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AFRPL-TR-71-46

**QUARTERLY PROGRESS REPORT
THERMALLY STABLE
BURNING RATE ACCELERATORS (U)**

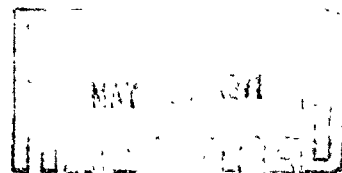
Contract F04611-70-C-0066

April 1971

**Prepared for
Air Force Systems Command
Air Force Propulsion Laboratory
Edwards Air Force Base, California 93523**

Prepared by

Thiokol
CHEMICAL CORPORATION
HUNTSVILLE DIVISION
HUNTSVILLE, ALABAMA



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QUARTERLY PROGRESS REPORT

1 December 1970 through 28 February 1971

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FOREWORD

This report is prepared in accordance with the requirements of Contract F04611-70-C-0066.

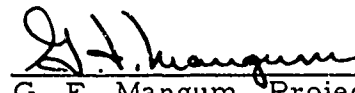
Program Structure Number	623148
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Prime Contractor	Thiokol Chemical Corporation Huntsville Division Huntsville, Alabama

This report, the third quarterly progress report to be issued under this program, covers effort performed for the period 1 December 1970 through 28 February 1971. This report has been assigned the Thiokol internal number 16-71 (Control No. C-71-16A).

The principal investigators on the program are Dr. W. D. Stephens, Dr. D. A. Flanigan, and Mr. J. O. Hightower. Dr. M. Miller is serving as Program Manager. In addition to those contributions by the principal investigators, major contributions to the program were made by Dr. T. C. Willis, Messrs. J. W. Blanks, C. M. Christian, Mrs. Patricia B. Walters, and Dr. R. E. Rogers. The over-all Project Director is Mr. G. F. Mangum.

STATEMENT OF APPROVAL

Publication of this report does not constitute Air Force approval of the report's findings or conclusions. It is published only for the exchange of ideas.


G. F. Mangum, Project Director

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ABSTRACT

THERMALLY STABLE UFAP

Two methods of producing thermally stable UFAP have been examined experimentally. These include ultrasonic grinding and spray-freeze drying. The ultrasonic grinding technique reduced the AP particle size from 200 micron to 3 micron; however, further reductions could not be realized using currently available commercial equipment and established techniques. The spray freeze drying process was utilized to prepare UFAP(e.g., 0.47 micron) which exhibited good thermal stability. Six pounds of thermally stable oxidizer have been prepared by the technique described in the previous quarterly report. This material will be used in subsequent particle size reduction technique evaluation.

Examination of propellant components in combination with AP has provided the following conclusions: A mixture of R45M/IPDI with thermally stable AP is eight times as stable as a mixture of HC-434/MAPO with the same oxidizer; mixtures of oxidizer, polymer, and iron oxide are stable so long as only two components are present; however, when all three materials are involved, thermal stability is much poorer. Polybutadiene/iron oxide mixtures give a slight increase in weight upon exposure to air at high temperature. Removal of volatile polymer components has been shown to help increase thermal stability of propellants.

THERMALLY STABLE CATALYSTS

A number of synthetic routes have been utilized in efforts to obtain model structures for correlating ferrocene-containing catalyst stability with structure. Certain groups which were desired for their electron withdrawal ability, or because they could be utilized to eliminate alpha-hydrogen atoms, could not be prepared because of synthetic problems. These groups include alpha-chloroalkyl, sulfone, sulfonate, and p-nitrobenzoyl.

More than twenty-five different compounds have been synthesized and of these, eight have been prepared and purified by techniques described earlier. These models have been tested by reaction with air at elevated temperature, and by thermal stability of propellants made from these model compounds. In general, the air uptake experiments are in good agreement with the TGA experiments using propellant. Significant conclusions from these experiments are that exclusion of alpha-hydrogen atoms and incorporation of electron withdrawal are primary factors in achieving good thermal and oxidative stability. It has been shown that excessive electron withdrawal can cause chemical vulnerability of the catalyst in propellant.

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INTRODUCTION

(C) The purpose of this program is to research the methods and materials that will produce thermally stable AP and burning rate catalyst. They will be used with other thermally stable propellant ingredients to produce a solid propellant that is capable of withstanding a temperature environment of -75° to 350°F .

(C) With the present capability of aircraft to fly at supersonic speeds, a new requirement now exists for missiles which can be carried externally. The major development problem arises from the effects of aerodynamic heating, causing missile skin temperatures of several hundred degrees Fahrenheit. In addition, the external carry means that a missile in the airstream is exposed to temperatures of about -65°F (the temperature in the isothermal layer). Missiles and missile components therefore are subject to temperatures from -65°F to well over 300°F .

(C) Propellants that will survive this treatment, and also possess the desirable ballistic properties, are not available. Two general approaches to assure the rocket motor's operation at all times are to insulate the motor or develop a propellant to withstand the environment. Internal or external insulations increase the motor cross sectional area and weight. Also, internal insulation decreases propellant volume. Since the missile is normally exposed to these conditions for a relatively short time, the insulation approach is inefficient. The penalties paid for insulation are too great; thermally stable propellant is a better solution.

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PROGRAM OBJECTIVES AND ORGANIZATION

OBJECTIVES:

(C) Very small particle oxidizer (less than one micron) and burning rate catalysts have been used to achieve high burning rates; however, commercially available ammonium perchlorate is prone to thermal decomposition which, in turn, degrades propellant. Some liquid burning rate catalysts also have undesirable characteristics such as crystallization, migration, or volatilization and are subject to thermal and oxidative decomposition. The objectives of this program are: (1) to develop methods of production of stable ultrafine ammonium perchlorate (UFAP), (2) to demonstrate improved methods of burning rate catalysis, and (3) utilization of these rate augmentation techniques in propellants which must survive temperature cycling up to 350°F.

PROGRAM ORGANIZATION:

(U) This program is divided into three tasks as follows:

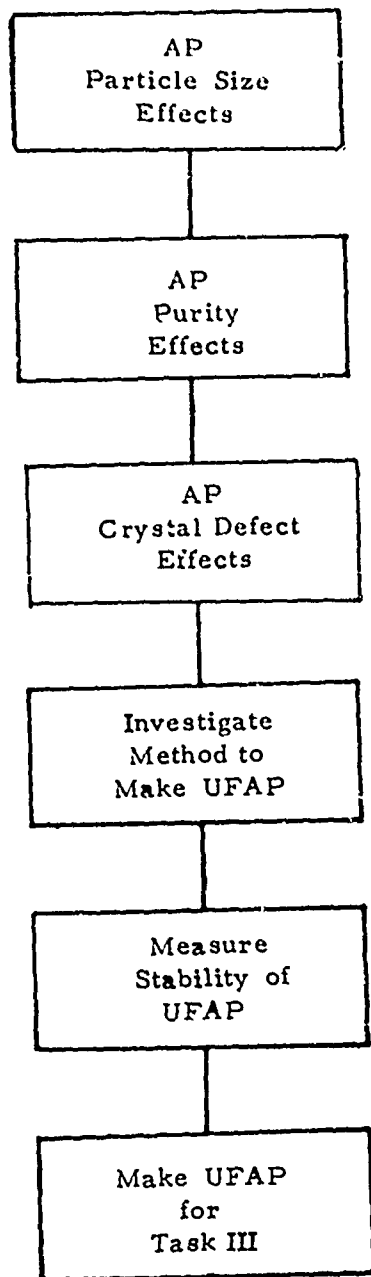
Task I, Thermally Stable UFAP, is subdivided into three subtasks. The first defines AP thermal stability and identifies the qualities that AP must have to be stable. In the second, new methods of preparing UFAP are examined and in the third subtask, the method of preparing UFAP is chosen and enough UFAP prepared for the demonstration part of the program.

Task II, Thermally Stable Catalysts, is divided into four subtasks. These include impurity investigations, structure investigations, improved catalysts, and synthesis of material for Task III.

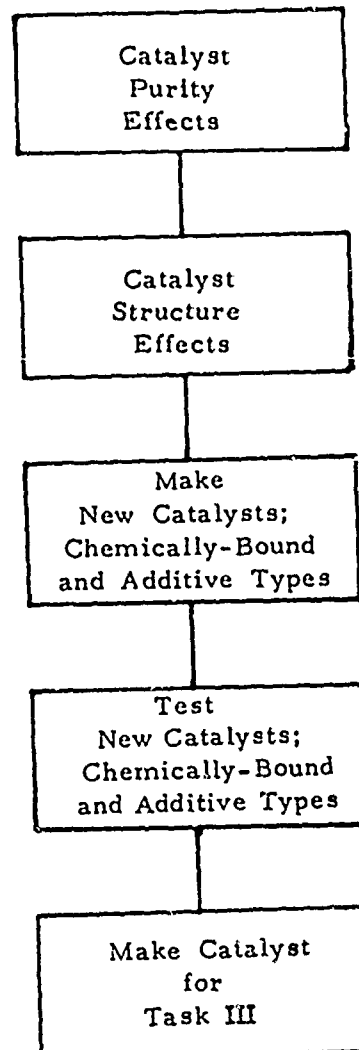
Task III, Demonstration, is that part of the program in which the thermally stable UFAP and an improved catalyst are combined into a propellant for demonstration.

