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NMR (NUCLEAR MAGNETIC RESONANCE) INVESTIGATIONS OF
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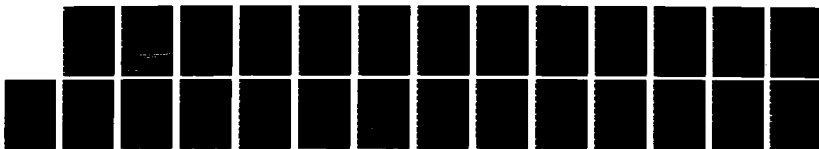
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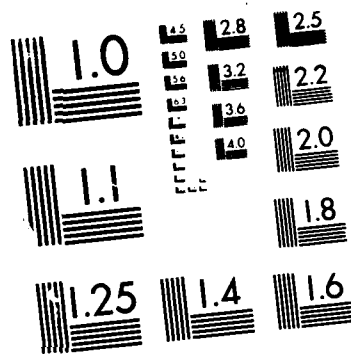
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TECHNICAL REPORT ARAED-TR-86028

**NMR INVESTIGATIONS OF LIQUID PROPELLANT SYSTEMS
CONSISTING OF ALKYL- AND HYDROXYL-AMMONIUM NITRATES**

**PART 1. CHARACTERIZATION OF CARBON-13, PROTON, AND NITROGEN-15
NMR SPECTRA AND ANALYTICAL APPLICATIONS**

S. BULUSU
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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) The proton, carbon-13, and nitrogen-15 (natural abundance) nuclear magnetic resonance (NMR) spectra of four aliphatic ammonium nitrates (AAN's) which could find application as the fuel components in liquid propellant systems and the nitrogen-15 NMR spectrum of hydroxyl ammonium nitrate (HAN) which serves as the oxidizer have been characterized in terms of their chemical shifts. (continued)		

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20. ABSTRACT (Continued)

The utility of NMR spectroscopy for analytical applications has been evaluated. It was found that while the AAN's can be analyzed by using either proton or carbon-13 NMR, NMR spectroscopy cannot be applied readily to the analysis of HAN. Nitrogen-15 NMR at natural abundance may be applied to analyze HAN, but the technique would be too time-consuming.

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INTRODUCTION

Liquid propellants which consist of hydroxyl ammonium nitrate (HAN), an aliphatic ammonium nitrate (AAN) and just sufficient water to keep them fluid over a wide temperature range, have been under investigation for some time to substitute solid propellants in guns (refs 1-4). These "monopropellant" fluids which contain both the fuel and the oxidizer for self-sustained combustion, offer high volumetric impetus and some important advantages of vehicle space utilization and improved mobility. However, impurities associated with the amine nitrates, especially metal ions (e.g., Cu^{2+} and Fe^{3+}) seem to adversely affect their ballistic performance and stability in general, calling for the use of sequestering agents to eliminate the metal ions. Methods of achieving and monitoring the purity and concentration of the liquid propellants during the production and storage are therefore in need of development.

Typically, the liquid propellants contain 80 weight percent or more of salt in aqueous solution and have, therefore, to be considered as extremely concentrated electrolytic solutions which may be compared to molten salts (ref 1) in many properties. The microstructure of these "liquids," that is, the ionic interactions and the coordination chemistry among the various molecular and ionic species present in them are poorly understood, but are of obvious importance to their proper application and prediction of performance. The present nuclear magnetic resonance (NMR) investigation was initiated for the dual purpose of exploring the potential use of NMR spectroscopy for analytical applications and studying the microstructure and ionic interactions mentioned above in the highly concentrated, ionic, liquid propellant systems. This Part I of the report deals with the characterization of the NMR spectra for analytical applications and the Part II addresses the ionic microstructure of the liquid propellants selected for the study.

Three AAN's were previously selected as prime candidates (ref 1) for use as fuels in the HAN-based liquid propellants based on their thermal behavior and detonation susceptibility. These were isopropyl ammonium nitrate (IPAN), trimethyl ammonium nitrate (TMAN), and triethanol-amine nitrate (TEAN). While these three AAN's were the primary focus of this investigation, n-butylammonium nitrate (NBAN) is also included since much information on the carbon-13 spin-relaxation times (T_1 , s) of several salts of n-butylamine relevant to Part II of this investigation, is available in the literature.

Previous analytical approaches (ref 5) to liquid propellants consisted of UV spectrophotometric and conductometric methods for the determination of NO_3^- and hydroxylamine and atomic absorption spectrometry for the parts per million level of transition metal ion impurities. Only a cursory attempt (ref 4) seemed to have been made to apply proton (^1H)-NMR to the analysis of the AAN's. In this report (Part I) the hydrogen-1 and the natural abundance carbon-13 (^{13}C) and nitrogen-15 (^{15}N) NMR spectra are presented with a discussion of their analytical usefulness.

EXPERIMENTAL

All the NMR spectra included in this report were obtained on a Varian Associates XL-200 pulse fourier transform spectrometer equipped with a proton probe and a separate broad-band probe for ^{13}C and ^{15}N . The spectrometer was operated with an internal D_2O lock for proton spectra and an external D_2O lock for the ^{13}C and ^{15}N spectra, respectively.

^1H Spectra

The proton spectra were obtained with approximately 50 mg of the AAN dissolved in 0.5 mL D_2O using 5 mm sample tubes. Typical acquisition parameters were: pulse width (pw) = 6-8 μsec , acquisition time (AT) = 2 sec, D_1 (pulse interval) = 10 sec, and NT (number of transients) $\approx$ 10. The spectra were referenced using 4,4-dimethyl-4-silapentane sodium sulfonate (DDS) ($\delta^1\text{H}$ = 0.015 ppm) as an internal standard.

^{13}C Spectra

For obtaining the ^{13}C spectra 0.5 g each of the AAN's was dissolved in 2.2 mL H_2O contained in a 10 mm NMR sample tube. A 5 mm coaxial tube filled with D_2O was inserted into the 10 mm sample tubes to serve as external D_2O lock. The following acquisition parameters were employed based on an approximate determination of the Ernst angle: pw = 5 μsec , AT = 2 sec, D_1 = 0 and NT $\approx$ 250. Adequate signal to noise ratio can in fact be obtained with a considerably smaller number of transients. The chemical shift scale was calibrated using $\delta^{13}\text{C}$ = 132.1 ppm (relative to TMS) for the aromatic carbons of hexamethyl benzene as an external standard.

