exxon states that as received, HT-43 may contain 5 volumes dissolved oxygen, or nearly 1% by weight oxygen. This gas must be removed initially to prevent oxidation during heating.

X 4.3.1 Open Hv-11 Tank Vent.

X 4.3.2 Verify Oil drum is on a pallet and on the scale.

X 4.3.3 Record total oil plus drum weight in log.

X 4.3.4 Place transfer pump in drum bung hole.

X 4.3.5 Plumb GN_2 or compressed air supply to pump air motor.

X 4.3.6 Attach pump outlet to tank fill/drain line.

X 4.3.7 Load pump supply reg R-45 to 40 psig.

X 4.3.8 Open Hv-03 fill isolation.

X 4.3.9 Verify drum is vented.

X 4.3.10 Open Hv-04 sight glass isolation.

X 4.3.11 Fill tank to the 50 gallon level. Record final weight of drum and oil in log. Take a tank delta P reading.

X 4.3.12 Close Hv-03 and Hv-11.

X 4.3.13 Unload R-45 to 0 psig.

X 4.3.14 Remove drum from scale and save for cleaning and draining of remaining oil.

X 4.3.15 Place full 55 gallon drum of oil on pallet on scale and insert transfer pump for future use.

X 4.3.16 Open Hv-18 Vacuum Gage ISO. Close Hv-26.

X 4.3.17 Turn on vacuum pump.

X 4.3.18 Open Hv-07 vacuum line ISO.

X 4.3.19 Start agitator air motor by loading R-10 to 60 osig. Adjust packing to stall the motor at 40-45 psig.

X 4.14.20 Allow oil to stir under vacuum for 24 hours. Maintain vacuum at 10 mm Hg max. as read on G-40.

4.4 Initial Heating of Oil

NOTE

The oil is now ready to be brought to temperature.

X 4.4.1 Actuate instrumentation system for monitoring TC13-22.

X 4.4.2 Check G-40 vacuum gage to be 5 mm Hg or less. Shut off vacuum pump when temperature reaches 260°F.

NOTE

If tank pressure is above 5 mm Hg, evacuation and stirring should be continued until pressure is below 5 mm Hg.

X 4.4.3 Check Power Inputs to Variacs and verify proper connection.

Variac 1 X Variac 2 X Variac 3 X

Variac 4 X Variac 5 X Variac 6 X

X 4.4.4 Check Heater Connections to Variacs and verify proper connection.

Heater 1 X Heater 2 X Heater 3 X

Heater 4 X Heater 5 X Heater 6 X

Heater 7 X Heater 8 X Heater 9 X

X 4.4.5 Check Thermocouple to readout connections and verify proper connections.

TC13 X TC14 X TC15 X TC16 X

TC17 X TC18 X TC19 X TC20 X

TC21 X TC22 X TC23 X

X 4.4.6 Check and verify that thermocouple readouts are operating properly. Enter inital tank temperature in log.

X 4.4.7 Set overtemperature kill at 655°F.

OT1 X OT2 X OT3 X

X 4.4.8 Check oil level in tank and record level in log.

X 4.4.9 Turn on Variacs 1, 2 and 3 to 240 V output, Variacs 4 and 5 to 100% power.

X 4.4.10 Record beginning warmup date and time here and in log.

Date 22 March Time 0820

X 4.4.11 Record all thermocouple temperatures on Data Sheet X every hour.

X 4.4.12 Reduce power on Variac 1 to 120 V. when TC22 reaches 602°F. Record date and time power is reduced.

X 4.4.13 Reduce power on variac 2 to 120 V. when TC21 reaches 604°F. Record date and time power is reduced.

X 4.4.14 Reduce power on Variac 3 to 120 V. when TC20 reaches 630°F. Record date and time power is reduced.

Date 23 March Time 1400

X 4.4.15 Reduce power on variac 4 to 79% when TC18 reaches 600°F. Record date and time when power is reduced.

Date 27 March Time 0730

X 4.4.16 Reduce power on Variac 5 to 55% when TC17 reaches 600°F. Record date and time when power is reduced.

Date 27 March Time 0730

X 4.4.17 As oil temperature begins to stabilize vary power level on variacs as needed to maintain an oil temperature of 600°F ± 5°F. Record date, time, variac number, and amount of change in log whenever a change is made.

X 4.4.18 Open Hv-12 low pressure relief isolation.

X 4.4.19 Monitor pressure on G-40.

X 4.4.20 Monitor TC13, TC14, TC15.

X 4.4.21 Continue heating to 600°F and insure over temperature kill on controllers is properly set.

X 4.4.22 Close Hv-18.

X 4.4.23 Close Hv-07.

X 4.4.24 When oil temperature stabilizes at 600°F, open Hv-01 tank pressure gage isolation. Remove the handle on Hv-01. (This valve must remain open.)

X 4.4.25 Close Hv-12 Low Pressure Relief Isolation.

X 4.4.26 Observe tank pressure on G-41. If pressure is ambient or above (0 psig), system is ready for long time hold, go to Section 4.5.

X 4.4.27 If pressure is less than ambient, load R- 09 Tank Pressure Regulator to 5 psig.

X 4.4.28 Open Hv-09 and observe pressure on G-41.

X 4.4.29 Unload R-09 to zero.

X 4.4.30 When pressure stabilizes, close Hv-08.

X 4.4.31 Note oil level in Data Log.

4.5 Long Duration Temperature Hold

NOTE

The oil will be held and stirred at temperature in the vented tank for the test duration (6 weeks) with weekday monitoring.

X 4.5.1 Monitor tank temperatures on a five day per week basis. Enter in data log.

X 4.5.2 A horn will indicate an over temperature problem.

X 4.5.3 Check for oil leaks

X 4.5.4 If pressure exceeds atmospheric or the horn is on call W. Morgan (781-3666), D. Beshore (979-1095, A. Anderson (789-3660), or Safety (ext. 4444).

4.6 Distillation Column Start-Up Procedure

NOTE

The distillation system is protected by an overtemperature kill on the bottoms cycle heater element and an overpressure kill on the column pressure. The overtemperature kill is set manually on the control panel while the over pressure kill is set internally at 40 mm Hg. The kills on the system are provided by setting the SHUTDOWN BYPASS switch in the "SAFE" position. During the first few steps of the start-up procedure, the SHUTDOWN BYPASS switch will be placed in the "BYPASS" position. Care must be exercised during the first few steps under this mode that the bottoms recycle the heater element temperature does not exceed 750°F.

4.6.1 Open valves, Hv-126 and Hv-127, for condenser and oil coolant heat exchanger water flow.

4.6.2 Verify closed all bypass valves around control valves, Hv-130, and Hv-139.

4.6.3 Close the following valves:

_X_ 1) Hv-100

_X_ 2) Hv-101

_X_ 3) Hv-102

_X_ 4) Hv-103

_X_ 5) Hv-104

_X_ 6) Hv-105

_X_ 7) Hv-106

_X_ 8) Hv-109

_X_ 9) Hv-110

_X_ 12) Hv-113

_X_ 13) Hv-132

X 14) Hv-121

X 16) Hv-117

X 17) Hv-131

4.6.4 Open the following valves:

X 1) Hv-123 - ½ turn

X 2) Hv-124 - ½ turn

X 3) Hv-122

X 4) Hv-134

X 5) Hv-135

X 6) Hv-128

X 7) Hv-129

X 8) Hv-140

X 9) PBV-1

X 10) PBV-2

X 11) Hv-133

X 12) Hv-115

X 13) Hv-116

X 15) Hv-111

X 16) Hv-112

X 17) Hv-120

X 18) Hv-110

X 4.6.5 Open Hv-125, 1/4 turn.

X 4.6.6 Fill vacuum pump with vacuum pump oil to proper level and turn vacuum pump on.

X 4.6.7 Open Hv-26 slowly to full open.

X 4.6.8 Set heater control temperatures for H-1, H-2 and H-3 at 000.

X 4.6.9 Turn SHUTDOWN BYPASS switch to "BYPASS".

X 4.6.10 Purge and fill side draw and bottom level control legs with oil. Use oil reservoir connected with DP cell oil ports to purge cell and legs at same time.

X 4.6.11 Fill side draw chimney tray to ½ full as evidenced in sight glass.

X 4.6.12 Switch TOWER FEED, TOWER PRESSURE, BOTTOM LEVEL, and SIDEDRAW level controllers to "MANUAL".

X 4.6.13 Switch MAIN POWER to "ON".

X 4.6.14 Manually close all control valves by reducing output signal to "0".

X 4.6.15 Open Hv-60 and Hv-61.

X 4.6.16 Purge tower feed rate DP cell with oil.

X 4.6.17 Manually open TOWER FEED and fill bottom of column to approximately ½ full as evidenced in sight glass.

X 4.6.18 The following steps will be performed to obtain operation of pump, P-1 to approximately 600 rpm and discharge pressure of 15 psig.

1) Adjust pressure regulator, R-64, to 40 psig.
2) Partially open valve, Hv-137.
3) Adjust pump bypass, PBV-1.
4) Perform steps 2) and 3) until smooth operation of P-1 is attained at 15 psig.

X 4.6.19 Set SHUTDOWN BYPASS switch to "SAFE".

X 4.6.20 Adjust bottom and sidedraw DP cell suppression screw so indicated level on control panel is approximately the same as the indicated level in the side glass. For example, an actual level of 80% full in sight glass would correspond to 8.0 on controller indicated level.

X 4.6.21 Open Hv-102 to full open.

X 4.6.22 Set the following setpoints on controllers.

1) Tower feed - 6.5
2) Tower pressure - 7.0
3) Tower bottom level - 8.5
4) SIDEDRAW LEVEL - 8.5

X 4.6.23 Switch all controllers to "AUTO".

X 4.6.24 Open Hv-25.

X 4.6.25 Begin heating column up to temperature by performing the following steps:

1) Increase H-1 set point in increments of 5 not exceeding 2800 watts on power meter

   {Stop increasing set point when bottoms level begins to decrease.

2) Partially close valve, Hv-140 until the difference in heater element temperature, TC9, is approximately 40°F. Wait 5 minutes after each valve adjustment for system to equilibrate.

3) Increase H-3 setpoint in increments of 5 not exceeding 1000 watts on power meter.

4) When sidedraw level begins to increase start and operate pump P-2 by performing the following steps:

   a) Adjust pressure regulator, R-65, to 40 psig.
   b) Partially open valve, Hv-138.
   c) Adjust pump bypass, PBV-2.
   d) Perform steps b) and c) until smooth operation of P-2 is attained at 24 - 27 psig.

X 4.6.26 Monitor system and adjust heater controls until the column is up to temperature and thermocouple readings TC-1 - 12 are constant (start up to constant temperature column profile will take 3 - 5 hours).

X 4.6.27 Adjust valve Hv-122 so temperature from TC-12 is between 450 - 500°F. Temperature on TC-12 should never exceed 500°F.

X 4.6.28 Once column is at steady state take a set of samples per steps outlined is section 4.8.

X 4.6.29 Adjust valve Hv-131 so a flow rate of 1.0 to 2.0 is indicated on internal reflux flowmeter.

4.7 Distillation Column Operation

The operation of the distillation column will require close surveillance to insure proper control over process variables such as temperatures, column pressures, and flowrates. Adequate levels of oil must be maintained in the column bottom and sidedraw chimney tray to keep pumps from running dry.

X 4.7.1 Monitor daily high boiler accumulation, record in log book and increase H-1 temperature setpoint if mass of oil in high boiler accumulator is greater than 1500 cc. If oil is less than 500 cc over a 24 hour period decrease H-1 set point by 3 - 5 °F. If oil accumulation is 500 to 1500 cc per day no adjustment is necessary.

X 4.7.2 Decrease H-1 temperature setpoint by an appropriate amount if no bottoms level is in sight glass. Adjust H-1 temperature as necessary to maintain bottoms level.

X 4.7.3 Check low boiler accumulation weekly and record in log book.

X 4.7.4 Adjust Hv-131 to maintain an internal reflux flowrate of 1.0 to 2.0 as indicated on control panel.

X 4.7.5 Record daily in log book the following items:

1) Oil level in storage tank.
2) Δ P on oil in storage tank as indicated by the blue pointer on recorder.
3) Amount of total noncondensible oil vented from storage tank as indicated on gas meter.
4) Weight of oil in bottoms accumulator.
5) Readings on control panel.

a. Indicated levels and set points.

(1) Product return

(2) Internal reflux

(3) Tower feed

(4) Tower pressure

(5) Bottoms level

(6) Side draw level.

b. Heater set points, H-1, H-2, and H3.

c. Heater power.

d. Hi-Temp indicated temperature.

6) Pump discharge pressures on P-1 and P-2.

7) Δ P across tower column as indicated by red pointer on recorder.

8) Vacuum pump oil level.

9) Oil tank thermocouple inserts, TC 13, TC 14, TC15.

X 4.7.6 Maintain pump discharge pressures on P-1 of 15 to 20 psia and on P-2 of 25 to 35 psia during test period. Adjust valves, Hv-137 or Hv-138, or bypass valves PBV-1 or PBV-2 to maintain nominal discharge pressures.

X 4.7.7 Every 3rd or 4th day of operation, switch the accumulation of low boilers from one of the low boiler accumulators to the other. For example, assume that the low boilers liquid has been collecting in the accumulator, ACC-1. To switch accumulators:

1) Close Hv-110

2) Open Hv-109

X 4.7.8 If level in LOW BOILER REFLUX RESERVOIR sight glass indicates that the reservoir is completely full, crack valve Hv-132 slightly to allow passage of low boilers into LOW BOILER ACCUMULATORS. Close Hv-132 whenever the liquid level is below the refux line port into the column.

X 4.7.9 Readjust level control setpoints or DP cell suppression screws on bottom level control or sidedraw level control as required to insure proper control of levels. Actuation of control valves by controllers should occur when bottom and sidedraw levels are 80 to 90% of total level height.

X 4.7.10 Take weekly samples per steps outlined in OIL SAMPLING PROCEDURE, Section 4.8.

X 4.7.11 Empty LOW BOILER ACCUMULATORS weekly after oil samples have been taken. The emptying of these accumulators will upset column vacuum momentarily so care must be taken in the following steps to insure continuous column operation.

1) Turn SHUTDOWN BYPASS SWITCH to "BYPASS".
2) Close valves:
    a) Hv-107
    b) Hv-108
    c) Hv-109
    d) Hv-110
3) Open valves Hv-105 and Hv-106.
4) Open valves Hv-104 and Hv-103 and drain oil into receiving vessel.
5) Record volume of oil collected.
6) Close valves Hv-103, Hv-104, Hv-105, Hv-106.
7) Slowly open Hv-109 and Hv-108 as they will affect column pressure. Try to maintain column pressure below 40 mm Hg while opening these valves.

8) Open valve Hv-109.

9) Turn SHUTDOWN BYPASS SWITCH to "SAFE".

10) After 3 days of operations close Hv-109 and open Hv-110.

X 4.7.12 On a weekly basis check for oil in CONDENSATE ACCUMULATOR by closing valve Hv-120 and opening valve Hv-121. After condensate has been collected close valve Hv-121 then open valve Hv-120.

X 4.7.13 Maintain temperature of oil to pump inlet of P-1, TC/12 in the range of 450^{o} - 500^{o}F by adjusting valve, Hv-122.

X 4.7.14 Turn on temperature recorder and monitor temperatures indicated by thermocouples TC1 - TC13 when system is unattended.

X 4.8 Oil Sampling Procedure

X 4.8.1 Tank Sample

X 4.8.1.1 Carefully open HV-15 and drain dead volume of oil from sample line into a beaker.

X 4.8.1.2 Shut valve Hv-15.

X 4.8.1.3 Attach clean, evacuated and labelled sample bottle to coupling downstream from Hv-15.

X 4.8.1.4 Open Hv-15.

X 4.8.1.5 Open Hv-16, and vacuum load sample.

X 4.8.1.6 Close Hv-16.

X 4.8.1.7 Close Hv-15.

X 4.8.1.8 Allow sample bottle to cool to a low enough temperature to handle.

X 4.8.1.9 Disconnect sample bottle.

X 4.8.1.10 Transport sample bottle and dead volume oil to lab for disposition and analysis.

X 4.8.2 Processor Samples

X 4.8.2.1 Take 200 ml samples of oil at the following sample points:

1) Hv-104 or Hv-103.

2) Hv-100.

3) Hv-49.

X 4.8.3 Lab Procedure

X 4.8.3.1 At the lab, weigh the sampled oil and dead volume oil.

X 4.8.3.2 Transfer sampled oil to bottles for shipment, storage and laboratory analysis.

X 4.8.3.3 Before sealing the sample bottles, blanket any dead space with nitrogen.

X 4.8.3.4 Record sampling data in the sample log and transmit the weight of oil removed to test log.

4.9 Test Termination Procedure

4.9.1 Processor System Shutdown

X 4.9.1.1 Take last set of oil samples per steps outlined in Section 4.8.

X 4.9.1.2 Reduce TOWER FEED to 0.

X 4.9.1.3 Reduce H-1 heater control setpoint to 000. Allow column to operate under this condition for 5 minutes.

X 4.9.1.4 Reduce H-2 and H-3 heater control setpoints to 000.

X 4.9.1.5 Send all sidedraw liquid collected on chimney tray back to storage tank by reducing SIDEDRAW LEVEL setpoint to zero. When liquid level is below sight glass close control valve LCV-2 by increasing setpoint on SIDEDRAW LEVEL above indicated level.

X 4.9.1.6 Turn pump P-2 off by closing valve Hv-138 and reducing regulated pressure on air motor supply to zero with R-65.

X 4.9.1.7 Turn pump P-1 off by closing valve Hv-137 and reducing regulated pressure on air motor supply to zero with R-64.

X 4.9.1.8 Shut MAIN POWER to "OFF".

X 4.9.1.9 Close valve Hv-26 to vacuum pump and shut off vacuum pump.

X 4.9.1.10 After processor column has cooled to below 100°F drain column DP cell legs, DP cells, and all accumulators filled with oil. Record volume of oil drained.

X 4.9.1.11 Unload R-43 and R-48 to zero psig.

X 4.9.1.12 Close Hv-126 and Hv-127 water coolant valves.

X 4.9.1.13 Close Hv-60 and Hv-61.

X 4.9.2 Tank System Shutdown

X 4.9.2.1 Check position of all valves.

X 4.9.2.2 Take oil sample from Hv-15 as per regular procedure.

X 4.9.2.3 Take data reading.

X 4.9.2.4 Close Hv-14.

X 4.9.2.5 Close Hv-28.

X 4.9.2.6 Place sample bottle underneath Hv-19.

X 4.9.2.7 Open Hv-19 and drain condensate into bottle.

X 4.9.2.8 When condensate has drained, close Hv-19.

X 4.9.2.9 Take oil sample from Hv-15 as per regular procedure.

X 4.9.2.10 Weigh the effluent and record in data log.

X 4.9.2.11 Open Hv-08.

X 4.9.2.12 Set R-09 to 15 psig and allow GN_2 to backfill into tank.

X 4.9.2.13 When G-41 reads 15 psig, close R-09.

X 4.9.2.14 Close Hv-08.

X 4.9.2.15 Turn off all variacs and verify.

X V1 X V2 X V3 X V4 X V5

X 4.9.2.16 Monitor and record oil temperature and tank pressure as needed.

X 4.9.2.17 When oil temperature reaches 200°F take oil sample from Hv-15 as per regular procedure.

X 4.9.2.18 If tank pressure is less than 5 psig, open Hv-08 and adjust R-09 until G41 reads 5 psig.

X 4.9.2.19 Weigh a clean, empty 55 gallon drum and record weight in log.

X 4.9.2.20 Attach oil drain line to drum and Hv-03.

X 4.9.2.21 Open Hv-02.

X 4.9.2.22 Open Hv-03 and allow oil to drain into drum.

X 4.9.2.23 Ensure oil tank pressure is a constant 5 psig by adjusting R-09 as necessary.

X 4.9.2.24 After all oil has drained into drum close Hv-03 and Hv-02.

X 4.9.2.25 Weigh the drum and record weight in log.

X 4.9.2.26 After waiting 30 minutes, open Hv-03 and Hv-02 to allow any residual oil to drain into drum.

X 4.9.2.27 After all residual oil drained into drum close Hv-03 and Hv-02.

X 4.9.2.28 Weigh drum and record weight in log.

X 4.9.2.29 After 30 minutes, repeat above procedure and keep repeating until no significant mass gain in drum is noted.

X 4.9.2.30 Clean system as per cleanout procedure, Section 4.12.

4.10 Chemical and Data Analysis

NOTE

The initial oil, the aged oil and the samples obtained during the test will be analyzed. A mass balance between the input materials and output materials will provide a baseline comparison between published data and test results.

4.10.1 The initial and final oil samples will be subjected to the following chemical analysis

___4.10.1.1 Infrared spectra 4000-650 cm.$^{-1}$ This will provide data on oil chemical structural changes.

___4.10.1.2 NMR spectra. This will support conclusions derived from the infrared analysis.

___4.10.1.3 Molecular weight distribution to be performed by Sandia Livermore.

4.10.2 The initial weight of the oil should be equal to the sum of the final weight plus the samples taken plus the total weight collected in catch basins.

4.10.3 Data Log

NOTE

All data collected will be compiled in a final data book. This will include at least the following:

4.10.3.1 Initial weight of oil.

4.10.3.2 Weight of oil collected in catch basin.

4.10.3.3 Sample weight.

4.10.3.4 Temperature history.

4.10.3.5 Chemical analysis of original oil at ambient.

4.10.3.6 Chemical analysis of original oil at 600°F.

4.10.3.7 Chemical analysis of final oil.

4.10.3.8 Chemical analysis of noncondensable, low boiler, and fluid samples during test.

4.10.3.9 Chemical analysis of final vent cold trap sample,

4.10.3.10 Mass balance during test.

4.11 Phase II Emergency Procedures

NOTE

For this procedure an emergency will be defined as any situation occurring to or by the test system that if left unchecked could

1) cause potential or real harm to personnel or equipment or

2) compromise the accuracy of data collection.

4.11.1 General Emergency Procedure

4.11.1.1 Call Safety (ext. 4444) and notify the extent and type of problem.

4.11.1.2 Tell any unneeded personnel to leave the area.

4.11.1.3 Don any appropriate Safety Equipment.

4.11.1.4 If the problem is electrical, switch main breaker OFF.

4.11.1.5 If a subsystem is leaking, shutoff the nearest upstream source of the leak.

4.11.1.6 Note time and nature of emergency in log book.

4.11.2 Fire Caused by Oil Leak

___4.11.2.1 Shut off main breaker to cut all power to test equipment.

___4.11.2.2 Open Hv-11 Tank Pressure Vent and dump tank pressure.

___4.11.2.3 If insulation is burning, remove the lagging surrounding the area affected.

___4.11.2.4 Cut out the insulation affected by the leak.

___4.11.2.5 If required douse any flames with CO_2.

___4.11.2.6 Ascertain the location of the leak source for determination of the best fix.

4.11.3 Fire Caused by Electrical Short

___4.11.3.1 Shut off main breaker to cut all power to test equipment.

___4.11.3.2 Douse any fire with CO_2 fire extinguisher.

4.11.4 High Temperature Shutdown

___4.11.4.1 Check to make sure all power to heaters has been cut off.

___4.11.4.2 Note time of shutdown and enter in data log.

___4.11.4.3 Note temperatures TC1-TC15 in data log.

___4.11.4.4 Do not restart heaters until cause of the shutdown has been determined, and restart has the concurrence of Test Conducter and Safety.

4.12 System Cleaning

NOTE

Upon the completion of Phase II and after the oil is back in its shipping drum and stored, the tank processor and associated hardware and plumbing will be cleaned. The tank and processor will be washed with an additive free gasoline cut petroleum solvent (petroleum ether).

___4.12.1 Tank Subsystem

___4.12.1.1 Verify closed all hand valves.

___4.12.1.2 Remove ullage cold trap and low boiler tank and lines between Hv-14, Hv-19 and Rv-42, and clean separately.

___4.12.1.3 Verify petroleum solvent tank is on hand and filled with solvent.

___4.12.1.4 Attach solvent outlet to the inlet of Hv-25, Dist. Column Return Isolation.

___4.12.1.5 Disconnect line between R-09 and Hv-08.

___4.12.1.6 Run flexible from R-09 outlet to solvent tank pressure inlet.

___4.12.1.7 Load R-09 to 5 psig.

___4.12.1.8 Open Hv-04, lower sight glass isolation.

___4.12.1.9 Place solvent catch basin under Hv-24.

___4.12.1.10 Open Hv-25 and wait one minute to allow pressure to stabilize.

___4.12.1.11 Close Hv-25.

___4.12.1.12 Slowly open Hv-24 and allow approx. one gallon of solvent to flow into catch basin.

___4.12.1.13 Close Hv-24.

___4.12.1.14 Move catch basin under Hv-15.

___4.12.1.15 Open Hv-15 and allow approx. one gallon of solvent to glow into catch basin.

___4.12.1.16 Close Hv-15.

___4.12.1.17 Move catch basin under Hv-03.

___4.12.1.18 Open Hv-03 and allow approx. one gallon of solvent to flow into catch basin.

___4.12.1.19 Close Hv-03.

___4.12.1.20 Open Hv-11 Tank Vent and zero the tank pressure.

___4.12.1.21 Close Hv-11.

___4.12.1.22 Repeat steps 4.11.9-4.11.21 a total of 4 times, then continue with step 4.11.23.

___4.12.1.23 Open Hv-03 and allow any remaining solvent to flow into catch basin.

___4.12.1.24 Close Hv-03.

___4.12.1.25 Replumb Cold Trap, Low Boiler Tank and Hardware into system.

___4.12.1.26 Unload R-09 to zero psig.

___4.12.1.27 Disconnect solvent tank from Hv-25.

___4.12.1.28 Replumb R-09 to Hv-08.

___4.12.1.29 Open Hv-04 drain plug.

___4.12.1.30 Load R-09 to 100 psig.

___4.12.1.31 Open Hv-15.

___4.12.1.32 Open Hv-14.

___4.12.1.33 Open Hv-25.

___4.12.1.34 Open Hv-03.

___4.12.1.35 Open Hv-25.

___4.12.1.36 Open Hv-24.

___4.12.1.37 Open Hv-28.

___4.12.1.38 Open Hv-19

___4.12.1.39 Open Hv-08 Tank Pressure Reg. Isolation.

___4.12.1.40 Purge system with GN_2 for 8 hours.

___4.12.1.41 Close Hv-14.

___4.12.1.42 Close Hv-19.

___4.12.1.43 Close Hv-28.

___4.12.1.44 Close Hv-24.

___4.12.1.45 Close Hv-25.

___4.12.1.46 Close Hv-03.

___4.12.1.47 Close Hv-15.

___4.12.1.48 Close Hv-08.

___4.12.1.49 Open Hv-11 and vent pressure.

___4.12.1.50 Close Hv-11.

___4.12.1.51 Replace Hv-04 drain plug.

___4.12.1.52 Unload R-09 to zero psig.

___4.12.1.53 Place a new charcoal filter in the filter housing F-32.

4.12.2 Processor Subsystem

___4.12.2.1 Drain column, reservoirs, accumulators, DP cells and feed and return lines between processor and tank subsystems.

APPENDIX C

COMPUTER PROGRAM LISTINGS

APPENDIX C: COMPUTER PROGRAM LISTINGS

The computer models, DISTILL and TMASS, developed under this program are listed in this report section. Also included are sample inputs and outputs of each program. The DISTILL model is a distillation process performance evaluation program based on an existing documented computer program formulation. The development and features of the model are described in more detail in the main report in Section IV.B.3.b. The TMASS model was developed to solve Eq IV-10 in main report Section IV.B.3.a for degradation products and tank mass as functions of time. Both models were developed under Fortran IV for use on CDC computers. Included in this report sections are:

DISTILL

PROGRAM

LISTING

```
      PROGRAM MAIN(INPUT,OUTPUT=64,TAPE5=INPUT,TAPE6=OUTPUT)
C
C    DISTILLATION PERFORMANCE COMPUTER PROGRAM
C
C        BASIC PROGRAM FROM HANSON AND SOMERFIELD--1962
C
C        PROGRAM UPDATED AND MODIFIED BY:
C           DAVID G. BESHORE
C           MARTIN MARIETTA AEROSPACE
C           ENGINEERING MECHANICS DEPARTMENT
C           DENVER, COLORADO 80201
C
      DIMENSION GENX(20,40,5),GENY(20,40,5),EXTFD(20,40,5).NSTGS(5),
     1A(20,5),B(20,5),C(20,5),ENTHK(20),ENTHL(20),ENTHU(20),ENTHW(20),
     2TOTFDH(40,5),VAPPRO(40,5),QUIPRO(40,5),VAPFLO(40,5),QUIFLO(40,5),
     3JFVFRO(40,5),KFVFRO(40,5),JFVTO(40,5),KFVTO(40,5),JFLFRO(40,5),
     4KFLFRO(40,5),JFLTO(40,5),KFLTO(40,5),JVVFRO(40,5),KVVFRO(40,5),
     5JVVTO(40,5),KVVTO(40,5),JVLFRO(40.5),KVLFRO(40,5),JVLTO(40,5),
     6KVLTO(40,5),JRETYP(5),JCOTYP(5),QUITOP(5),TEMP(40,5),VAPOR(40,5),
     7QUID(40,5),TCSET(5),SUMFD(20),FLFEED(20),VAPY(20).QUIDX(20),
     8SUMX(40,5),SUMY(40,5),QUIMOH(40,5),VAPMOH(40,5),QUNBAL(40,5),
     9FLVAP(20),FLLIQ(20),FLVAP1(20),FLLIQ1(20),PRDSUM(20),RFSUM(20)
      DIMENSION CONDLD(5),REBLD(5)
      COMMON GENX,GENY.EXTFD,NSTGS,A,B,C.ENTHK.ENTHL,ENTHU,ENTHW,TOTFDH,
     1VAPPRO.QUIPRO,VAPFLO,QUIFLO,JFVFRO,KFVFRO,JFVTO.KFVTO,JFLFRO,
     2KFLFRO,JFLTO,KFLTO,JVVFRO,KVVFRO,JVVTO,KVVTO,JVLFRO,KVLFRO.JVLTO,
     3KVLTO,JRETYP,JCOTYP,QUITOP,TEMP,VAPOR.QUID.TCSET,SUMFD,FLFEED,
     4VAPY.QUIDX.SUMX.SUMY,QUIMOH,VAPMOH,QUNBAL,FLVAP,FLLIQ,FLVAP1,
     5FLLIQ1,PRDSUM,RFSUM,CONDLD,REBLD,PRT
      EQUILKF(A,B,C,T)=EXP(A/(T+460.0)+B+C*(T+460.0))
      THALFF(Y,Z.T)=Y*T+Z
    1 CALL INPUT (NCOMPS,NCOLS,PRDERR,BDFERR)
      ITERAT=0
      FACTOR=10.0
      TOTFD=0.0
      DO 2 I=1,NCOMPS
      SUMFD(I)=0.0
      DO 2 K=1,NCOLS
      JT=NSTGS(K)
      DO 2 J=1,JT
      SUMFD(I)=SUMFD(I)+EXTFD(I,J,K)
    2 TOTFD=TOTFD+EXTFD(I,J,K)
    3 HCALIK=0.0
      DO 145 K=1,NCOLS
      JT=NSTGS(K)
      J=1
      JDELTA=1
      KTIMES=2*JT-1
      DO 125 KCOUNT=1,KTIMES

    DETERMINATION OF COMPONENT FEEDS AND ENTHALPIES TO THE STAGE

      FLFEES=0.0
      IF (J-1) 4,4,6
    4 DO 5 I=1,NCOMPS
      FLFEED(I)=GENX(I,2,K)*QUID(2,K)+EXTFD(I,1,K)
    5 FLFEES=FLFEES+FLFEED(I)
      QIN=TOTFDH(1,K)+QUID(2,K)*QUIMOH(2,K)
      GO TO 11
    6 IF (J-JT) 9,7,9
    7 DO 8 I=1,NCOMPS
      FLFEED(I)=GENY(I.JT-1,K)*VAPOR(JT-1,K)+EXTFD(I,JT,K)
    8 FLFEES=FLFEES+FLFEED(I)
```

```
      QIN=TOTFDH(JT,K)+VAPOR(JT-1,K)*VAPMOH(JT-1,K)
      GO TO 11
    9 DO 10 I=1,NCOMPS
      FLFEED(I)=GENX(I,J+1,K)*QUID(J+1,K)+GENY(I,J-1,K)*VAPOR(J-1,K)+
     1EXTFD(I,J,K)
   10 FLFEES=FLFEES+FLFEED(I)
      QIN=TOTFDH(J,K)+VAPOR(J-1,K)*VAPMOH(J-1,K)+QUID(J+1,K)*
     1QUIMOH(J+1,K)
   11 IF (KFVFRO(J,K)) 14,14,12
   12 JV=JFVFRO(J,K)
      KV=KFVFRO(J,K)
      DO 13 I=1,NCOMPS
      FLFEED(I)=FLFEED(I)+VAPFLO(JV,KV)*GENY(I,JV,KV)
   13 FLFEES=FLFEES+VAPFLO(JV,KV)*GENY(I,JV,KV)
      QIN=QIN+VAPFLO(JV,KV)*VAPMOH(JV,KV)
   14 IF (KVVFRO(J,K)) 17,17,15
   15 JV=JVVFRO(J,K)
      KV=KVVFRO(J,K)
      DO 16 I=1,NCOMPS
      FLFEED(I)=FLFEED(I)+GENY(I,JV,KV)*VAPOR(JV,KV)
   16 FLFEES=FLFEES+VAPOR(JV,KV)*GENY(I,JV,KV)
      QIN=QIN+VAPOR(JV,KV)*VAPMOH(JV,KV)
   17 IF (KFLFRO(J,K)) 20,20,18
   18 JV=JFLFRO(J,K)
      KV=KFLFRO(J,K)
      DO 19 I=1,NCOMPS
      FLFEED(I)=FLFEED(I)+QUIFLO(JV,KV)*GENX(I,JV,KV)
   19 FLFEES=FLFEES+QUIFLO(JV,KV)*GENX(I,JV,KV)
      QIN=QIN+QUIFLO(JV,KV)*QUIMOH(JV,KV)
   20 IF (KLLFRO(J,K)) 26,26.21
   21 JV=JLLFRO(J,K)
      KV=KLLFRO(J,K)
      IF (JV-1) 22,22.24
   22 DO 23 I=1,NCOMPS
      FLFEED(I)=FLFEED(I)+GENX(I,JV,KV)*QUID(JV,KV)
   23 FLFEES=FLFEES+QUID(JV,KV)*GENX(I,JV,KV)
      QIN=QIN+QUID(JV,KV)*QUIMOH(JV,KV)
      GO TO 26
   24 DO 25 I=1,NCOMPS
      FLFEED(I)=FLFEED(I)+GENX(I,JV,KV)*QUITOP(KV)
   25 FLFEES=FLFEES+QUITOP(KV)*GENX(I,JV,KV)
      QIN=QIN+QUITOP(KV)*QUIMOH(JV,KV)
C
C   CALCULATION OF REBOILER
C
   26 IF (J-1) 27.27.39
   27 IF (JRETYP(K)) 94.94,28
   28 SUMX(1,K)=0.0
      SUMY(1,K)=0.0
      QUIMOH(1,K)=0.0
      VAPMOH(1,K)=0.0
      FIXSUM=VAPOR(1,K)+VAPPRO(1,K)+VAPFLO(1,K)+QUIPRO(1,K)+QUIFLO(1,K)
      IF(FLFEES-FIXSUM) 29,31,35
   29 DO 30 I=1,NCOMPS
   30 FLFEED(I)=FLFEED(I)+GENX(I,2,K)*(FIXSUM-FLFEES)
      FLFEES=FIXSUM
   31 QUID(1,K)=0.0
      IF (QUIPRO(1,K)+QUIFLO(1,K)) 32,32.36
   32 DO 33 I=1,NCOMPS
   33 VAPY(I)=FLFEED(I)/FLFEES
      CALL DEWPT (VAPY,QUIDX.TEMP(1,K),A,B,C,NCOMPS,K,BDFERR)
      GO TO 37
   35 QUID(1,K)=FLFEES-FIXSUM
```

```
   36 FLVAS=VAPOR(1,K)+VAPPRO(1,K)+VAPFLO(1,K)
      CALL ISOVFL (FLFEED,FLFEES,FLVAS,TEMP(1,K),A,B,C,NCOMPS,K,BDFERR,
     1VAPY,QUIDX)
   37 DO 38 I=1,NCOMPS
      GENX(I,1,K)=QUIDX(I)
      GENY(I,1,K)=VAPY(I)
      SUMX(1,K)=SUMX(1,K)+GENX(I,1,K)
      SUMY(1,K)=SUMY(1,K)+GENY(I,1,K)
      QUIMOH(1,K)=QUIMOH(1,K)+GENX(I,1,K)*THALPF(ENTHK(I),ENTHL(I),
     1TEMP(1,K))
   38 VAPMOH(1,K)=VAPMOH(1,K)+GENY(I,1,K)*THALPF(ENTHU(I),ENTHW(I),
     1TEMP(1,K))
      GO TO 122
C
C     CALCULATION OF CONDENSER
C
   39 IF (J-JT) 94,40,40
   40 IF (JCOTYP(K)-1) 94,41,41
   41 SUMX(JT,K)=0.0
      SUMY(JT,K)=0.0
      QUIMOH(JT,K)=0.0
      VAPMOH(JT,K)=0.0
      FIXSUM=QUID(JT,K)+QUIPRO(JT,K)+QUIFLO(JT,K)+VAPPRO(JT,K)+
     1VAPFLO(JT,K)
      IF (FLFEES-FIXSUM) 42,44,45
   42 DO 43 I=1,NCOMPS
   43 FLFEED(I)=FLFEED(I)+GENY(I,JT-1,K)*(FIXSUM-FLFEES)
   44 FLFEES=FIXSUM
   45 IF (JCOTYP(K)-2) 46,54,63
C
C     CALCULATION OF PARTIAL CONDENSER
C
   46 QUITOP(K)=0.0
      IF (FLFEES-FIXSUM) 47,47,50
   47 VAPOR(JT,K)=0.0
      IF (VAPPRO(JT,K)+VAPFLO(JT,K)) 48,48,51
   48 DO 49 I=1,NCOMPS
   49 QUIDX(I)=FLFEED(I)/FLFEES
      CALL BUBPT (QUIDX,VAPY,TEMP(JT,K),A,B,C,NCOMPS,K,BDFERR)
      GO TO 52
   50 VAPOR(JT,K)=FLFEES-FIXSUM
   51 FLVAS=VAPOR(JT,K)+VAPPRO(JT,K)+VAPFLO(JT,K)
      CALL ISOVFL (FLFEED,FLFEES,FLVAS,TEMP(JT,K),A,B,C,NCOMPS,K,BDFERR,
     1VAPY,QUIDX)
   52 DO 53 I=1,NCOMPS
      GENX(I,JT,K)=QUIDX(I)
      GENY(I,JT,K)=VAPY(I)
      SUMX(JT,K)=SUMX(JT,K)+GENX(I,JT,K)
      SUMY(JT,K)=SUMY(JT,K)+GENY(I,JT,K)
      QUIMOH(JT,K)=QUIMOH(JT,K)+GENX(I,JT,K)*THALPF(ENTHK(I),ENTHL(I),
     1TEMP(JT,K))
   53 VAPMOH(JT,K)=VAPMOH(JT,K)+GENY(I,JT,K)*THALPF(ENTHU(I),ENTHW(I),
     1TEMP(JT,K))
      GO TO 122
C
C     CALCULATION OF TOTAL CONDENSER
C
   54 VAPOR(JT,K)=0.0
      IF (FLFEES-FIXSUM) 55,55,56
   55 QUITOP(K)=0.0
      GO TO 57
   56 QUITOP(K)=FLFEES-FIXSUM
   57 IF (VAPPRO(JT,K)+VAPFLO(JT,K)) 58,58,60
```

```
58 DO 59 I=1,NCOMPS
59 QUIDX(I)=FLFEED(I)/FLFEES
   CALL BUBPT (QUIDX,VAPY,TEMP(JT,K),A,B,C,NCOMPS,K,BDFERR)
   GO TO 61
60 FLVAS=VAPPRO(JT,K)+VAPFLO(JT,K)
   CALL ISOVFL (FLFEED,FLFEES,FLVAS,TEMP(JT,K),A,B,C,NCOMPS,K,BDFERR,
  1VAPY,QUIDX)
61 DO 62 I=1,NCOMPS
   GENX(I,JT,K)=QUIDX(I)
   GENY(I,JT,K)=VAPY(I)
   SUMX(JT,K)=SUMX(JT,K)+GENX(I,JT,K)
   SUMY(JT,K)=SUMY(JT,K)+GENY(I,JT,K)
   QUIMOH(JT,K)=QUIMOH(JT,K)+GENX(I,JT,K)*THALPF(ENTHK(I),ENTHL(I),
  1TEMP(JT,K))
62 VAPMOH(JT,K)=VAPMOH(JT,K)+GENY(I,JT,K)*THALPF(ENTHU(I),ENTHW(I),
  1TEMP(JT,K))
   GO TO 122
C
C  CALCULATION OF TWO PRODUCT CONDENSER
C
63 IF (FLFEES-FIXSUM) 64,64,74
64 QUITOP(K)=0.0
   VAPOR(JT,K)=0.0
   IF (VAPPRO(JT,K)+VAPFLO(JT,K)) 65,65,72
65 DO 66 I=1,NCOMPS
66 QUIDX(I)=FLFEED(I)/FLFEES
   CALL BUBPT (QUIDX,VAPY,TEMP(JT,K),A,B,C,NCOMPS,K,BDFERR)
   DO 67 I=1,NCOMPS
   GENX(I,JT,K)=QUIDX(I)
67 SUMX(JT,K)=SUMX(JT,K)+GENX(I,JT,K)
   IF (TEMP(JT,K)-TCSET(K)) 68,68,70
68 DO 69 I=1,NCOMPS
   GENY(I,JT,K)=VAPY(I)
69 SUMY(JT,K)=SUMY(JT,K)+GENY(I,JT,K)
   GO TO 91
70 DO 71 I=1,NCOMPS
71 GENY(I,JT,K)=0.0
   SUMY(JT,K)=1.0
   TEMP(JT,K)=TCSET(K)
   GO TO 91
72 FLVAS=VAPPRO(JT,K)+VAPFLO(JT,K)
   CALL ISOVFL (FLFEED,FLFEES,FLVAS,TEMP(JT,K),A,B,C,NCOMPS,K,BDFERR,
  1VAPY,QUIDX)
   DO 73 I=1,NCOMPS
   GENX(I,JT,K)=QUIDX(I)
   GENY(I,JT,K)=VAPY(I)
   SUMX(JT,K)=SUMX(JT,K)+GENX(I,JT,K)
73 SUMY(JT,K)=SUMY(JT,K)+GENY(I,JT,K)
   GO TO 91
74 IF (VAPPRO(JT,K)+VAPFLO(JT,K)) 75,75,81
75 DO 76 I=1,NCOMPS
76 QUIDX(I)=FLFEED(I)/FLFEES
   CALL BUBPT (QUIDX,VAPY,TEMP(JT,K),A,B,C,NCOMPS,K,BDFERR)
   IF (TEMP(JT,K)-TCSET(K)) 81,77,79
77 DO 78 I=1,NCOMPS
   GENX(I,JT,K)=QUIDX(I)
   GENY(I,JT,K)=VAPY(I)
   SUMX(JT,K)=SUMX(JT,K)+GENX(I,JT,K)
78 SUMY(JT,K)=SUMY(JT,K)+GENY(I,JT,K)
   QUITOP(K)=FLFEES-FIXSUM
   VAPOR(JT,K)=0.0
   GO TO 91
79 DO 80 I=1,NCOMPS
```

```
      GENX(I,JT,K)=QUIDX(I)
      GENY(I,JT,K)=0.0
   80 SUMX(JT,K)=SUMX(JT,K)+GENX(I,JT,K)
      SUMY(JT,K)=1.0
      QUITOP(K)=FLFEES-FIXSUM
      VAPOR(JT,K)=0.0
      TEMP(JT,K)=TCSET(K)
      GO TO 91
   81 FLLIS=QUID(JT,K)+QUIPRO(JT,K)+QUIFLO(JT,K)+QUITOP(K)
      FLVAS=FLFEES-FLLIS
      IF (FLVAS) 82,82,83
   82 FLLIS=QUID(JT,K)+QUIPRO(JT,K)+QUIFLO(JT,K)
      FLVAS=FLFEES-FLLIS
   83 TEMP(JT,K)=TCSET(K)
      FIXV=VAPPRO(JT,K)+VAPFLO(JT,K)
      FIXL=QUID(JT,K)+QUIPRO(JT,K)+QUIFLO(JT,K)
      CALL ISOTFL (FLFEED,FLFEES,FLVAS,FLLIS,FIXV,FIXL,TEMP(JT,K),A,B,C,
     1NCOMPS,K,BDFERR,VAPY,QUIDX)
      IF (FIXL-FLLIS) 84,84,87
   84 IF (FIXV-FLVAS) 85,85,89
   85 DO 86 I=1,NCOMPS
      GENX(I,JT,K)=QUIDX(I)
      GENY(I,JT,K)=VAPY(I)
      SUMX(JT,K)=SUMX(JT,K)+GENX(I,JT,K)
   86 SUMY(JT,K)=SUMY(JT,K)+GENY(I,JT,K)
      QUITOP(K)=FLLIS-FIXL
      VAPOR(JT,K)=FLVAS-FIXV
      GO TO 91
   87 FLVAS=FLFEES-FIXL
      CALL ISOVFL (FLFEED,FLFEES,FLVAS,TEMP(JT,K),A,B,C,NCOMPS,K,BDFERR,
     1VAPY,QUIDX)
      DO 88 I=1,NCOMPS
      GENX(I,JT,K)=QUIDX(I)
      GENY(I,JT,K)=VAPY(I)
      SUMX(JT,K)=SUMX(JT,K)+GENX(I,JT,K)
   88 SUMY(JT,K)=SUMY(JT,K)+GENY(I,JT,K)
      QUITOP(K)=0.0
      VAPOR(JT,K)=FLFEES-FIXSUM
      GO TO 91
   89 FLVAS=FIXV
      CALL ISOVFL (FLFEED,FLFEES,FLVAS,TEMP(JT,K),A,B,C,NCOMPS,K,BDFERR,
     1VAPY,QUIDX)
      DO 90 I=1,NCOMPS
      GENX(I,JT,K)=QUIDX(I)
      GENY(I,JT,K)=VAPY(I)
      SUMX(JT,K)=SUMX(JT,K)+GENX(I,JT,K)
   90 SUMY(JT,K)=SUMY(JT,K)+GENY(I,JT,K)
      VAPOR(JT,K)=0.0
      QUITOP(K)=FLFEES-FIXSUM
   91 DO 92 I=1,NCOMPS
      QUIMOH(JT,K)=QUIMOH(JT,K)+GENX(I,JT,K)*THALPF(ENTHK(I),ENTHL(I),
     1TEMP(JT,K))
   92 VAPMOH(JT,K)=VAPMOH(JT,K)+GENY(I,JT,K)*THALPF(ENTHU(I),ENTHW(I),
     1TEMP(JT,K))
   93 GO TO 122
C
C     CALCULATION OF COMPONENT FLOWS FROM TYPICAL STAGE
C
   94 FIXSUM=VAPPRO(J,K)+VAPFLO(J,K)+QUIPRO(J,K)+QUIFLO(J,K)
      IF (FLFEES-FIXSUM) 95,95,96
   95 VAPOR(J,K)=FLFEES*1.0E-4
      QUID(J,K)=FLFEES*1.0E-4
      GO TO 122
```

```
   96 IF(VAPOR(J,K).EQ.0.0) VAPOR(J,K)=FLFEES*1.0E-4
      IF(QUID(J,K).EQ.0.0) QUID(J,K) = FLFEES*1.0E-4
      FLVASO=VAPOR(J,K)+VAPPRO(J,K)+VAPFLO(J,K)
      FLLISO=QUID(J,K)+QUIPRO(J,K)+QUIFLO(J,K)
      IF (FLLISO-FLVASO) 97,98,98
   97 IF(PRT.EQ.7.0) WRITE(6.1000) FLVASO,FLLISO,FLFEES,FLV,FLL
 1000 FORMAT("FLVASO,FLLISO,FLFEES,FLV,FLL=",5E15.8)
      FLL=FLFEES/(FLVASO/FLLISO+1.0)
      FLV=FLFEES-FLL
      GO TO 99
   98 FLV=FLFEES/(FLLISO/FLVASO+1.0)
      FLL=FLFEES-FLV
   99 FLVASC=0.0
      FLLISC=0.0
      QUIH=0.0
      VAPH=0.0
      IF(PRT.EQ.7.0) WRITE(6,1000) FLVASO,FLLISO;FLFEES,FLV,FLL
      DO 100 I=1,NCOMPS
      EQCST=EQUILKF(A(I,K),B(I,K),C(I,K),TEMP(J,K))
      FLVAP(I)=FLFEED(I)/(FLL/FLV/EQCST+1.0)
      FLLIQ(I)=FLFEED(I)/(FLV*EQCST/FLL+1.0)
      FLVASC=FLVASC+FLVAP(I)
      FLLISC=FLLISC+FLLIQ(I)
      QUIH=QUIH+FLLIQ(I)*THALPF(ENTHK(I),ENTHL(I),TEMP(J,K))
  100 VAPH=VAPH+FLVAP(I)*THALPF(ENTHU(I),ENTHW(I),TEMP(J,K))
      SUMX(J,K)=FLLISC/FLL
      SUMY(J,K)=FLVASC/FLV
      T=TEMP(J,K)+1.0
      QUIH1=0.0
      VAPH1=0.0
      DO 101 I=1,NCOMPS
      EQCST=EQUILKF(A(I,K),B(I,K).C(I,K),T)
      FLVAP1(I)=FLFEED(I)/(FLL/FLV/EQCST+1.0)
      FLLIQ1(I)=FLFEED(I)/(FLV*EQCST/FLL+1.0)
      VAPH1=VAPH1+FLVAP1(I)*THALPF(ENTHU(I),ENTHW(I),T)
  101 QUIH1=QUIH1+FLLIQ1(I)*THALPF(ENTHK(I),ENTHL(I),T)
      HCAP=VAPH1+QUIH1-VAPH-QUIH
      DELT=(QIN-QUIH-VAPH)/HCAP
      TEMP(J,K)=TEMP(J.K)+DELT
      FLVASP=0.0
      FLLISP=0.0
      DO 107 I=1.NCOMPS
      DELV=DELT*(FLVAP1(I)-FLVAP(I))
      IF (DELV) 102.106.104
  102 IF (ABS(DELV)-FLVAP(I)) 106,103,103
  103 DELV=-FLVAP(I)
      GO TO 106
  104 IF (DELV-FLLIQ(I)) 106,106,105
  105 DELV=FLLIQ(I)
  106 FLVAP(I)=FLVAP(I)+DELV
      FLLIQ(I)=FLLIQ(I)-DELV
      FLVASP=FLVASP+FLVAP(I)
  107 FLLISP=FLLISP+FLLIQ(I)
      VAPMOH(J,K)=0.0
      QUIMOH(J,K)=0.0
      DO 108 I=1,NCOMPS
      IF(PRT.EQ.7.0) WRITE(6,1010) I,J,K,FLVASP,FLLISP
 1010 FORMAT("I,J,K,FLVASP,FLLISP=",3I5,2E15.8)
      GENY(I,J,K)=FLVAP(I)/(FLVASP+1.0E-20)
      GENX(I,J,K)=FLLIQ(I)/(FLLISP+1.0E-20)
      VAPMOH(J,K)=VAPMOH(J,K)+GENY(I,J,K)*THALPF(ENTHU(I),ENTHW(I),
     1TEMP(J,K))
  108 QUIMOH(J,K)=QUIMOH(J,K)+GENX(I,J,K)*THALPF(ENTHK(I).ENTHL(I),
```

```
     1TEMP(J,K))
      VAPORN=FLVASP-VAPPRO(J,K)-VAPFLO(J,K)
      QUIDN=FLLISP-QUIPRO(J,K)-QUIFLO(J,K)
      IF (FLVASP/FLV-1.0) 109,109,111
  109 IF (VAPORN/FLFEES-1.0E-4) 110,110,111
  110 HBALOK=1.0
      VAPOR(J,K)=FLFEES*1.0E-4
      QUID(J,K)=QUIDN
      GO TO 122
  111 IF (FLLISP/FLL-1.0) 112,112,114
  112 IF (QUIDN/FLFEES-1.0E-4) 113,113,114
  113 HBALOK=1.0
      QUID(J,K)=FLFEES*1.0E-4
      VAPOR(J,K)=VAPORN
      GO TO 122
  114 IF (ABS(FLV-FLVASP)/FLV-FACTOR*PRDERR/TOTFD) 115,115,116
  115 IF (ABS(FLL-FLLISP)/FLL-FACTOR*PRDERR/TOTFD) 117,117,116
  116 FLV=FLVASP
      FLL=FLLISP
      GO TO 99
  117 IF (ABS(FLVASP-FLVASO)/FLFEES-PRDERR/TOTFD) 119,119,118
  118 HBALOK=1.0
      GO TO 121
  119 IF (ABS(FLLISP-FLLISO)/FLFEES-PRDERR/TOTFD) 121,121,120
  120 HBALOK=1.0
  121 QUID(J,K)=QUIDN
      VAPOR(J,K)=VAPORN
  122 IF (J-JT) 124,123,123
  123 JDELTA=-1
  124 J=J+JDELTA
  125 CONTINUE
C
C     CALCULATION OF HEAT UNBALANCES
C
  126 DO 141 J=1,JT
      IF (J-1) 127,127,128
  127 QUNBAL(1,K)=TOTFDH(1,K)+QUID(2,K)*QUIMOH(2,K)
      GO TO 131
  128 IF (J-JT) 130,129,129
  129 QUNBAL(JT,K)=TOTFDH(JT,K)+VAPOR(JT-1,K)*VAPMOH(JT-1,K)
      GO TO 131
  130 QUNBAL(J,K)=TOTFDH(J,K)+VAPOR(J-1,K)*VAPMOH(J-1,K)+QUID(J+1,K)*
     1QUIMOH(J+1,K)
  131 QUNBAL(J,K)=QUNBAL(J,K)-(VAPOR(J,K)+VAPPRO(J,K)+VAPFLO(J,K))*
     1VAPMOH(J,K)-(QUID(J,K)+QUIPRO(J,K)+QUIFLO(J,K))*QUIMOH(J,K)
      IF (KFVFRO(J,K)) 133,133,132
  132 JV=JFVFRO(J,K)
      KV=KFVFRO(J,K)
      QUNBAL(J,K)=QUNBAL(J,K)+VAPFLO(JV,KV)*VAPMOH(JV,KV)
  133 IF (KVVFRO(J,K)) 135,135,134
  134 JV=JVVFRO(J,K)
      KV=KVVFRO(J,K)
      QUNBAL(J,K)=QUNBAL(J,K)+VAPOR(JV,KV)*VAPMOH(JV,KV)
  135 IF (KFLFRO(J,K)) 137,137,136
  136 JV=JFLFRO(J,K)
      KV=KFLFRO(J,K)
      QUNBAL(J,K)=QUNBAL(J,K)+QUIFLO(JV,KV)*QUIMOH(JV,KV)
  137 IF (KVLFRO(J,K)) 141,141,138
  138 JV=JVLFRO(J,K)
      KV=KVLFRO(J,K)
      IF (JV-1) 139,139,140
  139 QUNBAL(J,K)=QUNBAL(J,K)+QUID(JV,KV)*QUIMOH(JV,KV)
      GO TO 141
```

```
  140 QUNBAL(J,K)=QUNBAL(J,K)+QUITOP(KV)*QUIMOH(JV,KV)
  141 CONTINUE
      IF (JRETYP(K)) 143,143,142
  142 REBLD(K)=-QUNBAL(1,K)
      QUNBAL(1,K)=0.0
  143 IF (ICOTYP(K)) 145,145,144
  144 CONDLD(K)=QUNBAL(JT,K)-QUITOP(K)*QUIMOH(JT,K)
      QUNBAL(JT,K)=0.0
  145 CONTINUE
  146 CONTINUE
      FACTOR=0.50
C
C  CALCULATION OF COMPONENT RECOVERY FRACTIONS
C
      DO 147 I=1,NCOMPS
  147 PRDSUM(I)=0.0
      DO 163 K=1,NCOLS
      JT=NSTGS(K)
      DO 153 J=1,JT
      IF (VAPPRO(J,K)) 150,150,148
  148 DO 149 I=1,NCOMPS
  149 PRDSUM(I)=PRDSUM(I)+VAPPRO(J,K)*GENY(I,J,K)
  150 IF (QUIPRO(J,K)) 153,153,151
  151 DO 152 I=1,NCOMPS
  152 PRDSUM(I)=PRDSUM(I)+QUIPRO(J,K)*GENX(I,J,K)
  153 CONTINUE
      IF (KVLTD(1,K)) 154,154,156
  154 DO 155 I=1,NCOMPS
  155 PRDSUM(I)=PRDSUM(I)+QUID(1,K)*GENX(I,1,K)
  156 IF (KVVTD(JT,K)) 157,157,159
  157 DO 158 I=1,NCOMPS
  158 PRDSUM(I)=PRDSUM(I)+VAPOR(JT,K)*GENY(I,JT,K)
  159 IF (ICOTYP(K)-2) 163,160,160
  160 IF (KVLTD(JT,K)) 161,161,163
  161 DO 162 I=1,NCOMPS
  162 PRDSUM(I)=PRDSUM(I)+QUITOP(K)*GENX(I,JT,K)
  163 CONTINUE
      DO 164 I=1,NCOMPS
      RFSUM(I)=PRDSUM(I)/SUMFD(I)
  164 PRDSUM(I)=PRDSUM(I)-SUMFD(I)
C
C  OUTPUT
C
      ITERAT=ITERAT+1
      PRSERR=0.0
      DO 165 I=1,NCOMPS
  165 PRSERR=PRSERR+ABS(PRDSUM(I))
      IF (PRSERR-PRDERR) 166,166,169
  166 IF (HEALOK) 167,167,169
  167 CHECK=1.0
  168 CALL OUTPUT (ITERAT,NCOMPS,NCOLS,CHECK)
      GO TO 1
  169 IF (PRT.EQ.5.0) 170,171
  170 CHECK=1.0
      CALL OUTPUT (ITERAT,NCOMPS,NCOLS,CHECK)
      GO TO 3
  171 IF (PRT.EQ.6.0) 172,3
  172 CHECK=0.0
      CALL OUTPUT (ITERAT,NCOMPS,NCOLS,CHECK)
      GO TO 3
      END
C     PROGRAM 9 PROGRAM FOR INTERLINKED COLUMNS
C
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```
C  SUBROUTINE FOR INPUT
C
      SUBROUTINE INPUT (NCOMPS,NCOLS,PRDERR,BDFERR)
      DIMENSION GENX(20,40,5),GENY(20,40.5),EXTFD(20,40,5),NSTGS(5),
     1A(20,5),B(20.5),C(20,5),ENTHK(20),ENTHL(20),ENTHU(20),ENTHW(20),
     2TOTFDH(40,5),VAPPRO(40.5),QUIPRO(40.5),VAPFLO(40,5),QUIFLO(40.5).
     3JFVFRO(40,5),KFVFRO(40,5),JFVTO(40.5),KFVTO(40,5),JFLFRO(40.5).
     4KFLFRO(40,5).JFLTO(40,5),KFLTU(40,5).JVVFRO(40,5),KVVFRO(40,5).
     5JVVTO(40,5),KVVTO(40,5).JVLFRO(40,5),KVLFRO(40,5),JVLTO(40,5),
     6KVLTO(40,5),JRETYP(5),JCOTYP(5),QUITOP(5),TEMP(40,5),VAPOR(40,5),
     7QUID(40,5),TCSET(5),SUMFD(20).FLFEED(20),VAPY(20),QUIDX(20),
     8SUMX(40,5),SUMY(40,5),QUIMOH(40,5).VAPMOH(40,5),QUNBAL(40,5),
     9FLVAP(20),FLLIQ(20),FLVAP1(20),FLLIQ1(20),PROSUM(20),RFSUM(20)
      DIMENSION CONDLD(5),REBLD(5)
      DIMENSION JFROM(4,5), KFROM(4,5), JTO(4,5), KTO(4,5)
      COMMON GENX,GENY,EXTFD,NSTGS,A,B,C.ENTHK,ENTHL,ENTHU,ENTHW,TOTFDH,
     1VAPPRO,QUIPRO.VAPFLO,QUIFLO,JFVFRO.KFVFRO,JFVTO,KFVTO,JFLFRO,
     2KFLFRO,JFLTO,KFLTO,JVVFRO,KVVFRO,JVVTO,KVVTO,JVLFRO,KVLFRO,JVLTO,
     3KVLTO,JRETYP,JCOTYP,QUITOP,TEMP,VAPOR,QUID,TCSET,SUMFD,FLFEED,
     4VAPY,QUIDX.SUMX,SUMY,QUIMOH,VAPMOH,QUNBAL,FLVAP,FLLIQ,FLVAP1,
     5FLLIQ1,PRDSUM,RFSUM,CONDLD,REBLD,PRT
      NAMELIST /IN/ NCOMPS,NCOLS,NSTGS,A,B,C,ENTHK,ENTHL,ENTHU,
     1       ENTHW,PRDERR,BDFERR,NFEEDS,EXTFD,TOTFDH,NVPROS,NLPROS,
     2       VAPPRO,QUIPRO,NVFLOS,NLFLOS,JFROM,KFROM,JTO,KTO,QUIFLO,
     3       NVAPOS,NQUIDS,JRETYP,JCOTYP,TEMP,VAPOR,QUID,QUITOP,PRT,VAPFLO
      THALPF(Y,Z,T)=Y*T+Z
C
C  SPECIFY FEEDS NFEEDS IS THE TOTAL NUMBER OF EXTERNAL FEED STREAMS,
C  A PARTICULAR FEED ENTERING STAGE J OF COLUMN K
C
      READ (5,IN)
      IF(EOF(5)) 110,100
  110 CALL EXIT
  100 WRITE(6,IN)
C
C SPECIFY FIXED PRODUCT STREAMS NVPROS IS THE TOTAL NUMBER OF FIXED
C VAPOR PRODUCTS, NLPROS IS THE TOTAL NUMBER OF FIXED LIQUID PRODUCTS,
C A PARTICULAR PRODUCT OF AMOUNT VAPPRO OR QUIPRO BEING DRAWN FROM
C STAGE J OF COLUMN K
C
C
C  SPECIFY FIXED STREAMS INTERCONNECTING ANY STAGE IN ANY COLUMN WITH
C ANY OTHER STAGE IN ANY COLUMN NVFLOS IS THE TOTAL NUMBER OF FIXED
C VAPOR STREAMS, NLFLOS IS THE TOTAL NUMBER OF FIXED LIQUID STREAMS,
C A PARTICULAR STREAM LEAVING STAGE JFROM OF COLUMN KFROM AND ENTERING
C STAGE JTO OF COLUMN KTO
C
   13 DO 14 K=1,NCOLS
      JT=NSTGS(K)
      DO 14 J=1,JT
      JFVFRO(J,K)=0
      KFVFRO(J,K)=0
      JFVTO(J,K)=0
      KFVTO(J,K)=0
      JFLFRO(J,K)=0
      KFLFRO(J,K)=0
      JFLTO(J,K)=0
   14 KFLTO(J,K)=0
      IF (NVFLOS) 17,17,15
   15 DO 16 J=1,NVFLOS
      JFVFRO(JTO(1,J),KTO(1,J))=JFROM(1,J)
      KFVFRO(JTO(1,J),KTO(1,J))=KFROM(1,J)
      JFVTO(JFROM(1,J),KFROM(1,J))=JTO(1,J)
```

```
   16 KFVTO(JFROM(1,J),KFROM(1,J))=KTO(1,J)
   17 IF (NLFLOS) 20,20,18
   18 DO 19 J=1,NLFLOS
      JFLFRO(JTO(2,J),KTO(2,J))=JFROM(2,J)
      KFLFRO(JTO(2,J),KTO(2,J))=KFROM(2,J)
      JFLTO(JFROM(2,J),KFROM(2,J))=JTO(2,J)
   19 KFLTO(JFROM(2,J),KFROM(2,J))=KTO(2,J)
C
C SPECIFY VARIABLE STREAMS INTERCONNECTING ANY END STAGE IN ANY COLUMN
C WITH ANY STAGE IN ANY OTHER COLUMN NVAPOS IS THE TOTAL NUMBER OF
C VARIABLE VAPOR STREAMS LINKING COLUMNS, NQUIDS IS THE TOTAL NUMBER
C OF VARIABLE LIQUID STREAMS LINKING COLUMNS, A PARTICULAR STREAM
C LEAVING END STAGE JFROM OF COLUMN KFROM AND ENTERING STAGE JTO OF
C COLUMN KTO
C
   20 DO 21 K=1,NCOLS
      JT=NSTGS(K)
      DO 21 J=1,JT
      JVVFRO(J,K)=0
      KVVFRO(J,K)=0
      JVVTO(J,K)=0
      KVVTO(J,K)=0
      JVLFRO(J,K)=0
      KVLFRO(J,K)=0
      JVLTO(J,K)=0
   21 KVLTO(J,K)=0
      IF (NVAPOS) 24,24,22
   22 DO 23 JV=1,NVAPOS
      JVVFRO(JTO(3,JV),KTO(3,JV))=JFROM(3,JV)
      KVVFRO(JTO(3,JV),KTO(3,JV))=KFROM(3,JV)
      JVVTO(JFROM(3,JV),KFROM(3,JV))=JTO(3,JV)
   23 KVVTO(JFROM(3,JV),KFROM(3,JV))=KTO(3,JV)
   24 IF (NQUIDS) 27,27,25
   25 DO 26 JV=1,NQUIDS
      JVLFRO(JTO(4,JV),KTO(4,JV))=JFROM(4,JV)
      KVLFRO(JTO(4,JV),KTO(4,JV))=KFROM(4,JV)
      JVLTO(JFROM(4,JV),KFROM(4,JV))=JTO(4,JV)
   26 KVLTO(JFROM(4,JV),KFROM(4,JV))=KTO(4,JV)
C
C SPECIFY REBOILER TYPE AT BOTTOM OF COLUMN K FOR ALL COLUMNS
C JRETYP(K)=0 INDICATES NO REBOILER JRETYP(K)=1 INDICATES PARTIAL
C REBOILER, VAPOR FLOW FROM REBOILER WILL BE HELD CONSTANT AT VAPOR(1,K)
C
C
C SPECIFY CONDENSER TYPE AT TOP OF COLUMN K FOR ALL COLUMNS
C JCOTYP(K)=0 INDICATES NO CONDENSER JCOTYP(K)=1 INDICATES PARTIAL
   27 CONTINUE
C INDICATES TOTAL CONDENSER, REFLUX WILL BE HELD CONSTANT AT QUID(JT,K)
C JCOTYP(K)=3 INDICATES TWO PRODUCT CONDENSER, REFLUX WILL BE HELD
C CONSTANT AT QUID(JT,K) AND CONDENSER TEMPERATURE WILL BE HELD
C CONSTANT AT TEMP(JT,K) IF IT IS POSSIBLE TO PRODUCE FIXED STREAMS
C
C
C INITIAL ESTIMATES OF TEMPERATURES AND FLOWS
C
C
C INITIAL ESTIMATES OF LIQUID TOP PRODUCTS FOR COLUMNS WITH TOTAL OR TWO
C PRODUCT CONDENSERS
C
      DO 31 K=1,NCOLS
      IF (JCOTYP(K)-2) 31,29,30
   29 VAPOR(JT,K)=0.0
      GO TO 31
```

```
   30 TCSET(K)=TEMP(JT,K)
   31 CONTINUE
C
C PRINT INPUT DATA
C
   32 FORMAT (11H0INPUT DATA)
      WRITE (6,32)
   33 FORMAT (37H0NO. OF COMPONENTS      NO. OF COLUMNS/I10,I20)
      WRITE (6,33)NCOMPS,NCOLS
   34 FORMAT (63H0                                                  *****
     1 COLUMN I1,6H *****)
   35 FORMAT (20H0NUMBER OF STAGES = I3)
   36 FORMAT (24H EXTERNAL FEED TO STAGE I3,11H OF COLUMN I1)
   37 FORMAT (20H   COMPONENT AMOUNTS/(5E20.8))
   38 FORMAT (22H   ENTHALPY OF FEED = E15.8)
   39 FORMAT (12H0NO REBOILER)
   40 FORMAT (34H0REBOILER, FIXED REBOILER VAPOR = E15.8)
   41 FORMAT (13H0NO CONDENSER)
   42 FORMAT (45H0PARTIAL CONDENSER, FIXED SATURATED REFLUX = E15.8)
   43 FORMAT (43H0TOTAL CONDENSER, FIXED SATURATED REFLUX = E15.8)
   44 FORMAT (39H0TWO PRODUCT CONDENSER, FIXED REFLUX = E15.8,22H      CO
     1NDENSER TEMP = E15.8,12H IF POSSIBLE)
      DO 57 K=1,NCOLS
      JT=NSTGS(K)
      WRITE (6,34)K
      WRITE (6,35)NSTGS(K)
      DO 47 J=1,JT
      DO 45 I=1,NCOMPS
      IF (EXTFD(I,J,K)) 46,45,46
   45 CONTINUE
      GO TO 47
   46 WRITE (6,36)J,K
      WRITE (6,37)(EXTFD(I,J,K),I=1,NCOMPS)
      WRITE (6,38)TOTFDH(J,K)
   47 CONTINUE
      IF (JRETYP(K)) 48,48,49
   48 WRITE (6,39)
      GO TO 50
   49 WRITE (6,40)VAPOR(1,K)
   50 IF (JCOTYP(K)) 51,51,52
   51 WRITE (6,41)
      GO TO 57
   52 IF (JCOTYP(K)-1) 53,53,54
   53 WRITE (6,42)QUID(JT,K)
      GO TO 57
   54 IF (JCOTYP(K)-2) 55,55,56
   55 WRITE (6,43)QUID(JT,K)
      GO TO 57
   56 WRITE (6,44)QUID(JT,K),TEMP(JT,K)
   57 CONTINUE
   58 FORMAT (31H0FIXED VAPOR PRODUCT STREAM OF E15.8,18H MOLES FROM STA
     1GE I3,11H OF COLUMN I1)
   59 FORMAT (32H0FIXED LIQUID PRODUCT STREAM OF E15.8,18H MOLES FROM ST
     1AGE I3,11H OF COLUMN I1)
   60 FORMAT (39H0FIXED INTERCONNECTING VAPOR STREAM OF E15.8,17H MOLES
     1FROM STAGE I3,11H OF COLUMN I1,10H TO STAGE I3,11H OF COLUMN I1)
   61 FORMAT (40H0FIXED INTERCONNECTING LIQUID STREAM OF E15.8,17H MOLES
     1FROM STAGE I3,11H OF COLUMN I1,10H TO STAGE I3,11H OF COLUMN I1)
   62 FORMAT (50H0VARIABLE INTERCONNECTING VAPOR STREAM FROM STAGE I3,11
     1H OF COLUMN I1,10H TO STAGE I3,11H OF COLUMN I1)
   63 FORMAT (51H0VARIABLE INTERCONNECTING LIQUID STREAM FROM STAGE I3,1
     11H OF COLUMN I1,10H TO STAGE I3,11H OF COLUMN I1)
      DO 67 K=1,NCOLS
```

```
      JT=NSTGS(K)
      DO 67 J=1,JT
      IF (VAPPRO(J,K)) 64,65,64
   64 WRITE (6,58)VAPPRO(J,K),J,K
   65 IF (QUIPRO(J,K)) 66,67,66
   66 WRITE (6,59)QUIPRO(J,K),J,K
   67 CONTINUE
      DO 71 K=1,NCOLS
      JT=NSTGS(K)
      DO 71 J=1,JT
      IF (VAPFLO(J,K)) 68,69,68
   68 WRITE (6,60)VAPFLO(J,K),J,K,JFVTO(J,K),KFVTO(J,K)
   69 IF (QUIFLO(J,K)) 70,71,70
   70 WRITE (6,61)QUIFLO(J,K),J,K,JFLTO(J,K),KFLTO(J,K)
   71 CONTINUE
      DO 75 K=1,NCOLS
      JT=NSTGS(K)
      DO 75 J=1,JT
      IF (KVVTO(J,K)) 72,73,72
   72 WRITE (6,62)J,K,JVVTO(J,K),KVVTO(J,K)
   73 IF (KVLTO(J,K)) 74,75,74
   74 WRITE (6,63)J,K,JVLTO(J,K),KVLTO(J,K)
   75 CONTINUE
   76 FORMAT (82H0EQUILIBRIUM CONSTANTS A        EQUILIBRIUM CONSTANTS B
     1      EQUILIBRIUM CONSTANTS C/(E19.8,2E29.8))
      DO 77 K=1,NCOLS
      WRITE (6,34)K
   77 WRITE (6,76)(A(I,K),B(I,K),C(I,K),I=1,NCOMPS)
   78 FORMAT (99H ENTHALPY CONSTANTS K        ENTHALPY CONSTANTS L        EN
     1THALPY CONSTANTS U        ENTHALPY CONSTANTS W/(E18.8,3E26.8))
      WRITE (6,78)(ENTHK(I),ENTHL(I),ENTHU(I),ENTHW(I),I=1,NC
     1OMPS)
   79 FORMAT (52H0PRODUCT ERROR LIMIT     BUBBLE DEW FLASH ERROR LIMIT/(E
     117.8,E28.8))
      WRITE (6,79)PRDERR,BDFERR
   80 FORMAT (55H0          TEMPERATURES          VAPOR FLOWS        LIQUID FLOWS
     1/(3E19.8))
   81 FORMAT (E57.8)
      DO 83 K=1,NCOLS
      JT=NSTGS(K)
      WRITE (6,34)K
      WRITE (6,80)(TEMP(J,K),VAPOR(J,K),QUID(J,K),J=1,JT)
      IF (LCOTYP(K)-2) 83,82,82
   82 WRITE (6,81)QUITOP(K)
   83 CONTINUE
      FLFEES=0.0
      FIXV=0.0
      FIXL=0.0
      BDFERR=10.0*BDFERR
      DO 84 I=1,NCOMPS
      FLFEED(I)=0.0
      DO 84 K=1,NCOLS
      JT=NSTGS(K)
      DO 84 J=1,JT
      FLFEED(I)=FLFEED(I)+EXTFD(I,J,K)
   84 FLFEES=FLFEES+EXTFD(I,J,K)
      DO 88 K=1,NCOLS
      JT=NSTGS(K)
      DO 88 J=1,JT
      IF (J-1) 85,85,86
   85 FLVAS=.5*FLFEES
      FLLIS=FLFEES-FLVAS
   86 CALL ISOTFL (FLFEED,FLFEES,FLVAS,FLLIS,FIXV,FIXL,TEMP(J,K),A,B,C,
```

```
     1NCOMPS,K,BDFERR,VAPY,QUIDX)
      QUIMOH(J,K)=0.0
      VAPMOH(J,K)=0.0
      DO 87 I=1,NCOMPS
      GENX(I,J,K)=QUIDX(I)
      QUIMOH(J,K)=QUIMOH(J,K)+GENX(I,J,K)*THALPF(ENTHK(I),ENTHL(I),
     1TEMP(J,K))
      GENY(I,J,K)=VAPY(I)
   87 VAPMOH(J,K)=VAPMOH(J,K)+GENY(I,J,K)*THALPF(ENTHU(I),ENTHW(I),
     1TEMP(J,K))
   88 CONTINUE
      BDFERR=BDFERR/10.0
      RETURN
      END
C      PROGRAM 9     PROGRAM FOR INTERLINKED COLUMNS
C
C  SUBROUTINE FOR OUTPUT
C
      SUBROUTINE OUTPUT (ITERAT,NCOMPS,NCOLS,CHECK)
      DIMENSION GENX(20.40,5),GENY(20,40,5),EXTFD(20.40,5),NSTGS(5),
     1A(20,5),B(20,5),C(20,5),ENTHK(20).ENTHL(20),ENTHU(20),ENTHW(20),
     2TOTFDH(40,5).VAPPRO(40,5),QUIPRO(40,5),VAPFLO(40,5),QUIFLO(40,5),
     3JFVFRO(40,5),KFVFRO(40,5),JFVTO(40,5),KFVTO(40,5),JFLFRO(40,5),
     4KFLFRO(40,5),JFLTO(40,5),KFLTO(40,5),JVVFRO(40,5),KVVFRO(40,5),
     5JVVTO(40,5),KVVTO(40,5),JVLFRO(40,5),KVLFRO(40,5),JVLTO(40,5),
     6KVLTO(40,5),JRETYP(5),JCOTYP(5),QUITOP(5),TEMP(40,5),VAPOR(40,5),
     7QUID(40,5),TCSET(5),SUMFD(20),FLFEED(20),VAPY(20),QUIDX(20),
     8SUMX(40,5),SUMY(40,5),QUIMOH(40,5),VAPMOH(40,5),QUNBAL(40,5),
     9FLVAP(20),FLLIQ(20),FLVAP1(20),FLLIQ1(20),PRDSUM(20),RFSUM(20)
      DIMENSION CONDLD(5),REBLD(5)
      COMMON GENX,GENY,EXTFD,NSTGS,A,B,C,ENTHK,ENTHL,ENTHU,ENTHW,TOTFDH,
     1VAPPRO,QUIPRO,VAPFLO,QUIFLO,JFVFRO,KFVFRO,JFVTO,KFVTO,JFLFRO,
     2KFLFRO,JFLTO,KFLTO,JVVFRO,KVVFRO,JVVTO,KVVTO,JVLFRO,KVLFRO,JVLTO,
     3KVLTO,JRETYP,JCOTYP,QUITOP,TEMP,VAPOR,QUID,TCSET,SUMFD,FLFEED,
     4VAPY,QUIDX,SUMX,SUMY,QUIMOH,VAPMOH,QUNBAL,FLVAP,FLLIQ,FLVAP1,
     5FLLIQ1,PRDSUM,RFSUM,CONDLD,REBLD
    1 FORMAT (12H1PROBLEM NO./17H0ITERATION NO. = I3)
      WRITE (6,1)ITERAT
      DO 95 K=1,NCOLS
      JT=NSTGS(K)
    2 FORMAT (63H0                                                *****
    1 COLUMN I1,6H *****)
      WRITE (6,2)K
    3 IF (CHECK) 31,31,5
    4 FORMAT (63H0LIQUID AND VAPOR MOLE FRACTIONS LISTED AS COMPONENTS P
     1ER STAGE)
    5 WRITE (6,4)
    6 FORMAT (19H0       STAGE NO. = I3)
    7 FORMAT (19H0       STAGE NO. = I3,15H     (REBOILER))
    8 FORMAT (19H0       STAGE NO. = I3,24H     (PARTIAL CONDENSER))
    9 FORMAT (19H0       STAGE NO. = I3,22H     (TOTAL CONDENSER))
   10 FORMAT (19H0       STAGE NO. = I3,28H     (TWO PRODUCT CONDENSER))
   11 FORMAT (7H LIQUID (5E20.8))
   12 FORMAT (6H VAPOR/(5E20.8))
      JSTAGE=1
      IF (JRETYP(K)) 13,13,14
   13 WRITE (6,6)JSTAGE
      GO TO 15
   14 WRITE (6,7)JSTAGE
   15 WRITE (6,11)(GENX(I,1,K),I=1,NCOMPS)
      WRITE (6,12)(GENY(I,1,K),I=1,NCOMPS)
      JMIN=JT-1
      DO 16 J=2,JMIN
```

```
      JSTAGE=JSTAGE+1
      WRITE (6,6)JSTAGE
      WRITE (6,11)(GENX(I,J,K),I=1,NCOMPS)
   16 WRITE (6,12)(GENY(I,J,K),I=1,NCOMPS)
      IF (JCOTYP(K)-1) 17,18,19
   17 WRITE (6,6)JT
      GO TO 22
   18 WRITE (6,8)JT
      GO TO 22
   19 IF (JCOTYP(K)-2) 20,20,21
   20 WRITE (6,9)JT
      GO TO 22
   21 WRITE (6,10)JT
   22 WRITE (6,11)(GENX(I,JT,K),I=1,NCOMPS)
   23 WRITE (6,12)(GENY(I,JT,K),I=1,NCOMPS)
   24 FORMAT (27H0LIQUID PRODUCT FROM STAGE I3,5H  OF E15.8,7H  MOLES)
   25 FORMAT (26H0VAPOR PRODUCT FROM STAGE I3,5H  OF E15.8,7H  MOLES)
   26 FORMAT (19H0BOTTOM PRODUCT OF E15.8,7H  MOLES)
   27 FORMAT (23H0LIQUID TOP PRODUCT OF E15.8,7H  MOLES)
   28 FORMAT (22H0VAPOR TOP PRODUCT OF E15.8,7H  MOLES)
   29 FORMAT (21H     COMPONENT AMOUNTS/(5E20.8))
   30 FORMAT (32H     COMPONENT RECOVERY FRACTIONS/(5E20.8))
   31 DO 39 J=1,JT
      IF (QUIPRO(J,K)) 35,35,32
   32 WRITE (6,24)J,QUIPRO(J,K)
      DO 33 I=1,NCOMPS
   33 QUIDX(I)=QUIPRO(J,K)*GENX(I,J,K)
      WRITE (6,29)(QUIDX(I),I=1,NCOMPS)
      DO 34 I=1,NCOMPS
   34 QUIDX(I)=QUIDX(I)/SUMFD(I)
      WRITE (6,30)(QUIDX(I),I=1,NCOMPS)
   35 IF (VAPPRO(J,K)) 39,39,36
   36 WRITE (6,25)J,VAPPRO(J,K)
      DO 37 I=1,NCOMPS
   37 QUIDX(I)=VAPPRO(J,K)*GENY(I,J,K)
      WRITE (6,29)(QUIDX(I),I=1,NCOMPS)
      DO 38 I=1,NCOMPS
   38 QUIDX(I)=QUIDX(I)/SUMFD(I)
      WRITE (6,30)(QUIDX(I),I=1,NCOMPS)
   39 CONTINUE
      IF (KVLTO(1,K)) 43,40,43
   40 WRITE (6,26)QUID(1,K)
      DO 41 I=1,NCOMPS
   41 QUIDX(I)=QUID(1,K)*GENX(I,1,K)
      WRITE (6,29)(QUIDX(I),I=1,NCOMPS)
      DO 42 I=1,NCOMPS
   42 QUIDX(I)=QUIDX(I)/SUMFD(I)
      WRITE (6,30)(QUIDX(I),I=1,NCOMPS)
   43 IF (JCOTYP(K)-2) 49,44,44
   44 IF (QUITOP(K)) 49,49,45
   45 IF (KVLTO(JT,K)) 49,46,49
   46 WRITE (6,27)QUITOP(K)
      DO 47 I=1,NCOMPS
   47 QUIDX(I)=QUITOP(K)*GENX(I,JT,K)
      WRITE (6,29)(QUIDX(I),I=1,NCOMPS)
      DO 48 I=1,NCOMPS
   48 QUIDX(I)=QUIDX(I)/SUMFD(I)
      WRITE (6,30)(QUIDX(I),I=1,NCOMPS)
   49 IF (JCOTYP(K)-2) 51,57,50
   50 IF (VAPOR(JT,K)) 57,57,51
   51 IF (KVVTO(JT,K)) 57,52,57
   52 WRITE (6,28)VAPOR(JT,K)
      DO 53 I=1,NCOMPS
```

```
53 QUIDX(I)=VAPOR(JT,K)*GENY(I,JT,K)
   WRITE (6,29)(QUIDX(I),I=1,NCOMPS)
   DO 54 I=1,NCOMPS
54 QUIDX(I)=QUIDX(I)/SUMFD(I)
   WRITE (6,30)(QUIDX(I),I=1,NCOMPS)
55 FORMAT (22H0FIXED LIQUID FLOW OF E15.8,19H  MOLES FROM STAGE I3,11
  1H  TO STAGE I3,12H  OF COLUMN I3)
56 FORMAT (21H0FIXED VAPOR FLOW OF E15.8,19H  MOLES FROM STAGE I3,11H
  1  TO STAGE I3,12H  OF COLUMN I3)
57 DO 63 J=1,JT
   IF (QUIFLO(J,K)) 60,60,58
58 WRITE (6,55)QUIFLO(J,K),J,JFLTO(J,K),KFLTO(J,K)
   DO 59 I=1,NCOMPS
59 QUIDX(I)=QUIFLO(J,K)*GENX(I,J,K)
   WRITE (6,29)(QUIDX(I),I=1,NCOMPS)
60 IF (VAPFLO(J,K)) 63,63,61
61 WRITE (6,56)VAPFLO(J,K),J,JFVTO(J,K),KFVTO(J,K)
   DO 62 I=1,NCOMPS
62 QUIDX(I)=VAPFLO(J,K)*GENY(I,J,K)
   WRITE (6,29)(QUIDX(I),I=1,NCOMPS)
63 CONTINUE
64 FORMAT (16H0LIQUID FLOW OF E15.8,19H  MOLES FROM STAGE I3,11H  TO
  1STAGE I3,12H  OF COLUMN I3)
65 FORMAT (16H0LIQUID FLOW OF E15.8,31H  MOLES FROM REBOILER TO STAGE
  1 I3,12H  OF COLUMN I3)
66 FORMAT (16H0LIQUID FLOW OF E15.8,32H  MOLES FROM CONDENSER TO STAG
  1E I3,12H  OF COLUMN I3)
67 FORMAT (15H0VAPOR FLOW OF E15.8,19H  MOLES FROM STAGE I3,11H  TO S
  1TAGE I3,12H  OF COLUMN I3)
68 FORMAT (15H0VAPOR FLOW OF E15.8,32H MOLES FROM CONDENSER TO STAGE
  1 I3,12H  OF COLUMN I3)
69 IF (KVLTO(1,K)) 75,75,70
70 IF (JPETYP(K)) 71,71,72
71 J=1
   WRITE (6,64)QUID(1,K),J,JVLTO(1,K),KVLTO(1,K)
   GO TO 73
72 WRITE (6,65)QUID(1,K),JVLTO(1,K),KVLTO(1,K)
73 DO 74 I=1,NCOMPS
74 QUIDX(I)=QUID(1,K)*GENX(I,1,K)
   WRITE (6,29)(QUIDX(I),I=1,NCOMPS)
75 IF (KVLTO(JT,K)) 78,78,76
76 WRITE (6,66)QUITOP(K),JVLTO(JT,K),KVLTO(JT,K)
   DO 77 I=1,NCOMPS
77 QUIDX(I)=QUITOP(K)*GENX(I,JT,K)
   WRITE (6,29)(QUIDX(I),I=1,NCOMPS)
78 IF (KVVTO(JT,K)) 86,86,79
79 IF (JCOTYP(K)) 80,80,81
80 WRITE (6,67)VAPOR(JT,K),JT,JVVTO(JT,K),KVVTO(JT,K)
   GO TO 82
81 WRITE (6,68)VAPOR(JT,K),JVVTO(JT,K),KVVTO(JT,K)
82 DO 83 I=1,NCOMPS
83 QUIDX(I)=VAPOR(JT,K)*GENY(I,JT,K)
   WRITE (6,29)(QUIDX(I),I=1,NCOMPS)
84 FORMAT (16H0STAGE VARIABLES)
85 FORMAT( 120H0      SUMX              SUMY         TEMPERATURE        VAPO
  1R            LIQUID        VAP MOL ENTH   LIQ MOL ENTH  HEAT UNBALANCE
  2/(8E15.8))
86 WRITE (6,84)
   WRITE (6,85)(SUMX(J,K),SUMY(J,K),TEMP(J,K),VAPOR(J,K),Q
  1UID(J,K),VAPMOH(J,K),QUIMOH(J,K),QUNBAL(J,K),J=1,JT)
87 FORMAT (E75.8)
   IF (JCOTYP(K)-2) 91,88,88
88 WRITE (6,87)QUITOP(K)
```

```
   89 FORMAT (17H0REBOILER LOAD = E15.8)
   90 FORMAT (18H0CONDENSER LOAD = E15.8)
   91 IF (REBLD(K)) 93,93,92
   92 WRITE (6,89) REBLD(K)
   93 IF (CONDLD(K)) 95,95,94
   94 WRITE (6,90) CONDLD(K)
   95 CONTINUE
   96 FORMAT (2H0 /48H0RECOVERY FRACTION SUMMATIONS FOR EACH COMPONENT/(
     15E20.8))
      WRITE (6,96) (RESUM(I),I=1,NCOMPS)
   97 FORMAT (77H0EXCESS MOLES OF EACH COMPONENT LEAVING THE SYSTEM IN P
     1RODUCTS OVER MOLES FED/(5E20.8))
      WRITE (6,97) (PRDSUM(I).I=1,NCOMPS)
      RETURN
      END
C     PROGRAM 9 PROGRAM FOR INTERLINKED COLUMNS
C
C  SUBROUTINE FOR BUBBLE POINTS
C
      SUBROUTINE BUBPT (QUIDX,VAPY,T,A,B,C,NCOMPS,K,BDFERR)
      DIMENSION QUIDX(20),VAPY(20).A(20,5),B(20,5),C(20,5)
      EQUILKF(A,B,C,T)=EXP(A/(T+460.0)+B+C*(T+460.0))
      KTIMES=1
    1 SUMY=0.0
      DO 2 I=1,NCOMPS
      VAPY(I)=EQUILKF(A(I,K),B(I,K),C(I,K),T)*QUIDX(I)
    2 SUMY=SUMY+VAPY(I)
      IF (ABS(SUMY-1.0)-BDFERR) 6,6,3
    3 KTIMES=KTIMES-1
      IF (KTIMES) 5,4,4
    4 SUMYO=SUMY
      TO=T
      T=T+10.0
      GO TO 1
    5 SLOPE=(SUMY-SUMYO)/(T-TO)
      SUMYO=SUMY
      TO=T
      IF(SLOPE.EQ.0.0) SLOPE=1.0E-06
      T=((1.0-SUMY)/SLOPE)+T
      GO TO 1
    6 RETURN
      END
C     PROGRAM 9 PROGRAM FOR INTERLINKED COLUMNS
C
C  SUBROUTINE FOR DEW POINTS
C
      SUBROUTINE DEWPT (VAPY,QUIDX,T,A,B.C,NCOMPS,K,BDFERR)
      DIMENSION VAPY(20),QUIDX(20),A(20,5),B(20,5),C(20,5)
      EQUILKF(A,B.C,T)=EXP(A/(T+460.0)+B+C*(T+460.0))
      KTIMES=1
    1 SUMX=0.0
      DO 2 I=1,NCOMPS
      QUIDX(I)=VAPY(I)/EQUILKF(A(I.K),B(I,K),C(I,K).T)
    2 SUMX=SUMX+QUIDX(I)
      IF (ABS(SUMX-1.0)-BDFERR) 6,6,3
    3 KTIMES=KTIMES-1
      IF (KTIMES) 5,4,4
    4 SUMXO=SUMX
      TO=T
      T=T+10.0
      GO TO 1
    5 SLOPE=(SUMX-SUMXO)/(T-TO)
      SUMXO=SUMX
```

```
      TO=T
      IF(SLOPE.EQ.0.0) SLOPE=1.0E-06
      T=((1.0-SUMY)/SLOPE)+T
      GO TO 1
    6 RETURN
      END
C     PROGRAM 9 PROGRAM FOR INTERLINKED COLUMNS
C
C  SUBROUTINE FOR CALCULATION OF FLASH TEMPERATURE
C
      SUBROUTINE ISOVFL (FLFEED,FLFEES,FLVAS,T,A,B,C,NCOMPS,K,BDFERR
     1VAPY,QUIDX)
      DIMENSION FLFEED(20),A(20,5),B(20,5),C(20,5),VAPY(20),QUIDX(20)
      EQUILKF(A,B,C,T)=EXP(A/(T+460.0)+B+C*(T+460.0))
      FLLIS=FLFEES-FLVAS
      KTIMES=1
 1    SUMY=0.0
      SUMX=0.0
      DO 2 I=1,NCOMPS
      VAPY(I)=FLFEED(I)/(FLLIS/FLVAS/EQUILKF(A(I,K),B(I,K),C(I,K),T)+1.0
     1)
      QUIDX(I)=(FLFEED(I)-VAPY(I))/FLLIS
      VAPY(I)=VAPY(I)/FLVAS
      SUMY=SUMY+VAPY(I)
 2    SUMX=SUMX+QUIDX(I)
      IF (ABS(SUMY-1.0)-BDFERR) 3,3.4
 3    IF (ABS(SUMX-1.0)-BDFERR) 7,7,4
 4    KTIMES=KTIMES-1
      IF (KTIMES) 6,5,5
 5    SUMYO=SUMY
      TO=T
      T=T+10.0
      GO TO 1
 6    SLOPE=(SUMY-SUMYO)/(T-TO)
      SUMYO=SUMY
      TO=T
      IF(SLOPE.EQ.0.0) SLOPE=1.0E-06
      T=((1.0-SUMY)/SLOPE)+T
      GO TO 1
 7    RETURN
      END
C     PROGRAM 9 PROGRAM FOR INTERLINKED COLUMNS
C
C  SUBROUTINE FOR ISOTHERMAL FLASH
C
      SUBROUTINE ISOTFL (FLFEED,FLFEES,FLVAS,FLLIS,FIXV,FIXL,T,A,B,C,
     1NCOMPS,K,BDFERR,VAPY,QUIDX)
      DIMENSION FLFEED(20),A(20,5),B(20,5),C(20,5),VAPY(20),QUIDX(20),
     1EQK(20),FLVAP(20),FLLIS(20)
      EQUILKF(A,B,C,T)=EXP(A/(T+460.0)+B+C*(T+460.0))
      KTIMES=1
      DO 1 I=1,NCOMPS
    1 EQK(I)=EQUILKF(A(I,K),B(I,K),C(I,K),T)
    2 SUMY=0.0
      SUMX=0.0
      IF (FLVAS-FLLIS) 3,3,16
    3 DO 4 I=1,NCOMPS
      FLVAP(I)=FLFEED(I)/(FLLIS/FLVAS/EQK(I)+1.0)
      VAPY(I)=FLVAP(I)/FLVAS
      QUIDX(I)=(FLFEED(I)-FLVAP(I))/FLLIS
      SUMY=SUMY+VAPY(I)
    4 SUMX=SUMX+QUIDX(I)
      IF (ABS(SUMY-1.0)-BDFERR) 5,5,6
```

```
  5 IF (ABS(SUMX-1.0)-BDFERR) 15,15,6
  6 IF (FLVAS/FLFEES-1.0E-6) 29,29,7
  7 KTIMES=KTIMES-1
    IF (KTIMES) 12,8,8
  8 SUMYO=SUMY
    SUMXO=SUMX
    FLVASO=FLVAS-1.0E-06
    FLLISO=FLLIS
  9 FLVAS=0.0
    DO 10 I=1,NCOMPS
 10 FLVAS=FLVAS+FLVAF(I)
    FLLIS=FLFEES-FLVAS
    IF (FLVAS-FIXV) 15,15,11
 11 IF (FLLIS-FIXL) 15,15,2
 12 SLOPE=(SUMY-SUMYO)/(FLVAS-FLVASO)
    FLVASO=FLVAS
    FLLISO=FLLIS
    SUMYO=SUMY
    SUMXO=SUMX
    IF(SLOPE.EQ.0.0) SLOPE=1.0E-06
    FLVAS=((1.0-SUMY)/SLOPE)+FLVAS
    IF (FLVAS-FIXV) 9,9,13
 13 IF (FLFEES-FLVAS-FIXL) 9,9,14
 14 FLLIS=FLFEES-FLVAS
    GO TO 2
 15 RETURN
 16 DO 17 I=1,NCOMPS
    FLLIO(I)=FLFEED(I)/(FLVAS*EQK(I)/FLLIS+1.0)
    QUIDX(I)=FLLIO(I)/FLLIS
    VAPY(I)=(FLFEED(I)-FLLIO(I))/FLVAS
    SUMX=SUMX+QUIDX(I)
 17 SUMY=SUMY+VAPY(I)
    IF (ABS(SUMX-1.0)-BDFERR) 18,18,19
 18 IF (ABS(SUMY-1.0)-BDFERR) 28,28,19
 19 IF (FLLIS/FLFEES-1.0E-6) 29,29,20
 20 KTIMES=KTIMES-1
    IF (KTIMES) 25,21,21
 21 SUMXO=SUMX
    SUMYO=SUMY
    FLLISO=FLLIS-1.0E-06
    FLVASO=FLVAS
 22 FLLIS=0.0
    DO 23 I=1,NCOMPS
 23 FLLIS=FLLIS+FLLIO(I)
    FLVAS=FLFEES-FLLIS
    IF (FLLIS-FIXL) 28,28,24
 24 IF (FLVAS-FIXV) 28,28,2
 25 SLOPE=(SUMX-SUMXO)/(FLLIS-FLLISO)
    FLLISO=FLLIS
    FLVASO=FLVAS
    SUMXO=SUMX
    SUMYO=SUMY
    IF(SLOPE.EQ.0.0) SLOPE=1.0E-06
    FLLIS=((1.0-SUMX)/SLOPE)+FLLIS
    IF (FLLIS-FIXL) 22,22,26
 26 IF (FLFEES-FLLIS-FIXV) 22,22,27
 27 FLVAS=FLFEES-FLLIS
    GO TO 2
 28 RETURN
 29 DO 30 I=1,NCOMPS
    VAPY(I)=VAPY(I)/SUMY
 30 QUIDX(I)=QUIDX(I)/SUMX
    RETURN
```

