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Newsletter

No. 168

September, 1972

BRUKER SCIENTIFIC INC.

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Deadline Dates: No. 169: 2 October 1972 No. 170: 6 November 1972
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All Newsletter correspondence, etc. should be addressed to:

Bernard L. Shapiro  
Department of Chemistry  
Texas A&M University  
College Station, Texas 77843

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## THE UNIVERSITY OF BRITISH COLUMBIA

VANCOUVER 8, CANADA

DEPARTMENT OF CHEMISTRY

7 August, 1972

Professor B. L. Shapiro  
Department of Chemistry  
Texas A & M University  
College Station, Texas 77843 USA

Dear Barry,

IMPROVED TRANSISTORIZATION OF VARIAN HA-100 POWER SUPPLY

In common with Dr. C. A. Reilly (TAMU 166-29), we recently replaced our 304TL tubes in our HA-100 V2100B power supply with transistors according to the recipe of Pearson (Rev. Sci. Inst. 42, 713 (1971)), and we also experienced blowout of all five 1N3340 Zener diodes. Our treatment of the malady differs somewhat from that of Dr. Reilly, however, and may be of interest to your readers.

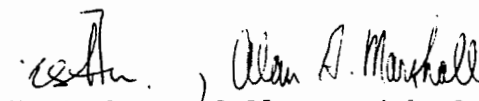
In the Pearson modification, when a high voltage suddenly appears across the Zeners, they will conduct heavily with only 5X20 Ohm to limit the current. Current easily climbs over 500 mA in a very short time (much faster than the normally slow-acting K1 relay) thus fusing off the Zeners before K1 has a chance to be energized. Mr. K. S. Au has modified the circuit to act as a fast-acting SCR clamp (see diagrams), set to operate at about 620V, protected by 2x1N1347A (or any 1 KV 5A diode) against induced surge voltage. As voltage across it goes over 620V, it clamps; to reset it, one has to decrease the voltage variac, switch H.T. off and turn it back on again.

At a magnet current of 2A, ripples across the DTS702's are measured as about 400V<sub>pp</sub>. Hence an operating voltage of 250V is barely sufficient, and larger d.c. shifts are not regulated. As a remedy, one could either increase the operating voltage or reduce the ripples; we chose the latter course, by adding an extra 4 MFD capacitor and a 0.2H choke. Ripples are now measured as about 60V<sub>pp</sub>, and operating voltage may now be lowered even to 200V.

In order to correct for poor regulation (it refused to lock with the V3506 super-stabilizer) a 0.05 MFD capacitor across R 754 and R 755 was added. To optimize the chopper amplifier regulation range, a range adjust control (50 K in series with 22 K, as shown in circuit diagram) was added.

This power supply is now in operation, with very little heat generated. Transient protection has proved to be satisfactory--last month we had water and power failures which resulted in switching-off without damage to any semiconductor device.

Sincerely,

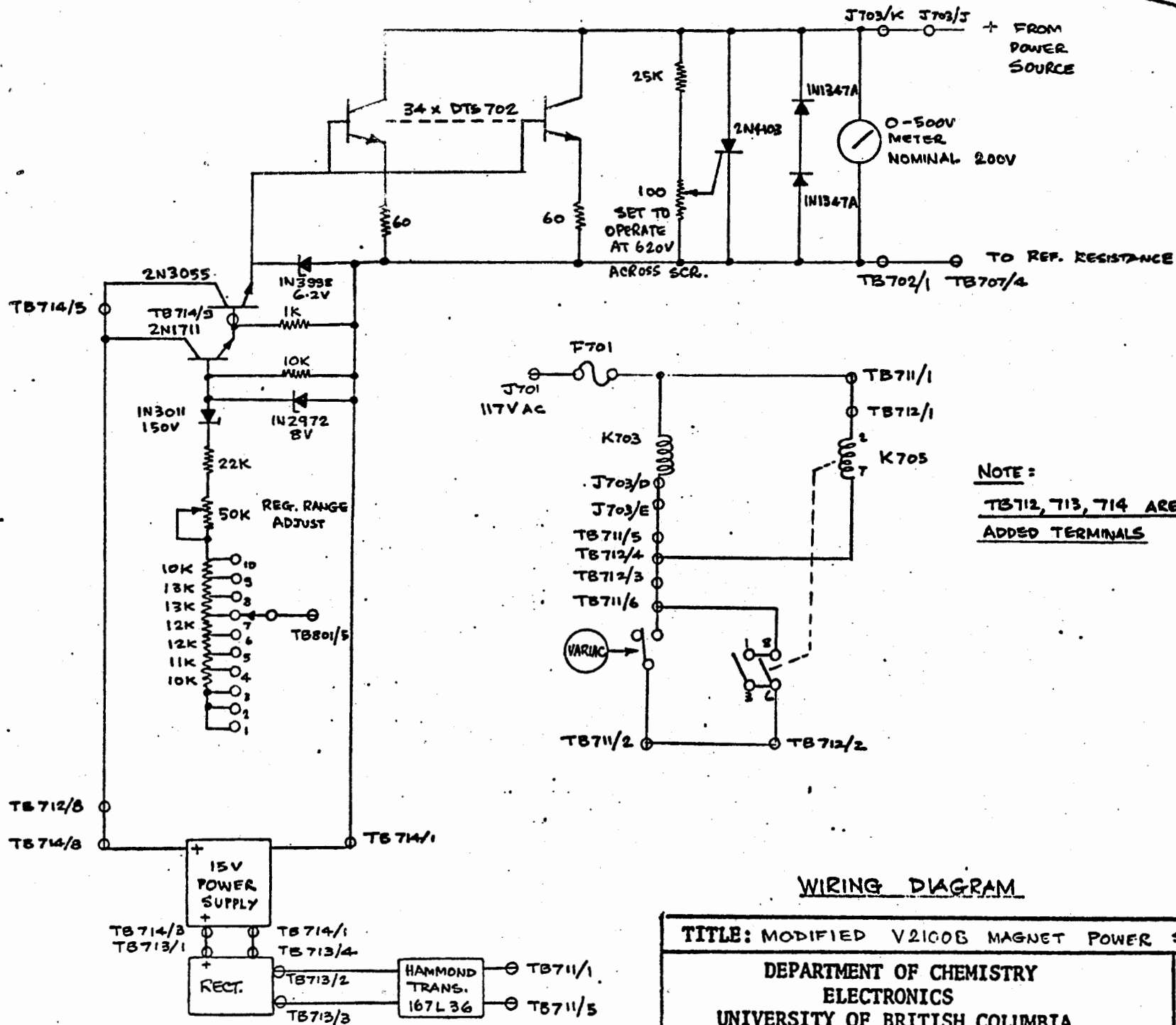


K. S. Au, J. Sallos, and A. G. Marshall\*

\* to whose subscription this contribution should be relegated.