^{15}N Spectra

In this preliminary work attempts were made to obtain ^{15}N -NMR spectra at natural abundance. However, good signal-to-noise ratios were difficult to obtain particularly for the NO_3 -nitrogen. Ammonium- ^{15}N signals could be obtained with reasonable signal-to-noise ratios using long (>30 sec) pulse intervals and DMSO-d_6 , as solvent.

RESULTS AND DISCUSSION

Proton (^1H) NMR Spectra

The proton NMR spectra of the AAN's, IPAN, TMAN, TEAN, and NBAN are shown in figures 1 to 4. In common to all the four AAN's, the H_2O peak derived from contamination in D_2O and exchange with the labile protons can be seen at ~ 4.8 ppm. The labile protons attached to the cationic nitrogen would be highly deshielded and may be observed only in a nonaqueous solvent. This fact was independently confirmed but not shown in the figures. The protons attached to the carbons in each of the salts can be readily assigned as indicated in the spectra using the spin-coupling information. All the proton chemical shift (δ , ppm) information is summarized in table 1 for convenience.

Since only one AAN will be present along with HAN in any given liquid propellant system, the use of the above proton peak areas is not expected to present any problem in quantitative analysis. However, HAN cannot be analyzed by this method since all the protons in this structure would be exchangeable and labile. The concentration of the protons attached to the carbons can be measured without interference from HAN.

^{13}C -NMR Spectra

The proton noise decoupled ^{13}C spectra of the four AAN's, IPAN, TMAN, TEAN, and NBAN, are shown in figures 5 and 6, respectively. In these spectra each chemically distinguishable carbon gives rise to a separate line readily assignable on the basis of the well known shielding and inductive effects. The assignments are indicated in the figures.

It may be pointed out that the peak intensities in carbon-13 NMR spectra do not necessarily bear an exact relationship to the number of carbons of each type in the structure. This is due to the interplay of two effects. Firstly, the large and widely varying relaxation (T_1) times of different carbon-13 nuclei compared to the protons, requiring different pulse widths and pulse intervals to avoid peak saturation. Secondly, the inherent low sensitivity of carbon-13 nucleus (1.7×10^{-4} relative to ^1H) commonly requires proton noise decoupling to enhance the sensitivity. The enhancement in sensitivity, up to a maximum of 3 times, is brought about by nuclear overhauser effect (NOE). These two effects make it often difficult to relate the peak intensities in a spectrum to the number of each type of carbon atoms in the structure under investigation. However, the peak intensity of any given carbon in the structure chosen for quantitative analysis can be optimized to maintain a linear relationship to the concentration of that substance in a fixed volume of solution. This is illustrated in figure 7 representing a plot of concentration versus peak height for the primary and the tertiary carbons in IPAN and in figure 8 for the carbons of TEAN and TMAN. For peaks which are considerably broader peak areas are preferable for quantitative analysis. This relationship was again found to be unaffected by the presence of HAN.

The proton noise decoupling which gives rise to the NOE also results in a simplification in the carbon-13 spectra which can be difficult to interpret otherwise. The strong proton couplings, such as those illustrated in figure 9 for IPAN, give rise to a multitude of overlapping lines in more complex structures.

¹⁵N-NMR Spectra

Nitrogen-15 NMR spectra at natural abundance (~0.35%) are often difficult to obtain especially when the spin-lattice relaxation times are long. The AAN's studied thus far gave poor signal-to-noise ratios even with pulse intervals of the order of 30 sec. Detectable signals from the ammonium nitrogen can be observed more readily owing to their shorter relaxation times resulting from the attached protons. The natural abundance ¹⁵N spectra of ¹⁵NH₄⁺, ¹⁵NO₃⁻, IPAN, TMAN, TEAN, and NBAN are collected together in figure 10. The acquisition parameters are described in the respective figure captions. The ¹⁵N- chemical shifts of the ammonium nitrogens in the AAN's taken from these spectra are summarized below. In these spectra, ¹⁵NH₄⁺, ¹⁵NO₃⁻, IPAN, TEAN, and NBAN show ¹⁵NO₃⁻ absorptions also in addition to the ammonium nitrogen lines. The higher noise levels in some of the spectra can be readily improved by accumulating more transients. However, because of the long delay times required and the NOE effects on nitrogen absorptions it would be impractical to use these spectra for purely analytical applications.

AAN in DMSO-d ₆ Solution	$\delta(^{15}\text{N}^+)$, ppm from NO ₃ -nitrogen
IPAN	-328.8
TEAN	-330.3
TMAN	-345.3
NBAN	-343.3
HAN	-292.5

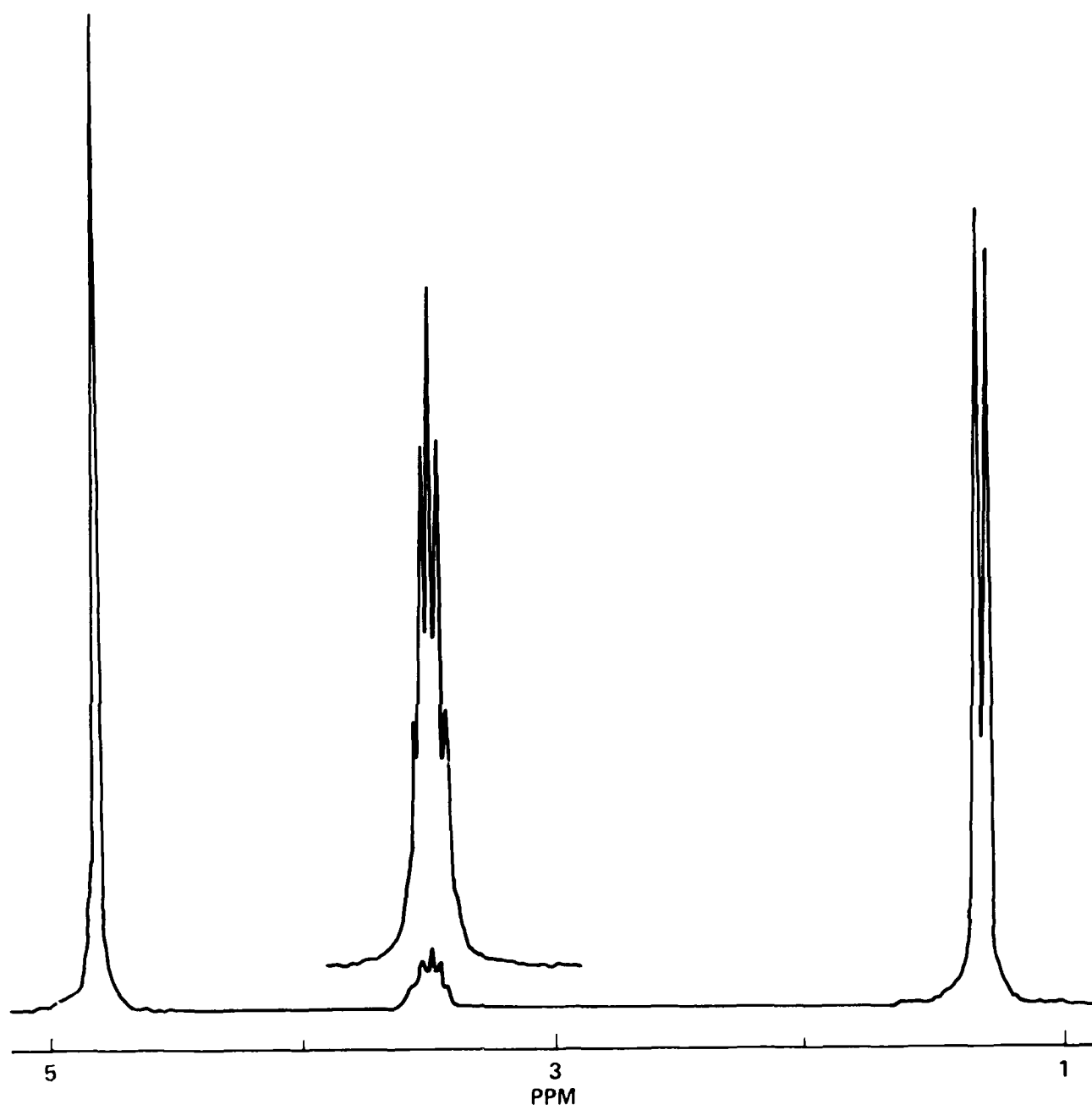
Attempts will be made in the future to synthesize the AAN's enriched with ¹⁵N in both ammonium and nitrate nitrogens in order to characterize the N-15 spectra better and investigate the spin-lattice relaxation times (see Part II of this report).