DISTILL

INPUT

DISTILL INPUT

The input to the DISTILL model is in the form of NAMELIST input. Inputs to the model are described in Ref.5. The sample input list on the following page was actual input for a case run on the eight theoretical stage column modeled for the Phase II sidestream processor. The feed oil consisted of a 10 component mixture with properties of oil typified from Sample B-1 (oil used in Phase II).

```
$IN
NCOMPS=10,
NCOLS=1,
NSTGS=8,
A(1,1)=-10013.3,-13921.3,-15449.5.-17012.4,-17935.6,-19155.1,-20520.3
   -21993.7,-22907.2,-24669.3,
B(1,1)=15.802,17.523,17.924.19.673,18.958.19.509.20.346,21,064,
   21.396,22.365,
C(1,1)=10*0.,
ENTHK(1)=120.95,183.36,213.87,236.75,255.80,277.53,297.95,311.22
   325.23,348.03,
ENTHL(1)=-6195.,-11165.,-13794.,-15808.,-17340.,-19278.,-21238..
   -22372.,-23660..-25970..
ENTHU(1)=109.15,146.16,162.55,173.73,185.20,195.83,204.60,211.00,
   217.53,226.13,
ENTHW(1)=15930..29940.,37218.,43110..47270.,52870.,58410.,61870.,
   65550.,72080.,
PRDERR = 0.01,
BDFERR = 0.0001,
NFEEDS = 1,
EXTFD(1,1,1)=2*.05,2*.10,2*.20,2*.10,2*.05,
TOTFDH(1,1)=99394.,-32135.,-25272.,-17553.,-14980.,-12406.,
 -10691.,
NVPROS = 0,
NLPROS = 1,
VAPPRO = 0,
QUIPRO(3,1)=0.8,
NVFLOS = 0,
NLFLOS=0,
JRETYP(1)=1,
JCOTYP(1)=3,
TEMP=560.,530.,510.,500.,445.,405.,300.,70.,
VAPOR = 3.6,6*1.0,
QUID=8*0.75,
QUITOP=0.04,
$END
```

DISTILL

OUTPUT

DISTILL OUTPUT

Output from a sample case run using the inputs previously described are listed on the following pages. The column temperatures, stage vapor and liquid flows, along with process heat balances are summarized on the last page of the listing.

```
$IN

NCOMPS   = 10,

NCOLS    = 1,

NSTGS    = 8, 0, 0, 0, 0,

A        = -.100133E+05, -.139213E+05, -.154495E+05, -.170124E+05, -.179356E+05, -.191551E+05, -.208203E+05, -.219937E+05,
           -.229072E+05, -.246693E+05, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,   0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
           0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
           0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
           0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
           0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,

B        = .15802E+02, .17523E+02, .17924E+02, .18673E+02, .18958E+02,          .19509E+02, .20346E+02, .21064E+02,
           .21396E 02, .22365E+02, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,  0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
           0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
           0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
           0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
           0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,

C        = 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
           0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
           0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
           0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
           0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,

ENTHK    = .12095E+03, .18336E+03, .21387E+03,  23675E+03, .2558E+03,           .27753E+03, .29795E+03, .3112E+03,
           .32523E+03, .34803E+03, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,  0.0,

ENTHL    = -.6195E+04, -.11165E+05, -.13794E+05, -.15808E+05, -.1734E+05,        -.19278E+05, -.21238E+05, -.22372E+05,
           -.2366E+05, -.2597E+05, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,  0.0,

ENTHU    = .10915E+03, .14616E+03, .16255E+03, .17373E+03, .1852E+03,           .19583E+03, .2046E+03, .211E+03, .21753E+03,
           .22613E+03, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,

ENTHW    = .1593E+05, .2994E+05, .37218E+05, .4311E+05, .4727E+05, .5287E+05,    .5841E+05, .6187E+05, .6555E+05, .7208E+05,
           0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,

PRDERR   = .1E-01,

BDFERR   = .1E-03,

NFEEDS   = 1,

EXTFD    = .5E-01, .5E-01, .1E+00, .1E+00, .2E+00, .2E+00, .1E+00, .1E+00,       .5E-01, .5E-01, 0.0, 0.0, 0.0, 0.0,
           0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
           0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
           0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
           0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
           0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
           0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
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           0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
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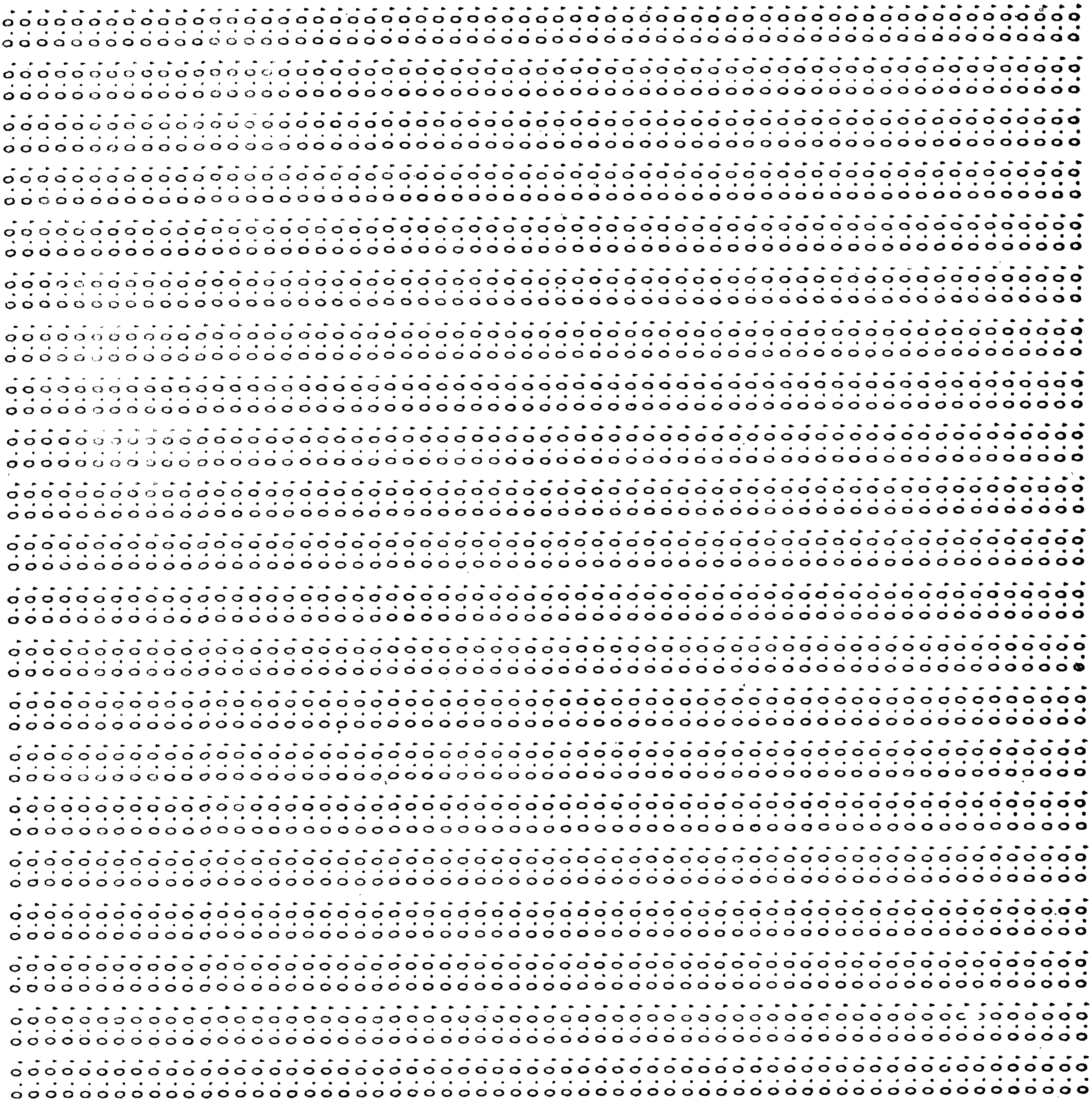
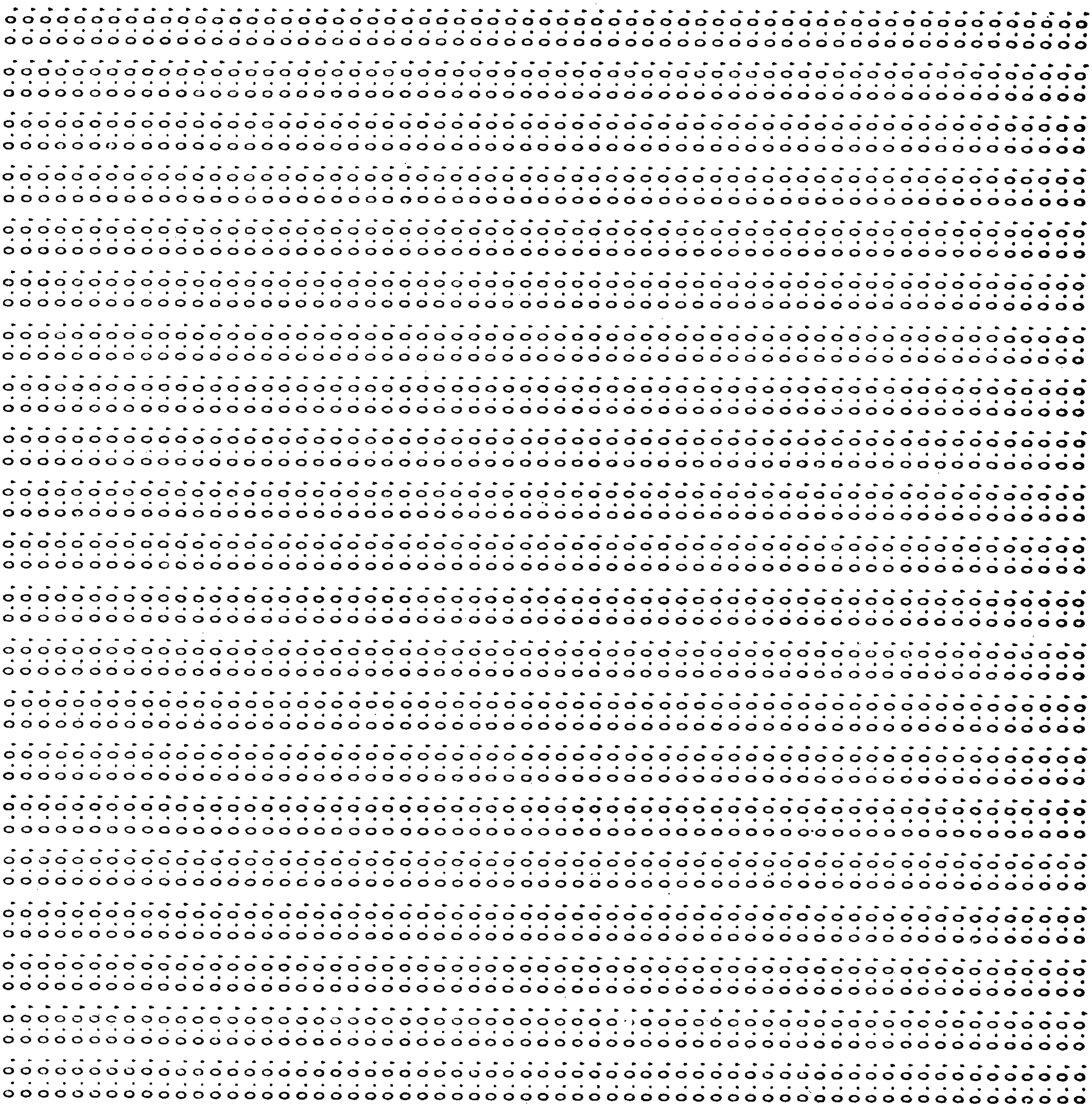
           0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
           0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
           0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
           0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
```





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            0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
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            0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
            0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
            0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
            0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
            0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
            0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
            0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
            0.0, 0.  , 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
            0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
            0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
            0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
            0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
            0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
            0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
            0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
            0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
            0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
            0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
            0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
            0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
            0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
            0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
            0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
            0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
            0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
            0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
            0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
            0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
            0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
            0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
            0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
            0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
            0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
            0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
            0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
            0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
            0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
            0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
            0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,

TOTFDH   = .99394E+05, -.32135E+05, -.25272E+05, -.17553E+05, -.1498E+05,        -.12406E+05, -.10691E+05, 0.0, 0.0,
            0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
            0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
            0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
            0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
            0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
            0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
            0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
            0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
            0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
            0.0, 0.0,

NVPROS   = 0,
```

```
NLPROS  = 1,

VAPPRO  = 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
          0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
          0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
          0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
          0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
          0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
          0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
          0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
          0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
          0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,

QUIPRO  = 0.0, 0.0, .8E+00, 0.0, 0.0, 0.0, 0.0, 0.0, 0 0, 0.0, 0.0, 0.0, 0.0,   0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
          0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
          0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
          0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
          0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
          0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
          0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
          0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
          0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
          0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,

NVFLOS  = 0,

NLFLOS  = 0,

JFROM   = 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0,

KFROM   = 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0,

JTO     = 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0,

KTO     = 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0,

QUIFLO  = 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
          0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
          0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
          0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
          0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
          0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
          0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
          0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
          0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
          0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,

NVAPOS  = 0,

NQUIDS  = 0,

JRETYP  = 1, 0, 0, 0, 0,

JCOTYP  = 3, 0, 0, 0, 0,

TEMP    = .56E+03  .53E+03, .51E+03, .5E+03, .445E+03, .405E+03, .3E+03,        .7E+02, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
          0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
          0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
          0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
          0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
          0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
          0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
          0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
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          0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
          0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0.

 VAPOR  = .36E+01, .1E+01, .1E+01, .1E+01, .1E+01, .1E+01, .1E+01, 0.0, 0.0,    0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
          0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
          0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
          0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
          0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
          0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
          0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
          0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
          0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
          0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,

 QUID   = .75E+00, .75E+00, .75E+00, .75E+00, .75E+00, .75E+00, .75E+00,      .75E+00, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
          0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
          0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
          0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
          0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
          0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
          0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
          0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
          0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
          0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,

 QUITOP = .4E-01, 0.0, 0.0, 0.0, 0.0,

 PRT    = 0.0,

 VAPFLO = 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
          0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
          0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
          0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
          0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
          0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
          0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
          0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
          0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,
          0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0, 0.0,

 $END

 INPUT DATA

 NO. OF COMPONENTS     NO. OF COLUMNS
        10                   1

                                     ***** COLUMN 1 *****

 NUMBER OF STAGES =    8
 EXTERNAL FEED TO STAGE   1 OF COLUMN  1
   COMPONENT AMOUNTS
       .50000000E-01       .50000000E-01       .10000000E+00       .10000000E+00       .20000000E+00
       .20000000E+00       .10000000E+00       .10000000E+00       .50000000E-01       .50000000E-01
   ENTHALPY OF FEED =    .99394000E+05

 REBOILER, FIXED REBOILER VAPOR =    .36000000E+01

 TWO PRODUCT CONDENSER, FIXED REFLUX =    .75000000E+00     CONDENSER TEMP =    .70000000E+02 IF POSSIBLE

 FIXED LIQUID PRODUCT STREAM OF   .80000000E+00 MOLES FROM STAGE   3 OF COLUMN  1

                                     ***** COLUMN 1 *****
```

```
EQUILIBRIUM CONSTANTS A      EQUILIBRIUM CONSTANTS B      EQUILIBRIUM CONSTANTS C
   -.10013300E+05               .15602000E+02                 0.
   -.13921300E+05               .17523000E+02                 0.
   -.15449500E+05               .17924000E+02                 0.
   -.17012400E+05               .18673000E+02                 0.
   -.17935600E+05               .18958000E+02                 0.
   -.19155100E+05               .19509000E+02                 0.
   -.20820300E+05               .20346000E+02                 0.
   -.21993700E+05               .21064000E+02                 0.
   -.22907200E+05               .21396000E+02                 0.
   -.24669300E+05               .22365000E+02                 0.
ENTHALPY CONSTANTS K         ENTHALPY CONSTANTS L         ENTHALPY CONSTANTS U         ENTHALPY CONSTANTS W
   .12095000E+03               -.61950000E+04                .10915000E+03                .15930000E+05
   .18336000E+03               -.11165000E+05                .14616000E+03                .29940000E+05
   .21387000E+03               -.13794000E+05                .16255000E+03                .37218000E+05
   .23675000E+03               -.15808000E+05                .17373000E+03                .43110000E+05
   .25580000E+03               -.17340000E+05                .18520000E+03                .47270000E+05
   .27753000E+03               -.19278000E+05                .19583000E+03                .52870000E+05
   .29795000E+03               -.21238000E+05                .20460000E+03                .58410000E+05
   .31120000E+03               -.22372000E+05                .21100000E+03                .61870000E+05
   .32523000E+03               -.23660000E+05                .21753000E+03                .65550000E+05
   .34803000E+03               -.25970000E+05                .22613000E+03                .72080000E+05

PRODUCT ERROR LIMIT       BUBBLE DEW FLASH ERROR LIMIT
  .10000000E-01                 .10000000E-03

                                      ***** COLUMN 1 *****

    TEMPERATURES          VAPOR FLOWS         LIQUID FLOWS
   .56000000E+03        .36000000E+01        .75000000E+00
   .53000000E+03        .10000000E+01        .75000000E+00
   .51000000E+03        .10000000E+01        .75000000E+00
   .50000000E+03        .10000000E+01        .75000000E+00
   .44500000E+03        .10000000E+01        .75000000E+00
   .40500000E+03        .10000000E+01        .75000000E+00
   .30000000E+03        .10000000E+01        .75000000E+00
   .70000000E+02        .                    .75000000E+00
                                             .40000000E-01
```

```
PROBLEM NO.

ITERATION NO. = 160

                                          *****  COLUMN 1 *****

LIQUID AND VAPOR MOLE FRACTIONS LISTED AS COMPONENTS PER STAGE

       STAGE NO. =    1      (REBOILER)
LIQUID
       .29143983E-04        .23498231E-03        .16927848E-02        .46265672E-02        .22892767E-01
       .64147807E-01        .11889655E+00        .22734249E+00        .21814408E+00        .34198045E+00
VAPOR
       .13935330E-01        .14635902E-01        .36200770E-01        .46531269E-01        .12597446E+00
       .18949788E+00        .16347180E+00        .20729004E+00        .11513399E+00        .87328984E-01

       STAGE NO. =    2
LIQUID
       .62726297E-04        .99846818E-03        .11215371E-01        .25009096E-01        .94156173E-01
       .18001675E+00        .18478934E+00        .24760386E+00        .14368265E+00        .11246558E+00
VAPOR
       .19491638E-01        .34157210E-01        .12334494E+00        .12093573E+00        .23941032E+00
       .23315499E+00        .10368742E+00        .87597575E-01        .28289278E-01        .99309034E-02

       STAGE NO. =    3
LIQUID
       .17312536E-03        .22419880E-01        .12832302E+00        .12488676E+00        .24701971E+00
       .24049169E+00        .10693451E+00        .90337124E-01        .29173127E-01        .10241050E-01
VAPOR
       .23059279E-01        .23621446E+00        .38194236E+00        .14320028E+00        .13774673E+00
       .61615151E-01        .10309785E-01        .49725317E-02        .82721581E-03        .11220174E-03

       STAGE NO. =    4
LIQUID
       .44264669E-03        .22990797E+00        .39725604E+00        .14866254E+00        .14295790E+00
       .63939389E-01        .10698501E-01        .51601311E-02        .85844182E-03        .11644409E-03
VAPOR
       .22899509E-01        .65051855E+00        .27487032E+00        .34190242E-01        .14654574E-01
       .26838835E-02        .14440371E-03        .35594149E-04        .27982968E-05        .12418289E-06

       STAGE NO. =    5
LIQUID
       .89241050E-03        .66003943E+00        .28541673E+00        .35474842E-01        .15202624E-01
       .27841885E-02        .14981185E-03        .36930302E-04        .29035395E-05        .12887211E-06
VAPOR
       .26942559E-01        .88314015E+00        .86014280E-01        .32668127E-02        .59375644E-03
       .41697876E-04        .65962758E-06        .78013613E-07        .27594833E-08        .36444523E-10

       STAGE NO. =    6
LIQUID
       .34556695E-02        .90310375E+00        .89385442E-01        .33942256E-02        .61683208E-03
       .43316266E-04        .68523857E-06        .81045194E-07        .28667945E-08        .37864100E-10
VAPOR
       .84154006E-01        .89627647E+00        .19335734E-01        .21696092E-03        .16394182E-04
       .43006455E-06        .19299326E-08        .10678897E-09        .16664581E-11        .63063208E-14

       STAGE NO. =    7
LIQUID
       .53819796E-01        .92561992E+00        .20314079E-01        .22847897E-03        .17273241E-04
       .45321932E-06        .20340100E-08        .11255012E-09        .17563799E-11        .66466924E-14
VAPOR
       .65117398E+00        .34732826E+00        .14931823E-02        .44491220E-05        .13111865E-06
       .11801756E-08        .13374255E-11        .31896876E-13        .20601285E-15        .19749298E-18
```

```
        STAGE NO. =    8      (TWO PRODUCT CONDENSER)
   LIQUID
        .65128736E+00        .34721483E+00        .14932353E-02        .44486586E-05        .13110142E-06
        .11800097E-08        .13372375E-11        .31892486E-13        .20598506E-15        .19746757E-18
   VAPOR
       0.                   0.                   0.                   0.                   0.
       0.                   0.                   0.                   0.                   0.

   LIQUID PRODUCT FROM STAGE   3  OF   .80000000E+00  MOLES
      COMPONENT AMOUNTS
        .13850029E-03        .17935904E-01        .10265842E+00        .99909411E-01        .19761577E+00
        .19239335E+00        .85547605E-01        .72269699E-01        .23338502E-01        .81928402E-02
      COMPONENT RECOVERY FRACTIONS
        .27700058E-02        .35871807E+00        .10265842E+01        .99909411E+00        .98807885E+00
        .96196676E+00        .85547605E+00        .72269699E+00        .46677004E+00        .16385680E+00

   BOTTOM PRODUCT OF    .12211041E+00  MOLES
      COMPONENT AMOUNTS
        .35587836E-05        .28693785E-04        .20670664E-03        .56495201E-03        .27954451E-02
        .78331149E-02        .14518506E-01        .27760885E-01        .26637663E-01        .41759372E-01
      COMPONENT RECOVERY FRACTIONS
        .71175673E-04        .57387571E-03        .20670664E-02        .56495201E-02        .13977226E-01
        .39165574E-01        .14518506E+00        .27760885E+00        .53275326E+00        .83518744E+00

   LIQUID TOP PRODUCT OF   .76940714E-01  MOLES
      COMPONENT AMOUNTS
        .50110515E-01        .26714957E-01        .11489059E-03        .34228297E-06        .10087037E-07
        .90790786E-10        .10288801E-12        .24538307E-14        .15848638E-16        .15193296E-19
      COMPONENT RECOVERY FRACTIONS
        .10022103E+01        .53429914E+00        .11489059E-02        .34228297E-05        .50435186E-07
        .45395393E-09        .10288801E-11        .24538307E-13        .31697276E-15        .30386592E-18

   STAGE VARIABLES

         SUMX             SUMY          TEMPERATURE        VAPOR            LIQUID        VAP MOL ENTH     LIQ MOL ENTH   HEAT UNBALANCE
    .99998762E+00   .10000004E+01   .57957969E+03   .36000000E+01   .12211041E+00   .17214459E+06   .16283437E+06 0.
    .10000406E+01   .99995718E+00   .53504681E+03   .25801383E+01   .27221104E+01   .14849290E+06   .13935361E+06  .95142573E+01
    .99976973E+00   .10002616E+01   .45779813E+03   .22029448E+01   .17022565E+01   .11408093E+06   .10274133E+06 -.51878145E+02
    .99933560E+00   .10006242E+01   .38460616E+03   .22623240E+01   .21247397E+01   .90649668E+05   .70826117E+05 -.55452459E+02
    .99979575E+00   .10002113E+01   .34788034E+03   .21113147E+01   .21840736E+01   .81272706E+05   .55744242E+05  .33796717E+02
    .99999177E+00   .10000111E+01   .33411199E+03   .15070348E+01   .20339658E+01   .76806187E+05   .50776012E+05  .20366673E+02
    .10001204E+01   .99979175E+00   .29235902E+03   .82676269E+00   .14300820E+01   .56520530E+05   .41857999E+05  .14966529E+02
    .10000000E+01   .10000000E+01   .70000000E+02 0.                .75000000E+00 0.                .20610783E+04 0.
                                                                    .76940714E-01

   REBOILER LOAD =    .16087441E+06

   CONDENSER LOAD =    .45024676E+05


   RECOVERY FRACTION SUMMATIONS FOR EACH COMPONENT
        .10050515E+01        .89359108E+00        .10298002E+01        .10047471E+01        .10020561E+01
        .10011323E+01        .10006611E+01        .10003058E+01        .99952330E+00        .99904424E+00

   EXCESS MOLES OF EACH COMPONENT LEAVING THE SYSTEM IN PRODUCTS OVER MOLES FED
        .25257369E-03       -.53204458E-02        .29800159E-02        .47470560E-03        .41122424E-03
        .22646610E-03        .66110652E-04        .30583802E-04       -.23835035E-04       -.47787845E-04
```

TMASS

PROGRAM

LISTING

```
      PROGRAM MAIN(INPUT,OUTPUT,TAPE5=INPUT,TAPE6=OUTPUT)
C
C      PROGRAM TO SOLVE OIL DECOMPOSITION CONCENTRATION IN PSR
C
C                    CHANGING MASS SYSTEM
C
C
C           PROGRAMMER:  D. G. BESHORE(MMC/DEPT 0482)
C
C      DEFINE FUNCTION IN DIFFERENTIAL EQUATION
C
      INTEGER PRINT,PRNT
      NAMELIST /IN/ X1,Y1,H,EPS,MAX,NC,RATE,WSS,TMASS,PRNT
      FCT(ARG2,ARG3) = RATE + ARG2**2.*WSS/ARG3 -ARG2*(RATE+WSS/ARG3)
C
C         INPUT VALUES
C
      X1=0.0
      H=.5
      EPS=1.0E-05
      MAX=10
      PRNT=20
      NC=4000
    5 READ(5,IN)
      IF(EOF(5)) 99,70
   70 WRITE(6,IN)
      WRITE(6,100)
      X2 = X1
      Y2 = Y1
      PRINT = 0
      DO 40 N=1,NC
      X1 = X2
      Y1= Y2
      M = 0
      FN = FCT(Y1,TMASS)
      YP = Y1 + H*FN
      X2 = X1 + H
   10 FP = FCT(YP,TMASS)
      YC = Y1 + H/2.*(FN + FP)
      DELY = ABS(YC-YP)
      IF(DELY-EPS) 30,30,15
   15 IF(M-MAX) 20,20,98
   20 YP = YC
      M = M + 1
      GO TO 10
   30 Y2 = YC
      IF(PRINT) 50,50,60
   50 WRITE(6,110) X1,Y1,TMASS
      PRINT = PRNT
   60 PRINT = PRINT - 1
      TMASS = TMASS - WSS*YC
   40 CONTINUE
      GO TO 5
   99 STOP
   98 NLAST = N
      WRITE(6,113) NLAST
      WRITE(6,110) X1,Y1,TMASS
      GO TO 99
  100 FORMAT(1H1,4X,"TIME(HRS)",9X,"CONC(LB/LB)",9X,"MASS(LB)")
  110 FORMAT(1X,3E18.8)
  113 FORMAT(5X,"FAILURE TO CONVERGE FOR N =",I3)
      END
```

TMASS

INPUT

TMASS INPUT

The input to the TMASS model is in the form of NAMELIST input. The inputs to the model are listed below:

Variable	Definition	Units
X1	Initial time	Hours
H	Time step	Hours
Y1	Initial decomposition product concentration	Weight Fraction
RATE	Decomposition rate	Weight Fraction per hour
WSS	Sidestream flowrate	Lbm/HOUR
TMASS	Initial oil mass	Lbm
EPS	Error limit for numerical integration convergence criteria (1.0E-05 recommended	-
MAX	Maximum number of interactions for numerical convergence	-
PRNT	Number of time steps before a output is printed	-
NC	Maximum number of time steps performed for case	-

A sample input is listed below:

```
$IN
     Y1    =  0.00932
     RATE  =  5.7078 E-06
     WSS   =  1.348
     TMASS =  219.4
$END
```

TMASS

OUTPUT

TMASS OUTPUT

Output from execution of the TMASS model using the inputs previously described is listed on the following pages. As shown output from the program consists of an input listing and decomposition product concentration, expressed as weight fraction, and tank oil mass as functions of time.

```
$IN
X1       = 0.0,
Y1       = .932E-02,
H        = .5E+00,
EPS      = .1E-04,
MAX      = 10,
NC       = 4000,
RATE     = .57078E-05,
WSS      = .1348E+01,
TMASS    = .2194E+03,
PRNT     = 20,
$END
```

```
TIME(HRS)             CONC(LB/LB)           MASS(LB)
0.                     .93200000E-02        .21940000E+03
  .10000000E+02        .88240854E-02        .21915582E+03
  .20000000E+02        .83567067E-02        .21892460E+03
  .30000000E+02        .79162998E-02        .21870559E+03
  .40000000E+02        .75013778E-02        .21849810E+03
  .50000000E+02        .71105277E-02        .21830145E+03
  .60000000E+02        .67424082E-02        .21811501E+03
  .70000000E+02        .63957462E-02        .21793818E+03
  .80000000E+02        .60693346E-02        .21777042E+03
  .90000000E+02        .57620293E-02        .21761118E+03
  .10000000E+03        .54727464E-02        .21745997E+03
  .11000000E+03        .52004596E-02        .21731632E+03
  .12000000E+03        .49441975E-02        .21717977E+03
  .13000000E+03        .47030412E-02        .21704992E+03
  .14000000E+03        .44761219E-02        .21692637E+03
  .15000000E+03        .42626181E-02        .21680875E+03
  .16000000E+03        .40617537E-02        .21669670E+03
  .17000000E+03        .38727954E-02        .21658989E+03
  .18000000E+03        .36950510E-02        .21648802E+03
  .19000000E+03        .35278567E-02        .21639079E+03
  .20000000E+03        .33706257E-02        .21629793E+03
  .21000000E+03        .32227459E-02        .21620917E+03
  .22000000E+03        .30836784E-02        .21612427E+03
  .23000000E+03        .29529052E-02        .21604301E+03
  .24000000E+03        .28299383E-02        .21596515E+03
  .25000000E+03        .27143173E-02        .21589051E+03
  .26000000E+03        .26056086E-02        .21581889E+03
  .27000000E+03        .25034033E-02        .21575010E+03
  .28000000E+03        .24073164E-02        .21568398E+03
  .29000000E+03        .23169851E-02        .21562037E+03
  .30000000E+03        .22320678E-02        .21555912E+03
  .31000000E+03        .21522426E-02        .21550008E+03
  .32000000E+03        .20772067E-02        .21544313E+03
  .33000000E+03        .20066748E-02        .21538814E+03
  .34000000E+03        .19403784E-02        .21533499E+03
  .35000000E+03        .18780648E-02        .21528356E+03
  .36000000E+03        .18194960E-02        .21523377E+03
  .37000000E+03        .17644483E-02        .21518550E+03
  .38000000E+03        .17127111E-02        .21513867E+03
  .39000000E+03        .16640861E-02        .21509319E+03
  .40000000E+03        .16183869E-02        .21504898E+03
  .41000000E+03        .15754382E-02        .21500596E+03
  .42000000E+03        .15350750E-02        .21496407E+03
  .43000000E+03        .14971420E-02        .21492322E+03
  .44000000E+03        .14614933E-02        .21488337E+03
  .45000000E+03        .14279918E-02        .21484445E+03
  .46000000E+03        .13965082E-02        .21480640E+03
  .47000000E+03        .13669213E-02        .21476917E+03
  .48000000E+03        .13391169E-02        .21473272E+03
  .49000000E+03        .13129877E-02        .21469699E+03
  .50000000E+03        .12884327E-02        .21466194E+03
  .51000000E+03        .12653572E-02        .21462753E+03
  .52000000E+03        .12436720E-02        .21459373E+03
  .53000000E+03        .12232933E-02        .21456049E+03
  .54000000E+03        .12041421E-02        .21452778E+03
  .55000000E+03        .11861445E-02        .21449558E+03
  .56000000E+03        .11692309E-02        .21446384E+03
  .57000000E+03        .11533357E-02        .21443255E+03
  .58000000E+03        .11383975E-02        .21440167E+03
  .59000000E+03        .11243585E-02        .21437118E+03
  .60000000E+03        .11111642E-02        .21434105E+03
  .61000000E+03        .10987638E-02        .21431127E+03
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.64000000E+03      .10653592E-02      .21422380E+03
.65000000E+03      .10561818E-02      .21419520E+03
.66000000E+03      .10470855E-02      .21416686E+03
.67000000E+03      .10385350E-02      .21413875E+03
.68000000E+03      .10304973E-02      .21411086E+03
.69000000E+03      .10229413E-02      .21408319E+03
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.71000000E+03      .10091596E-02      .21402842E+03
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.74000000E+03      .99142569E-03      .21394755E+03
.75000000E+03      .98620545E-03      .21392090E+03
.76000000E+03      .98129621E-03      .21389438E+03
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.80000000E+03      .96440859E-03      .21378952E+03
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.82000000E+03      .95739139E-03      .21373771E+03
.83000000E+03      .95419026E-03      .21371195E+03
.84000000E+03      .95117760E-03      .21368627E+03
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.87000000E+03      .94315942E-03      .21360967E+03
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.90000000E+03      .93646490E-03      .21353367E+03
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.94000000E+03      .92921140E-03      .21343310E+03
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.96000000E+03      .92617796E-03      .21338308E+03
.97000000E+03      .92478907E-03      .21335813E+03
.98000000E+03      .92347853E-03      .21333322E+03
.99000000E+03      .92224157E-03      .21330834E+03
.10000000E+04      .92107371E-03      .21328349E+03
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.10300000E+04      .91794398E-03      .21320913E+03
.10400000E+04      .91701297E-03      .21318439E+03
.10500000E+04      .91613244E-03      .21315968E+03
.10600000E+04      .91529933E-03      .21313499E+03
.10700000E+04      .91451074E-03      .21311033E+03
.10800000E+04      .91376396E-03      .21308568E+03
.10900000E+04      .91305645E-03      .21306106E+03
.11000000E+04      .91238582E-03      .21303645E+03
.11100000E+04      .91174982E-03      .21301186E+03
.11200000E+04      .91114633E-03      .21298729E+03
.11300000E+04      .91057339E-03      .21296274E+03
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.12000000E+04      .90727500E-03      .21279122E+03
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.12300000E+04      .90616519E-03      .21271789E+03
.12400000E+04      .90582717E-03      .21269346E+03
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.13500000E+04       .90288804E-03       .21242530E+03
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.13800000E+04       .90226817E-03       .21235230E+03
.13900000E+04       .90207397E-03       .21232798E+03
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.16200000E+04       .89863526E-03       .21176977E+03
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.16800000E+04       .89791532E-03       .21162446E+03
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.18200000E+04       .89634616E-03       .21128585E+03
.18300000E+04       .89623788E-03       .21126169E+03
.18400000E+04       .89612996E-03       .21123753E+03
.18500000E+04       .89602237E-03       .21121337E+03
.18600000E+04       .89591510E-03       .21118922E+03
.18700000E+04       .89580813E-03       .21116506E+03
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.19100000E+04      .89538286E-03      .21106848E+03
.19200000E+04      .89527713E-03      .21104435E+03
.19300000E+04      .89517160E-03      .21102021E+03
.19400000E+04      .89506627E-03      .21099608E+03
.19500000E+04      .89496112E-03      .21097195E+03
.19600000E+04      .89485615E-03      .21094782E+03
.19700000E+04      .89475134E-03      .21092370E+03
.19800000E+04      .89464668E-03      .21089958E+03
.19900000E+04      .89454216E-03      .21087546E+03
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APPENDIX D

CHEMICAL DATA

APPENDIX D: CHEMICAL DATA

Oil samples were taken from the storage tank condensate trap, and processor product streams throughout the six week test periods of Phase I and Phase II. Chemical analyses of these samples were subsequently performed to determine changes in physical and chemical properties of the oil with time at the 600°F operating temperature. Shown in Tables D-1 and D-2 are the lists of chemical analyses performed on the oil samples. Hand valve locations described as "HV-" correspond with those illustrated in Appendix A. Sandia conducted the viscosity, GPC, and GC distillation tests. Martin Marietta was responsible for the other analyses shown in Tables D-1 and D-2. The analyses for chloride and iron were not completed on all samples when only traces of these elements were detected in the first few samples. The unreduced data from these analyses are included in the following pages of this appendix. Also included in this section are the analyses performed on the "wax-like" residue that was formed in the system during Phase II tests and the independently performed tests conducted by Exxon on Phase I oil samples.

TABLE D-1
OIL EVALUATION PROGRAM SAMPLE ANALYSIS

SAMPLE NUMBER	SOURCE	DATE	TEST PROGRAM STATUS	ANALYSIS STATUS													REMARKS
				MOLECULAR WT. DISTRIBUTION BY GPC	VISCOSITY	GC DISTILLATION		CHLORIDE	IRON	ACIDS	IR	NMR	MOLECULAR WT. DISTRIBUTION BY GC-MS	DENSITY			
A-1	Barrel A	1/18/78				X					X	X					
B-1	Barrel B	1/18/78				X			X		X	X					
C-1	Barrel C	1/18/78		X	X	X		X	X	X	X	X		X			
A-2	Barrel A	1/26/78															Entire sample 5 gal. sent to Quest Inc.
1	HV-15	1/25/78															Entire sample held for storage, insufficient for oil analysis. Retake of sample of oil at temperature.
2	HV-15	1/26/78		X				X	X	X	X	X		X			
3	HV-15	2/1/78		X	X			X	X	X	X	X		X			
4	HV-15	2/8/78		X	X			X	X	X	X	X	X	X			
5	HV-15	2/15/78		X	X	X			X	X	X	X	X	X			
6	HV-15	2/16/78				X											Sample taken for storage after pressure loss on test fixture.
7	HV-15	2/22/78		X	X	X			X	X	X	X	X	X			
8	HV-15	2/28/78		X	X	X			X	X	X	X		X			
9	HV-29	2/28/78															Gas sample from vent down tank at 28 psig.
10	HV-29	2/28/78											X				Gas sample from vent down tank at 16 psig.
11	HV-15	2/28/78		X	X	X				X	X	X	X	X			
12	HV-19	2/28/78		X						X	X	X	X	X			Ice water condensables from vent down.
13	HV-15	3/8/78		X	X	X			X	X	X	X		X			
14	HV-29	3/8/78											X				Gas sample from vent down 38 psig.
15	HV-19	3/8/78		X						X	X	X	X	X			Ice water condensables from vent down.
16	HV-29	3/8/78															Gas sample from vent down 6 psig.
17	HV-19	3/8/78		X						X	X	X	X	X			Ice water condensables from vent down.
18	HV-15	3/8/78		X	X	X				X	X	X		X			
19	HV-15	3/13/78				X			X	X	X	X		X			Taken when oil cooled to 121°F-end of Ph. 1
20	HV-19	3/13/78								X	X	X	X	X			From an uncontrolled vent 3/8-13/78. Caught in condenser.

TABLE D-2
PHASE II OIL EVALUATION PROGRAM SAMPLE ANALYSIS

SAMPLE NUMBER	SOURCE	DATE	TEST PROGRAM STATUS	ANALYSIS STATUS									REMARKS
				MOLECULAR WT. BY GPC	VISCOSITY	GC DISTILLATION	IR	NMR	MOLECULAR WT. DISTRIBUTION BY GC - MS	DENSITY			
21	HV-49	5/11/78					X	X		X			
22	HV100	5/11/78					X	X					
23	HV-15	5/11/78				X	X	X		X			
24	HV-100	5/11/78					X	X		X			
25	HV-15	5/18/78				X	X	X		X			
26	HV-15	5/26/78				X	X	X		X			
27	HV-15	6/1/78		X	X	X	X	X		X			
28	HV-100	6/1/78				X	X	X		X			
29	HV-15	6/8/78		X	X	X	X	X		X			
30	HV-49	6/8/78				X	X	X		X			
31	HV-103	6/8/78				X	X	X	X	X			
32	HV-100	6/8/78				X	X	X		X			
33	HV-103	6/15/78				X	X	X		X			
34	HV-100	6/15/78				X	X	X		X			
35	HV-49	6/15/78				X	X	X		X			
36	HV-15	6/15/78		X	X	X	X	X		X			
37	HV-15	6/22/78		X	X	X	X	X		X			
38	HV-49	6/22/78				X	X	X		X			
39	HV-100	6/22/78				X	X	X		X			
40	HV-103	6/22/78				X	X	X		X			
41	HV-15	6/29/78		X	X	X	X	X		X			
42	HV-49	6/29/78				X	X	X		X			
43	HV-100	6/29/78				X	X	X		X			
44	HV-103	6/29/78				X	X	X	X	X			
45	HV-15	7/6/78		X	X	X	X	X		X			
46	HV-49	7/6/78				X	X	X		X			
47	HV-100	7/6/78				X	X	X		X			
48	HV-103	7/6/78				X	X	X		X			
49	HV-15	7/14/78		X	X	X	X	X		X			
50	HV-49	7/14/78				X	X	X		X			
51	HV-100	7/14/78				X	X	X		X			

Density Measurements

PHASE I & II DENSITY DETERMINATION SUMMARY

Sample Number	Density at 21 ± 1°C (g/ml)
A-1	Not done
B-1	Not done
C-1	0.851
1	Not done
2	0.861
3	0.851 ± 0.002 (Average of 3)
4	0.849
5	0.852
6	Not done
7	0.850
8	0.850
9	Vent gas
10	Vent gas
11	0.853
12	0.752
13	0.854
14	Vent gas
15	0.762
16	Vent gas
17	0.782
18	0.851
19	0.856
20	0.764
21	0.829
22	Not done
23	0.849
24	0.847
25	0.847

PHASE I & II DENSITY DETERMINATION SUMMARY (CONTINUED)

Sample Number	Density at 21 ± 1°C (g/ml)
26	0.850
27	0.846
28	0.861
29	0.841
30	0.842
31	0.822
32	0.866
33	0.818
34	0.861
35	0.847
36	0.846
37	0.844
38	0.842
39	0.862
40	0.819
41	0.843
42	0.838
43	0.860
44	0.818
45	0.842
46	0.841
47	0.851

Viscosity Tests

PHASE I VISCOSITY MEASUREMENTS

TEMPERATURE:	21°C
VISCOMETER:	Cannon-Ubbelohde #2
METHOD:	ASTM D445
VISCOMETER CONSTANT:	.0902
DATE:	22 February 1978
INVESTIGATOR:	V. Burolla, Sandia Labs - Livermore

Sample Number	Time (Seconds)	Viscosity (CSt)
C-1	689.2	61.8
#2	664.6 669.7	60.2
#3	608.7 579.4	53.6
#4	520.7 525.0	47.2
#5	429.5 438.8	39.2

PHASE I VISCOSITY MEASUREMENTS

TEMPERATURE:	19°C
VISCOMETER:	Cannon-Ubbelohde #2
METHOD:	ASTM D445
VISCOMETER CONSTANT:	.0902
DATE:	15 March 78
INVESTIGATOR:	V. Burolla, Sandia Labs - Livermore

Sample Number	Time (Seconds)	Viscosity (CSt)
#7	480.9	43.3
	482.9	43.6
#8	420.6	NG
	436.0	NG
	444.9	40.1
	446.7	40.3
#11	510.9	46.1
	508.6	45.9
#13	444.7	40.1
	441.2	39.8
#18	545.0	NG
	549.9	49.6
	550.7	49.7

PHASE II VISCOSITY MEASUREMENTS

TEMPERATURE:	$21^{\circ} - 24^{\circ}$ C
VISCOMETER:	Cannon-Ubbelohde #2
METHOD:	ASTM D445
VISCOMETER CONSTANT:	0.0902
INVESTIGATOR:	V. Burolla, Sandia Labs - Livermore

Sample Number	Viscosity (Centistokes)	Sample Number	Viscosity (Centistokes)
#23	73.5	#44	4.97
#25	56.1	#45	31.0
#26	51.6	#46	29.6
#27	49.5	#47	85.2
#29	46.6	#48	4.82
#36	38.4	#49	26.8
#37	35.3	#50	28.3
#41	36.6	#51	88.5
#42	34.0	#52	3.34
#43	147.7		

Chlorine, Iron, and Acids Tests

ANALYSES OF CHLORINE, IRON, AND ACIDS

	ANALYTE CONCENTRATION			
	CHLORINE	IRON	ACIDS	
SAMPLE NUMBER	µg Cl/g Sample	µg Fe/g Sample	µg KOH/g Sample	µg Myristic Acid/g Sample
A-1	Test deleted	Test deleted	Test deleted	
B-1	Test deleted	2.3	Test deleted	
C-1	16.7	1.8	0.2	0.8
1	Sample held for storage			
2	17.7	2.3	1.0	3.2
3	11.6	2.0	<0.2	0.8
4	9.0	1.4	<0.5	1.6
5	Test deleted	1.7	<2.0	6.5
6	Sample held for storage			
7	Test deleted	2.3	4.2	13.7
8	Test deleted	2.6	<4.3	<14.2
11	Test deleted	Test deleted	<4.4	14.3
12	Test deleted	Test deleted	4.3	14.3
13	Test deleted	2.1	<4.4	14.6
15	Test deleted	Test deleted	<4.0	13.1
17	Test deleted	Test deleted	4.2	13.7
18	Test deleted	Test deleted	3.6	11.9
19	Test deleted	1.9	<4.3	<14.0
20	Test deleted	Test deleted	4.2	13.8

GAS CHROMATOGRAPH

DISTILLATION TESTS

GAS CHROMATOGRAPH DISTILLATION TESTS

The GC distillation tests were independently performed by two laboratories. The results of the tests performed by Lawrence Livermore Labs is presented first followed by tests conducted by West Coast Technical Service, Inc.

Sandia Laboratories Data
Victor Burolla

SAMPLE #A-1

Interval	% Area	Boiling Point°C
1	0.01	154
2	0.02	161
3	0.05	183
4	0.09	200
5	0.16	216
6	0.23	233
7	0.31	249
8	0.48	265
9	0.67	282
10	1.4	298
11	2.68	315
12	4.53	331
13	6.96	347
14	10.12	364
15	14.22	380
16	19.81	397
17	27.77	413
18	38.95	430
19	53.62	446
20	69.56	462
21	83.08	479
22	91.52	495
23	95.71	512
24	97.86	528
25	98.96	545
26	99.5	562
27	99.7	578
28	100	595

SAMPLE #B-1

Interval	% Area	Boiling Point°C
1	0.01	233
2	0.09	249
3	0.3	265
4	0.79	282
5	1.82	298
6	3.61	315
7	5.91	331
8	8.91	347
9	12.87	364
10	18.22	380
11	25.79	397
12	36.64	413
13	50.88	430
14	66.86	446
15	80.96	462
16	90.58	479
17	95.53	495
18	98.05	512
19	99.27	528
20	99.74	545
21	100	561

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SAMPLE #C-1

Interval	% Area	Boiling Point°C
1	0.02	233
2	0.1	249
3	0.31	265
4	0.82	282
5	1.89	298
6	3.73	315
7	6.05	331
8	9.13	347
9	13.18	364
10	18.73	380
11	26.56	397
12	37.77	413
13	52.35	430
14	68.45	446
15	82.51	462
16	91.61	479
17	96.23	495
18	98.41	512
19	99.33	528
20	99.53	545

SAMPLE #1

Interval	% Area	Boiling Point°C
1	0.04	134
2	0.16	150
3	0.81	167
4	1.98	183
5	3.6	200
6	5.72	216
7	7.98	233
8	10.65	249
9	13.45	265
10	16.58	282
11	20.17	298
12	24.12	315
13	28.39	331
14	33.11	347
15	38.31	364
16	44.35	380
17	51.74	397
18	60.86	413
19	71.7	430
20	82.58	446
21	91.28	462
22	96.65	479
23	99.02	495
24	99.87	512

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SAMPLE #5

Interval	% Area	Boiling Point °C
1	0.77	97.3
2	1.55	110.7
3	2.46	124.1
4	3.55	137.5
5	4.69	150.9
6	5.86	164.3
7	7.1	177.7
8	8.37	191.1
9	9.68	204.5
10	11.05	217.9
11	12.48	231.3
12	14.03	244.8
13	15.78	258.2
14	17.66	271.6
15	19.89	285
16	22.57	298.4
17	25.93	311.8
18	30.28	325.2
19	36.21	338.6
20	44.11	352
21	53.79	365.4
22	63.78	378.8
23	72.31	392.3
24	78.4	405.7
25	82.74	419.1
26	86.13	432.5
27	88.97	445.9
28	91.39	459.3
29	93.52	472.7
30	95.38	486.1
31	97	499.5
32	98.42	512.9
33	99.67	526.3

SAMPLE #6

Interval	% Area	Boiling Point °C
1	0.57	150.9
2	1.19	164.3
3	1.83	177.7
4	2.52	191.1
5	3.2	204.5
6	4.06	217.9
7	4.97	231.3
8	6.15	244.8
9	7.77	258.2
10	10.23	271.6
11	13.05	285.2
12	17	298.4
13	21.68	311.8
14	27.47	325.2
15	35.83	338.6
16	47.47	352
17	58.86	365.4
18	70.09	378.8
19	78.58	392.3
20	83.87	405.7
21	87.4	419.1
22	90.16	432.5
23	92.3	445.9
24	94.02	459.3
25	95.48	472.7
26	96.72	486.1
27	97.79	499.5
28	98.7	512.9
29	99.48	526.3
30	100	539.8

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SAMPLE #7

Interval	% Area	Boiling Point °C
1	0.7	124.1
2	1.47	137.5
3	2.25	150.9
4	3.06	164.3
5	3.95	177.7
6	4.92	191.1
7	5.96	204.5
8	7.06	217.9
9	8.29	231.3
10	9.74	244.8
11	11.6	258.2
12	13.82	271.6
13	16.49	285
14	19.76	298.4
15	23.78	311.8
16	28.84	325.2
17	35.66	338.6
18	44.65	352
19	55.56	365.4
20	66.6	378.8
21	75.64	392.3
22	81.61	405.7
23	85.65	419.1
24	88.62	432.5
25	90.98	445.9
26	92.93	459.3
27	94.66	472.7
28	96.05	486.1
29	97.27	499.5
30	98.35	512.9
31	99.32	526.3
32	100	539.8

SAMPLE #8

Interval	% Area	Boiling Point °C
1	0.97	110.7
2	2	124.1
3	3.1	137.5
4	4.24	150.9
5	5.41	164.3
6	6.65	177.7
7	7.95	191.1
8	9.29	204.5
9	10.72	217.9
10	12.19	231.3
11	13.66	244.8
12	15.43	258.2
13	17.6	271.6
14	20.11	285
15	23.16	298.4
16	26.83	311.8
17	31.46	325.2
18	37.51	338.6
19	45.49	352
20	55.32	365.4
21	65.61	378.8
22	74.53	392.3
23	80.86	405.7
24	85.09	419.1
25	88.16	432.5
26	90.52	445.9
27	92.44	459.3
28	94.04	472.7
29	95.38	486.1
30	96.55	499.5
31	97.55	512.9
32	98.42	526.3
33	99.17	539.8
34	99.81	553.2

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SAMPLE #11

Interval	% Area	Boiling Point °C
1	0.85	150.9
2	1.77	164.3
3	2.81	177.7
4	3.93	191.1
5	5.15	204.5
6	6.49	217.9
7	7.94	231.3
8	9.59	244.8
9	11.62	258.2
10	14.12	271.6
11	16.99	285
12	20.37	298.4
13	24.43	311.8
14	29.45	325.2
15	35.89	338.6
16	44.43	352
17	55.05	365.4
18	66.17	378.8
19	75.46	392.3
20	81.44	405.7
21	85.89	419.1
22	88.75	432.5
23	90.93	445.9
24	92.66	459.3
25	94.08	472.7
26	95.29	486.1
27	96.32	499.5
28	97.24	512.9
29	98.12	526.3
30	99.12	539.8
31	99.82	553.2

SAMPLE #13

Interval	% Area	Boiling Point °C
1	1.9	97.3
2	3.01	110.7
3	4.23	124.1
4	5.46	137.5
5	6.71	150.9
6	8.01	164.3
7	9.39	177.7
8	10.81	191.1
9	12.3	204.5
10	13.88	217.9
11	15.52	231.3
12	17.38	244.8
13	19.66	258.2
14	22.19	271.6
15	25.03	285
16	28.29	298.4
17	32.13	311.8
18	36.88	325.2
19	42.83	338.6
20	50.23	352
21	58.92	365.4
22	67.66	378.8
23	75.12	392.3
24	80.37	405.7
25	84.1	419.1
26	86.95	432.5
27	89.29	445.9
28	91.27	459.3
29	93.05	472.7
30	94.66	486.1
31	95.99	499.5
32	97.11	512.9
33	98.09	526.3
34	98.97	539.8
35	99.75	553.2

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SAMPLE #18

Interval	% Area	Boiling Point °C
1	1.31	97.3
2	2.07	110.7
3	2.9	124.1
4	3.79	137.5
5	4.76	150.9
6	5.78	164.3
7	6.87	177.7
8	8.05	191.1
9	9.35	204.5
10	10.75	217.9
11	12.27	231.3
12	13.97	244.8
13	15.88	258.2
14	18.26	271.6
15	21.02	285
16	24.24	298.4
17	28.03	311.8
18	32.55	325.2
19	38.09	338.6
20	45.3	352
21	54.38	365.4
22	64.55	378.8
23	74.07	392.3
24	81.35	405.7
25	86.01	419.1
26	89.16	432.5
27	91.46	445.9
28	93.22	459.3
29	94.63	472.7
30	95.8	486.1
31	96.88	499.5
32	97.8	512.9
33	98.61	526.3
34	99.3	539.8
35	99.87	553.2

SAMPLE #19

Interval	% Area	Boiling Point °C
1	1.04	110.7
2	2	124
3	2.94	137.5
4	3.87	150.9
5	4.81	164.3
6	5.81	177.7
7	6.86	191.1
8	8	204.5
9	9.19	217.9
10	10.48	231.3
11	12.02	244.8
12	14.04	258.2
13	16.55	271.6
14	19.55	285
15	23.27	298.4
16	27.85	311.8
17	33.69	325.2
18	41.51	338.6
19	51.58	352
20	63.63	365.4
21	75.05	378.8
22	84.55	392.3
23	89.85	405.7
24	92.85	419.1
25	94.71	432.5
26	95.98	445.9
27	96.97	459.3
28	97.99	472.7
29	98.44	486.1
30	98.99	499.5
31	99.43	512.9
32	99.75	526.3
33	99.96	539.8
34	100	553.2

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SAMPLE #23

Interval	% Area	Boiling Point °C
1	0.03	134
2	0.06	167
3	0.09	216
4	0.11	233
5	0.22	249
6	0.4	265
7	0.83	282
8	1.73	298
9	3.23	315
10	5.53	331
11	8.43	347
12	12.13	364
13	17.13	380
14	23.93	397
15	33.47	413
16	46.21	430
17	61.49	446
18	76.99	462
19	88.69	479
20	95.06	495
21	98.24	512
22	99.74	528
23	100	545

SAMPLE #25

Interval	% Area	Boiling Point °C
1	0.08	134
2	0.18	150
3	0.33	169
4	0.47	185
5	0.62	200
6	0.82	216
7	1.07	233
8	1.35	249
9	1.74	265
10	2.36	282
11	3.35	298
12	4.97	315
13	7.26	331
14	10.29	347
15	14.08	364
16	19.05	380
17	25.92	397
18	35.21	413
19	47.16	430
20	61.37	446
21	75.45	462
22	86.66	479
23	93.37	495
24	96.58	512
25	98.45	528
26	99.68	545
27	100	561

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SAMPLE #26

Interval	% Area	Boiling Point °C
1	0.06	128
2	0.18	145
3	0.35	161
4	0.56	178
5	0.83	194
6	1.19	211
7	1.59	227
8	2.08	243
9	2.7	260
10	3.52	276
11	4.77	293
12	6.69	309
13	9.28	326
14	12.59	342
15	16.69	368
16	21.79	375
17	28.59	391
18	37.69	408
19	49.63	424
20	63.61	441
21	77.67	457
22	89.17	473
23	95.66	490
24	98.82	506
25	99.93	523

SAMPLE #27

Interval	% Area	Boiling Point °C
1	0.03	134
2	0.05	150
3	0.15	167
4	0.31	183
5	0.52	200
6	0.8	216
7	1.11	233
8	1.52	249
9	2.06	265
10	2.74	282
11	3.78	298
12	5.51	315
13	8.11	331
14	11.25	347
15	15.38	364
16	20.4	386
17	27.17	397
18	36.34	413
19	48.54	430
20	63.05	446
21	77.87	462
22	89.59	479
23	96.23	495
24	99.06	512
25	99.89	528
26	100	539

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SAMPLE #28

Interval	% Area	Boiling Point °C
1	0.08	150
2	0.27	167
3	0.63	183
4	1.09	200
5	1.67	216
6	2.29	233
7	3.1	249
8	4.07	265
9	5.17	282
10	6.63	298
11	8.73	315
12	11.84	331
13	15.48	347
14	19.96	364
15	25.45	380
16	32.54	397
17	41.8	413
18	53.92	430
19	67.96	446
20	82.13	462
21	92.95	479
22	98.47	495
23	100	512

SAMPLE #29

Interval	% Area	Boiling Point °C
1	0.02	128
2	0.05	150
3	0.15	167
4	0.25	183
5	0.38	200
6	0.55	216
7	0.78	233
8	1.05	249
9	1.37	265
10	1.75	282
11	2.64	298
12	5.74	315
13	10.08	331
14	15.4	347
15	21.69	364
16	29.58	386
17	39.55	397
18	52.52	413
19	68.24	430
20	85.27	446
21	97.08	462
22	99.57	479
23	100	495

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SAMPLE #41

Interval	% Area	Boiling Point °C
1	0.04	175
2	0.07	190
3	0.12	208
4	0.25	224
5	0.32	240
6	0.45	256
7	0.56	272
8	0.73	288
9	1.16	304
10	4.21	316
11	8.67	332
12	14.51	350
13	21.8	370
14	31.49	386
15	42.68	402
16	56.72	416
17	72.88	435
18	89.72	446
19	96.98	466
20	97.98	480
21	98.51	495
22	98.93	505
23	99.29	515
24	99.59	524
25	99.85	533
26	100	543

SAMPLE #42

Interval	% Area	Boiling Point °C
1	0.02	196
2	0.17	220
3	0.19	234
4	0.37	256
5	0.45	272
6	0.75	288
7	0.9	304
8	1.45	316
9	4.23	332
10	12.5	350
11	23.88	370
12	37.44	390
13	54.9	404
14	79.44	421
15	95.62	438
16	99.04	452
17	99.67	468
18	99.83	481
19	99.9	488

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SAMPLE #43

Interval	% Area	Boiling Point °C
1	0.09	210
2	0.12	228
3	0.65	275
4	0.67	292
5	0.81	308
6	0.94	326
7	1.15	340
8	1.49	356
9	2.02	370
10	2.89	390
11	4.41	404
12	7.14	421
13	12.96	438
14	34.99	452
15	76.35	468
16	86.63	483
17	91.23	495
18	94.03	508
19	95.97	516
20	97.33	525
21	98.29	534
22	98.96	546
23	99.43	554
24	99.7	564
25	100	575

SAMPLE #44

Interval	% Area	Boiling Point °C
1	0.09	132
2	0.52	148
3	1.58	164
4	3.97	181
5	7.76	196
6	13.84	210
7	21.06	228
8	29.14	244
9	39.32	260
10	50.55	275
11	64.16	292
12	80.79	308
13	89.8	326
14	89.8	340
15	99.75	356
16	99.9	370
17	100	390

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SAMPLE #45

Interval	% Area	Boiling Point °C
1	0.13	288
2	1.33	304
3	5.67	316
4	12.03	332
5	19.41	350
6	28.45	370
7	39.12	391
8	52.15	404
9	68.67	421
10	84.22	438
11	91.56	452
12	95.03	468
13	97.16	482
14	98.13	495
15	98.85	508
16	99.13	516
17	99.4	525
18	100	542

SAMPLE #46

Interval	% Area	Boiling Point °C
1	0.01	288
20.	0.88	304
3	5.6	316
4	12.18	332
5	20.36	350
6	29.88	370
7	41.18	385
8	55.05	402
9	71.95	416
10	89.94	435
11	99.09	446
12	99.89	466
13	99.92	502

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SAMPLE #47

Interval	% Area	Boiling Point °C
1	0.02	265
2	0.04	282
3	0.07	296
4	0.16	316
5	0.35	330
6	0.66	350
7	1.18	363
8	2.02	382
9	3.41	394
10	6.17	414
11	13.49	428
12	44.41	444
13	92.52	459
14	98.98	476
15	99.27	488
16	99.56	502
17	99.86	512
18	100	522
19		

SAMPLE #48

Interval	% Area	Boiling Point °C
1	0.26	148
2	0.9	164
3	2.61	181
4	5.73	200
5	11.2	211
6	18.15	227
7	25.8	241
8	35.3	256
9	45.86	272
10	58.85	288
11	78.58	304
12	88.64	316
13	96.44	332
14	99.42	350
15	99.55	370
16	99.57	390
17	99.61	404
18	99.7	421
19	99.8	438
20	99.9	452

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SAMPLE #49

Interval	% Area	Boiling Point °C
1	0.01	181
2	0.02	200
3	0.05	211
4	0.08	227
5	0.14	241
6	0.21	256
7	0.29	272
8	0.44	288
9	1.75	304
10	6.89	316
11	13.94	332
12	22.77	350
13	32.8	370
14	43.95	385
15	58.82	402
16	76.86	416
17	93.4	435
18	98.14	446
19	98.66	466
20	99.28	480
21	99.9	495

SAMPLE #50

Interval	% Area	Boiling Point °C
1	0.99	304
2	6.18	316
3	13.64	332
4	22.96	350
5	33.77	370
6	46.69	385
7	62.34	402
8	80.85	416
9	95.93	435
10	99.6	446
11	99.76	466

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SAMPLE #51

Interval	% Area	Boiling Point °C
1	0.06	316
2	0.23	330
3	0.51	350
4	0.96	363
5	3.2	394
6	6.49	414
7	17.49	428
8	58.82	444
9	93.17	459
10	97.56	476
11	98.62	488
12	99.2	502
13	99.56	512
14	99.86	522
15	99.98	532
16	100	543

SAMPLE #52

Interval	% Area	Boiling Point °C
1	0.06	132
2	0.37	148
3	1.17	166
4	3.46	181
5	8.35	200
6	17.22	211
7	28.58	227
8	42.83	245
9	59.88	262
10	78.12	276
11	95.42	293
12	99.2	307
13	99.45	323
14	99.55	342
15	99.62	359

	Sample #A-1			Sample #C-1	
Interval	% Area	Boiling Point °C	Interval	% Area	Boiling Point °C
1	0.01	253	1	0.01	253
2	0.05	271	2	0.07	271
3	0.12	287	3	0.24	287
4	0.34	302	4	0.81	302
5	1.11	317	5	1.75	317
6	2.26	331	6	3.08	331
7	3.77	344	7	4.72	344
8	5.55	356	8	6.61	356
9	7.84	369	9	8.98	369
10	10.66	380	10	11.83	380
11	14.07	391	11	15.24	391
12	18.37	402	12	19.48	402
13	23.75	412	13	26.67	412
14	32.69	422	14	33.65	422
15	41.35	432	15	42.18	432
16	48.62	441	16	49.21	441
17	57.40	450	17	57.54	450
18	65.16	459	18	65.18	459
19	72.28	468	19	72.45	468
20	80.01	476	20	76.17	476
21	84.66	483	21	79.70	483
22	91.53	491	22	84.30	491
23	94.97	498	23	90.81	498
24	96.99	505	24	94.75	505
25	98.27	5012	25	97.25	512
26	99.12	518	26	98.76	518
27	99.61	525	27	99.54	525
28	99.85	531	28	99.89	531
29	99.95	537	29	99.97	537
30	99.98	543	30	100.00	543
31	100.00	548			

Sample #30

Interval	% Area	Boiling Point °C
1	0.01	271
2	0.27	287
3	1.57	302
4	3.25	317
5	6.97	331
6	11.23	344
7	15.79	356
8	21.08	369
9	27.22	380
10	34.18	391
11	42.02	402
12	49.87	412
13	61.19	422
14	72.72	432
15	78.41	441
16	86.77	450
17	93.68	459
18	99.97	468
19	100.00	476

Sample #31

Interval	% Area	Boiling Point °C
1	0.06	69
2	0.14	98
3	0.29	126
4	1.38	151
5	4.84	174
6	9.40	196
7	16.28	216
8	21.48	235
9	30.92	253
10	40.08	271
11	51.84	287
12	67.86	302
13	74.73	317
14	80.67	331
15	84.79	344
16	88.79	356
17	92.98	369
18	96.18	380
19	97.78	391
20	98.11	402
21	98.68	412
22	99.16	422
23	100.00	432