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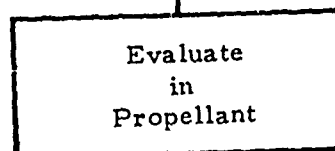
TASK I



TASK II



TASK III



PROGRAM OUTLINE

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DISCUSSION

ACCOMPLISHMENTS

Task I - Thermally Stable Ammonium Perchlorate

Primary effort during this report period was concerned with an investigation of the feasibility of reducing the particle size of ultrapure ammonium perchlorate to a submicron particle size using ultrasonic irradiation techniques. Data presented in earlier reports showed that ultrasonic fracture did not have any deleterious effect upon thermal stability. Reports of disintegration of tissue, minerals, etc., to their basic units of structure with ultrasonics and dispersion of paint pigments or agglomerates to submicron particles suggested the possibility of application to AP particle size reduction. At a given ultrasonic frequency and power input, the AP particle should fracture and dissociate to the desired size, and the reduction would be achieved without product contamination. A continuous process is envisioned, if the technique proves successful, in which large quantities of ultrapure, ultrafine AP could be produced in a practical manner.

Experiments, shown in Tables I and II and described in detail below, demonstrated that ultrasonic irradiation effected a reduction in the particle size of the AP; however, a size reduction below about 3 microns was not achieved in these initial experiments which utilized various diluents, exposure times, dispersing aids, power inputs (at a frequency of 20KHz) and slurry concentrations. Data in the literature indicate an optimum ultrasonic frequency exists for breaking agglomerates, for example, dispersing of paint pigments. Consequently, to realize the full potential of the technique, the optimum ultrasonic frequency for dissociating an AP particle must be utilized. A higher (than 20KHz) frequency is apparently needed and efforts to acquire such a transducer have been unsuccessful.

Twenty-eight experimental runs were made to evaluate this technique. The first group of experiments was conducted with equipment obtained from Heat Systems - Ultrasonic, Inc. The apparatus consisted of an ultrasonic transducer containing a lead zirconate titanate crystal mounted to a stainless steel probe which tapered to a titanium tip of 1/2 inch diameter to concentrate the energy. A generator supplied a variable power of 0-200 watts to the transducer which operated at a frequency of 20 KHz. The sample of AP was placed in a one inch diameter cylindrical glass container holding a suitable solvent, such as Freon 113, to act as a slurry medium and heat sink; in some cases, a coating agent was added to the slurry to prevent particle agglomeration. The container was placed in a cold bath to restrict the temperature rise of the contents. AP particle size was determined by MSA analysis.

*January Quarterly

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TABLE 1

ULTRASONIC GRINDING OF AP

(Using 20 KHz Sonifier Cell Disruptor Model W-140-E)

Sample No.	Composition	Size before grind (Micron)	Power (watts)	Grind Time (min)	Size After Grind (Micron)
S-1	1gAP/6% Monawet 15 ml Freon 113	200	100	25	7.00
S-2	1gAP/6.5% Monawet/ 20 ml. Freon 113	200	200	68	3.35
UG-1	SVM-213 AP/Freon 113 (86.1/13.9) 2% COPA-3	1.03	100	60	1.05
UG-2	SVM-213	1.03	160	45	1.01
UG-3	SVM-213	1.03	160	28	1.12
UG-4	SVM-213	1.03	160	15	1.07
UG-5	AP/Freon 113	27	200	45	19
UG-6	AP/Freon 113	79	200	60	10.5
UG-7	1gAP/2% Monawet/ 25 ml Freon 112	200	200	68	3.25
UG-8	2gAP/3% Monawet/ 25 ml Freon 112	200	200	68	6.30
UG-9	1gAP/3% Lecithin/ 25 ml Freon 112	200	200	68	5.20
UG-10	MCM-42, S-4 AP/Hexane (75/25)/ 2% Monawet	0.72	200	68	0.79
UG-11	1gAP/2% Monawet/ 25 ml Hexane	200	200	68	---
UG-12	1gAP/1% Lecithin/ 25 ml Hexane	200	200	68	8.5

Surfactant Concentration Based on AP Weight

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TABLE II

ULTRASONIC GRINDING OF AP USING A MICRO-TIP DEVICE

<u>Sample No.</u>	<u>Composition</u>	<u>Size before Grind (Micron)</u>	<u>Power (Watt)</u>	<u>Grind Time (min)</u>	<u>Size after Grind/ micron</u>
USR-1	1 gm purified AP/ 30 cc Freon 113 No Surfactant	23.1	300 (microtip)	60	4
USR-2, 3	1 gm purified AP/ 30 cc Freon 113/ No Surfactant	23.1 (microtip)	300	60	----
UG-13	1 gm purified AP/ 30 cc Freon 113/ No Surfactant	23.1	300 (microtip)	60	7.2
UG-14	1 gm purified AP/ 30 cc Freon 113/ No Surfactant	23.1	300 (microtip)	120	5.3
UG-15	1 gm purified AP/ 30 cc Freon 113	23.1	300 (microtip)	60	4
UG-16	No Surfactant	200	300 (microtip)	60	6.7
UG-17	No Surfactant	23.1	300 (microtip)	60	5.7
UG-18	5 gm purified AP/ 30 cc Freon 113/ No Surfactant	23.1	300 (microtip)	300	1.0

Surfactant Concentration Based on AP Weight

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Initial experiments, S-1 and S-2, (Table I) utilized a Freon 113/AP/Monawet MT slurry in which 200 micron unground AP was subjected to the irradiation for about an hour. Particle size was reduced to 3.35 micron WMD utilizing the maximum 200 watts power capability. Continuing experiments, UG-1, 2, 3, 4 were conducted with a slurry (initially processed in the vibro-energy mill) which contained Freon 113, COPA-3 and 1.03 micron WMD AP. The slurry was irradiated at 160 watts power level for times varying from 15 to 60 minutes. No reduction in the AP size was achieved at any of the irradiation exposure times. Experiments UG-5 and UG-6, utilized a slurry of Freon 113/AP with no surfactant and 27 and 79 micron WMD AP. Irradiation for one hour at 200 watts resulted in a particle size of 10.5 microns. Tests UG-7, UG-8 and UG-9 were made with Freon 112 as the slurry medium. Results were basically the same as with Freon 113; namely, the AP was reduced in size from 200 microns to 3.25 micron in about one hour. Similar results were obtained with lecithin and Monawet MT (Table I). Experiments performed with n-hexane as the slurring medium (using the slurry processed in the vibro-energy mill which had an AP particle size of 0.72 micron) indicated no reduction in size was achieved with the ultrasonics irradiation; however, as with the other diluents, particle size reduction did occur with the 200 micron feed.

The apparatus was exchanged for a unit with a capability of 300 watts power input and having a "microtip", i.e. a probe, that tapered to 1/4 inch diameter tip, to concentrate more energy where the slurry contacted the probe. Twelve experiments were conducted with this equipment. Generally, with a similar procedure, particle size could be reduced to the same lower limit as achieved with the less powerful unit. Processing was extended to two hours in one experiment; after one hour the 23 micron AP had been reduced to 7.2 microns and after two hours to 5.3 microns. (UG-13 and UG-14 Table II)

Effort was continued to study interactions between the freeze dried thermally stable ammonium perchlorate and CTPB and HTPB polymers. The sample of ARCO HTPB R-45M, lot 3475, was subjected to an isothermal TGA at 375°F. The polymer lost no weight in 26 hours, but did polymerize quickly. Efforts at removing impurities and volatiles from this lot of HTPB polymer through extraction with ethyl alcohol/benzene mixtures resulted in a less thermally stable material, particularly in the early stages of exposure to temperature.

A sample of HTPB polymer (lot 3475) was mixed in a 1/1 ratio with the freeze dried thermally stable AP and exposed to 375°F. Approximately 50 hours at 375°F was required before 1% weight loss was measured. As was the case with the neat polymer, polymerization occurred shortly after exposure to the elevated temperature.