CONCLUSIONS

The ¹H and ¹³C NMR spectra of the four AAN's studied may be readily adapted for the analysis of these materials in the liquid propellant systems. The natural abundance ¹⁵N spectra of the AAN's and the HAN, however, take typically 15 to 20 hours each, to yield reasonable signal-to-noise ratios and hence are judged impractical for analytical applications. It is concluded, therefore, that the HAN cannot readily be analyzed using NMR spectroscopy while the AAN's can be analyzed by either ¹Hp or ¹³C-NMR.

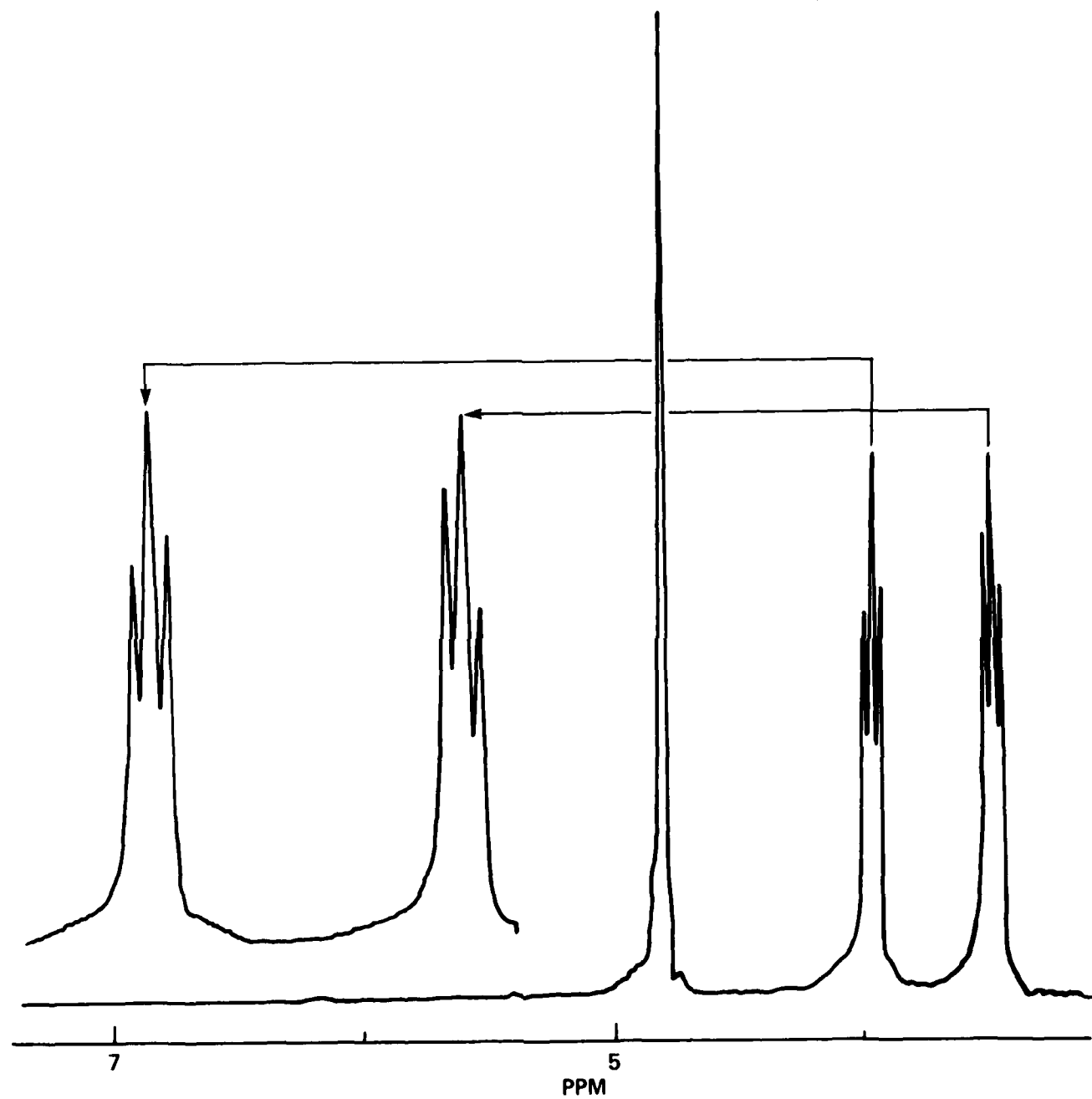
Table 1. Summary of the proton chemical shifts δ of the AAN's:
IPAN, TMAN, TEAN, and NBAN