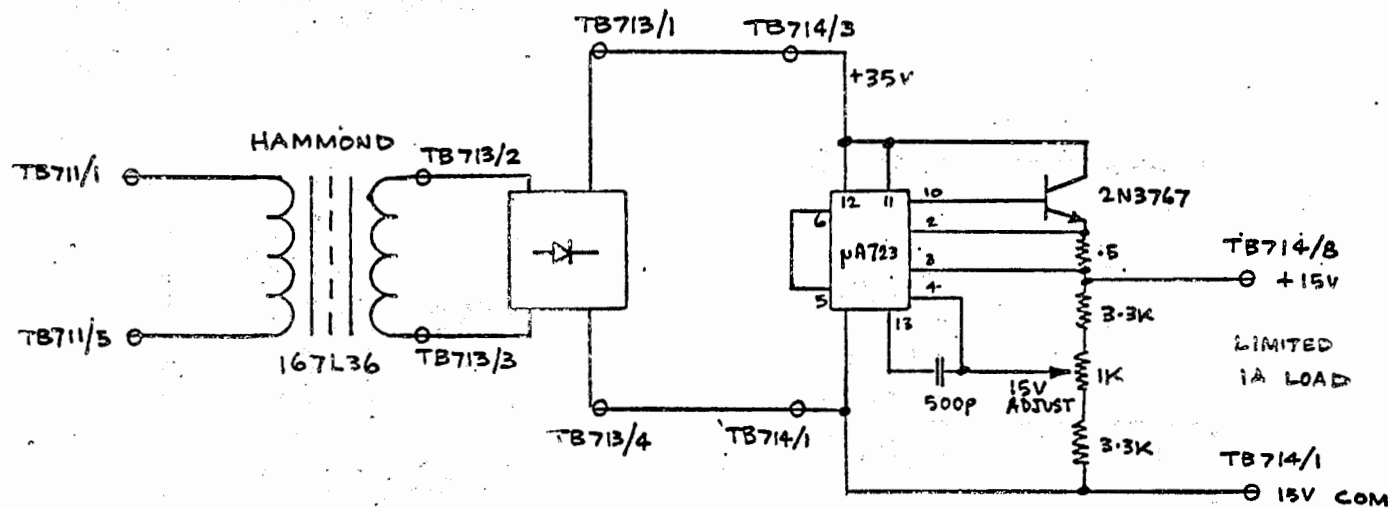


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מחלקת איזוטופים

Prof. Bernard L. Shapiro  
TAMU NMR Newsletter  
Department of Chemistry  
Texas A & M University  
College Station, Texas 77843  
USA

August 22nd 1972

Shift Reagent Sessions at the 10th

Rare Earth Research Conference.

Dear Professor Shapiro:

The 10th Rare Earth Research Conference, chaired by Professor Therald Moeller (Department of Chemistry, Arizona State University, Tempe, Arizona 85281), will take place at Carefree, Arizona, April 30 - May 3, 1973, and for the first time will include sessions devoted to Lanthanide Shift Reagents. Intended to provide a forum to present the advances and resolve the controversies in the field, these sessions will include short introductory talks (20-30 mins each) followed by longer discussions (30-40 mins each) on the following topics:

Pseudocontact and Contact Contributions to Lanthanide Induced Shifts.  
Molecular Structure Elucidation Using Induced Shifts  
Chiral Shift Reagents  
Applications of Shift Reagents to Natural Products  
Physicochemical Studies and Chemical Aspects (Central Ions, Stoichiometry, Functional Groups, Isotope Effects)

In addition to the regular sessions and depending upon the interest round the table, discussions will be arranged.

Those wishing to participate should write to the Conference Chairman. Short (one typewritten page) abstracts on subjects related to the above topics should be sent to me before November 15, 1972. The full papers will be published in the Conference Proceedings and special forms will be provided in due course.

Sincerely yours,

*Jacques Reuben*  
Jacques Reuben

JR:dr

Enc.

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Professor Bary L. Shapiro  
 Dept. of Chemistry  
 Texas A & M University  
 College Station, Texas 77843

August 7th, 1972

Dear Bary,

 **$^{17}\text{O}$  Fourier Transform Spectroscopy**

In our Elmsford application laboratory, we have recently been quite successful in applying Fourier-Transform techniques to that neglected nuclei  $^{17}\text{O}$ .

$^{17}\text{O}$ , with a natural abundance of only 0.037%, spin of  $5/2$  and the low resonance frequency 12.2 MHz at 21.1kGauss, has always been difficult to study. Only wide-line techniques have been used and often with  $^{17}\text{O}$  enriched compounds.

In the FT mode, we typically collected data in about 15 minutes, and with the system field/frequency locked on protons, the spectra are easily computer calibrated.

Included are some spectra obtained here recently. Neat liquids were used and about 2 to 3 K of scans were required to obtain the results.