Sample #32

Interval	% Area	Boiling Point °C
1	0.01	287
2	0.03	302
3	0.06	307
4	0.08	331
5	0.11	344
6	0.14	356
7	0.22	369
8	0.39	380
9	0.67	391
10	1.07	402
11	1.71	412
12	2.62	422
13	3.78	432
14	5.32	441
15	17.59	450
16	30.43	459
17	39.59	468
18	44.12	476
19	51.46	483
20	60.62	491
21	68.63	498
22	74.97	505
23	79.73	512
24	87.83	518
25	92.91	525
26	96.11	531
27	97.91	537
28	98.84	543
29	99.36	548
30	99.64	553
31	99.78	558
32	99.85	563
33	99.90	567
34	99.94	571
35	100.00	575

Sample #33

Interval	% Area	Boiling Point °C
1	0.01	69
2	0.04	98
3	0.24	126
4	1.68	151
5	6.61	174
6	13.19	196
7	21.28	216
8	31.39	235
9	43.99	253
10	55.67	271
11	69.62	287
12	86.85	302
13	90.59	317
14	92.58	331
15	93.99	344
16	95.32	356
17	96.79	369
18	98.32	380
19	99.28	391
20	99.52	402
21	99.68	412
22	99.84	422
23	100.00	432

Sample #34

Interval	% Area	Boiling Point °C
1	0.01	287
2	0.01	302
3	0.05	317
4	0.07	331
5	0.11	344
6	0.12	356
7	0.22	369
8	0.52	380
9	0.92	391
10	1.45	402
11	2.19	412
12	3.24	422
13	6.61	432
14	9.71	441
15	16.34	450
16	34.45	459
17	49.89	468
18	56.36	476
19	64.26	483
20	71.72	491
21	76.27	498
22	83.49	505
23	88.38	512
24	92.13	518
25	95.05	525
26	97.20	531
27	98.41	537
28	99.15	543
29	99.56	548
30	99.77	553
31	99.89	558
32	99.95	563
33	99.98	567
34	99.99	571
35	100.00	575

Sample #35

Interval	% Area	Boiling Point °C
1	0.01	287
2	0.52	302
3	2.15	317
4	5.62	331
5	9.70	344
6	13.96	356
7	18.91	369
8	24.53	380
9	30.88	391
10	38.35	402
11	49.67	412
12	59.68	422
13	72.89	432
14	79.64	441
15	85.66	450
16	93.54	459
17	99.04	468
18	99.53	476
19	100.00	483

Sample #37

Interval	% Area	Boiling Point °C
1	0.03	69
2	0.09	98
3	0.14	126
4	0.18	151
5	0.28	174
6	0.41	196
7	0.52	216
8	0 56	235
9	0.62	253
10	0.68	271
11	0.71	287
12	1.52	302
13	3.34	317
14	6.75	331
15	10.83	344
16	15.15	356
17	20.18	369
18	25.99	380
19	30.17	391
20	35.34	402
21	43.56	412
22	52.54	422
23	62.98	432
24	77.04	441
25	86.32	450
26	96.17	459
27	99.83	468
28	99.96	476
29	100.00	483

Sample #38

Interval	% Area	Boiling Point °C
1	0.01	287
2	0.97	302
3	4.11	317
4	8.05	331
5	12.66	344
6	17.45	356
7	23.02	369
8	29.31	380
9	36.48	391
10	48.09	402
11	57.75	412
12	68.79	422
13	82.22	432
14	94.14	441
15	98.16	450
16	99.06	459
17	99.59	468
18	99.83	476
19	100.00	483

Sample #39			Sample #40		
Interval	% Area	Boiling Point °C	Interval	% Area	Boiling Point °C
1	0.01	287	1	0.10	69
2	0.04	302	2	0.23	98
3	0.06	317	3	0.41	126
4	0.07	331	4	2.29	151
5	0.10	344	5	5.61	174
6	0.11	356	6	11.84	196
7	0.18	369	7	20.07	216
8	0.34	380	8	27.60	235
9	0.63	391	9	38.65	253
10	1.41	402	10	48.50	271
11	2.59	412	11	59.62	287
12	4.90	422	12	69.84	302
13	6.45	432	13	75.16	317
14	8.89	441	14	79.35	331
15	14.89	450	15	83.62	344
16	35.67	459	16	87.74	356
17	61.70	468	17	91.85	369
18	69.92	476	18	96.75	380
19	74.51	483	19	99.39	391
20	79.02	491	20	99.65	402
21	81.82	498	21	99.84	412
22	86.26	505	22	99.91	422
23	90.23	512	23	100.00	432
24	93.41	518			
25	95.82	525			
26	97.50	531			
27	98.56	537			
28	99.23	543			
29	99.61	548			
30	99.85	553			
31	99.98	558			
32	100.00	563			

Sample #41	Bp°C	Percent Area
C_7 hydrocarbons	98	.01
C_8 hydrocarbons	126	.02
C_9 hydrocarbons	151	.04
C_{10} hydrocarbons	174	.06
C_{11} hydrocarbons	196	.07
C_{12} hydrocarbons	216	.10
C_{13} hydrocarbons	235	.13
C_{14} hydrocarbons	253	.16
C_{15} hydrocarbons	271	.19
C_{16} hydrocarbons	287	.24
C_{17} hydrocarbons	302	.56
C_{18} hydrocarbons	317	1.12
C_{19} hydrocarbons	331	2.44
C_{20} hydrocarbons	344	6.45
C_{21} hydrocarbons	356	10.94
C_{22} hydrocarbons	369	16.14
C_{23} hydrocarbons	380	24.23
C_{24} hydrocarbons	391	31.40
C_{25} hydrocarbons	402	39.45
C_{26} hydrocarbons	412	48.36
C_{27} hydrocarbons	422	57.99
C_{28} hydrocarbons	432	68.66
C_{29} hydrocarbons	441	82.19
C_{30} hydrocarbons	450	93.18
C_{31} hydrocarbons	459	99.78
C_{32} hydrocarbons	468	99.87
C_{33} hydrocarbons	476	100.00

Sample #42	Bp°C	Percent Area
C_{19} hydrocarbons	331	.07
C_{20} hydrocarbons	344	1.29
C_{21} hydrocarbons	356	5.75
C_{22} hydrocarbons	369	13.79
C_{23} hydrocarbons	380	25.96
C_{24} hydrocarbons	391	36.33
C_{25} hydrocarbons	402	48.48
C_{26} hydrocarbons	412	64.66
C_{27} hydrocarbons	422	89.26
C_{28} hydrocarbons	432	96.64
C_{29} hydrocarbons	441	99.28
C_{30} hydrocarbons	450	100.00

Sample #43	Bp°C	Percent Area
C_{20} hydrocarbons	344	.09
C_{21} hydrocarbons	356	.10
C_{22} hydrocarbons	369	.13
C_{23} hydrocarbons	380	.20
C_{24} hydrocarbons	391	.38
C_{25} hydrocarbons	402	.95
C_{26} hydrocarbons	412	1.76
C_{27} hydrocarbons	422	3.56
C_{28} hydrocarbons	432	5.37
C_{29} hydrocarbons	441	8.81
C_{30} hydrocarbons	450	15.34
C_{31} hydrocarbons	459	32.29
C_{32} hydrocarbons	468	59.83
C_{33} hydrocarbons	476	83.84
C_{34} hydrocarbons	483	86.66
C_{35} hydrocarbons	491	88.78
C_{36} hydrocarbons	498	91.08
C_{37} hydrocarbons	505	93.54
C_{38} hydrocarbons	512	96.90
C_{39} hydrocarbons	518	99.349
C_{40} hydrocarbons	525	100.00

Sample #44	Bp°C	Percent Area
C_8 hydrocarbons	126	.28
C_9 hydrocarbons	151	1.08
C_{10} hydrocarbons	174	3.50
C_{11} hydrocarbons	196	8.27
C_{12} hydrocarbons	216	15.02
C_{13} hydrocarbons	235	24.28
C_{14} hydrocarbons	253	35.86
C_{15} hydrocarbons	271	46.12
C_{16} hydrocarbons	287	57.47
C_{17} hydrocarbons	302	75.66
C_{18} hydrocarbons	317	83.30
C_{19} hydrocarbons	331	91.82
C_{20} hydrocarbons	344	98.06
C_{21} hydrocarbons	356	99.66
C_{22} hydrocarbons	369	100.00

Sample #45	Bp°C	Percent Area
C_9 hydrocarbons	151	.13
C_{10} hydrocarbons	174	.19
C_{11} hydrocarbons	196	.24
C_{12} hydrocarbons	216	.28
C_{13} hydrocarbons	235	.33
C_{14} hydrocarbons	253	.40
C_{15} hydrocarbons	271	.45
C_{16} hydrocarbons	287	.55
C_{17} hydrocarbons	302	.93
C_{18} hydrocarbons	317	3.34
C_{19} hydrocarbons	331	8.44
C_{20} hydrocarbons	344	13.76
C_{21} hydrocarbons	356	19.05
C_{22} hydrocarbons	369	25.62
C_{23} hydrocarbons	380	30.33
C_{24} hydrocarbons	391	38.69
C_{25} hydrocarbons	402	45.50
C_{26} hydrocarbons	412	56.43
C_{27} hydrocarbons	422	67.19
C_{28} hydrocarbons	432	79.35
C_{29} hydrocarbons	441	92.83
C_{30} hydrocarbons	450	97.18
C_{31} hydrocarbons	459	100.00

Sample #46	Bp°C	Percent Area
C_{18} hydrocarbons	317	.54
C_{19} hydrocarbons	331	3.27
C_{20} hydrocarbons	344	8.61
C_{21} hydrocarbons	356	14.23
C_{22} hydrocarbons	369	20.30
C_{23} hydrocarbons	380	29.71
C_{24} hydrocarbons	391	38.23
C_{25} hydrocarbons	402	48.04
C_{26} hydrocarbons	412	59.58
C_{27} hydrocarbons	422	70.62
C_{28} hydrocarbons	432	83.19
C_{29} hydrocarbons	441	99.46
C_{30} hydrocarbons	450	100.00

Sample #47	Bp°C	Percent Area
C_{14} hydrocarbons	253	.01
C_{15} hydrocarbons	271	.06
C_{16} hydrocarbons	287	.08
C_{17} hydrocarbons	302	.10
C_{18} hydrocarbons	317	.14
C_{19} hydrocarbons	331	.22
C_{20} hydrocarbons	344	.32
C_{21} hydrocarbons	356	.41
C_{22} hydrocarbons	369	.56
C_{23} hydrocarbons	380	.66
C_{24} hydrocarbons	391	.10
C_{25} hydrocarbons	402	1.59
C_{26} hydrocarbons	412	2.39
C_{27} hydrocarbons	422	3.79
C_{28} hydrocarbons	432	6.31
C_{29} hydrocarbons	441	47.39
C_{30} hydrocarbons	450	49.49
C_{31} hydrocarbons	459	66.98
C_{32} hydrocarbons	468	87.76
C_{33} hydrocarbons	476	90.22
C_{34} hydrocarbons	483	92.88
C_{35} hydrocarbons	491	92.89
C_{36} hydrocarbons	498	96.27
C_{37} hydrocarbons	505	97.55
C_{38} hydrocarbons	512	97.79
C_{39} hydrocarbons	518	100.00

Sample #48	Bp°C	Percent Area
C_8 hydrocarbons	126	.18
C_9 hydrocarbons	151	.61
C_{10} hydrocarbons	174	1.98
C_{11} hydrocarbons	196	5.48
C_{12} hydrocarbons	216	11.22
C_{13} hydrocarbons	235	19.87
C_{14} hydrocarbons	253	30.90
C_{15} hydrocarbons	271	40.79
C_{16} hydrocarbons	287	51.80
C_{17} hydrocarbons	302	68.73
C_{18} hydrocarbons	317	83.32
C_{19} hydrocarbons	331	90.27
C_{20} hydrocarbons	344	97.41
C_{21} hydrocarbons	356	99.51
C_{22} hydrocarbons	369	100.00

Sample #49	Bp°C	Percent Area
C_{13} hydrocarbons	235	.20
C_{14} hydrocarbons	253	.25
C_{15} hydrocarbons	271	.29
C_{16} hydrocarbons	287	.36
C_{17} hydrocarbons	302	1.08
C_{18} hydrocarbons	317	4.14
C_{19} hydrocarbons	331	10.19
C_{20} hydrocarbons	344	16.52
C_{21} hydrocarbons	356	23.23
C_{22} hydrocarbons	369	30.90
C_{23} hydrocarbons	380	33.41
C_{24} hydrocarbons	391	42.66
C_{25} hydrocarbons	402	53.29
C_{26} hydrocarbons	412	64.91
C_{27} hydrocarbons	422	76.82
C_{28} hydrocarbons	432	94.02
C_{29} hydrocarbons	441	97.70
C_{30} hydrocarbons	450	100.00

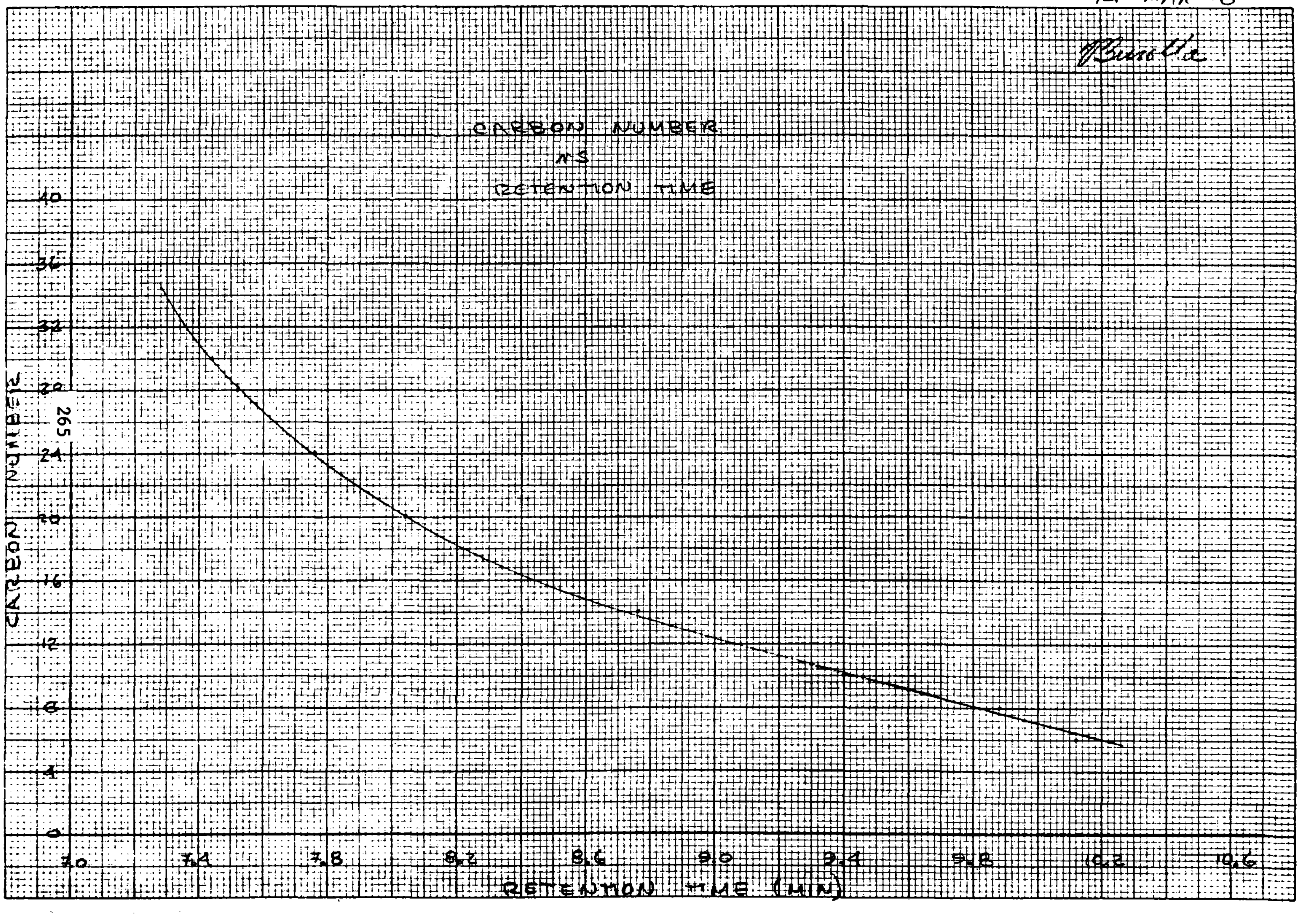
Sample #50	Bp°C	Percent Area
C_{18} hydrocarbons	317	.61
C_{19} hydrocarbons	331	3.39
C_{20} hydrocarbons	344	9.17
C_{21} hydrocarbons	356	15.10
C_{22} hydrocarbons	369	21.67
C_{23} hydrocarbons	380	32.16
C_{24} hydrocarbons	391	41.91
C_{25} hydrocarbons	402	53.06
C_{26} hydrocarbons	412	69.51
C_{27} hydrocarbons	422	78.34
C_{28} hydrocarbons	432	95.32
C_{29} hydrocarbons	441	99.39
C_{30} hydrocarbons	450	100.00

Sample #51	Bp°C	Percent Area
C_{19} hydrocarbons	331	.03
C_{20} hydrocarbons	344	.06
C_{21} hydrocarbons	356	.09
C_{22} hydrocarbons	369	.17
C_{23} hydrocarbons	380	.41
C_{24} hydrocarbons	391	.90
C_{25} hydrocarbons	402	1.95
C_{26} hydrocarbons	412	3.43
C_{27} hydrocarbons	422	6.51
C_{28} hydrocarbons	432	9.16
C_{29} hydrocarbons	441	25.25
C_{30} hydrocarbons	450	66.93
C_{31} hydrocarbons	459	86.46
C_{32} hydrocarbons	468	90.35
C_{33} hydrocarbons	476	90.40
C_{34} hydrocarbons	483	90.73
C_{35} hydrocarbons	491	92.06
C_{36} hydrocarbons	498	94.47
C_{37} hydrocarbons	505	95.57
C_{38} hydrocarbons	512	97.80
C_{39} hydrocarbons	519	100.00

Sample #52	Bp°C	Percent Area
C_9 hydrocarbons	151	.18
C_{10} hydrocarbons	174	.71
C_{11} hydrocarbons	196	2.64
C_{12} hydrocarbons	216	8.28
C_{13} hydrocarbons	235	18.39
C_{14} hydrocarbons	253	34.19
C_{15} hydrocarbons	271	54.54
C_{16} hydrocarbons	287	71.95
C_{17} hydrocarbons	302	96.21
C_{18} hydrocarbons	317	98.22
C_{19} hydrocarbons	331	99.92
C_{20} hydrocarbons	344	100.00

Gel Permeation Chromatography Tests

14 MAR 78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vs
RETENTION TIME
CARBON NUMBER
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36
32
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24
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16
12
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7.0
7.4
7.8
8.2
8.6
9.0
9.4
9.8
10.2
10.6
RETENTION TIME (MIN)

DATE : 23 MAY 78
SAMPLE : CALORIA HT43- MARTIN M- A-1
INJECTION VOL : 2.0 µℓ
COLUMNS : 1000A°, 500A°, 100A°, 100A° MICROSTYRAGEL
CARRIER : [✓ THF] TOLUENE ____________
FLOW RATE : 1.5 [✓ 2.0] 2.5 3.0 ____________ mℓ/min
DETECTORS : UV-VIS --
WAVELENGTH 254 nm
BANDWIDTH 2 4 [✓ 8] 16 nm
ABS. RANGE 2 [✓ 1] .5 .2 .1 .05
TIME CONSTANT FAST NORMAL [✓ SLOW]
BEAM SINGLE [✓ DOUBLE]

RI --
ATTENUATION 64 32 16 [✓ 8] 4 2

RECORDER : CHART SPEED .25 .5 [✓ 1.25] 2.5 cm/min
RANGE --
UV-VIS 5 10 [✓ 20] 50 mv/FS
RI 5 [✓ 10] 20 50 mv/FS

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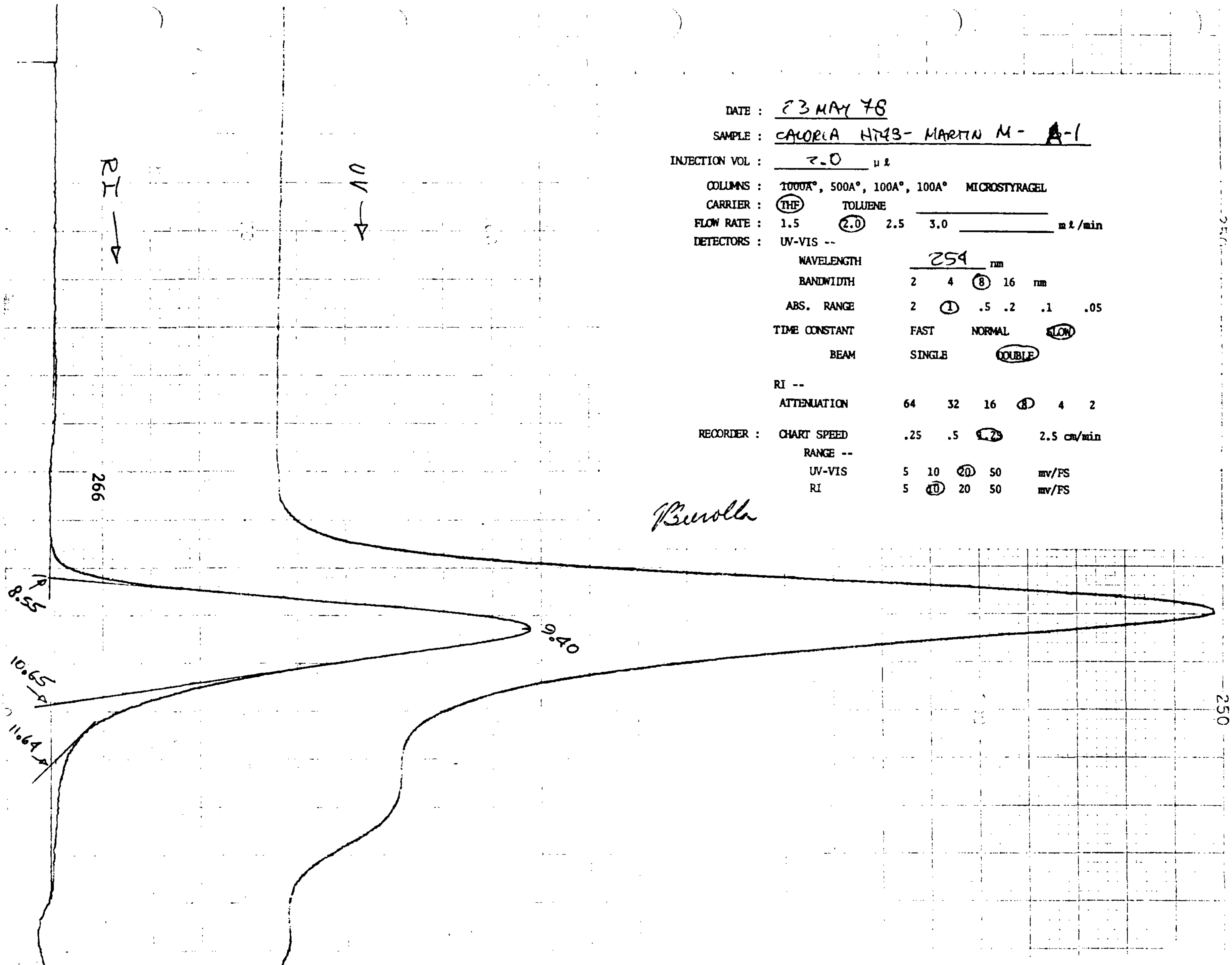
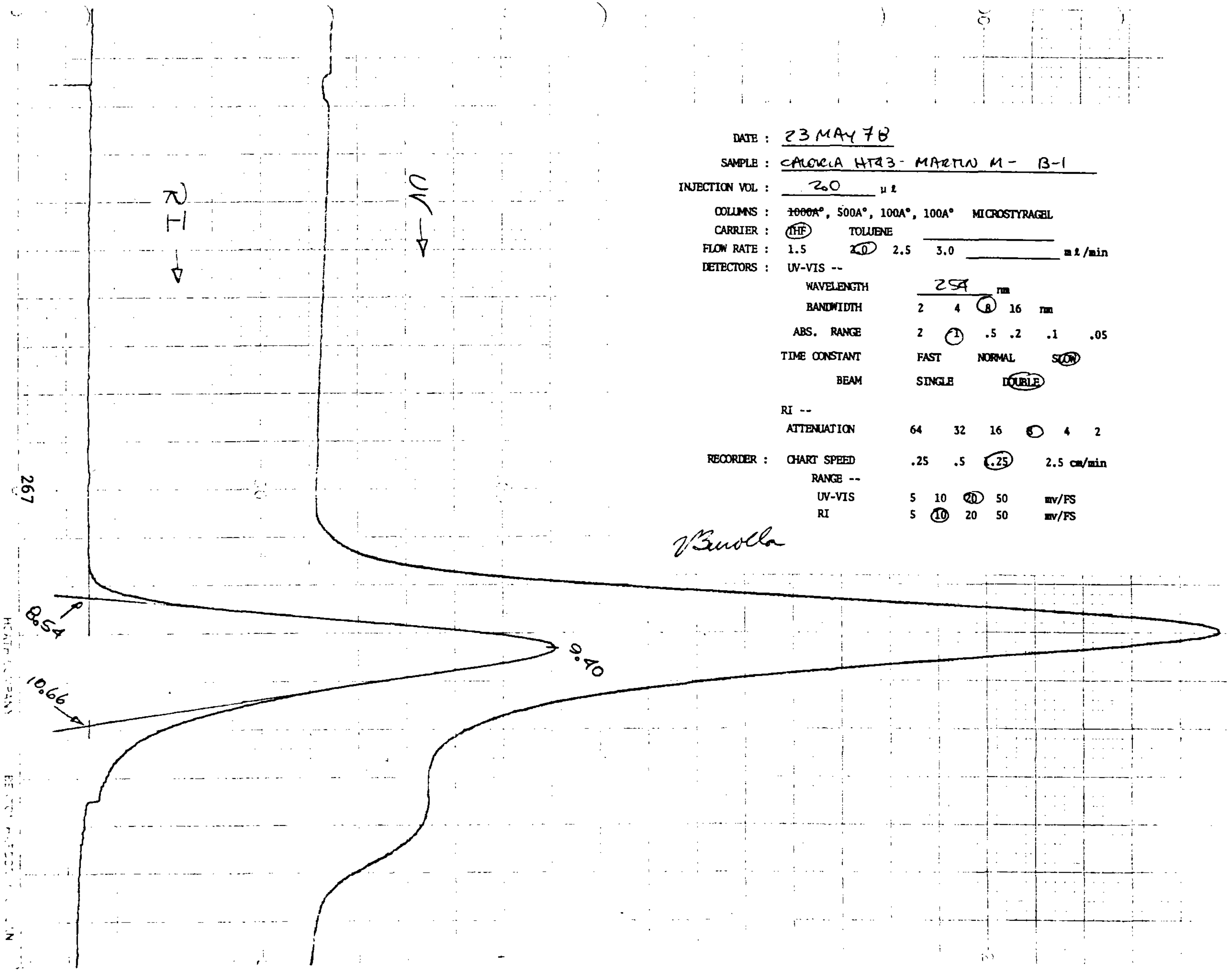




DATE : 23 MAY 78

SAMPLE : CALORIA HT43- MARTIN M- B-1

INJECTION VOL : 20 μl

COLUMNS : ~~1000A°~~, 500A°, 100A°, 100A° MICROSTYRAGEL

CARRIER : [✓ THF] TOLUENE ______

FLOW RATE : 1.5 [✓ 2.0] 2.5 3.0 ______ ml/min

DETECTORS : UV-VIS --

WAVELENGTH 254 nm

BANDWIDTH 2 4 [✓ 8] 16 nm

ABS. RANGE 2 [✓ 1] .5 .2 .1 .05

TIME CONSTANT FAST NORMAL [✓ SLOW]

BEAM SINGLE [✓ DOUBLE]

RI --

ATTENUATION 64 32 16 [✓ 8] 4 2

RECORDER : CHART SPEED .25 .5 [✓ 1.25] 2.5 cm/min

RANGE --

UV-VIS 5 10 [✓ 20] 50 mv/FS

RI 5 [✓ 10] 20 50 mv/FS

VBuolla

DATE : 23 MAY 78

SAMPLE : CALORIA HT43 - MARTIN M - C-1

INJECTION VOL : 200 μℓ

COLUMNS : ~~1000A°~~, 500A°, 100A°, 100A° MICROSTYRAGEL

CARRIER : [✓ THF] TOLUENE ______

FLOW RATE : 1.5 [✓ 2.0] 2.5 3.0 ______ mℓ/min

DETECTORS : UV-VIS --

WAVELENGTH 254 nm

BANDWIDTH 2 4 [✓ 8] 16 nm

ABS. RANGE 2 [✓ 1] .5 .2 .1 .05

TIME CONSTANT FAST NORMAL [✓ SLOW]

BEAM SINGLE [✓ DOUBLE]

RI --

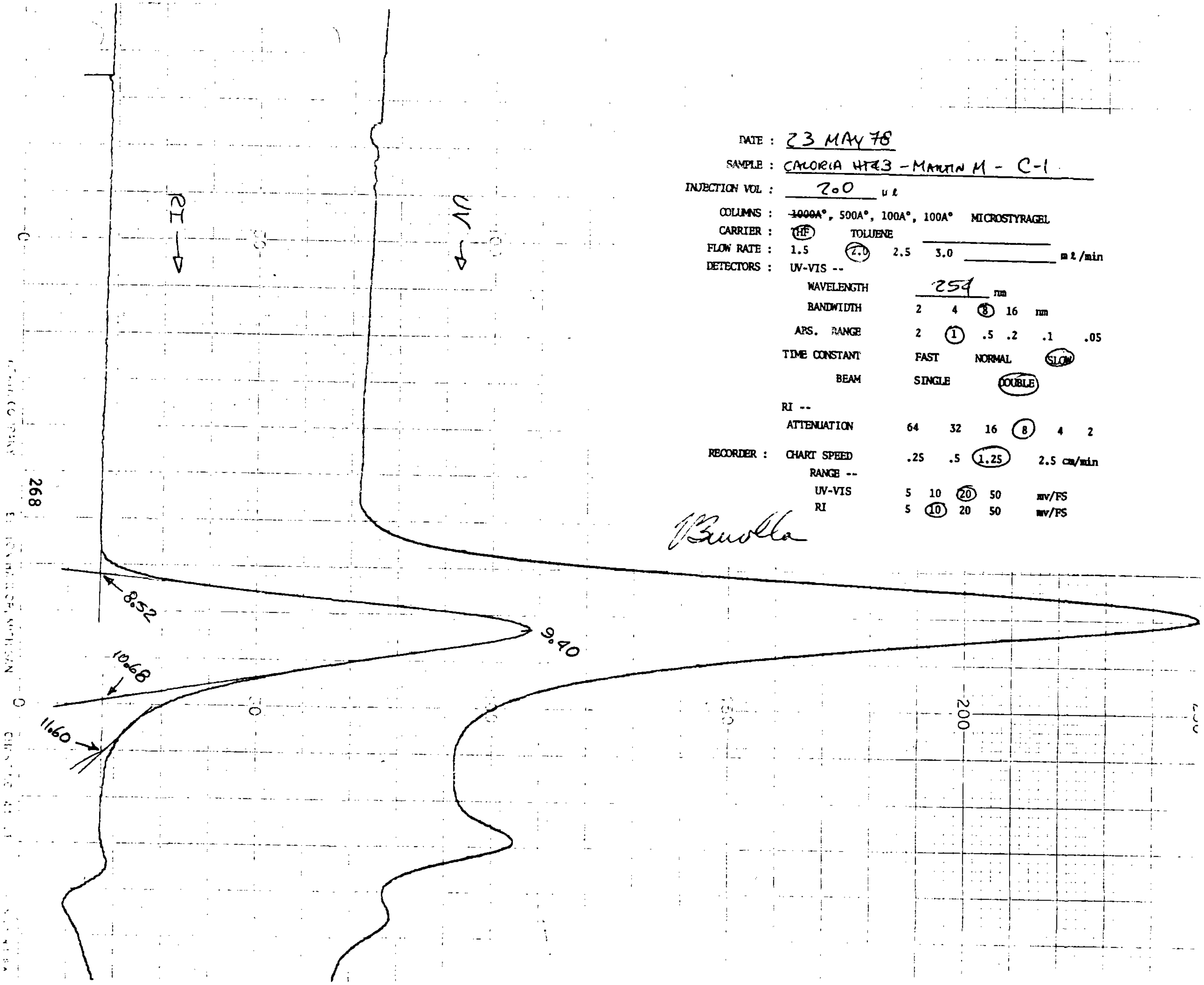
ATTENUATION 64 32 16 [✓ 8] 4 2

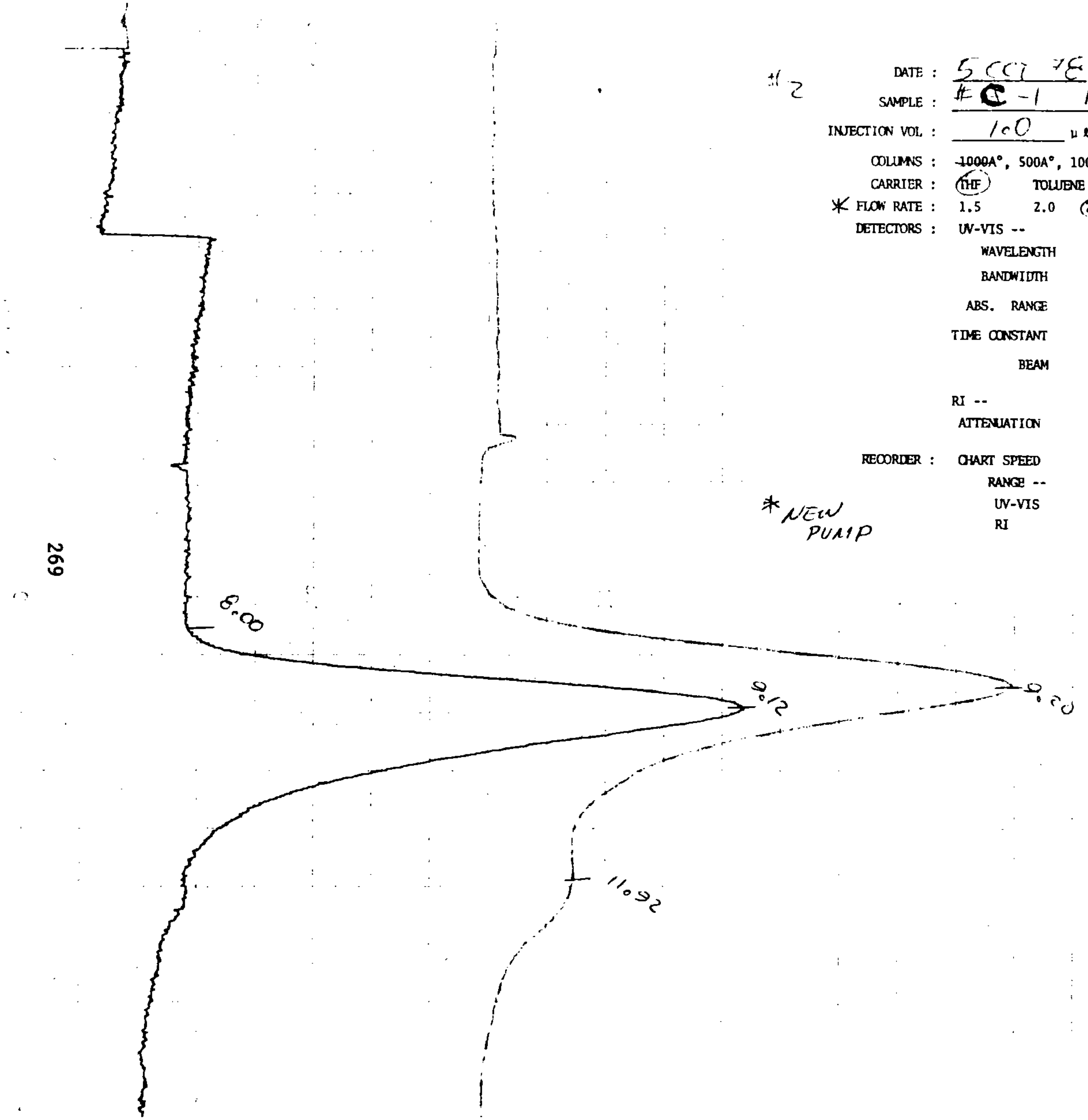
RECORDER : CHART SPEED .25 .5 [✓ 1.25] 2.5 cm/min

RANGE --

UV-VIS 5 10 [✓ 20] 50 mv/FS

RI 5 [✓ 10] 20 50 mv/FS





#2

DATE : 5 OCT 78

SAMPLE : #C-1 MANITOU

INJECTION VOL : 100 µℓ

COLUMNS : ~~1000A°~~, 500A°, 100A°, 100A° MICROSTYRAGEL

CARRIER : [✓ THF] TOLUENE ______

* FLOW RATE : 1.5 2.0 [✓ 2.5] 3.0 ______ mℓ/min

DETECTORS : UV-VIS --

WAVELENGTH 254 nm

BANDWIDTH 2 4 [✓ 8] 16 nm

ABS. RANGE 2 [✓ 1] .5 .2 .1 .05

TIME CONSTANT FAST NORMAL [✓ SLOW]

BEAM SINGLE [✓ DOUBLE]

RI --

ATTENUATION 64 32 16 [✓ 8] 4 2

RECORDER : CHART SPEED .25 .5 [✓ 1.25] 2.5 cm/min

RANGE --

UV-VIS 5 10 [✓ 20] 50 mv/FS

RI [✓ 5] 10 20 50 mv/FS

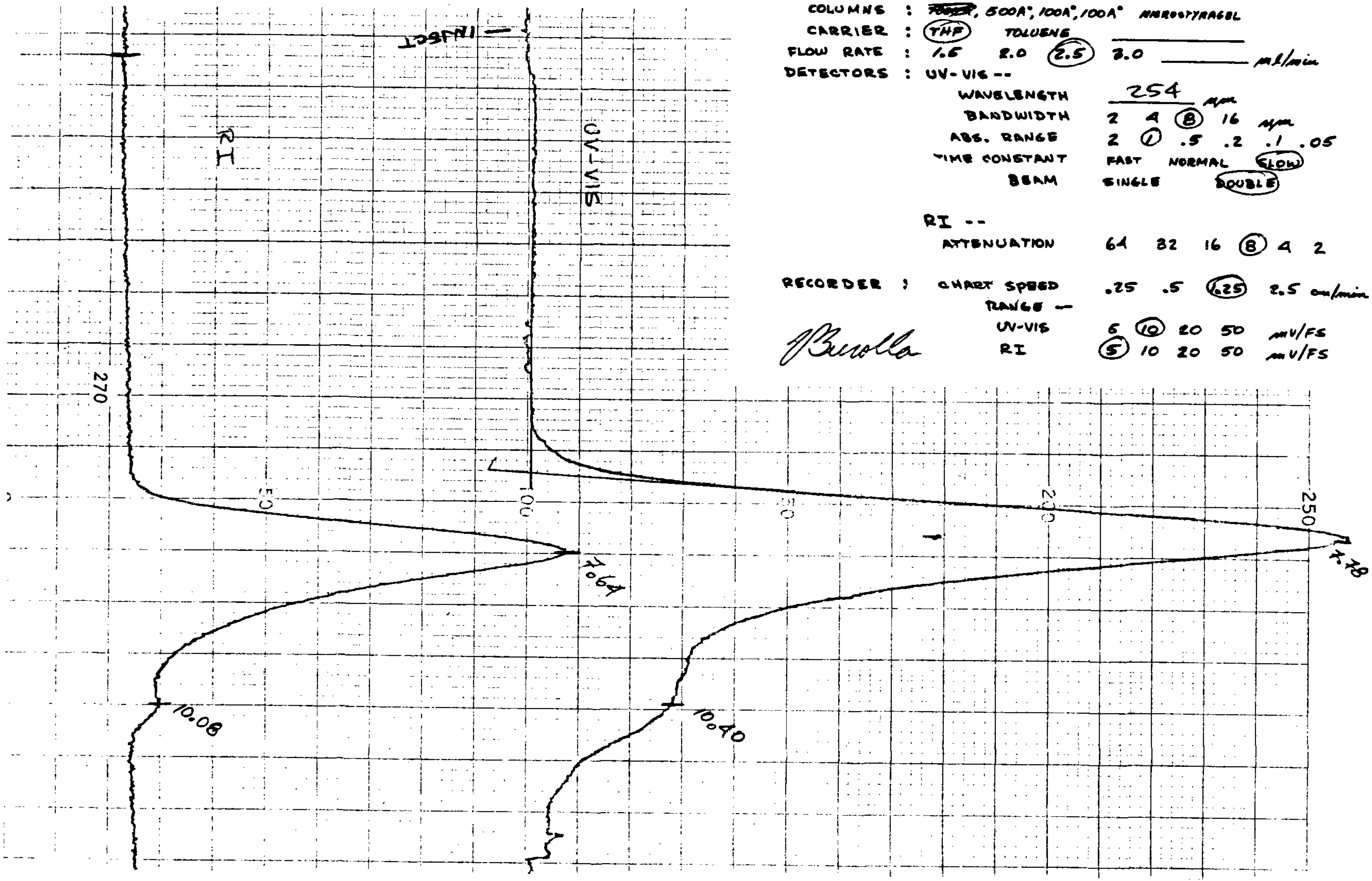
* NEW PUMP

DATE : 14 MAR 78
SAMPLE : C-1 CALORIA HT43 - MARTIN M.
INJECTION VOL : 1.0 μl
COLUMNS : ~~[illegible]~~, 500Å, 100Å, 100Å MICROSTYRAGEL
CARRIER : [✓ THF] TOLUENE ________
FLOW RATE : 1.5 2.0 [✓ 2.5] 3.0 ________ ml/min
DETECTORS : UV-VIS --
WAVELENGTH 254 nm
BANDWIDTH 2 4 [✓ 8] 16 nm
ABS. RANGE 2 [✓ 1] .5 .2 .1 .05
TIME CONSTANT FAST NORMAL [✓ SLOW]
BEAM SINGLE [✓ DOUBLE]

RI --
ATTENUATION 64 32 16 [✓ 8] 4 2

RECORDER : CHART SPEED .25 .5 [✓ 1.25] 2.5 cm/min
RANGE --
UV-VIS 5 [✓ 10] 20 50 mV/FS
RI [✓ 5] 10 20 50 mV/FS

J Burolla



DATE : 15 MAR 78
SAMPLE : #2 - CALORIA HT43 - MARTIN M.
INJECTION VOL : 1.0 µl
COLUMNS : [redacted], 500A°, 100A°, 100A° µSTYRAGEL
CARRIER : [✓ THF] TOLUENE ____
FLOW RATE : 1.6 2.0 [✓ 2.5] 3.0 ____ ml/min
DETECTORS : UV-VIS --
WAVELENGTH 254 nm
BANDWIDTH 2 4 [✓ 8] 16 nm
ABS. RANGE 2 [✓ 1] .5 .2 .1 .05
TIME CONSTANT FAST NORMAL [✓ SLOW]
BEAM SINGLE [✓ DOUBLE]

RI --
ATTENUATION 64 32 16 [✓ 8] 4 2

RECORDER : CHART SPEED .25 .5 [✓ 1.25] 2.5 cm/min
RANGE --
UV-VIS 5 10 [✓ 20] 50 mV/FS
RI [✓ 5] 10 20 50 mV/FS

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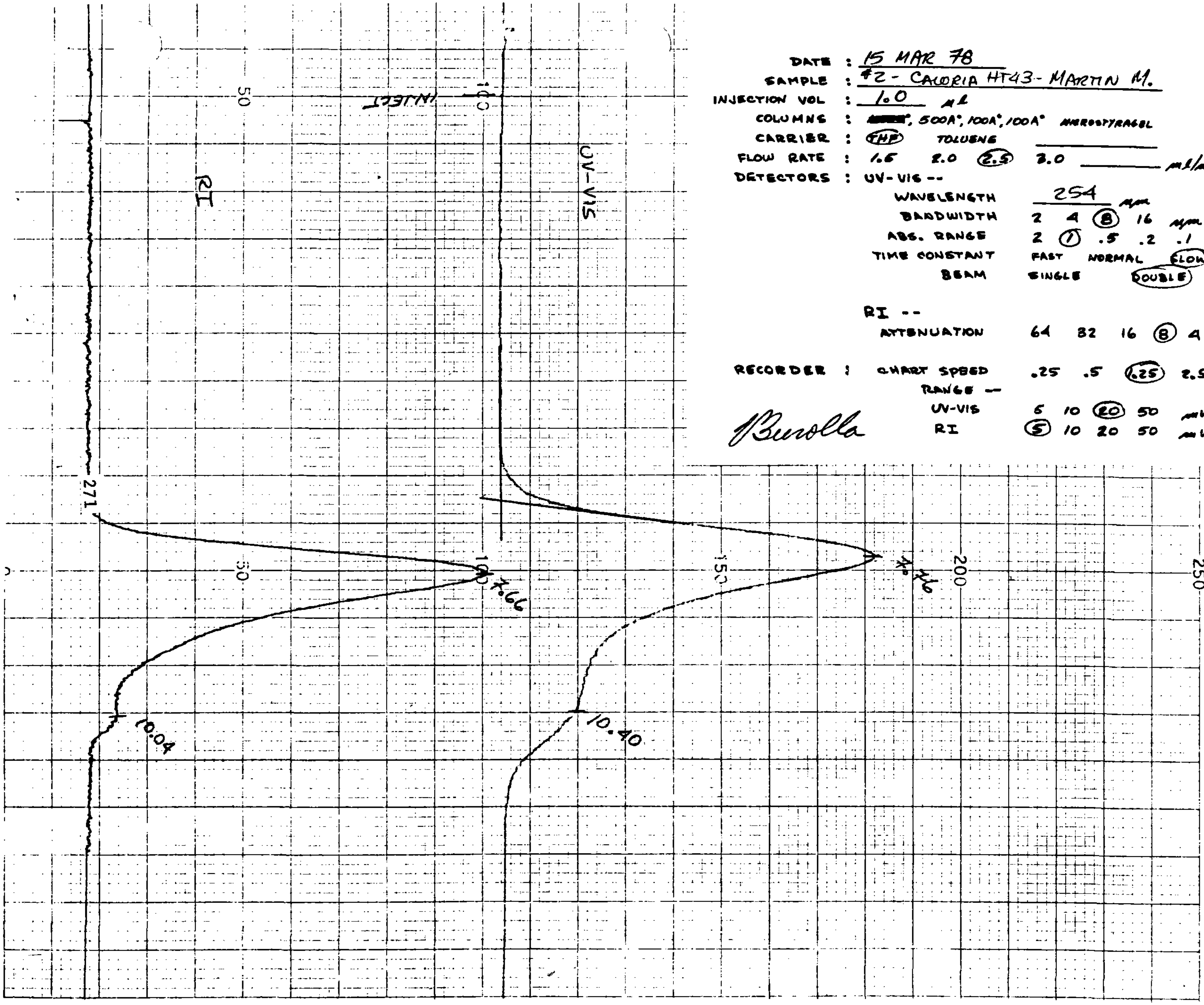
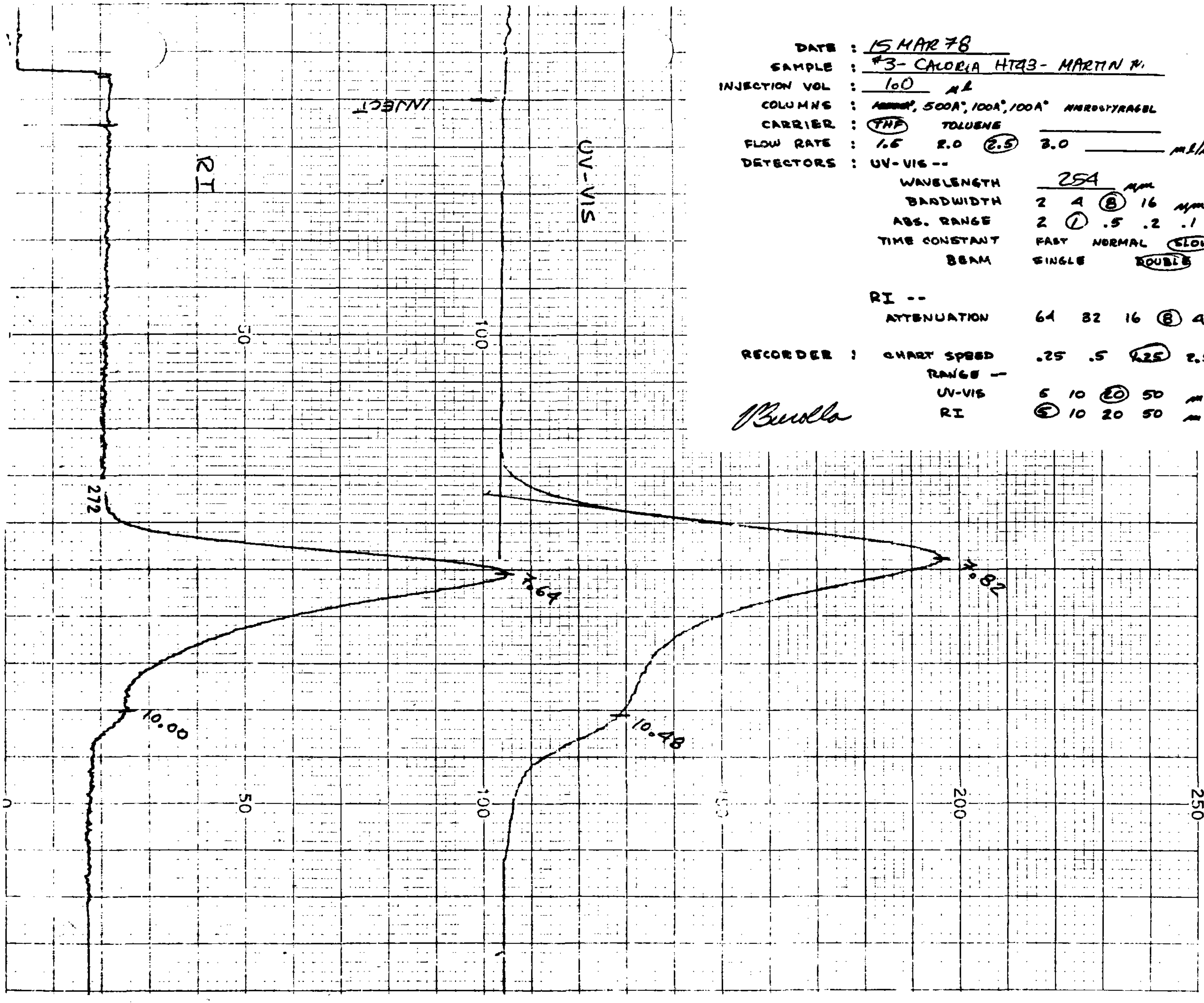


DATE : 15 MAR 78
SAMPLE : #3- CALORIA HT43- MARTIN R.
INJECTION VOL : 100 μl
COLUMNS : [illegible], 500A°, 100A°, 100A° MICROSTYRAGEL
CARRIER : [✓ THF] TOLUENE ________
FLOW RATE : 1.6 2.0 [✓ 2.5] 3.0 ________ ml/min
DETECTORS : UV-VIS --
WAVELENGTH 254 mμ
BANDWIDTH 2 4 [✓ 8] 16 mμ
ABS. RANGE 2 [✓ 1] .5 .2 .1 .05
TIME CONSTANT FAST NORMAL [✓ SLOW]
BEAM SINGLE [✓ DOUBLE]

RI --
ATTENUATION 64 32 16 [✓ 8] 4 2

RECORDER : CHART SPEED .25 .5 [✓ 1.25] 2.5 cm/min
RANGE --
UV-VIS 5 10 [✓ 20] 50 mV/FS
RI [✓ 5] 10 20 50 mV/FS

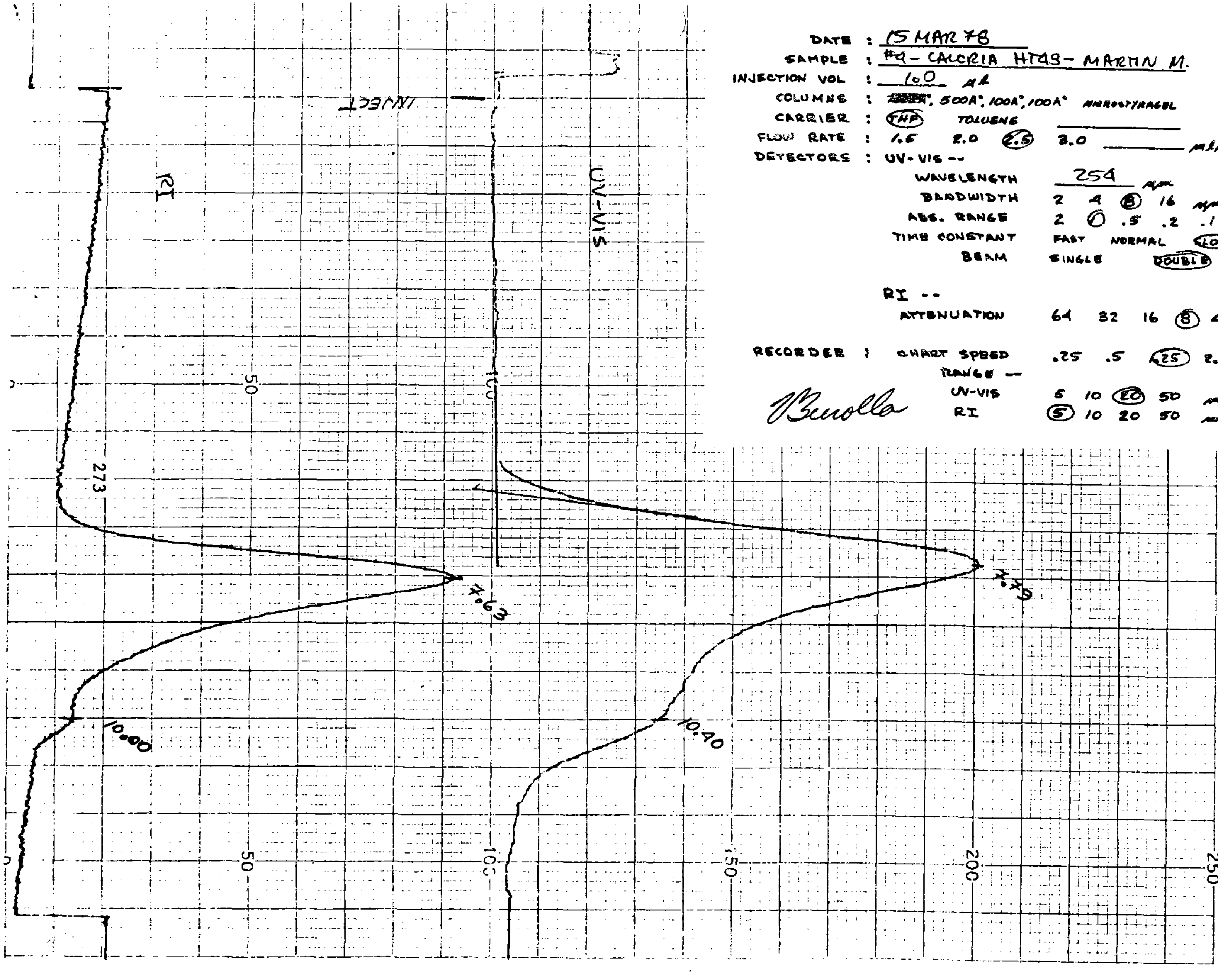
Burolla



DATE : 15 MAR 78
SAMPLE : #9 - CALCRIA HT43 - MARTIN M.
INJECTION VOL : 100 µl
COLUMNS : ~~[illegible]~~, 500A°, 100A°, 100A° MICROSTYRAGEL
CARRIER : [✓ THF] TOLUENE ______
FLOW RATE : 1.6 2.0 [✓ 2.5] 3.0 ______ ml/min
DETECTORS : UV-VIS --
WAVELENGTH 254 nm
BANDWIDTH 2 4 [✓ 8] 16 nm
ABS. RANGE 2 [✓ 1] .5 .2 .1 .05
TIME CONSTANT FAST NORMAL [✓ SLOW]
BEAM SINGLE [✓ DOUBLE]

RI --
ATTENUATION 64 32 16 [✓ 8] 4 2

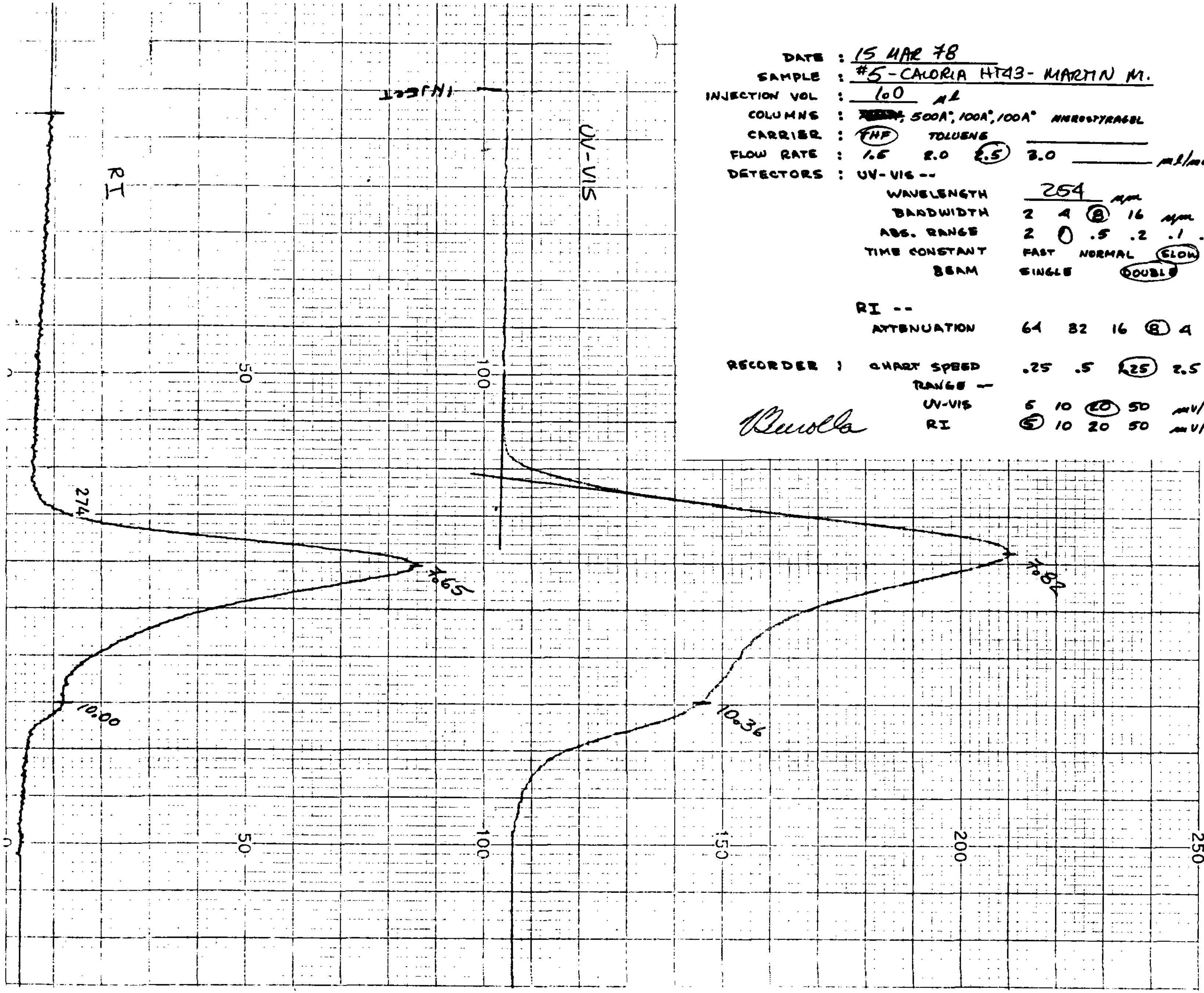
RECORDER : CHART SPEED .25 .5 [✓ 1.25] 2.5 cm/min
RANGE --
UV-VIS 5 10 [✓ 20] 50 mV/FS
RI [✓ 5] 10 20 50 mV/FS



DATE : 15 MAR 78
SAMPLE : #5-CALORIA HT43- MARTIN M.
INJECTION VOL : 100 μl
COLUMNS : ~~[illegible]~~, 500A°, 100A°, 100A° MICROSTYRAGEL
CARRIER : [✓ THF] TOLUENE ______
FLOW RATE : 1.5 2.0 [✓ 2.5] 3.0 ______ ml/min
DETECTORS : UV-VIS --
WAVELENGTH 254 nm
BANDWIDTH 2 4 [✓ 8] 16 nm
ABS. RANGE 2 [✓ 1] .5 .2 .1 .05
TIME CONSTANT FAST NORMAL [✓ SLOW]
BEAM SINGLE [✓ DOUBLE]

RI --
ATTENUATION 64 32 16 [✓ 8] 4 2

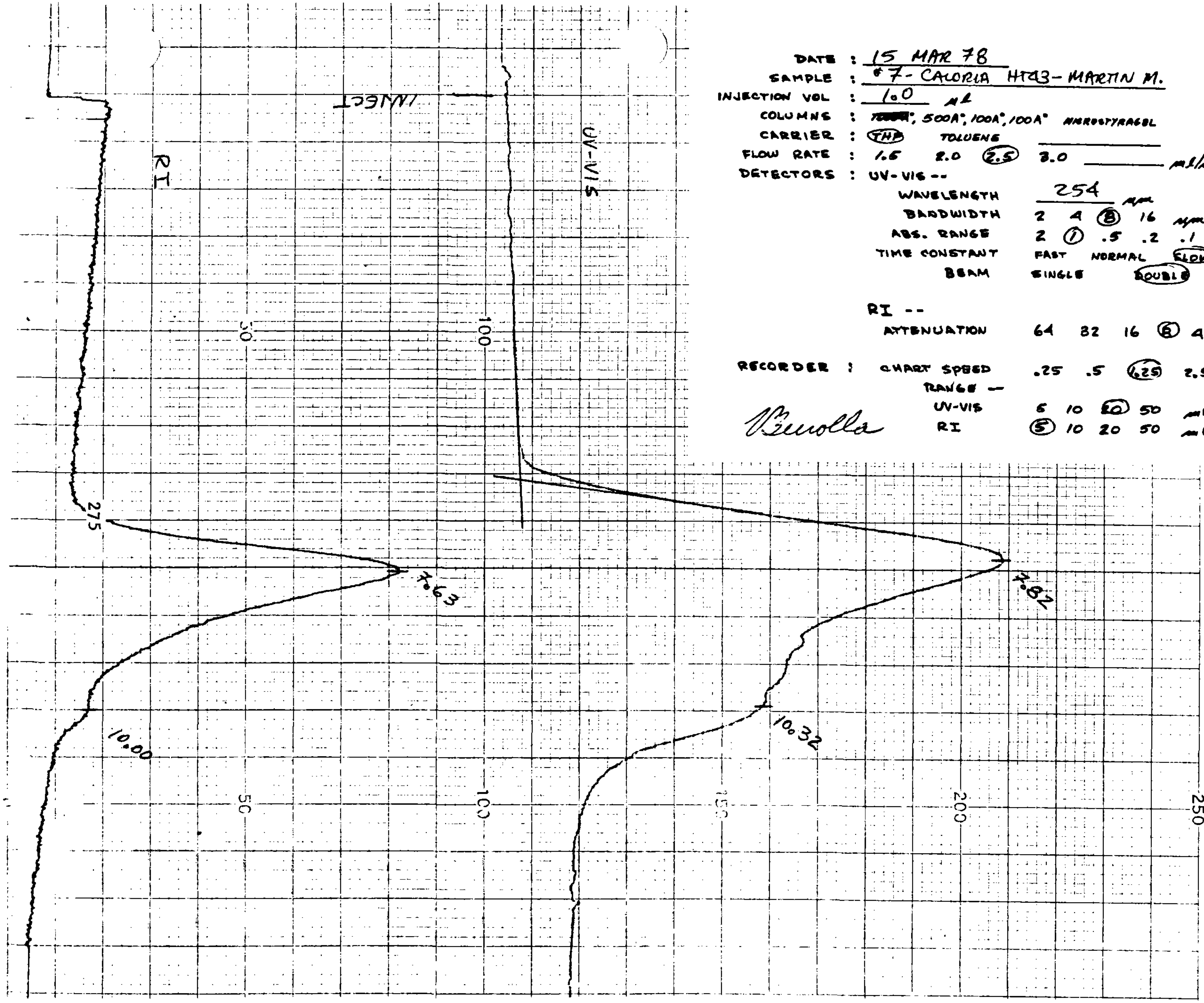
RECORDER : CHART SPEED .25 .5 [✓ 1.25] 2.5 cm/min
RANGE --
UV-VIS 5 10 [✓ 20] 50 mV/FS
RI [✓ 5] 10 20 50 mV/FS



DATE : 15 MAR 78
SAMPLE : #7- CALORIA HT43- MARTIN M.
INJECTION VOL : 10.0 μl
COLUMNS : [illegible], 500A°, 100A°, 100A° MICROSTYRAGEL
CARRIER : [✓ THF] TOLUENE ______
FLOW RATE : 1.6 2.0 [✓ 2.5] 3.0 ______ ml/min
DETECTORS : UV-VIS --
WAVELENGTH 254 nm
BANDWIDTH 2 4 [✓ 8] 16 nm
ABS. RANGE 2 [✓ 1] .5 .2 .1 .05
TIME CONSTANT FAST NORMAL [✓ SLOW]
BEAM SINGLE [✓ DOUBLE]

RI --
ATTENUATION 64 32 16 [✓ 8] 4 2

RECORDER : CHART SPEED .25 .5 [✓ 1.25] 2.5 cm/min
RANGE --
UV-VIS 5 10 [✓ 20] 50 mV/FS
RI [✓ 5] 10 20 50 mV/FS

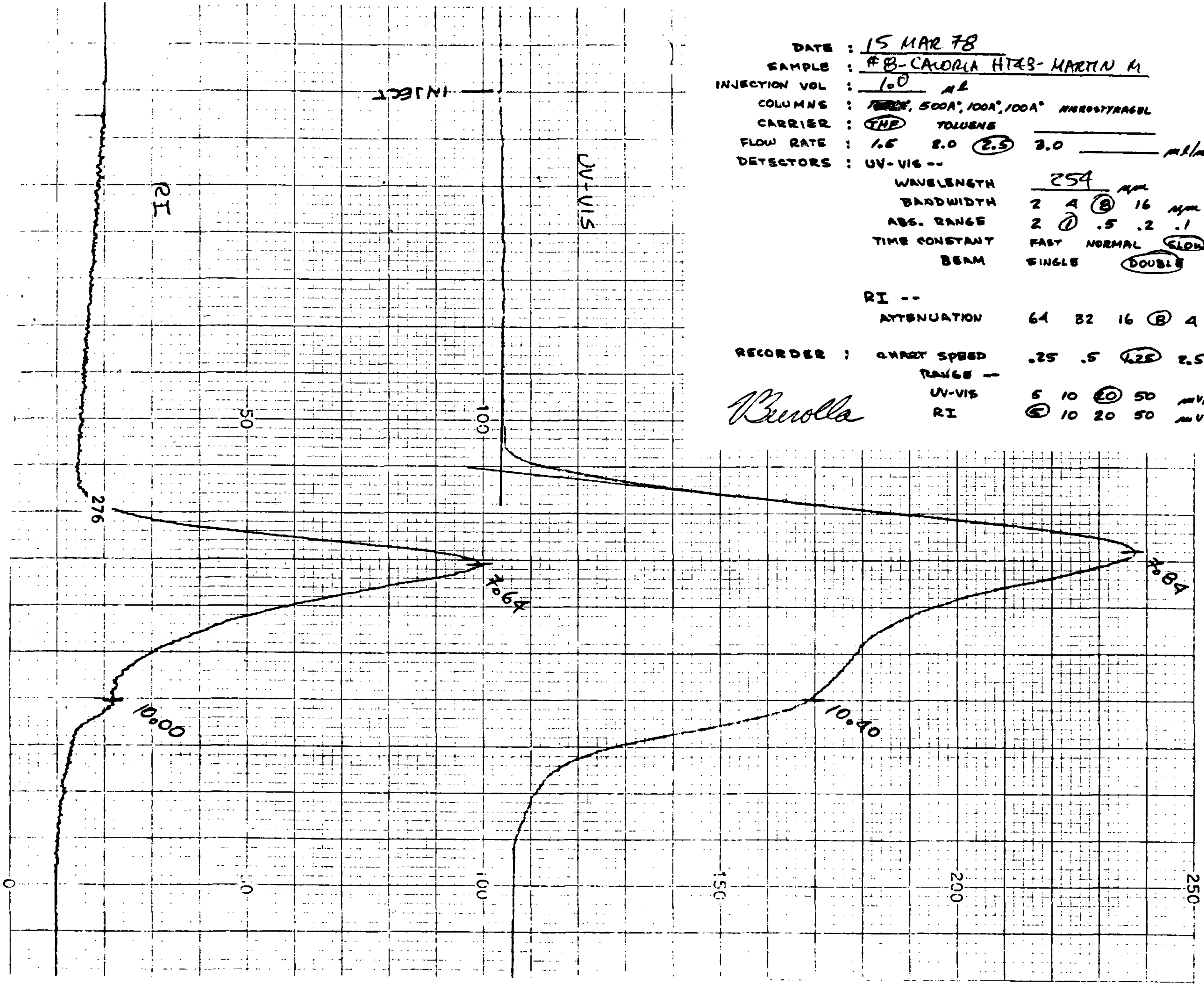


DATE : 15 MAR 78
SAMPLE : #B-CALORIA HT43- MARTIN M
INJECTION VOL : 1.0 µl
COLUMNS : [illegible], 500A°, 100A°, 100A° µSTYRAGEL
CARRIER : [✓ THF] TOLUENE ________
FLOW RATE : 1.6 2.0 [✓ 2.5] 3.0 ________ ml/min
DETECTORS : UV-VIS --
WAVELENGTH 254 nm
BANDWIDTH 2 4 [✓ 8] 16 nm
ABS. RANGE 2 [✓ 1] .5 .2 .1 .05
TIME CONSTANT FAST NORMAL [✓ SLOW]
BEAM SINGLE [✓ DOUBLE]

RI --
ATTENUATION 64 32 16 [✓ 8] 4 2

RECORDER : CHART SPEED .25 .5 [✓ 1.25] 2.5 cm/min
RANGE --
UV-VIS 5 10 [✓ 20] 50 mV/FS
RI [✓ 5] 10 20 50 mV/FS

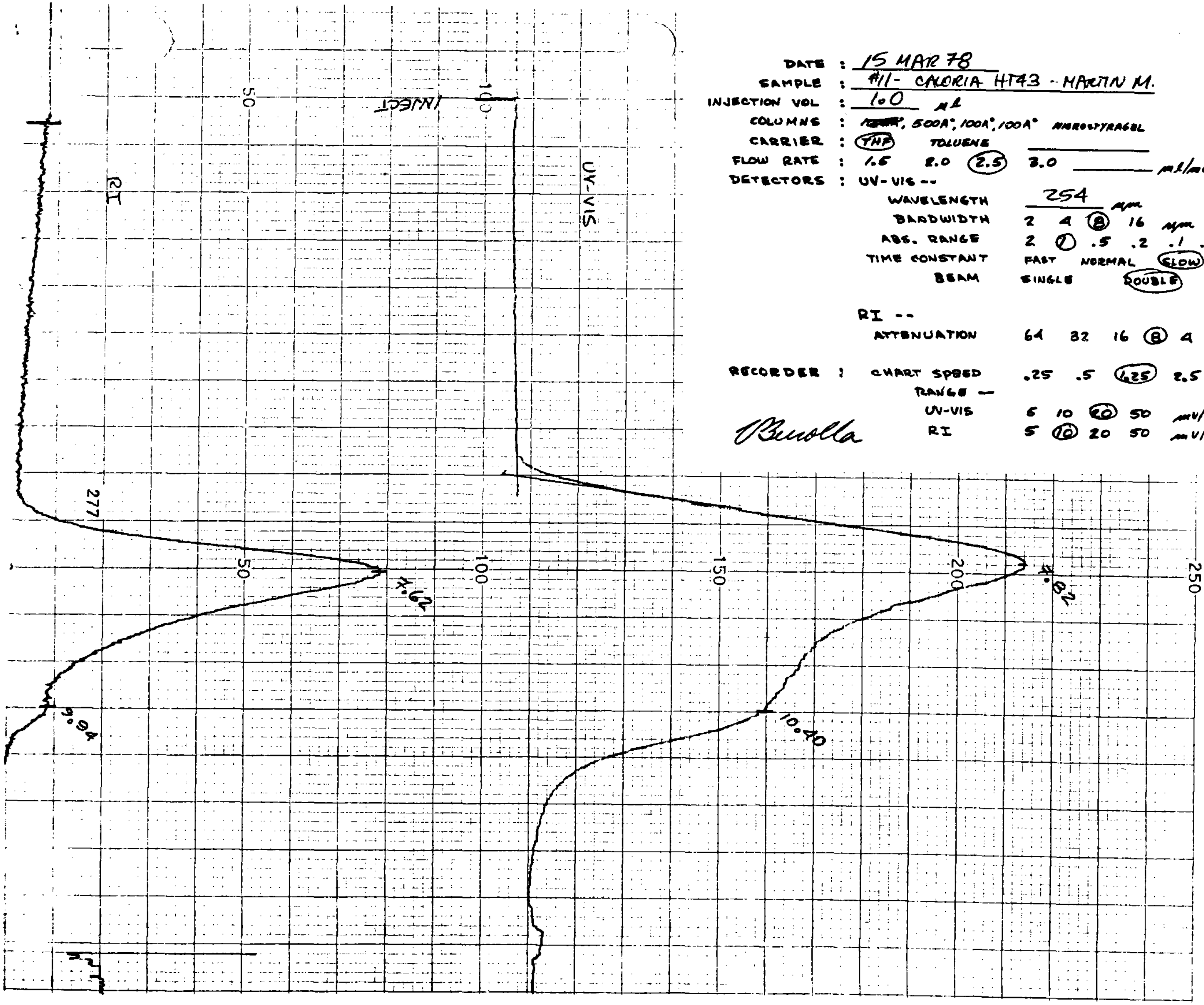
VBenolla



DATE : 15 MAR 78
SAMPLE : #11- CALORIA HT43 - MARTIN M.
INJECTION VOL : 100 μl
COLUMNS : ~~[illegible]~~°, 500A°, 100A°, 100A° μ-STYRAGEL
CARRIER : [✓ THF] TOLUENE ________
FLOW RATE : 1.5 2.0 [✓ 2.5] 3.0 ________ ml/min
DETECTORS : UV-VIS --
WAVELENGTH 254 nm
BANDWIDTH 2 4 [✓ 8] 16 nm
ABS. RANGE 2 [✓ 1] .5 .2 .1 .05
TIME CONSTANT FAST NORMAL [✓ SLOW]
BEAM SINGLE [✓ DOUBLE]

RI --
ATTENUATION 64 32 16 [✓ 8] 4 2

RECORDER : CHART SPEED .25 .5 [✓ 1.25] 2.5 cm/min
RANGE --
UV-VIS 5 10 [✓ 20] 50 mV/FS
RI 5 [✓ 10] 20 50 mV/FS

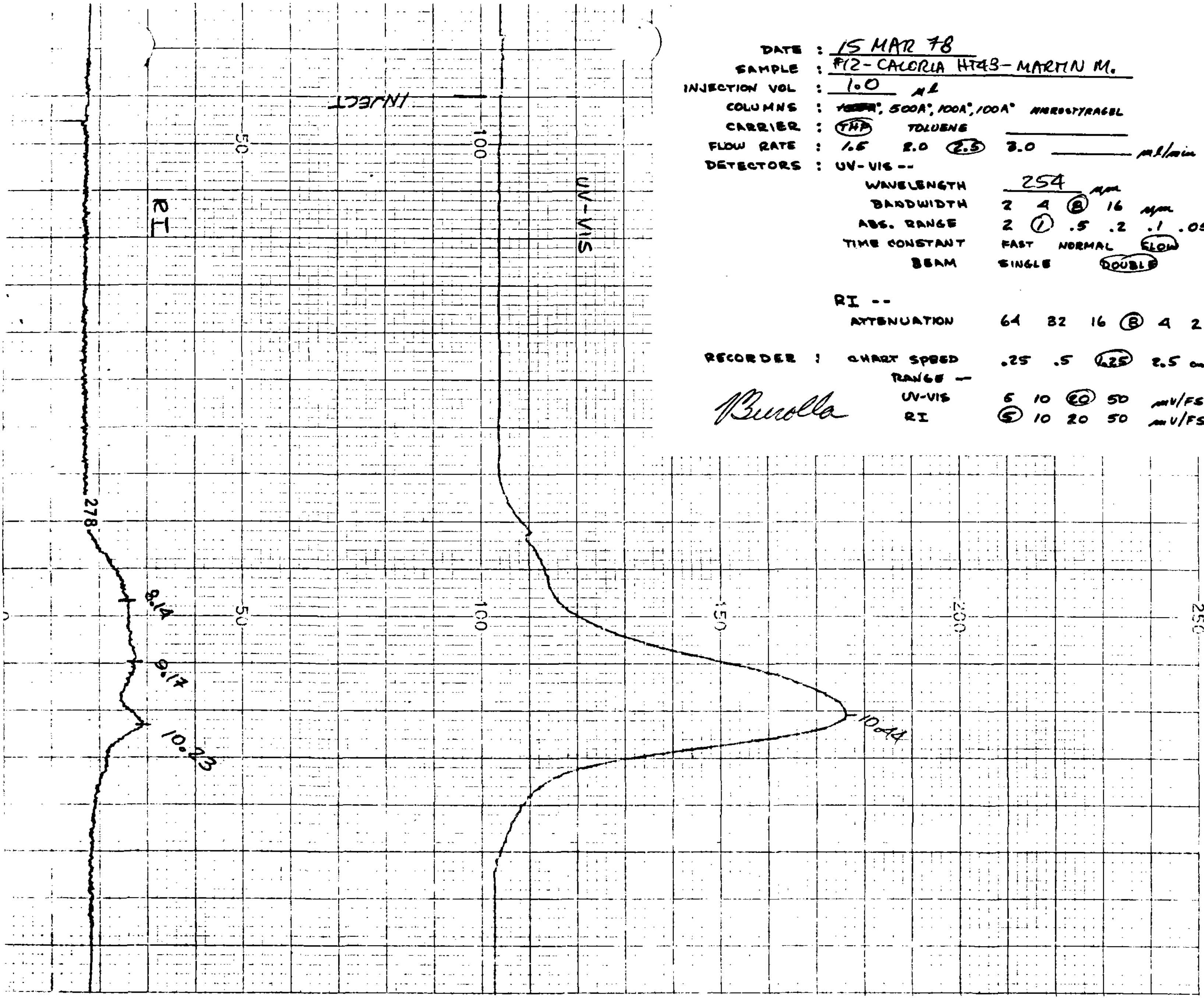


DATE : 15 MAR 78
SAMPLE : #12-CALORIA HT43-MARTIN M.
INJECTION VOL : 1.0 μl
COLUMNS : [illegible], 500A°, 100A°, 100A° MICROSTYRAGEL
CARRIER : [✓ THF] TOLUENE ____________
FLOW RATE : 1.5 2.0 [✓ 2.5] 3.0 ____________ ml/min
DETECTORS : UV-VIS --
WAVELENGTH 254 nm
BANDWIDTH 2 4 [✓ 8] 16 nm
ABS. RANGE 2 [✓ 1] .5 .2 .1 .05
TIME CONSTANT FAST NORMAL [✓ SLOW]
BEAM SINGLE [✓ DOUBLE]