	$\delta^1\text{H}$, ppm (TMS = 0)	Hz
TEAN in D ₂ O		
Downfield triplet	3.99	789
	3.97	793
	3.94	788
Upfield triplet	3.52	704
	3.49	699
	3.47	694
H ₂ O in D ₂ O	4.79	959
DSS	0.02	3
IPAN in D ₂ O		
H ₂ O in D ₂ O	4.81	963
Center of septet	3.50	699
CH ₃ doublet	1.32	253
	1.28	257
TMAN in D ₂ O		
H ₂ O in D ₂ O	4.88	976
CH ₃	2.88	576
NBAN in D ₂ O		
H ₂ O in D ₂ O	4.77	955
¹ C-triplet	2.98	596
² C-quintet	1.62	324
³ C-center doublet	1.38, 1.34	276, 268
⁴ C-triplet	0.90	181



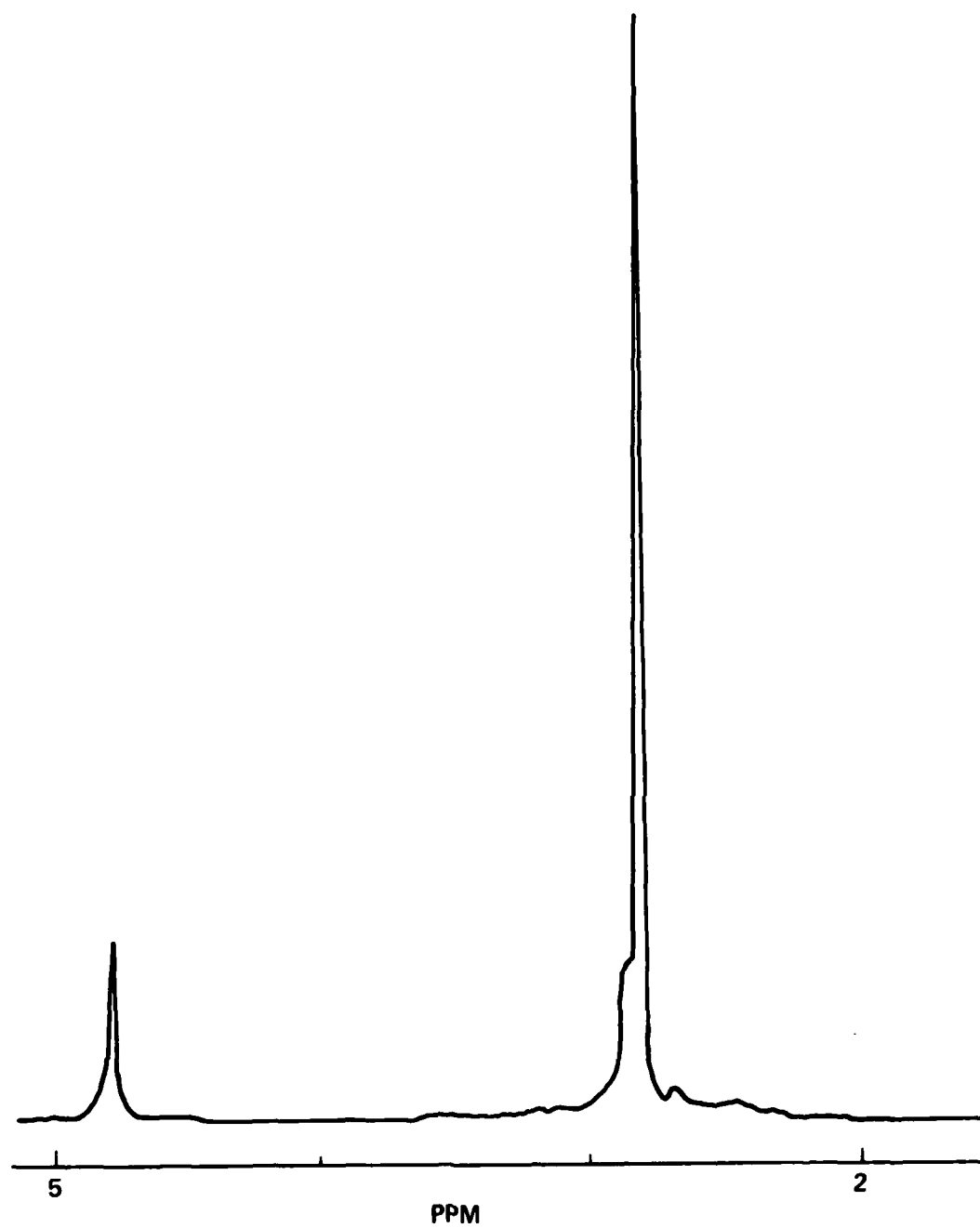
^1H -NMR Spectrum
 $(\text{CH}_3)_2\text{CH-NH}^+\text{NO}_3^-$