**EXPERIMENTAL DATA :**

Tube size : 13 mm

Sweep width : 10,000 Hz

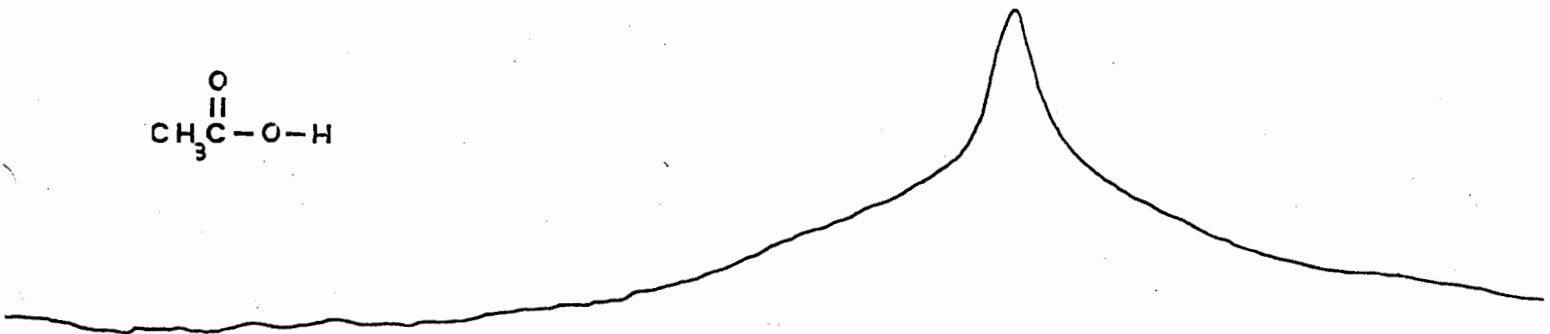
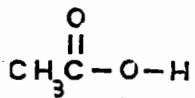
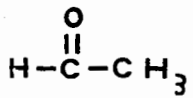
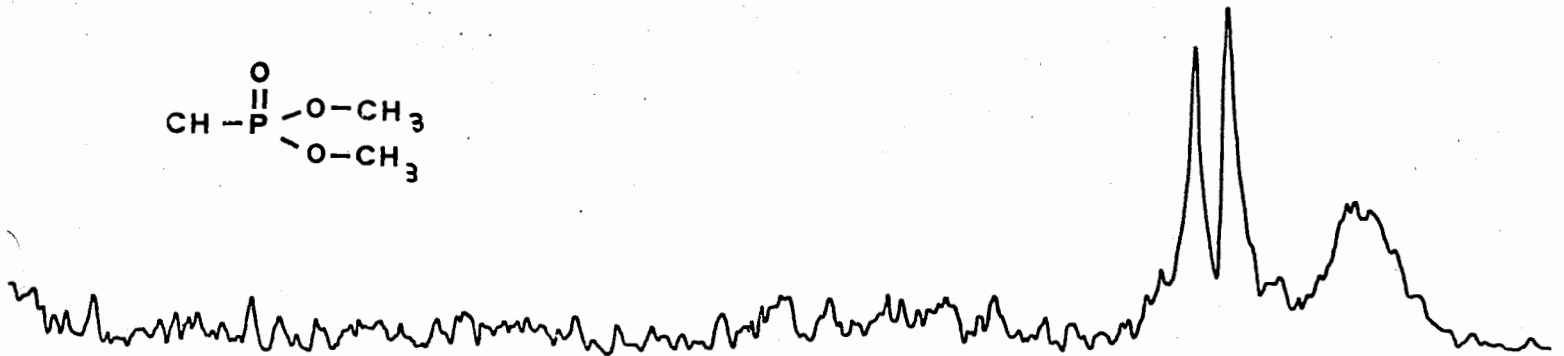
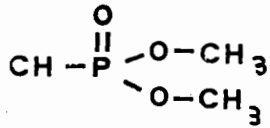
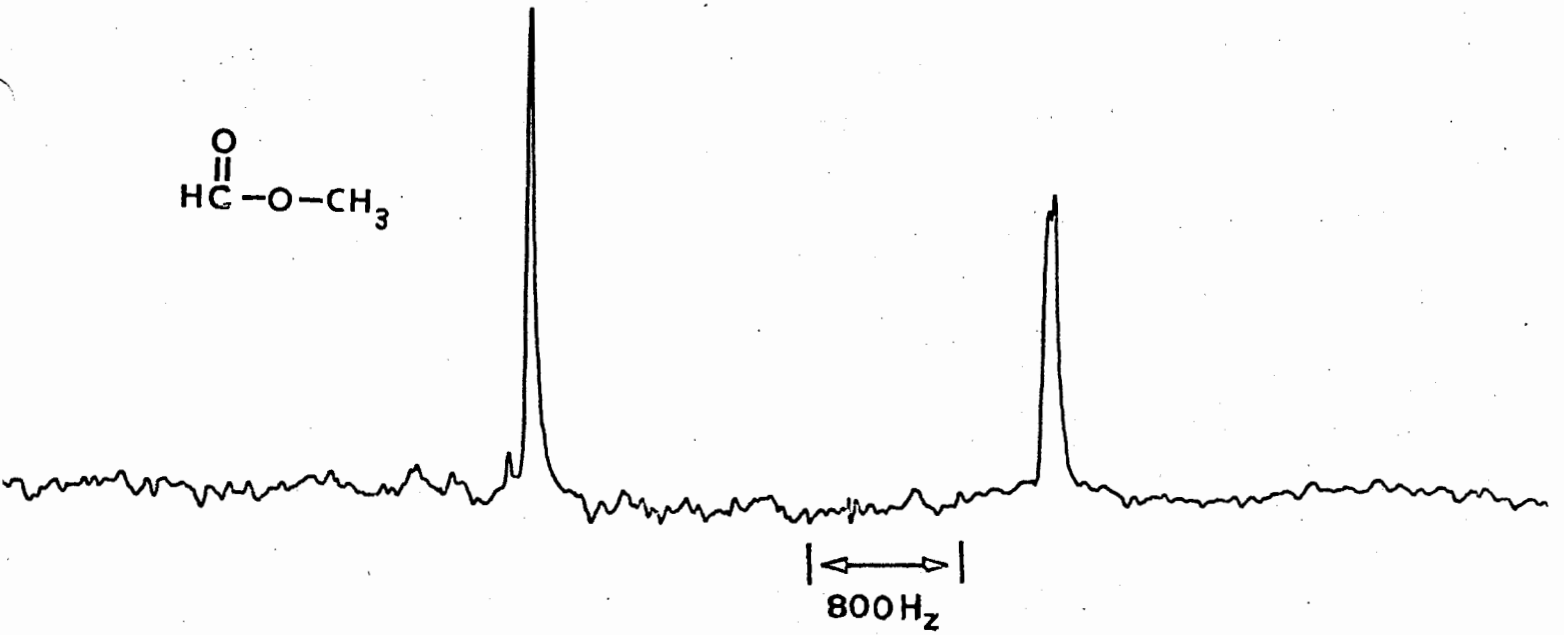
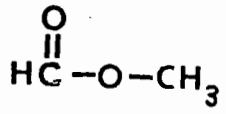
Frequency scale on attached spectra : 400 Hz/cm

No. of data points : 8 K

SYSTEM : BRUKER HX-90 FT incorporating the NIC-1085 data system.

Christian I. Tanzer

Dieter Hiss



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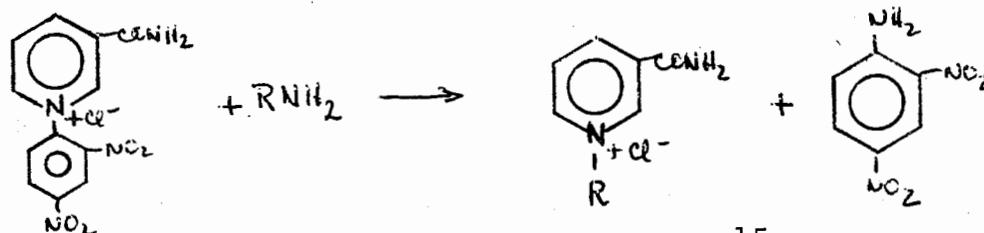
DEPARTMENT OF  
 CHEMISTRY AND GEOLOGY

August 15, 1972

Dr. Bernard L. Shapiro  
 Department of Chemistry  
 Texas A & M University  
 College Station, Texas 77834

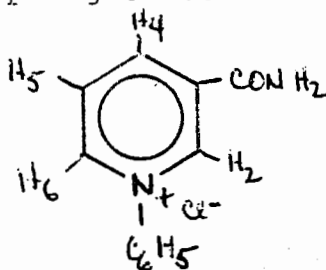
Dear Barry:

In connection with our nmr work<sup>1</sup> on NAD and NADH it became necessary to determine the stereochemistry (at the  $\alpha$  carbon of the side chain) of the Zincke-Lettre<sup>2</sup> reaction (equation). Utilization of 99+% enriched  $^{15}\text{N}$ -aniline as the amine component afforded 1-phenyl-nicotinamide chloride containing 99+%  $^{15}\text{N}$  in the 1-position, i.e., the amine component is introduced intact.