RI --
ATTENUATION 64 32 16 [✓ 8] 4 2

RECORDER : CHART SPEED .25 .5 [✓ 1.25] 2.5 cm/min
RANGE --
UV-VIS 6 10 [✓ 20] 50 mV/FS
RI [✓ 5] 10 20 50 mV/FS

Burolla

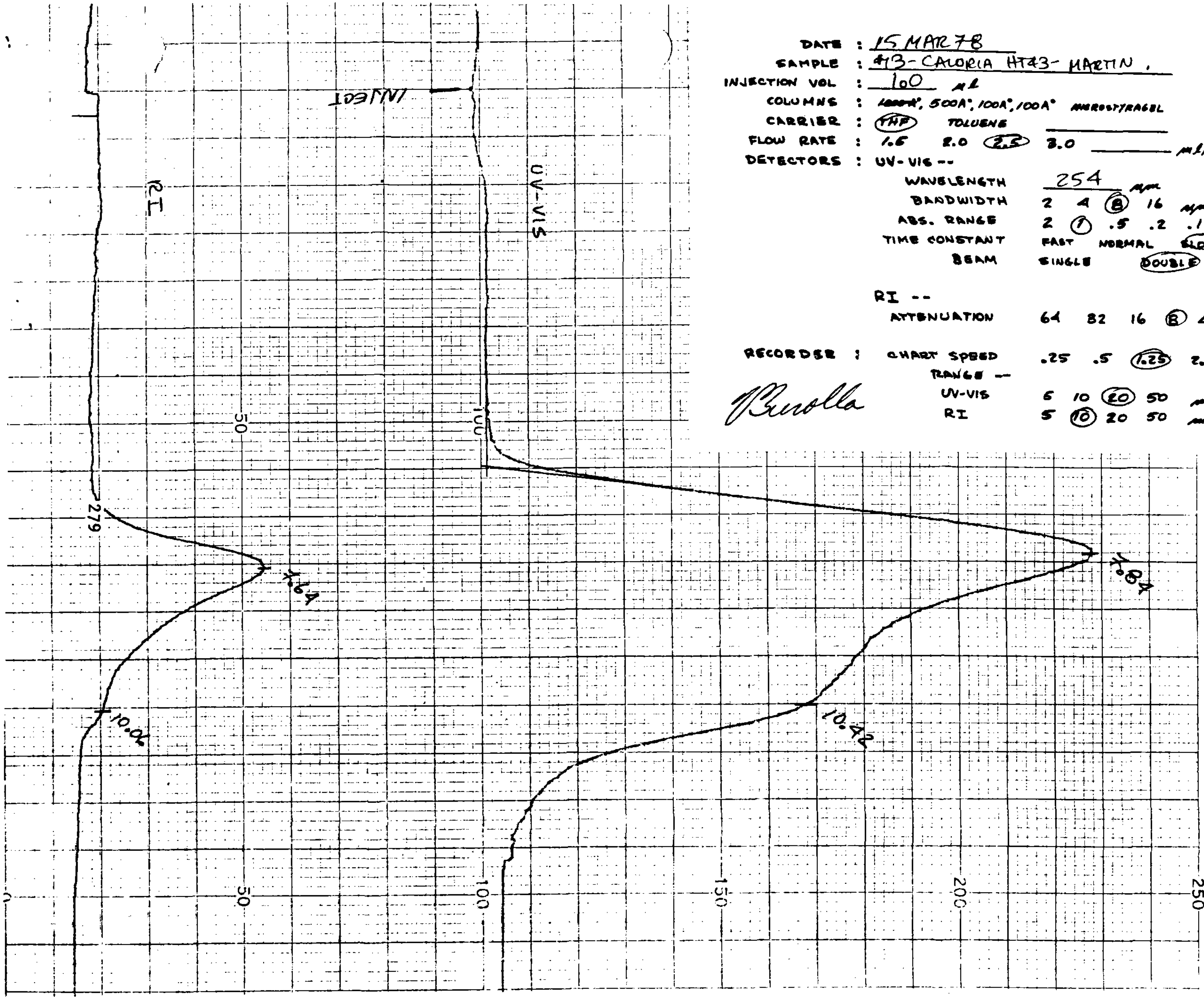


DATE : 15 MAR 78
SAMPLE : #13-CALORIA HT43- MARTIN .
INJECTION VOL : 100 μl
COLUMNS : ~~1000A°~~, 500A°, 100A°, 100A° MICROSTYRAGEL
CARRIER : [✓ THF] TOLUENE ________
FLOW RATE : 1.6 2.0 [✓ 2.5] 3.0 ________ ml/min
DETECTORS : UV-VIS --
WAVELENGTH 254 nm
BANDWIDTH 2 4 [✓ 8] 16 nm
ABS. RANGE 2 [✓ 1] .5 .2 .1 .05
TIME CONSTANT FAST NORMAL [✓ SLOW]
BEAM SINGLE [✓ DOUBLE]

RI --
ATTENUATION 64 32 16 [✓ 8] 4 2 -

RECORDER : CHART SPEED .25 .5 [✓ 1.25] 2.5 cm/min
RANGE --
UV-VIS 5 10 [✓ 20] 50 mV/FS
RI 5 [✓ 10] 20 50 mV/FS

Burolla

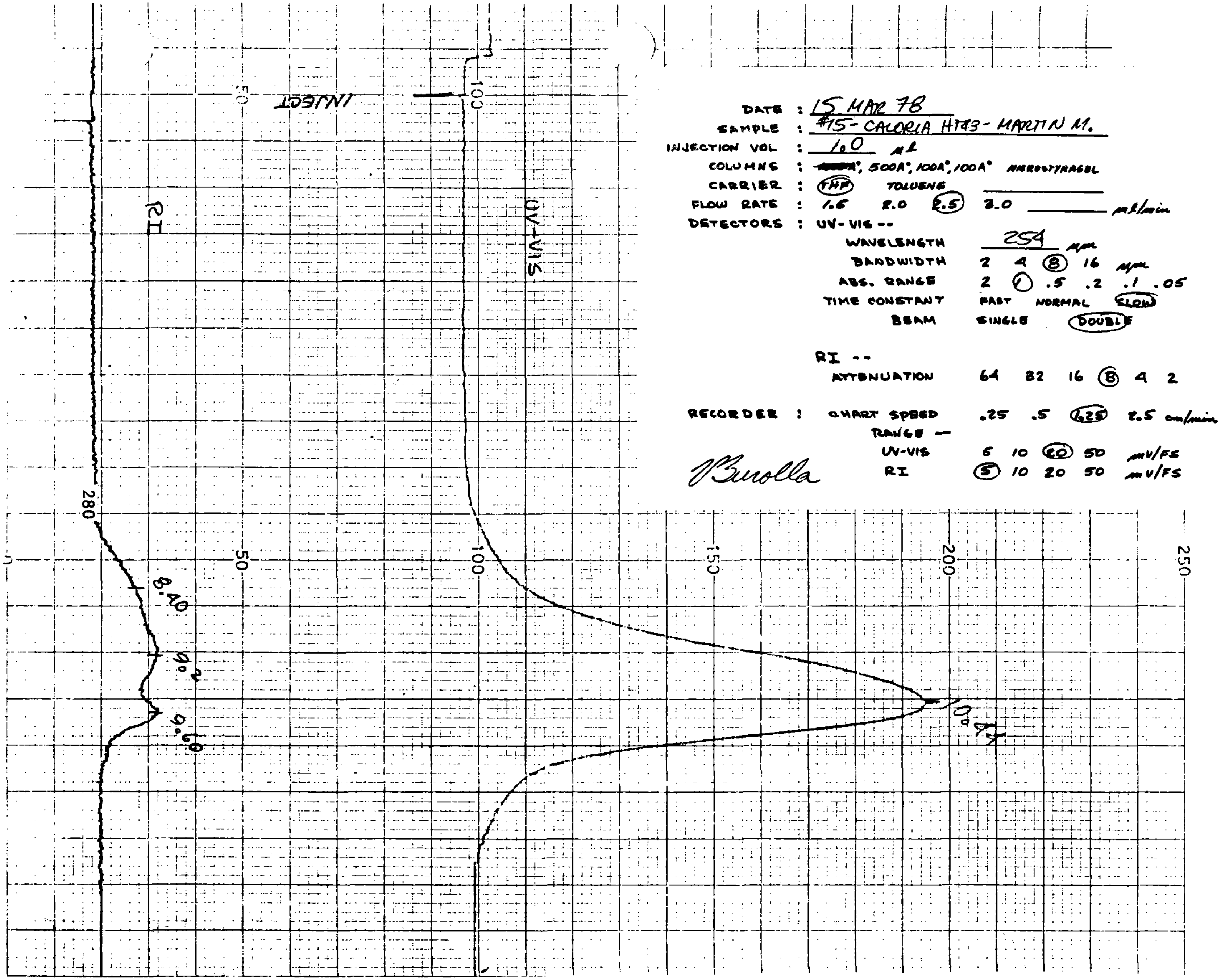


DATE : 15 MAR 78
SAMPLE : #15- CALORIA HT43- MARTIN M.
INJECTION VOL : 1.0 μl
COLUMNS : ~~[illegible]~~, 500A°, 100A°, 100A° μSTYRAGEL
CARRIER : [✓ THF] TOLUENE ________
FLOW RATE : 1.6 2.0 [✓ 2.5] 3.0 ________ ml/min
DETECTORS : UV-VIS --
WAVELENGTH 254 nm
BANDWIDTH 2 4 [✓ 8] 16 nm
ABS. RANGE 2 [✓ 1] .5 .2 .1 .05
TIME CONSTANT FAST NORMAL [✓ SLOW]
BEAM SINGLE [✓ DOUBLE]

RI --
ATTENUATION 64 32 16 [✓ 8] 4 2

RECORDER : CHART SPEED .25 .5 [✓ 1.25] 2.5 cm/min
RANGE --
UV-VIS 5 10 [✓ 20] 50 mV/FS
RI [✓ 5] 10 20 50 mV/FS

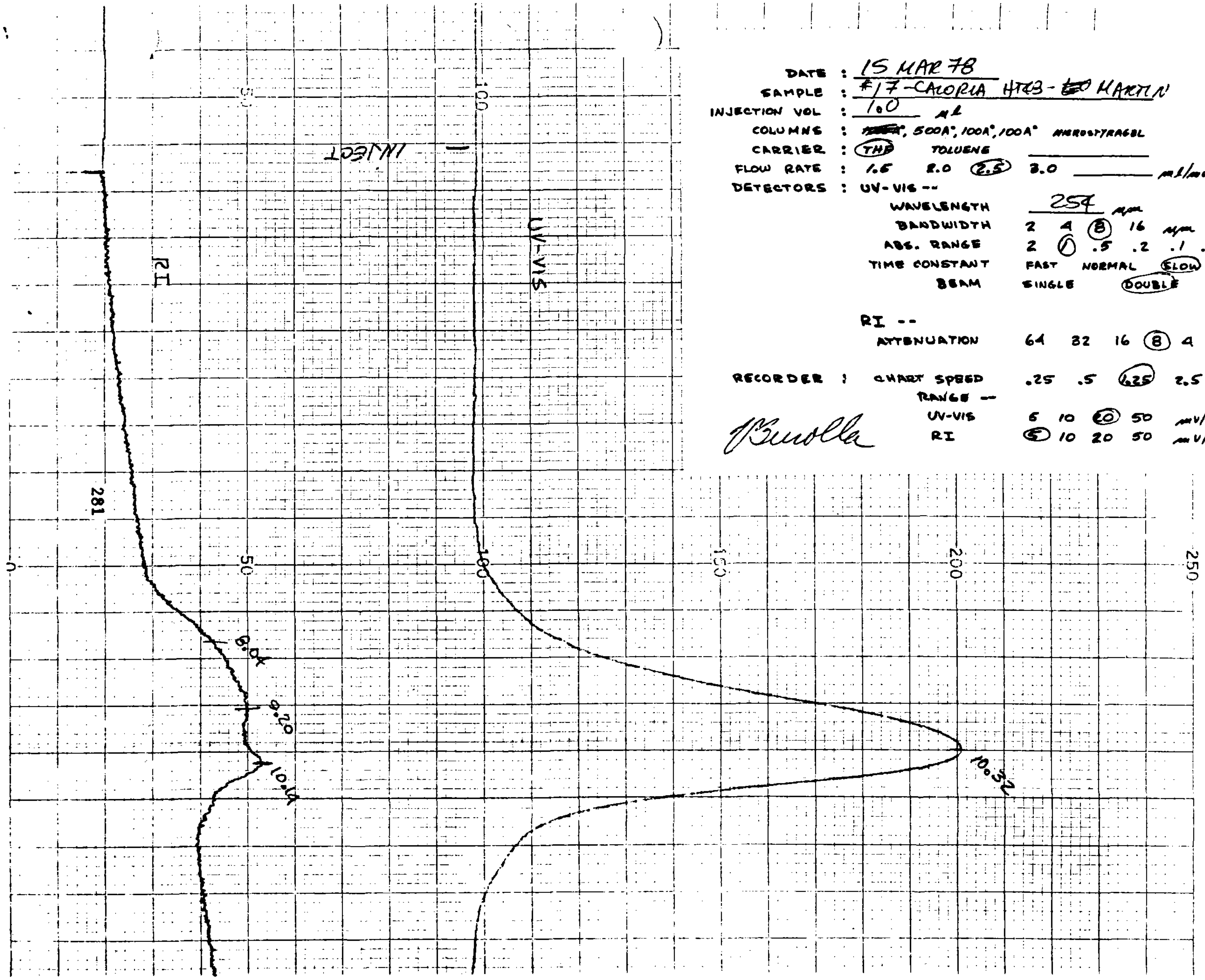
Burolla



DATE : 15 MAR 78
SAMPLE : #17-CALORIA HT43 - MARTIN
INJECTION VOL : 100 μl
COLUMNS : 500A°, 100A°, 100A° μSTYRAGEL
CARRIER : [✓ THF] TOLUENE ______
FLOW RATE : 1.6 2.0 [✓ 2.5] 3.0 ______ ml/min
DETECTORS : UV-VIS --
WAVELENGTH 254 nm
BANDWIDTH 2 4 [✓ 8] 16 nm
ABS. RANGE 2 [✓ 1] .5 .2 .1 .05
TIME CONSTANT FAST NORMAL [✓ SLOW]
BEAM SINGLE [✓ DOUBLE]

RI --
ATTENUATION 64 32 16 [✓ 8] 4 2

RECORDER : CHART SPEED .25 .5 [✓ 1.25] 2.5 cm/min
RANGE --
UV-VIS 5 10 [✓ 20] 50 mV/FS
RI [✓ 5] 10 20 50 mV/FS

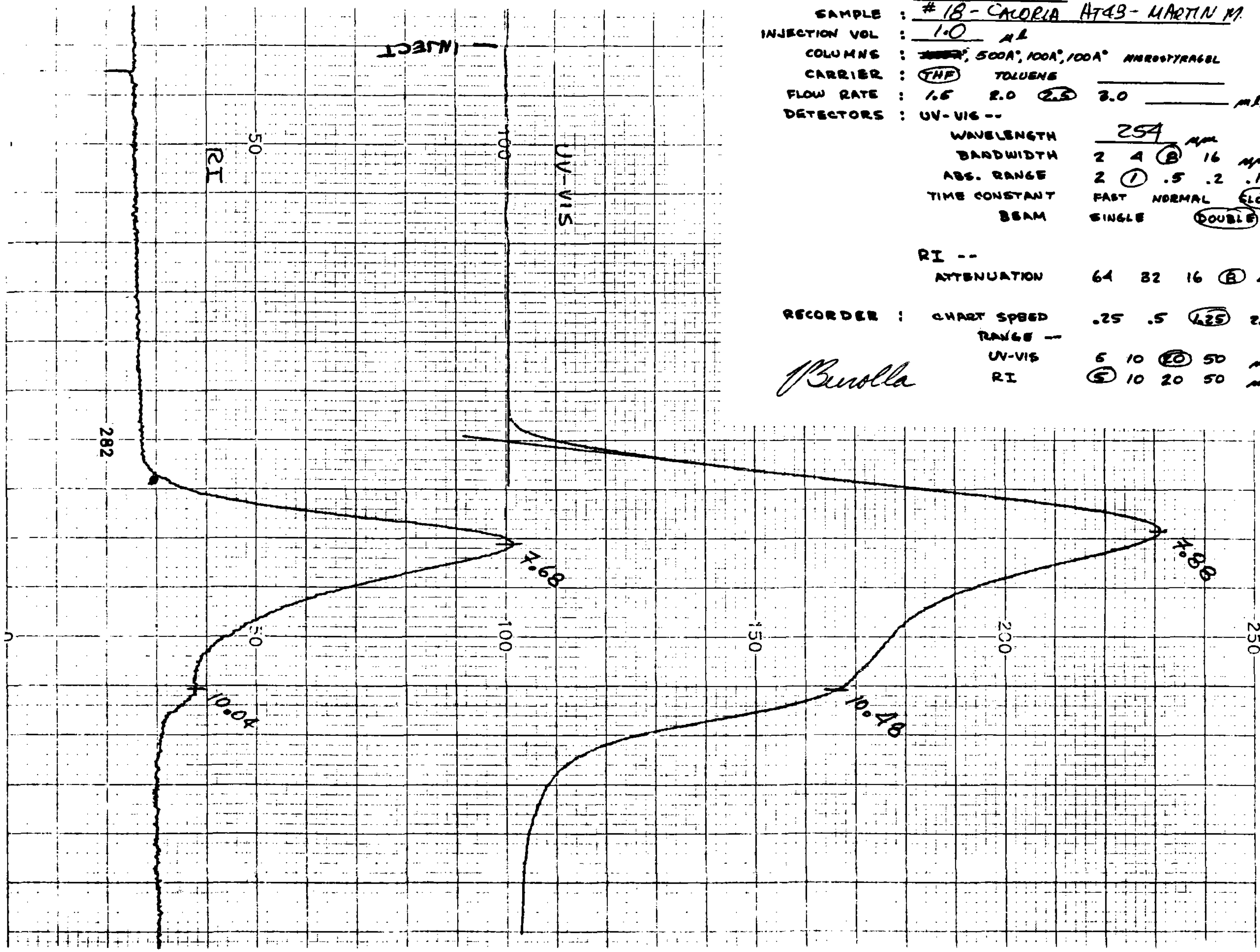


DATE : 14 MAR 78
SAMPLE : # 18 - CALORIA AT43 - MARTIN M.
INJECTION VOL : 1.0 μl
COLUMNS : ~~[illegible]~~, 500A°, 100A°, 100A° μSTYRAGEL
CARRIER : [✓ THF] TOLUENE ________
FLOW RATE : 1.6 2.0 [✓ 2.5] 3.0 ________ ml/min
DETECTORS : UV-VIS --
WAVELENGTH 254 nm
BANDWIDTH 2 4 [✓ 8] 16 nm
ABS. RANGE 2 [✓ 1] .5 .2 .1 .05
TIME CONSTANT FAST NORMAL [✓ SLOW]
BEAM SINGLE [✓ DOUBLE]

RI --
ATTENUATION 64 32 16 [✓ 8] 4 2

RECORDER : CHART SPEED .25 .5 [✓ 1.25] 2.5 cm/min
RANGE --
UV-VIS 5 10 [✓ 20] 50 mV/FS
RI [✓ 5] 10 20 50 mV/FS

Burolla



DATE : 3 OCT 78.

SAMPLE : 427 CACORIA - MARTIN MARIETTA

INJECTION VOL : 100 µℓ

COLUMNS : ~~1000A°~~, 500A°, 100A°, 100A° MICROSTYRAGEL

CARRIER : [✓ THF] TOLUENE ____

FLOW RATE : 1.5 2.0 [✓ 2.5] 3.0 ____ mℓ/min

DETECTORS : UV-VIS --

WAVELENGTH 254 nm

BANDWIDTH 2 4 [✓ 8] 16 nm

ABS. RANGE 2 [✓ 1] .5 .2 .1 .05

TIME CONSTANT FAST NORMAL [✓ SLOW]

BEAM SINGLE [✓ DOUBLE]

RI --

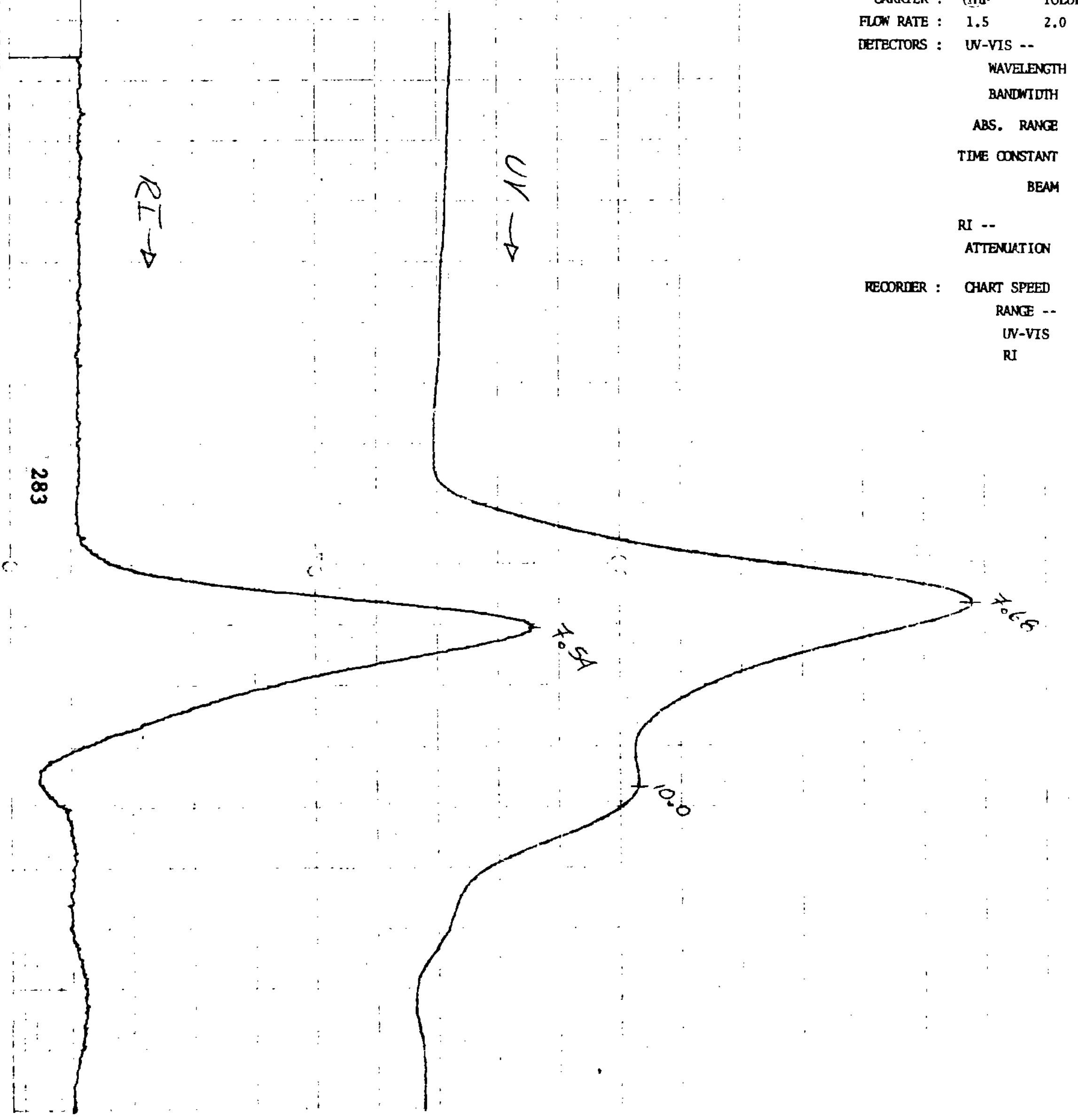
ATTENUATION 64 32 16 [✓ 8] 4 2

RECORDER : CHART SPEED .25 .5 [✓ 1.25] 2.5 cm/min

RANGE --

UV-VIS 5 10 [✓ 20] 50 mv/FS

RI [✓ 5] 10 20 50 mv/FS



DATE : 3 Oct 78

SAMPLE : #29 Calcium [illegible] [illegible]

INJECTION VOL : 10 μℓ

COLUMNS : ~~1000A°~~, 500A°, 100A°, 100A° MICROSTYRAGEL

CARRIER : [✓ THE] TOLUENE ______

FLOW RATE : 1.5 2.0 [✓ 2.5] 3.0 ______ mℓ/min

DETECTORS : UV-VIS --

WAVELENGTH 254 nm

BANDWIDTH 2 4 [✓ 8] 16 nm

ABS. RANGE 2 [✓ 1] .5 .2 .1 .05

TIME CONSTANT FAST NORMAL [✓ SLOW]

BEAM SINGLE [✓ DOUBLE]

RI --

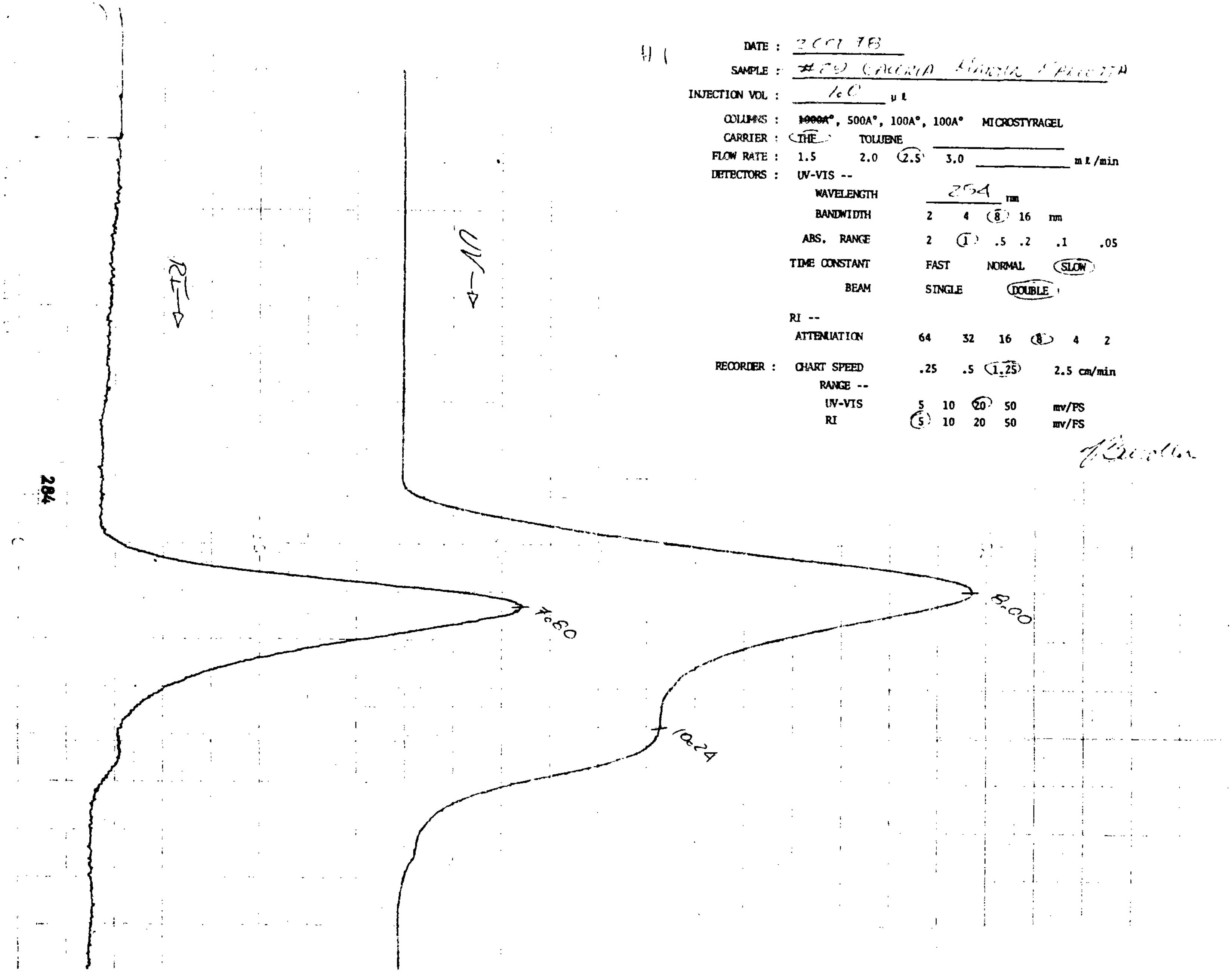
ATTENUATION 64 32 16 [✓ 8] 4 2

RECORDER : CHART SPEED .25 .5 [✓ 1.25] 2.5 cm/min

RANGE --

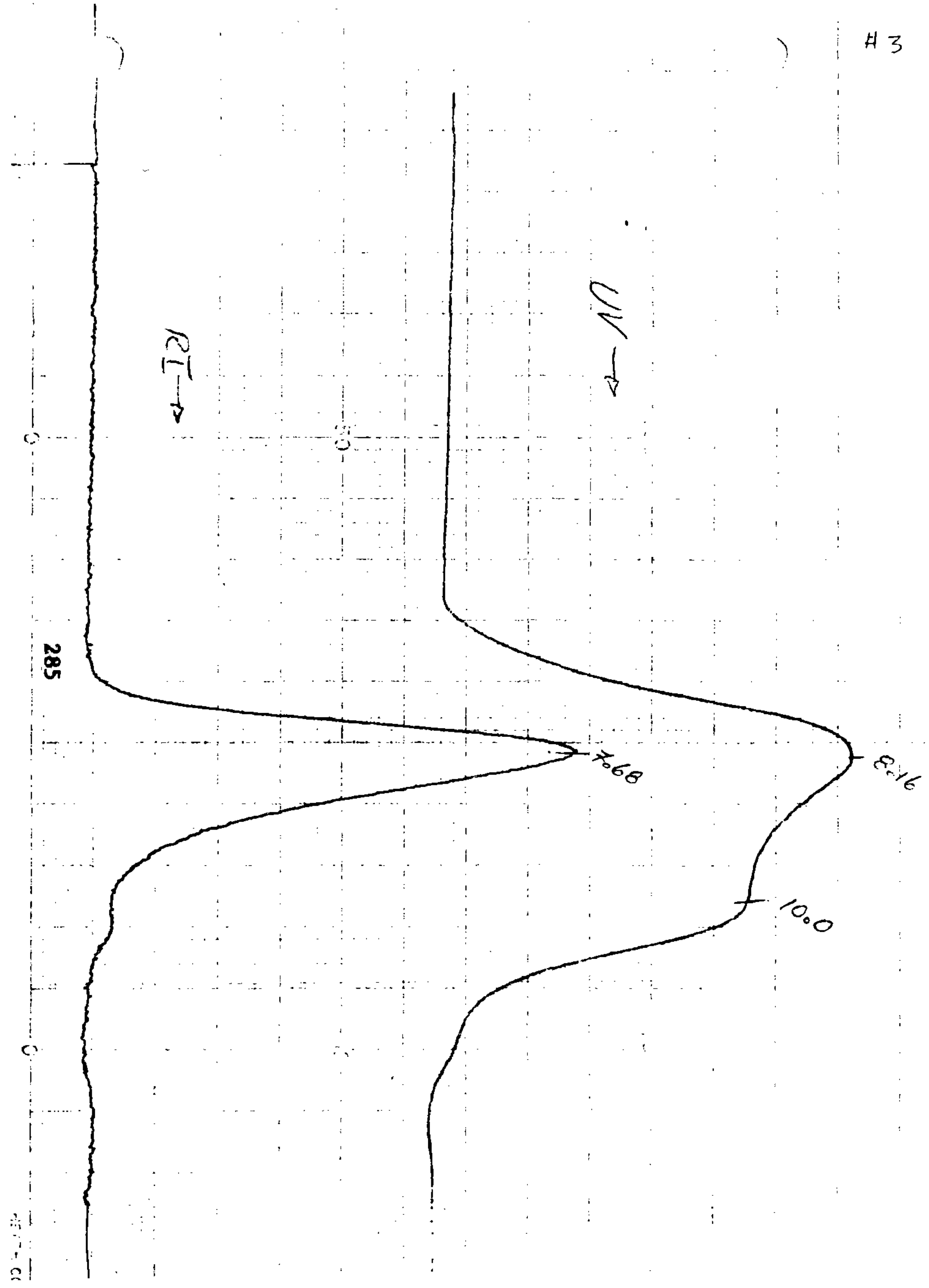
UV-VIS 5 10 [✓ 20] 50 mv/FS

RI [✓ 5] 10 20 50 mv/FS



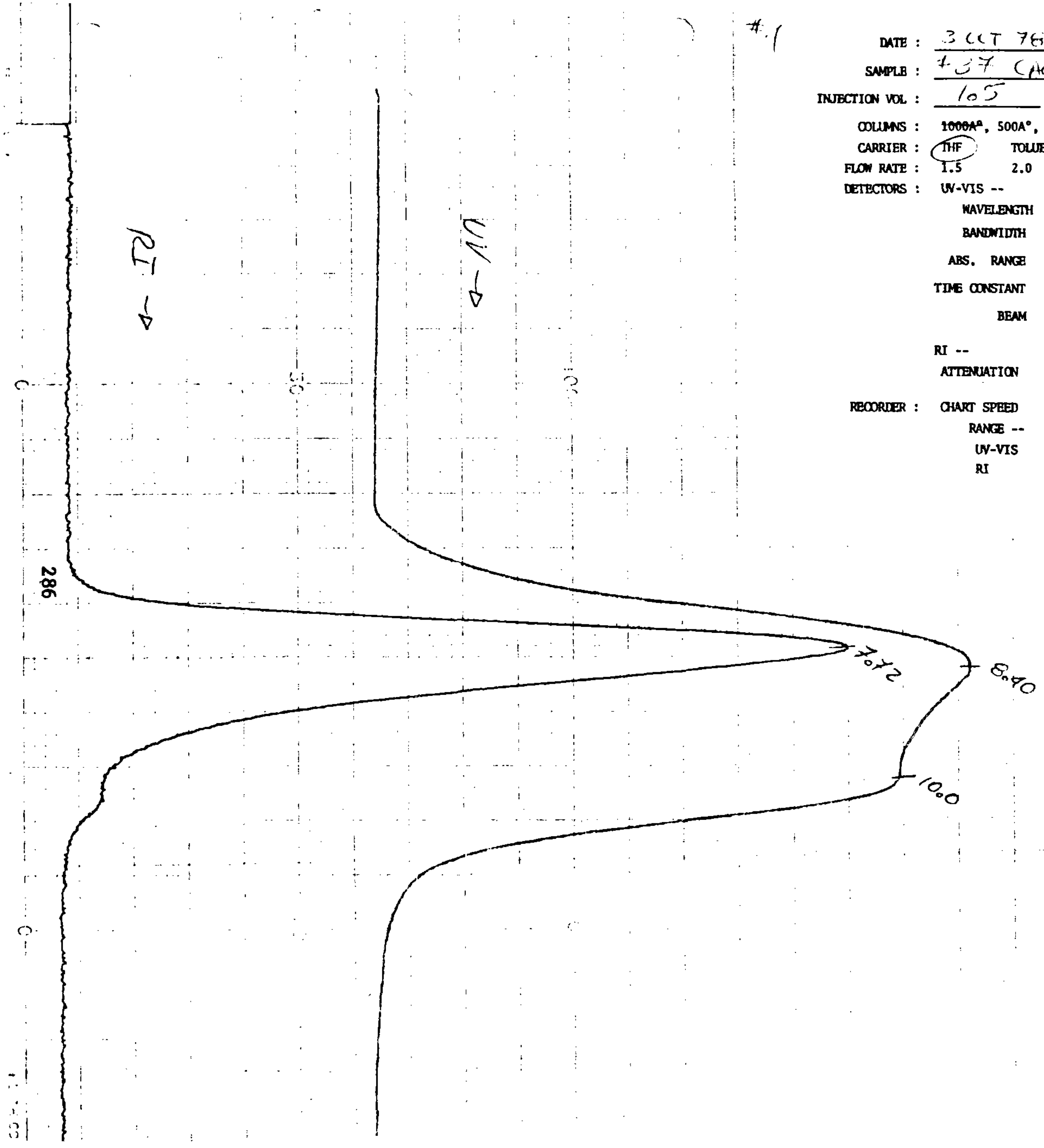
#3

DATE : 3 OCT 76
SAMPLE : # 26 CALORIA MARTIN MARIETTA
INJECTION VOL : 100 μℓ
COLUMNS : ~~1000A°~~, 500A°, 100A°, 100A° MICROSTYRAGEL
CARRIER : [✓ THF] TOLUENE ______
FLOW RATE : 1.5 2.0 [✓ 2.5] 3.0 ______ mℓ/min
DETECTORS : UV-VIS --
WAVELENGTH 254 nm
BANDWIDTH 2 4 [✓ 8] 16 nm
ABS. RANGE 2 [✓ 1] .5 .2 .1 .05
TIME CONSTANT FAST NORMAL [✓ SLOW]
BEAM SINGLE [✓ DOUBLE]
RI --
ATTENUATION 64 32 16 [✓ 8] 4 2
RECORDER : CHART SPEED .25 .5 [✓ 1.25] 2.5 cm/min
RANGE --
UV-VIS 5 10 [✓ 20] 50 mv/FS
RI [✓ 5] 10 20 50 mv/FS



#1

DATE : 3 OCT 76
SAMPLE : 437 CACCIA MARTIN MARIETTA
INJECTION VOL : 105 μℓ
COLUMNS : ~~1000A°~~, 500A°, 100A°, 100A° MICROSTYRAGEL
CARRIER : [✓ THF] TOLUENE ______
FLOW RATE : 1.5 2.0 [✓ 2.5] 3.0 ______ mℓ/min
DETECTORS : UV-VIS --
WAVELENGTH 254 nm
BANDWIDTH 2 4 [✓ 8] 16 nm
ABS. RANGE 2 [✓ 1] .5 .2 .1 .05
TIME CONSTANT FAST NORMAL [✓ SLOW]
BEAM SINGLE [✓ DOUBLE]
RI --
ATTENUATION 64 32 16 [✓ 8] 4 2
RECORDER : CHART SPEED .25 .5 [✓ 1.25] 2.5 cm/min
RANGE --
UV-VIS 5 10 [✓ 20] 50 mv/FS
RI [✓ 5] 10 20 50 mv/FS

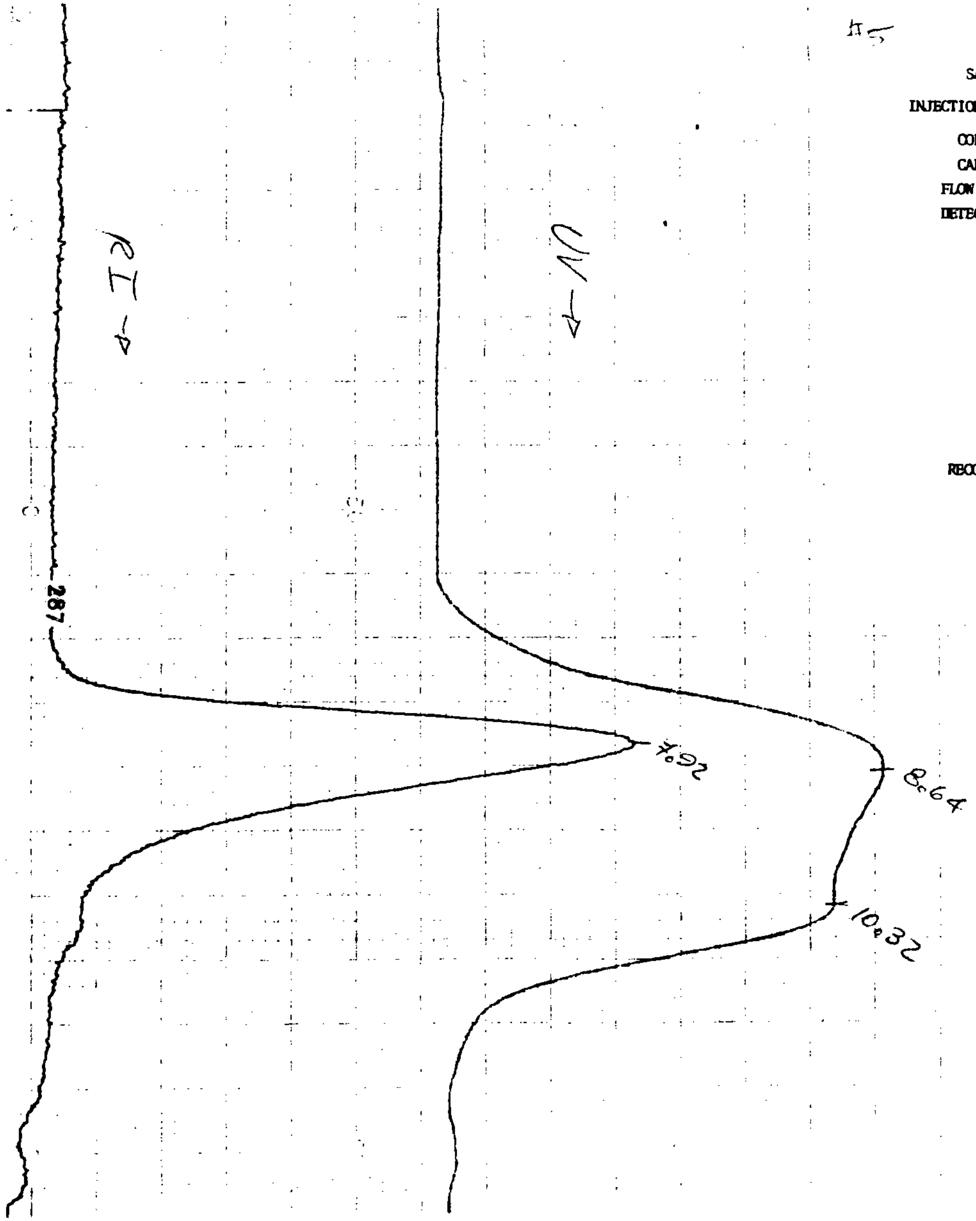


#5

DATE : 3 OCT 78
SAMPLE : #57 CACORIA MARINO
INJECTION VOL : 1.0 µℓ
COLUMNS : ~~1000A~~°, 500A°, 100A°, 100A° MICROSTYRAGEL
CARRIER : [✓ THF] TOLUENE ______
FLOW RATE : 1.5 2.0 [✓ 2.5] 3.0 ______ mℓ/min
DETECTORS : UV-VIS --
WAVELENGTH 254 nm
BANDWIDTH 2 4 [✓ 8] 16 nm
ABS. RANGE 2 [✓ 1] .5 .2 .1 .05
TIME CONSTANT FAST NORMAL [✓ SLOW]
BEAM SINGLE [✓ DOUBLE]

RI --
ATTENUATION 64 32 16 [✓ 8] 4 2

RECORDER : CHART SPEED .25 .5 [✓ 1.25] 2.5 cm/min
RANGE --
UV-VIS 5 10 [✓ 20] 50 mv/FS
RI [✓ 5] 10 20 50 mv/FS

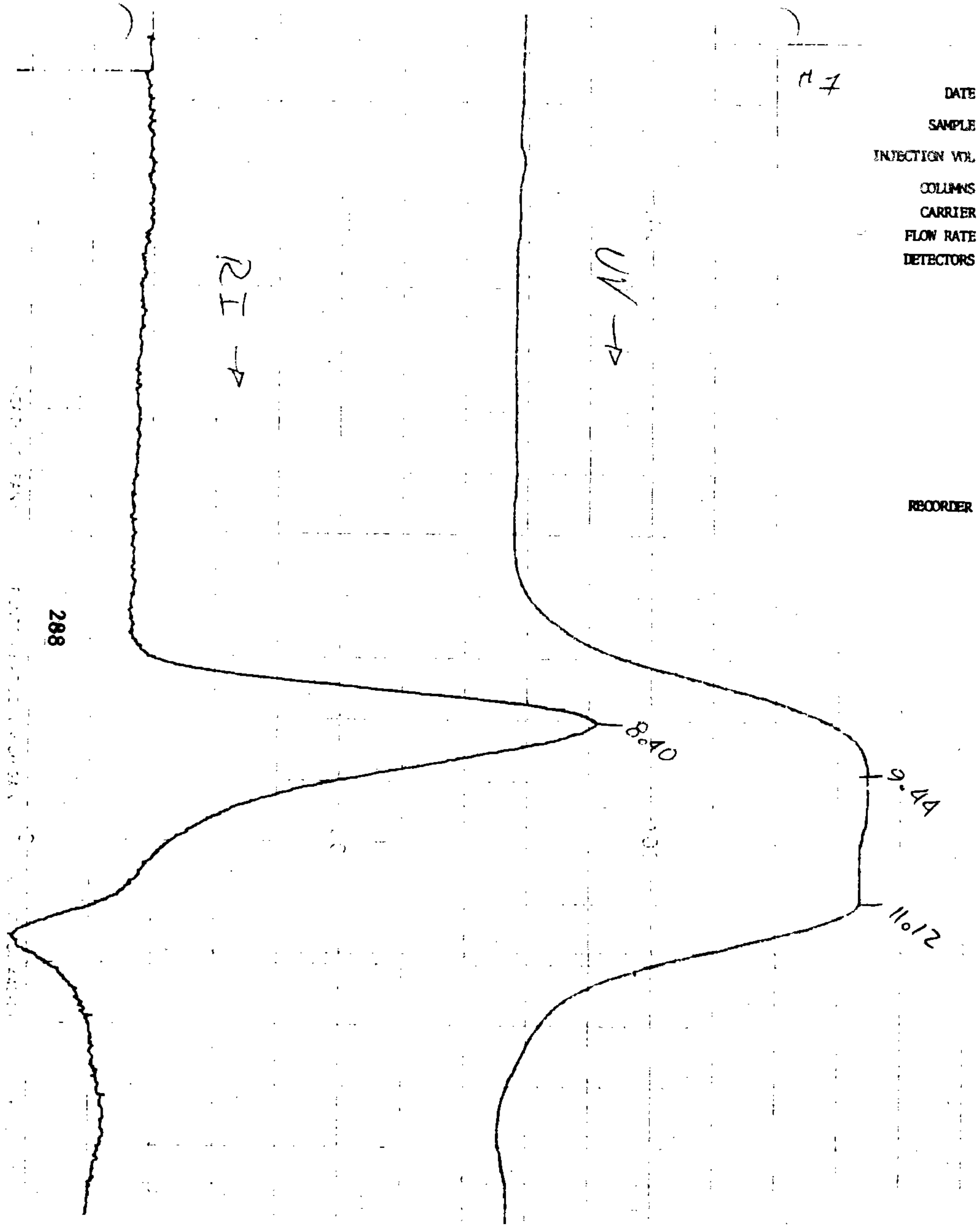


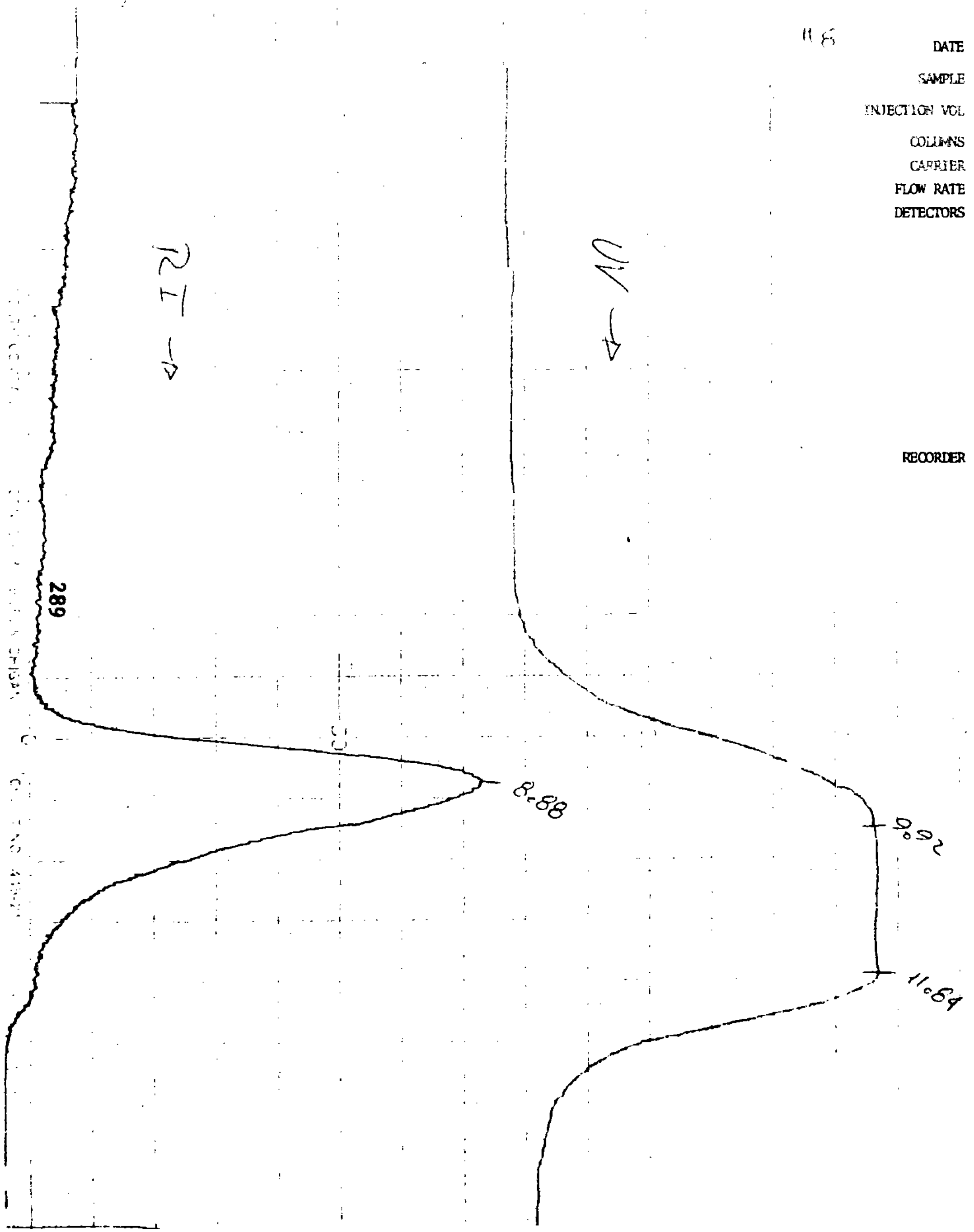
#7

DATE : 3 OCT 78
SAMPLE : #.11 CALCRIA MARTIN
INJECTION VOL. : 100 µℓ
COLUMNS : ~~1000A°~~, 500A°, 100A°, 100A° MICROSTYRAGEL
CARRIER : [✓ THF] TOLUENE ______
FLOW RATE : 1.5 2.0 [✓ 2.5] 3.0 ______ mℓ/min
DETECTORS : UV-VIS --
WAVELENGTH 254 nm
BANDWIDTH 2 4 [✓ 8] 16 nm
ABS. RANGE 2 [✓ 1] .5 .2 .1 .05
TIME CONSTANT FAST NORMAL [✓ SLOW]
BEAM SINGLE [✓ DOUBLE]

RI --
ATTENUATION 64 32 16 [✓ 8] 4 2

RECORDER : CHART SPEED .25 .5 [✓ 1.25] 2.5 cm/min
RANGE --
UV-VIS 5 10 [✓ 20] 50 mv/FS
RI [✓ 5] 10 20 50 mv/FS





11 8

DATE : 3 OCT 78

SAMPLE : #45 CALCIA [illegible]

INJECTION VOL : 1.0 µl

COLUMNS : ~~1000A°~~, 500A°, 100A°, 100A° MICROSTYRAGEL

CARRIER : [✓ THF] TOLUENE ______

FLOW RATE : 1.5 2.0 [✓ 2.5] 3.0 ______ ml/min

DETECTORS : UV-VIS --

WAVELENGTH 254 nm

BANDWIDTH 2 4 [✓ 8] 16 nm

ABS. RANGE 2 [✓ 1] .5 .2 .1 .05

TIME CONSTANT FAST NORMAL [✓ SLOW]

BEAM SINGLE [✓ DOUBLE]

RI --

ATTENUATION 64 32 16 [✓ 8] 4 2

RECORDER : CHART SPEED .25 .5 [✓ 1.25] 2.5 cm/min

RANGE --

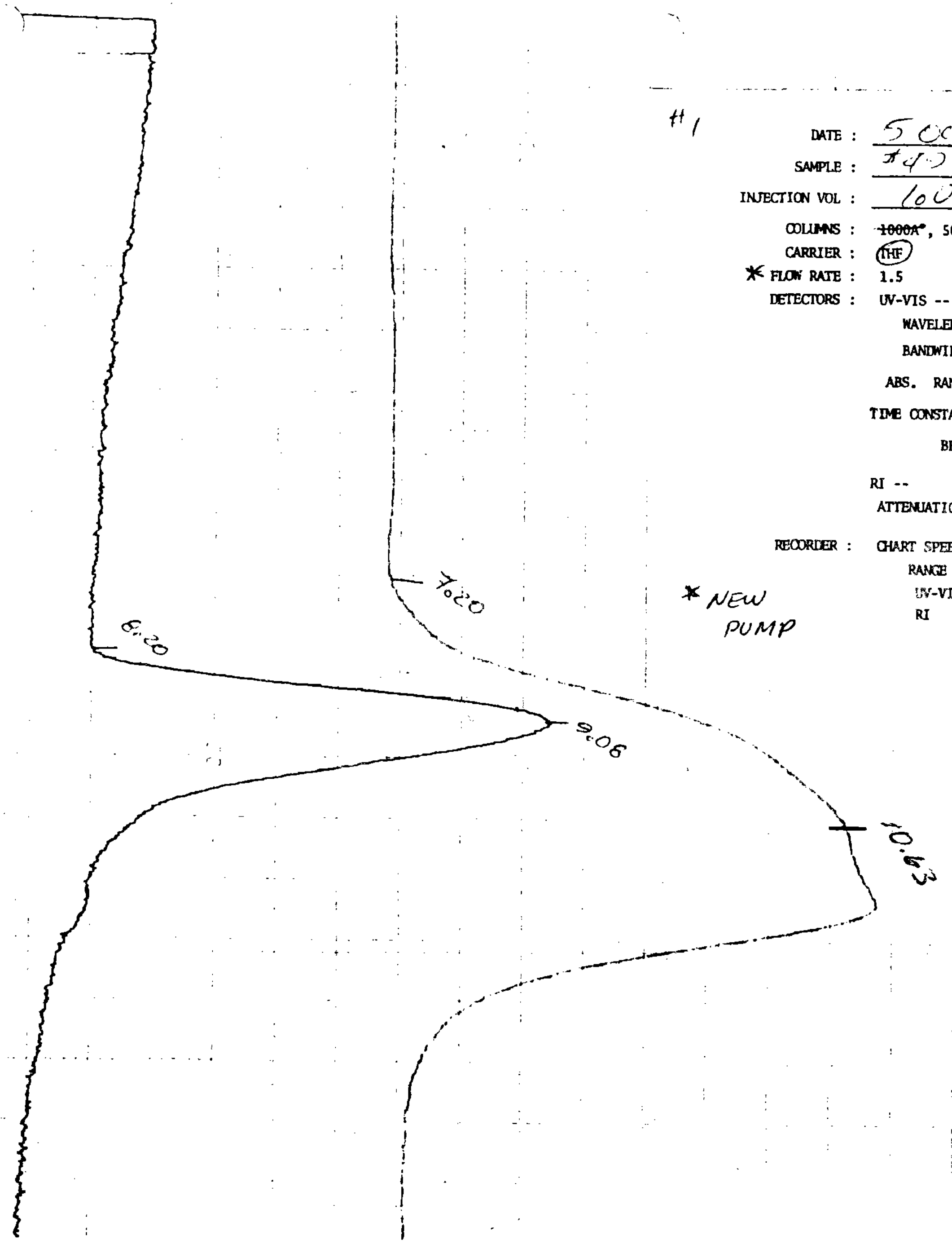
UV-VIS 5 10 [✓ 20] 50 mv/FS

RI [✓ 5] 10 20 50 mv/FS

#1

DATE : 5 OCT 78
SAMPLE : #40 MARTIN CACCIA
INJECTION VOL : 100 μℓ
COLUMNS : ~~1000A°~~, 500A°, 100A°, 100A° MICROSTYRAGEL
CARRIER : [✓ THF] TOLUENE
* FLOW RATE : 1.5 2.0 [✓ 2.5] 3.0 mℓ/min
DETECTORS : UV-VIS --
WAVELENGTH 254 nm
BANDWIDTH 2 4 [✓ 8] 16 nm
ABS. RANGE 2 [✓ 1] .5 .2 .1 .05
TIME CONSTANT FAST NORMAL [✓ SLOW]
BEAM SINGLE [✓ DOUBLE]
RI --
ATTENUATION 64 32 16 [✓ 8] 4 2
RECORDER : CHART SPEED .25 .5 [✓ 1.25] 2.5 cm/min
RANGE --
UV-VIS 5 10 [✓ 20] 50 mv/FS
RI [✓ 5] 10 20 50 mv/FS

* NEW PUMP



Gas Chromatograph/Mass Spectrometer Tests

GC/MS MEAN MOLECULAR WEIGHT DETERMINATIONS

Volatile components in tank vent gas, tank condensate trap, low boilers separated from the Phase II processor, and bulk oil samples were analyzed. Separation was performed on 60/80 mesh Tenax-GC, a high temperature porous polymer support material. The GC oven was temperature programmed from 90-220 or 280°C; the injection port at 225°C. The higher upper limit was used for liquid samples. Other instrumental parameters were:

Flow Rate (ml/min)	25
MS Peak Detect Threshold	2000
Samples per 0.1 AMU	2
Electron Multiplier	1800
GC Peak Detect Threshold	1000

From the mass chromatogram it was possible to assign a component identification and ion abundance for the peaks. Mean molecular weights were calculated as follows:

$$\text{Mean Molecular Weight (g/mole)} = \frac{\left(\text{Peak Abundance} \times \begin{array}{c}\text{Assigned}\\ \text{Molecular}\\ \text{Weight}\end{array}\right)}{\text{Total Abundance}}$$

GS/MS DATA

SAMPLE NO. 4
SAMPLE SIZE: 0.5cc

SPECTRUM NO.	NO. CARBONS	RETENTION TIME (MIN)	ABUNDANCE	COMPOUND	MOLECULAR WEIGHT
16	1	0.3	14129	CO_2	44
17	2	0.7	163450	ethane	30
18	0	1.2	5276	H_2O	18
19	3	2.3	74874	propane	44
23	4	5.2	136969	butane	58
24	5	5.9	14180	iso-pentane	72
36 (29)	12	7.3	1764*	dodecane	170
25	5	6.1	68413	pentane	72
28	6	6.8	43717	hexane	86
26 (34)	5	6.3	5935*	pentene	72
30	7	7.5	13918	heptane	100
31 (37)	11-14	8.0	750*	undecane	156
32	8	8.3	7175	octane	114
33	12	9.1	4666	170 M.W. Bleed dodecane	

* Subtracted previous large peak

GC/MS DATA

SAMPLE NO. 5
SAMPLE SIZE:

SPECTRUM NO.	NO. CARBONS	RETENTION TIME (MIN)	ABUNDANCE	COMPOUND	MOLECULAR WEIGHT
3	2	0.6	143231	ethane	30
4	0	0.9	4686	H_2O	18
5	3	1.6	92146	propane	44
7	4	3.2	4225	iso-butane	58
8	4	4.0	65294	butane	58
11	5	5.7	50403	pentane	72
12 (41)	5	6.0	7252	1-pentene	72
14	6	6.8	42407	hexane	86
15 (43)	12	7.3	1858	170 M.W.	
16 (44)	7	7.6	,20603	heptane	100
18 (46)	14	8.1	2298	198	
19 (58)	8	8.3	21790	octane	114
22 (55)	?	9.1	841	aromatic	
23 (56)	10	9.3	1408	decane	142
26 (47)	7	10.1	3495	heptene	98
27	?	10.6	11391	Hydrocarbon (octane)	114
29 (48)	9	11.6	4512	Nonane	128
30 (49)	16c's	12.1	12333	226.0 M.W.	
31 (50)	12c's	12.6	16090	Bleed + H.Carb.	170
32 (51)	17c's	12.8	3841	240.0 M.W.	
33 (52)	18c's	14.0	7016	254	
34 (53)	22c's/ oxygen	14.7	5409	326	

GC/MS DATA

SAMPLE NO. 7
SAMPLE SIZE: 0.5 cc

SPECTRUM NO.	NO. CARBONS	RETENTION TIME (MIN)	ABUNDANCE	COMPOUND	MOLECULAR WEIGHT
4	2	0.6	2876	ethane + air	
5	0	0.9	3594	H_2O	
6	3	1.5	3124	propane	
7	4	3.3	2458	butane	
8	7	6.9	2422	heptane	
9		9.1	2971	Bleed	
10	?	11.4	3265	?	

GC/MS DATA

SAMPLE NO. 8
SAMPLE SIZE: 0.5 cc

SPECTRUM NO.	NO. CARBONS	RETENTION TIME (MIN)	ABUNDANCE	COMPOUND	MOLECULAR WEIGHT
3	2	0.8	3788	ethane	30
4	0	1.5	1183	H_2O	18
5	3	2.3	5261	propane	
6	4	4.3	5100	butane	
7	5	5.6	4711	pentane	
8	6-7	6.3	2783	96.0	
9	6	6.6	4808	hexane	
10	7	7.2	3147	iso-heptane	
11	7	7.4	6471	heptane	
12	8	7.9	4854	iso-octane	
13	8	8.1	6128	octane	
1	1	0.3	3830	CO_2	44
2	-	0.3	94005	air	

GC/MS DATA

SAMPLE NO. 9
SAMPLE SIZE: 0.5 cc

SPECTRUM NO.	NO. CARBONS	RETENTION TIME (MIN)	ABUNDANCE	COMPOUND	MOLECULAR WEIGHT
18	2	0.6	3683	ethane	
19	3	1.4	5884	propane	
20	4	3.4	4083	butane	
21	5	5.4	3708	pentane	
22	6	6.6	2417	hexane	
23	7	7.4	2255	heptane	

GC/MS DATA

SAMPLE NO. 10
SAMPLE SIZE: 0.1cc

SPECTRUM NO.	NO. CARBONS	RETENTION TIME (MIN)	ABUNDANCE	COMPOUND	MOLECULAR WEIGHT
3	2	0.5	2472	acetaldehyde	
4	2	0.8	148300	ethane	
5	3	2.1	345763	propane	
6	4	3.4	49462	butane	
7	4	4.2	16852	iso butane	
8	5	5.4	5231	pentane	
9	6-7	6.3	1880	?	
10	6	6.5	3273	hexane	
11	7	7.3	2492	heptane	

GC/MS DATA

SAMPLE NO. 11
SAMPLE SIZE: 0.5 cc

SPECTRUM NO.	NO. CARBONS	RETENTION TIME (MIN)	ABUNDANCE	COMPOUND	MOLECULAR WEIGHT
2	1	0.6	4589	CO_2	
3	2	0.9	13968	ethane	
4	3	2.4	19945	propane	
5	4	4.2	12689	butane	
6	4	4.5	2934	butene	
7	5	5.4	2442	iso-pentane	
8	5.8	5.8	9640	pentane	
9	5	6.1	5143	pentene	
10	6	6.6	4418	hexane	
				C = C-C-C-C-C = C	
11	6	6.9	8552	hexane	
12	7	7.4	4587	heptane	
13	7	7.7	9714	heptane	
14	8	8.2	6487	octane	
15	8	8.4	8190	Octane	
16	9	8.9	5339	nonane	

GC/MS DATA

SAMPLE NO. 12
SAMPLE SIZE: 0.1

SPECTRUM NO.	NO. CARBONS	RETENTION TIME (MIN)	ABUNDANCE	COMPOUND	MOLECULAR WEIGHT
1	-	0.4	1602	air	
2	3	0.6	5178	propane	
3	4	0.8	2367	init. butane	
4	4	0.9	19653	butane	
5	5	1.5	5573	pentane	
6	5	1.7	26419	pentane	
7	5	2.0	12736	pentene	
8	6	2.6	12308	hexane	
9	6	2.8	39734	hexane	
10	7	3.6	28520	heptane	
11	7	3.8	92515	heptane	
12	8	4.6	77669	octane	
13	8	4.8	127616	octane	
14	9	5.3	61566	nonane	
15	9	5.5	87245	nonane	
16	9	5.8	128056	nonane	
17	9-10	6.1	63728	nonene (decane)	
18	9-10	6.3	96379	nonane (2 unsat) decane	
19	10	6.6	130006	decane	
20	11	7.3	107546	undecane	
21	12	7.9	102496	dodecane	
22	-				
23	13	8.5	104532	13 chain	
24	13	8.8	89940	1 unsat.	
26	14	9.1	92717	-	
28	14	9.7	81296	-	

GC/MS DATA

SAMPLE NO. 12
SAMPLE SIZE: 0.1

SPECTRUM NO.	NO. CARBONS	RETENTION TIME (MIN)	ABUNDANCE	COMPOUND	MOLECULAR WEIGHT
1	2	0.6	1977	ethane	
2	3	0.9	18334	propane	
3	4	1.7	2316	(iso-) butane	
4	4	2.4	29500	butane	
5	4	2.9	4689	butene	
9	5	5.1	9095	(iso-) pentane	
10	5	5.5	74306	pentane	
11	5	5.9	43735	pentene	
12	6	6.7	60822	hexane	
13	6	6.9	214247	hexane	
14	7	7.8	526155	heptane	
15	8	8.3	481910	octane	
16	8	8.4	625832	octane	
17	9	8.8	525807	nonane	
18	9	8.9	620573	nonane	
19	10	9.4	589814	decane	
10	11	9.9	534412	undecane	
21	11	10.0	411499	11c's aryl alkyl	
22	12	10.3	491055	dodecane	
23	13	10.5	334778	tridecane	
24	13	10.7	377248	tridecane	
25	14	11.1	270945	tetradecane	
26	15	11.7	186088	pentadecane	
27	15-16	11.9	148639	(penta)(hexa)-decane	
28	16	12.3	140544	hexadecane	
29	17	13.2	102920	heptadecane	
32	18	14.4	72718	octadecane	
35	19	16.0	46234	nonadecane	
37	20	18.1	27902	eicosane	

GC/MS DATA

SAMPLE NO. 13
SAMPLE SIZE: 0.5

SPECTRUM NO.	NO. CARBONS	RETENTION TIME (MIN)	ABUNDANCE	COMPOUND	MOLECULAR WEIGHT
1	-	0.4	5439	air + CO_2	
2	2	0.6	21109	ethane	
3	-	0.8	3385	H_2O	
4	3	0.9	77135	propane	
5	4	1.6	3581	(iso) butane	
6	4	2.3	27317	butane	
9	5	5.6	16782	pentane	
10	5	6.1	8478	pentene	
12	6	7.3	7839	(iso) hexane	
13	6	7.6	15035	hexane	
16	7	9.0	12738	heptane	
17	8	9.7	7841	octane	
18	8	9.9	9062	octane	
19	9	10.6	6127	nonane	

GC/MS DATA

SAMPLE NO. 14
SAMPLE SIZE: 0.5

SPECTRUM NO.	NO. CARBONS	RETENTION TIME (MIN)	ABUNDANCE	COMPOUND	MOLECULAR WEIGHT
1	-	0.4	24995	air	
2	2	0.6	148209	ethane	
3	3	0.9	550453	propane	
4	4	1.7	10572	iso-butane	
5	4	2.2	93698	butane	
6	4	2.6	3809	butene	
8	5	4.9	3534	pentane	
9-10-11			bleed	-	

GC/MS DATA

SAMPLE NO. 15
SAMPLE SIZE: 0.1

SPECTRUM NO.	NO. CARBONS	RETENTION TIME (MIN)	ABUNDANCE	COMPOUND	MOLECULAR WEIGHT
1	2	0.6	3727	ethane	
2	3	1.0	24133	propane	
3	4	1.8	3250	iso-butane	
4	4	2.4	39123	butane	
5	4	2.8	5370	butene	
11	5	4.6	8131	iso-pentane	
12	5	5.4	58237	pentane	
13	5	6.0	28963	pentene	
15	6	7.1	37808	(iso) hexane	
16	6	7.4	122924	hexane	
17	7	8.8	286936	heptane	
18	8	9.4	257602	octane	
19	8	9.6	419325	octane	
20	9	10.3	447738	nonane	
21	10	10.6	327674	decane	
22	10	10.8	385062	decane	
23	11	11.1	192918	undecane	
24	11	11.4	270834	undecane	
25	12	11.8	163147	dodecane	
26	12	12.1	179608	dodecane	
28	13	12.9	108644	tridecane	
29	13	13.1	110006	tridecane	
32	14	14.4	62027	tetradecane	
34	15	16.3	35118	pentadecane	

GC/MS DATA

SAMPLE NO. 16
SAMPLE SIZE: 0.5

SPECTRUM NO.	NO. CARBONS	RETENTION TIME (MIN)	ABUNDANCE	COMPOUND	MOLECULAR WEIGHT
2	-	0.4	25588	air + ? (15)	
3	2	0.6	118774	ethane	
4	-	0.9	2292	H_2O + (air?)	
5	3	1.2	242122	propane	
8	4	2.2	9285	iso-butane	
9	4	2.8	95678	butane	
10	4	3.3	10242	butene	
12	5	5.0	13007	iso-pentane	
13	5	5.5	86795	pentane	
14	5	5.9	37316	pentene	
15	6	6.3	7634	hexane	
16	6	6.6	17530	hexane	
17-18	-	7.8	- bleed	-	

GC/MS DATA

SAMPLE NO. 17
SAMPLE SIZE: 0.1

SPECTRUM NO.	NO. CARBONS	RETENTION TIME (MIN)	ABUNDANCE	COMPOUND	MOLECULAR WEIGHT
1	2	0.6	2478	ethane	
2	3	1.0	14729	propane	
3	4	1.8	1820	iso-butane	
4	4	2.6	16959	butane	
8	5	5.7	29901	pentane	
9	5	6.1	15320	pentene	
10	6	6.7	6337	hexane (+ bleed)	
13	6	7.5	64022	hexane	
14	7	8.6	49009	heptane	
15	7	8.9	147596	heptane	
16	8	9.3	50174	octane	
18	8	9.9	264972	octane	
19	9	10.2	135570	nonane	
20	9	10.3	136580	nonane	
22	9	10.8	334100	nonane	
23	10	11.3	269743	decane	
24	10	11.6	380178	decane	
25	11	12.3	324364	undecane	
26	12	13.1	257592	dodecane	
27	13	13.6	151552	tridecane	
29	13	13.9	168222	tridecane	
31	14	15.0	101484	tetradecane	
34	15	16.1	70241	pentadecane	
39	15	17.2	69250	pentadecane	
40	16	19.4	39512	hexadecane	
44	16	20.4	40610	hexadecane	

GC/MS DATA

SAMPLE NO. 20
SAMPLE SIZE: 0.1

SPECTRUM NO.	NO. CARBONS	RETENTION TIME (MIN)	ABUNDANCE	COMPOUND	MOLECULAR WEIGHT
2	-	0.5	2205	air + CO_2	
3	2	0.6	1712	air + ethane	
5	3	0.9	25504	propane	
6	4	1.6	7650	(iso-) butane	
7	4	1.9	68006	butane	
8	4	2.2	13912	butene	
9	5	3.1	30997	(iso) pentane	
10	5	3.3	105822	pentane	
11	5	3.6	65782	pentene	
12	6	4.4	171751	hexane	
13	7	5.1	161151	heptane	
14	7	5.3	280317	heptane	
15	8	5.8	281842	octane	
16	8	5.9	358097	octane	
17	9	6.3	319030	nonane	
18	9	6.4	386906	nonane	
19	10	6.9	382258	decane	
20	11	7.4	345373	undecane	
21	12	7.6	284058	dodecane	
22	12	7.8	326908	dodecane	
23	13	8.0	249041	tridecane	
24	13	8.2	272422	tridecane	
25	14	8.6	242899	tetra-decane	
26	15	8.8	207371	pentadecane	
27	15	8.9	207201	pentadecane	
28	16	9.1	185742	hexadecane	
29	16	9.3	192104	hexadecane	
30	17	9.5	163728	heptadecane	
31	17	9.7	165988	heptadecane	
32	18	9.9	139342	octa decane	
33	18	10.1	142926	octa decane	
34	19	10.7	113266	nonadecane	
35	20	11.4	83892	eicosane	

GC/MS DATA

SAMPLE NO. 31
SAMPLE SIZE: 0.05

SPECTRUM NO.	NO. CARBONS	RETENTION TIME (MIN)	ABUNDANCE	COMPOUND	MOLECULAR WEIGHT
1	0	0.8	12209	H_2O	18
2	7	7.1	1248	heptane	100
3	8	8.1	6251	octane	114
4	9	9.1	27815	nonane	128
5	10	9.8	60293	decane	142
6	11	10.6	81342	undecane	156
7	12	11.2	100002	dodecane	170
8	13	11.8	113107	tridecane	184
9	14	12.4	122172	tetradecane	198
10	15	12.9	130892	pentadecane	211
11	16	13.5	141835	hexadecane	226
14	17	13.9	142560	heptadecane	240
15	17	14.1	140188	heptadecane	240
16	18	14.6	124417	octadecane	254
18	19	15.1	107675	nonadecane	268
22	20	15.7	96786	eicosane	282
26	20	16.2	91356	1 ring possible	282
33	20	16.9		1 ring possible	

GC/MS DATA

SAMPLE NO. 44
SAMPLE SIZE: 0.05

SPECTRUM NO.		RETENTION TIME(MIN)	ABUNDANCE	COMPOUND	MOLECULAR WEIGHT
1	0	0.8	10504	H_2O	18
2	6	4.8	837	hexane	86
3	7	5.7	2010	heptane	100
5	8	6.6	5690	octane	114
6	9	7.4	15509	nonane	128
7	10	8.4	36375	decane	142
8	11	9.3	59649	undecane	156
9	12	10.1	81379	dodecane	170
10	13	11.0	95143	tridecane	184
11	14	11.8	105069	tetradecane	198
14	15	12.6	111751	pentadecane	212
16	16	13.2	101277	hexadecene	226
17	16	13.4	120701	hexadecane	226
20	17	14.2	133421	heptadecane	240
21	18	15.0	111371	octadecane	254
22	19	15.8	100139	nonadecane	268
24	1-20	16.2	83920	nonadecene	268
26	20	16.6	82258	eicosane	282

GC/MS DATA

SAMPLE NO. 52
SAMPLE SIZE: 0.05

SPECTRUM NO.	NO. CARBONS	RETENTION TIME(MIN)	ABUNDANCE	COMPOUND	MOLECULAR WEIGHT
1	0		13363	H_2O	18
2	C_7	7.2	954	heptane	100
4	8	8.3	2205	octane	114
5	9	9.3	7365	nonane	128
6	10	10.1	26414	decane	142
7	11	10.8	64102	undecane	156
8	12	11.4	107367	dodecane	170
9	13	11.9	132546	tridecane	184
10	14	12.6	139609	tetradecane	198
11	15	13.1	141004	pentadecane	212
12	16	13.7	137205	hexadecane	226

Infrared Analysis Data

INFRARED ANALYSIS

Infrared spectra were run of heat samples between NaCl plates from 4000 to 600 cm^{-1}. Analysis was performed within 1-2 days of the sampling time.