Figure 1. ^1H -NMR spectrum of IPAN in D_2O solution



^1H -NMR Spectrum
 $(\text{HOCH}_2\text{CH}_2)_3\text{NH}^+\text{NO}_3^-$

Figure 2. ^1H -NMR spectrum of TMAN in D_2O solution



^1H -NMR Spectrum
 $(\text{CH}_3)_3\text{NH}^+\text{NO}_3^-$

Figure 3. ^1H -NMR spectrum of TEAN in D_2O solution

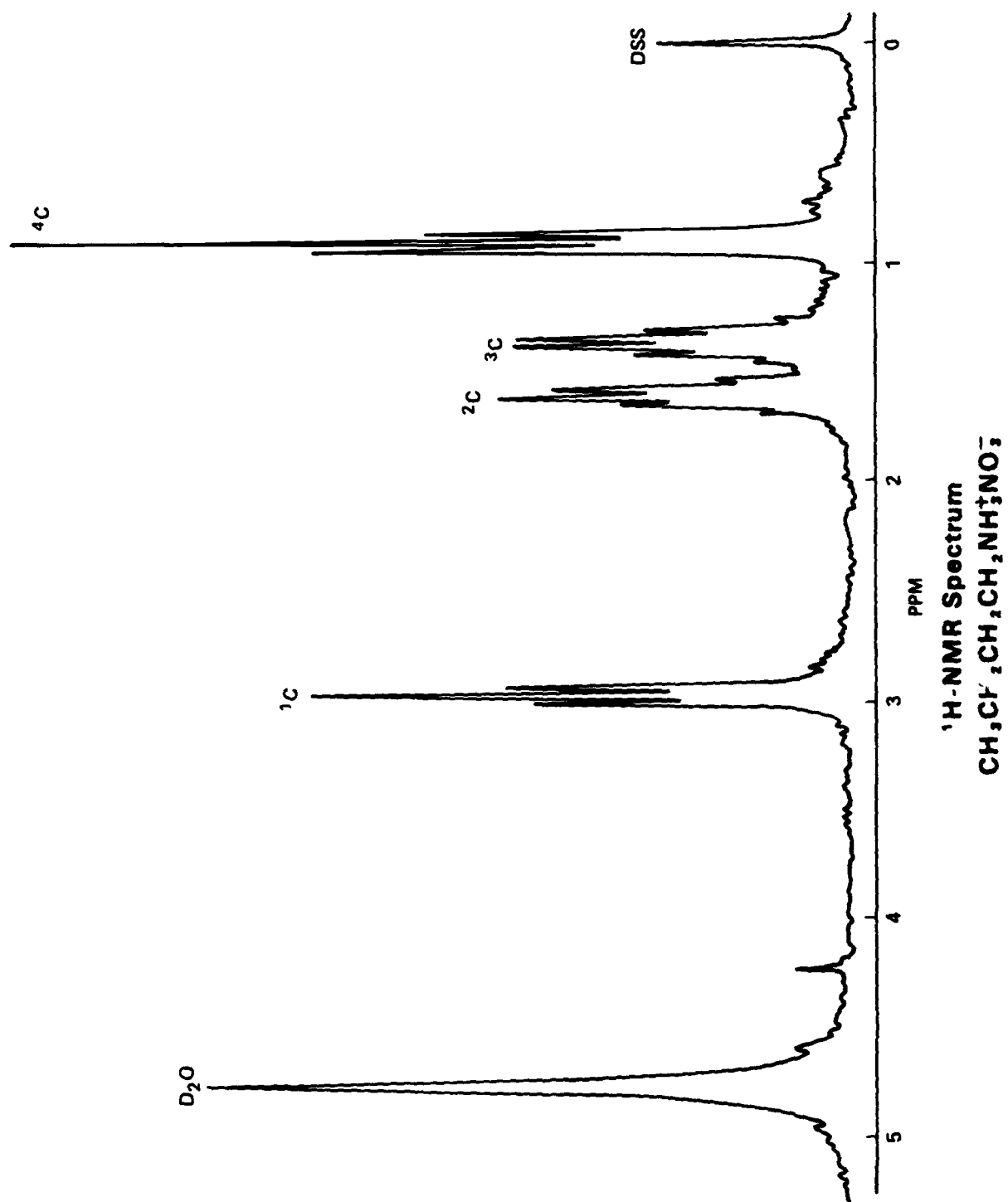
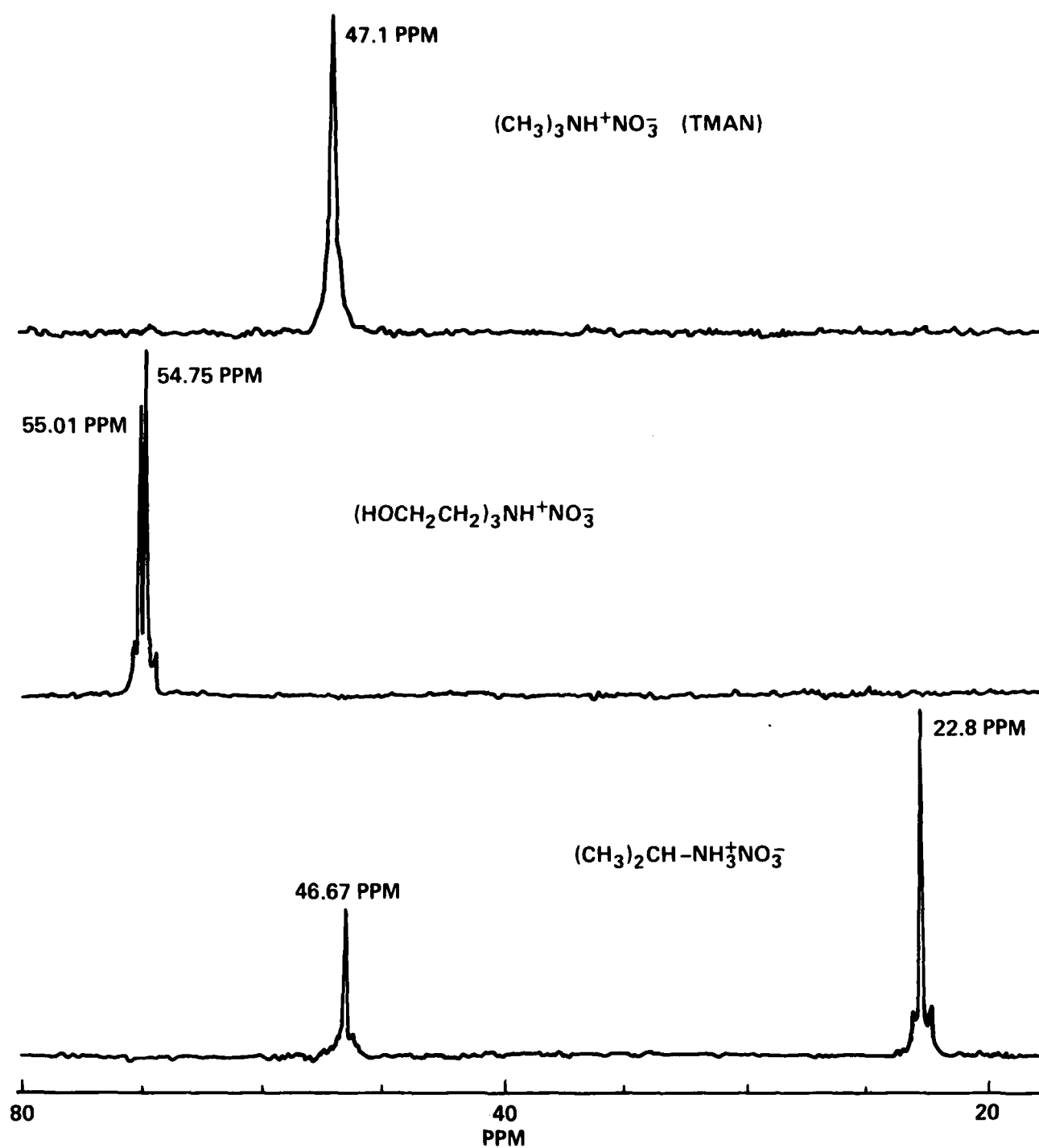
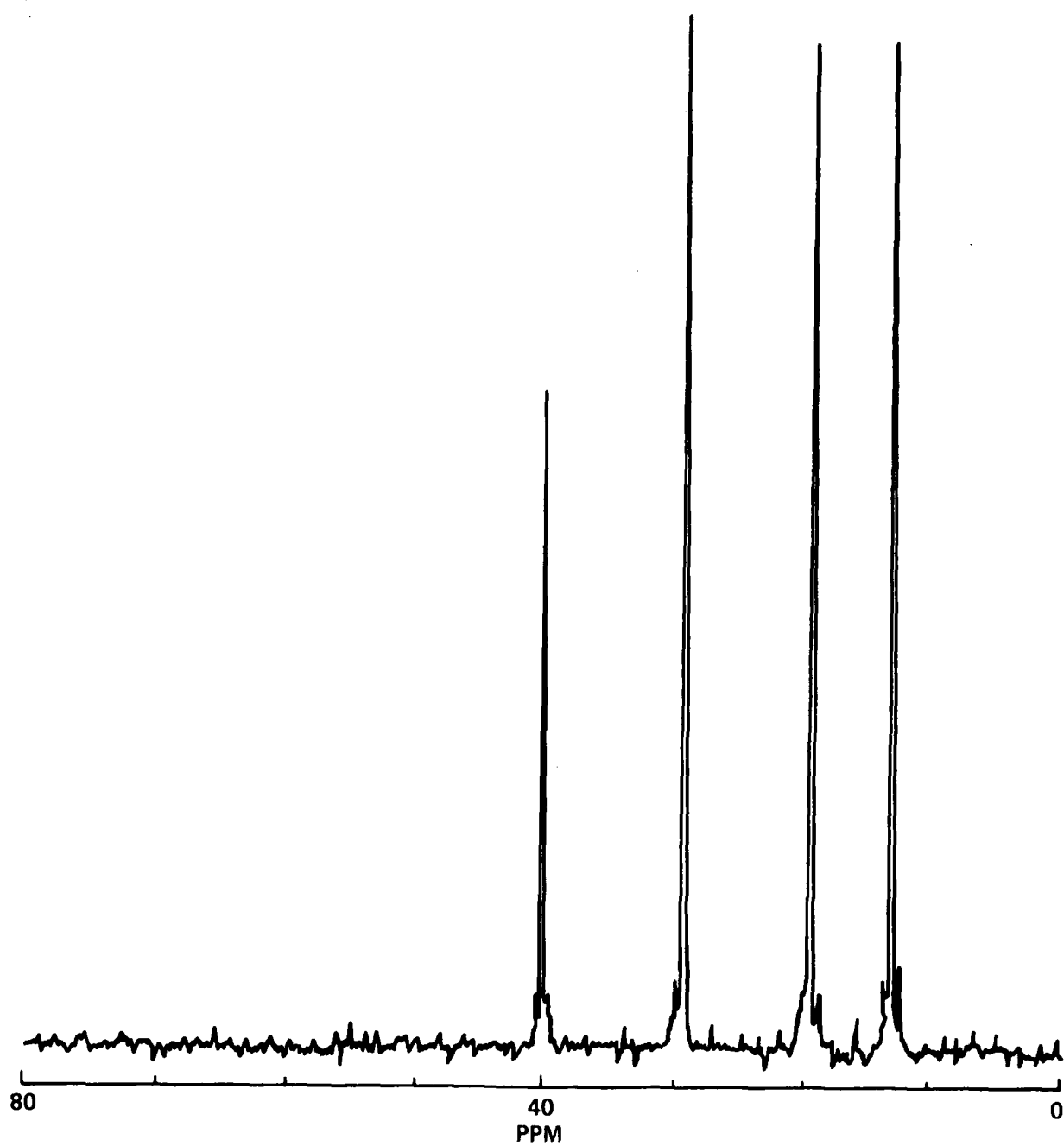