This is an extremely facile introduction of  $^{15}\text{N}$  into the pyridine nucleus. The pertinent H-H and N-H coupling constants are given in the table.

Coupling Constants<sup>a</sup>



$J_{24}$	1.72	$J_{56}$	6.18
$J_{25}$	0.64	$J_{12}$	-1.72
$J_{26}$	-1.41	$J_{14}$	$\pm 1.14$
$J_{45}$	8.03	$J_{15}$	-4.96
$J_{46}$	1.28	$J_{16}$	-1.75

<sup>a</sup>0.5 M solution in  $\text{D}_2\text{O}$



We are currently applying this reaction to the preparation of specifically  $^{15}\text{N}$  labelled nicotinamide mononucleotide for nmr studies.

In collaboration with Dr. William Egan, currently at the Lund Institute of Technology, we have measured the NOE's of NMN and NAD in an effort to determine the solution conformation of NAD. Quantitative analysis<sup>3</sup> of the results are currently under way.

Details of the labeling reaction or the NOE experiments will be provided to interested parties.

Sincerely yours,



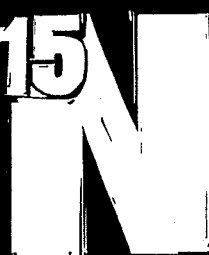

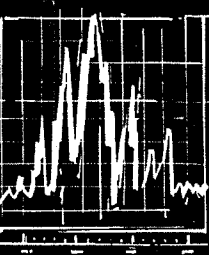
*John Jacobus*

John Jacobus

1. J. Jacobus, Biochemistry, 10, 161(1971).
2. H. Lettre, W. Haeda, and E. Rahbaum, Ann. Chem., 579, 123(1953) and references cited therein.
3. R. E. Schirmer, J. P. Davis, J. H. Noggle, and P. A. Hart, J. Amer. Chem. Soc., 94, 2561(1972).

Suggested Title:  $^{15}\text{N}$ - $^1\text{H}$  Coupling: Facile Introduction of  $^{15}\text{N}$  into the Pyridine Nucleus. NAD-NOE.

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DEPARTMENT OF CHEMISTRY  
REVELLE COLLEGEPOST OFFICE BOX 109  
LA JOLLA, CALIFORNIA 92037

## EFFECTS OF TRANSVERSE RELAXATION IN SPIN ECHO SPECTRA

Dear Barry,

Our continued interest in the details of transverse relaxation mechanisms has lately led us to measure spin echo spectra of different types of  $A_2B$  systems by Fourier transformation of Carr-Purcell envelopes.

The accompanying figure shows an example: the spin echo spectrum of 1,1,2-trichloroethane obtained at 55 MHz and a Carr-Purcell pulse spacing  $2\tau$  of 14 msec. The spectrum is moderately complex, consisting of a single line at zero frequency and an assortment of lines of varying width and intensity.

The differential linewidths are doubtless due to the existence of different scalar coupling constants between protons and various rapidly relaxing chlorine nuclei. However, analysis by means of formulae used for cw linewidths is not possible because the proton spin states associated with each spin echo line are not "pure" in this coupled system, and it is particularly relevant that the  $180^\circ$  refocussing pulses introduce a further scrambling. We are in process of mimicing the relaxation pathways in this molecule by density matrix calculations. Unfortunately, your famous pink slip and a month long shutdown of the campus computer center arrived simultaneously so we have no quantitative results at this point.

However, one effect of interest is apparent in the figure. Allerhand<sup>1</sup> has calculated the line frequencies and intensities to be expected for an  $A_2B$  spin system without relaxation. We have checked his truly formidable algebra and find no mistakes; a maximum of seven distinct lines is predicted. Depending on the personal bias of the observer, there are eight or nine lines in the spectrum shown. High resolution cw spectra reveal no impurities, the spin echo spectrum is reproducible, and this and other  $A_2B$  systems give more than seven lines at various short pulse spacings. Our explanation for the appearance of extra lines is as follows.

For an  $A_2B$  system, there are at most nine spectral magnetizations which precess in the transverse plane following a nonselective  $90^\circ$  pulse. Their vector sum along some fixed axis (in the rotating frame) is measured at the time of each echo, and the Fourier transform reveals the effective precession frequencies which are determined by the coupling constant, chemical shift, and the pulse spacing. Allerhand's treatment shows that in absence of relaxation, some eigenvalues of the recursion matrix are degenerate which leads to only seven distinct lines.

However, if relaxation is included, elements of the relaxation matrix which are nonsecular in the sense of Redfield result in additional coupling of the differential equations of motion for the spectral magnetizations<sup>2</sup> and can therefore lead to small shifts of the precession

frequencies<sup>3</sup>. We have previously observed such small shifts in spin echo spectra of 1,1-difluoro-2,2-dichloroethane<sup>3</sup>, and in the case of A<sub>2</sub>B systems, the effects of off diagonal relaxation matrix elements is apparently large enough to break the degeneracy mentioned above.

The small effect of relaxation on precession frequencies is normally obscured<sup>3</sup> in high resolution spectra by field inhomogeneity and is also expected to be negligible in spin echo spectra with long pulse spacings. At high pulse rates, the 180° pulses act to "enhance" the effects of off diagonal relaxation matrix elements.

In support of this interpretation, we find that the number of distinctly observable lines is never more than nine at any pulse spacing, and at large pulse spacings (>150 msec) the spectra agree satisfactorily with those calculated by Allerhand's formulae.

#### References

1. A. Allerhand, J. Chem. Phys. 44, 1, (1966)
2. R.L. Vold and S.O. Chan, J. Chem. Phys. 56, 28 (1972)
3. R.L. Vold and R.R. Shoup, J. Chem. Phys. 56, 4787 (1972)