IR ANALYSIS

SAMPLE: A-1

DATE: 1/18/78

RESULTS (PEAK RATIO = 0.61):

ABSORPTION WAVELENGTH (CM^{-1})	PEAK INTENSITY (CM)
3150	0.16
2950	18.18
2920	20.74
2870	15.72
2850	18.53
2715	0.88
2660	0.84
2610	0.68
1650	0.23
1579	0.86
1458	10.05
1437	4.59
1376	6.12
1365	3.83
1350	1.11
1338	0.62
1295	1.00
1140	0.50
756	0.08
715	1.05

SAMPLE: B-1

DATE: 1/18/78

RESULTS (PEAK RATIO = 0.59):

ABSORPTION WAVELENGTH (CM^{-1})	PEAK INTENSITY (CM)
3170	0.15
2950	20.26
2920	21.72
2870	18.06
2850	20.53
2720	1.03
2660	0.94
1646	0.33
1584	1.13
1462	11.50
1441	4.34
1378	6.77
1368	4.08
1340	2.03
1290	0.98
1145	0.42
760	0.05
716	0.98

IR ANALYSIS

SAMPLE: C-1

DATE:

RESULTS (PEAK RATIO = 0.60):

ABSOPRTION WAVELENGTH (CM^{-1})	PEAK INTENSITY (CM)
3150	0.10
2950	19.69
2915	21.10
2870	17.29
2850	20.08
2710	0.98
2660	0.88
1643	0.26
1586	0.80
1457	11.72
1436	4.21
1375	6.98
1365	3.98
1350	2.12
1336	0.60
1289	1.02
1136	0.39
756	0.08
713	1.12

SAMPLE: OIl Sample No. 2

DATE:

RESULTS (PEAK RATIO = 0.63):

WAVELENGTH (CM^{-1})	PEAK INTENSITY (CM)
3140	0.36
2940	20.82
2910	21.58
2860	19.79
2850	21.02
2710	1.50
2660	1.96
2580	0.98
1636	0.88
1588	0.74
1457	13.98
1436	5.30
1375	8.80
1366	4.70
1350	2.74
1338	0.88
1288	1.22
1138	0.55
756	0.15
715	1.74

IR ANALYSIS

SAMPLE: 3
DATE:
RESULTS (PEAK RATIO = 0.63):

ABSORPTION WAVELENGTH (CM^{-1})	PEAK INTENSITY (CM)
3150	0.06
2935	14.18
2900	17.90
2850	11.78
2835	14.74
2685	0.51
2580	0.33
1645	0.12
1590	0.18
1455	6.23
1433	2.56
1374	3.92
1364	2.08
1347	1.11
1335	0.88
1283	0.56
710	0.62

SAMPLE: 4
DATE: 2/8/78
RESULTS (PEAK RATIO = 0.55):

ABSORPTION WAVELENGTH (CM^{-1})	PEAK INTENSITY (CM)
3140	0.18
2945	19.58
2915	22.10
2865	17.18
2850	19.98
2700	0.97
2650	0.89
2595	0.58
1572	0.54
1455	10.15
1436	4.67
1374	5.60
1364	3.02
1337	1.58
1284	1.03
709	0.84

IR ANALYSIS

SAMPLE: 4 (Designated 4A on IR, actual duplicate of 4)

DATE: 2/8/78

RESULTS (PEAK RATIO = 0.57):

ABSORPTION WAVELENGTH (CM^{-1})	PEAK INTENSITY (CM)
2945	17.25
2915	19.32
2860	15.22
2850	17.51
2700	0.68
2650	0.60
2590	0.49
1582	0.57
1454	9.36
1434	4.28
1373	5.33
1363	3.22
1346	1.79
1334	1.40
1285	0.80
707	0.89

SAMPLE: Oil Sample 4 (Numbered 4B, run on independent day)

DATE: 3/2/78

RESULTS (PEAK RATIO = 0.67):

ABSORPTION WAVELENGTH (CM^{-1})	PEAK INTENSITY (CM)
3160	0.19
2940	20.18
2910	20.35
2845	20.11
2710	1.70
2655	1.52
2595	1.03
2355	0.12
1586	0.10
1459	14.55
1374	9.80
1339	2.88
1291	1.63
1255	1.05
11.96	0.48
1139	0.50
712	1.71

IR ANALYSIS

SAMPLE: 5

DATE: 2/15/77

RESULTS (PEAK RATIO = 0.59):

ABSORPTION WAVELENGTH (CM^{-1})	PEAK INTENSITY (CM)
3150	0.18
2925	19.58
2915	21.37
2860	17.92
2850	19.95
2710	1.23
2650	1.12
1576	0.59
1457	11.83
1446	7.84
1435	5.08
1375	7.02
1364	3.72
1337	1.74
1286	1.14
1134	0.46
752	0.12
714	1.27

SAMPLE: Oil Sample No. 7

DATE: 2/24/78

RESULTS (PEAK RATIO = 0.54):

ABSORPTION WAVELENGTH (CM^{-1})	PEAK INTENSITY (CM)
3160	0.15
2950	19.94
2915	20.33
2850	19.67
2715	1.40
2660	1.26
2625	1.01
2530	0.32
1590	0.05
1454	11.63
1370	6.30
1354	3.96
1344	2.79
1290	1.34
1140	0.34
713	1.19

IR ANALYSIS

SAMPLE: Oil Sample No. 8

DATE: 3/2/78

RESULTS (PEAK RATIO = 0.63):

ABSORPTION WAVELENGTH (CM^{-1})	PEAK INTENSITY (CM)
3150	0.15
2945	20.12
2910	20.42
2845	20.00
2710	1.52
2650	1.46
1590	0.06
1457	13.13
1373	8.28
1286	1.33
1261	0.85
1195	0.30
1148	0.23
710	1.54

SAMPLE: Oil Sample No. 11

DATE: 3/2/78

RESULTS (PEAK RATIO = 0.59):

ABSORPTION WAVELENGTH (CM^{-1})	PEAK INTENSITY (CM)
3145	0.07
2945	19.60
2910	20.44
2845	19.52
2705	1.03
2650	0.93
2590	0.65
1457	11.86
1373	6.97
1336	1.98
1285	1.12
1186	0.38
1135	0.31
758	0.03
710	1.12

IR ANALYSIS

SAMPLE: Oil Sample No. 12

DATE: 3/2/78

RESULTS (PEAK RATIO = 0.55):

ABSORPTION WAVELENGTH (CM^{-1})	PEAK INTENSITY (CM)
2950	18.61
2915	19.97
2865	14.98
2850	17.02
2710	0.60
2650	0.50
1455	6.21
1370	3.42
1294	0.43
1253	0.33
1221	0.24
709	0.32

SAMPLE: Oil Sample No. 13

DATE: 3/10/78

RESULTS (PEAK RATIO = 0.63):

ABSORPTION WAVELENGTH (CM^{-1})	PEAK INTENSITY (CM)
3155	0.45
2940	19.84
2900	19.94
2860	19.18
2840	19.72
2710	2.16
2650	1.98
2600	1.50
2360	0.23
1585	0.20
1457	13.52
1372	8.49
1361	4.87
1347	2.88
1287	1.59
1136	0.59
753	0.08
710	1.61

IR ANALYSIS

SAMPLE: Oil Sample No. 15

DATE: 03/10/78

RESULTS (PEAK RATIO = 0.78):

ABSORPTION WAVELENGTH (CM^{-1})	PEAK INTENSITY
3600	0.11
3340	0.30
3150	0.52
2950	18.52
2910	18.87
2860	16.46
2850	17.12
2710	1.46
2650	1.10
2590	0.81
1653	0.15
1626	0.54
1583	1.13
1456	12.31
1372	9.63
1359	6.92
1338	2.97
1293	1.60
1245	1.02
1146	0.69
953	0.43
867	0.22
775	1.82
758	1.62
711	1.10

SAMPLE: Oil Sample No. 17

DATE: 03/10/78

RESULTS (PEAK RATIO = 0.78):

ABSORPTION WAVE LENGTH (CM^{-1})	PEAK INTENSITY (CM)
3605	0.15
3360	0.28
3160	0.69
2950	17.61
2915	18.00
2870	16.15
2850	16.79
2715	1.93
2660	1.43
2600	1.12
2315	0.20
1704	0.15
1634	0.68
1586	1.15
1491	1.94
1462	13.40
1376	10.50
1337	3.09
1296	1.91
1250	1.31
1152	0.97
956	0.71
900	0.22
875	0.32
798	0.30
760	0.23
735	0.92
715	1.51

IR ANALYSIS

SAMPLE: Oil Sample No. 18
DATE: 03/10/78
RESULTS (PEAK RATIO = 0.70):

ABSORPTION WAVELENGTH (CM^{-1})	PEAK INTENSITY (CM)
3390	0.30
2945	17.05
2910	19.11
2860	14.63
2845	17.08
2700	0.96
2650	0.92
2595	0.70
2350	0.15
1574	0.77
1456	10.10
1370	7.11
1350	4.81
1288	1.35
1134	0.49
1000	0.40
949	0.19
709	0.83

SAMPLE: Oil Sample No. 19
DATE: 03/16/78
RESULTS (PEAK RATIO = 0.79):

ABSORPTION WAVELENGTH (CM^{-1})	PEAK INTENSITY (CM)
3345	0.24
3145	0.68
2940	19.32
2900	19.42
2840	19.22
2710	2.62
2655	2.30
2610	1.92
2360	0.31
1630	0.42
1580	0.94
1455	16.69
1370	13.20
1349	7.03
1339	4.32
1290	2.83
1138	1.13
1000	0.53
945	0.48
864	0.22
755	0.30
712	3.22

IR ANALYSIS

SAMPLE: Oil Sample No. 20
DATE: 03/16/78
RESULTS (PEAK RATIO = 0.75):

ABSORPTION WAVELENGTH (CM^{-1})	PEAK INTENSITY (CM)
3355	0.25
2950	16.04
2910	16.88
2860	13.26
2850	14.00
2710	0.93
2645	0.71
2590	0.51
2350	0.09
1630	0.43
1582	0.73
1455	9.29
1370	6.98
1352	4.50
1337	2.29
1290	1.10
1244	0.67
1150	0.55
950	0.36
867	0.11
775	1.73
760	1.50
708	0.53

SAMPLE: Oil Sample No. 21
DATE: 05/17/78
RESULTS (PEAK RATIO = 0.69):

ABSORPTION WAVELENGTH (CM^{-1})	PEAK INTENSITY
2940	17.18
2910	19.20
2840	17.00
2695	0.81
2600	0.60
1670	0.10
1642	0.20
1575	0.61
1450	9.51
1365	6.59
1290	1.21
1135	0.60
995	0.50
710	0.71

IR ANALYSIS

SAMPLE: Oil #22

DATE: 5/17/78

RESULTS: (Peak Ratio = 0.83):

ABSORPTION WAVELENGTH (CM^{-1})	PEAK INTENSITY
3155	0.6
2935	20.2
2890	20.3
2845	30.2
2710	3.6
2660	3.2
2590	2.1
1595	0.4
1455	18.7
1371	15.6
1290	3.0
1266	2.4
1200	0.8
1145	1.1
752	1.1
713	5.2

SAMPLE: Oil #23

DATE: 5/17/78

RESULTS: (Peak Ratio = 0.89):

ABSORPTION WAVELENGTH (CM^{-1})	PEAK INTENSITY
2940	9.3
2910	13.9
2840	9.3
1446	3.5
1361	3.1
710	0.2
1332	1.2

IR ANALYSIS

SAMPLE: Oil #24
DATE: 5/17/78
RESULTS: (PEAK RATIO = 0.71):

ABSORPTION WAVELENGTH (CM^{-1})	PEAK INTENSITY
3130	0.3
2940	19.6
2910	20.1
2840	19.5
2710	2.6
2450	2.2
1455	23.1
1370	16.3
1290	2.7
1139	1.1
1000	0.4
750	0.9
711	3.2

SAMPLE: Oil #25
DATE: 5/26/78
RESULTS: (PEAK RATIO = 0.74):

ABSORPTION WAVELENGTH (CM^{-1})	PEAK INTENSITY
3150	0.39
2940	16.91
2910	17.81
2840	16.91
2710	1.89
2660	1.71
2600	1.41
1620	0.29
1572	0.41
1456	13.78
1370	10.20
1340	3.30
1290	2.17
1140	0.99
759	0.21
712	2.39

IR ANALYSIS

SAMPLE: Oil #26
DATE: 6/14/78
RESULTS: (PEAK RATIO = 0.75):

ABSORPTION WAVELENGTH (CM^{-1})	PEAK INTENSITY
3140	0.4
2935	17.7
2900	18.4
2840	17.7
2705	2.3
2650	2.1
1515	0.4
1455	15.2
1370	11.4
1290	2.6
1138	1.0
947	0.4
752	0.5
710	2.9

SAMPLE OIL #27
DATE: 6/14/78
RESULTS: (PEAK RATIO = 0.41):

ABSORPTION WAVELENGTH (CM^{-1})	PEAK INTENSITY
2940	12.8
2910	16.8
2840	12.7
1450	5.5
1365	2.3
762	0.1
705	0.4

IR ANALYSIS

SAMPLE: Oil #28
DATE: 6/14/78
RESULTS: (PEAK RATIO = 0.47):

ABSORPTION WAVELENGTH (CM^{-1})	PEAK INTENSITY
2945	17.1
2910	19.6
2845	17.6
2700	0.7
2650	0.6
1455	9.6
1370	4.5
1332	1.4
1285	0.9
710	0.8

SAMPLE: Oil #29
DATE: 6/14/78
RESULTS: (PEAK RATIO = 0.43):

ABSORPTION WAVELENGTH (CM^{-1})	PEAK INTENSITY
2940	10.6
2910	14.7
2840	10.6
2450	0.2
1447	4.2
1435	2.6
1415	0.8
1365	1.8
1325	0.5
705	0.3

IR ANALYSIS

SAMPLE: Oil #30

DATE: 6/14/78

RESULTS: (PEAK RATIO = 0.51):

ABSORPTION WAVELENGTH (CM^{-1})	PEAK INTENSITY
2940	17.2
2910	19.3
2840	17.6
2700	1.3
2650	1.0
1455	11.4
1370	5.8
1335	1.9
1285	1.3
1135	0.5
710	1.2

SAMPLE: Oil #31

DATE: 6/14/78

RESULTS: (PEAK RATIO = 0.46):

ABSORPTION WAVELENGTH (CM^{-1})	PEAK INTENSITY
2945	11.1
2910	13.7
2845	9.5
1449	4.1
1365	1.9
702	0.3

IR ANALYSIS

SAMPLE: Oil #32
DATE: 6/14/78
RESULTS: (PEAK RATIO = **0.63**):

ABSORPTION WAVELENGTH (CM^{-1})	PEAK INTENSITY
2940	19.0
2905	19.7
2840	19.2
2700	1.5
2650	1.4
2590	1.0
1457	14.0
1372	8.8
1340	2.8
1290	1.8
1135	0.7
1000	0.4
710	1.8

SAMPLE: Oil #33
DATE: 6/21/78
RESULTS: (PEAK RATIO = **0.45**):

ABSORPTION WAVELENGTH (CM^{-1})	PEAK INTENSITY
2940	15.7
2910	17.4
2840	14.2
2690	0.5
2640	0.4
1450	7.6
1366	3.4
1330	1.0
1281	0.6
710	0.5

IR ANALYSIS

SAMPLE: OIL#34 HIGH BOILERS
DATE: 6/21/78
RESULTS (PEAK RATIO = 0.46):

ABSORPTION WAVELENGTH (CM^{-1})	PEAK INTENSITY (CM)
2940	14.7
2910	17.8
2845	15.2
2630 (PEAK PROBABLY FROM "SALT" BLANK)	0.5
1452	7.8
1368	3.6
1281	0.7
711	0.7

SAMPLE: OIL#35 SIDEDRAW
DATE: 6/21/78
RESULTS (PEAK RATIO = 0.44):

ABSORPTION WAVELENGTH (CM^{-1})	PEAK INTENSITY (CM)
2945	13.1
2915	16.7
2850	13.2
1451	5.7
1415	1.0
1368	2.5
1335	0.8
770	0.6
710	0.4

IR ANALYSIS

SAMPLE: OIL#36 HOT OIL FEED

DATE: 6/21/78

RESULTS (PEAK RATIO = 0.54):

ABSORPTION WAVELENGTH (CM^{-1})	PEAK INTENSITY (CM)
2945	16.6
2910	18.5
2845	17.0
2705	1.0
2650	0.9
1457	10.9
1370	5.9
1335	1.2
1284	1.0
771	0.3
710	1.0

SAMPLE: OIL#37 HOT OIL FEED

DATE: 6/29/78

RESULTS (PEAK RATIO = 0.63):

ABSORPTION WAVELENGTH (CM^{-1})	PEAK INTENSITY (CM)
3165	0.5
2945	18.8
2910	19.3
2850	18.7
2710	1.8
2650	1.6
2590	1.2
1581	0.4
1458	14.3
1376	9.0
1338	2.9
1289	2.0
1140 (PEAK PROBABLY	1.0
1002 FROM "SALT" BLANK)	0.5
867	0.2
7.2	2.0

IR ANALYSIS

SAMPLE: OIL#38 SIDEDRAW

DATE: 6/29/78

RESULTS (PEAK RATIO = 0.42):

ABSORPTION WAVELENGTH (CM^{-1})	PEAK INTENSITY (CM)
2940	13.2
2910	17.5
2845	13.3
1451	5.2
1363	2.2
1334	0.8
705	0.4

SAMPLE: OIL#39 HIGH BOILERS

DATE: 6/29/78

RESULTS (PEAK RATIO = 0.48):

ABSORPTION WAVELENGTH (CM^{-1})	PEAK INTENSITY (CM)
2945	16.5
2920	19.1
2850	16.8
2640	0.7
1455	9.4
1371	4.5
1339	1.6
1285	1.1
1000	0.4
770	0.4
710	0.7

IR ANALYSIS

SAMPLE: OIL#40 LOW BOILERS

DATE: 6/29/78

RESULTS (PEAK RATIO = 0.48):

ABSORPTION WAVELENGTH (CM^{-1})	PEAK INTENSITY (CM)
2945	13.1
2915	16.1
2845	11.1
1450	4.6
1400	0.7
1367	2.2
1281 (PEAK IS PROBABLY CAUSED BY "SALT" BLANK)	0.5
999	0.4
770	0.2
710	0.2

SAMPLE: OIL#41 HOT OIL FEED

7/7/78

RESULTS (PEAK RATIO = 0.61):

ABSORPTION WAVELENGTH (CM^{-1})	PEAK INTENSITY (CM)
2945	18.1
2910	18.7
2845	18.0
2710	1.3
2650	1.2
2580	0.7
1455	12.9
1373	7.9
1335	2.2
1286	1.3
1122	0.4
945	0.4
755	0.3
710	1.9

IR ANALYSIS

SAMPLE: OIL#43 HIGH BOILERS

DATE: 7/7/78

RESULTS (PEAK RATIO = 0.44):

ABSORPTION WAVELENGTH (CM^{-1})	PEAK INTENSITY (CM)
2940	14.9
2910	18.6
2840	15.5
1453	7.5
1368	3.3
1286	0.7
755	0.2
710	1.0

SAMPLE: OIL#42 SIDEDRAW

DATE: 7/7/78

RESULTS (PEAK RATIO = 0.43):

ABSORPTION WAVELENGTH (CM^{-1})	PEAK INTENSITY (CM)
2945	14.6
2915	18.1
2845	14.6
2700	0.5
2645	0.4
1453	6.9
1415	1.2
1367	3.0
1326	0.8
1278	0.6
710	0.9

IR ANALYSIS

SAMPLE: OIL#45 HOT OIL FEED

DATE: 7/7/78

RESULTS (PEAK RATIO = 0.58):

ABSORPTION WAVELENGTH (CM^{-1})	PEAK INTENSITY (CM)
2940	17.8
2910	18.8
2840	17.6
2700	1.2
2640	1.1
2580	0.8
1455	12.2
1416	1.9
1371	7.1
1335	1.9
1281	1.1
1140	0.2
940	0.2
754	0.3
710	1.2

SAMPLE OIL#44 LOW BOILERS

DATE: 7/7/78

RESULTS (PEAK RATIO = 0.48):

ABSORPTION WAVELENGTH (CM^{-1})	PEAK INTENSITY (CM)
2945	11.9
2915	14.4
2845	10.3
2700	0.5
1450	4.8
1366	2.3
1279	0.4
752	0.4
710	0.6

IR ANALYSIS

SAMPLE: OIL#47 HIGH BOILERS

DATE: 7/7/78

RESULTS (PEAK RATIO = 0.46):

ABSORPTION WAVELENGTH (CM^{-1})	PEAK INTENSITY (CM)
2950	14.6
2920	17.6
2850	14.7
2450	0.7
1457	8.0
1419	1.3
1371	3.7
1339	1.2
1285	0.8
1010	NOT A PEAK
710	1.1

SAMPLE: OIL#46 SIDE DRAW

DATE: 7/7/78

RESULTS (PEAK RATIO = 0.60):

ABSORPTION WAVELENGTH (CM^{-1})	PEAK INTENSITY (CM)
2940	17.7
2905	18.5
2840	17.5
2700	1.3
2640	1.2
1455	12.7
1370	7.6
1332	2.1
1280	1.2
1141	0.4
938	0.2
765	0.6
705	1.9

IR ANALYSIS

SAMPLE: OIL#48 LOW BOILERS
DATE: 7/7/78
RESULTS (PEAK RATIO = 0.58):

ABSORPTION WAVELENGTH (CM^{-1})	PEAK INTENSITY (CM)
3055	1.2
2950	18.8
2910	19.3
2845	17.8
2710	1.1
2645	0.9
1634*	0.6
1584	0.5
1453	11.6
1372	6.7
1335	1.6
1285	1.0
1145	0.5
950	0.8
899	0.5
871	0.5
800	0.1
766	0.6
710	0.9

SAMPLE: OIL#49 HOT OIL FIELD
DATE: 7/19/78
RESULTS (PEAK RATIO = 0.47):

ABSORPTION WAVELENGTH (CM^{-1})	PEAK INTENSITY (CM)
2950	14.2
2920	17.8
2850	14.3
2705	0.5
2650	0.5
2585	0.3
2550	0.3
2520	0.2
1690	0.1
1455	7.0
1417	1.3
1370	3.3
1333	1.1
1285	0.7
774	0.8
712	0.8

IR ANALYSIS

SAMPLE: OIL#50 HIGH BOILERS

DATE: 7/19/78

RESULTS (PEAK RATIO = 0.45):

ABSORPTION WAVELENGTH (CM^{-1})	PEAK INTENSITY (CM)
2945	15.1
2910	18.2
2845	15.1
2705	0.6
2640	0.6
2570	0.4
2550	0.4
2510	0.3
2365	0.2
1455	7.8
1370	3.5
1335	1.2
1283	0.8
708	0.9

SAMPLE: OIL#51 HIGH BOILERS

DATE: 7/19/78

RESULTS (PEAK RATIO = 0.66):

ABSORPTION WAVELENGTH (CM^{-1})	PEAK INTENSITY (CM)
3180	0.2
2945	19.6
2915	19.7
2895	19.8
2840	19.6
2710	1.8
2660	1.6
2490	1.7
1647	0.1
1458	15.4
1372	10.1
1336	3.0
1290	2.0
1137	0.8
1050	0.5
934	0.3
765	0.9
710	2.4

IR ANALYSIS

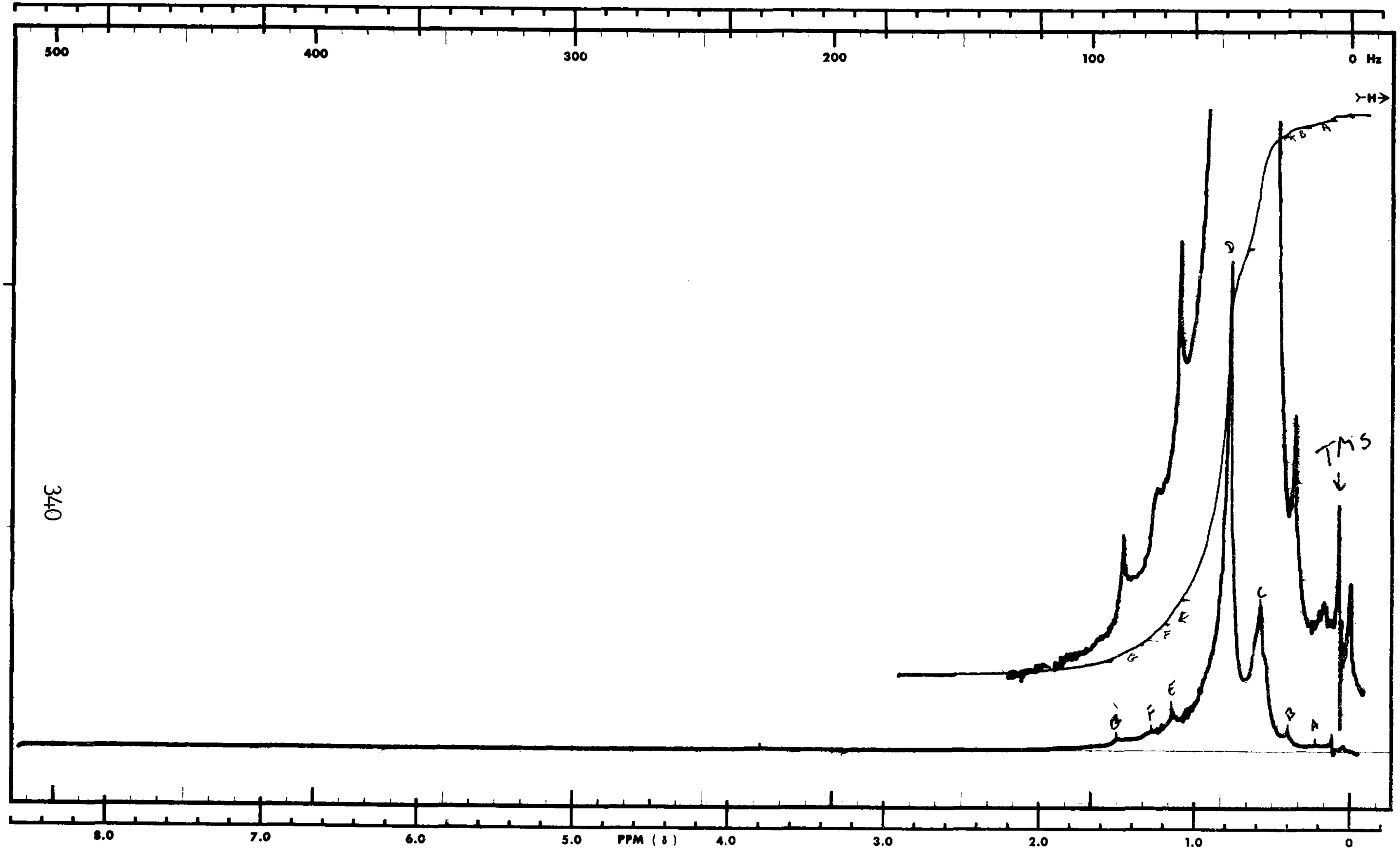
SAMPLE: OIL#52 LOW BOILERS

DATE: 7/19/78

RESULTS (PEAK RATIO = 0.55):

ABSORPTION WAVELENGTH (CM^{-1})	PEAK INTENSITY (CM)
3060	1.1
2950	18.8
2915	19.6
2845	17.2
2710	0.9
2650	0.7
2590	0.5
2510	0.2
1650	0.2
1630	0.6
1580	0.4
1455	10.4
1372	5.7
1327	1.4
1284	1.1
1135	0.4
982	0.4
950	0.4
900	0.8
873	0.8
795	0.1
757	0.1
710	0.9

NMR Analysis Data



MANUAL

SWEEP OFFSET (Hz): 0

SPECTRUM AMPLITUDE: 1.25

INTEGRAL AMPLITUDE: 7

SPINNING RATE (RPS): 40

SWEEP TIME (SEC): 50 [✓ 250]

SWEEP WIDTH (Hz): 25 50 100 250 500

FILTER: [✓ 1] 2 3 4 5 6 7 8

RF POWER LEVEL: .05

AUTO ☐

(250)

(500)

(2)

(.05)

SAMPLE: Sample A-1

0:1

50%V/V in CDCl3 + 2% TMS

SOLVENT: CDCl3 ≥ TMS

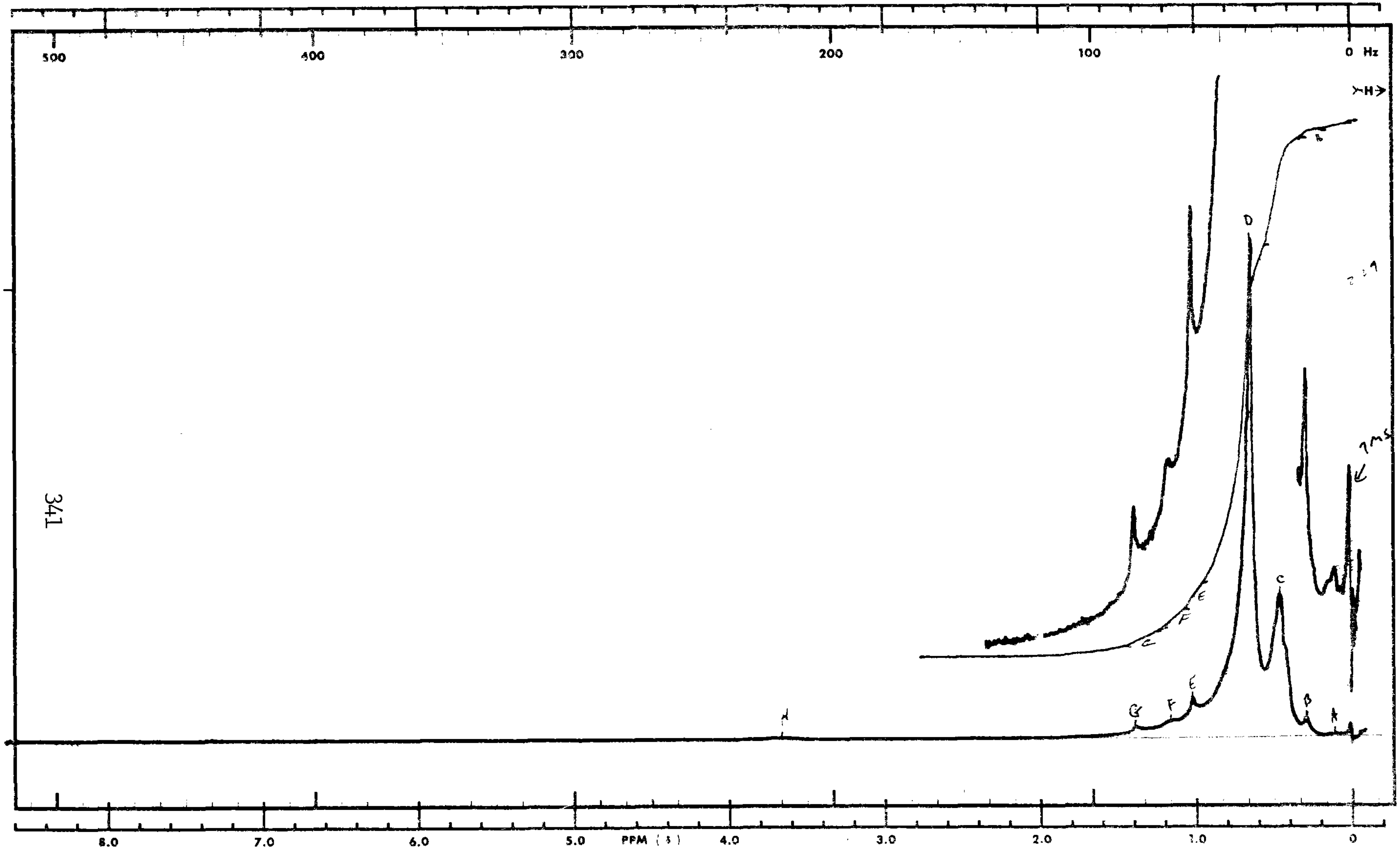
REMARKS:

varian
analytical instrument division

DATE: 1/26/78

OPERATOR: T. Giordano

60 MHz NMR
SPECTRUM NO.



MANUAL

AUTO ☐

SWEEP OFFSET (Hz): 0

SPECTRUM AMPLITUDE: 1.25

INTEGRAL AMPLITUDE: 7

SPINNING RATE (RPS): 40

SWEEP TIME (SEC): 50 [✓ 250] (250)

SWEEP WIDTH (Hz): 25 50 100 250 500 1000 (500)

FILTER: ☒ 2 3 4 5 6 7 8 (2)

RF POWER LEVEL: 0.05 (.05)

SAMPLE: Oil Sample B-1

50% 40

SOLVENT: CDCl3 + 2% TMS

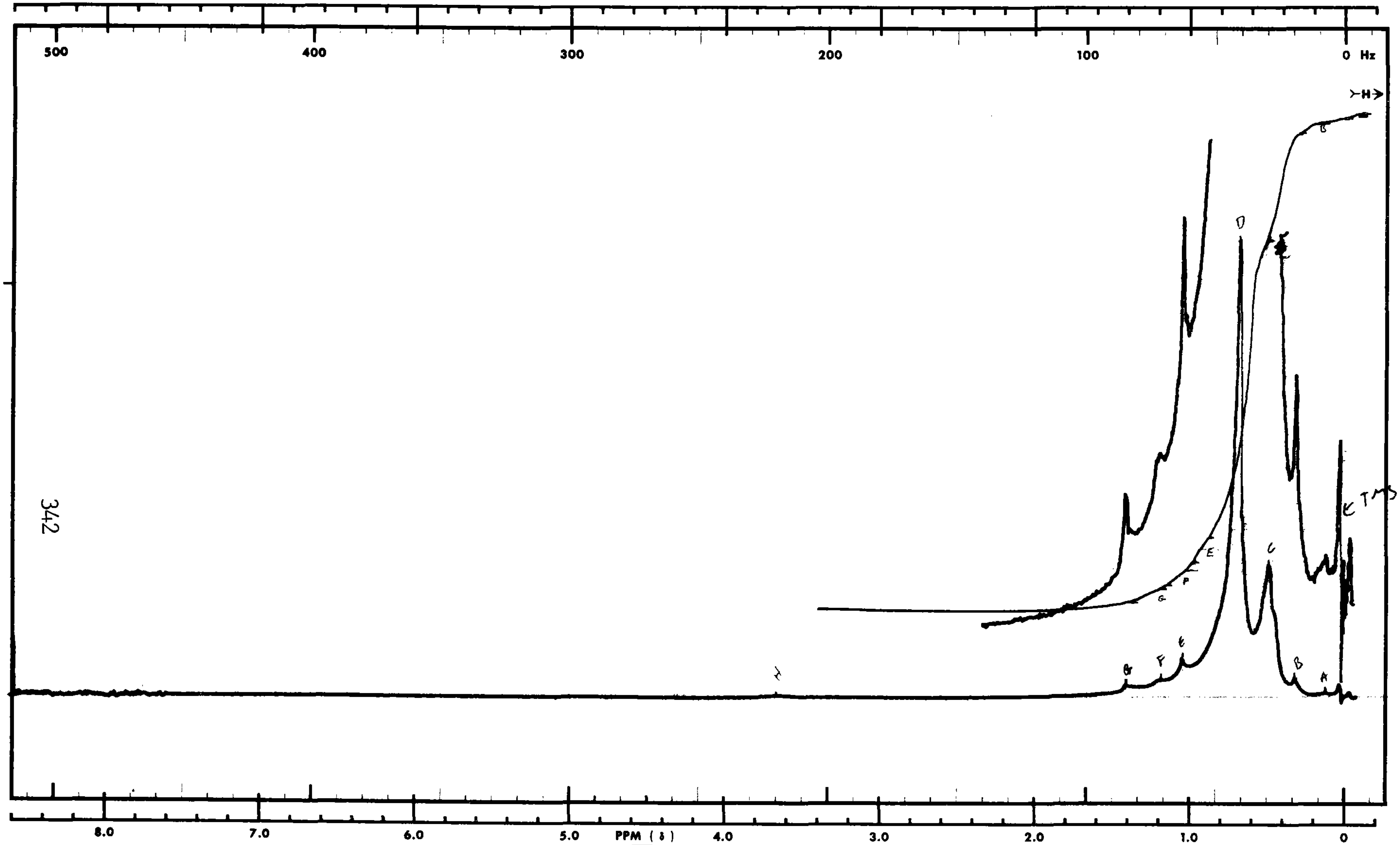
REMARKS:

varian
analytical instrument division

DATE: 1/21/78.

OPERATOR: T. Giordano

60 MHz NMR
SPECTRUM NO.



SWEEP OFFSET (Hz):	0
SPECTRUM AMPLITUDE:	1.25
INTEGRAL AMPLITUDE:	7
SPINNING RATE (RPS):	40

MANUAL

SWEEP TIME (SEC): 50 [✓ 250]

SWEEP WIDTH (Hz): 25 50 100 250 500 1000

FILTER: [✓ 1] 2 3 4 5 6 7 8

RF POWER LEVEL: 0.05

AUTO ☐

(250)

(500)

(2)

(.05)

SAMPLE: Oil Sample # C-1

REMARKS:

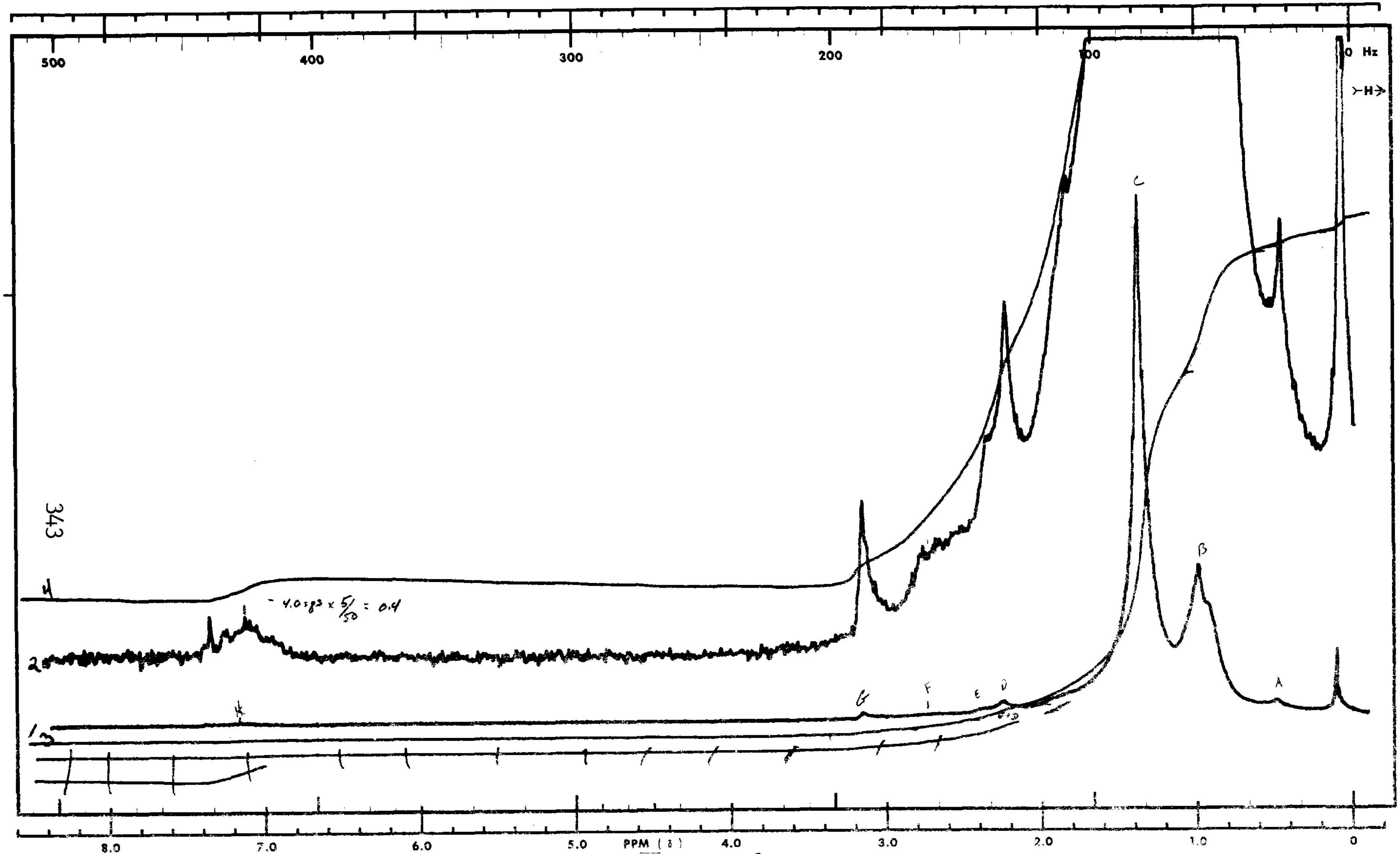
SOLVENT: CDCl$_3$ w/ 1% TMS

varian
analytical instrument division

DATE: 1/26/78

OPERATOR: T. J. Giordano

60 MHz NMR
SPECTRUM NO.



MANUAL | **AUTO** ☐

SWEEP OFFSET (Hz): 0

SPECTRUM AMPLITUDE: 1.25 + 25 (#2)

INTEGRAL AMPLITUDE: 1 × 5.0 (× 50 #2) 0.4

SPINNING RATE (RPS): 50

SWEEP TIME (SEC): 50 | 250 500 (250)

SWEEP WIDTH (Hz): #2 25 | 50 | 100 | 250 | ~~500~~ (500)

FILTER: ~~1~~ | 2 | ~~3~~ | 4 | 5 | 6 | 7 | 8 (2)

RF POWER LEVEL: 0.05 (.05)

SAMPLE: 0.1 Sample C-1

REMARKS:

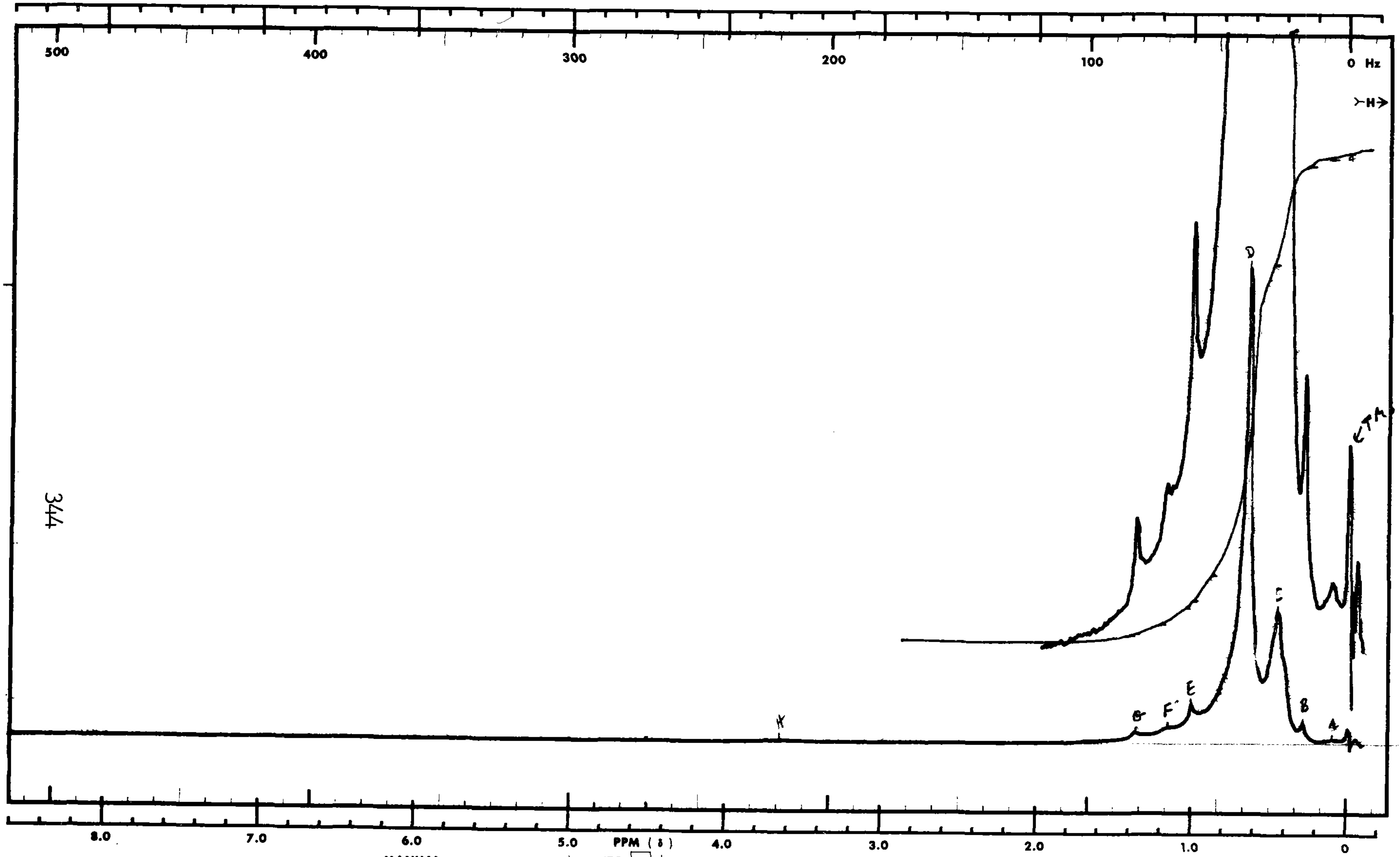
SOLVENT: 50% v/v in CDCl$_3$ + 2% TMS

varian
analytical instrument division

DATE: 3/2/78

OPERATOR: M. Barth

60 MHz NMR
SPECTRUM NO. A



MANUAL

SWEEP OFFSET (Hz): 0

SPECTRUM AMPLITUDE: 1.25 + 12.5

INTEGRAL AMPLITUDE: 7

SPINNING RATE (RPS): 40

SWEEP TIME (SEC): 50 | 25 | ☒

SWEEP WIDTH (Hz): 25 | 50 | 100 | 250 | 500 | 1000

FILTER: ☒ | 2 | 3 | 4 | 5 | 6 | 7 | 8

RF POWER LEVEL: 0.05

AUTO ☐

(250)

(500)

(2)

(.05)

SAMPLE: Oil Sample #2

50% v/v in

SOLVENT: $CDCl_3$ w/ 2% TMS

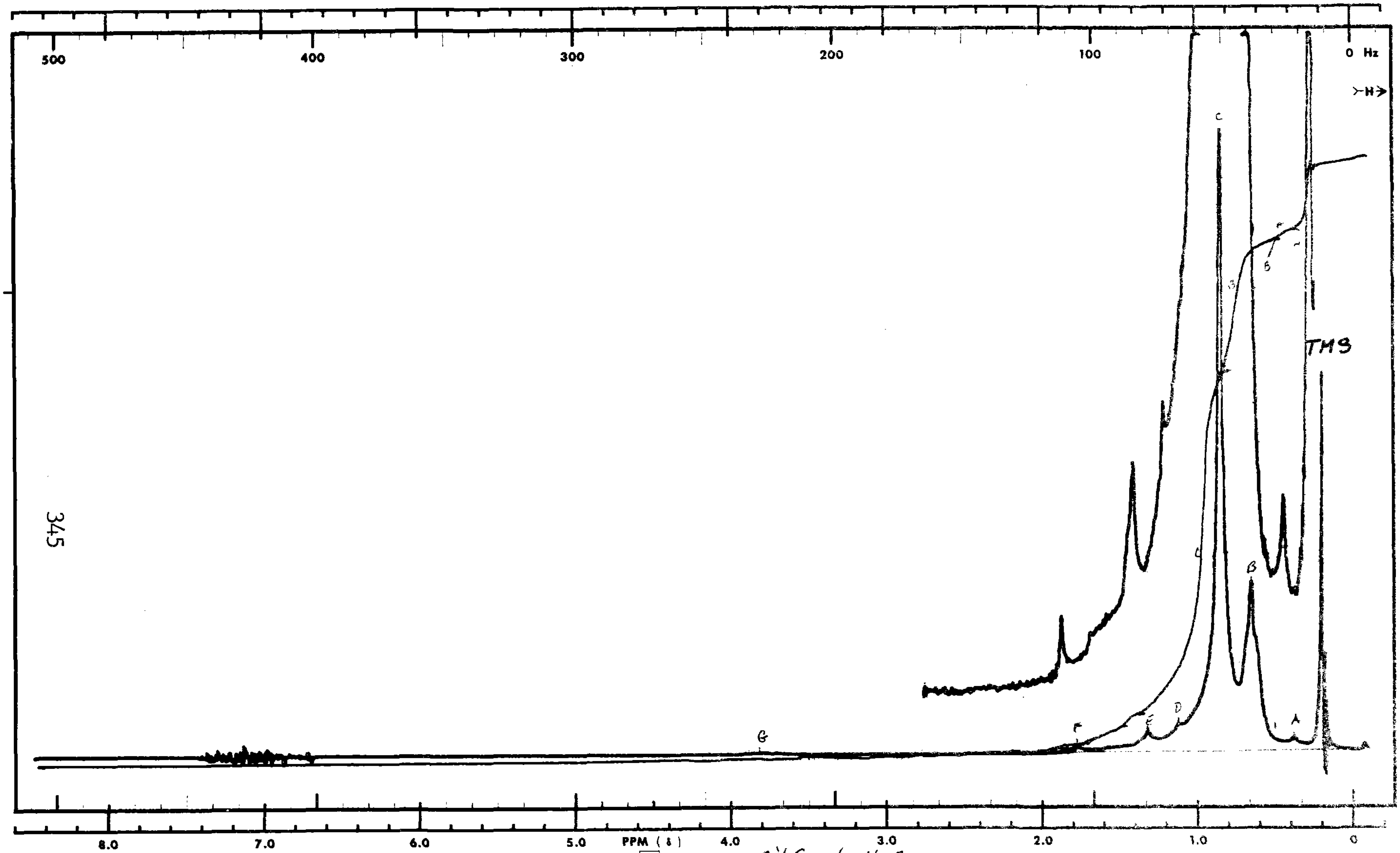
REMARKS:

varian
analytical instrument division

DATE: 1/26/78

OPERATOR: T. Giordano

60 MHz NMR
SPECTRUM NO. A



MANUAL

SWEEP OFFSET (Hz): 0

SPECTRUM AMPLITUDE: 1.25 + 12.5

INTEGRAL AMPLITUDE: 7

SPINNING RATE (RPS): 50

SWEEP TIME (SEC): 50 [✓ 250]

SWEEP WIDTH (Hz): 25 50 100 250 500 1000

FILTER: [✓ 1] 2 3 4 5 6 7 8

RF POWER LEVEL: 0.05

AUTO ☐

(250)

(500)

(2)

(.05)

SAMPLE: Oil Sample No. 3

REMARKS:

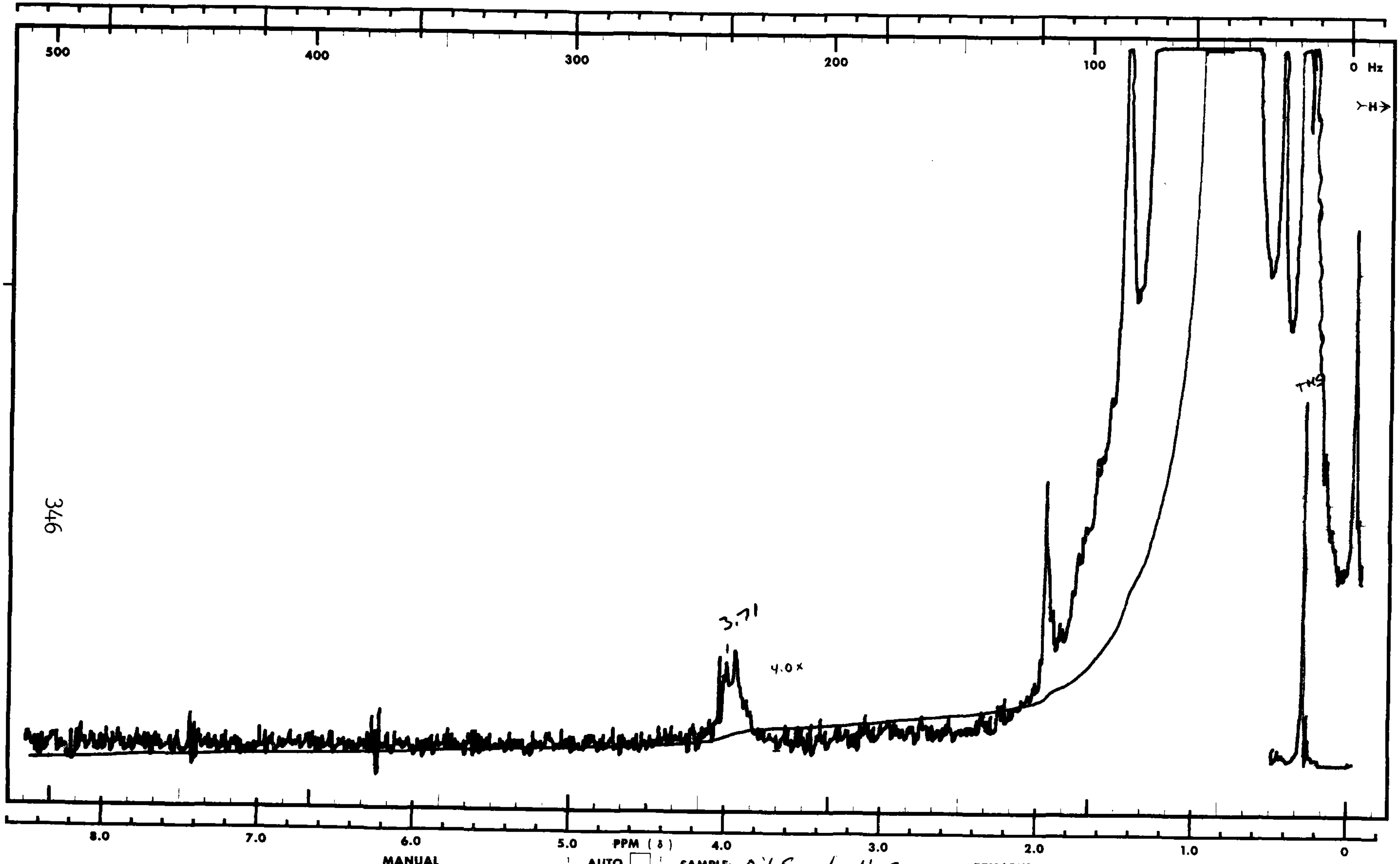
SOLVENT: 50% ¼ in D-Chloroform with 20% TMS

varian
analytical instrument division

DATE: 2-6-78

OPERATOR: C. H. Mosher

60 MHz NMR SPECTRUM NO A



MANUAL

SWEEP OFFSET (Hz): 0

SPECTRUM AMPLITUDE: [illegible]

INTEGRAL AMPLITUDE: LOAT 25.0

SPINNING RATE (RPS): 50

SWEEP TIME (SEC): ☐50 ☒250

SWEEP WIDTH (Hz): ☐25 ☐50 ☐100 ☐250 ☐500 1000

FILTER: ☒1 ☐2 ☐3 ☐4 ☐5 ☐6 ☐7 ☐8

RF POWER LEVEL: 0.05

AUTO ☐
(250)
(500)
(2)
(.05)

SAMPLE: Oil Sample No. 3

REMARKS:

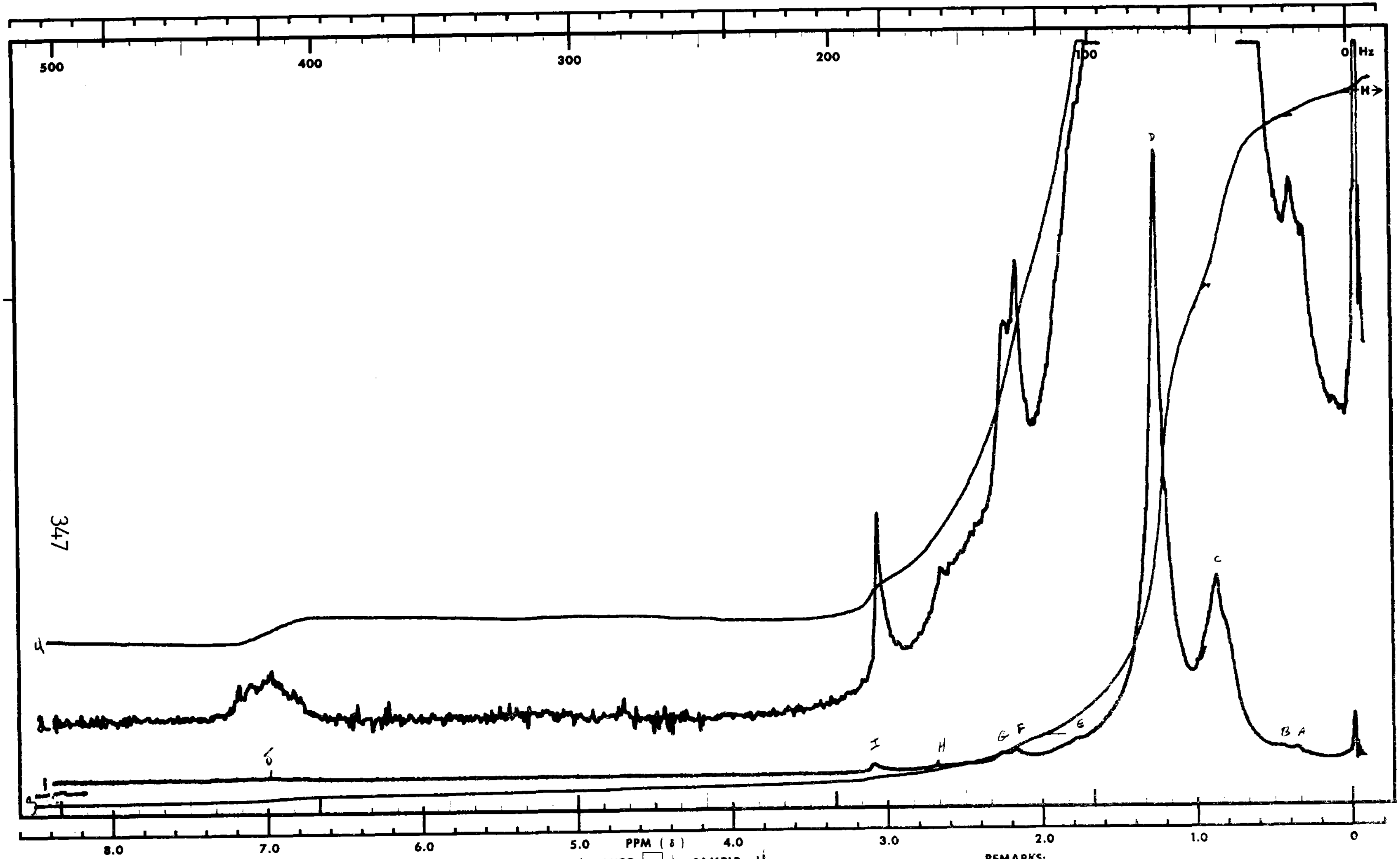
SOLVENT: 50% chloroform with 20%TMS

varian
analytical instrument division

DATE: 2-6-78

OPERATOR: C. H. Moshe

60 MHz NMR
SPECTRUM NO. B



MANUAL

SWEEP OFFSET (Hz): 0

SPECTRUM AMPLITUDE: 1.25 + 25

INTEGRAL AMPLITUDE: 5×1.0 #4-5×10.0

SPINNING RATE (RPS): 50

SWEEP TIME (SEC): 50 250 500

SWEEP WIDTH (Hz): 25 50 100 250 [✓ 500]

FILTER: [✓ 1] 2 [✓ 3] 4 5 6 7 8 #2

RF POWER LEVEL: 0.05

AUTO ☐

(250)

(500)

(2)

(.05)

SAMPLE: 4

REMARKS:

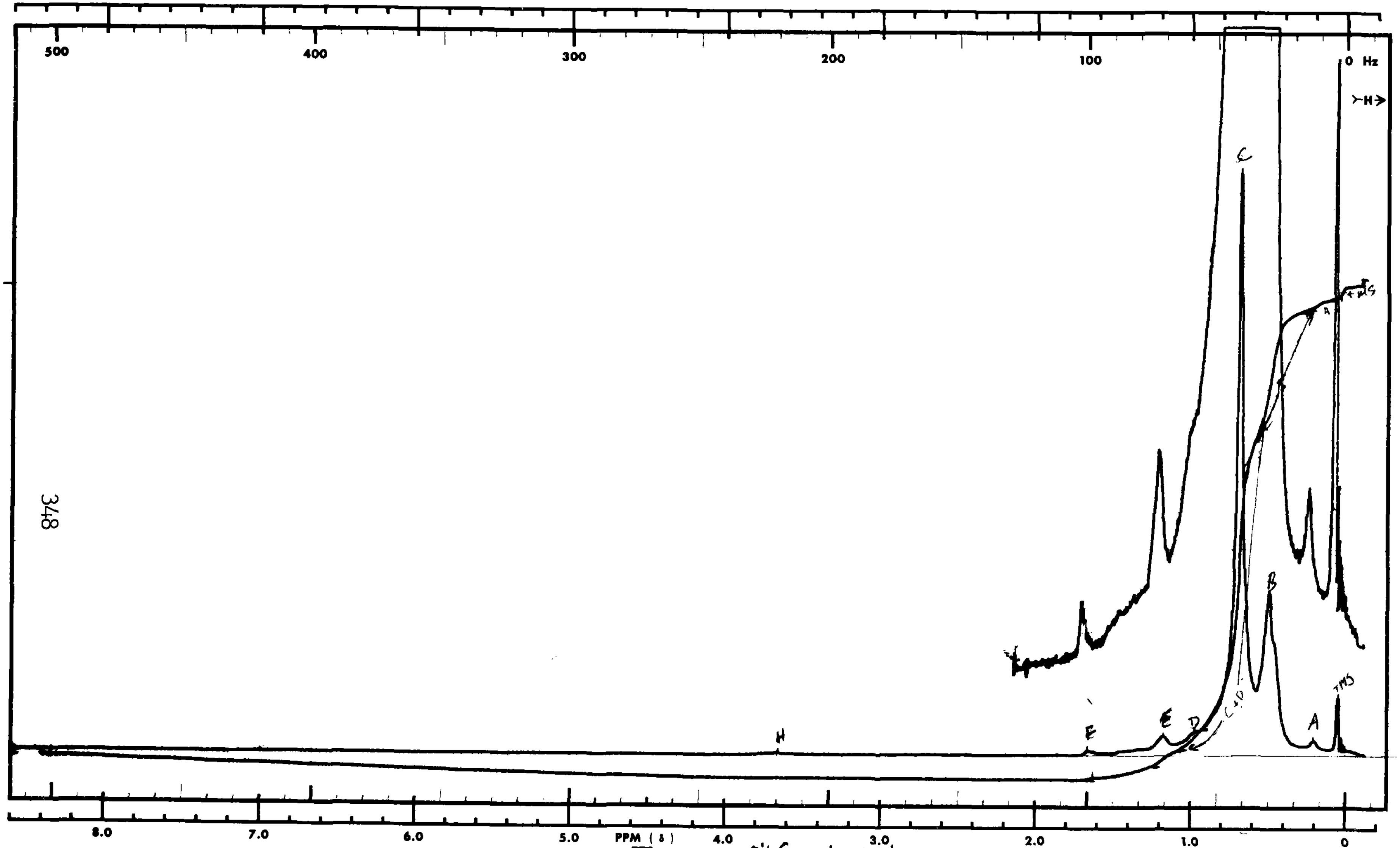
SOLVENT: 50% v/v in $CDCl_3$ + 2% TMS

varian
analytical instrument division

DATE: 3/2/78

OPERATOR: M. Barth

60 MHz NMR
SPECTRUM NO.



MANUAL

AUTO ☐

SWEEP OFFSET (Hz): 0

SPECTRUM AMPLITUDE: 125 + 12.5

INTEGRAL AMPLITUDE:

SPINNING RATE (RPS): 53

SWEEP TIME (SEC): 50 ~~250~~ (250)

SWEEP WIDTH (Hz): 25 | 50 | 100 | 250 | 500 /1000 (500)

FILTER: ☒ 2 3 4 5 6 7 8 (2)

RF POWER LEVEL: 0.05 (.05)

SAMPLE: Oil Sample No. 4

REMARKS:

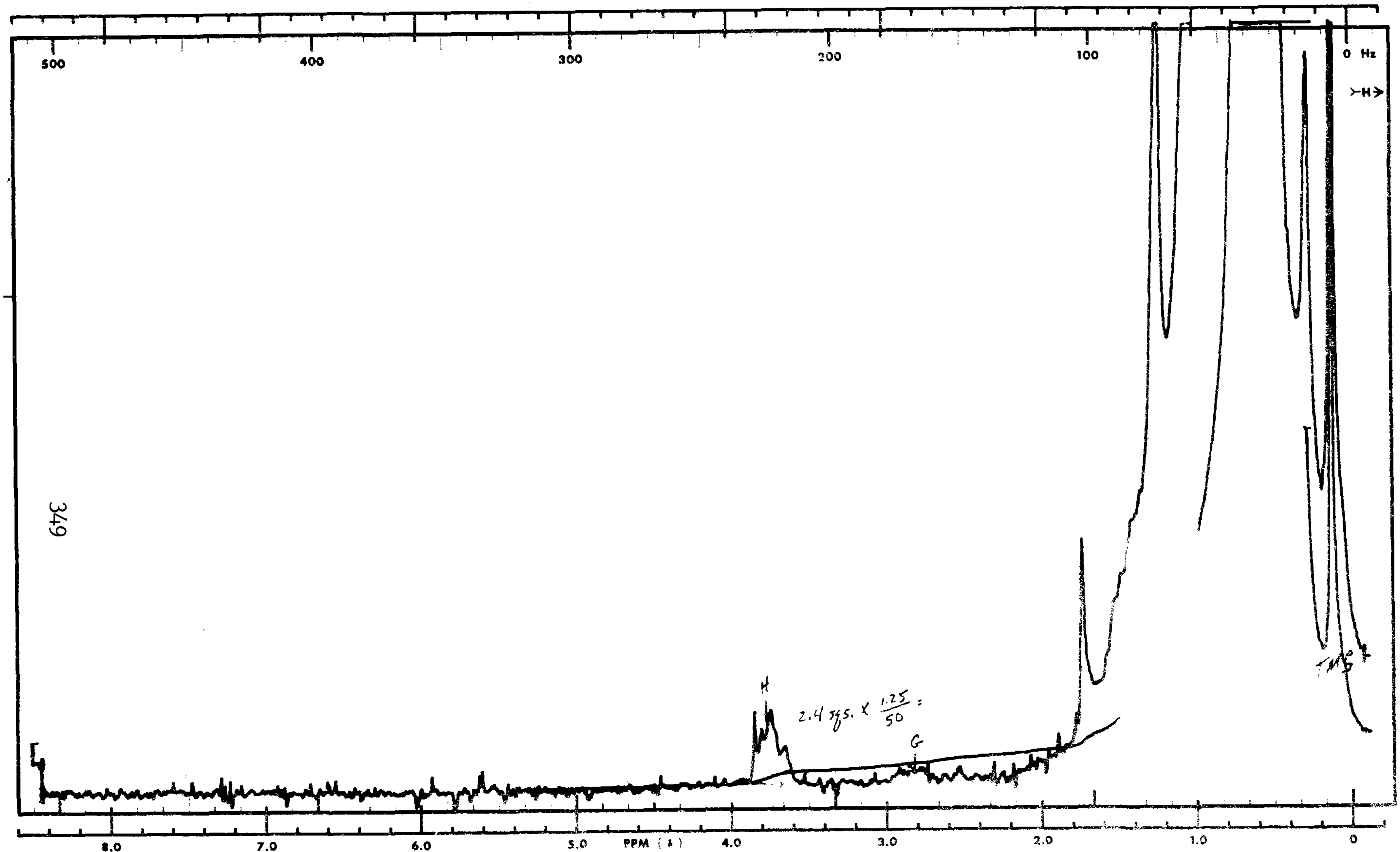
SOLVENT: 50% v/v ~~[illegible]~~ $CDCl_3$ w/ 2% TMS

varian
analytical instrument division

DATE: 2/10/78

OPERATOR: M. Barth

60 MHz NMR
SPECTRUM NO.



2.4 sqs. × $\frac{1.25}{50}$ =

349

MANUAL

SWEEP OFFSET (Hz): 0

SPECTRUM AMPLITUDE: 50

INTEGRAL AMPLITUDE: 1.0 at 25

SPINNING RATE (RPS): 53

SWEEP TIME (SEC): 50 250 1000 (250)

SWEEP WIDTH (Hz): 25 50 100 250 500 1000 (500)

FILTER: 1 2 3 ☒ 5 6 7 8 (2)

RF POWER LEVEL: 0.05 (.05)

AUTO ☐

SAMPLE: Oil Sample #4

REMARKS:

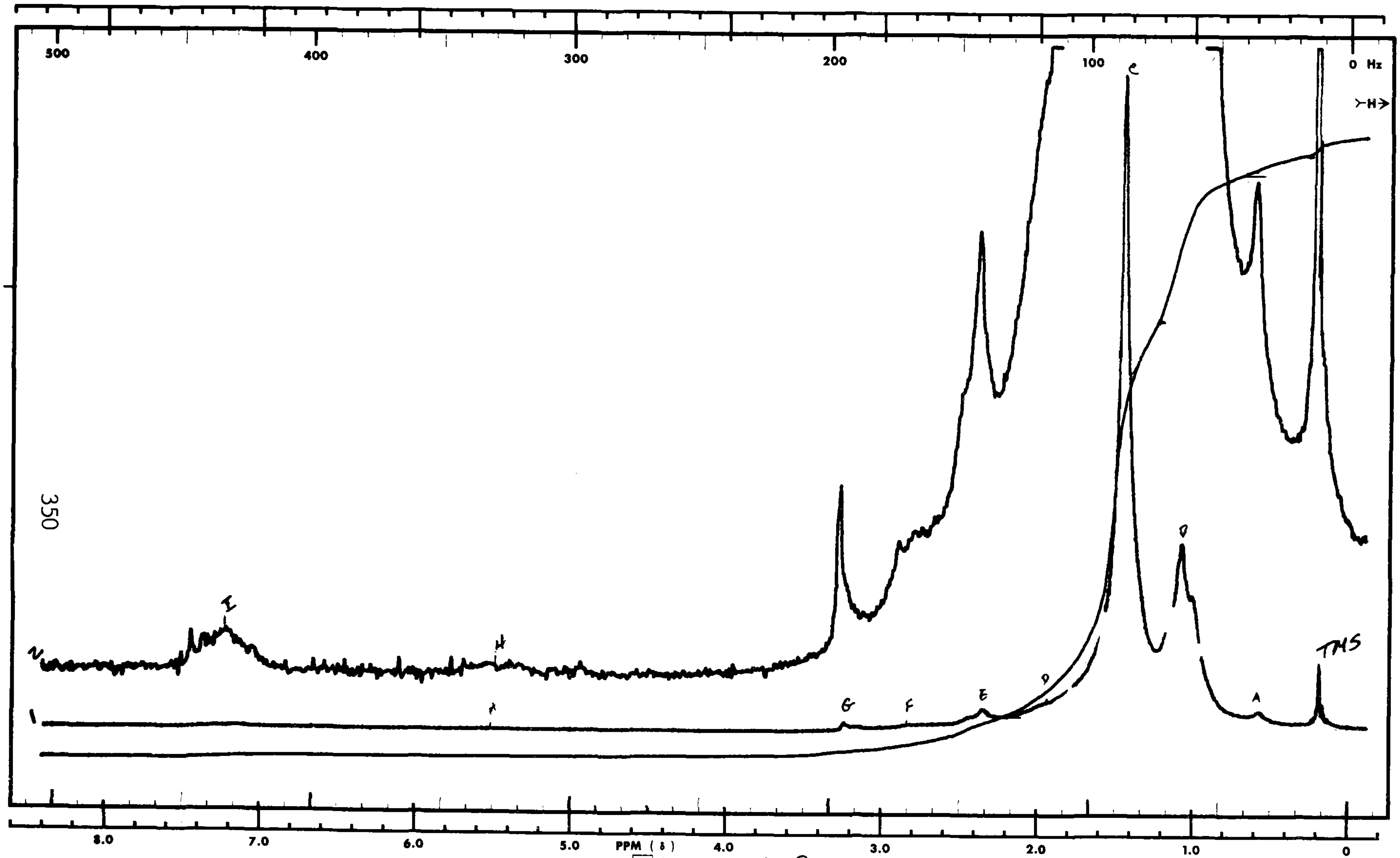
SOLVENT: 50% CDCl₃ + 2% TMS

varian
analytical instrument division

DATE: 2/10/78

OPERATOR: M. Barth

60 MHz NMR
SPECTRUM NO.



MANUAL

SWEEP OFFSET (Hz): 0

SPECTRUM AMPLITUDE: 1.25 + 2.5

INTEGRAL AMPLITUDE: 5×1.0

SPINNING RATE (RPS): 50

SWEEP TIME (SEC): 50 250 500

SWEEP WIDTH (Hz): #1 25 50 100 250 ☒5

FILTER: ☒ 2 ☒ #2 4 5 6 7 8

RF POWER LEVEL: 0.05

AUTO ☐

(250)

(500)

(2)

(.05)

SAMPLE: Oil Sample #5

REMARKS:

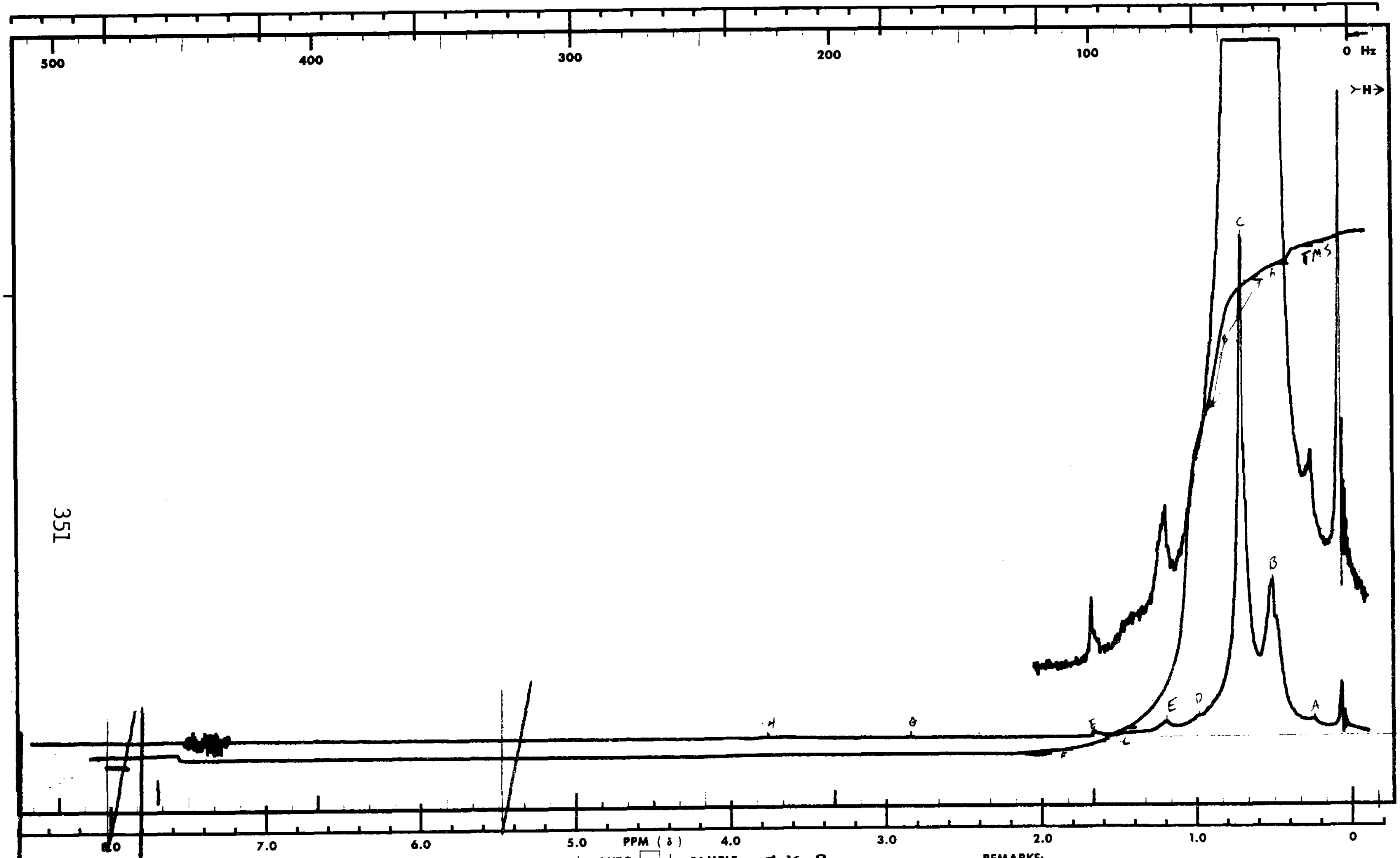
SOLVENT: 50% v/v $CDCl_3$ + 2% TMS

varian
analytical instrument division

DATE: 3/2/78

OPERATOR: M. Barth

60 MHz NMR
SPECTRUM NO.



MANUAL

AUTO ☐

SWEEP OFFSET (Hz): 0

SPECTRUM AMPLITUDE: 1.25 + 12.5

INTEGRAL AMPLITUDE: 7

SPINNING RATE (RPS): 53

SWEEP TIME (SEC): 50 250 (250)

SWEEP WIDTH (Hz): 25 50 100 250 500 1000 (500)

FILTER: ☒ 2 3 4 5 6 7 8 (2)

RF POWER LEVEL: 0.05 (.05)

SAMPLE: Oil Sample No. 5

REMARKS:

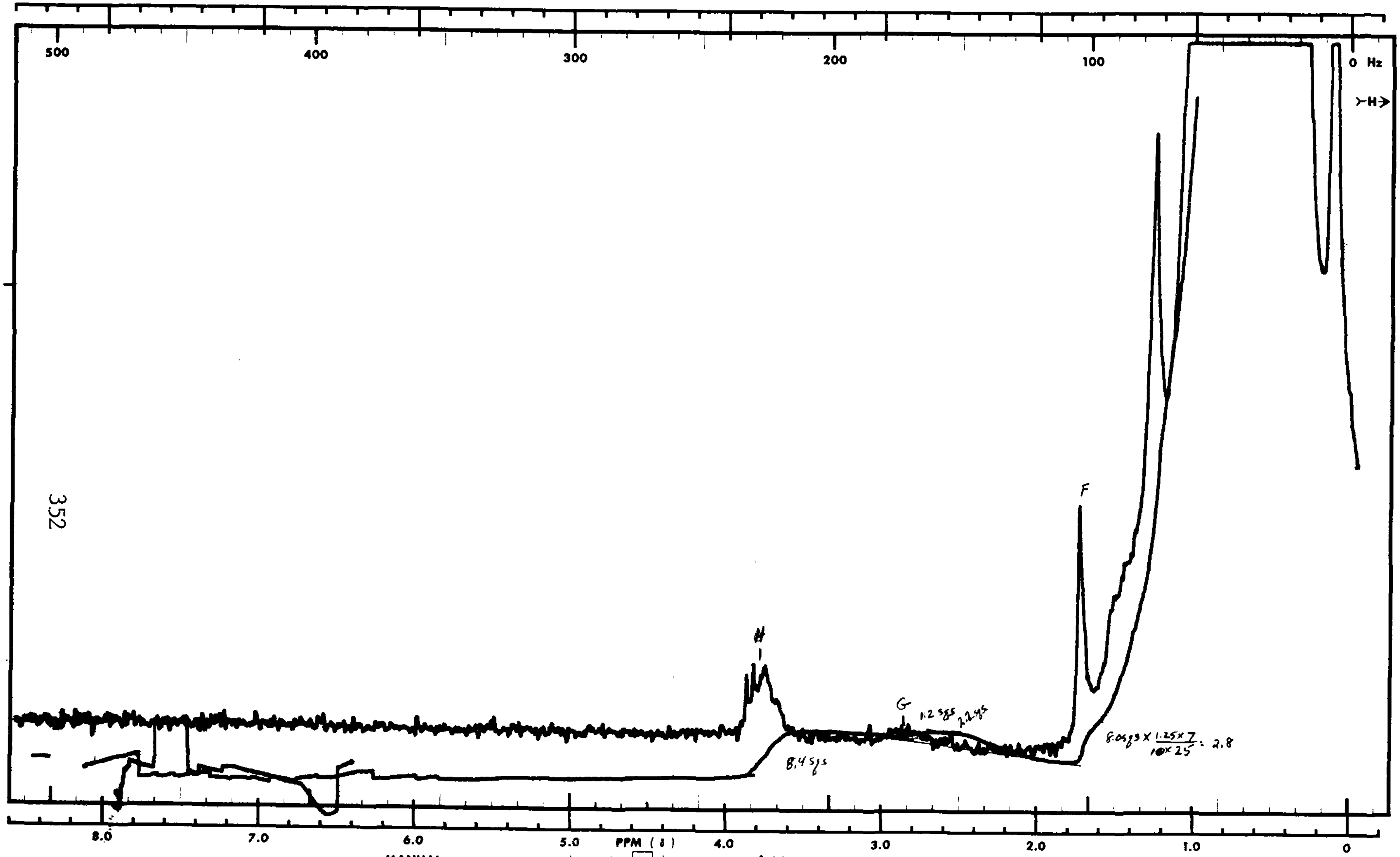
SOLVENT: 50% $CDCl_3$ + 2% TMS

varian
analytical instrument division

DATE: 7/22/78

OPERATOR: M. BARTH

60 MHz NMR
SPECTRUM NO.



MANUAL

SWEEP OFFSET (Hz): 0
SPECTRUM AMPLITUDE: 50
INTEGRAL AMPLITUDE: 10 1.0 x 25
SPINNING RATE (RPS): 53

SWEEP TIME (SEC): 50 250 1000
SWEEP WIDTH (Hz): 25 50 100 250 500 1000
FILTER: 1 2 3 ☒ 5 6 7 8
RF POWER LEVEL: 0.05

AUTO ☐
(250)
(500)
(2)
(.05)

SAMPLE: 0.7 Sample #5
REMARKS:

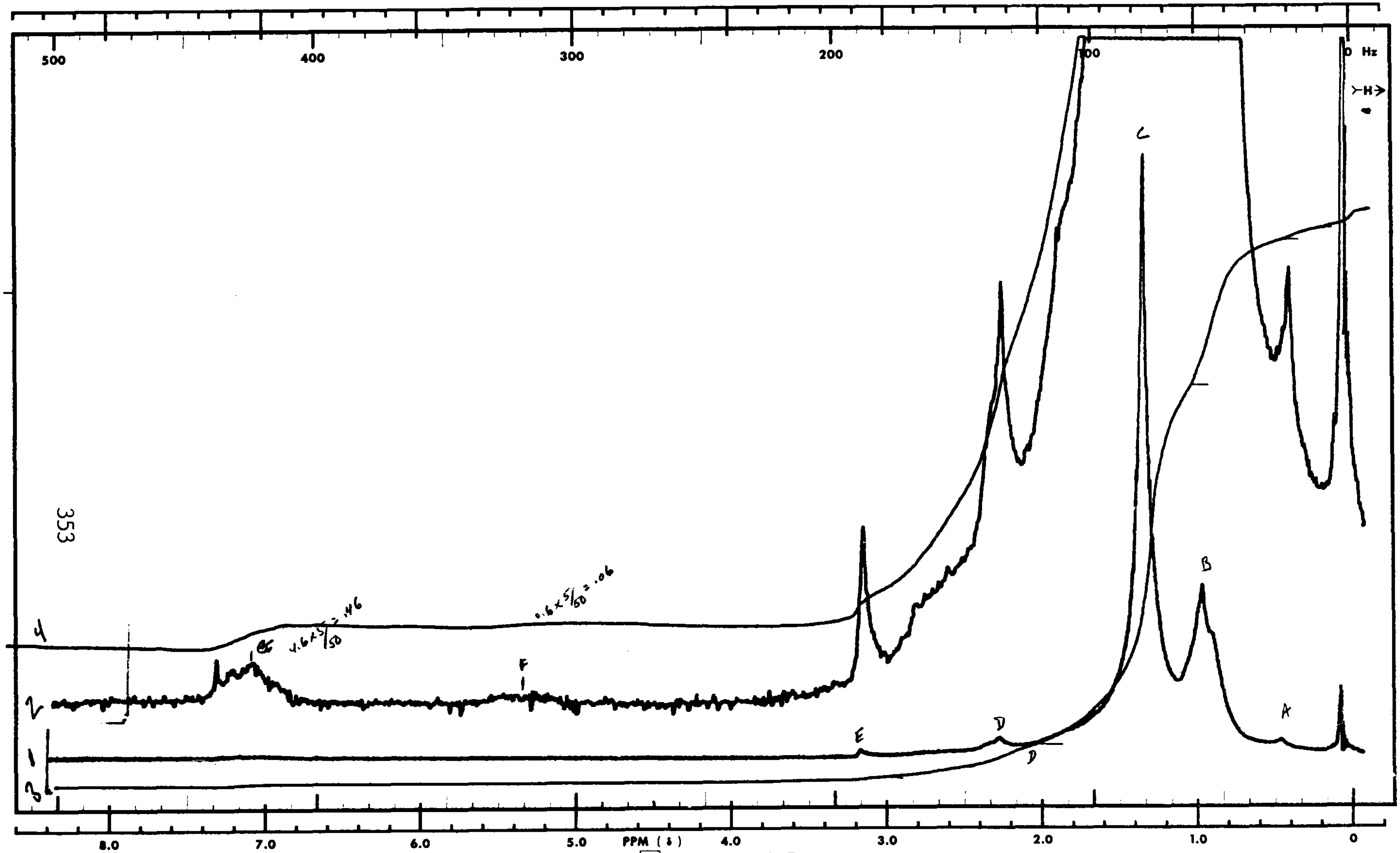
SOLVENT: 98% CDCl3 + 2% TMS

varian
analytical instrument division

DATE: 2/22/78

OPERATOR: M. Barth

60 MHz NMR
SPECTRUM NO.



MANUAL

AUTO ☐

SWEEP OFFSET (Hz): 0

SPECTRUM AMPLITUDE: 1.25 + 25

INTEGRAL AMPLITUDE: 5 × 1.0 (×4 5×10

SPINNING RATE (RPS): 51

SWEEP TIME (SEC): 50 ☒250 ~~500~~ (250)

SWEEP WIDTH (Hz): 25 50 100 250 ☒500 (500)

FILTER: ☒ 2 ☒ 4 5 6 7 8 (2) #2

RF POWER LEVEL: 0.05 (.05)

SAMPLE: Oil Sample No. 7

REMARKS:

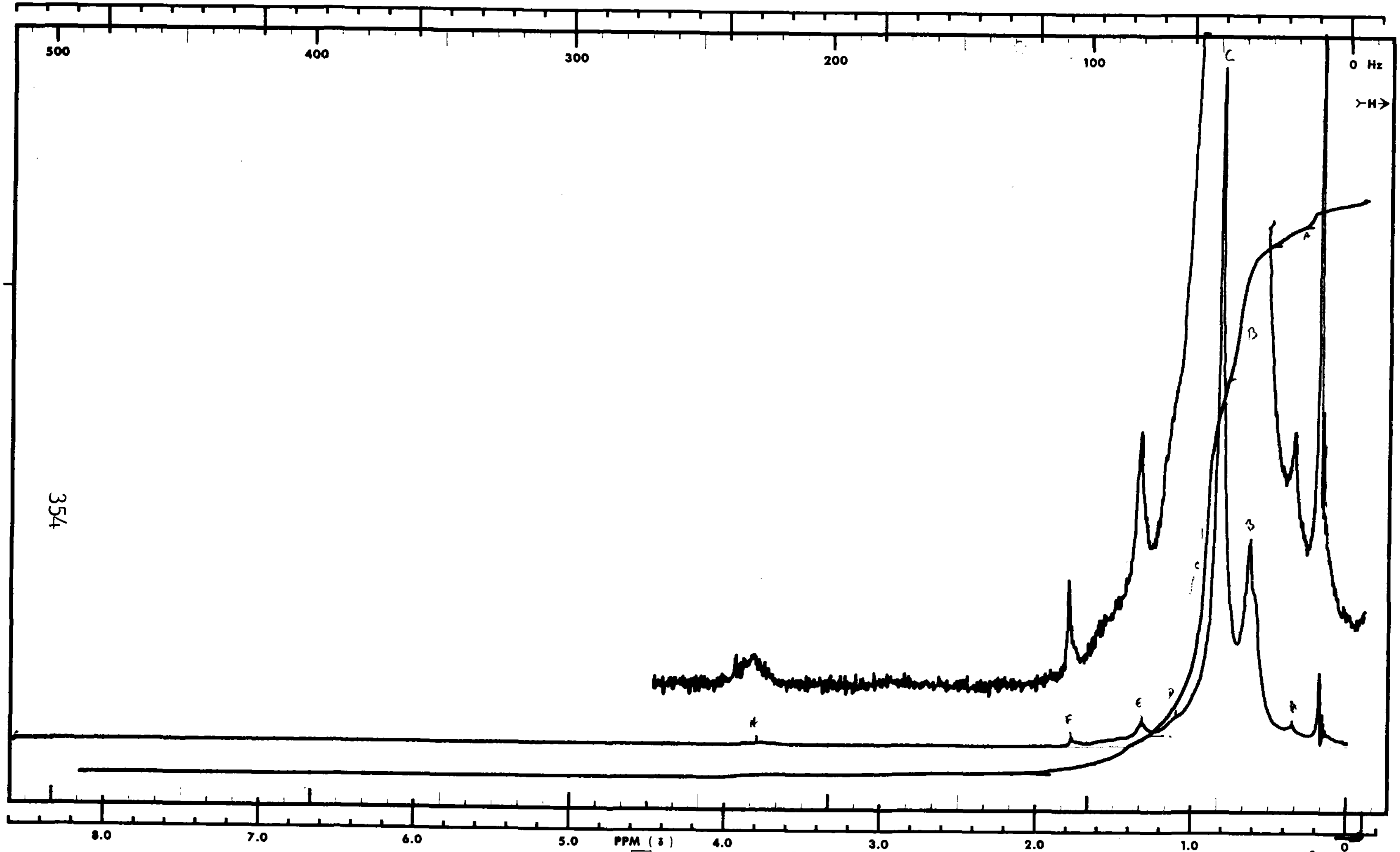
SOLVENT: 50% v/v in CDCl₃, 2% TMS

varian
analytical instrument division

DATE: 3/2/78

OPERATOR: M. Barrett

60 MHz NMR
SPECTRUM NO. A



MANUAL

SWEEP OFFSET (Hz): 0

SPECTRUM AMPLITUDE: 625 + 125

INTEGRAL AMPLITUDE: 7

SPINNING RATE (RPS): 50

SWEEP TIME (SEC): 50 ~~250~~ 1000

SWEEP WIDTH (Hz): 25 50 100 250 500

FILTER: 1 2 3 4 5 6 7 8

RF POWER LEVEL: 0.07

AUTO ☐

(250)