Figure 4. ^1H -NMR spectrum of NBAN in D_2O solution



^{13}C -NMR Spectra

Figure 5. Carbon-13 NMR spectra of aqueous solutions of TMAN (1.8 moles/l), TEAN (1.07 moles/l), and IPAN (1.8 moles/l)



^{13}C -NMR Spectrum
 $\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{NH}_3^+\text{NO}_3^-$

Figure 6. Carbon-13 NMR spectrum of NBAN in H_2O

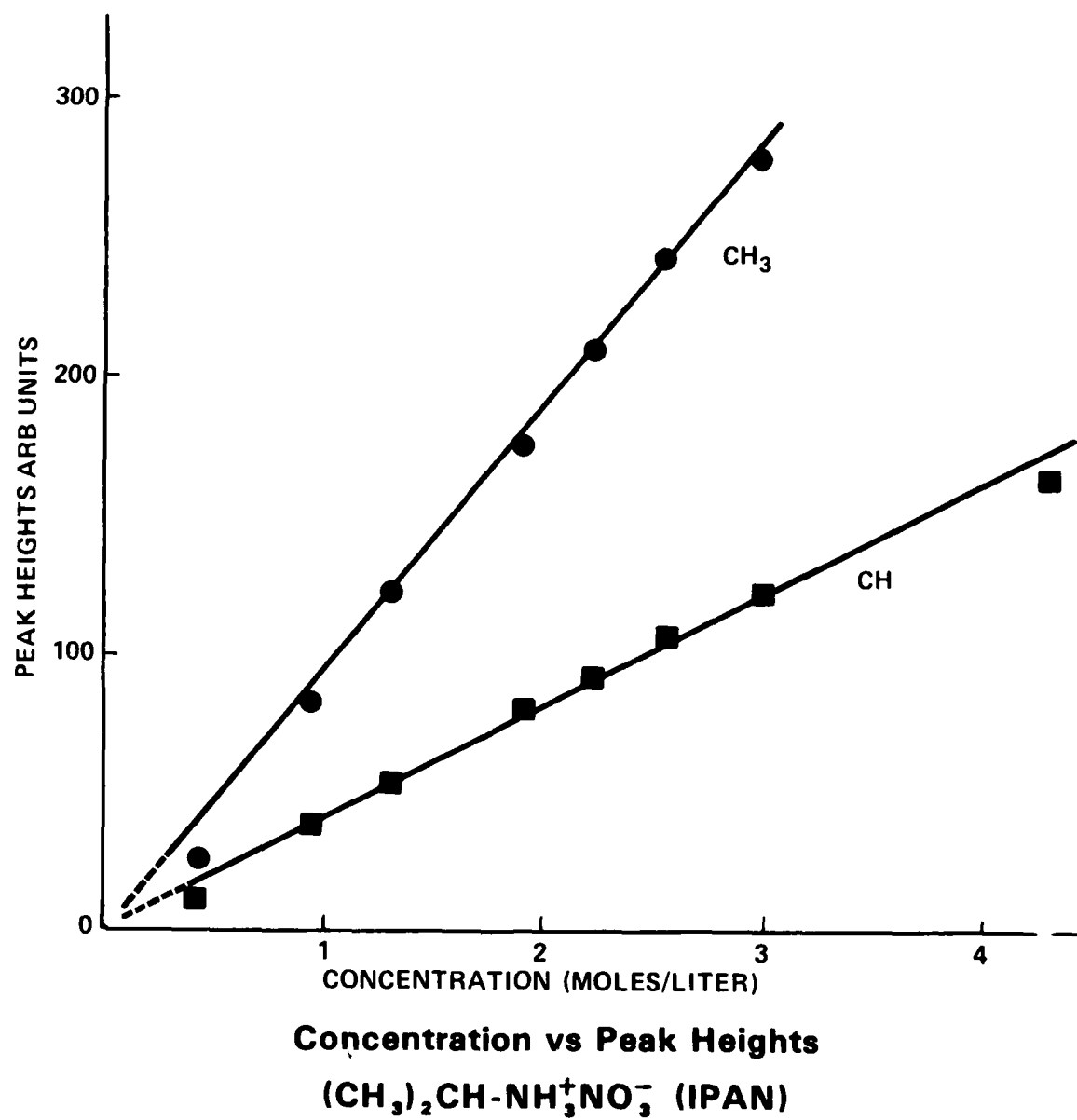


Figure 7. Plots of concentration versus peak heights for IPAN

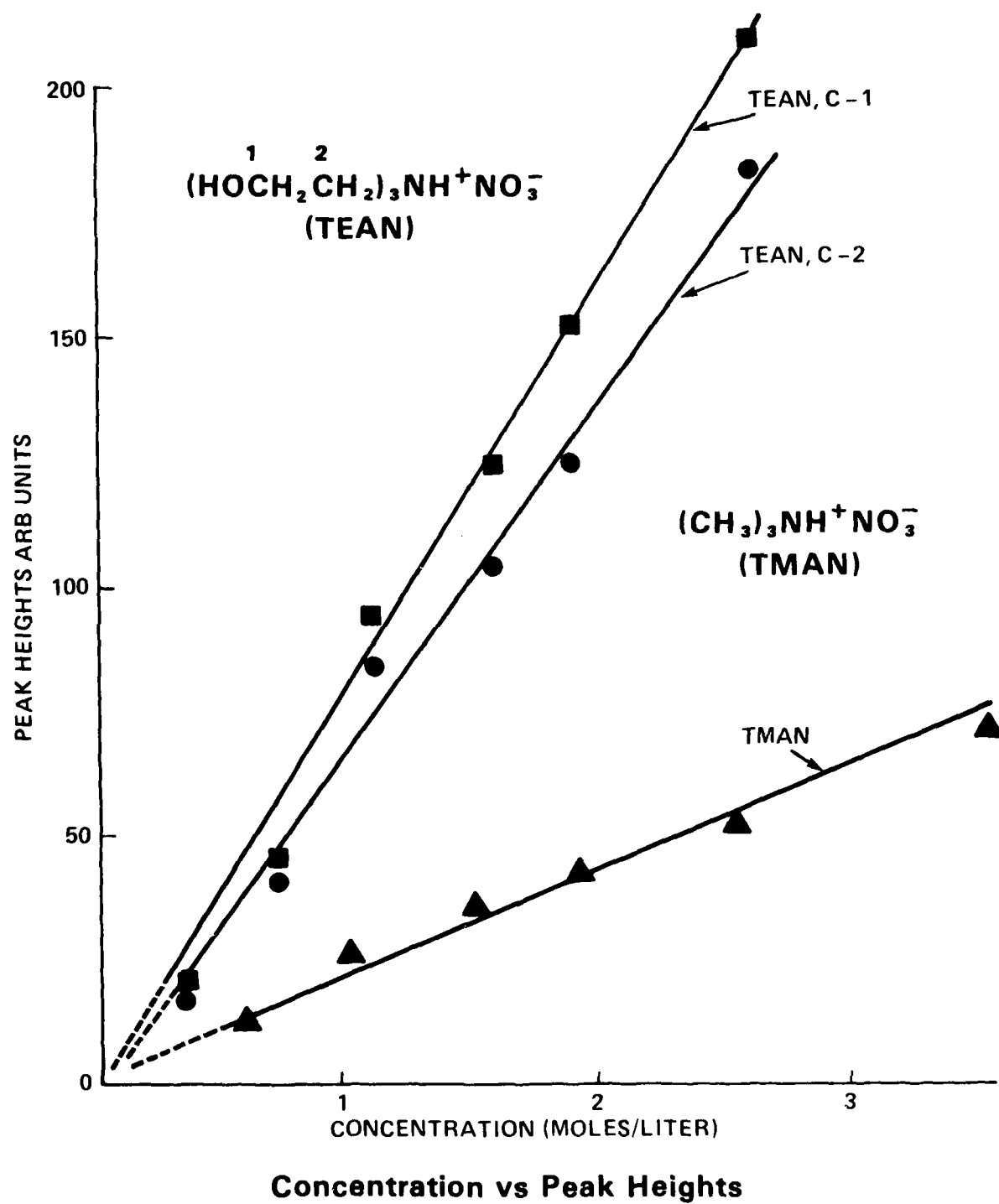
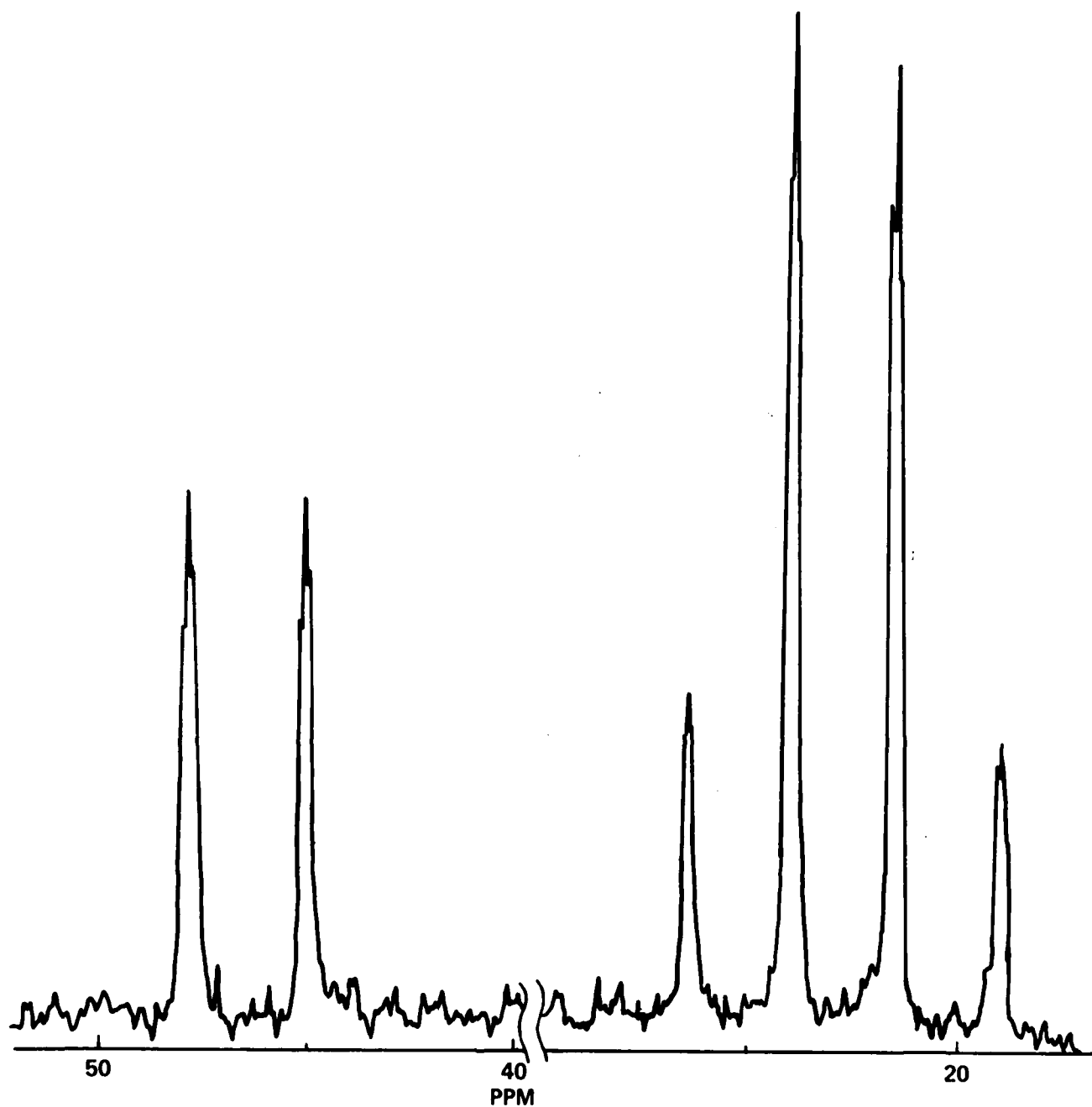