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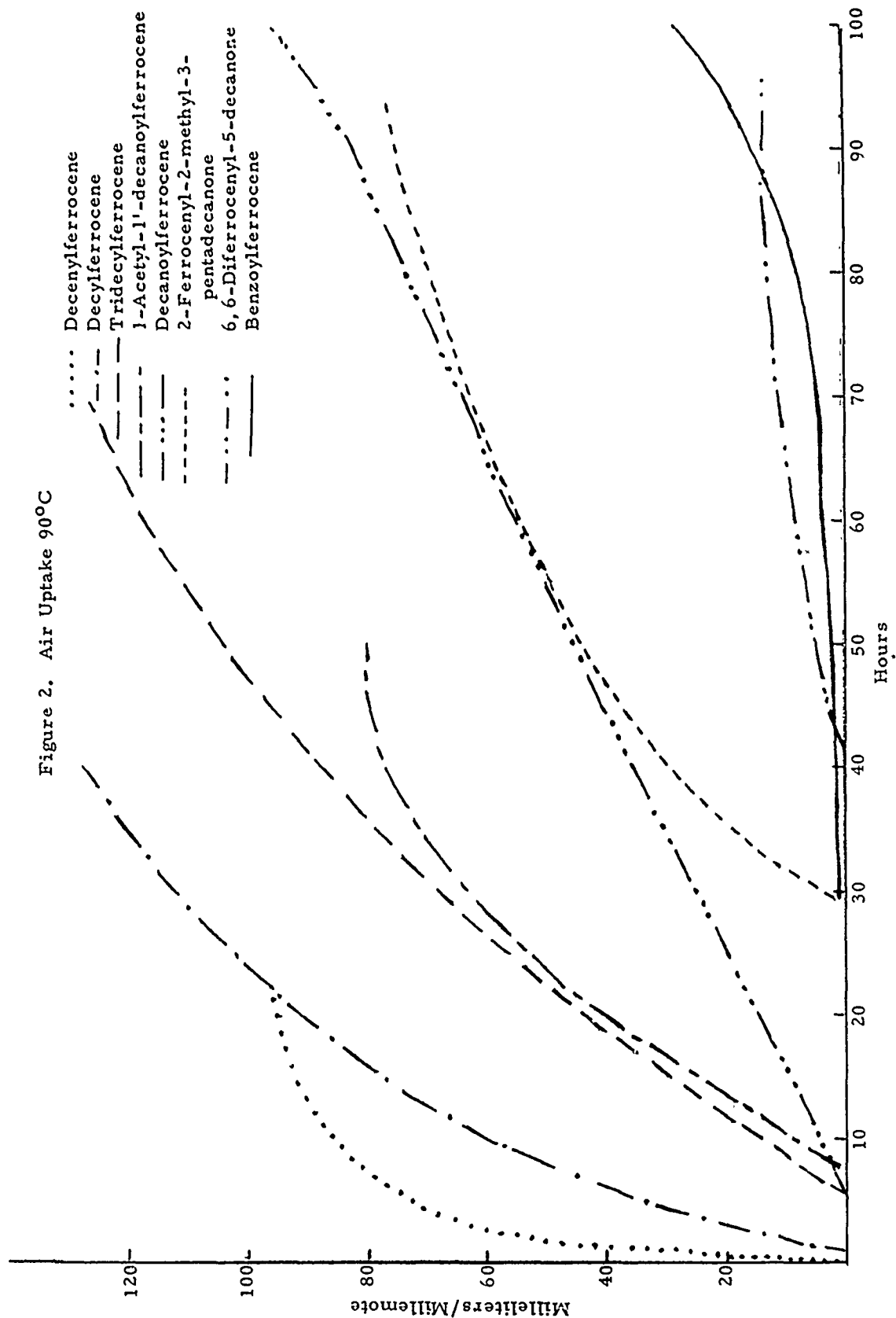


Figure 1. Air Uptake 90°C

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was even less as evidenced by a 1% weight loss in two and one-half hours. Initial attempt at determining the source of this instability was directed at possible interactions between the iron oxide and the polymers. Therefore, mixtures of polymer and iron oxide were subjected to isothermal TGA studies at 375° F and in hopes that a weight loss producing reaction could be detected. However, in mixtures of iron oxide with both HC-434 and R-45M polymer, a slow weight gain was observed which indicated some degree of oxidation and shows that iron oxide is a good oxidation catalyst for the binder. Further effort is now underway to elucidate the source of this instability when all the propellant ingredients are mixed together. Primary effort will be directed toward removing the moisture from the iron oxide and the polymer and eliminating the ionic impurities from the iron oxide.

A sample of "as received" ammonium perchlorate was spray freeze-dried and coated with 1% Strodex PK-90. The particle size of this sample was 0.47 micron. Exposure to 375° F in an isothermal TGA test showed an initial weight loss due, primarily, to the volatile constituents in the Strodex PK-90, and also to the surface moisture of the oxidizer; however, after this initial weight loss of less than 0.5%, no additional weight loss was noted up to 90 hours of exposure at which time the test was terminated. A similar experiment using UFAP manufactured from material produced from the pre-purified ammonium perchlorate is planned. The fact that the very high level of thermal stability was obtained from non-prepurified ammonium perchlorate adds further credence to the fact that moisture, particularly internal moisture, plays a significant role in facilitating thermal instability of ammonium perchlorate. The internal moisture content of the spray freeze dried ammonium perchlorate is present in only trace quantities.

A six-pound sample of ammonium perchlorate has been shock precipitated from isopropanol in order to obtain a large enough sample so that extensive studies with the various particle size reduction techniques can be made. The sample contained excessive amounts of moisture following recrystallization and several drying techniques have been utilized in order to effect further improvements in the thermal stability of the oxidizer before proceeding on the particle size reduction studies. Vacuum drying at elevated temperatures (75° C) has provided the thermal stability of 37 hours at 375° F for the six-pound sample. Studies are presently being initiated on spray freeze-drying, fluid energy milling, spray drying, and solvent-non-solvent precipitation.

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TASK II - THERMALLY STABLE CATALYSTS

The technical approach to achieving thermally stable burning rate catalysts was initiated with a study of the impurities present in alkylferrocenes, and the effects of these impurities upon propellant stability. These experiments showed that three critical classes of impurities had to be eliminated to improve thermal stability. These classes are the hexane insoluble components, the volatile materials and the ionic contaminants.

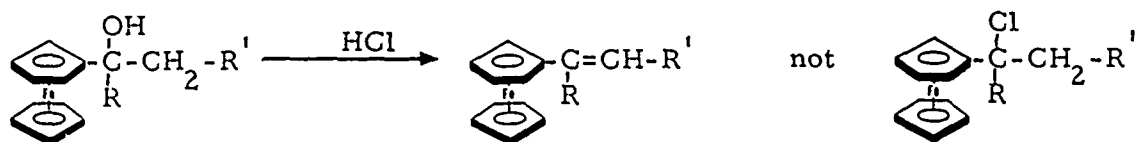
The second part of Task II involves an assessment of those features of molecular structure which contribute most to the oxidative and thermal stability of ferrocene compounds.

Synthesis of Model Compounds

At the present time a number of model compounds have been synthesized and purified and are being evaluated in thermal stability tests.

α -Chloroalkylferrocenes

The proposed synthesis of α -chlorodecylferrocene and α -chloro- α -methyltridecylferrocene from the corresponding alcohols has been unsuccessful. These alcohols dehydrate very readily under acidic conditions and



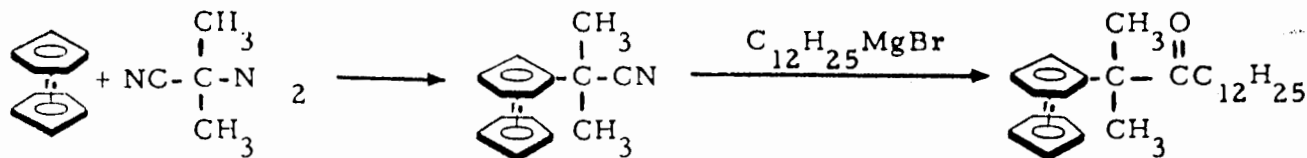
addition to the double bond is not easily accomplished. No further work is planned on these compounds. The α -chloro group was desired to replace oxidizable α -hydrogen atoms.

Branched Ferrocenes

The synthesis of branched ferrocenes by Friedel-Crafts alkylation with tertiary alkyl halides and tertiary alcohols has been attempted. Low yields and complex mixtures of products have resulted. A tertiary compound has been prepared by the reaction of ferrocenium ion with azo-bis-isobutyro-nitrile to yield 2-cyano-2-ferrocenyl-propane in approximately

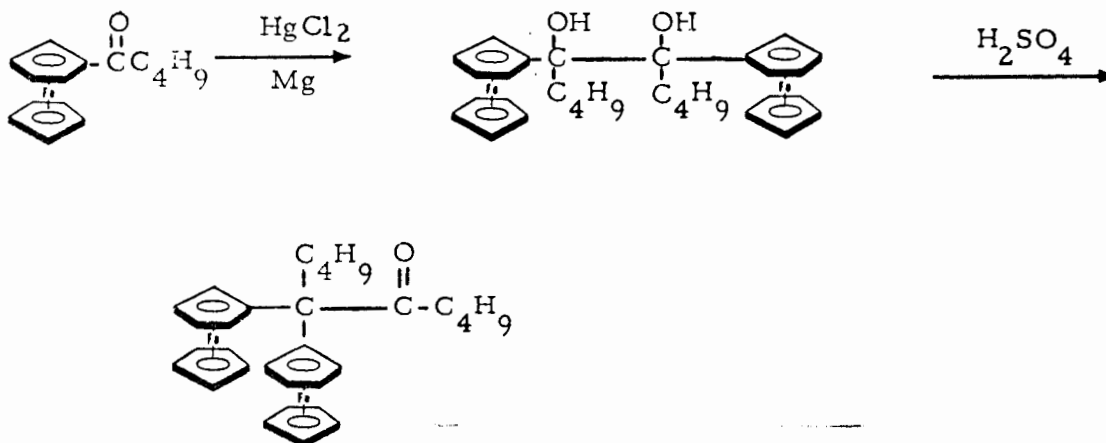
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30% yield. Reaction of the nitrile with dodecylmagnesium bromide gave



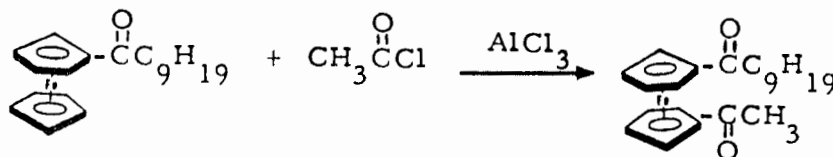
approximately 50% yield of 2-ferrocenyl-2-methyl-3-pentadecanone.

6,6-Diferrocenyl-5-decanone was previously prepared in this laboratory from valerylferrocene and provided a model with low volatility, and no susceptible alpha-hydrogen atoms. Valerylferrocene was converted to 5,6-diferrocenyl-5,6-decanediol via a bimolecular reduction. The diol undergoes a pinacol-pinacolone type rearrangement to give the diferrocenyl ketone.