(500)

(2)

(.05)

SAMPLE: Oil Sample #7

50% v/v in

SOLVENT: ~~[illegible]~~ $CDCl_3$ + 2% TMS

REMARKS:

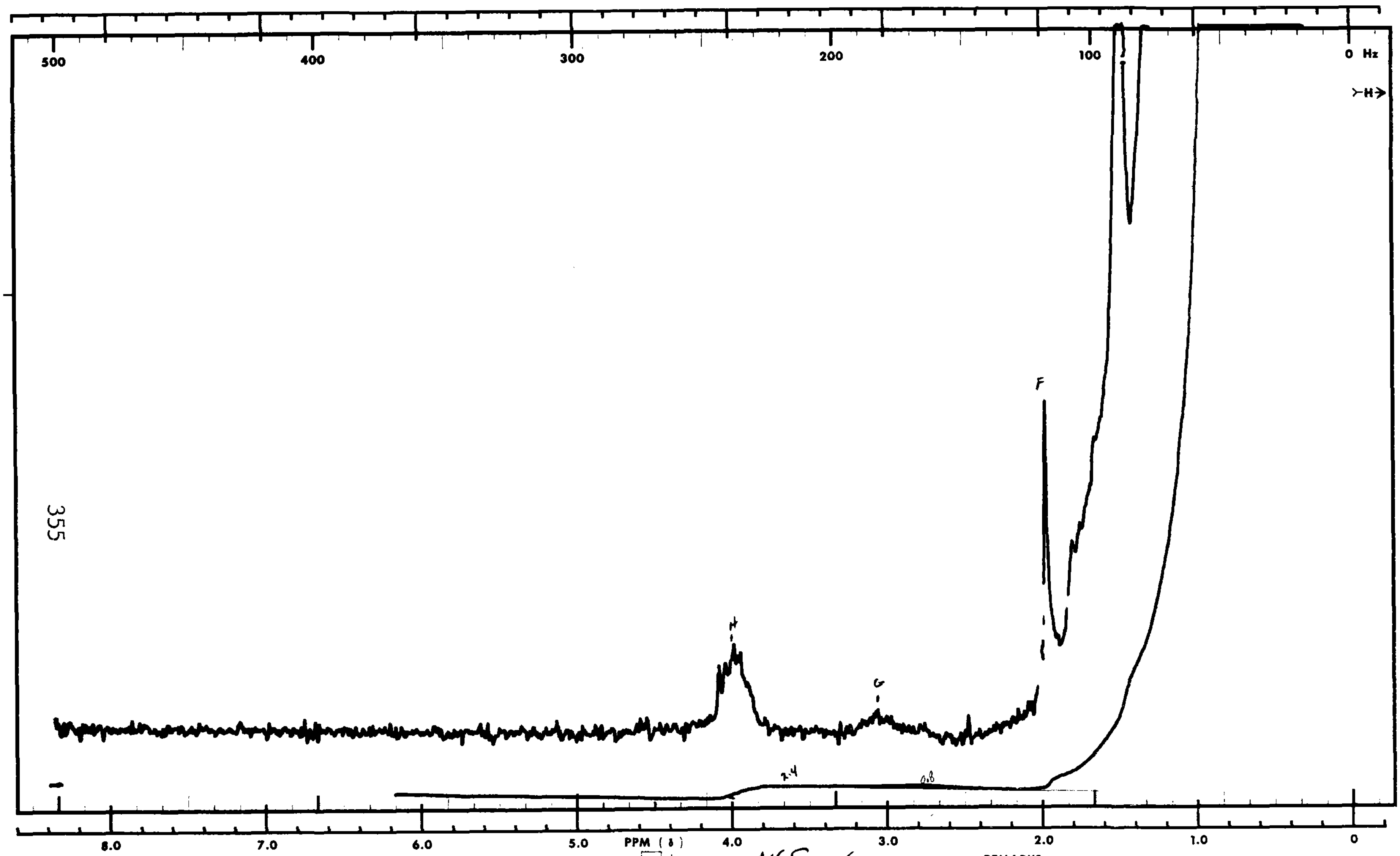
+ R.I. $B/C = \frac{69.2}{25.8} = 2.68$

varian
analytical instrument division

DATE: 2/23/78

OPERATOR: M. Beutler

60 MHz NMR
SPECTRUM NO.



MANUAL

AUTO ☐

SWEEP OFFSET (Hz): 0

SPECTRUM AMPLITUDE: 50

INTEGRAL AMPLITUDE: 1 X 25

SPINNING RATE (RPS): 50

SWEEP TIME (SEC): 50 ~~250~~ 1000 (250)

SWEEP WIDTH (Hz): 25 50 100 250 500 1000 (500)

FILTER: 1 2 3 ☒ 5 6 7 8 (2)

RF POWER LEVEL: 0.07 (.05)

SAMPLE: Oil Sample # 7

REMARKS:

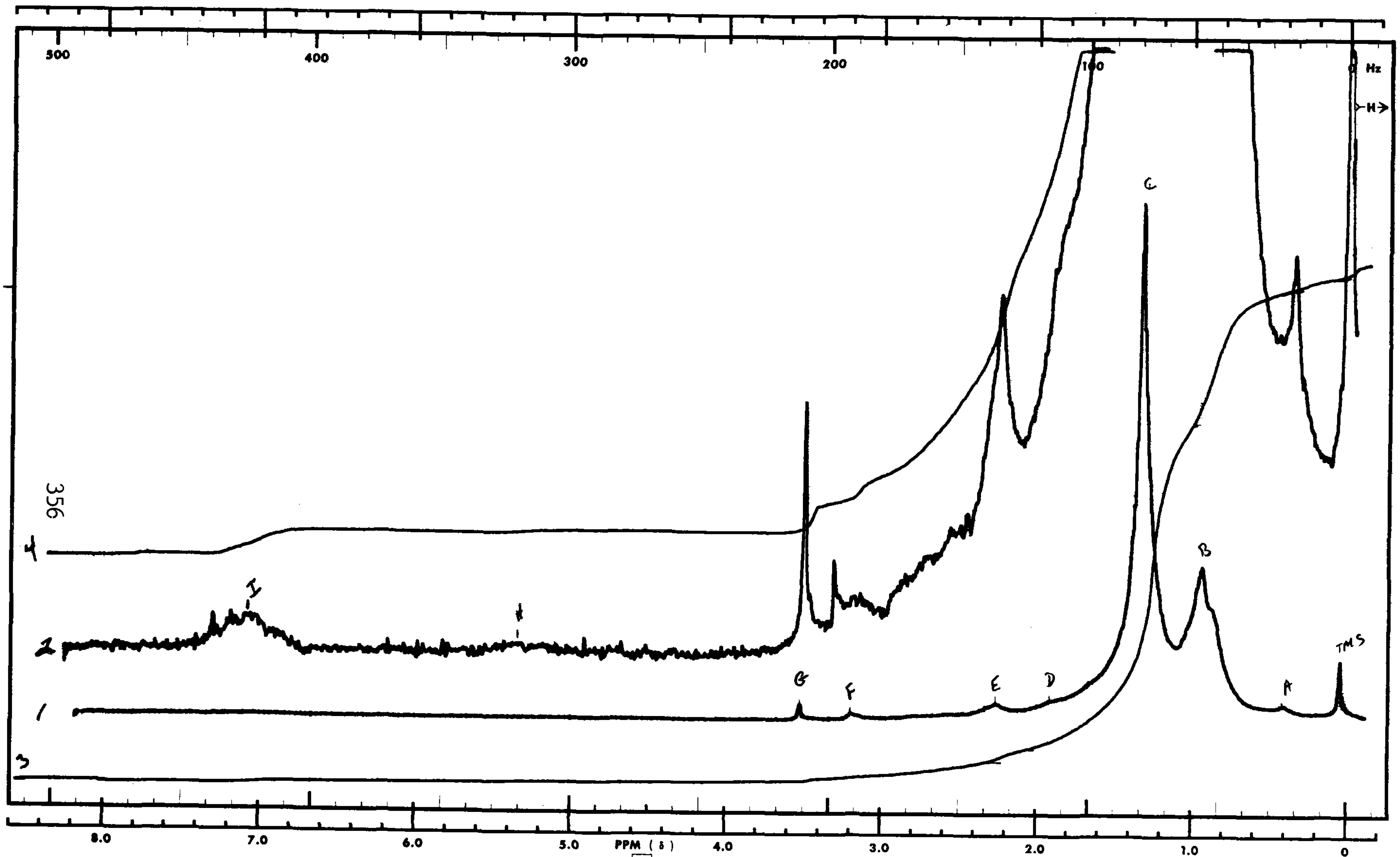
SOLVENT: 50% CDCl$_3$ + 2% TMS

varian
analytical instrument division

DATE: 2/23/78

OPERATOR: M. Barth

60 MHz NMR
SPECTRUM NO.



MANUAL

SWEEP OFFSET (Hz): 0

SPECTRUM AMPLITUDE: 1.25 25 (#2)

INTEGRAL AMPLITUDE: 1[illegible]5.0 ([illegible] for #4)

SPINNING RATE (RPS): 50

SWEEP TIME (SEC): 50 | 250   500

SWEEP WIDTH (Hz): ~~#2~~ 25 | 50 | 100 | 250 | [✓ 500]

FILTER: ~~1~~ | 2 | ~~3~~ | 4 | 5 | 6 | 7 | 8

RF POWER LEVEL: 0.05

AUTO ☐

(250)

(500)

(2)

(.05)

SAMPLE: Oil Sample #9

REMARKS:

SOLVENT: 50% v/v in $CDCl_3$+2% TMS

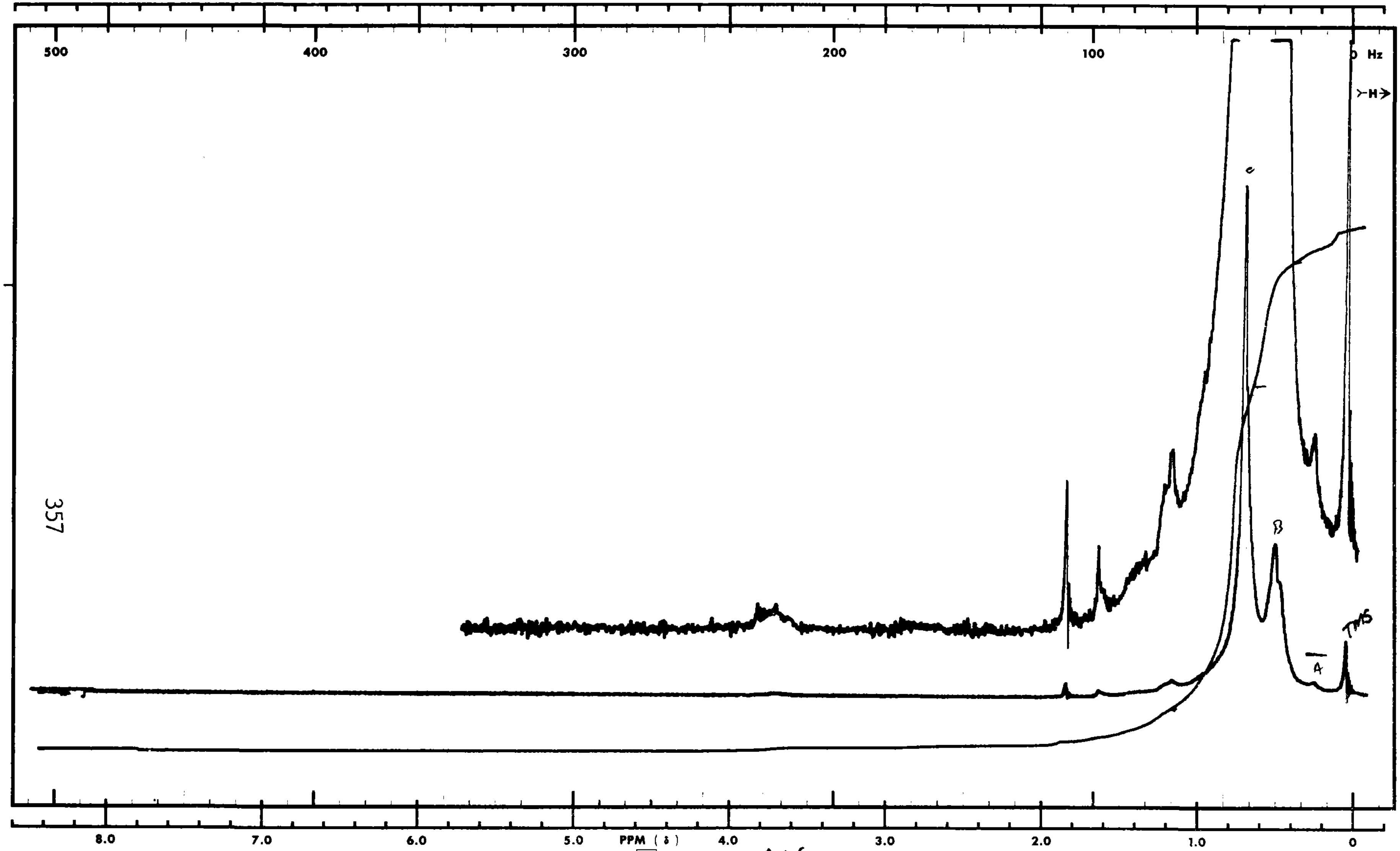
varian
analytical instrument division

DATE: 3/2/78

OPERATOR: M. Beth

60 MHz NMR

SPECTRUM NO.



MANUAL

AUTO ☐

SWEEP OFFSET (Hz): 0

SPECTRUM AMPLITUDE: 125 + 12.5

INTEGRAL AMPLITUDE: 7 x 1.25

SPINNING RATE (RPS): 50

SWEEP TIME (SEC): 50 ~~250~~ 1000 (250)

SWEEP WIDTH (Hz): 25 50 100 250 500 1000 (500)

FILTER: ☒ 2 3 4 5 6 7 8 (2)

RF POWER LEVEL: 0.05 (.05)

SAMPLE: Oil Sample #8

REMARKS: $B/_c = \frac{62}{24} = 2.58$

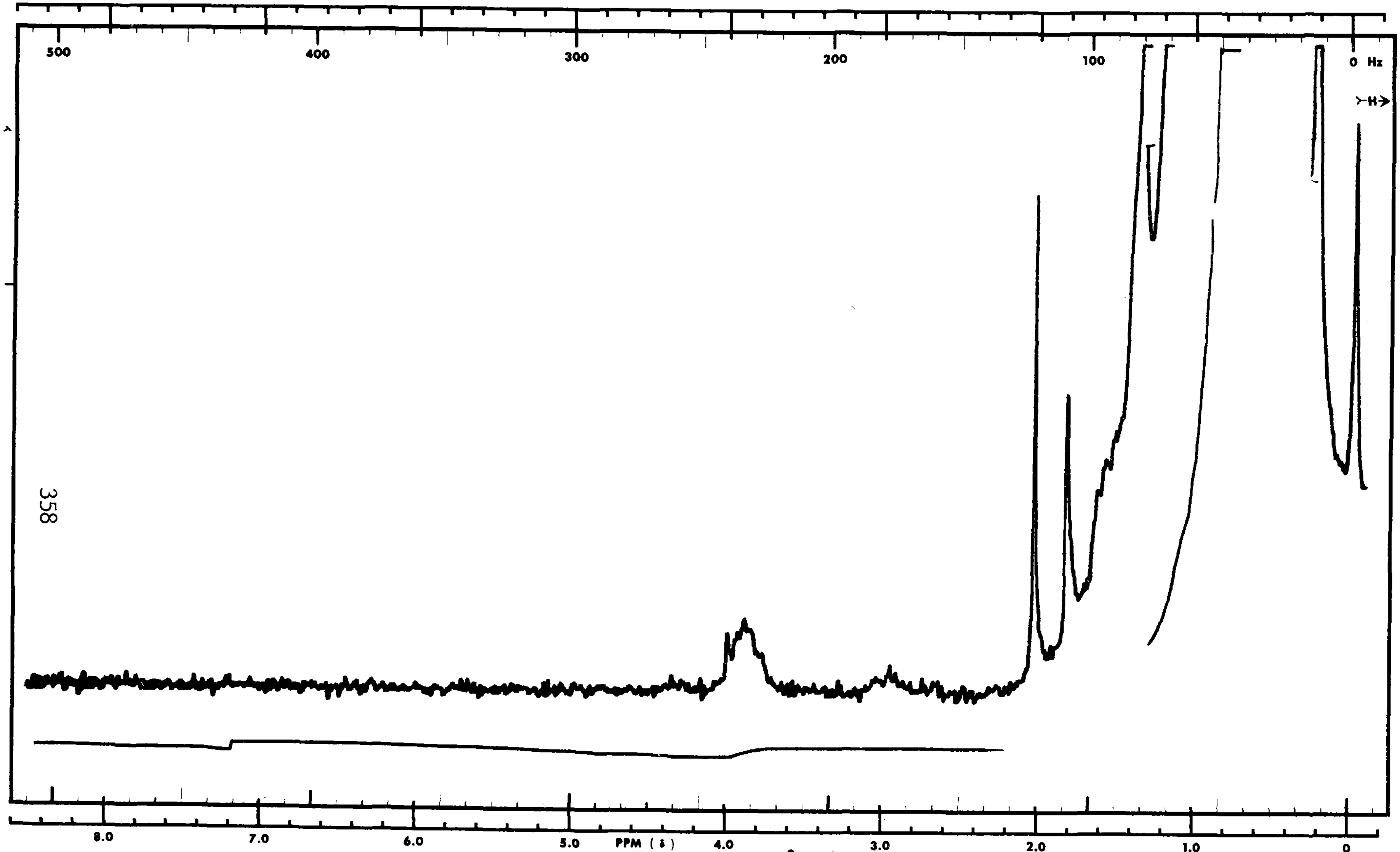
SOLVENT: 50% v/v in $CDCl_3$ + 2% TMS

varian analytical instrument division

DATE: 3/2/78

OPERATOR: M. Barth

60 MHz NMR SPECTRUM NO. A B



MANUAL

AUTO ☐

(250)
(500)
(2)
(.05)

SWEEP OFFSET (Hz): 0
SPECTRUM AMPLITUDE: 50
INTEGRAL AMPLITUDE: 1 × 25
SPINNING RATE (RPS): 50

SWEEP TIME (SEC): 50 250 (600
SWEEP WIDTH (Hz): 25 50 100 250 500 600
FILTER: 1 2 3 [✓ 4] 5 6 7 8
RF POWER LEVEL:

SAMPLE: Oil Sample No. 8

REMARKS:

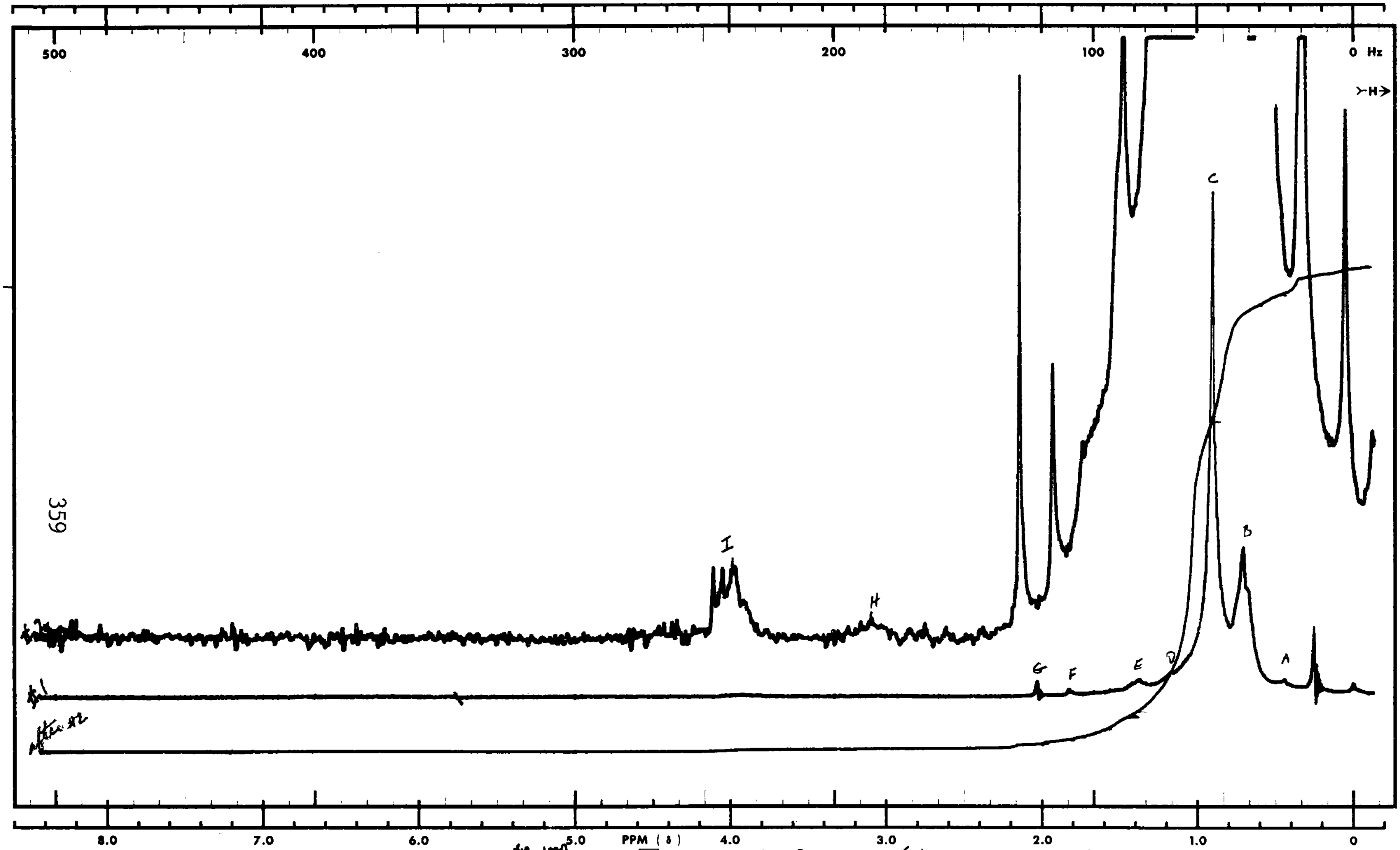
SOLVENT: 50% v/v in $CDCl_3$ + 2% TMS

varian
analytical instrument division

DATE: 3/2/78

OPERATOR: M. Barth

60 MHz NMR
SPECTRUM NO. B



MANUAL #2 · 1000

AUTO ☐

SWEEP OFFSET (Hz): 0 ~~[illegible]~~ 60

SPECTRUM AMPLITUDE: 1.25 + ~~1.25~~

INTEGRAL AMPLITUDE: 7 × 1.25

SPINNING RATE (RPS): 50

SWEEP TIME (SEC): 50 ☒250 (250)

SWEEP WIDTH (Hz): 25 50 100 250 500 1000 (500)

FILTER: ☒1 2 3 4 5 6 7 8 ← #1-4 (2)

RF POWER LEVEL: 0.05 (.05)

SAMPLE: Oil Sample # ~~12~~ 11

REMARKS: Had to adjust sample + spin from #1 → #2

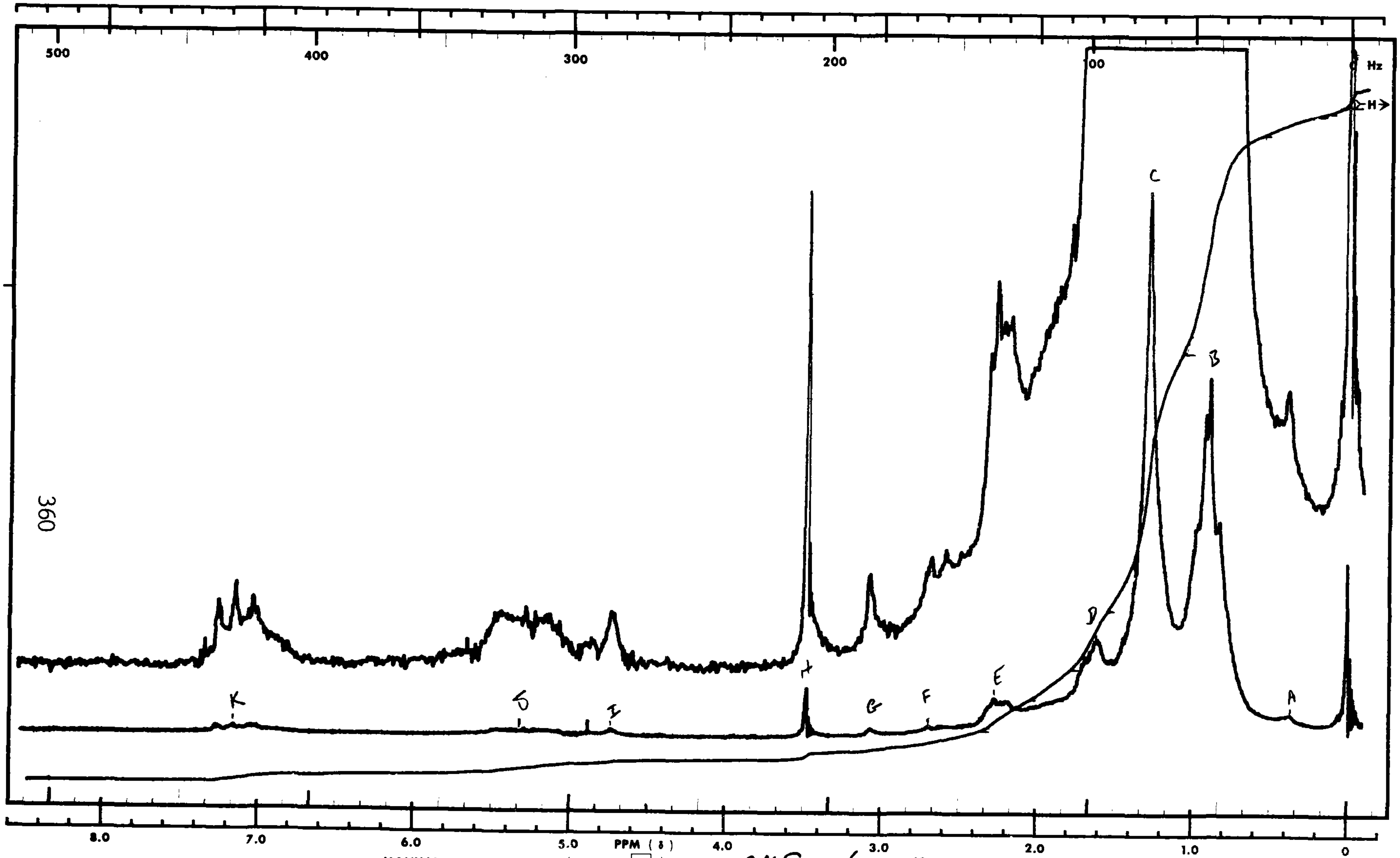
SOLVENT: 50% v/v in $CDCl_3$ + 2% TMS

varian
analytical instrument division

DATE: 3/2/78

OPERATOR: M. Barth

60 MHz NMR
SPECTRUM NO.



MANUAL

SWEEP OFFSET (Hz): 0

SPECTRUM AMPLITUDE: 25 + 25

INTEGRAL AMPLITUDE: 6 x 1.25

SPINNING RATE (RPS): ~~[illegible]~~ 50

SWEEP TIME (SEC): 50 ~~250~~ 500

SWEEP WIDTH (Hz): #1 ~~20~~ 50 100 250 ~~500~~

FILTER: X X X 4 5 6 7 8

RF POWER LEVEL: 0.05

AUTO ☐

(250)

(500)

(2)

(.05)

SAMPLE: Oil Sample #12

REMARKS:

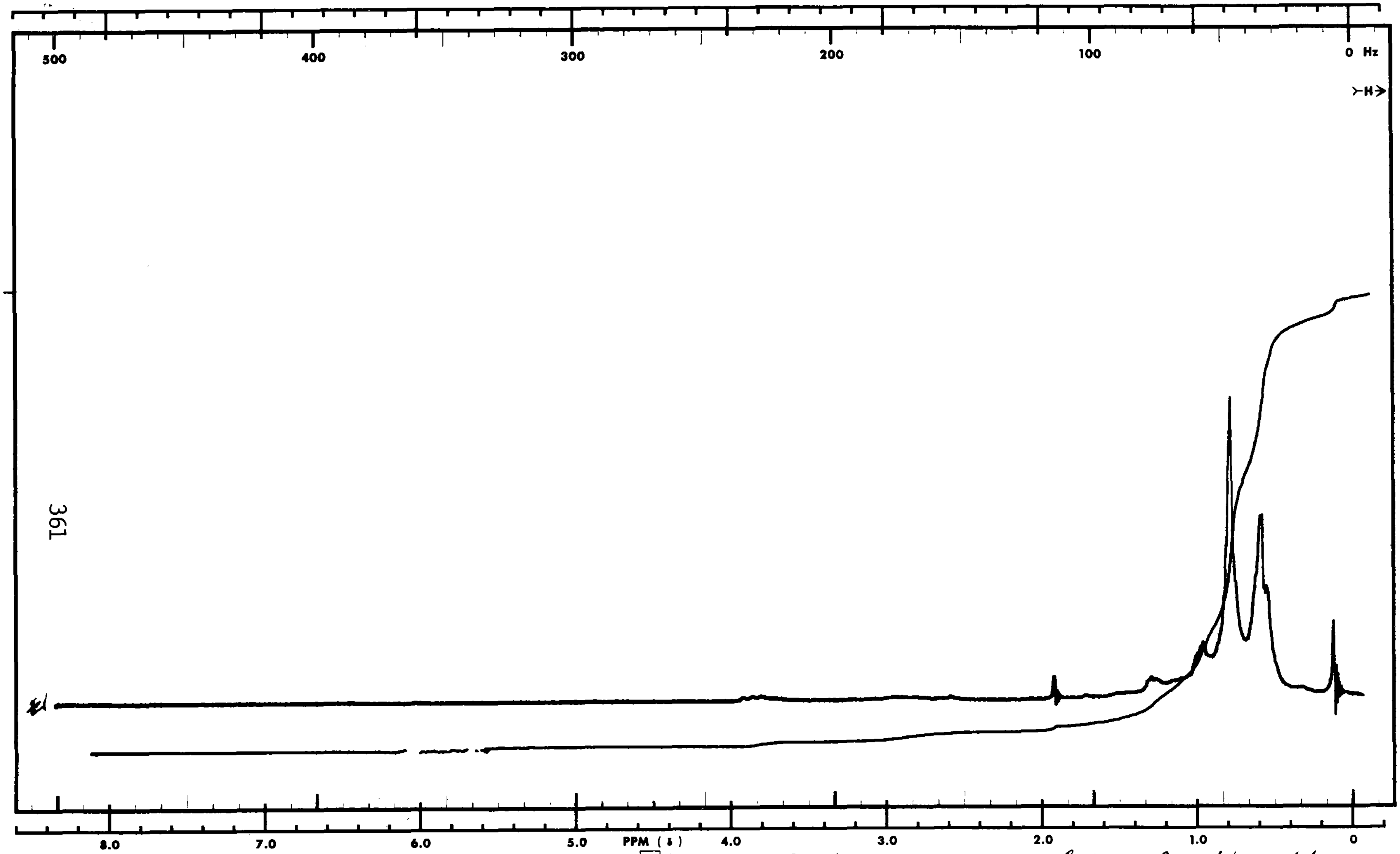
SOLVENT: 50% v/v in $CDCl_3$ + 2% TMS

varian
analytical instrument division

DATE: 3/2/78

OPERATOR: M. Barth

60 MHz NMR
SPECTRUM NO. B



MANUAL

SWEEP OFFSET (Hz): 0

SPECTRUM AMPLITUDE: 1.25

INTEGRAL AMPLITUDE: 7

SPINNING RATE (RPS): 50

SWEEP TIME (SEC): 50 ~~250~~ 1000 #2

SWEEP WIDTH (Hz): 25 50 100 250 500 1000

FILTER: ~~1~~ 2 3 4 5 6 7 8

RF POWER LEVEL: 0.05 2nd

AUTO ☐

(250)

(500)

(2)

(.05)

SAMPLE: Oil Sample #12

REMARKS: Condensate from 2/8/78 Vent down.

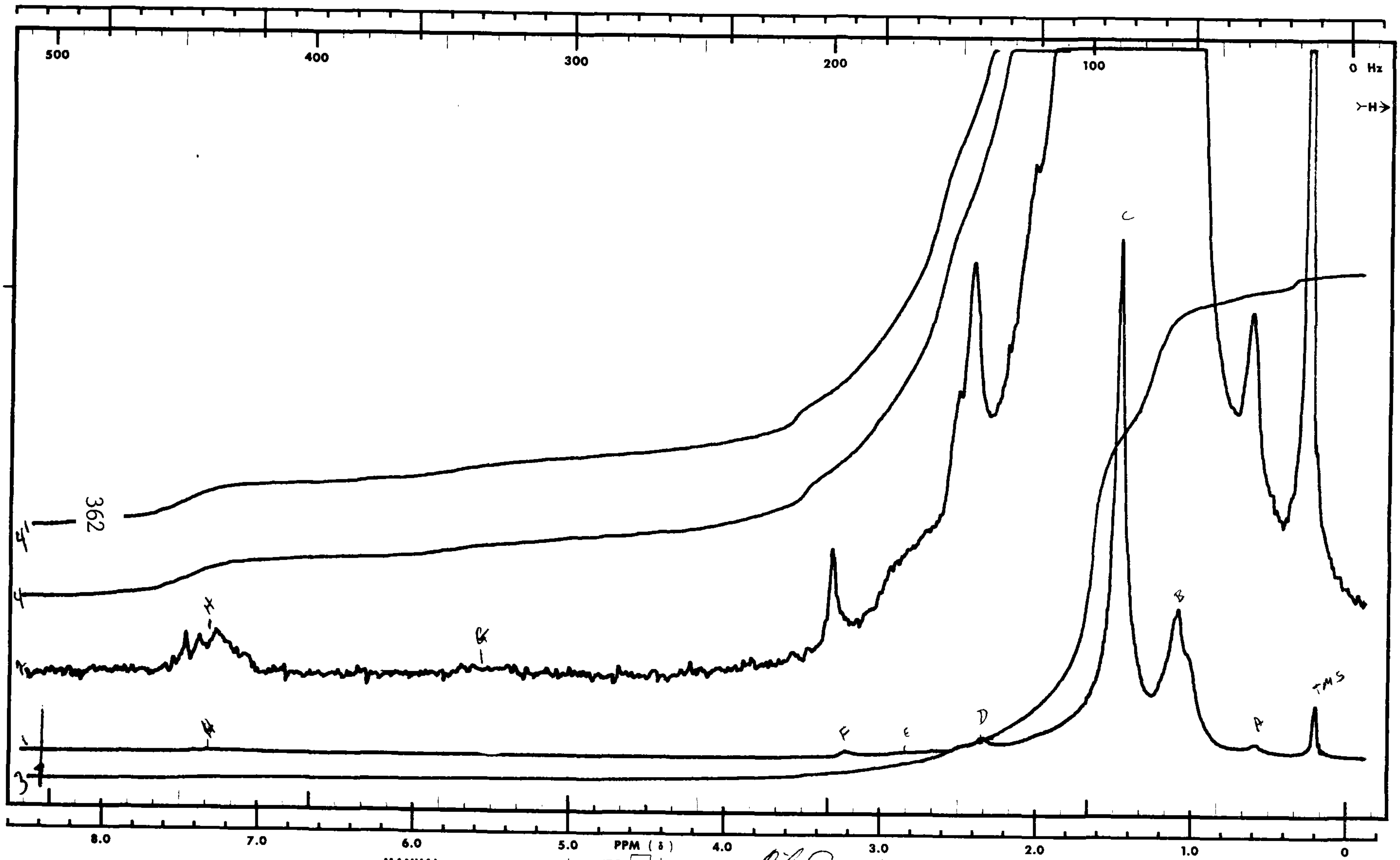
SOLVENT: 50% v/v in $CDCl_3$ + 2% TMS.

varian
analytical instrument division

DATE: 3/2/78

OPERATOR: M. Barth

60 MHz NMR
SPECTRUM NO. A



MANUAL

SWEEP OFFSET (Hz): 0
SPECTRUM AMPLITUDE: 1.25 + 25
INTEGRAL AMPLITUDE: x5 5x1.0 (#4 5x10)
SPINNING RATE (RPS): 50

SWEEP TIME (SEC): 50 ☒250
SWEEP WIDTH (Hz): 25 50 100 250 ☑500
FILTER: ☒1 2 ☒3 4 5 6 7 8
RF POWER LEVEL 0.05 #2

AUTO ☐
(250)
(500)
(2)
(.05)

SAMPLE: Oil Sample No. B
REMARKS:

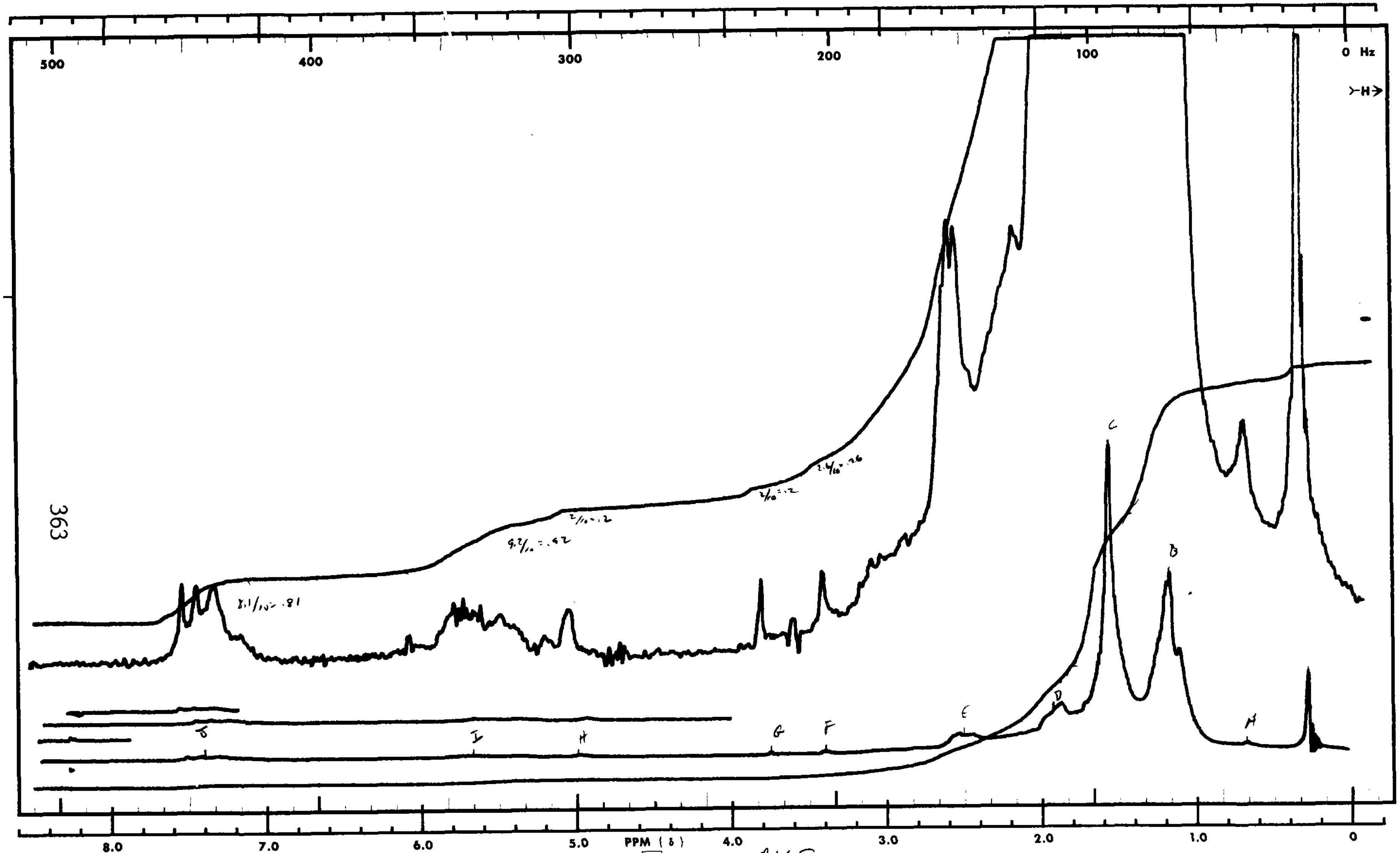
SOLVENT: 50% v/vm CDCl$_3$ + 2% TMS

varian
analytical instrument division

DATE: 3/17/78

OPERATOR: M. Barth

60 MHz NMR
SPECTRUM NO.



MANUAL

SWEEP OFFSET (Hz): 0

SPECTRUM AMPLITUDE: 1.25 + 25

INTEGRAL AMPLITUDE: (3) 5×1.0 + 5×10 (4)

SPINNING RATE (RPS): 50

SWEEP TIME (SEC): 50 ☒ 250

SWEEP WIDTH (Hz): 25 50 100 250 ☒ 500

FILTER: ☒ 1 2 ☒ 3 4 5 6 7 8 #2

RF POWER LEVEL: 0.05

AUTO ☐

(250)

(500)

(2)

(.05)

SAMPLE: Oil Sample #15

REMARKS:

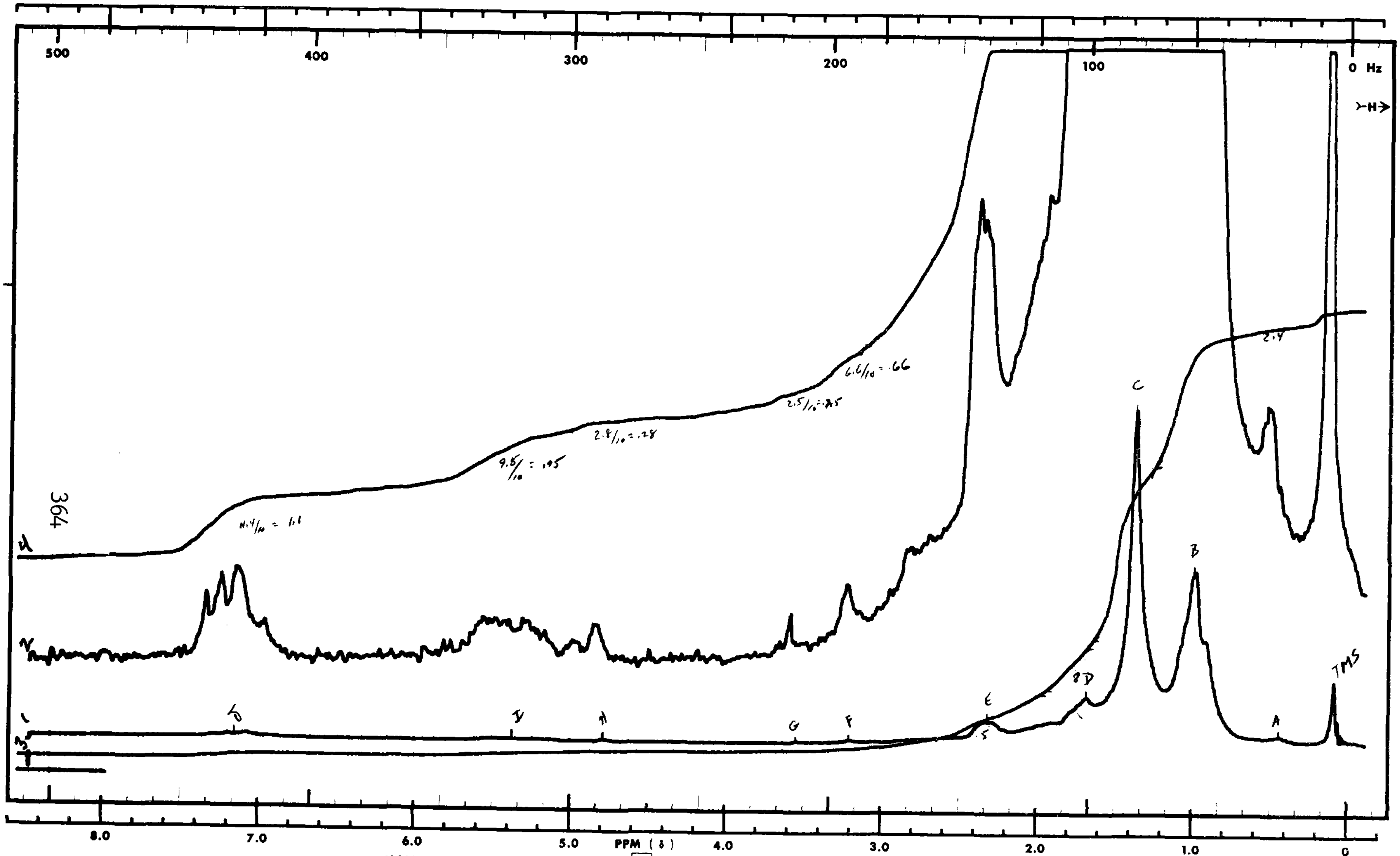
SOLVENT: 50% v/v in CDCl$_3$ + 2% TMS

varian
analytical instrument division

DATE: 3/17/78

OPERATOR: M. Barth

60 MHz NMR
SPECTRUM NO.



MANUAL

SWEEP OFFSET (Hz): 0

SPECTRUM AMPLITUDE: 1.25 + 25

INTEGRAL AMPLITUDE: (#3) 5 × 1.0 + 5 × 10 (#4)

SPINNING RATE (RPS): 51

SWEEP TIME (SEC): 50 ~~250~~

SWEEP WIDTH (Hz): 25 50 100 250 ~~500~~

FILTER: ~~1~~ 2 ~~3~~ 4 5 6 7 8 #2

RF POWER LEVEL: 0.05

AUTO ☐

(250)

(500)

(2)

(.05)

SAMPLE: Oil Sample #17

REMARKS:

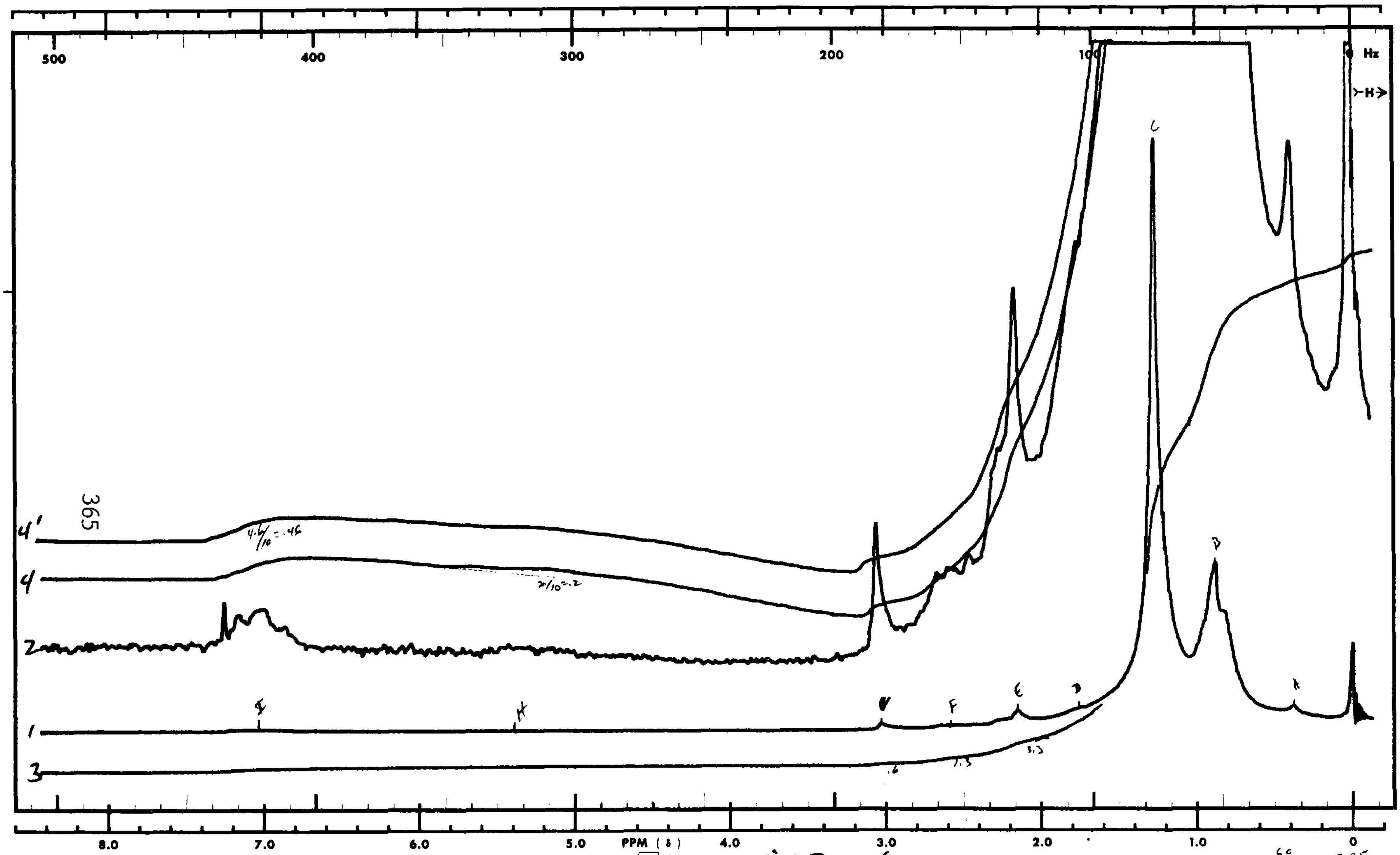
SOLVENT: 50% v/v in $CDCl_3$ + 2% TMS

varian
analytical instrument division

DATE: 3/17/78

OPERATOR M. Barth

60 MHz NMR
SPECTRUM NO.



MANUAL

AUTO ☐

SWEEP OFFSET (Hz): 0

SPECTRUM AMPLITUDE: 1.25 + 25

INTEGRAL AMPLITUDE: #2 5X1 + 5X10 #4

SPINNING RATE (RPS): 50

SWEEP TIME (SEC): 50 25 ☒ (250)

SWEEP WIDTH (Hz): 25 50 100 250 ☒ (500)

FILTER: #1 ☒ 2 ☒ 4 5 6 7 8 (2)

RF POWER LEVEL: 0.05 2#2 (.05)

SAMPLE: Oil Sample #18

REMARKS: c/B = 60/25.5 = 2.35

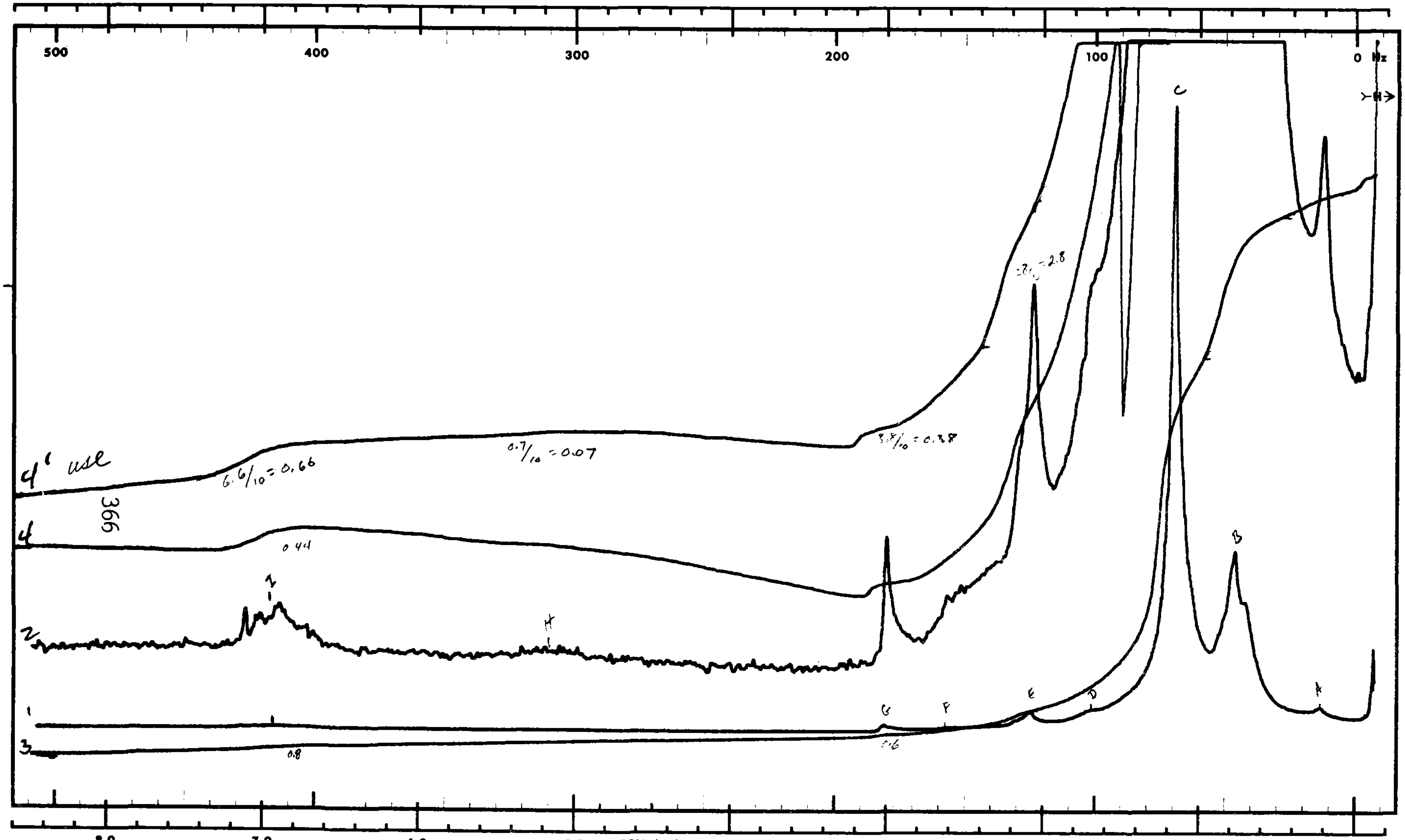
SOLVENT: 50% v/v in CDCl$_3$ + 2% TMS

varian
analytical instrument division

DATE: 3/20/78

OPERATOR: M. Barth

60 MHz NMR
SPECTRUM NO.



MANUAL

SWEEP OFFSET (Hz): 0

SPECTRUM AMPLITUDE: 1.25 + 2.5

INTEGRAL AMPLITUDE: 5×1 (#2) + 5×10 (#4)

SPINNING RATE (RPS): 50

SWEEP TIME (SEC): 50 | ~~250~~

SWEEP WIDTH (Hz): 25 | 50 | 100 | 250 | ~~500~~

FILTER: ~~1~~ | 2 | ~~3~~ | 4 | 5 | 6 | 7 | 8

RF POWER LEVEL: 0.05 #3

AUTO ☐

(250)

(500)

(2)

(.05)

SAMPLE: Oil Sample #19

REMARKS:

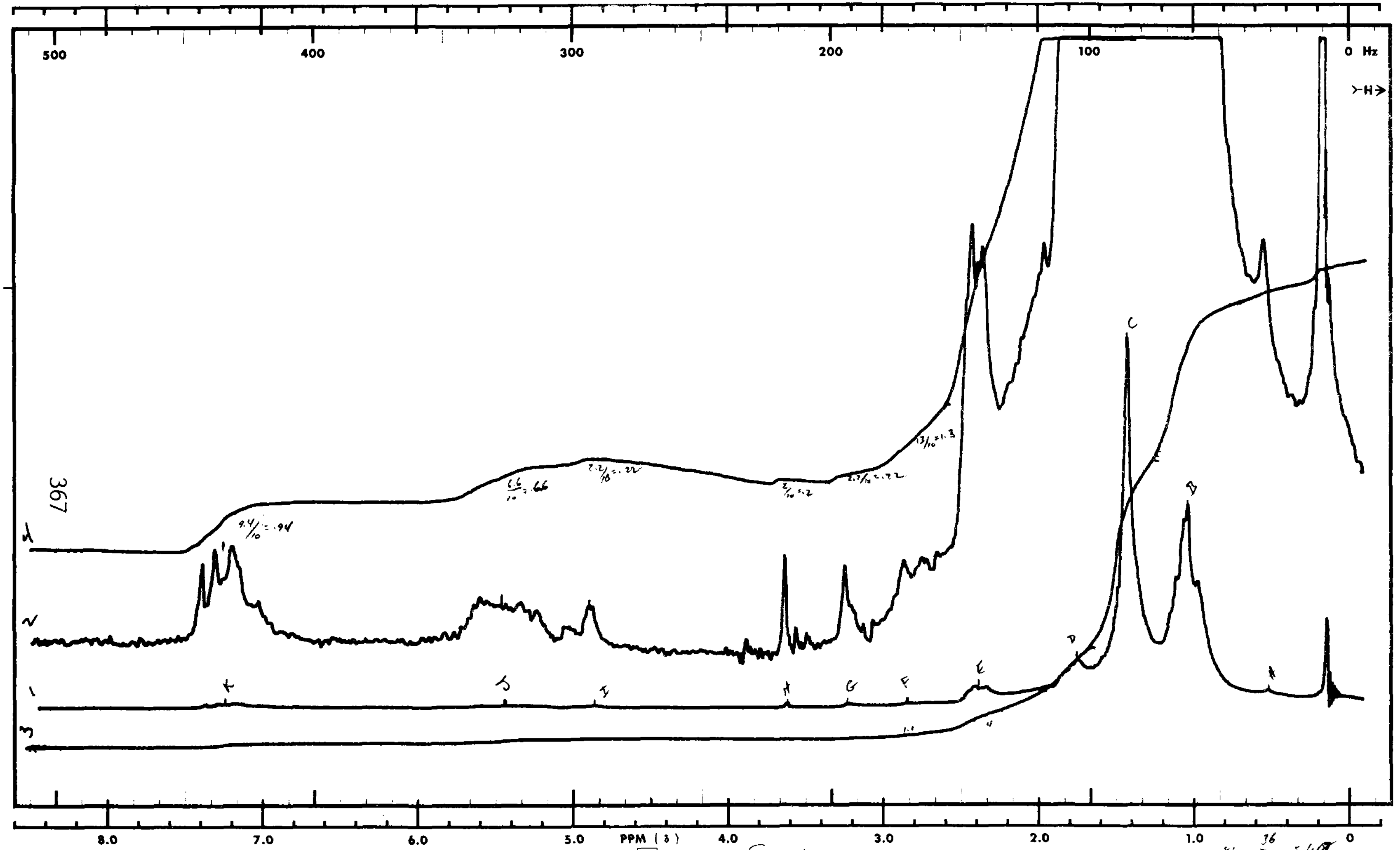
SOLVENT: 50% v/v in CDCl$_3$ + 2% TMS

varian
analytical instrument division

DATE: 3/20/78

OPERATOR: M. Booth

60 MHz NMR
SPECTRUM NO.



MANUAL

SWEEP OFFSET (Hz): 0

SPECTRUM AMPLITUDE: 1.25 + 2.5

INTEGRAL AMPLITUDE: 5X1 (#2) 5X10 (#4)

SPINNING RATE (RPS): 50

SWEEP TIME (SEC): 50 [✓ 250]

SWEEP WIDTH (Hz): 25 50 100 250 [✓ 500]

FILTER: [✓ 1] 2 [✓ 3] 4 5 6 7 8 #1 #3+4

RF POWER LEVEL: 0.05

AUTO ☐

(250)

(500)

(2)

(.05)

SAMPLE: Sample # 20

REMARKS:

c/3 36/30.4 = 1.18

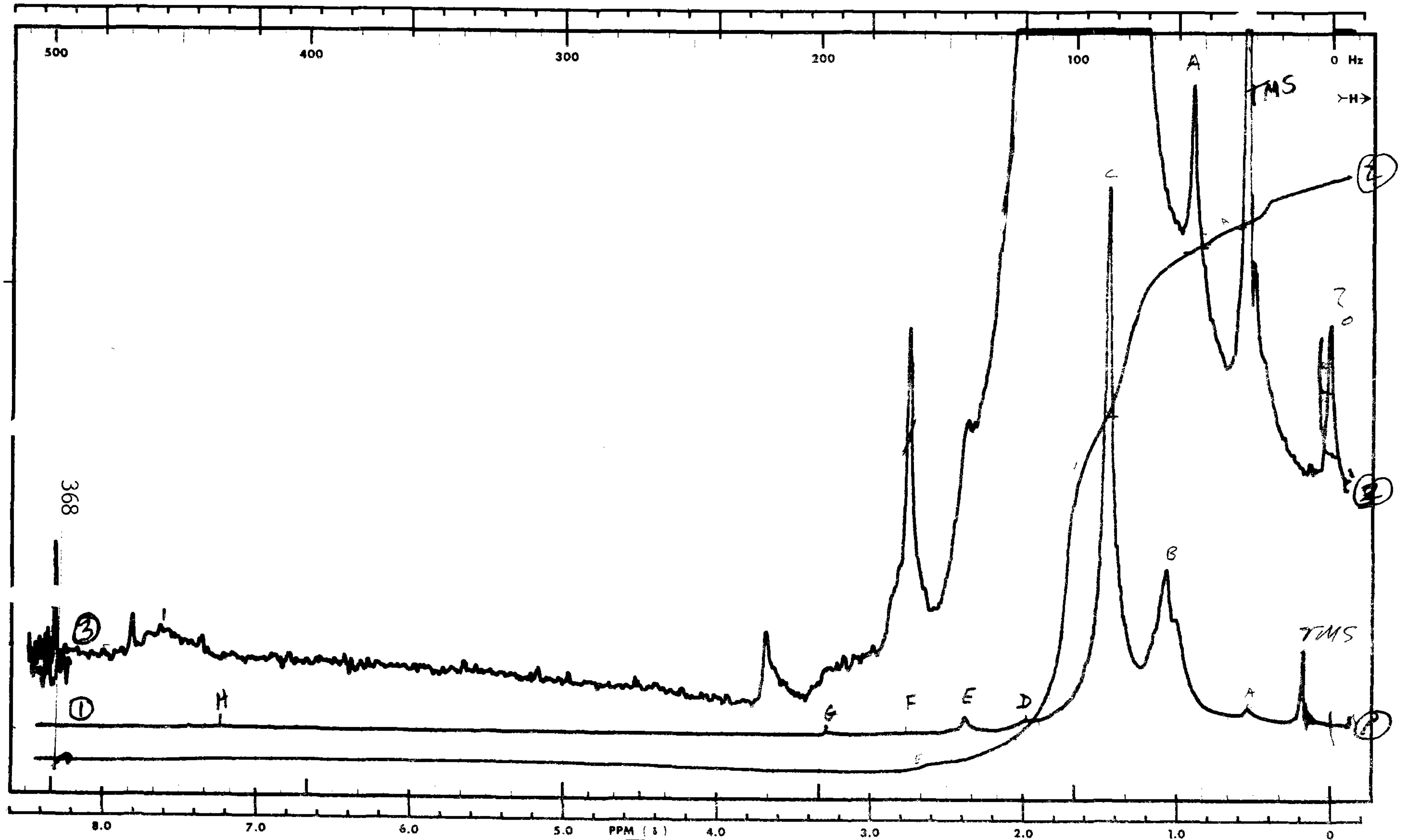
SOLVENT: 50% V/V in CDCl$_3$ + 2% TMS

varian
analytical instrument division

DATE: 3/20/78

OPERATOR: M. Beuth

60 MHz NMR
SPECTRUM NO.



MANUAL

SWEEP OFFSET (Hz): ① 0 ③

SPECTRUM AMPLITUDE: 1.25 + 25

INTEGRAL AMPLITUDE: 5(1.25) 5(40)

SPINNING RATE (RPS): 51

SWEEP TIME (SEC): 50 ☒250

SWEEP WIDTH (Hz): 25 50 100 250 ☒500

FILTER: ☒1 2 ☒3 4 5 6 7 8

RF POWER LEVEL: .05

AUTO ☐

(250)
(500)
(2)
(.05)

SAMPLE: Oil No. [illegible] 21

REMARKS:

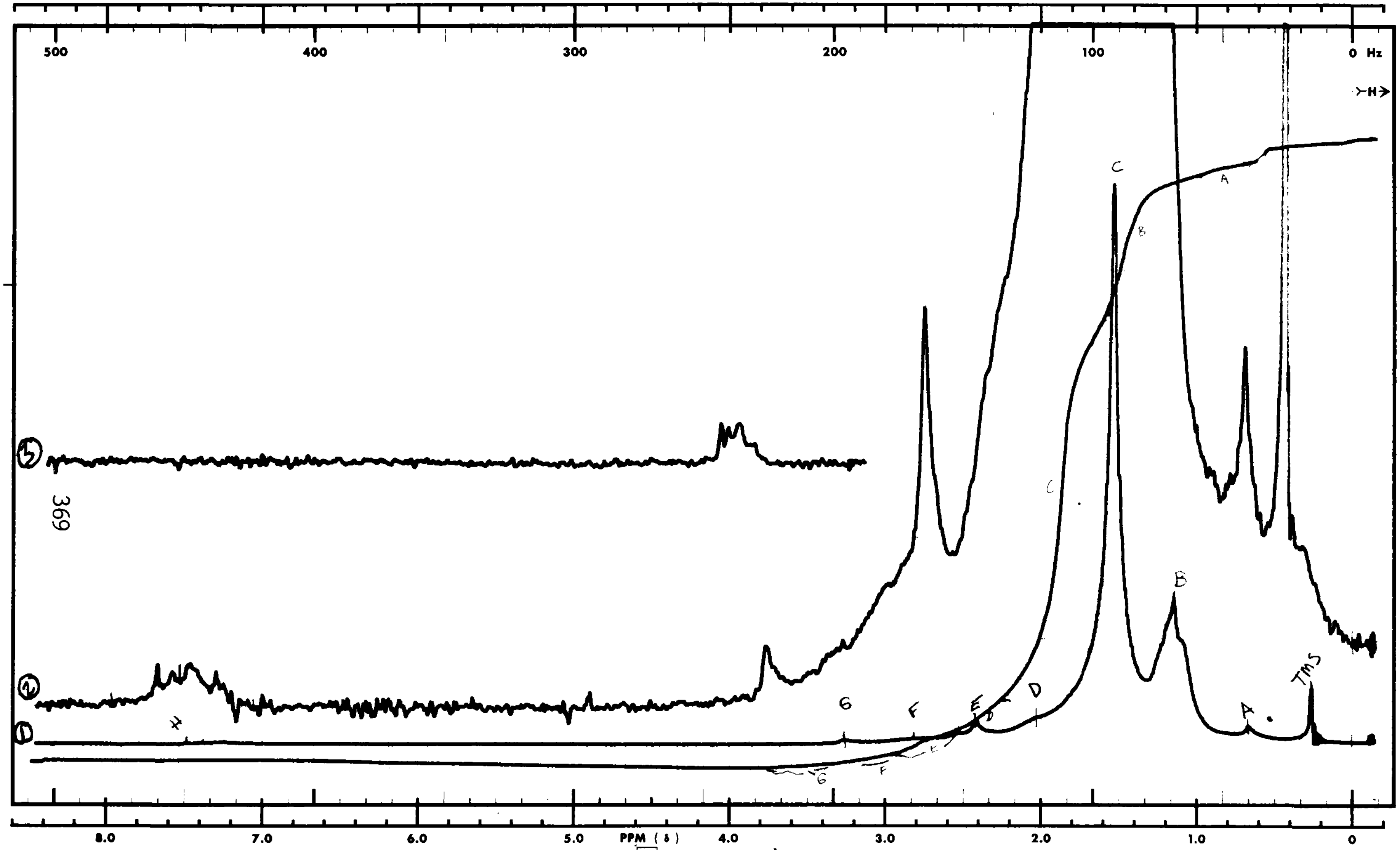
SOLVENT: 1:1 in $CDCl_3$ + 2% TMS

varian
analytical instrument division

DATE: 6/21/78

OPERATOR: M. Barth

60 MHz NMR
SPECTRUM NO.



MANUAL | **AUTO** ☐

SWEEP OFFSET (Hz): 0

SPECTRUM AMPLITUDE: 1.25 ① 25 ②+③

INTEGRAL AMPLITUDE: 5 at 1.25

SPINNING RATE (RPS): 50 on 2 went up to 58

SWEEP TIME (SEC): 50 ~~250~~ 1000 for ③ (250)

SWEEP WIDTH (Hz): 25 50 100 250 ~~500~~ ③ (500)

FILTER: 1 2 ~~3~~ 4 5 6 7 8 (2)

RF POWER LEVEL: 0.05 — for ②+③ (.05)

SAMPLE: ~~Ro~~ Oil No. 22

REMARKS:

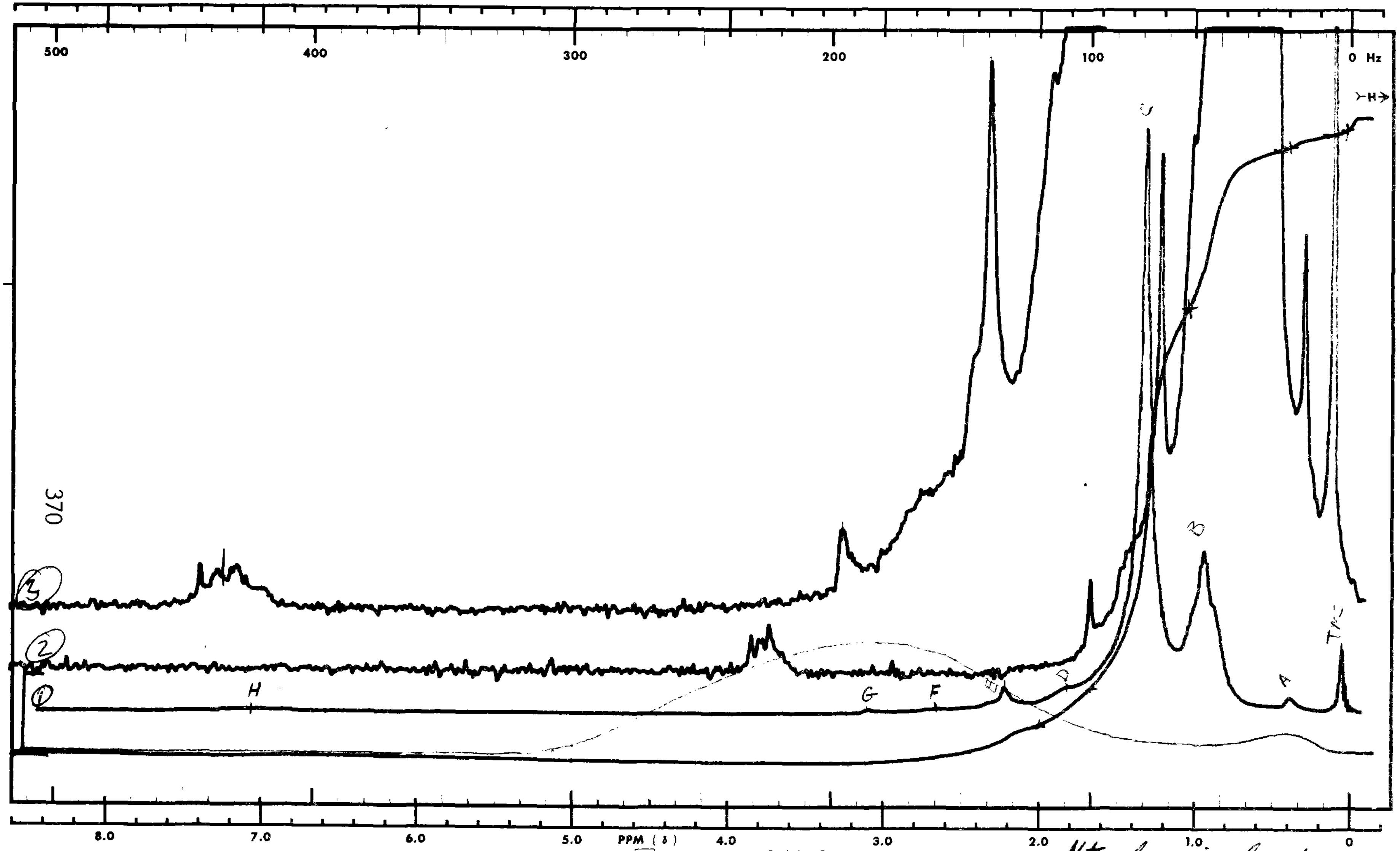
SOLVENT: 1:1 in CDCl₃ + 2% TMS

varian
analytical instrument division

DATE: 6/23/78

OPERATOR: M. Barth

60 MHz NMR
SPECTRUM NO.



MANUAL

AUTO ☐

SWEEP OFFSET (Hz): 0

SPECTRUM AMPLITUDE: 1.25 (1) + 25 (2+3)

INTEGRAL AMPLITUDE: 5 at 1.25 amp.

SPINNING RATE (RPS): 50

SWEEP TIME (SEC): 50 ~~250~~ 1000 for 2 (250)

SWEEP WIDTH (Hz): 1 25 50 100 250 ~~500~~ (500)

FILTER: ~~1~~ 2 ~~3~~ ~~4~~ 5 6 7 8 2+3 (2)

RF POWER LEVEL: 0.05 (.05)

SAMPLE: Oil Sample No 23

REMARKS: Note change in order of scans from those made for other samples.