Best regards,

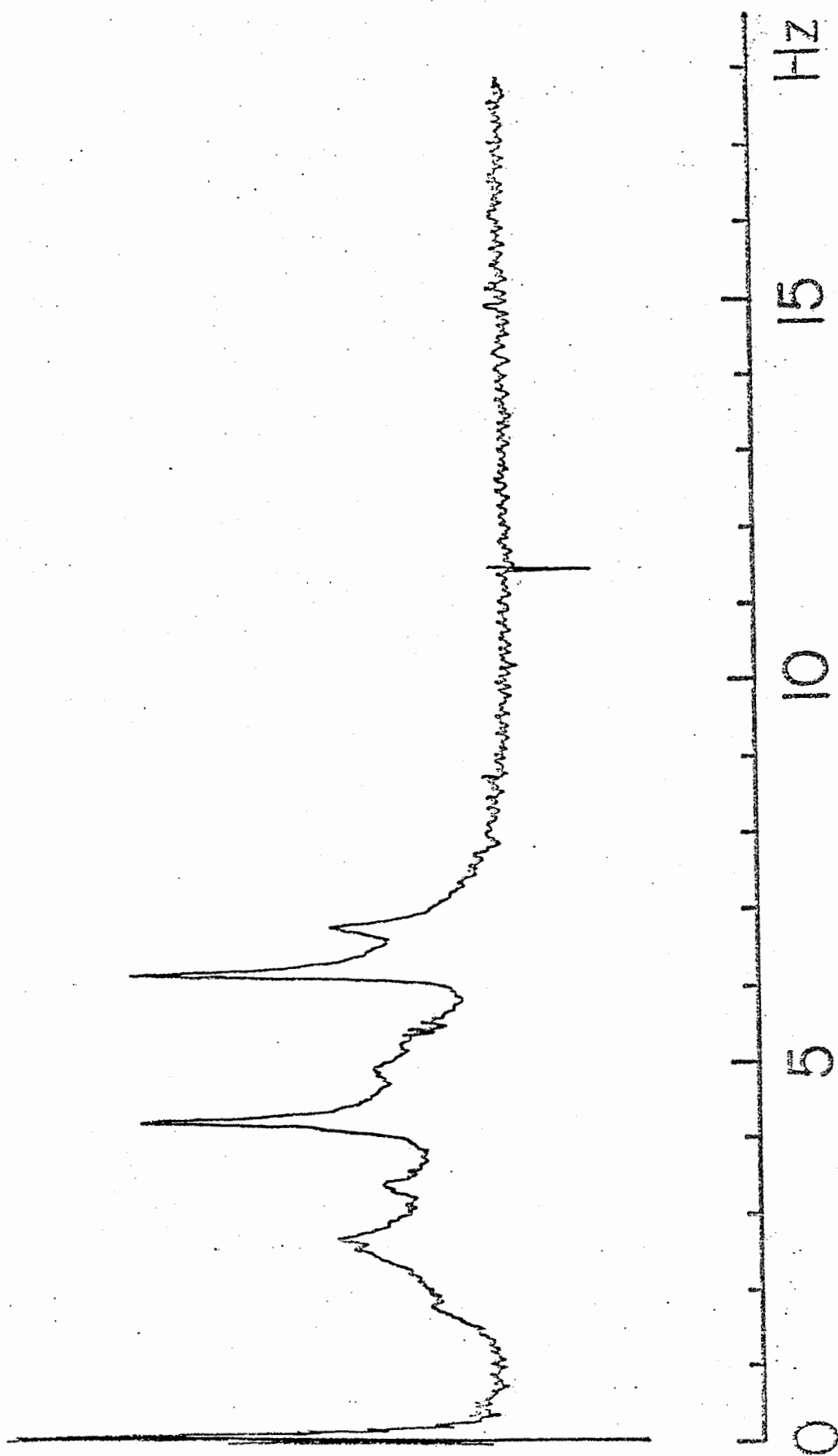
R.L. Vold and R.R. Shoup

*Bob & Gille*



# Spin Echo Spectrum of $\text{CH}_2\text{Cl}\cdot\text{CHCl}_2$

$2\tau = 14 \text{ msec}$





## THE OHIO STATE UNIVERSITY

August 18, 1972

Professor Bernard L. Shapiro  
 Department of Chemistry  
 Texas A and M University  
 College Station, Texas 77843

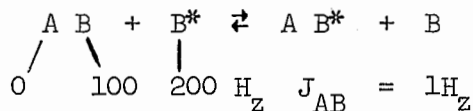
Nmr Line-Shapes at High RF in Chemically Exchanging Systems.

Dear Barry:

Sorry you had to send two reminders. I promise to do better next time.

The procedure for calculating nmr line-shapes for chemically exchanging systems at low RF is now quite well known<sup>1</sup>. What makes it easy is that due to the low power approximation the equations become linear in the density matrix elements and hence these can be solved with standard matrix inversion techniques.

Recently Dr. Ping Pin Yang has been looking into what happens to these line-shapes for exchanging systems at higher RF fields, specifically for systems undergoing bimolecular exchange. Now one cannot approximate the diagonal elements with Boltzman factors so the equations become second order in the density matrix elements. Further, it is necessary to solve for all density matrix elements, not just those for  $\Delta m = \pm 1$ . We allow for different nuclear relaxation times and solve the equations by iteration starting with the low power solution and going in steps to high power. We chose a hypothetical system, a two proton molecule exchanging its B spin with an outside source of protons.



Line-shapes were calculated as a function of the RF field and rate constant, see below B part of AB for a rough sketch. You can see how at low RF the line-shape is not very sensitive to the rate constant; the opposite is the case at high RF. Since this kind of experiment is easy to carry out, we are hoping the technique will be useful for measuring the smaller rate constants ( $10^{-2}$  to  $10 \text{ sec}^{-1}$ ) with

<sup>1</sup> G. Fraenkel and J. Kaplan, J. Amer. Chem. Soc., 94, 2907 (1972) and references therein.

Professor B. L. Shapiro  
Texas A and M University

August 18, 1972  
Page

much greater precision than is possible at low RF power.

Our current problem is to find a more efficient method to solve the second order equations.