Diacylferrocenes

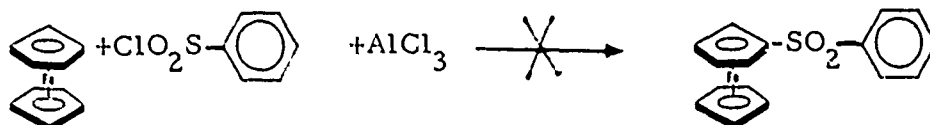
In order to assess the effect of two electron withdrawing groups on the stability of ferrocene compounds, a diacyl compound was prepared. Acylation of decanoylferrocene with acetyl chloride gave a 30% yield of 1-acetyl-1'-decanoylferrocene.



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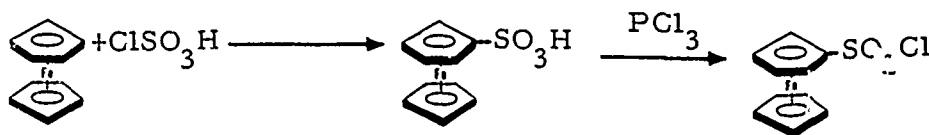
Ferrocenylsulfones and Sulfonates

Attempts were made to sulfonylate with benzene sulfonyl chloride and o-nitrobenzene sulfonyl chloride under Friedel-Crafts conditions. A number of variations in reaction conditions were used, including use of the sulfonic anhydride instead of the sulfonyl chloride. The desired models were unobtainable by these reactions.



The reaction of ferrocenyllithium with o-nitrobenzene sulfonyl chloride gave a very small yield (TLC) of an unidentified product. Further pursuit by this method was considered impractical.

The reaction of chlorosulfonic acid with ferrocene gave ferrocene sulfonic acid in good yield. Treatment with phosphorous trichloride produced ferrocene sulfonyl chloride.



The reaction of ferrocenesulfonyl chloride with 1,4-butanediol gave a product whose NMR shows only ferrocenyl and methylene protons. However, GPC indicates this product is a mixture. Further purification and characterization are needed.

p-Nitrobenzoylferrocene

The Friedel-Crafts acylation of ferrocene with p-nitrobenzoyl chloride has been attempted. Variations in solvent, temperature, catalyst ratio, and order of addition were unable to effect reaction. Likewise, reaction of ferrocenyllithium with p-nitrobenzoyl chloride failed to produce the desired result.

Purification of Model Compounds

Eight model compounds containing alkyl, alkenyl, acyl, diacyl, benzoyl and tertiary groups were purified for use in the stability evaluations.

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All compounds contain only a single component as indicated by column chromatography and thin layer chromatography. The critical impurities as determined in Part I of Task II were removed as follows.

The hexane insolubles were removed by extracting the compound with hexane. Ionic impurities were removed by passing a solution of the model compound through Amberlyst 15 (acidic) and Amberlyst 21 (basic) ion exchange resins. Volatile impurities were removed by prolonged warming at approximately 50°C under high vacuum.

Stability Tests - Model Compounds

To evaluate thermal stability a number of experiments have been performed. The absorption of air (Figure 1) by the model compounds has been measured at 90°C. TGA (Figures 2 and 3) and DSC of the model compounds have been recorded. The model compounds have been evaluated in a propellant of the following formulation:

HC Binder	34.3%
Al	10%
AP	50%
Model Compound	4.04%
ERLA-510	1.65%
Chrome Octoate	.01%

The rate of weight loss during TGA of the model propellants initially was thought to be volatility of the catalyst. Later experiments showed this to be incorrect.

Data from DSC analysis of the model compounds did not indicate significant differences in stability.

The data from air uptake experiments are shown in Figure 1, and reflect oxidation susceptibility at elevated temperature (200°F).

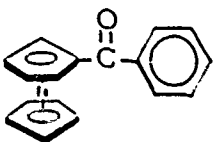
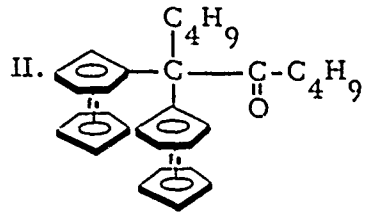
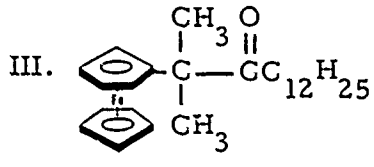
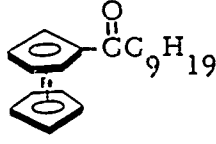
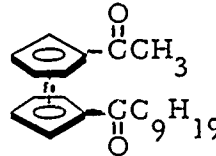
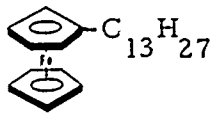
TABLE I

<u>Model Compound</u>	<u>% Wt. loss from Propellant in 5 hrs. at 160°C</u>
Decenylferrocene (VIII)	2.2
Benzoylferrocene (I)	2.4
6, 6'-Diferrocenyl-5-decanone (II)	3.0
Tridecylferrocene (VI)	3.0
Decanoylferrocene (IV)	3.1
2-Ferrocenyl-2-methyl-3-pentadecanone (III)	3.2
1-Acetyl-1'-decanoylferrocene (V)	3.8
Decylferrocene (VII)	3.8

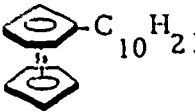
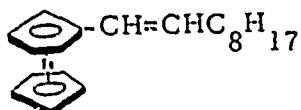
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From the air uptake and TGA of propellants, the model structures have been ranked with the numbers increasing with decreasing stability.

TABLE II

<u>Model Compound</u>	<u>Air Uptake 90°C</u>	<u>TGA of Propellant 160°C</u>
I. 	1	2
II. 	2	3
III. 	3	4
IV. 	4	6
V. 	5	7
VI. 	6	5

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<u>Model Compound</u>	<u>Air Uptake 90°C</u>	<u>TGA of Propellant 160°C</u>
VII. 	7	8
VIII. 	8	1

Conclusions

The unexpected difficulty of Friedel-Crafts reactions involving nitrobenzoyl chloride, benzenesulfonyl chloride, and nitrobenzenesulfonyl chloride suggests that even if these structures proved attractive, the difficulty in obtaining these structures precludes their use in a practical sense.

Stability studies conducted to date have given some unexpected data. Compound VIII which reacted very rapidly with air at 90°C was expected to show very poor results in propellant. The contrary effect was noted. For propellants made with sand or aluminum instead of oxidizer, there is essentially no weight loss in 24 hours. Therefore, it is assumed that propellant weight losses result from loss of volatile fragments due to oxidizer decomposition in the presence of catalyst. The relatively low weight loss from propellant containing VIII may involve a reaction between the polymer and the unsaturated link in VIII. Alternatively, the air uptake tests suggests that VIII may act as a trap which prevents oxidizer decomposition fragments from reacting further to give volatile products.

Another unexpected result of the stability studies was noted in the behavior of compound V. It was expected that greater electron withdrawal in the heteroannularly substituted diacyl derivative would lend greater thermal and oxidative stability since the mono ketones are significantly better than alkyl derivatives. In essence the idea was that "if one acyl group is good, two are better." Test results reveal this is not true.

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It should be noted that for a monoacyl derivative the nuclear magnetic resonance absorptions for the ring protons in the substituted ring are shifted down field. In diacyl derivatives protons on both rings are shifted. This effect may deshield the iron atom and render it vulnerable to chemical attack. (Figures 4 - 11).

Early results from experiments with propellants using benzoyl-ferrocene and oxidizer of varying stabilities are not easily correlated because the three oxidizers used were of different particle sizes. There appears to be a better correlation to oxidizer particle size than to stability of the oxidizer (Figure 3). This would indicate a surface-dependent interaction between catalyst and AP.