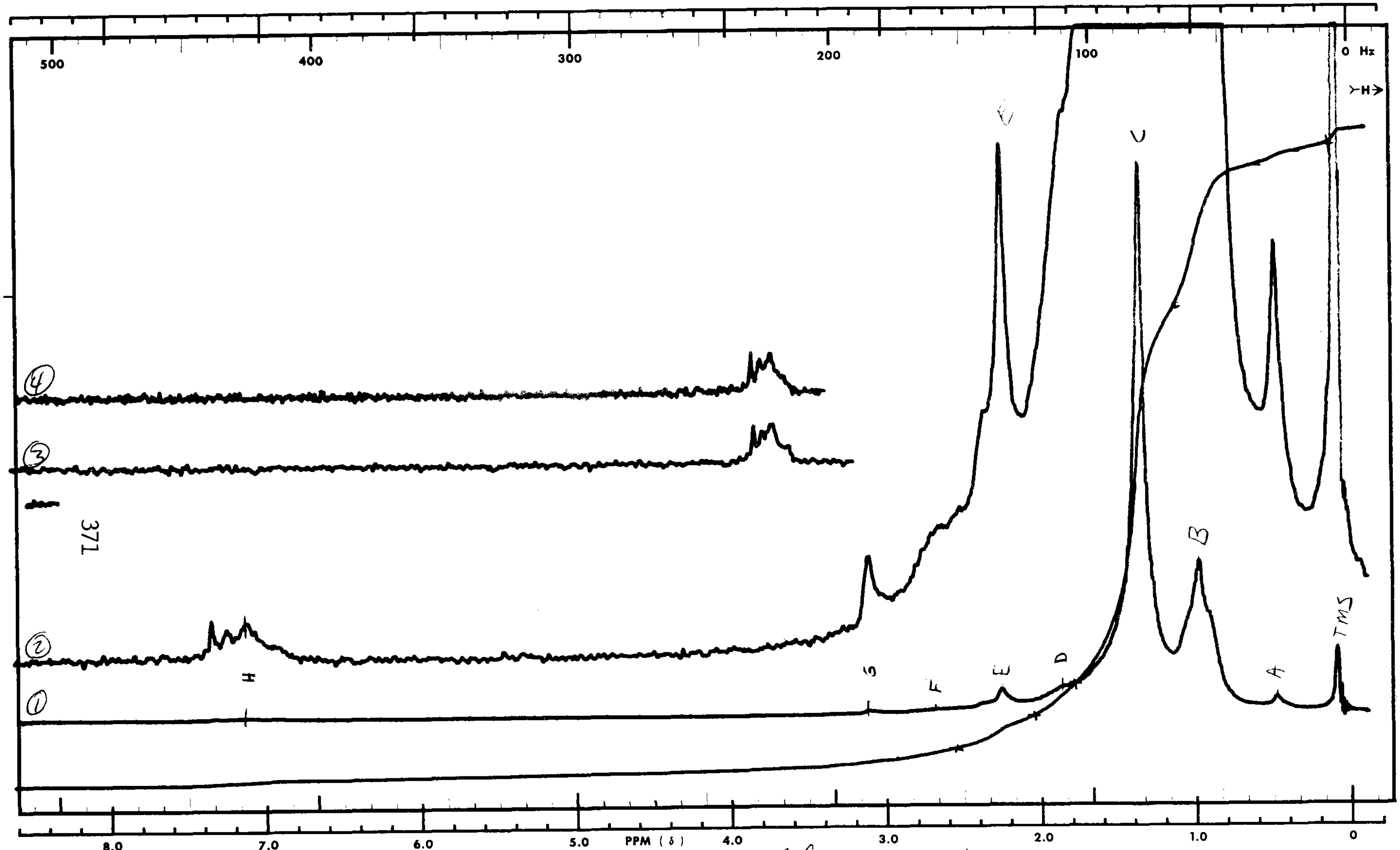
SOLVENT: 50% v/v in ($CDCl_3$ + 2% TMS)

varian
analytical instrument division

DATE: 6/27/78

OPERATOR: M. Barth

60 MHz NMR
SPECTRUM NO.



MANUAL #4-500

AUTO ☐

SWEEP OFFSET (Hz): 0

SPECTRUM AMPLITUDE: 1.25(1) 25(2-4)

INTEGRAL AMPLITUDE: 5 at 1.25 amp

SPINNING RATE (RPS): 49

SWEEP TIME (SEC): ☐ 50 ☒ 250 1000 for 3+4 (250)

SWEEP WIDTH (Hz): ☐ 25 ☐ 50 ☐ 100 ☐ 250 ☒ 500 (500)

FILTER: ☒ ☐ 2 ☒ ☐ ☐ 5 ☐ 6 ☐ 7 ☐ 8 (2)

RF POWER LEVEL: 0.05 — 2-4 (.05)

SAMPLE: Oil Sample No 24 REMARKS:

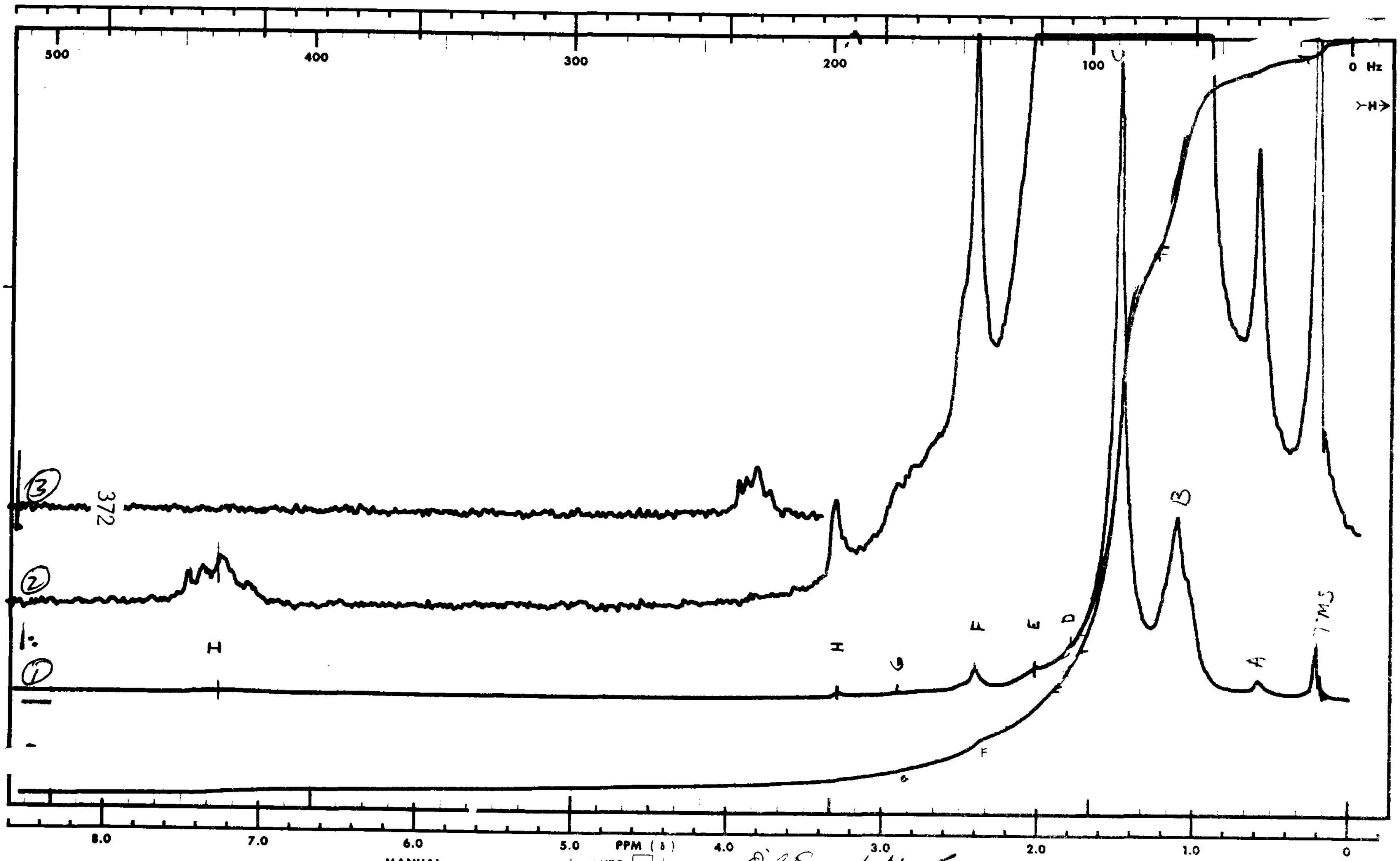
SOLVENT: 50% v/v in ($CDCl_3$ + 2% TMS)

varian
analytical instrument division

DATE: 6/27/78

OPERATOR: M. Barth

60 MHz NMR
SPECTRUM NO.



MANUAL

SWEEP OFFSET (Hz): 0

SPECTRUM AMPLITUDE: 1.25(1) 25(2+3)

INTEGRAL AMPLITUDE: 5 at 1.25amp

SPINNING RATE (RPS): 50

SWEEP TIME (SEC): 50 ☒ 1000 #3

SWEEP WIDTH (Hz): 25 50 100 250 ☒

FILTER: ☒ 2 ☒ 4 5 6 7 8

RF POWER LEVEL: 0.05 ~#2+3

AUTO ☐

(250)

(500)

(2)

(.05)

SAMPLE: Oil Sample No. 25

REMARKS:

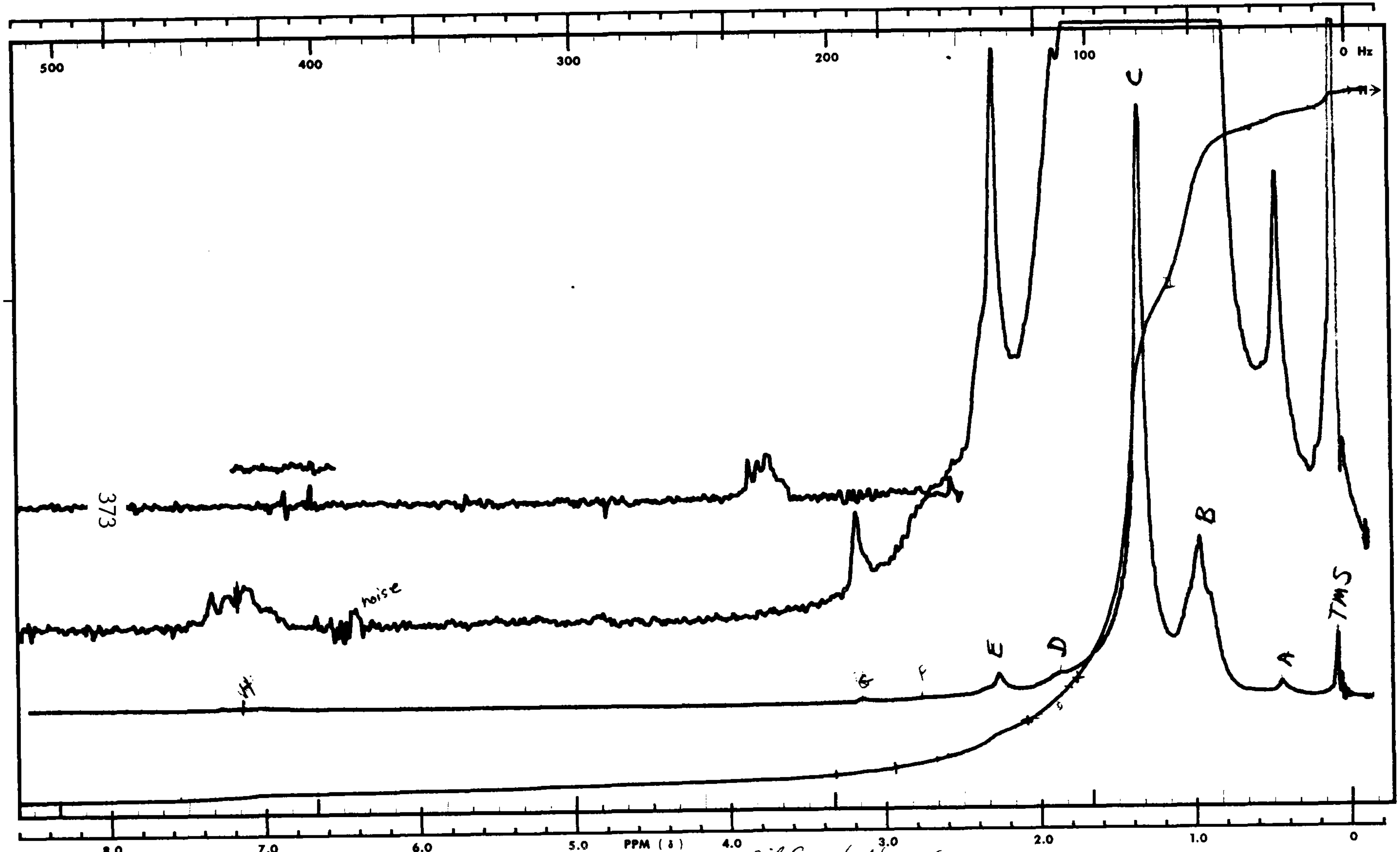
SOLVENT: 50% v/v in (CDCl3 + 2% TMS)

varian
analytical instrument division

DATE: 6/27/78

OPERATOR: M. Barth

60 MHz NMR
SPECTRUM NO.



MANUAL

AUTO ☐

SWEEP OFFSET (Hz): 0

SPECTRUM AMPLITUDE: 1.25(1) 25(2+3)

INTEGRAL AMPLITUDE: 5 at 1.25 amp

SPINNING RATE (RPS): 50

SWEEP TIME (SEC): 50 [✓ 250] 1000 for #3 (250)

SWEEP WIDTH (Hz): 25 50 100 250 [✓ 500] (500)

FILTER: [✓ 1] 2 [✓ 3] 4 5 6 7 8 2+3 (2)

RF POWER LEVEL: 0.05 (.05)

SAMPLE: Oil Sample No. 26

REMARKS:

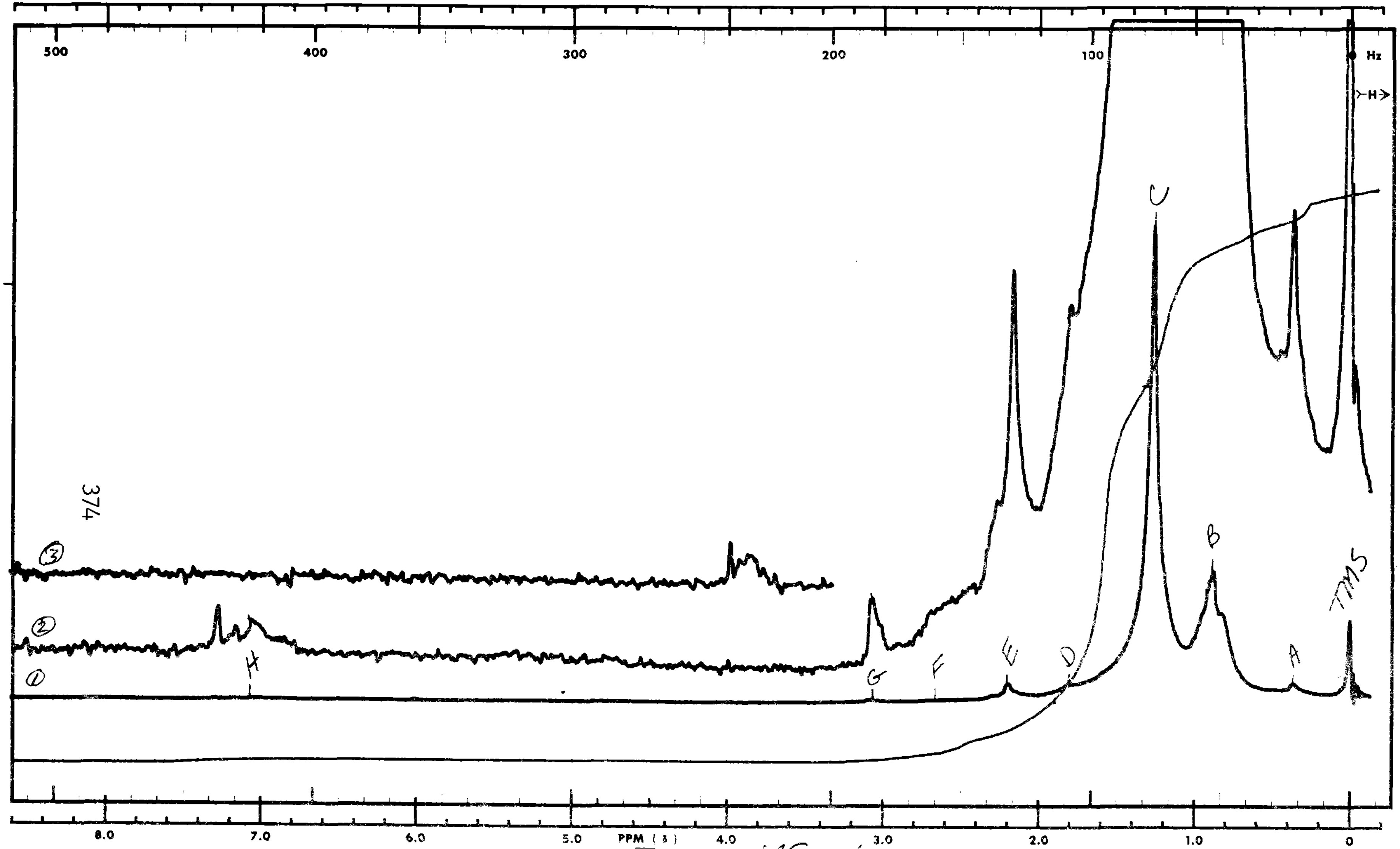
SOLVENT: 50% v/v in (CDCl$_3$ + 2% TMS)

varian
analytical instrument division

DATE: 6/27/78

OPERATOR: M. Barth

60 MHz NMR
SPECTRUM NO.



MANUAL | AUTO ☐

SWEEP OFFSET (Hz): 0
SPECTRUM AMPLITUDE: 1.25(1) 25(2+3)
INTEGRAL AMPLITUDE: 5 at 1.25 Spec. amp
SPINNING RATE (RPS): 51

SWEEP TIME (SEC): 50 ☒250 1000(3) (250)
SWEEP WIDTH (Hz): 25 50 100 250 ☒500 (500)
FILTER: ☒ 2 ☒ 4 5 6 7 8 (2)
RF POWER LEVEL: .05 (2+3) (.05)

SAMPLE: Oil Sample #27
REMARKS:

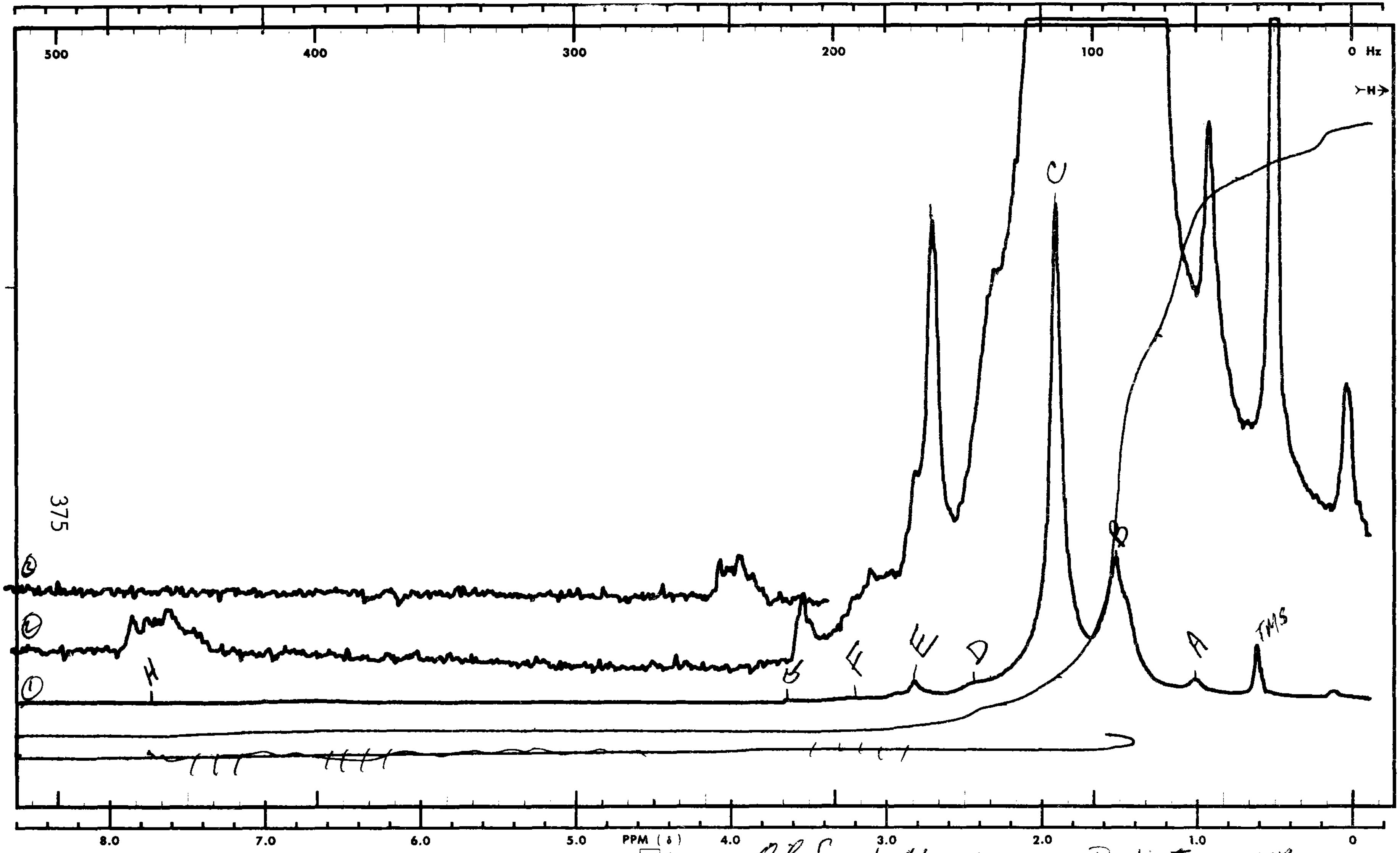
SOLVENT: 50% v/v in (CDCl$_3$ + 2% TMS)

varian
analytical instrument division

DATE: 7/20/78

OPERATOR: M. Barth

60 MHz NMR
SPECTRUM NO.



MANUAL

AUTO ☐

SWEEP OFFSET (Hz): 0

SPECTRUM AMPLITUDE: 1.25 (1) 25 (2+3)

INTEGRAL AMPLITUDE: 5.0 at 1.25 spec amp

SPINNING RATE (RPS): 45

SWEEP TIME (SEC): 50 250 ☒ 1000 (3) (250)

SWEEP WIDTH (Hz): 25 50 100 250 ~~500~~ (500)

FILTER: ☒ 2 ☒ 4 5 6 7 8 (2)

RF POWER LEVEL: .05 (2+3) (.05)

SAMPLE: Oil Sample No. 28

REMARKS: Duplicate - new NMR prep

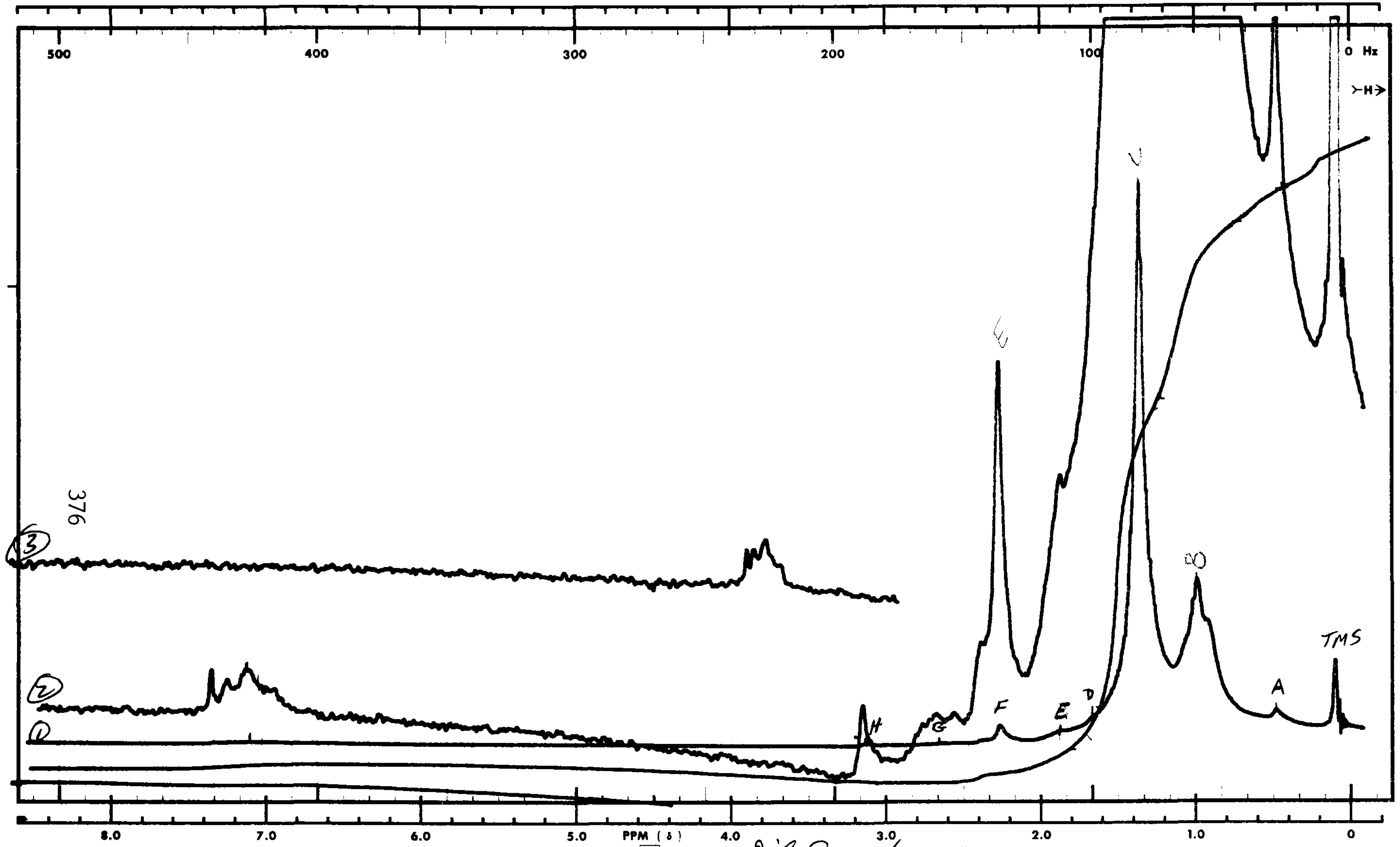
SOLVENT: 50% v/v in (CDCl$_3$ + 2% TMS)

varian
analytical instrument division

DATE: 7/20/78

OPERATOR: M. Barth

60 MHz NMR
SPECTRUM NO.



MANUAL

AUTO ☐

SWEEP OFFSET (Hz): 0 (250)

SPECTRUM AMPLITUDE: 1.25 (1) 25 (2+3)

INTEGRAL AMPLITUDE: 5.0 at 1.25 amp.

SPINNING RATE (RPS): 49

SWEEP TIME (SEC): 50 ~~250~~ 1000 for 2+3 (250)

SWEEP WIDTH (Hz): 25 50 100 250 ~~500~~ (500)

FILTER: ~~1~~ 2 ~~3~~ 4 5 6 7 8 2+3 (2)

RF POWER LEVEL: .05 (.05)

SAMPLE: Oil Sample #28

REMARKS:

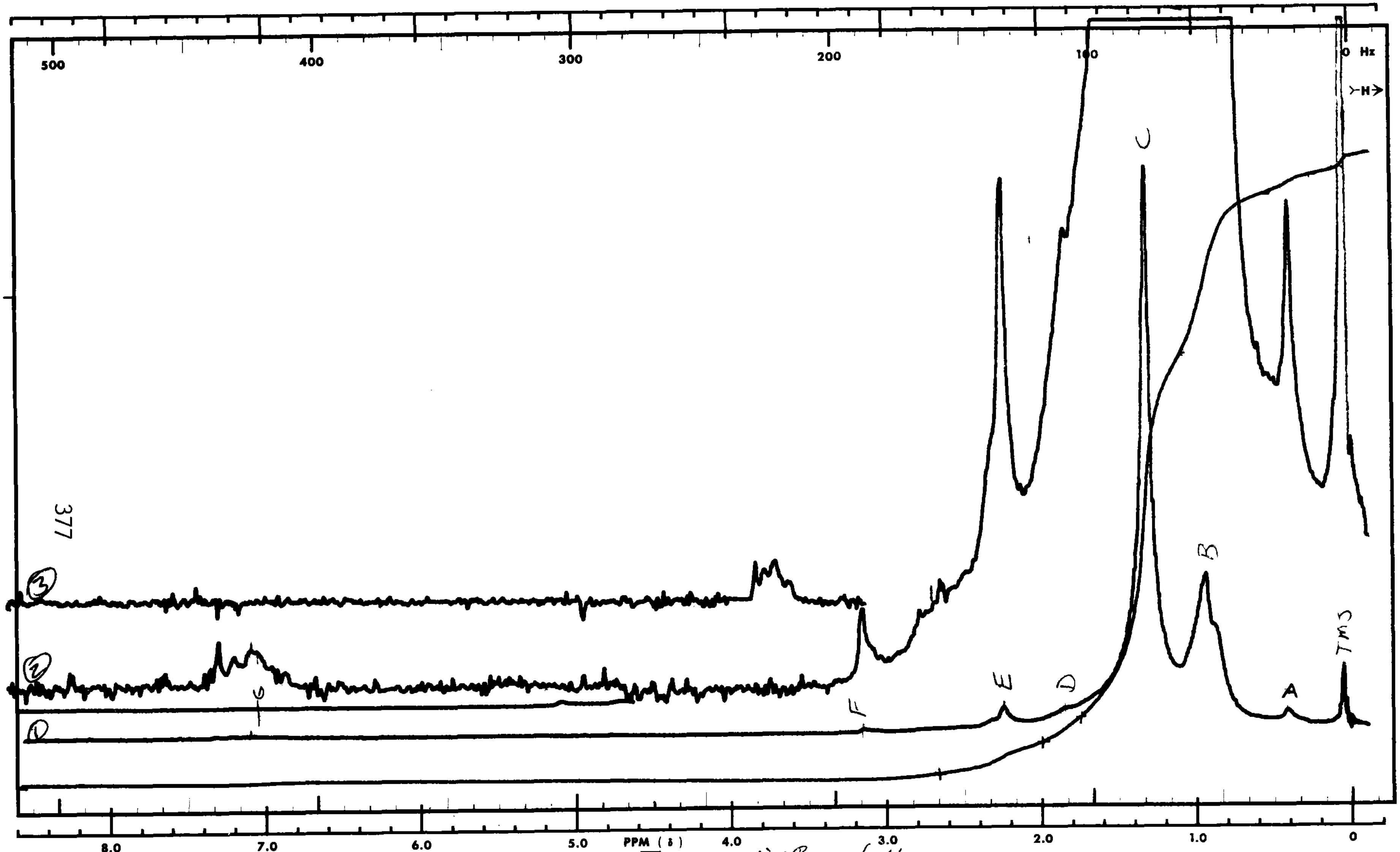
SOLVENT: 50% v/v in (CDCl$_3$ + 2% TMS)

varian
analytical instrument division

DATE: 6/29/78

OPERATOR: M. Barth

60 MHz NMR
SPECTRUM NO.



MANUAL

AUTO ☐

SWEEP OFFSET (Hz): 0

SPECTRUM AMPLITUDE: 1.25(1) 25(2+3)

INTEGRAL AMPLITUDE: 5 at 1.25 amp.

SPINNING RATE (RPS): 50

SWEEP TIME (SEC): 50 ☒250 1000 2,3 (250)

SWEEP WIDTH (Hz): 25 50 100 250 ☒500 (500)

FILTER: ☒ 2 ☒ 4 5 6 7 8 2+3 (2)

RF POWER LEVEL: 0.05 (.05)

SAMPLE: Oil Sample No 29

REMARKS:

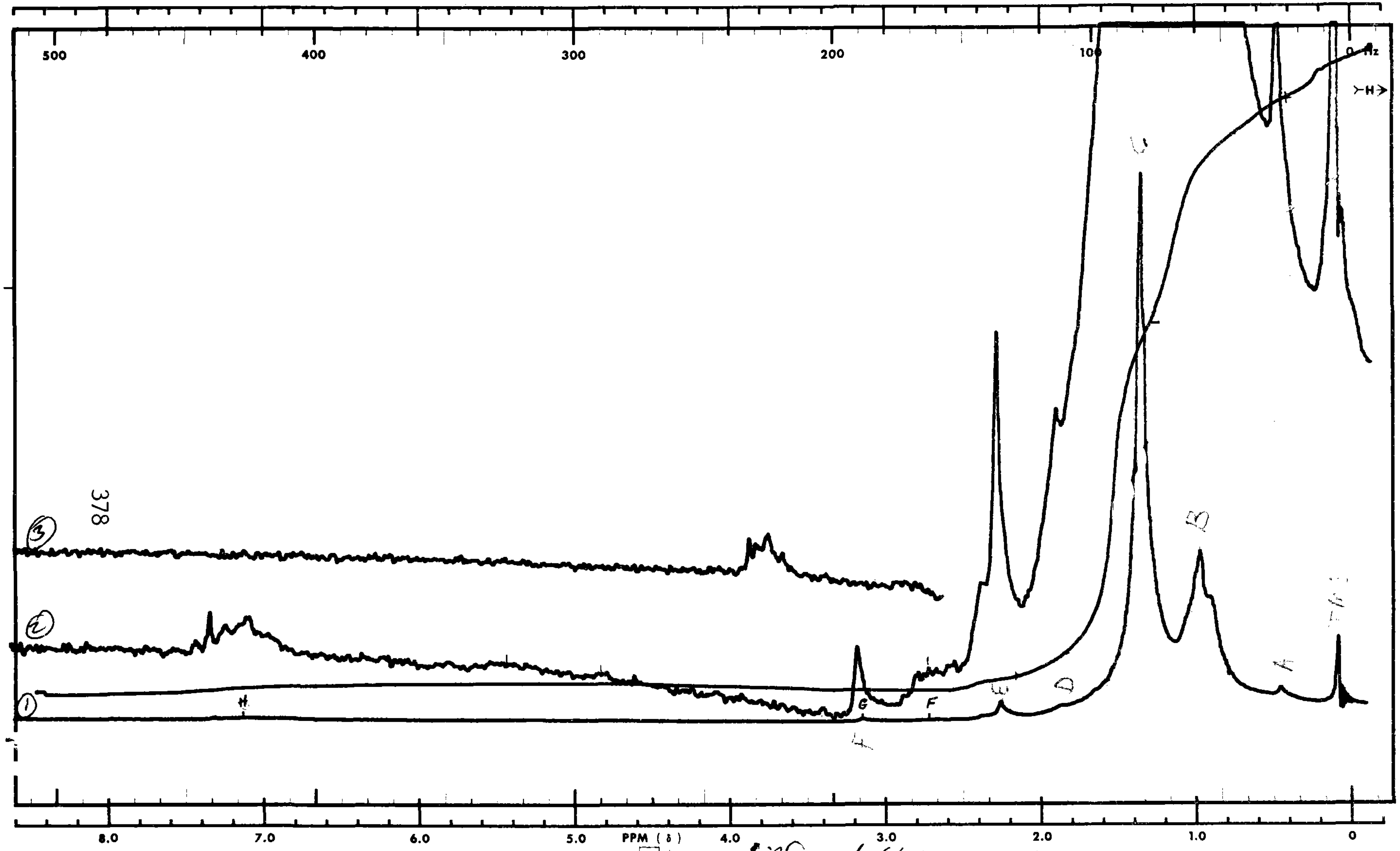
SOLVENT: 50% v/v in (CDCl$_3$ + 2% TMS)

varian
analytical instrument division

DATE: 6/29/78

OPERATOR: M. Barth

60 MHz NMR
SPECTRUM NO.



MANUAL

AUTO ☐

SWEEP OFFSET (Hz): 0

SPECTRUM AMPLITUDE: 1.25 (1) 25.0 (2+3)

INTEGRAL AMPLITUDE: 5 at 1.25 amp

SPINNING RATE (RPS): 50

SWEEP TIME (SEC): 50 ~~250~~ 1000 2+3 (250)

SWEEP WIDTH (Hz): 25 50 100 250 ~~500~~ (500)

FILTER: (1) → ☒ 2 ☒ 4 5 6 7 8 2+3 (2)

RF POWER LEVEL: 0.05 (.05)

SAMPLE: Oil Sample No. 30

REMARKS:

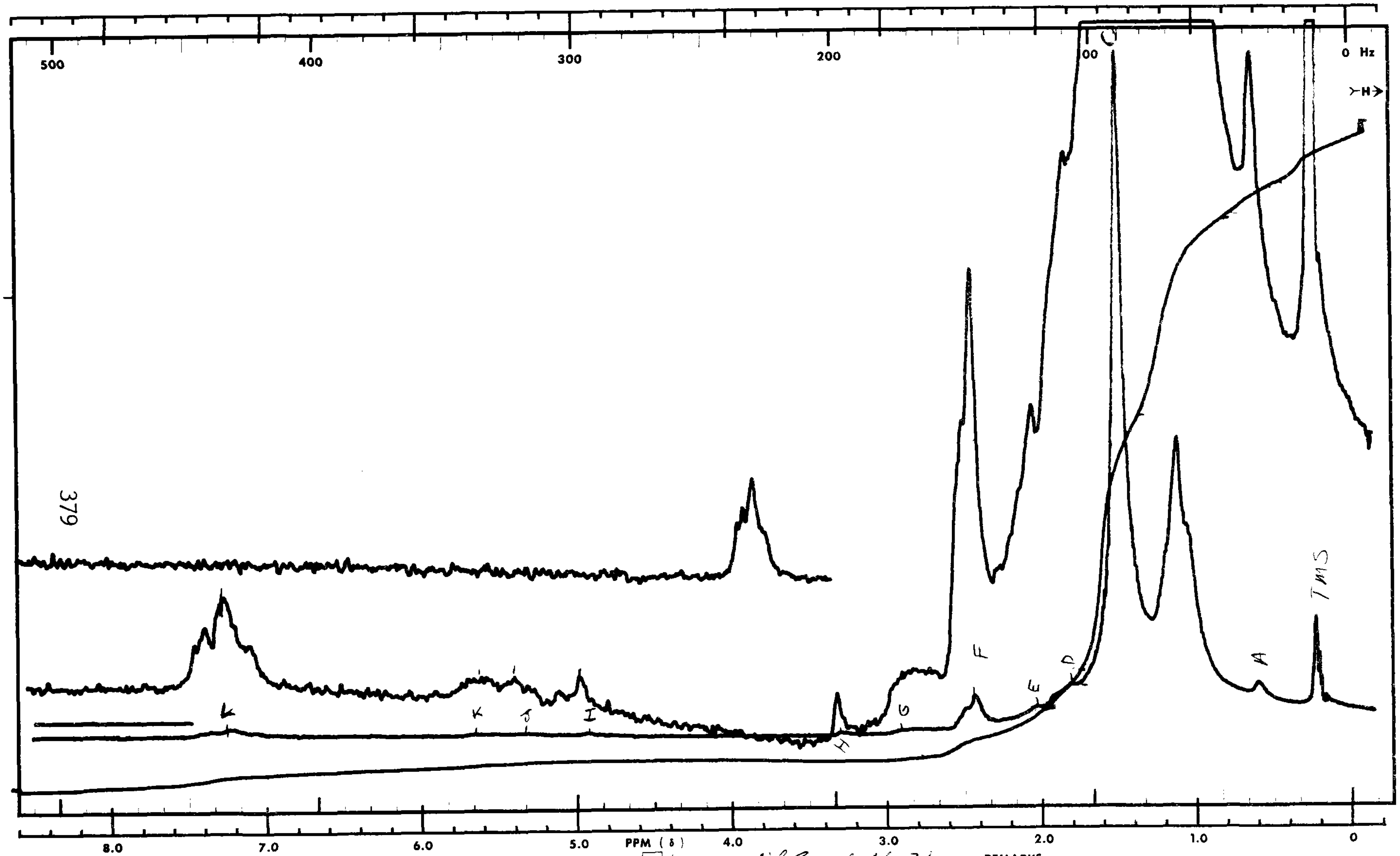
SOLVENT: 50% V/V in (CDCl$_3$ + 2% TMS)

varian
analytical instrument division

DATE: 6/30/78

OPERATOR: M. Barth

60 MHz NMR
SPECTRUM NO.



MANUAL

AUTO ☐

SWEEP OFFSET (Hz): 0

SPECTRUM AMPLITUDE: 2.5(1), 25(2+3)

INTEGRAL AMPLITUDE: 5.0 at 1.25

SPINNING RATE (RPS):

SWEEP TIME (SEC): 50 ☒250 1000 #3 (250)

SWEEP WIDTH (Hz): 25 50 100 250 ☒500 (500)

FILTER: 1 → ☒1 2 ☒3 4 5 6 7 8 2+3 (2)

RF POWER LEVEL: 0.05 (.05)

SAMPLE: Oil Sample No 31

REMARKS:

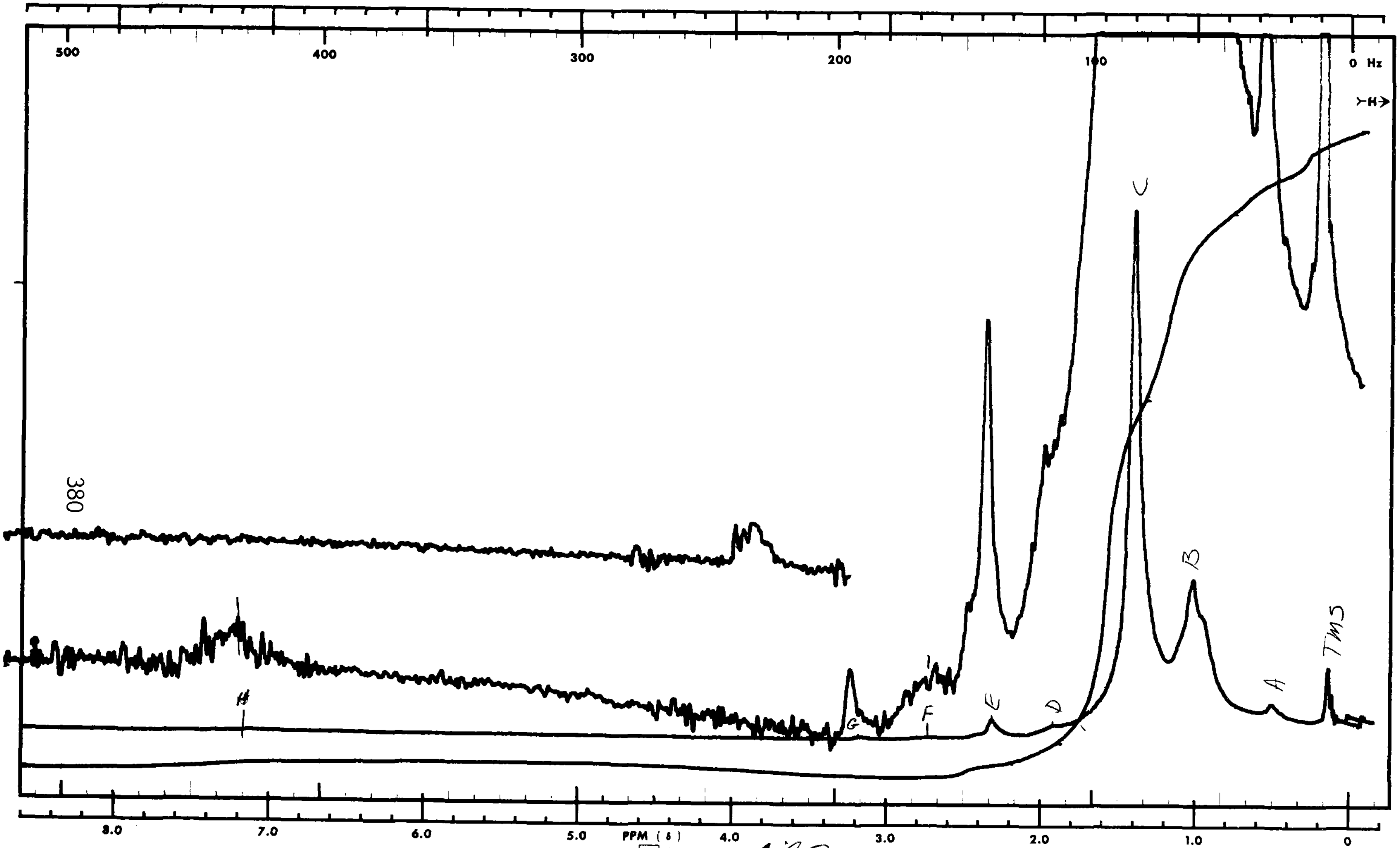
SOLVENT: 50% v/v in ($CDCl_3$ + 2% TMS)

varian
analytical instrument division

DATE: 6/30/78

OPERATOR: M. Barth

60 MHz NMR
SPECTRUM NO.



MANUAL

SWEEP OFFSET (Hz): 0

SPECTRUM AMPLITUDE: 1.25 (1) 25.0 (2+3)

INTEGRAL AMPLITUDE: 5.0 at 1.25 amp

SPINNING RATE (RPS): 49

SWEEP TIME (SEC): 50 [✓ 250] (#3) 1000

SWEEP WIDTH (Hz): 25 50 100 250 [✓ 500]

FILTER: 1 — [✓ 1] 2 3 4 5 6 7 8 (2+3)

RF POWER LEVEL: .05

AUTO ☐

(250)

(500)

(2)

(.05)

SAMPLE: Oil Sample No. 32

REMARKS:

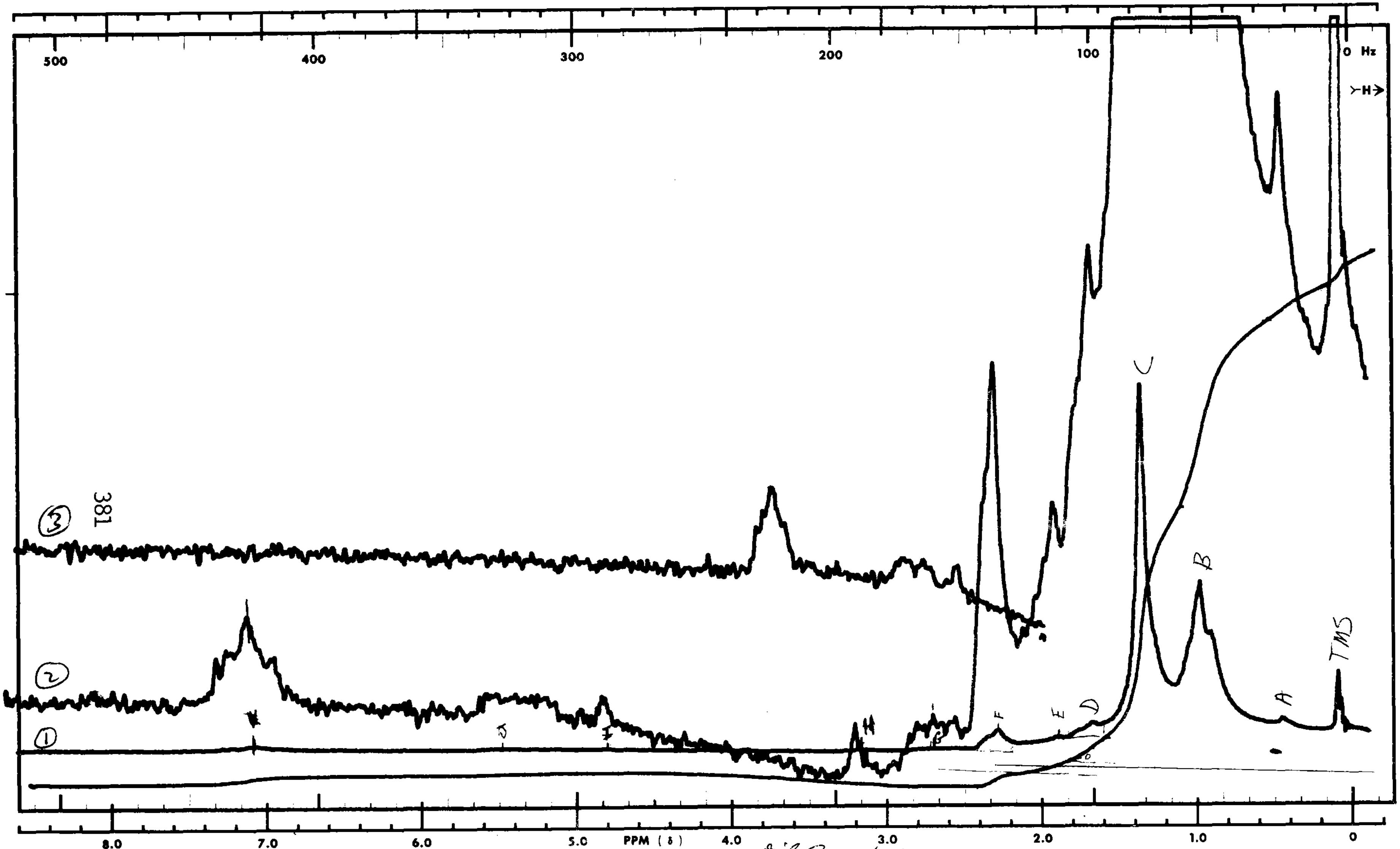
SOLVENT: 50% v/v in (CDCl$_3$ + 2% TMS)

varian
analytical instrument division

DATE: 6-30-78

OPERATOR: M. Barth

60 MHz NMR
SPECTRUM NO.



MANUAL

AUTO ☐

SWEEP OFFSET (Hz): 0

SPECTRUM AMPLITUDE: 1.25(1) 25(2+3)

INTEGRAL AMPLITUDE: 5.0 at 1.25 r

SPINNING RATE (RPS): 51

SWEEP TIME (SEC): 50 [✓ 250] 1000 for 3 (250)

SWEEP WIDTH (Hz): 25 50 100 250 [✓ 500] (500)

FILTER: [✓ 1] 2 [✓ 3] 4 5 6 7 8 2+3 (2)

RF POWER LEVEL .05 (.05)

SAMPLE: Oil Sample No. 33

REMARKS:

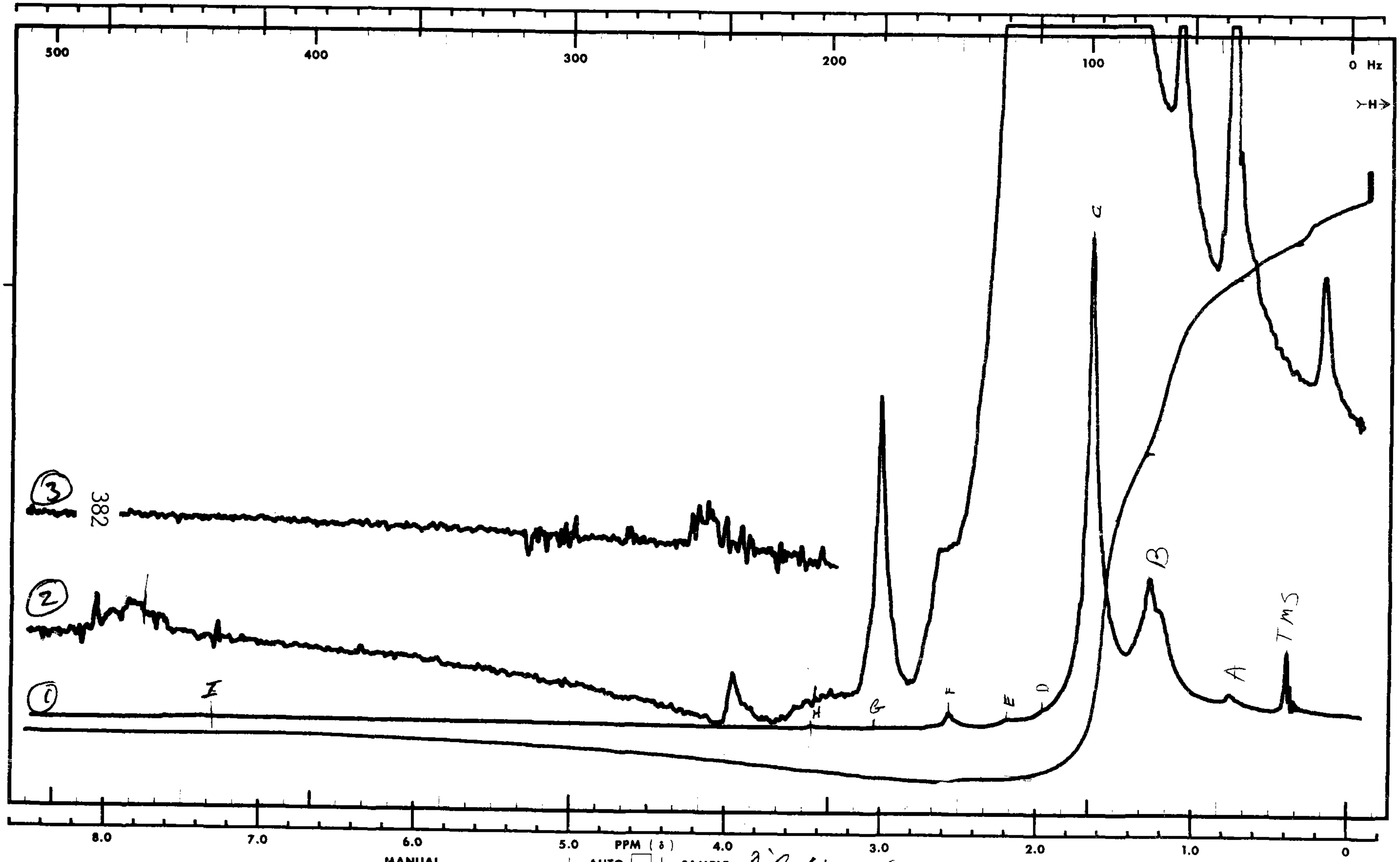
SOLVENT: 50% v/v in (CDCl$_3$ + 2% TMS

varian
analytical instrument division

DATE: 7/5/78

OPERATOR: M. Barth

60 MHz NMR
SPECTRUM NO.



MANUAL

AUTO ☐

SWEEP OFFSET (Hz): 0

SPECTRUM AMPLITUDE: 1.25(1) 25(2+3)

INTEGRAL AMPLITUDE: 5.0 at 1.25

SPINNING RATE (RPS): 49-51 Varying

SWEEP TIME (SEC): 50 [✓ 250] 1000 for #3 (250)

SWEEP WIDTH (Hz): 25 50 100 250 [✓ 500] (500)

FILTER: [✓ 1] 2 [✓ 3] 4 5 6 7 8 2+3 (2)

RF POWER LEVEL: .05 (.05)

SAMPLE: Oil No. 34

REMARKS:

SOLVENT: 50% V/V in (CDCl$_3$ + 2% TMS)

varian
analytical instrument division

DATE: 7/5/76

OPERATOR: M. Barth

60 MHz NMR
SPECTRUM NO.

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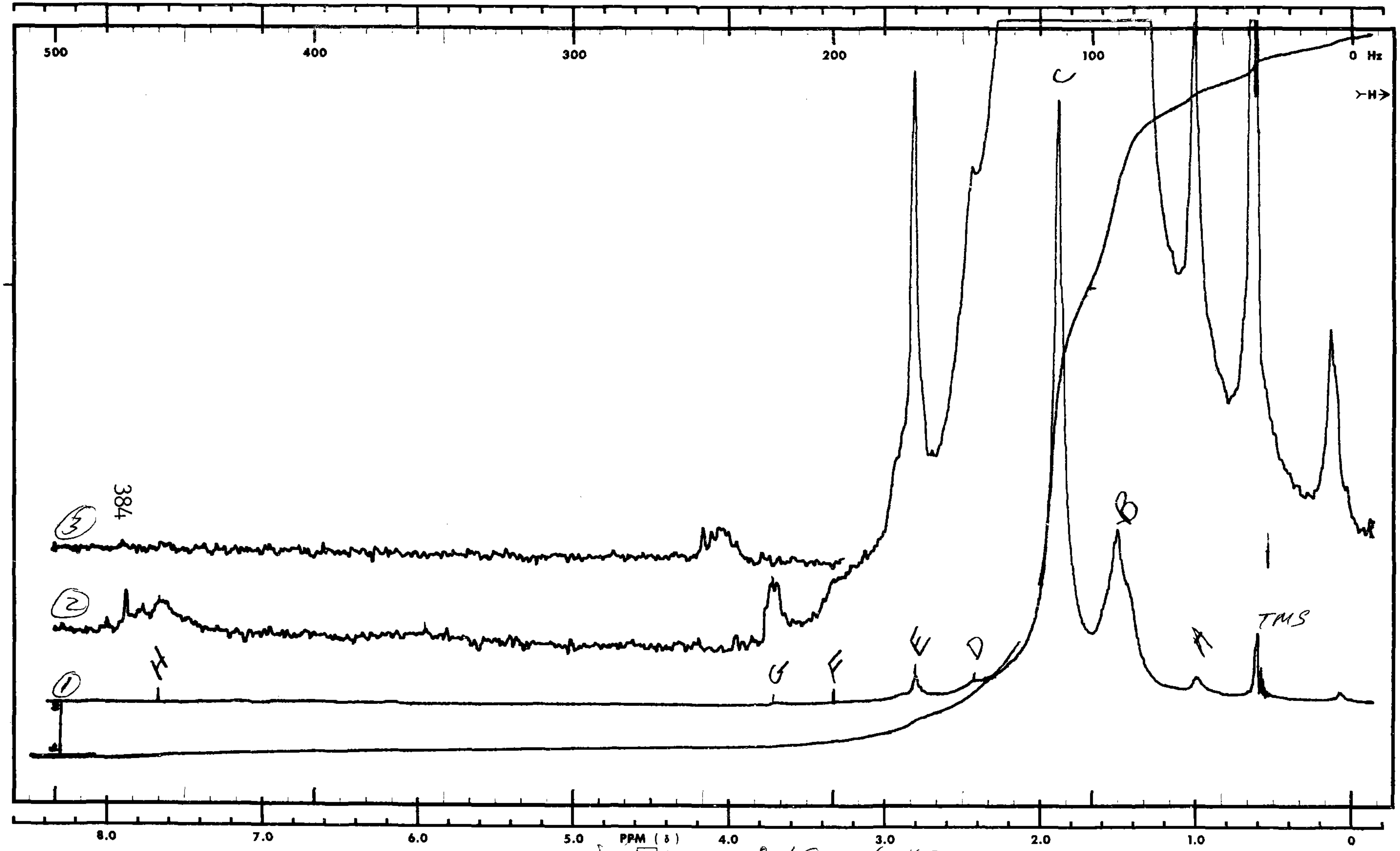


MANUAL

SWEEP OFFSET (Hz): 0

SPECTRUM AMPLITUDE: 1.25(1) + 25(2+3)

INTEGRAL AMPLITUDE: 5 at 1.25 spec. amp.

SPINNING RATE (RPS): 49

SWEEP TIME (SEC): 50 ☒250 1000(3)

SWEEP WIDTH (Hz): 25 50 100 250 ☒500

FILTER: ☒1 2 ☒3 4 5 6 7 8 (2+3)

RF POWER LEVEL: 0.05

AUTO ☐ (250) (500) (2) (.05)

SAMPLE: Oil Sample #35

REMARKS:

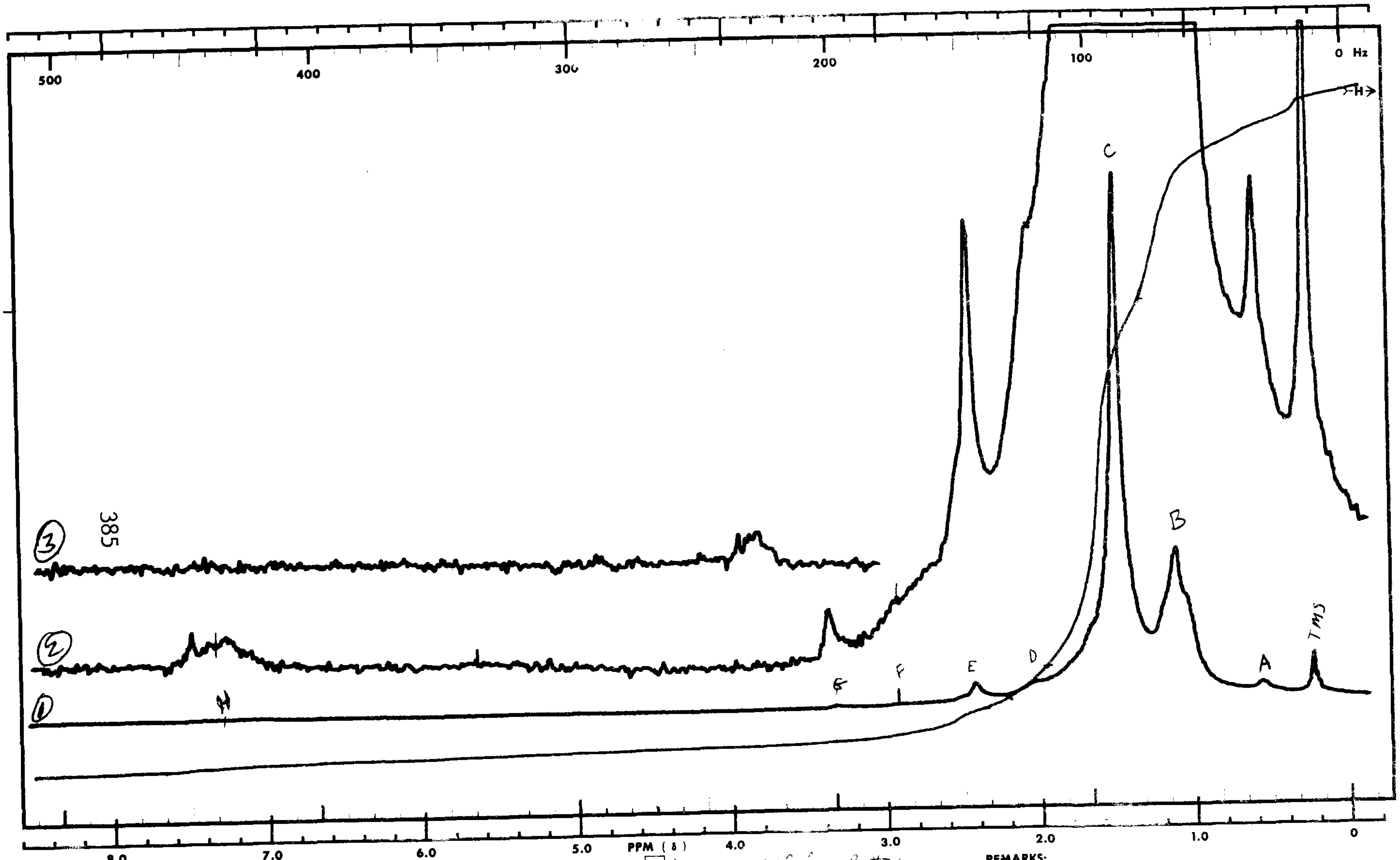
SOLVENT: 50% v/v in ($CDCl_3$ + 2% TMS)

varian analytical instrument division

DATE: 7/19/78

OPERATOR: M. Bauth

60 MHz NMR SPECTRUM NO.



MANUAL

AUTO ☐

SWEEP OFFSET (Hz): 0

SPECTRUM AMPLITUDE: 1.25(1)+25(2+3)

INTEGRAL AMPLITUDE: 5 at 1.25 spec. ampl.

SPINNING RATE (RPS): 51

SWEEP TIME (SEC): 50 ☒250 1000(3) (250)

SWEEP WIDTH (Hz): 25 50 100 250 ☒500 (500)

FILTER: ☒1 2 ☒3 (2+3) 4 5 6 7 8 (2)

RF POWER LEVEL: 0.05 (.05)

SAMPLE: Oil sample #36

REMARKS:

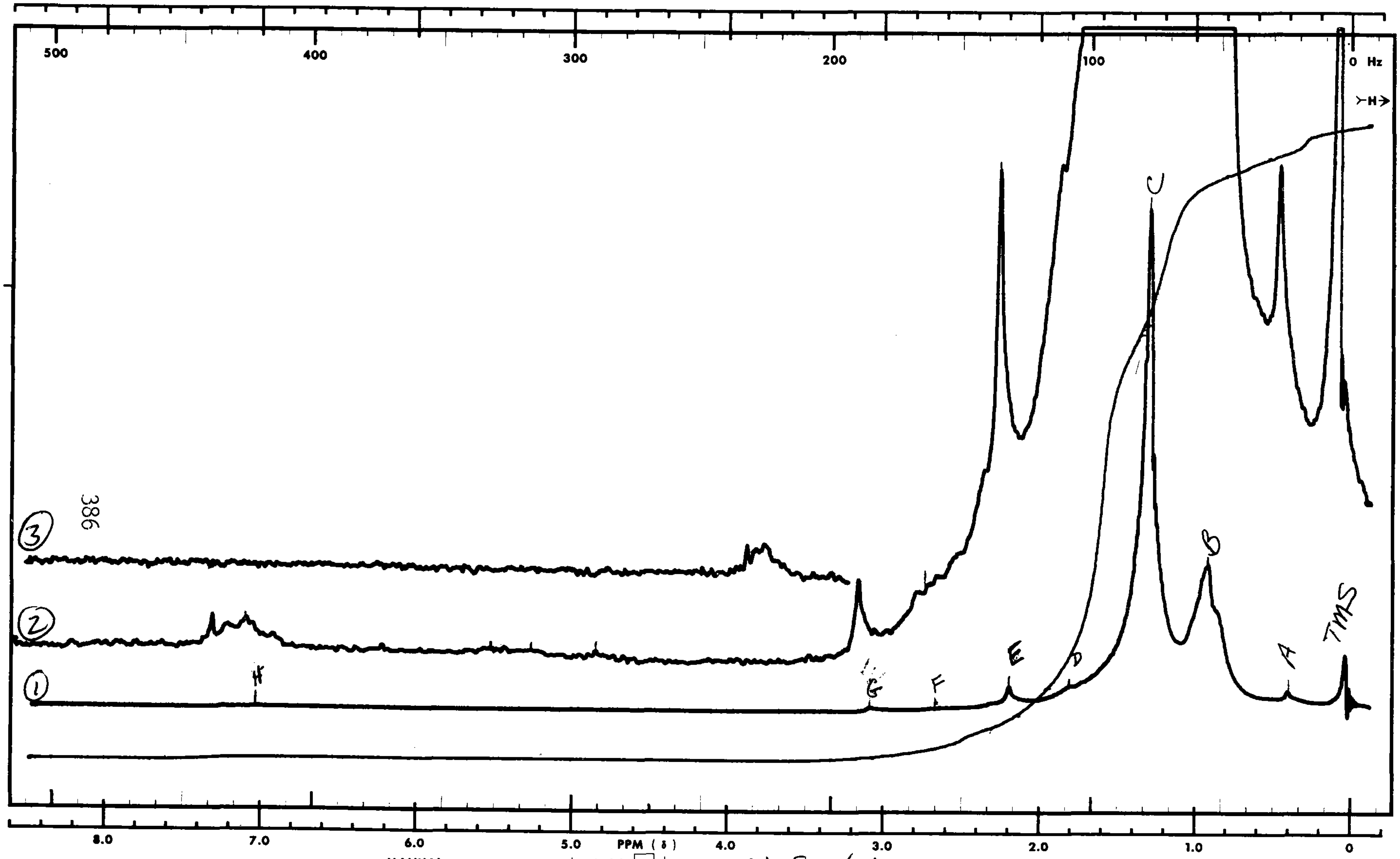
SOLVENT: 50% V/V in (CDCl3 + 2% TMS)

varian
analytical instrument division

DATE: 7/19/78

OPERATOR: B. Dowell

60 MHz NMR
SPECTRUM NO.



MANUAL

SWEEP OFFSET (Hz): 0

SPECTRUM AMPLITUDE: 1.25(1) + 25(2+3)

INTEGRAL AMPLITUDE: 5.0 at 1.25 spec amp.

SPINNING RATE (RPS): 50

SWEEP TIME (SEC): 50 ~~250~~ 1000(3)

SWEEP WIDTH (Hz): 25 50 100 250 ~~500~~

FILTER: ~~1~~ 2 ~~3~~ 4 5 6 7 8 2+3

RF POWER LEVEL: 0.05

AUTO ☐

(250)

(500)

(2)

(.05)

SAMPLE: Oil Sample No. 37

REMARKS:

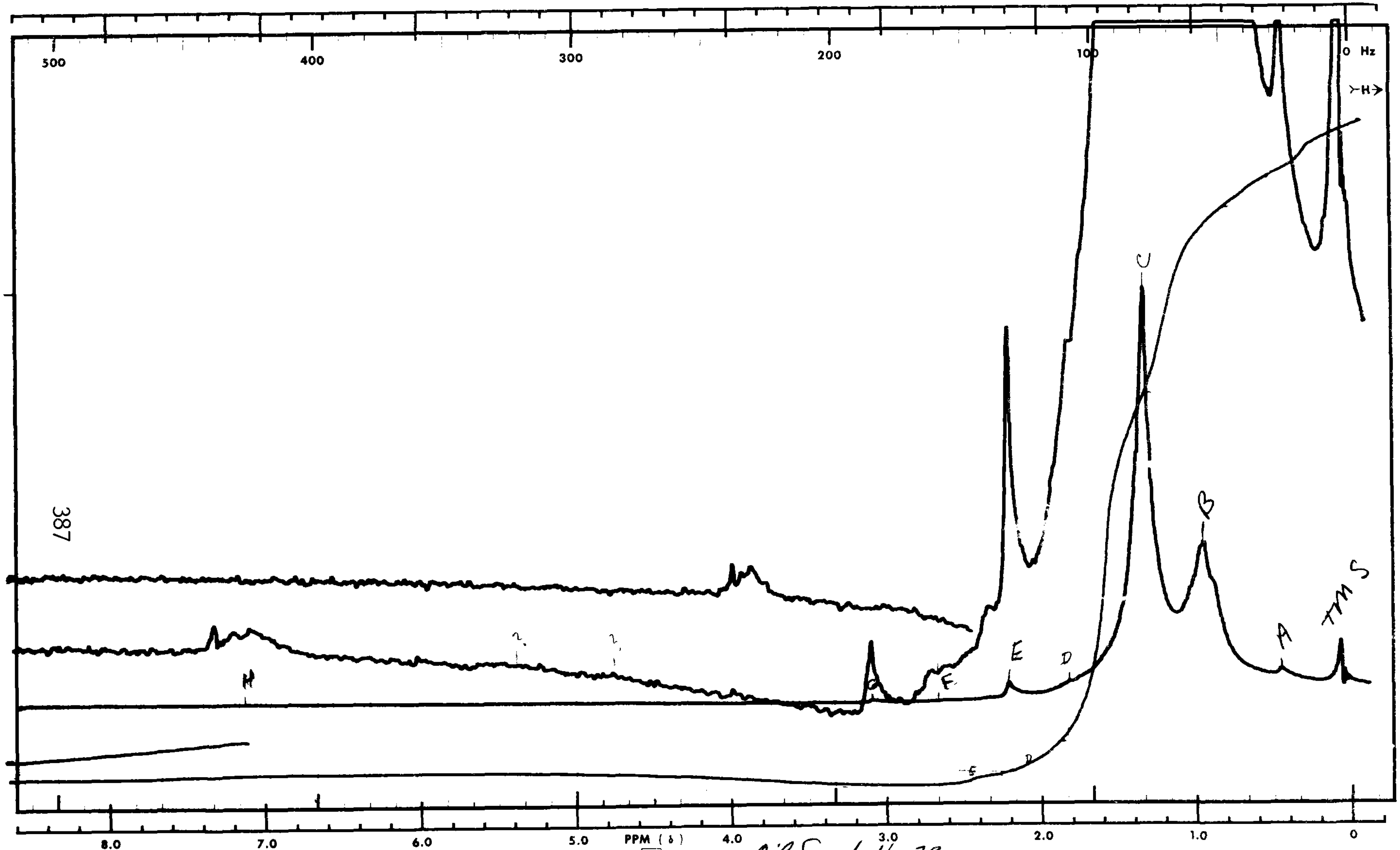
SOLVENT: 50% v/v in (CDCl$_3$ + 2% TMS)

varian
analytical instrument division

DATE: 7/25/78

OPERATOR: M. Barth

60 MHz NMR
SPECTRUM NO.



MANUAL

AUTO ☐

SWEEP OFFSET (Hz): 0
SPECTRUM AMPLITUDE: 1.25(1) 25(2+3)
INTEGRAL AMPLITUDE: 5.0 1
SPINNING RATE (RPS): 50 [illegible]

SWEEP TIME (SEC): 50 ☒ 1000(3) (250)
SWEEP WIDTH (Hz): 25 50 100 250 ☒ (500)
FILTER: 1 2 ☒ 4 5 6 7 8 (2)
RF POWER LEVEL: .05 (2+3) (.05)

SAMPLE: Oil Sample No. 38

REMARKS:

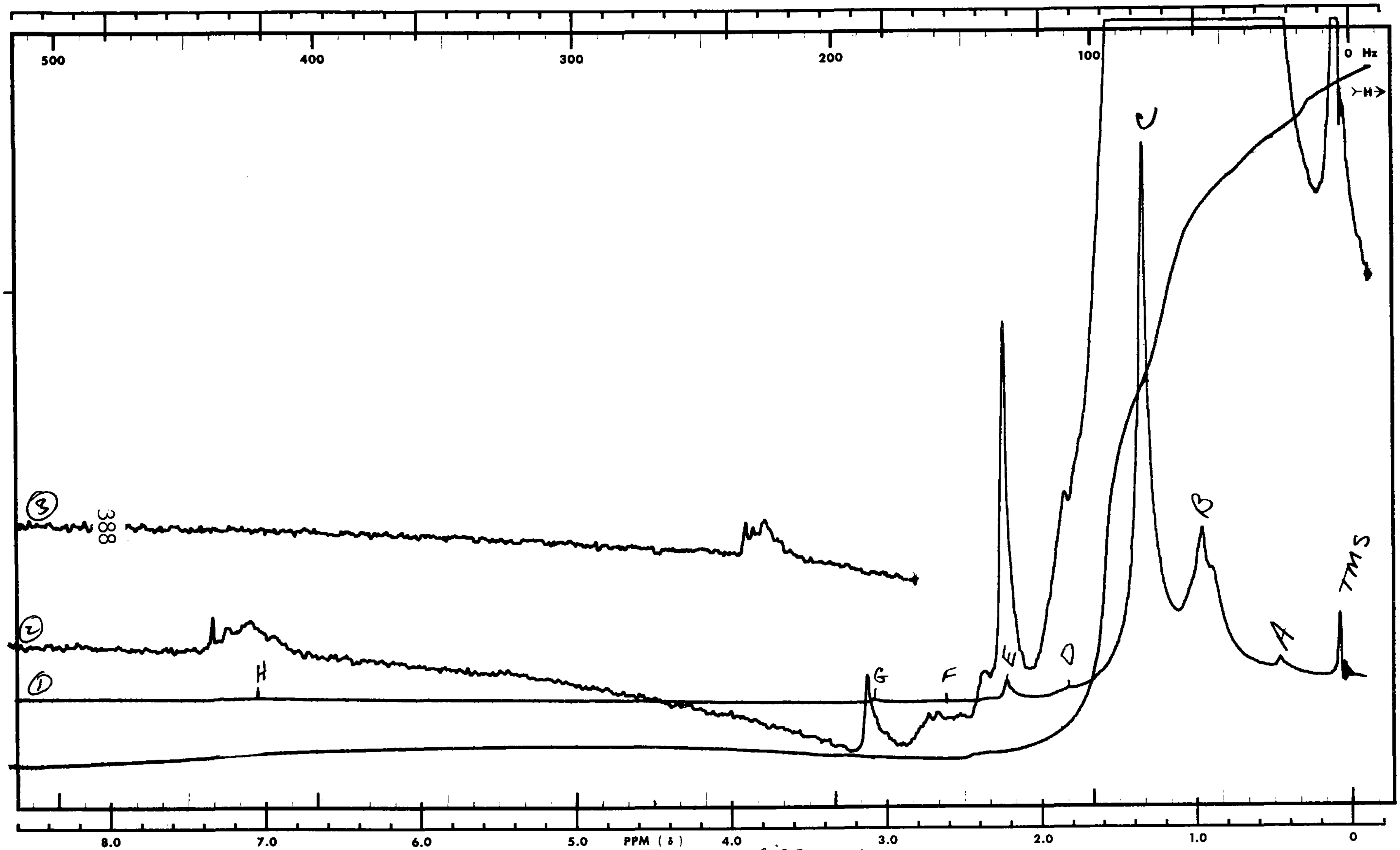
SOLVENT: 50% v/v in (CDCl$_3$ +2% TMS)

varian
analytical instrument division

DATE:

OPERATOR: M. Barth

60 MHz NMR
SPECTRUM NO



MANUAL | AUTO ☐

SWEEP OFFSET (Hz): 0

SPECTRUM AMPLITUDE: 1.25(1) 25(2+3)

INTEGRAL AMPLITUDE: 5.0 at 1.25

SPINNING RATE (RPS): 49

SWEEP TIME (SEC): 50 [✓ 250] 1000(3) (250)

SWEEP WIDTH (Hz): 25 50 100 250 [✓ 500] (500)

FILTER: [✓ 1] 2 [✓ 3] 4 5 6 7 8 (2)

RF POWER LEVEL: .05 (2+3) (.05)

SAMPLE: Oil Sample #39

REMARKS:

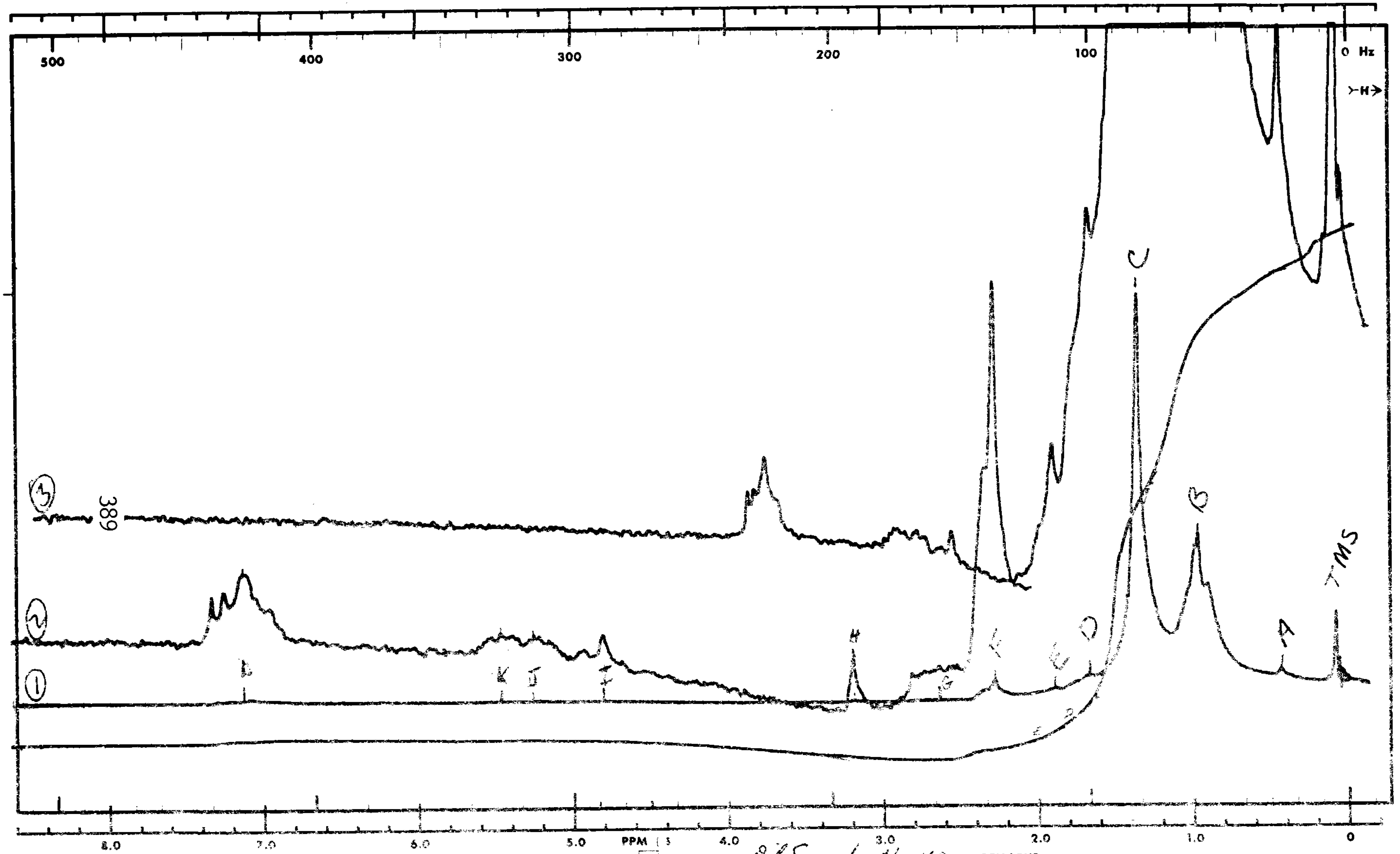
SOLVENT: 50% v/v in (CDCl$_3$ + 2% TMS)

varian
analytical instrument division

DATE: 7/26/78

OPERATOR: M. Barth

60 MHz NMR
SPECTRUM NO.



MANUAL

AUTO ☐

SWEEP OFFSET (Hz): 0

SPECTRUM AMPLITUDE: 1.25(1) + 25(2+3)

INTEGRAL AMPLITUDE: 5.0 at 1.25 spec amp

SPINNING RATE (RPS): 50

SWEEP TIME (SEC): 50 ~~250~~ 1000(3) (250)

SWEEP WIDTH (Hz): 25 50 100 250 ~~500~~ (500)

FILTER: ☒ 2 ☒ → (2+3) (2)

RF POWER LEVEL: .05 (.05)

SAMPLE: Oil Sample No. 40

REMARKS:

SOLVENT: 50% v/v in (CDCl$_3$ + 2% TMS)

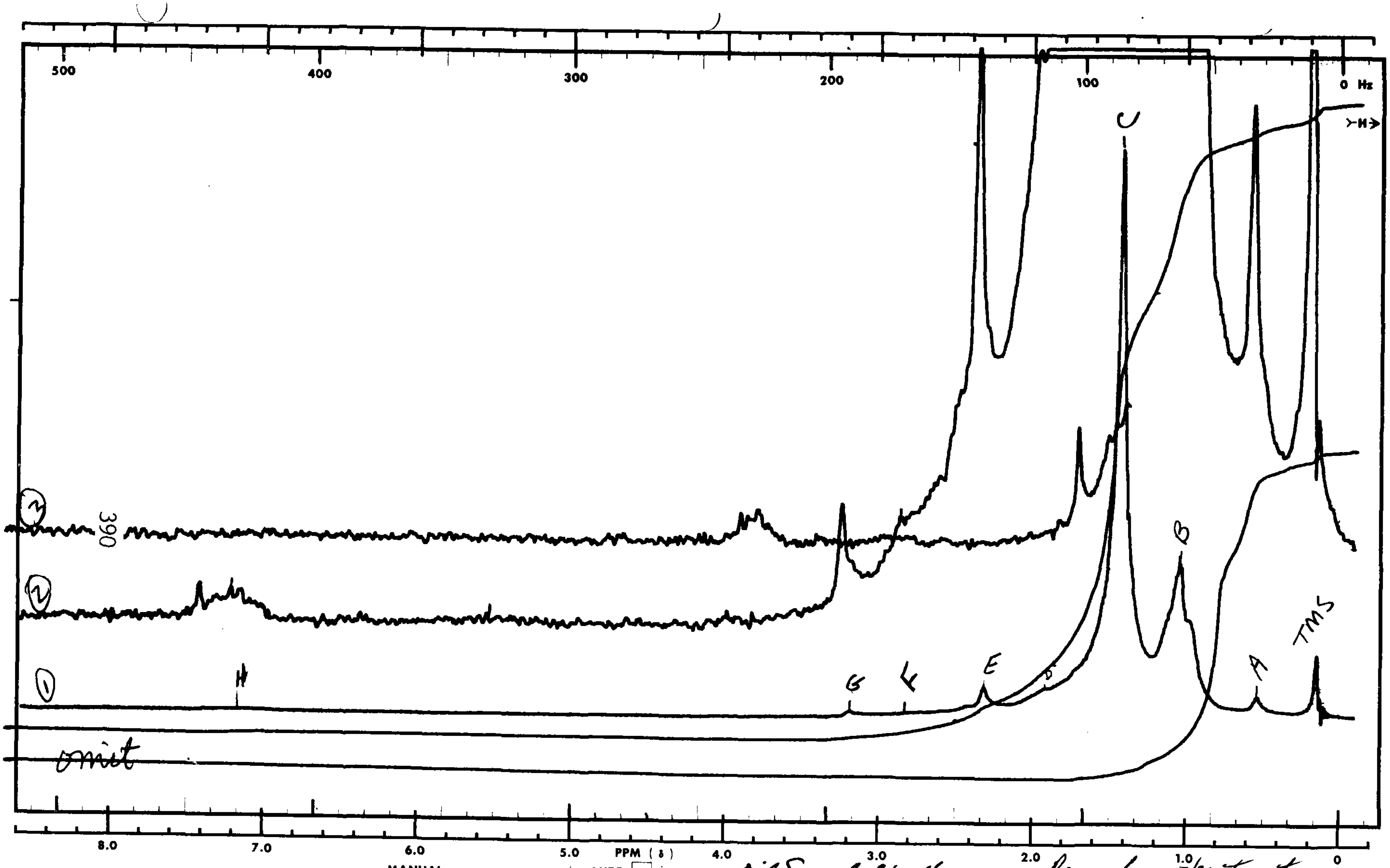
DATE: 7/27/28

OPERATOR: M. Barth

60 MHz NMR

SPECTRUM NO.

varian
analytical instrument division



MANUAL

AUTO ☐

SWEEP OFFSET (Hz): 0

SPECTRUM AMPLITUDE: 1.25(1) + 25(2+3)

INTEGRAL AMPLITUDE: 5.0 at 1.25 spec amp

SPINNING RATE (RPS): 50

SWEEP TIME (SEC): 50 ~~250~~ 100 (3) (250)

SWEEP WIDTH (Hz): 25 50 100 250 ~~500~~ (500)

FILTER: ~~1~~ 2 ~~3~~ 4 5 6 7 8 (2)

RF POWER LEVEL: 0.05 (2+3) (.05)

SAMPLE: Oil Sample No. 41

REMARKS: Rerun from 7/31 to get better spectrum.