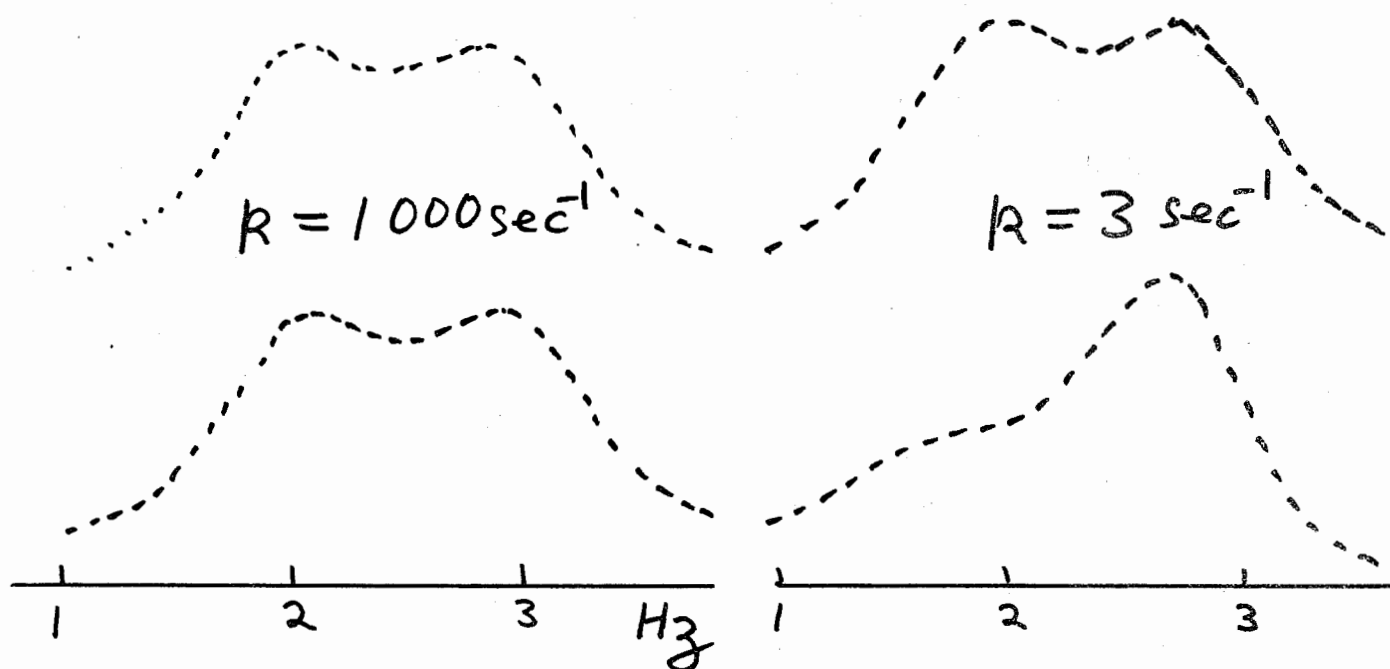
With best regards,

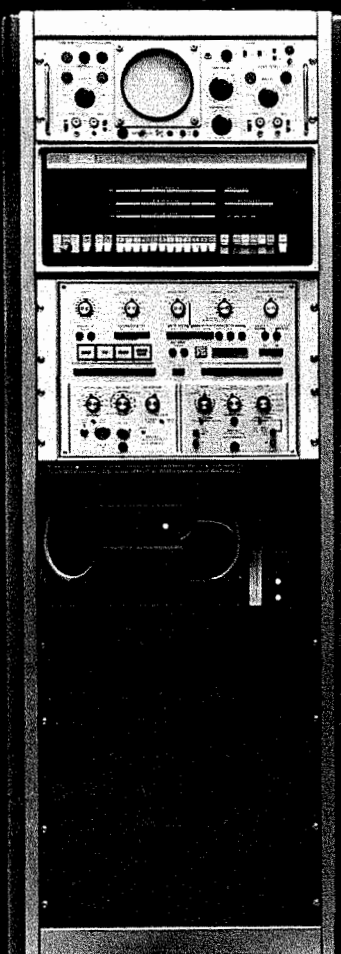
Sincerely yours,

Gideon

Gideon Fraenkel  
Professor of Chemistry

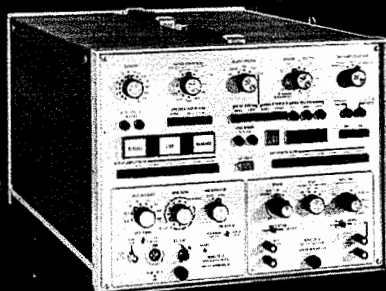
Line shape, B of AB  $H_1 = 0.2 H_3$





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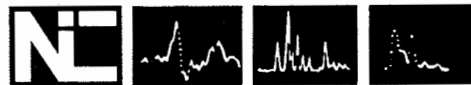


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# TEXAS CHRISTIAN UNIVERSITY

Fort Worth, Texas 76129

Department of Chemistry

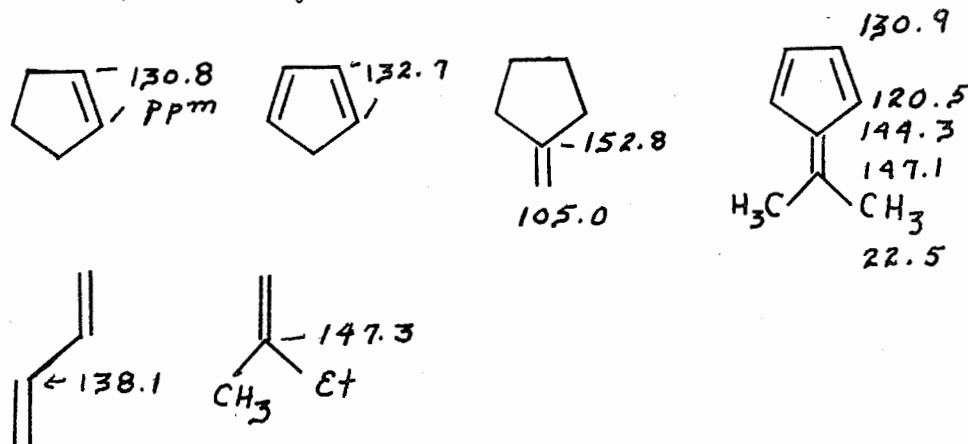
August 22, 1972

Dr. B. L. Shapiro  
Department of Chemistry  
Texas A and M University  
College Station, Texas 77843

Dear Barry:

Over the years our interest in the fulvenes has maintained itself. They are isoelectronic with the benzenes, and HMO theory predicts a sizeable delocalization energy and dipole moment. Of course in such nonalternant systems the HMO procedure leads to distorted expectations. SCF and PMO calculations are more in accord with the chemistry of these molecules which is clearly that of cyclic dienes.

Having converted our HA-100 to C-13, it was natural that we take a quick look at dimethylfulvene. The spectrum was taken in cyclohexane solution with the solvent as the lock. Chemical shifts are referenced to TMS. The two quaternary carbons and the remaining ring carbons are readily differentiated, but the assignments among these groups is open to question. The assignments shown were conjured up by a consideration of related structures taken from the literature (pertinent examples shown below), charge density effects ( $\sim 160$  ppm/electron), and the known effects of methyl substituents on olefinic carbons.



Yours sincerely,

*Bill*

W. B. Smith  
Chairman  
Department of Chemistry

WBS/dc



2000 NINTH AVENUE SOUTH  
BIRMINGHAM, ALABAMA 35205  
TELEPHONE 205-323-6592

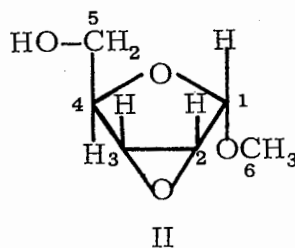
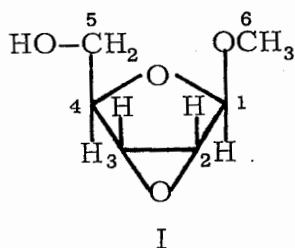
August 28, 1972

Prof. B. L. Shapiro  
Department of Chemistry  
Texas A & M University  
College Station, Texas 77843

Dear Barry:

$^{13}\text{C}$  NMR of Anomeric Sugar Derivatives

We have recently investigated the  $^{13}\text{C}$  spectra of two sugar derivatives, I and II.



We had hoped that the  $^{13}\text{C}$  spectra would show greater differences than were apparent in the proton spectra, but the  $^{13}\text{C}$  spectra are also disappointingly similar. Values for the chemical shifts and some of the coupling constants are tabulated below.

The assignments of carbons 2 and 3 are tentative, and are based primarily on the greater multiplicity of the absorption assigned to  $\text{C}_3$  in the undecoupled spectra. This seems reasonable in view of the proximity of the two protons at  $\text{C}_5$ . This, surprisingly, however, is the absorption which shows the greatest chemical shift difference between I and II.