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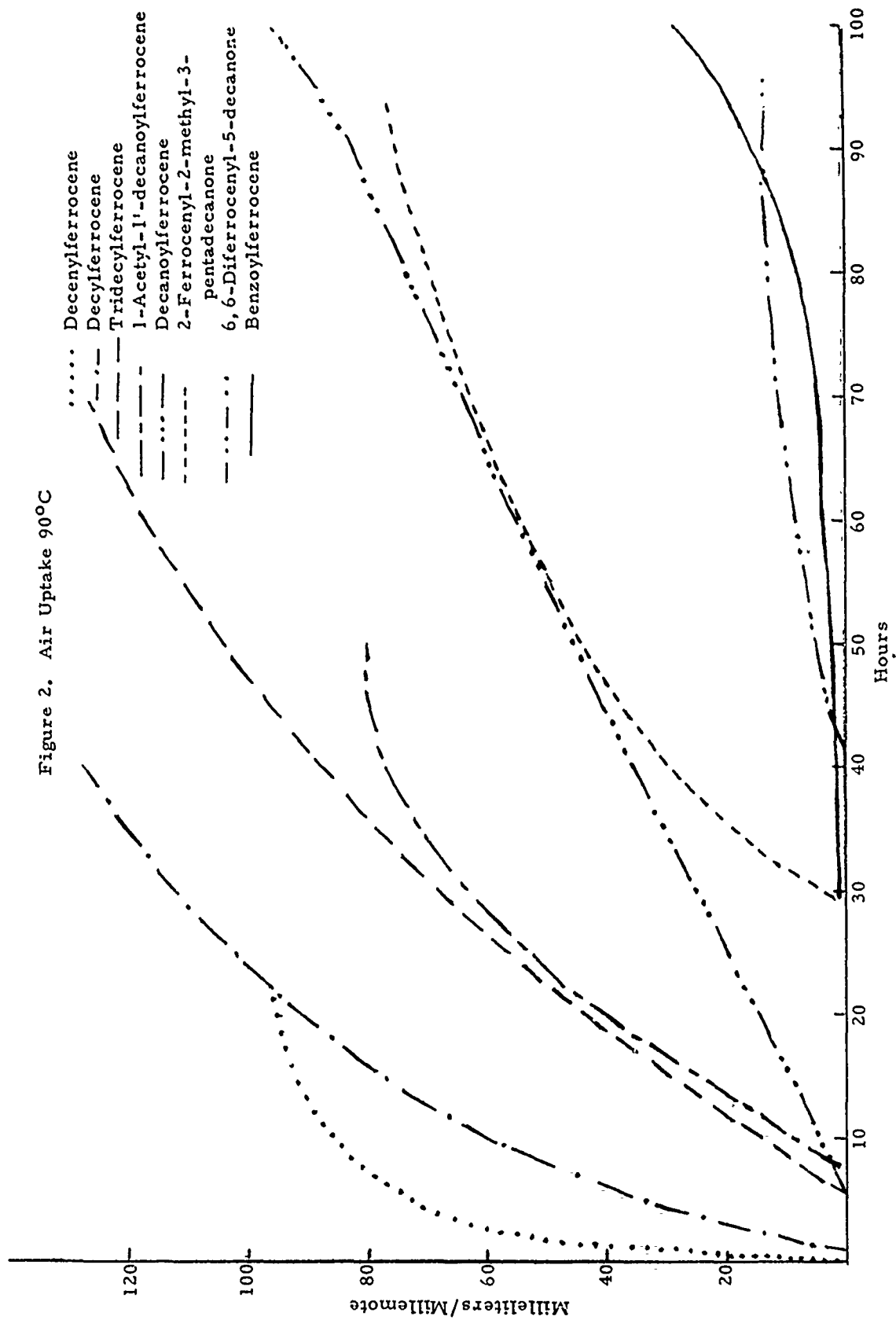


Figure 1. Air Uptake 90°C

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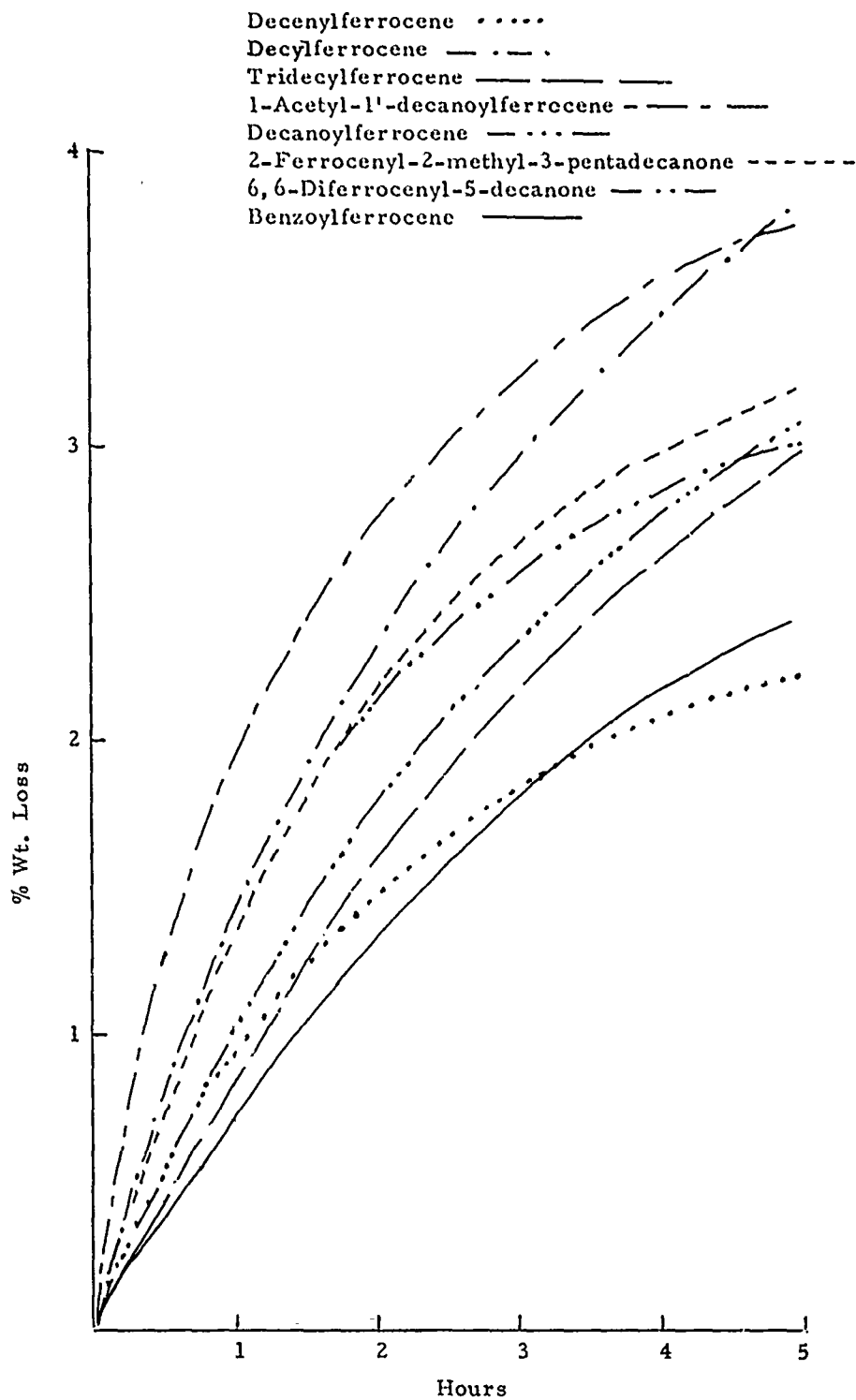


Figure 2. TGA of Propellants at 160°C

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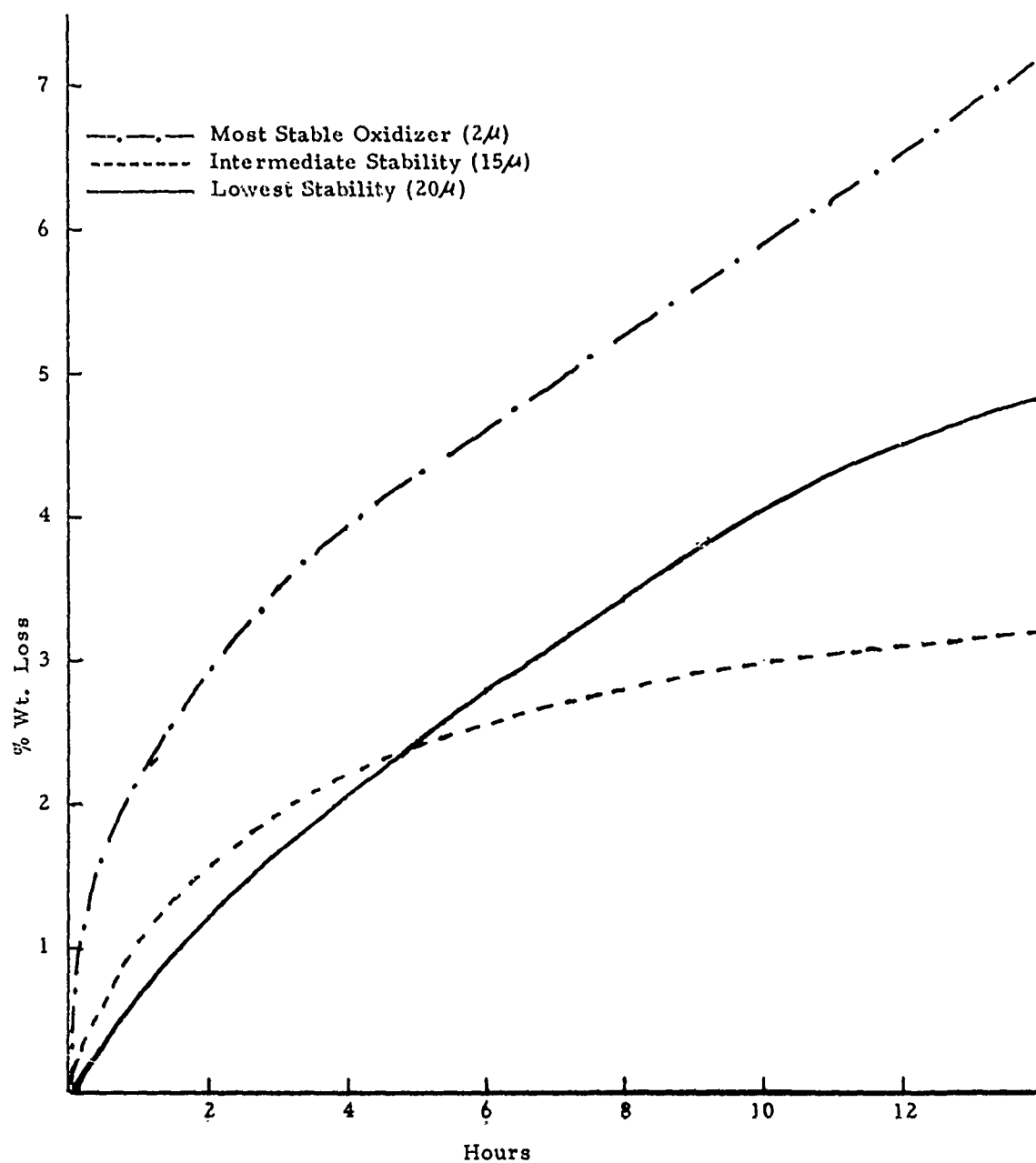