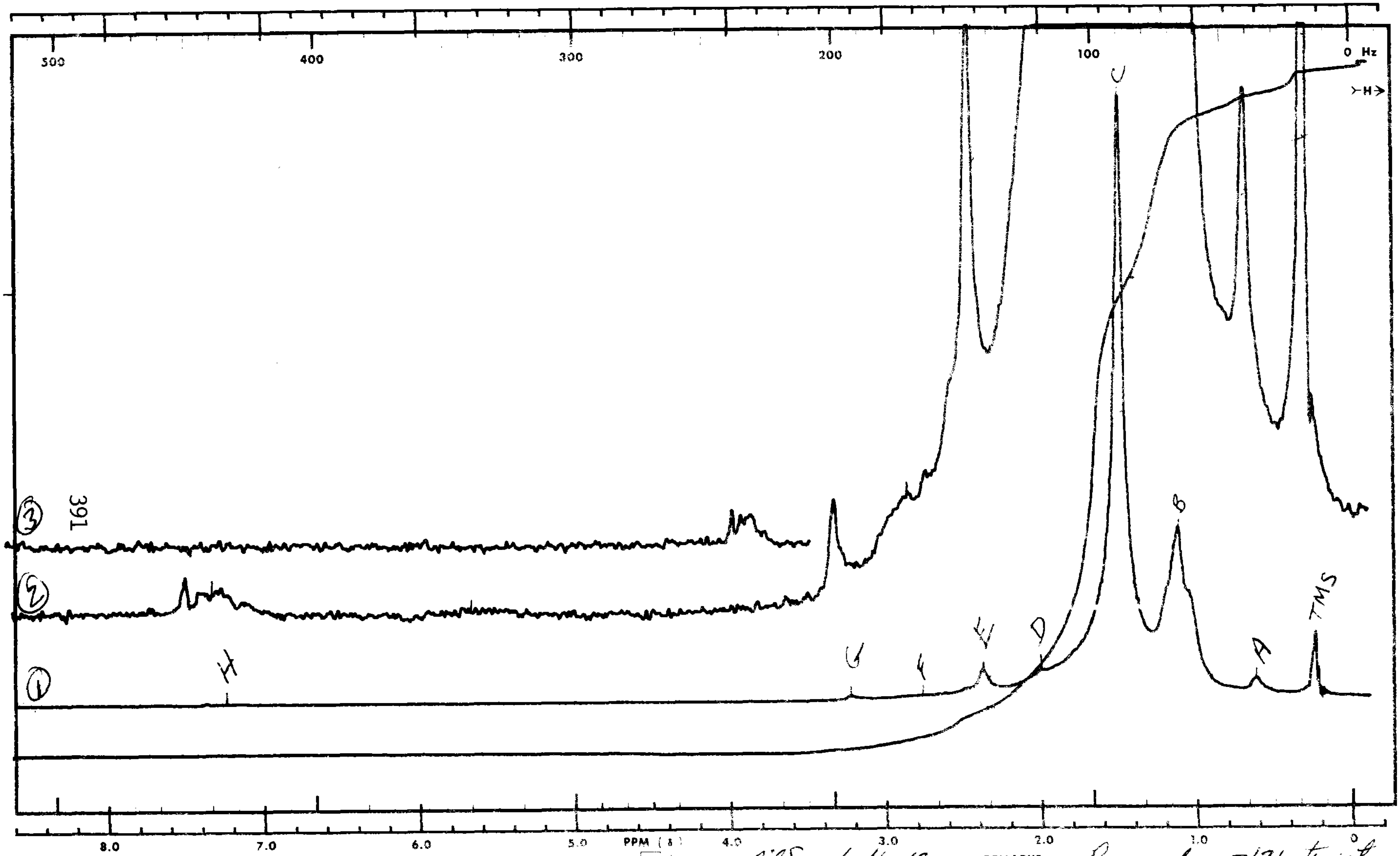
SOLVENT: 50% v/v in ($CDCl_3$ + 2% TMS)

varian
analytical instrument division

DATE: 8/9/78

OPERATOR: M. Barth

60 MHz NMR
SPECTRUM NO.



MANUAL AUTO ☐

SAMPLE: Oil Sample No. 42

REMARKS: Rerun from 7/31 to get better spectrum

SWEEP OFFSET (Hz): 0

SPECTRUM AMPLITUDE: 1.25 (1) & 25 (2+3)

INTEGRAL AMPLITUDE: 5.0 at 1.25 spec amp.

SPINNING RATE (RPS): 50

SWEEP TIME (SEC): 50 ~~250~~ 1000 (3) (250)

SWEEP WIDTH (Hz): 25 50 100 250 ☒ 500 (500)

FILTER: ☒ 1 2 ☒ 3 4 5 6 7 8 (2)

RF POWER LEVEL: 0.05 (2+3) (.05)

SOLVENT: 50% v/v in (CDCl₃ + 2% TMS)

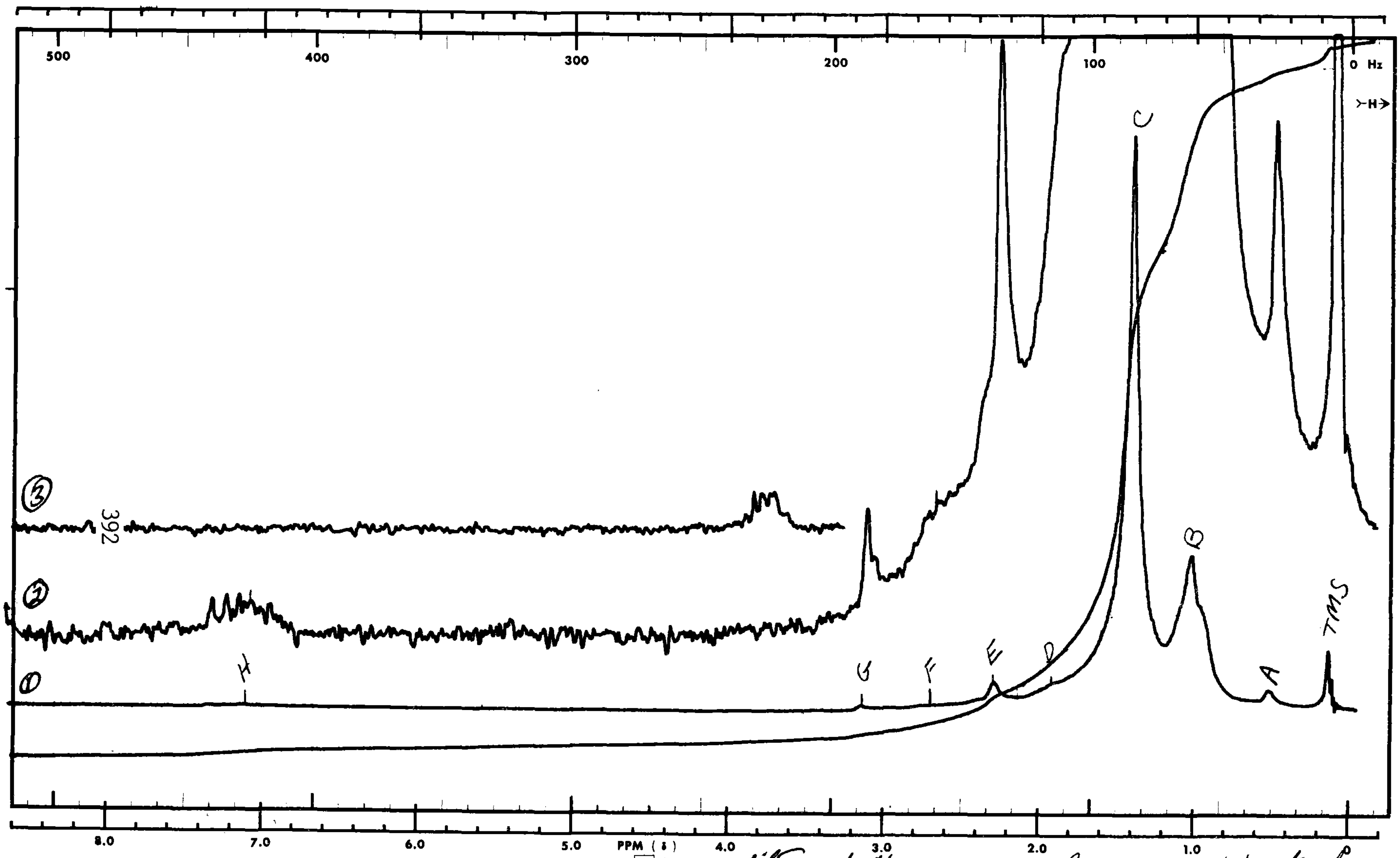
varian analytical instrument division

DATE: 8/2/78

OPERATOR: Baxter

60 MHz NMR

SPECTRUM NO.



PPM (δ)

MANUAL

SWEEP OFFSET (Hz): 0
SPECTRUM AMPLITUDE: 1.25(1) + 25(2+3)
INTEGRAL AMPLITUDE: 5.0 at 1.25 spec. ampl.
SPINNING RATE (RPS): 50

SWEEP TIME (SEC): 50 ☒250 1000(3)
SWEEP WIDTH (Hz): 25 50 100 250 ☒500
FILTER: ☒1 2 ☒3 4 5 6 7 8
RF POWER LEVEL: 0.05 (2+3)

AUTO ☐
(250)
(500)
(2)
(.05)

SAMPLE: Oil Sample No. 43

REMARKS: Rerun from 7/31/78 to get better spectrum.

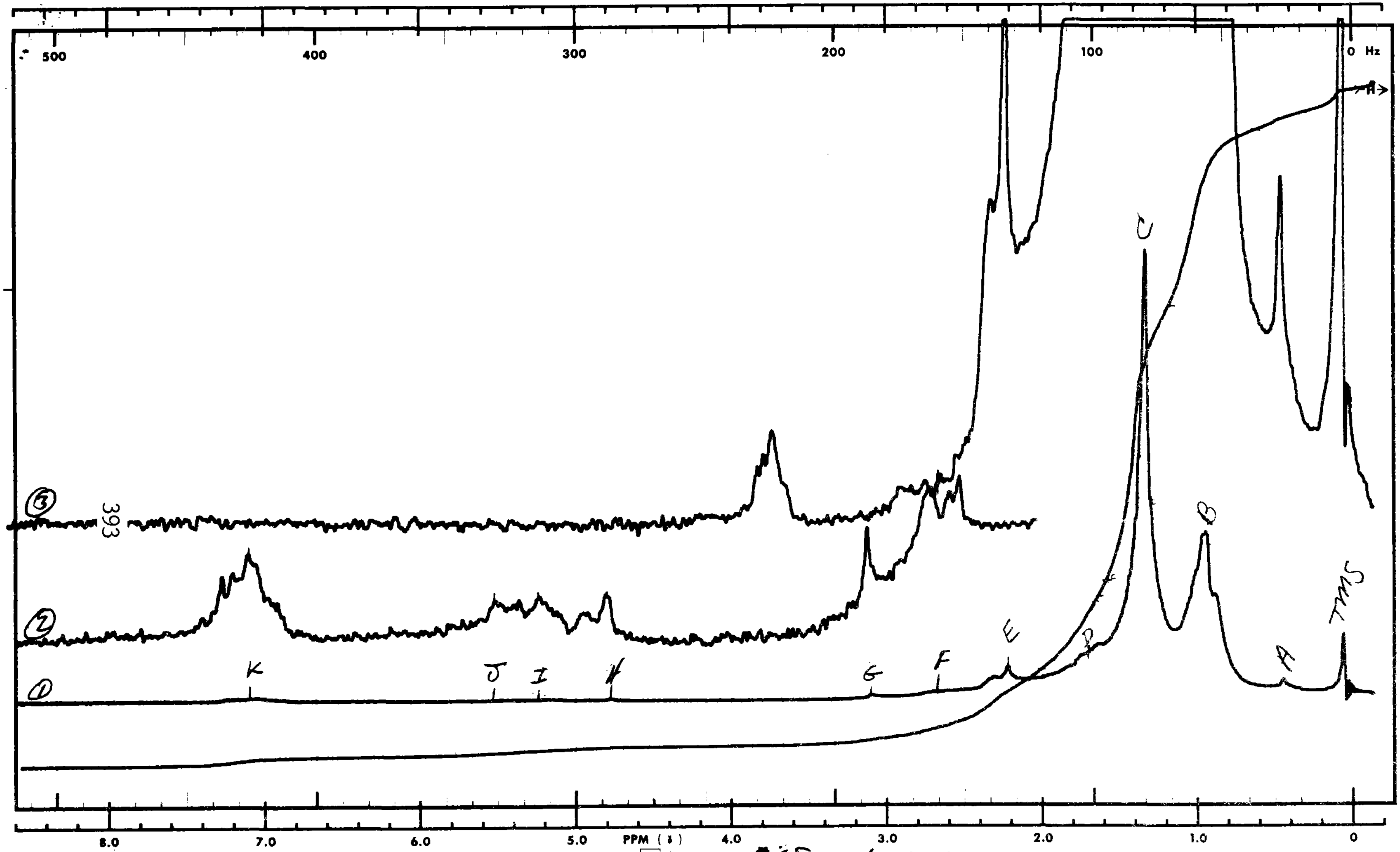
SOLVENT: 50% v/v in (CDCl$_3$ + 2% TMS)

varian
analytical instrument division

DATE: 8/9/78

OPERATOR: M. Barth

60 MHz NMR
SPECTRUM NO.



MANUAL

AUTO ☐

SWEEP OFFSET (Hz): 0

SPECTRUM AMPLITUDE: 12.5(1) + 25(2+3)

INTEGRAL AMPLITUDE: 5.0 at 1.25 spec. amp.

SPINNING RATE (RPS): 50

SWEEP TIME (SEC): 50 ☒250 1000(3) (250)

SWEEP WIDTH (Hz): 25 50 100 250 ☒500 (500)

FILTER: ☒1 2 ☒3 4 5 6 7 8 (2+3) (2)

RF POWER LEVEL: 0.05 (.05)

SAMPLE: Oil Sample #44

REMARKS:

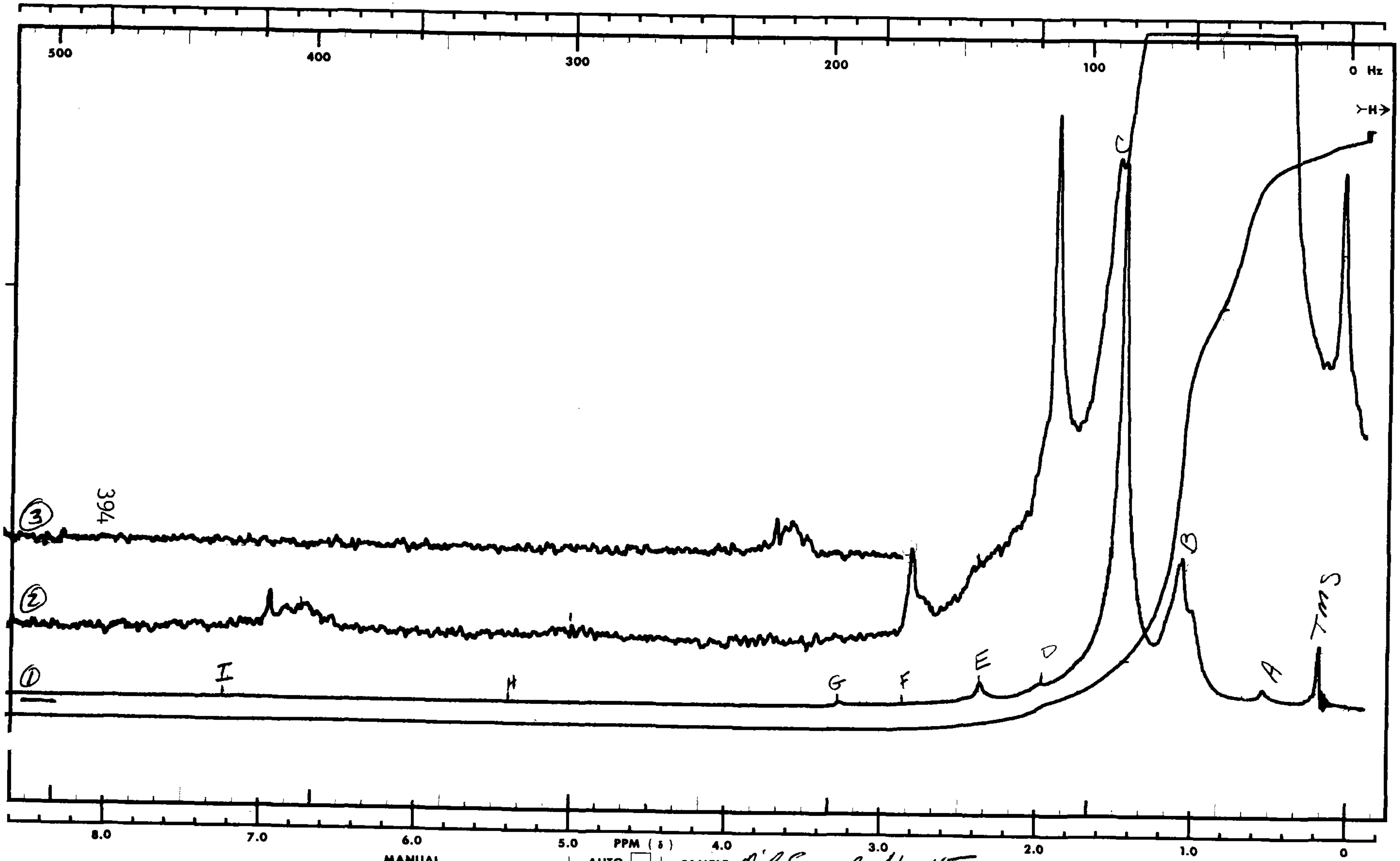
SOLVENT: 50% v/v in (CDCl$_3$ + 2% TMS)

varian
analytical instrument division

DATE: 6/7/78

OPERATOR: M. Barth

60 MHz NMR
SPECTRUM NO.



MANUAL

SWEEP OFFSET (Hz): 0

SPECTRUM AMPLITUDE: 1.25(1) + 25(2+3)

INTEGRAL AMPLITUDE: 5.0 at 1.25 spec amp.

SPINNING RATE (RPS): 50

SWEEP TIME (SEC): 50 [✓ 200] 1000(3)

SWEEP WIDTH (Hz): 25 50 100 250 [✓ 500]

FILTER: [✓ 1] 2 [✓ 3] 4 5 6 7 8 2+3

RF POWER LEVEL: 0.05

AUTO ☐

(250)

(500)

(2)

(.05)

SAMPLE: Oil Sample No. 45

REMARKS:

SOLVENT: 50% v/v in ($CDCl_3$ + 2% TMS)

varian

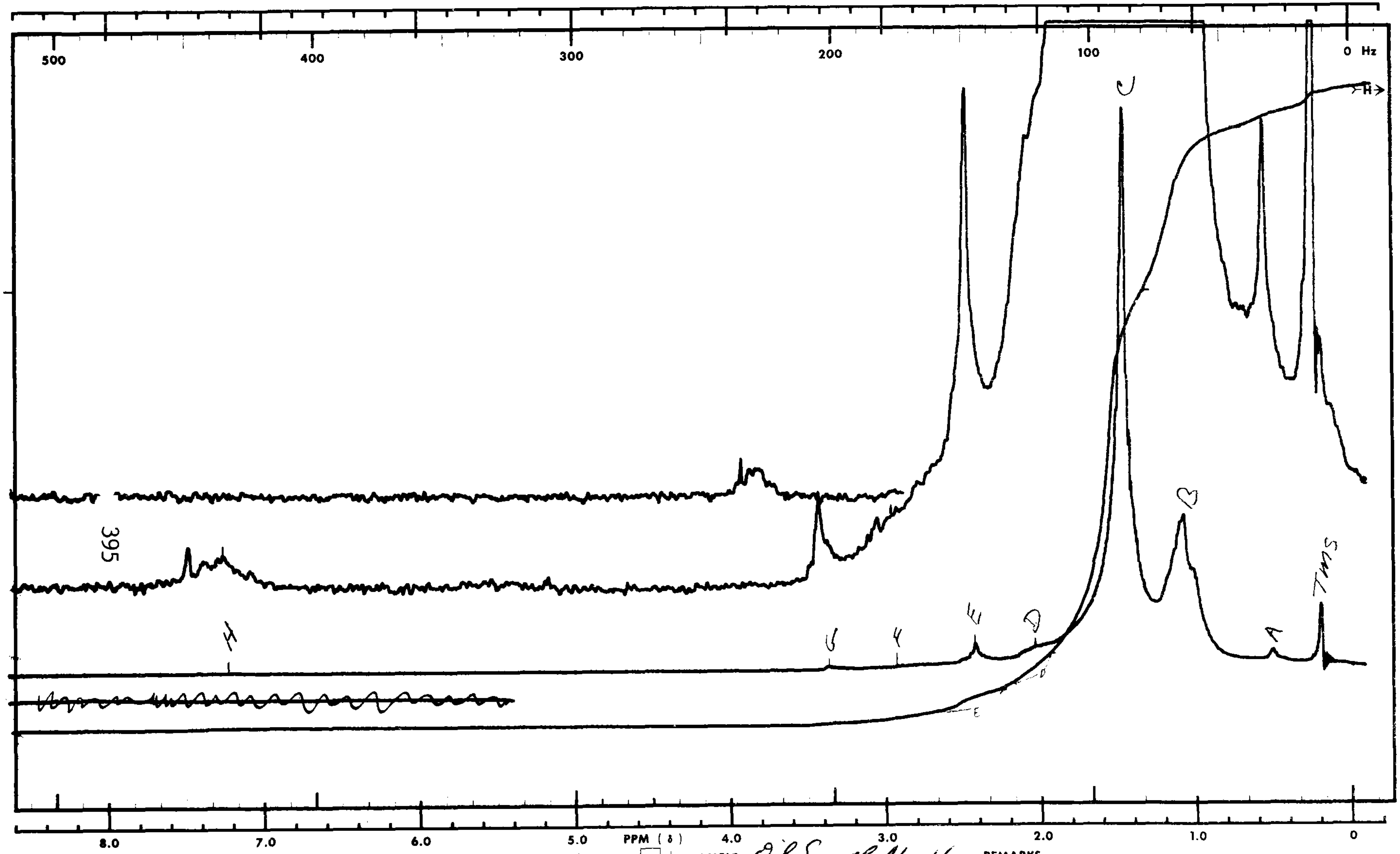
analytical instrument division

DATE: 8/7/78

OPERATOR: M. Barth

60 MHz NMR

SPECTRUM NO.



MANUAL **AUTO** ☐

SWEEP OFFSET (Hz): 0

SPECTRUM AMPLITUDE: 1.25(1)+25(2+3)

INTEGRAL AMPLITUDE: 5.0 at 1.25 spec amp.

SPINNING RATE (RPS): 53

SWEEP TIME (SEC): 50 ~~250~~ 1000 (2+3) (250)

SWEEP WIDTH (Hz): 25 50 100 250 ~~500~~ (500)

FILTER: ~~1~~ 2 ~~3~~ 4 5 6 7 8 2+3 (2)

RF POWER LEVEL: 0.05 (.05)

SAMPLE: Oil Sample No. 46

REMARKS:

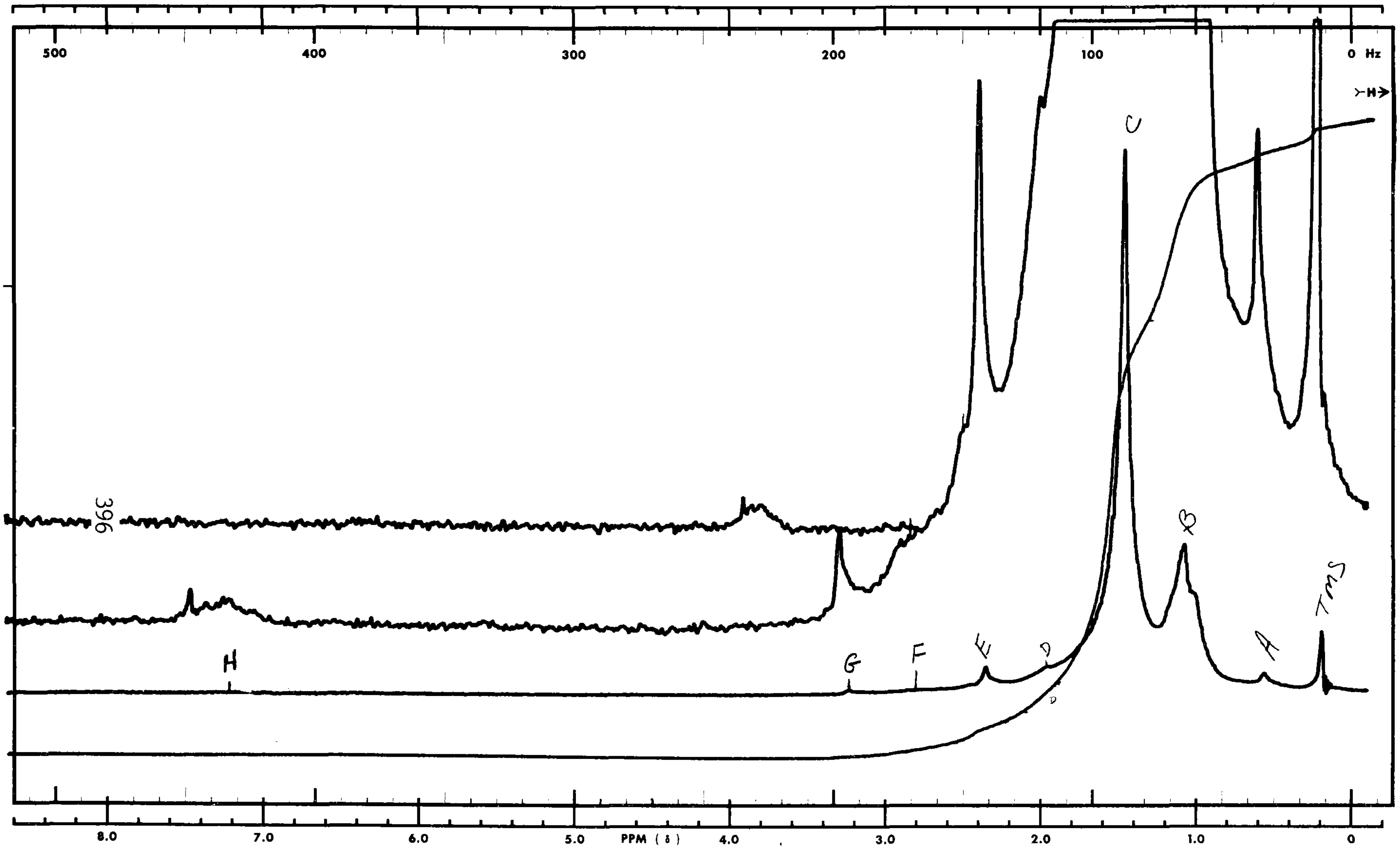
SOLVENT: 50% v/v in (CDCl₃ + 2% TMS)

varian
analytical instrument division

DATE: 8/7/78

OPERATOR: M. Barth

60 MHz NMR
SPECTRUM NO.



MANUAL

SWEEP OFFSET (Hz): 0

SPECTRUM AMPLITUDE: 1.25(1) 25(2+3)

INTEGRAL AMPLITUDE: 5.0 at 1.25 spec. amp.

SPINNING RATE (RPS): 50

SWEEP TIME (SEC): 50 ☒250 1000 (2+3)

SWEEP WIDTH (Hz): 25 50 100 250 ☒500

FILTER: ☒1 2 ☒3 4 5 6 7 8 (2+3)

RF POWER LEVEL: 0.05

AUTO ☐
(250)
(500)
(2)
(.05)

SAMPLE: Oil Sample No. 47

REMARKS:

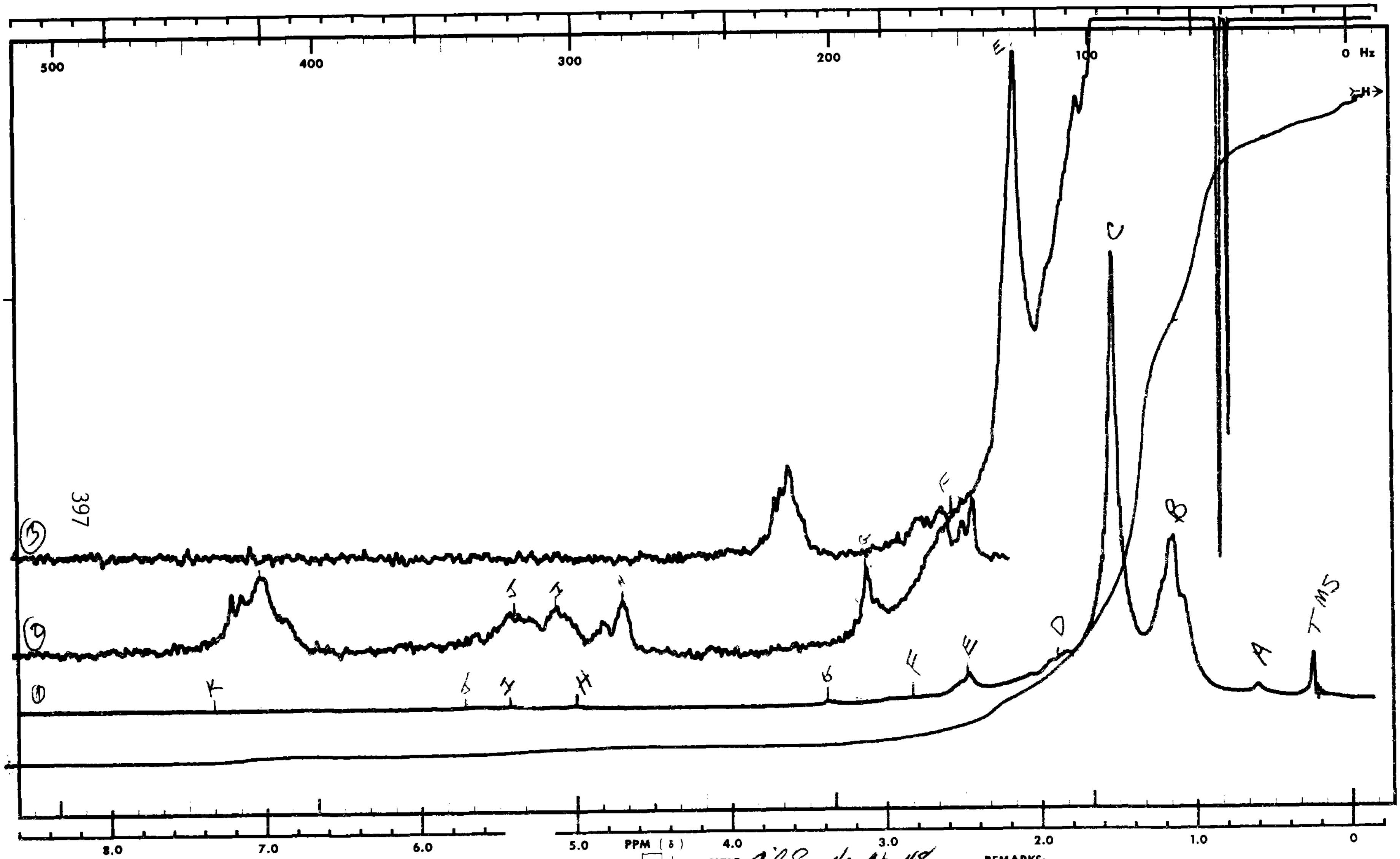
SOLVENT: 50% v/v in ($CDCl_3$ + 2% TMS)

varian
analytical instrument division

DATE: 8/7/78

OPERATOR: M. Barth

60 MHz NMR
SPECTRUM NO.



MANUAL

AUTO ☐

(250)

(500)

(2)

(.05)

SAMPLE: Oil Sample No. 48

REMARKS:

SWEEP OFFSET (Hz): 0

SPECTRUM AMPLITUDE: 1.25(1) + 25 (2+3)

INTEGRAL AMPLITUDE: 5.0 at 1.25 spec amp.

SPINNING RATE (RPS): 50(1) 52 (2+3 + int)

SWEEP TIME (SEC): 50 ☒250 1000(3)

SWEEP WIDTH (Hz): 25 50 100 250 ☒500

FILTER: ☒1 2 ☒3 4 5 6 7 8

RF POWER LEVEL: 0.05 (2+3)

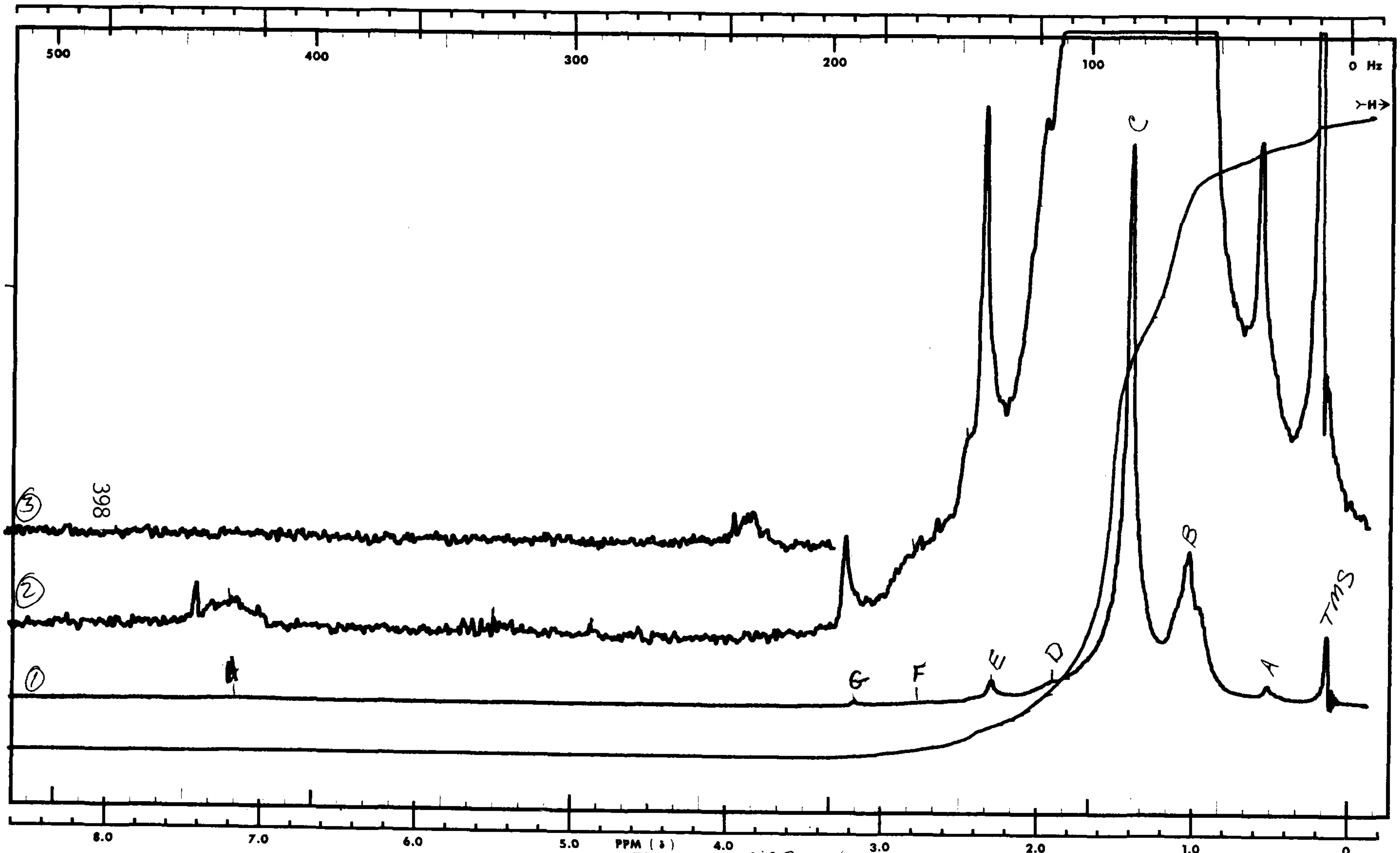
SOLVENT: 50% V/V in (CDCl$_3$ + 2% TMS)

varian
analytical instrument division

DATE: 8/8/78

OPERATOR: M. Burth

60 MHz NMR
SPECTRUM NO.



MANUAL

SWEEP OFFSET (Hz): 0

SPECTRUM AMPLITUDE: 1.25 (1) + 25 (2 + 3)

INTEGRAL AMPLITUDE: 5.0 at 1.25 Spec, Ampl.

SPINNING RATE (RPS): 50

SWEEP TIME (SEC): 50 ☒ 250

SWEEP WIDTH (Hz): 25 50 100 250 ☒ 500 1000 (3)

FILTER: ☒ 1 2 ☒ 3 4 5 6 7 8 (2+3)

RF POWER LEVEL: .05

AUTO ☐

(250)

(500)

(2)

(.05)

SAMPLE: Oil Sample No. 49

REMARKS:

SOLVENT: 50% v/v in (CDCl$_3$ + 2% TMS)

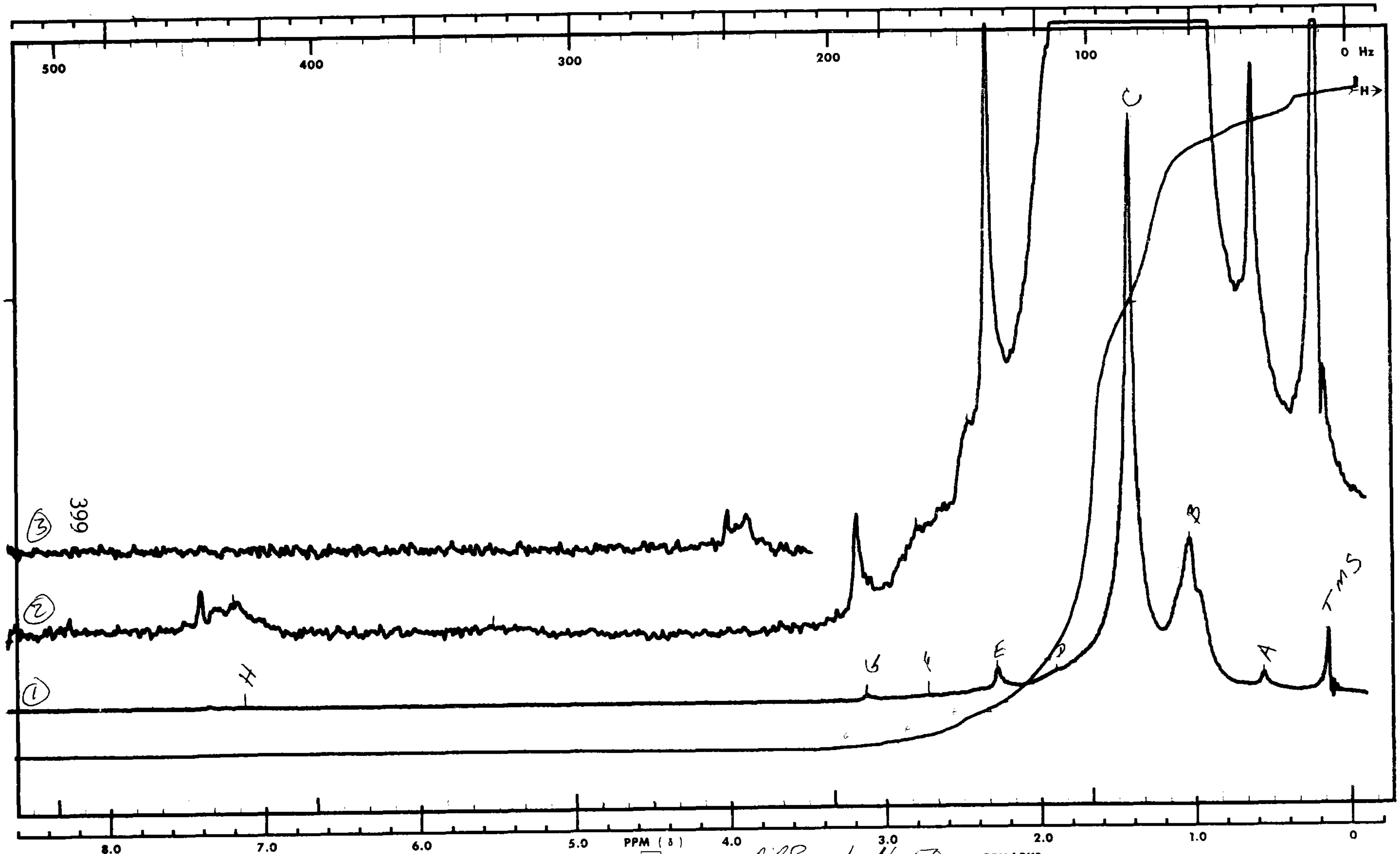
varian
analytical instrument division

DATE: 8/8/78

OPERATOR: M. Barth

60 MHz NMR

SPECTRUM NO.



MANUAL

AUTO ☐

SWEEP OFFSET (Hz): 0

SPECTRUM AMPLITUDE: 1.25 (1) + 25 (2+3)

INTEGRAL AMPLITUDE: 5.0 at 1.25 spec Amp.

SPINNING RATE (RPS): 48

SWEEP TIME (SEC): 50 ☒250 1000 (3) (250)

SWEEP WIDTH (Hz): 25 50 100 250 ☒500 (500)

FILTER: ☒1 2 ☒3 4 5 6 7 8 (2)

RF POWER LEVEL: 0.05 (2+3) (.05)

SAMPLE: Oil Sample No. 50

REMARKS:

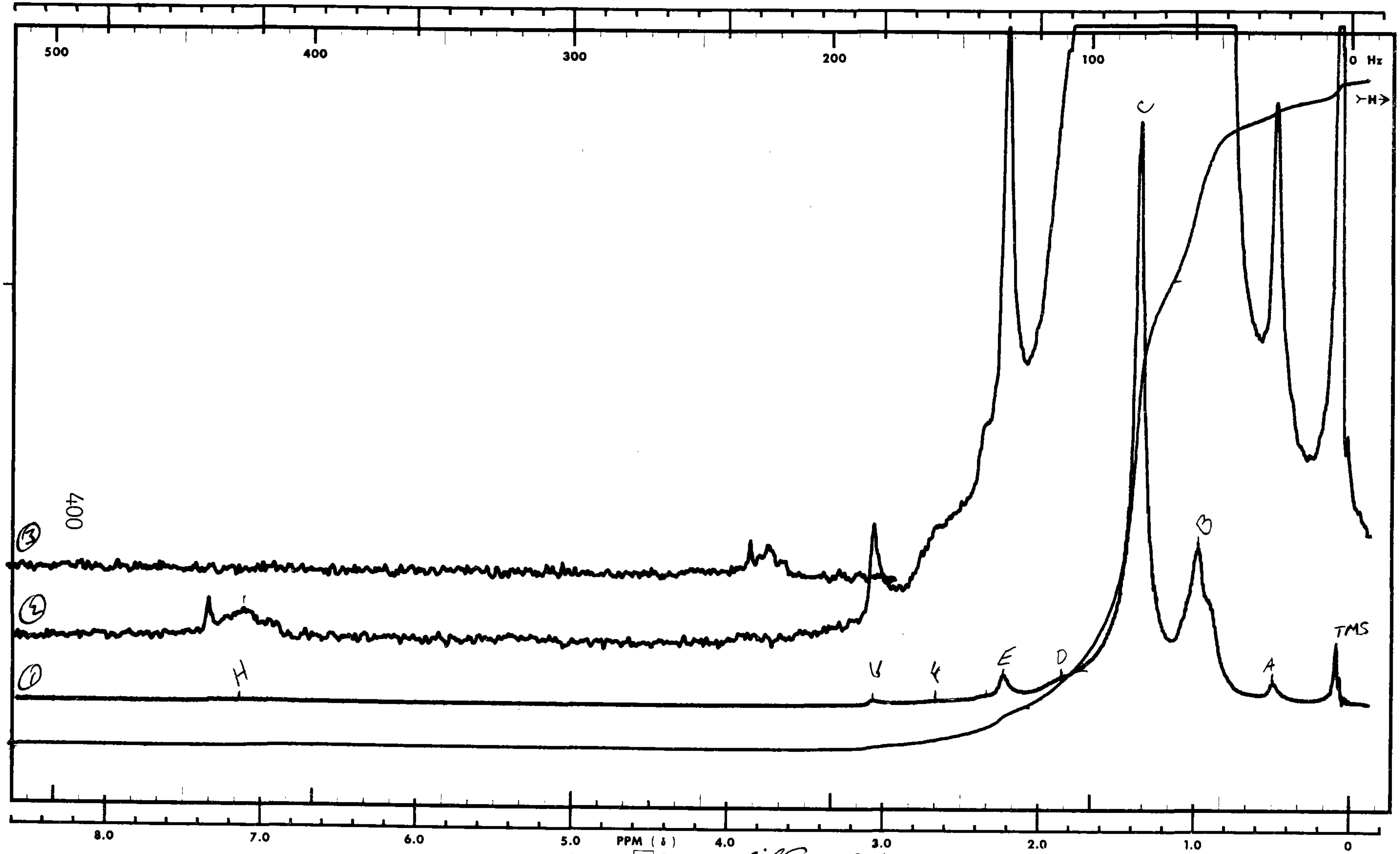
SOLVENT: 50% v/v in (CDCl$_3$ + 2% TMS)

varian
analytical instrument division

DATE: 8/8/78

OPERATOR: M. Barth

60 MHz NMR
SPECTRUM NO.



MANUAL

AUTO ☐

SWEEP OFFSET (Hz): 0

SPECTRUM AMPLITUDE: 1.25(1) + 25(2+3)

INTEGRAL AMPLITUDE: 5.0 at 1.25 spec. amp.

SPINNING RATE (RPS): 49

SWEEP TIME (SEC): 50 ☒100 1000(3) (250)

SWEEP WIDTH (Hz): 25 50 100 250 ☒500 (500)

FILTER: ☒1 2 ☒3 4 5 6 7 8 (2)

RF POWER LEVEL: 0.05 (2+3) (.05)

SAMPLE: Oil Sample No 51

REMARKS:

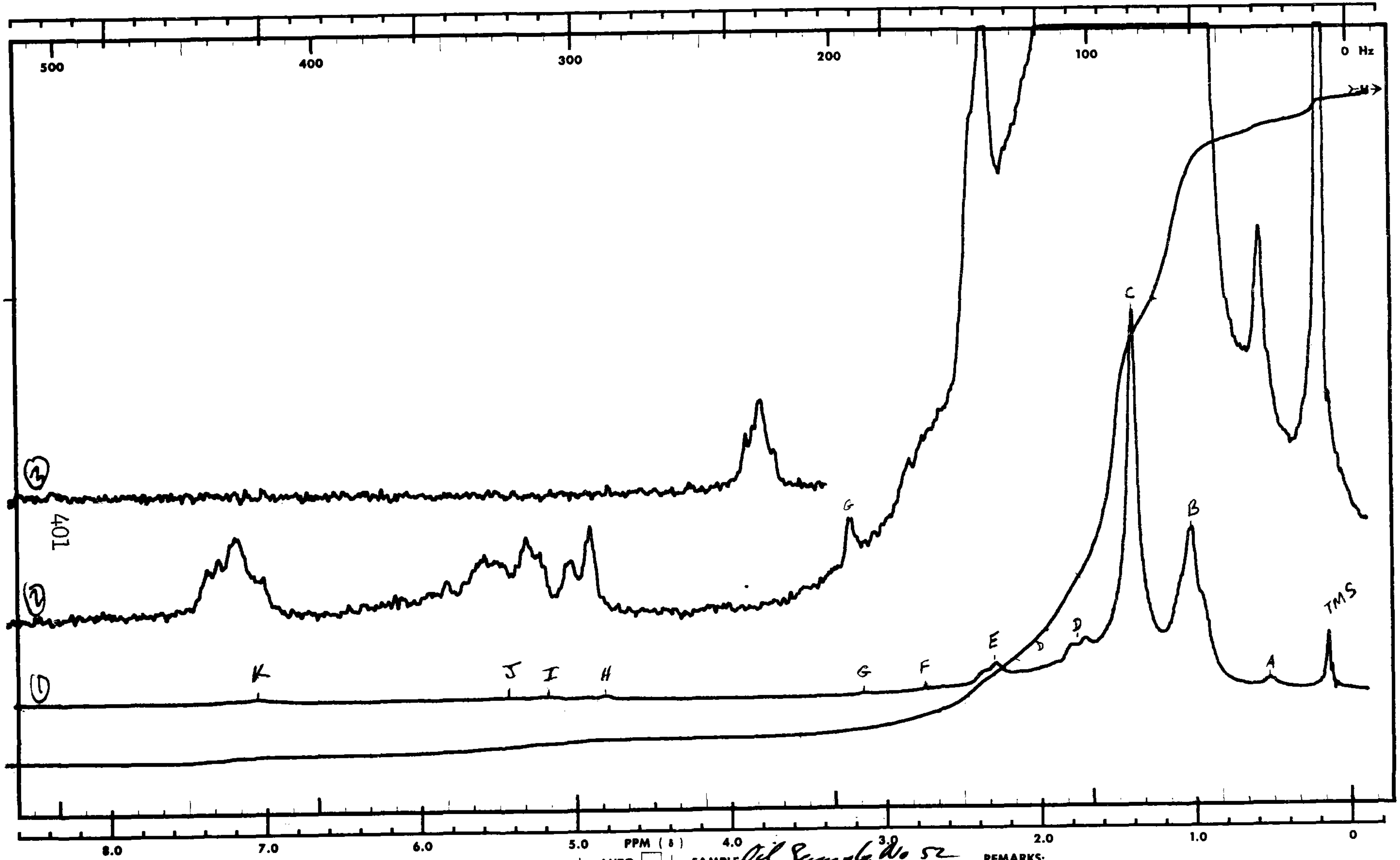
SOLVENT: 50% v/v in (CDCl$_3$ and 2% TMS)

varian
analytical instrument division

DATE: 8/9/78

OPERATOR: M. Barth

60 MHz NMR
SPECTRUM NO.



MANUAL

AUTO ☐

SWEEP OFFSET (Hz): 0

SPECTRUM AMPLITUDE: 125(1) + 25(2+3)

INTEGRAL AMPLITUDE: 5.0 at 1.25 [illegible]

SPINNING RATE (RPS): 50

SWEEP TIME (SEC): 50 ~~250~~ 1000(3) (250)

SWEEP WIDTH (Hz): 25 50 100 250 ~~500~~ (500)

FILTER: ~~1~~ 2 ~~3~~ 4 5 6 7 8 2+3 (2)

RF POWER LEVEL: 0.05 (.05)

SAMPLE: Oil Sample No 52

REMARKS:

SOLVENT: 50% v/v in (CDCl₃ and 2% TMS)

varian
analytical instrument division

DATE: 8/9/78

OPERATOR: M. Barth

60 MHz NMR
SPECTRUM NO.

"RESIDUE" ANALYSIS

"WAX-LIKE" RESIDUE ANALYSIS

Melting point experiments using a hot plate microscope indicated a 130-150°C melting point for the "wax-like" material. The solid appeared to melt in two areas 130 and 150° (Majority). Microscopic examination indicates that the particles are coated with a black material.

Solubility tests were run in solvents as follows. A few hundredths of mgs. of solid was mixed with 0.5 - 1 ml of solvent.

Solvent	Result (Solubility)
CCl_4 Carbon tetrachloride	Slight, partial
$CH\ Cl_3$ Chloroform	Slight, partial
CH_2Cl_2 Methylene Chloride	Slight, partial
Acetone	Slight, partial (white part)
Carbon Disulfide	Insoluble
p-Dioxane	Insoluble
Hexanes	Insoluble - slight
Toluene	Slight
Nitrobenzene	Slight

The solubility tests showed nitrobenzene, toluene, acetone to be the best solvents in that order. In spite of solubility at low levels (.1%) it was not possible to completely dissolve sufficient solid even to get a 0.5% solution. To obtain usable information from nuclear magnetic resonance spectra a concentration of at least 1% is desired.

NMR spectra were run of the nitrobenzene, acetone and carbontetrachloride solution. The spectra are as follows:

A. Nitrobenzene with TMS reference

B. Nitrobenzene + Sample + TMS Reference

C. Acetone + TMS reference

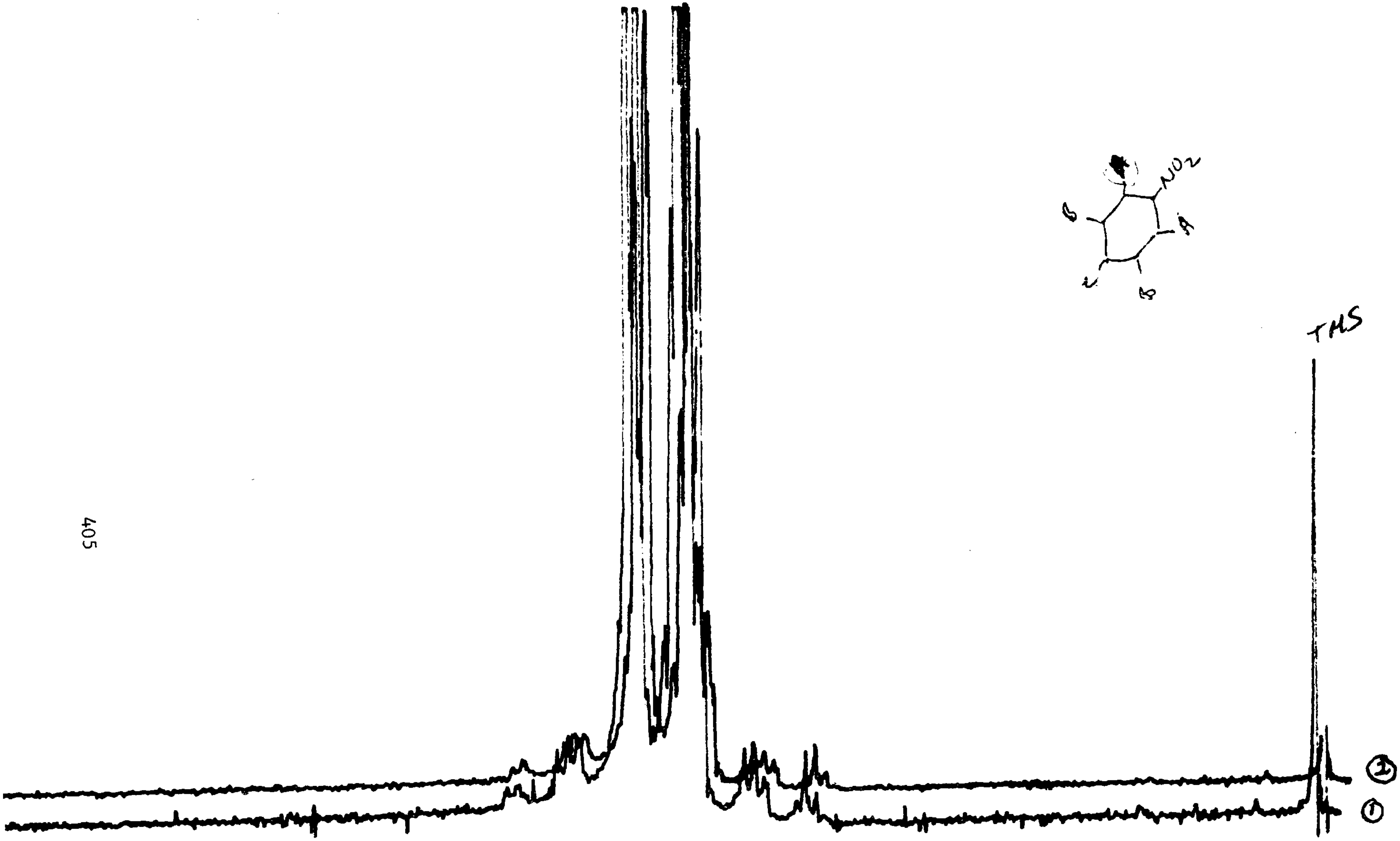
D. Acetone + Sample + TMS Reference (Clear supernatant only)

E. CCl_4 + Sample and CCl_4 alone

In none of the above spectra is there evidence of compounds other than those attributable to the solvent itself. The distortion evidenced in spectrum H is due to the presence of undissolved solids in the solution. On the basis of the available data the material appears to not be an ordinary hydrocarbon wax. These melt around 70-100°C.

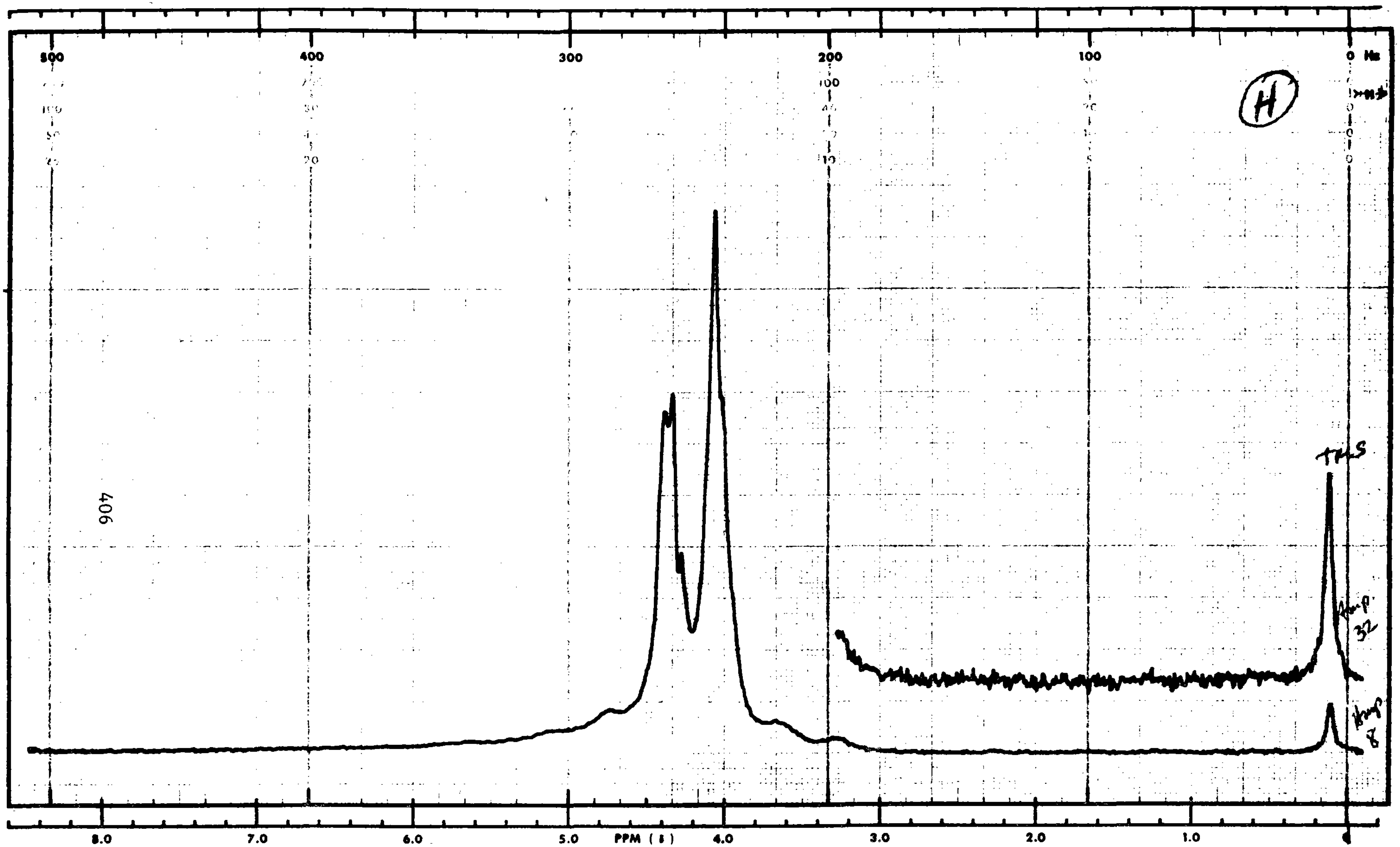
An infrared spectrum of a pressed pellet indicated that the material was a hydrocarbon. The observed absorptions were not intense enough to distinguish more about the structure. The 750-850 cm^{-1} region showed only a weak, unsplit absorption. A hydrocarbon solid such as a wax would exhibit a more intense absorption in this region. Also, the absorption may be split in solids. This was not observed.

On the basis of the available data, the material appears not to be an ordinary hydrocarbon wax. These melt about 50-80°C. The partial solubility, microscopic examination, and melting characteristics indicate that the solid is a mixture. The NMR and IR data are inconclusive since the sample size or solubility behavior limited the instrumental response. Additional information may be obtained by more solubility tests and possibly mass spectral examination of a dilute solution. In terms of the distillation and refinement of the bulk heat transfer oil the material is readily removed by vacuum filtration.



Nitrobenzene 0.5ml
+ ~0.4ml 2% TMS in CDCl3

Spin 42
Spec. Amp ① 1.25X10 ② 8.0
Rf .05
Filter 2
SW 1000
ST 250.



MANUAL

SWEEP OFFSET (Hz): 0

SPECTRUM AMPLITUDE: 8 + 32

INTEGRAL AMPLITUDE:

SPINNING RATE (RPS): 42

SWEEP TIME (SEC): 50 ~~250~~

SWEEP WIDTH (Hz): 25 50 100 250 500

FILTER: 1 ~~2~~ 3 4 5 6 7 8

RF POWER LEVEL:

AUTO ☐

(250)

(500)

(2)

(.05)

SAMPLE: Waxy Residue from 6/5/78 Side Drum Oil Sample partially dissolved in ϕ-NO_2 ~0.5% w/v

REMARKS:

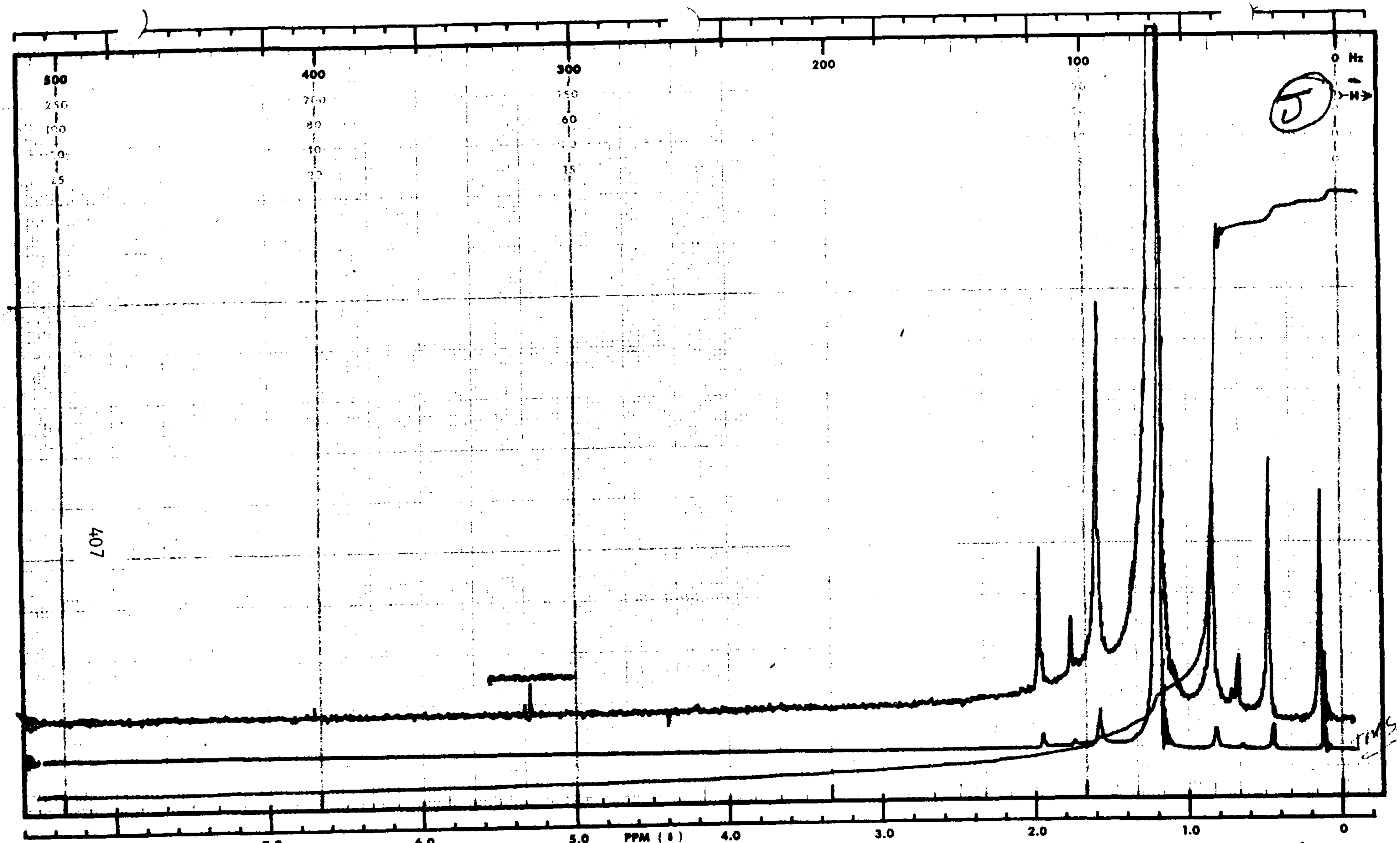
SOLVENT: ϕ-NO_2 + ~10% ($CDCl_3$ + 2% TMS)

varian
analytical instrument division

DATE: 6/14/78

OPERATOR: M. Barth

60 MHz NMR
SPECTRUM NO.



MANUAL

SWEEP OFFSET (Hz): 0 a b

SPECTRUM AMPLITUDE: 0.8 + 8.0

INTEGRAL AMPLITUDE: 6.5

SPINNING RATE (RPS): 42

SWEEP TIME (SEC): 50 [✓ 250]

SWEEP WIDTH (Hz): 25 50 100 250 500 1000

FILTER: [✓ 1] 2 3 4 5 6 7 8

RF POWER LEVEL: 0.05

AUTO ☐

(250)

(500)

(2)

(.05)

SAMPLE: Wax residue from 6/5/78 Oil Sediment - Partially dissolved in acetone.

REMARKS:

SOLVENT: Acetone + a little CDCl$_3$ + 2% TMS

varian

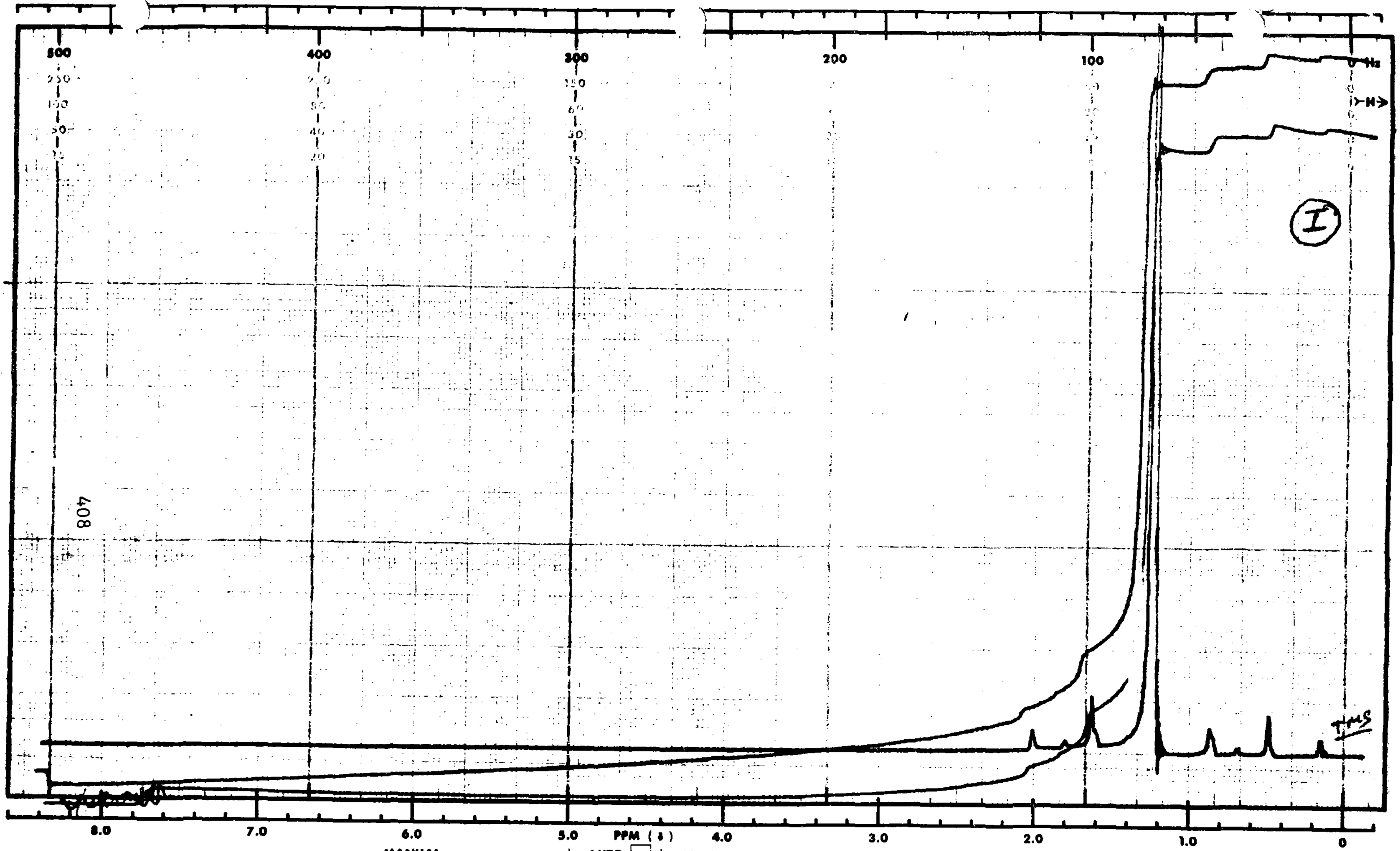
analytical instrument division

DATE: 6/14/78

OPERATOR: M. Booth

60 MHz NMR

SPECTRUM NO.



MANUAL

SWEEP OFFSET (Hz): 0

SPECTRUM AMPLITUDE: 0.8

INTEGRAL AMPLITUDE:

SPINNING RATE (RPS): 42

SWEEP TIME (SEC): 50 [✓ 250]

SWEEP WIDTH (Hz): 25 50 100 250 500 1000

FILTER: [✓ 1] 2 3 4 5 6 7 8

RF POWER LEVEL: 0.05

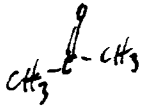
AUTO ☐
(250)
(500)
(2)
(.05)

SAMPLE: GC Grade Acetone

+ ~10% V/V (CDCl3 + 2% TMS)

SOLVENT:

REMARKS:



$CH_3-\overset{O}{\overset{\|}{C}}-CH_3$

varian
analytical instrument division

DATE: 6/14/78

OPERATOR: M. Barth

60 MHz NMR
SPECTRUM NO.

EXXON ANALYSIS

OF

PHASE I SAMPLES

EXXON COMPANY, U.S.A.
POST OFFICE BOX 2180 · HOUSTON, TEXAS 77001

MARKETING DEPARTMENT
MARKETING TECHNICAL SERVICES

July 25, 1978

Analysis of Caloria(R) HT 43 Sample
455.8020-1

Mr. Dave Beshore
Martin Marietta Aerospace
Mail Stop 50403
P. O. Box 179
Denver, Colorado 80201

Dear Sir:

Attached are data on three samples received from Mr. John Myers. These samples were identified as follows:

- Sample C-1, Barrel C, Caloria HT 43 as received from manufacturer, 1-18-78.
- Sample 13, HV-15, Bulk Oil Before Vent Down, 3-8-78.
- Sample 15, HV-19, Sample of Ice Water Condensate of Vented Volatiles, 3-8-78.

Dr. G. V. Chambers of Exxon Research and Engineering supplied the data and comments which are included in this letter. These samples were analyzed by a combination of techniques. The techniques included:

(1) Distribution of components by boiling point using a gas chromatographic procedure (ASTM D2887). This was used on Samples C-1 and 13. See Attachment 1 and Table 1.

(2) Differential absorption by infrared spectroscopy to compare Sample 13 with Sample C-1 (thermally exposed oil vs. non-exposed oil), see Figure I.

(3) Hydrogen-type distribution by nuclear magnetic resonance on all three samples. See Table 2.

(4) Combined gas chromatography and mass spectroscopy on Sample 19(vented volatiles condensate). See Table 3.

Caloria HT 43 After Thermal Exposure

The composition of the Caloria HT 43 after thermal exposure (HV-15) was characteristic for thermally exposed Caloria HT 43. That is, cracking/dealkylation resulted in a slight increase in aromatics and olefins as well as the formation of lighter boiling point components. This increase in either olefins or aromatic content is very small, but evident in both MNR and IR spectra.

A calculated 8 vol% of additional material was formed with a boiling point less than 600°F (12.2 vs 3.9 vol% at 600°F). In contrast, higher boiling point material was not formed. Thus, polymerization was not evident.

This sample is also characteristic for thermally exposed Caloria HT 43 in that the range of lower boiling point components formed is wide. This uniformity in lower boiling components formed is unlike high temperature cracking which results in predominately C_2-C_3 olefins. The carbon number here ranges from C_1 to C_{20}.

<u>Vented Volatiles</u>

The sample of vented volatiles which is described as an ice water condensate is composed primarily of normal paraffins and isoparaffins, which peak at C_7-C_8, but also contains relatively large amounts of one-ring naphthenes and olefins (Table 3). This general description is based on a GC analysis through C_{12} with additional interpretation from mass spectra data. It is necessary to utilize this combination since the GC has not been calibrated for each individual component above C_7. In regard to the C_8 unknowns shown in Table 3, these are likely olefins in view of the distribution of C_4-C_7 olefins in the sample.

The tabulation provided in Table 4 has been normalized to 100% based on the total sample. The computerized GC program is only good through C_{12} (Table 3). The sample actually contains 25% higher boiling point materials than C_{12}, and this is shown as C_{12} + unresolved components.

It should be pointed out that the vented volatiles probably contain larger amounts of C_1-C_4 components than reflected by the sample. These extremely low boiling point components (C_1-C_4) would not likely be quantitatively recovered in the sample using ice water for condensation.

The numbers given in the tables should be viewed with caution, as there is variability in any of the analytical methods. The values given are for single determinations, due to the very small sample size. Also, as a result of the small sample size, it was not possible to determine typical data such as viscosity, gravity, etc. These would have been desirable.

Please let me know if you have any questions about the data.

Very truly yours,

T. G. Lipscomb

T. G. Lipscomb

TGL:sah
Attachment

c: E. K. Cooper
G. V. Chambers (Baytown)

Mr. J. E. Reynolds

ATTACHMENT 1

GC DISTILLATION PROCEDURE

(ASTM D-2887)

Column:	1/4 inch stainless steel, 30 inches long packed with UCW 98 (5%) on Chromosorb G
Helium Flow Rate:	$\sim$70 ml/min
Detector Temperature:	400°C
Injection Port Temp.:	400°C
Oven Temperature Program:	40° → 390°C at 16°/min
Sample Size:	$\sim 3 \mu l$

TABLE I

GC DISTILLATION
CUMULATIVE PERCENT OFF

			After Thermal Exposure	
Temp. °F	°C	Caloria HT 43	Liquid HV-15	Vented Volatiles HV-19
300	149	0	1.2	40.3
350	177	0	2.1	53.8
400	205	0	3.1	64.0
430	222	0	3.7	69.9
450	233	0	4.2	72.8
500	260	0.4	5.7	79.8
550	288	1.5	8.3	85.1
600	316	3.9	12.2	89.2
650	344	7.7	17.7	92.8
700	372	13.3	24.9	95.4
750	399	22.4	35.0	97.4
800	427	38.2	50.0	98.8
850	455	60.7	68.4	99.6
900	483	80.6	83.1	100
950	510	91.0	90.7	100
1000	538	95.7	94.7	100
1050	566	100	98.7	100
1100	954	100	100	100

TABLE 2

HYDROGEN DISTRIBUTION BY NMR

	% Of Hydrogens		
		After Thermal Exposure	
Type of Hydrogen	Caloria^R HT 43	Liquid HV-15	Vented Volatiles HV-19
∂+ CH_3	27.6	26.7	39.3
∂+ CH, CH_2, β CH_3	59.7	57.7	37.8
β CH, CH_2	9.8	10.7	13.9
Olefins	0	0.5	2.0
α groups (to aromatic ring)	2.2	3.7	5.5
α^2 groups (to aromatic ring)	0	0	0
Uncondensed aromatic ring	0.7	0.5	1.3
Condensed aromatic rings	0	0.2*	0.2*

---

* At the detection level; actual level may be "0"

Table 3

GC/MS Combination Analysis on Condensate Sample

Sample: 15, Source HV-19, 3/8/78

Carbon Number	Paraffins	Naphthenes		Benzenes	Indans and Tetralins	Napthalenes	Olefins	Unresolved Components
		1-Ring	2-Ring					
1	<0.1	-	-	-	-	-	-	-
2	0.1	-	-	-	-	-	0.0	0.0
3	1.0	0.0	-	-	-	-	0.0	0.0
4	2.3	0.0	-	-	-	-	0.9	0.0
5	4.1	0.2	-	-	-	-	2.5	0.0
6	5.1	0.9	-	0.0	-	-	2.6	0.0
7	6.9	1.4	-	0.0	-	-	2.4	0.0
8	9.5	2.3	0.0	1.5	-	-	0.0	3.2*
9	5.2	2.0	2.5	1.4	0.0	-	0.0	0.0
10	3.5	1.3	0.8	0.9	0.3	0.2	0.0	0.0
11	3.0	1.2	0.6	0.4	0.4	0.1	0.0	0.0
12	1.9	1.0	0.4	0.3	0.5	0.1	0.0	0.0
12+	-	-	-	-	-	-	-	25.0#
Total	42.7	10.3	4.3	4.5	1.2	0.4	8.4	28.2

* Believed to be mostly olefins.

Not resolvable.

FIGURE I

SPECTRUM NO.
DATE 6-15-78
SAMPLE Salicia
T-43 after exp vs
Saladia NT 43
SOURCE GVc
STRUCTURE

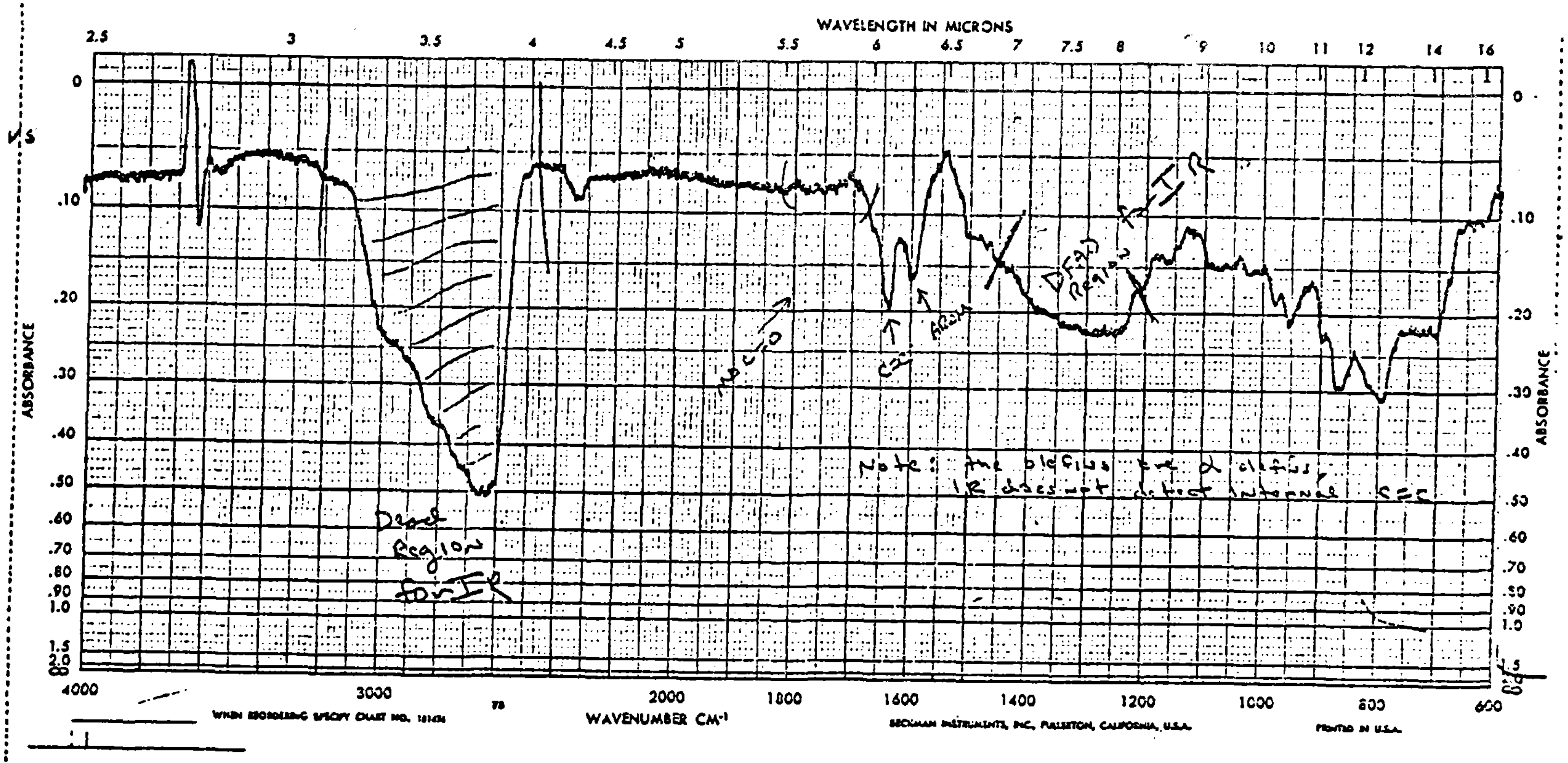
416

PATH 1.02 mm
SOLVENT
CONCENTRATIO
PHASE 416
COMMENTS
Scans 120

ANALYST TR

Beckman®

INFRARED
SPECTROPHOTOMETER



Note: the olefins are α olefins, IR does not detect internal C=C

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