There are some other interesting long-range coupling constants which we have not completely assigned. The greatest difference between I and II as far as long-range coupling constants are concerned is on the multiplet assigned as  $\text{C}_2$ .

This work was done on our XL-100-15 equipped with the Digilab NMR-3 FT pulser and data system.

Sincerely,

Martha C. Thorpe

## Southern Research Institute

Prof. Shapiro

August 28, 1972

<u>Compound</u>	<u>Carbon</u>	<u><math>\delta^*</math></u>	<u>J's (Hz)</u>
I	1	102.38	$^J\text{C}_1, \text{H}_1 = 174.3 \pm .2$ $^J\text{C}_1, \text{H}_6 = 4.6 \pm .2$
II	1	102.75	$^J\text{C}_1, \text{H}_1 = 167.5 \pm .2$
I	2	56.30	$^J\text{C}_2, \text{H}_2 = 205.2 \pm .2$
II	2	56.43	$^J\text{C}_2, \text{H}_2 = 191.8 \pm .2$
I	3	55.13	$^J\text{C}_3, \text{H}_3 = 202.0 \pm .2$
II	3	56.85	$^J\text{C}_3, \text{H}_3 = 190 \pm 1$
I	4	79.83	$^J\text{C}_4, \text{H}_4 = 151 \pm 1$
II	4	79.05	$^J\text{C}_4, \text{H}_4 = 151 \pm 1$
I	5	62.95	$^J\text{C}_5, \text{H}_5 = 142.3 \pm .2$
II	5	63.15	$^J\text{C}_5, \text{H}_5 = 142.3 \pm .2$
I	6	56.01	$^J\text{C}_6, \text{H}_6 = 143.3 \pm .2$ $^J\text{C}_6, \text{H}_1 = 4.3 \pm .2$
II	6	56.75	$^J\text{C}_6, \text{H}_6 = 142.9 \pm .2$ $^J\text{C}_6, \text{H}_1 = 4.6 \pm .2$

\* Chemical shifts are in ppm downfield from internal TMS, and are accurate to  $\pm 0.02$  ppm. Both samples were run in  $\text{CDCl}_3$  at a concentration of 70 mg/ml.

WESTERN  
RESEARCH CENTER

## Stauffer Chemical Company

1200 South 47th Street / Richmond, California 94804 / Tel. (415) 233-9361

September 1, 1972

Professor B. L. Shapiro  
Department of Chemistry  
Texas A & M University  
College Station, Texas 77843

Stereoisomers of 2-methyl-5-ethyl piperidines

Dear Barry:

We have separated the two isomeric 2-methyl-5-ethyl piperidines by the method of Levy and Wolffenstein.<sup>1</sup> The configurational assignments are made on the basis of 220 MHz proton spectra.<sup>2</sup> The lower field region of the spectra corresponding to the three protons adjacent to the nitrogen atom can be analyzed to obtain the configurational information. Due to the ring inversion, the pmr parameters of the cis isomer ( $n_D^{30}$  1.4438) are not readily obtainable. The  $H_2$  proton of the cis isomer exhibits a multiplet at 2.73 ppm and the  $H_{6ae}$  protons give a doublet centered at 2.82 ppm. The pmr parameters of  $H_{2a}$ ,  $H_{6a}$ , and  $H_{6e}$  protons of the trans isomer ( $n_D^{30}$  1.4410) are listed in the table.

Sincerely yours,

A handwritten signature in dark ink, appearing to read "CKTeng", written in a cursive style.

C. K. Tseng

CKT/lh  
Attachment



<u>Protons</u>	<u>Shifts (ppm)<sup>3</sup></u>	<u>Coupling Constants (Hz)<sup>3</sup></u>
H <sub>2a</sub>	2.54	J <sub>2a-3a</sub> , 10.2; J <sub>2a-3e</sub> , 2.8; J <sub>2a-Me</sub> , 7.0
H <sub>6a</sub>	2.25	J <sub>6a-5a</sub> , 10.2; J <sub>6a-6e</sub> , 11.8
H <sub>6e</sub>	3.08	J <sub>6e-6a</sub> , 11.8; J <sub>6e-5a</sub> , 3.5; J <sub>6e-4e</sub> , 2.0

1. L. Levy and R. Wolffenstein, Ber., 29, 1959 (1896)
2. We wish to thank Mr. L. F. Johnson, NMR Application Laboratories, Varian Associates, Palo Alto, California for obtaining 220 MHz NMR spectra.
3. In CDCl<sub>3</sub> solution.

THE UNIVERSITY OF NORTH CAROLINA  
AT  
CHAPEL HILL  
27514

DEPARTMENT OF CHEMISTRY

August 30, 1972

Professor Bernard L. Shapiro  
Department of Chemistry  
Texas A & M University  
College Station, Texas 77843

Partially Relaxed Fourier Transform Spectra of Solids

Dear Barry:

We have been using Fourier transform methods to obtain wide line spectra of protons in solids. This avoids saturation and modulation broadening and permits the simultaneous determination of spin-lattice relaxation times. The attached figure shows a series of spectra of  $\text{CH}_3\text{CN}$  dissolved in  $\text{CD}_3\text{CN}$  (1:10 by volume) at  $77^\circ\text{K}$ . The method used here is the same as that demonstrated by Vold, *et al.*,<sup>1</sup> for high-resolution NMR, namely a  $\pi$ - $\tau$ - $\pi/2$  sequence where  $\tau$  is varied. A Biomation Transient Recorder was used to record 1024 points on the FID at the rate of 1 point/ $\mu\text{sec}$  with a repetition period of 1.2 seconds and an accumulation time of 8 min. The signals were accumulated in the memory of a Nuclear Data 812 computer and were then transformed with a Raytheon 706 computer. The experiment was carried out on resonance with only points in the real domain being used in the transformation.

The powder spectrum shown in the figure indicates that the  $\text{CH}_3$  group in  $\text{CH}_3\text{CN}$  is rotating rapidly around its symmetry axis. It is expected that  $T_1$  will depend on the orientation of the symmetry axis with respect to the applied field and, therefore, will vary with position in the spectrum.<sup>2</sup> A variation is also expected because of the weak  $H_1$  pulse used.<sup>3</sup> The width of the spectrum is of the order of 50 kHz and the  $\pi$ -pulse length was  $\sim 8\mu\text{sec}$ . In contrast to these expectations our analysis indicates that  $T_1 = 0.22$  sec for the outer components as well as the central component. We conclude that at this spin concentration the spin diffusion time is much less than  $T_1$ . For very dilute spin systems, where spin diffusion is negligible, the variation of spin-lattice relaxation rates may permit the selection of spin systems on the basis of orientation in a manner somewhat analogous to photo selection in optical spectroscopy.