Figure 3. TGA of Propellants with Benzoylferrocene and Oxidizer of Varying Stability

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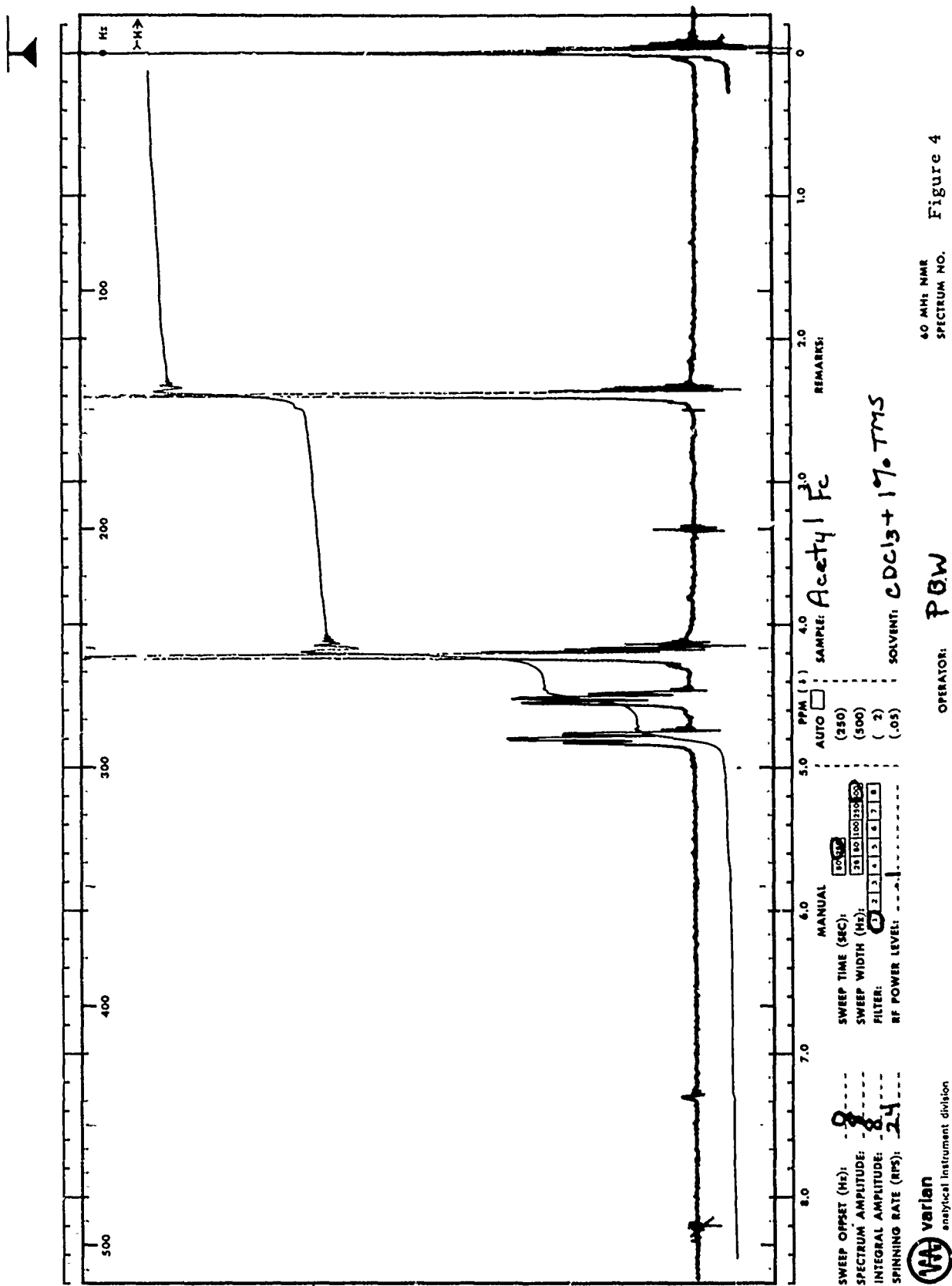


Figure 4. NMR Characteristics of Acetyl Ferrocene

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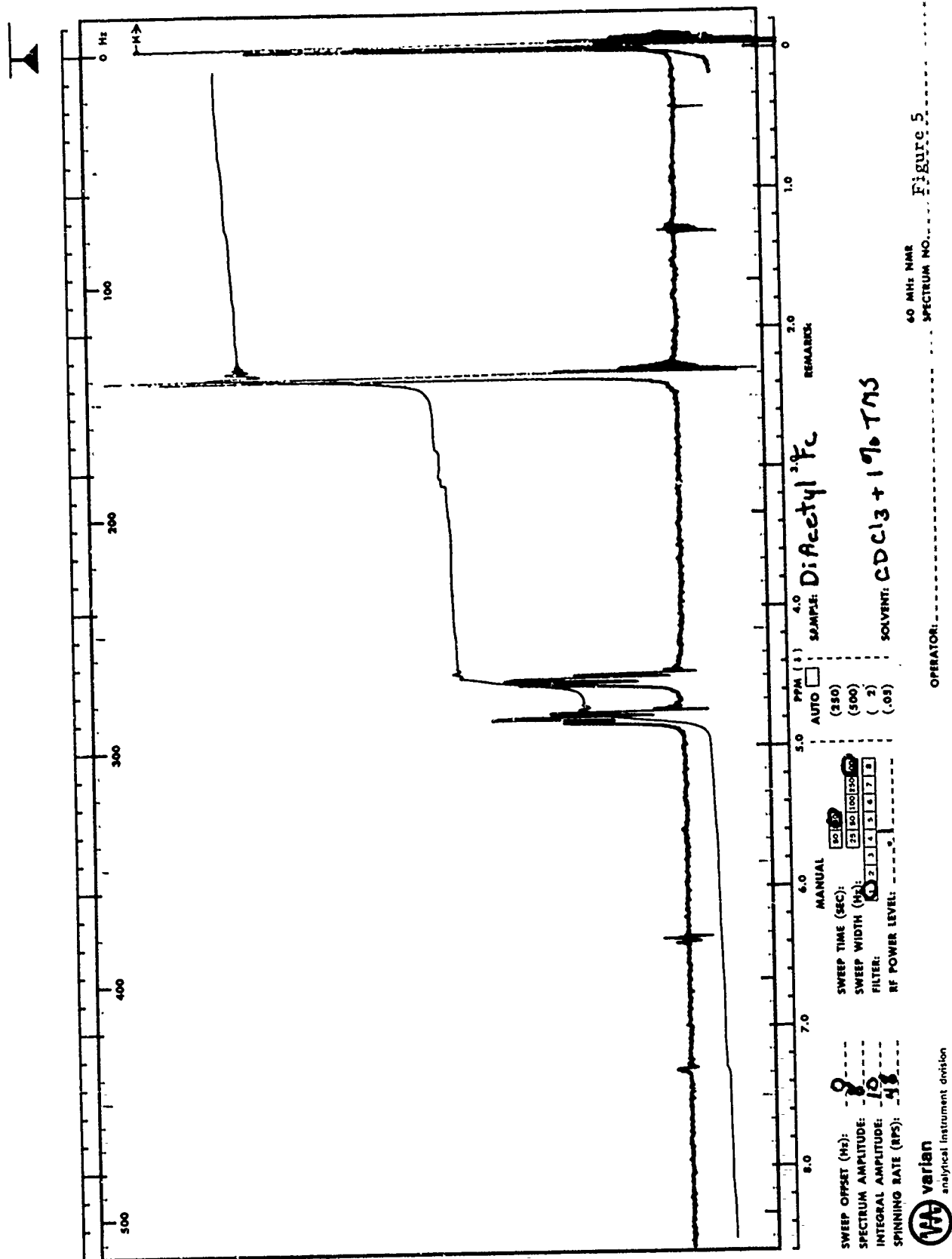