Figure 8. Plots of concentration versus peak heights for TEAN and TMAN



^1H -Coupled ^{13}C -Spectrum of IPAN
 $(\text{CH}_3)_2\text{CHNH}_3^+\text{NO}_3^-$

Figure 9. ^1H -coupled C-13 spectrum of IPAN. $J(^{13}\text{C}-^1\text{H})$: CH_3 , 125.8 Hz;
 CH , 143.3 Hz

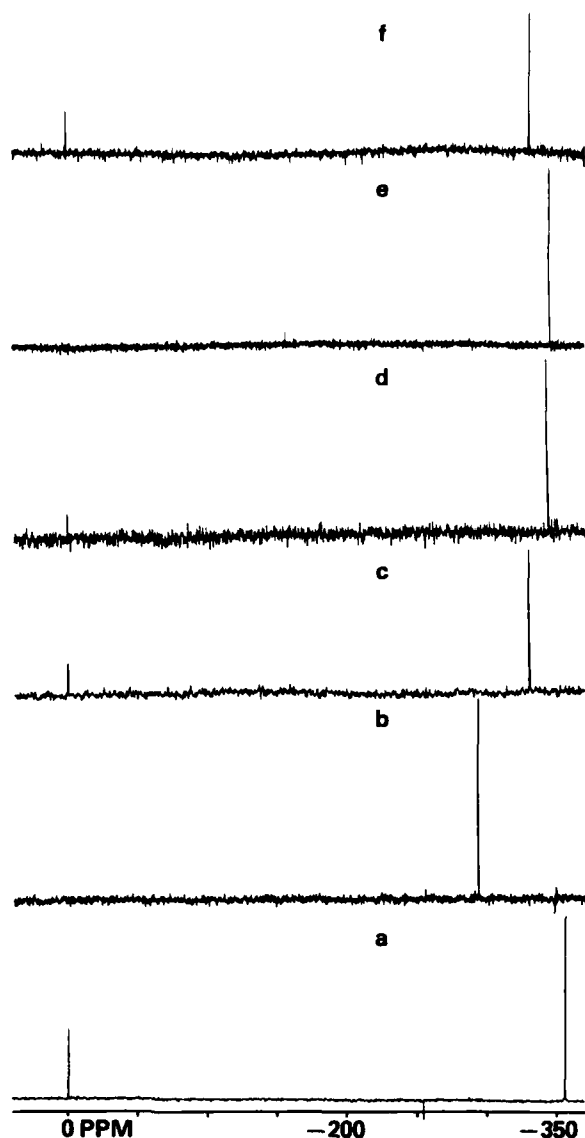


Figure 10. Proton decoupled nitrogen 15 NMR spectra of: A) Nitrogen-15 labeled AN in H_2O with a D_2O lock. Delay time (D_1) 60 secs; number of transients, (NT) 16; acquisition time (AT), 0.5 sec. B) Hydroxylammonium nitrate (80%) in H_2O with a D_2O lock. D_1 , 15 secs; NT, 500; AT, 0.5 sec. C) Isopropyl AN in DMSO with a D_2O lock, D_1 , 10 secs; NT, 4456; AT, 0.5 secs. D) n-butyl AN in DMSO with a D_2O lock, D_1 , 10 secs; NT, 3900; AT, 0.5 secs. E) Triethanol AN in DMSO with a D_2O lock. D_1 , 15 secs; NT, 5000; AT, 0.5 secs. F) Trimethyl AN in DMSO with D_2O lock. D_1 , 15 secs; NT, 15, 224; AT, 0.5 secs.

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