Figure 5. NMR Characteristics of Diacetyl Ferrocene

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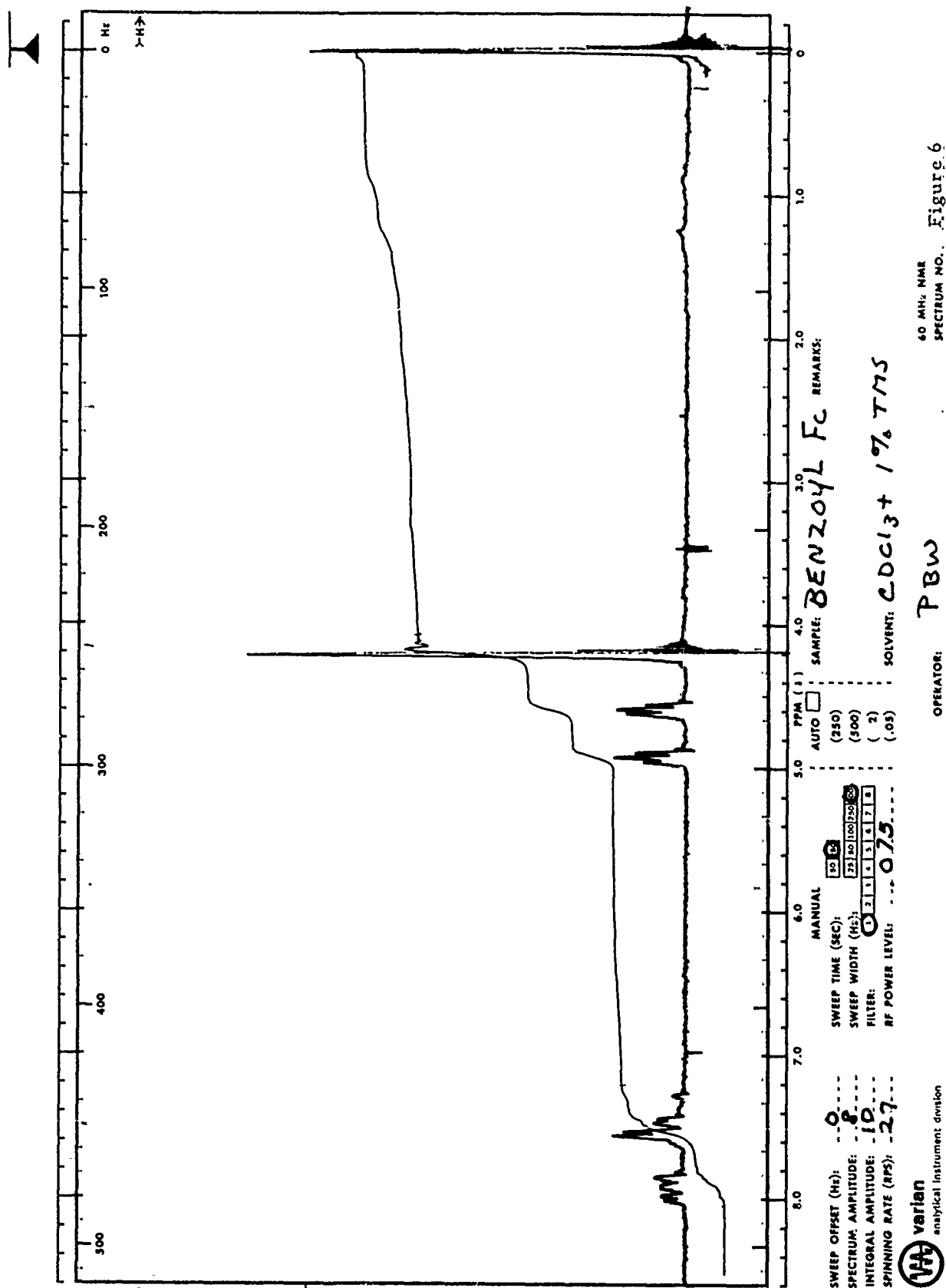


Figure 6. NMR Characteristics of Benzoyl Ferrocene

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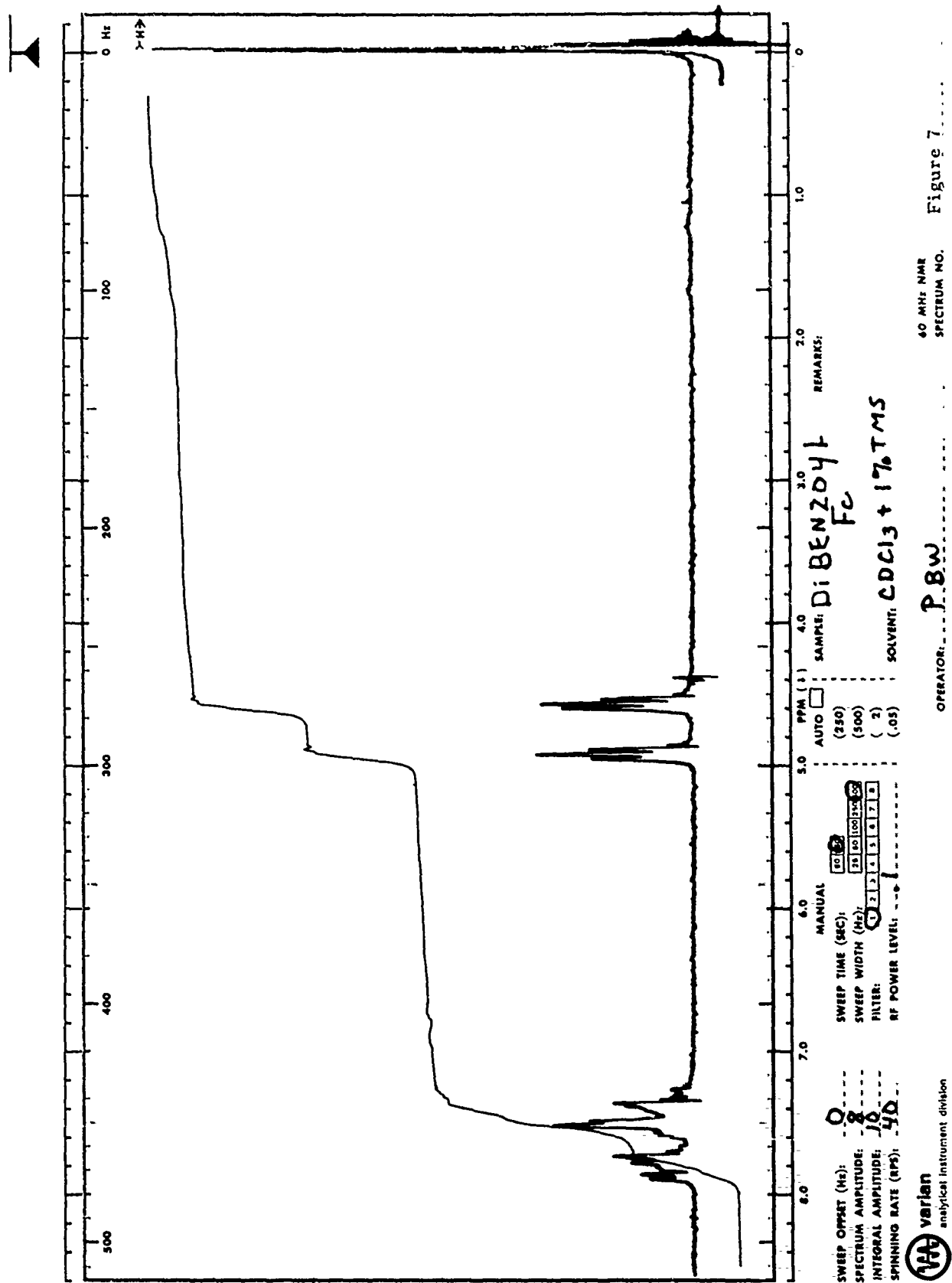


Figure 7

Figure 7. NMR Characteristics of Dibenzoyl Ferrocene

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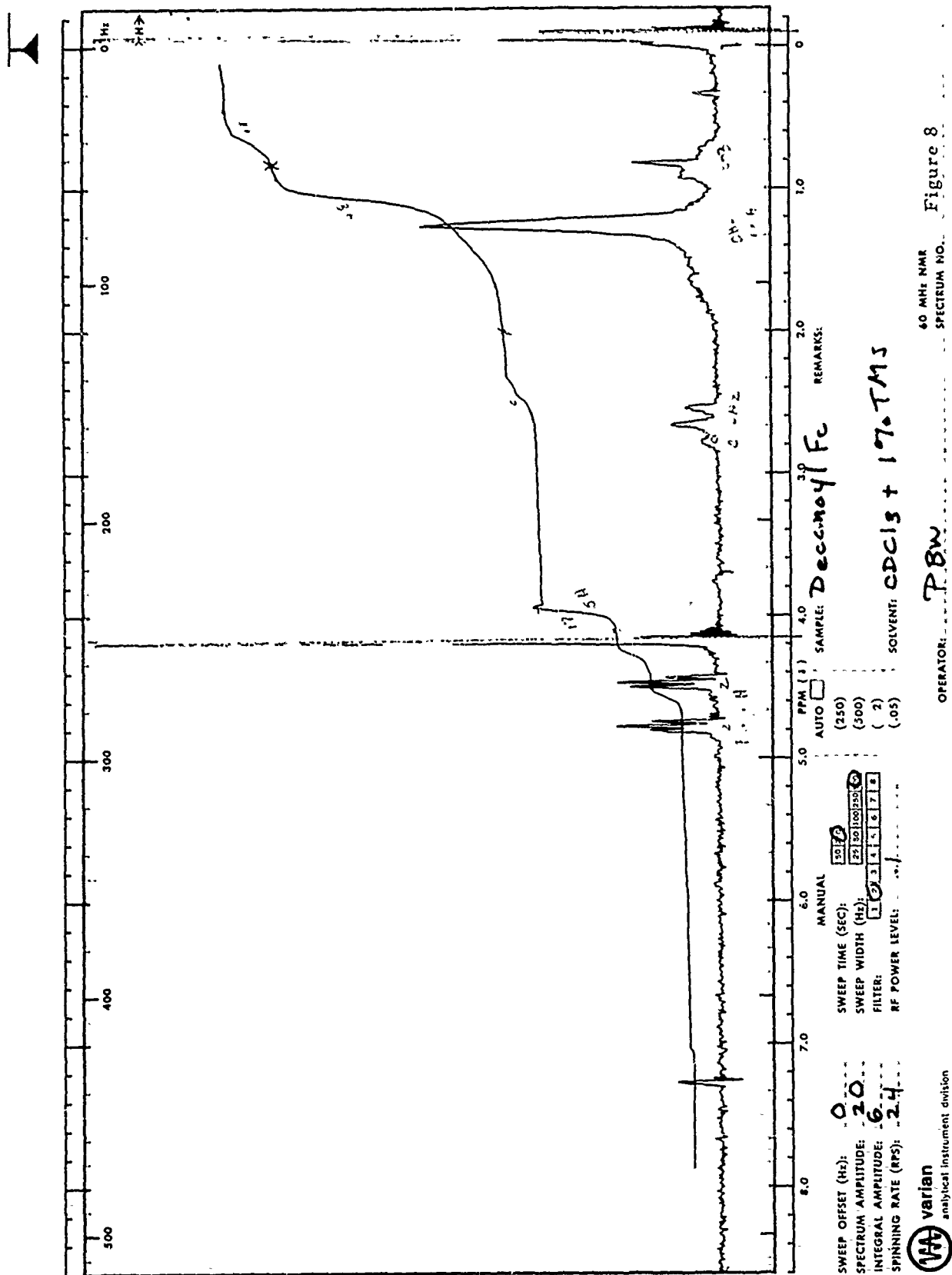


Figure 8. NMR Characteristics of Decanoyl Ferrocene

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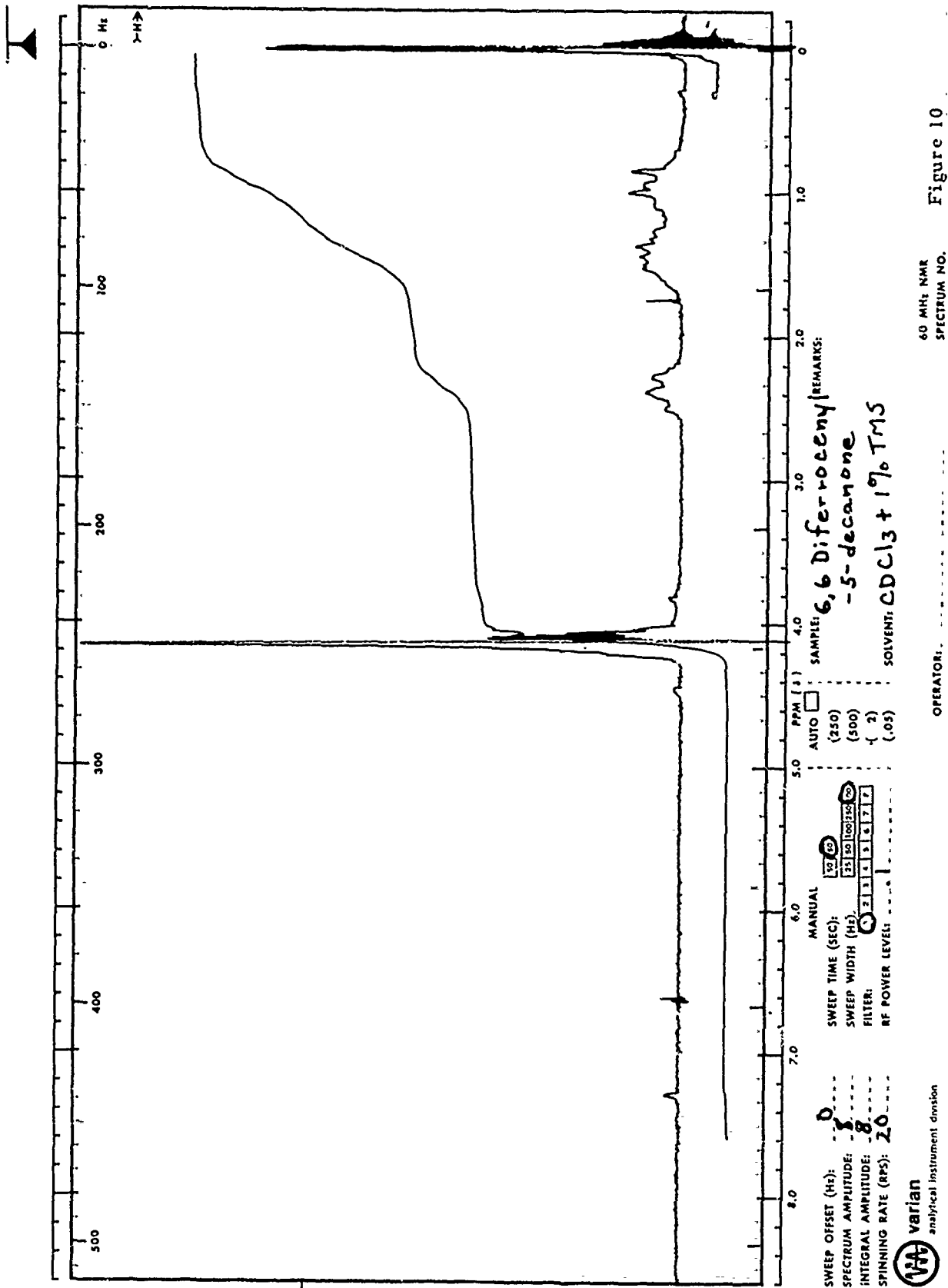


Figure 10. NMR Characteristics of 6,6 Diferrocenyl-5-decanone

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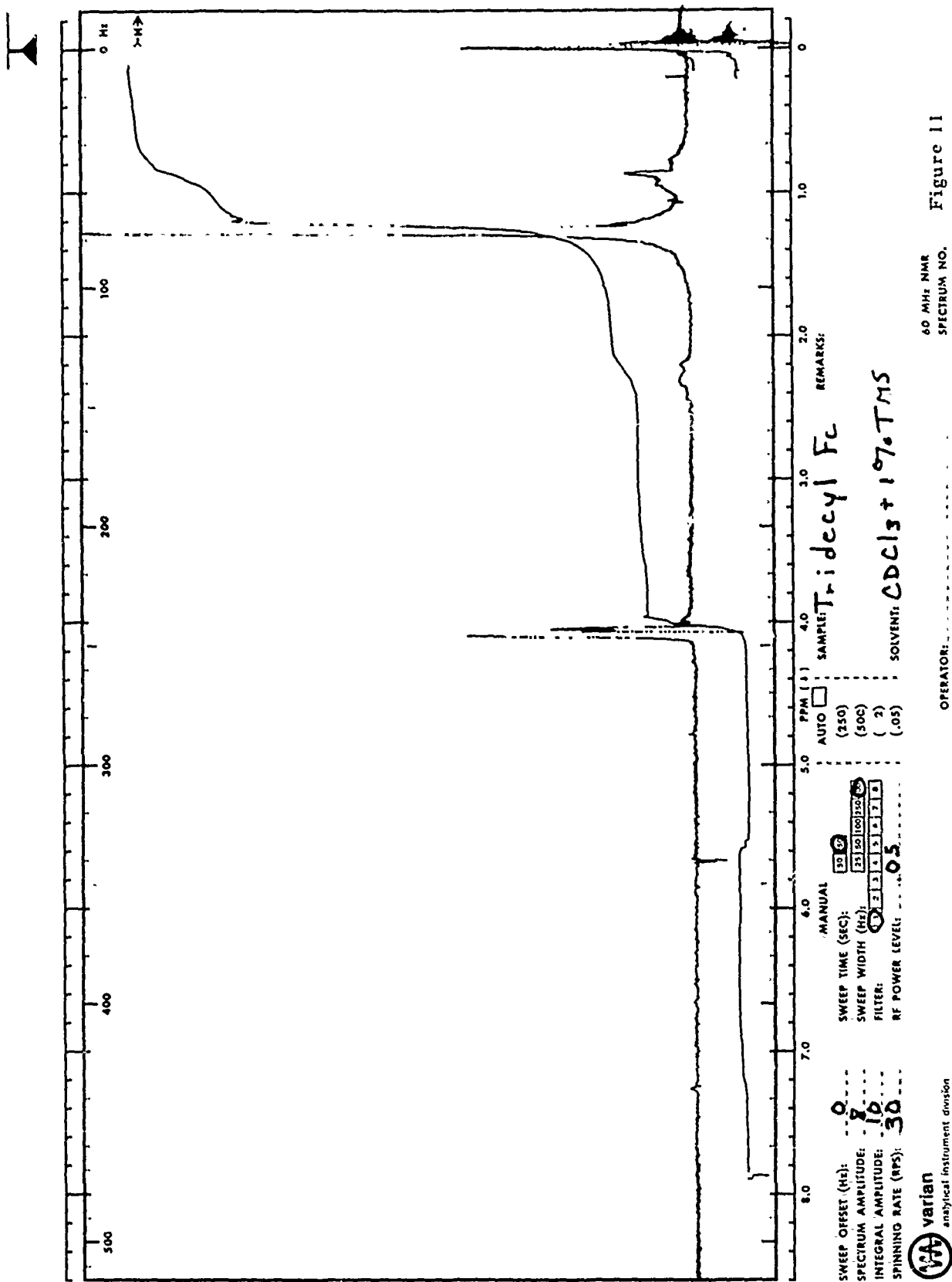


Figure 11. NMR Characteristics of Tridecyl Ferrocene

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