Sincerely,

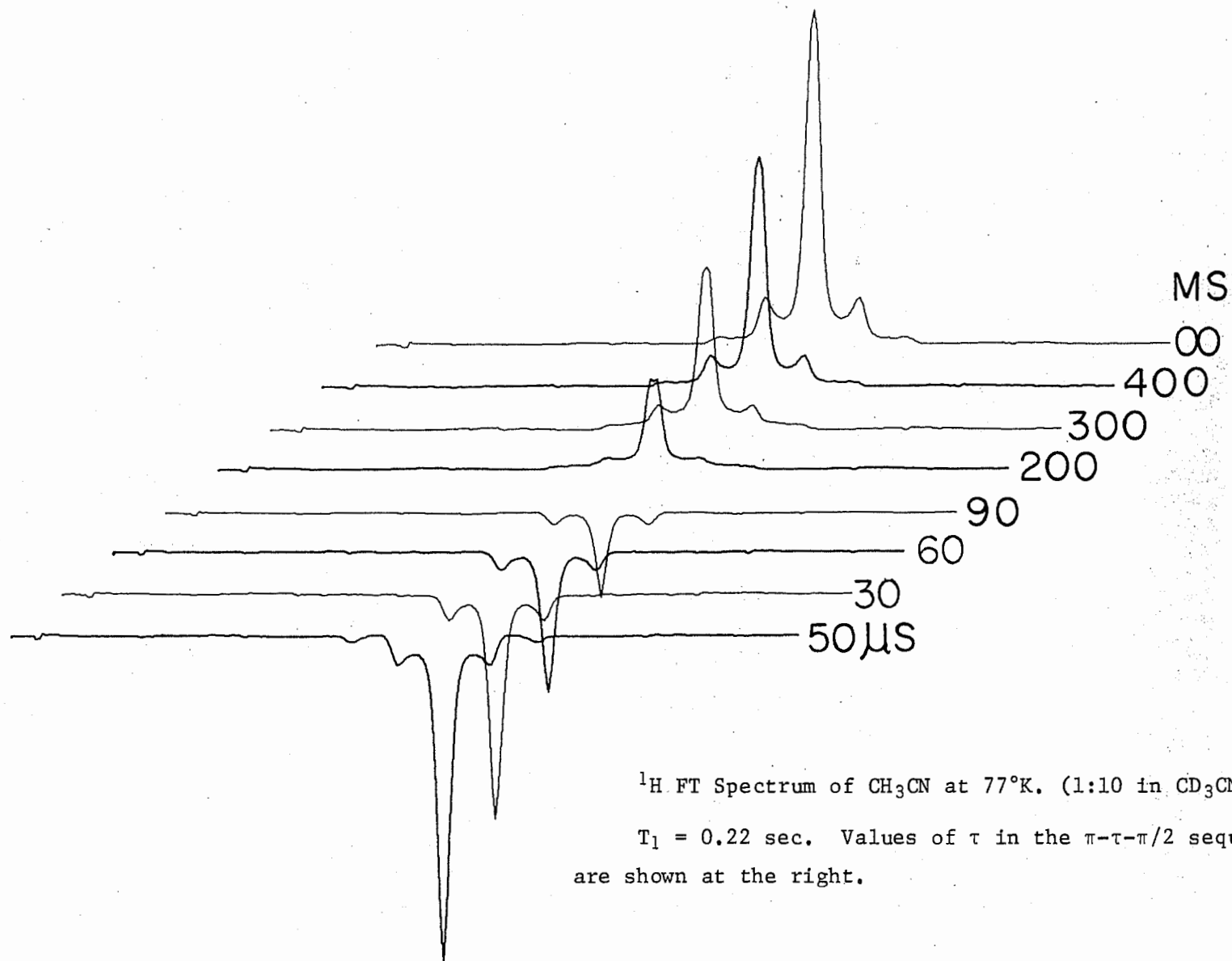
*Anil Kumar* *Charles*  
Anil Kumar Charles S. Johnson, Jr.

AK:CSJ:bs

Enclosure

1. R. L. Vold, J. S. Waugh, M. P. Klein and D. E. Phelps, J. Chem. Phys. 48, 3831 (1968).
2. R. H. Hilt and P. S. Hubbard, Phys. Rev. A134, 392 (1964).
3. D. E. Jones and H. Sternlicht, J. Magn. Resonance, 6, 167 (1972).

P.S. C.S.J. is on leave from September, 1972, through July, 1973. His address is: University Chemical Laboratory, Lensfield Road, Cambridge CB2-1EW, ENGLAND.



$^1\text{H}$  FT Spectrum of  $\text{CH}_3\text{CN}$  at  $77^\circ\text{K}$ . (1:10 in  $\text{CD}_3\text{CN}$ )

$T_1 = 0.22$  sec. Values of  $\tau$  in the  $\pi$ - $\tau$ - $\pi/2$  sequence are shown at the right.

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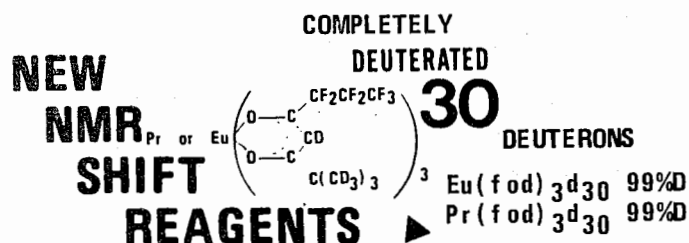
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547

August 30, 1972

Dr. Bernard L. Shapiro  
Department of Chemistry  
Texas A & M University  
College Station, Texas 77843

Dear Barry:

As Program Chairman for the 1973 Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy, I would like to announce the organization of a

SYMPOSIUM ON INDUSTRIAL APPLICATIONS OF  $^{13}\text{C}$  NMR

to be held at our 24th meeting. NMR experts actively involved in applying  $^{13}\text{C}$  NMR in the petroleum, polymer, pharmaceutical, and other industries will present invited papers. Frank Dickson, Gulf Research and Development Co., P. O. Box 2038, Pittsburgh, Pa. 15230, may be contacted for further details. Persons desiring to contribute papers in any field of NMR spectrometry are invited to do so.

A complete list of symposia which will be held at the Conference is given below:

1. LIQUID CHROMATOGRAPHY - Analytical Bridge to Spectroscopy.
2. THREE DIMENSIONAL MICROCOMPOSITIONAL ANALYSIS: The Advent of Ion Probe Mass Spectrometry.
3. INDUSTRIAL APPLICATIONS OF  $^{13}\text{C}$  NMR.
4. FORENSIC SCIENCE.
5. WOMEN IN MASS SPECTROMETRY.
6. INDUSTRIAL RESEARCH - Is It Worth It?
7. GUIDELINES FOR DEFINING AND IMPLEMENTING THE COMPUTERIZED LABORATORY SYSTEM (ASTM).
8. THE ANALYSIS OF SLAGS AND RELATED OXIDE-TYPE MATERIALS (ASTM).
9. COBLENTZ SOCIETY AWARD SYMPOSIUM.
10. SPECTROSCOPY SOCIETY OF PITTSBURGH AWARD SYMPOSIUM.

Papers are not restricted to the symposium topics and original papers on all phases of analytical chemistry and spectroscopy are invited. Abstract forms and related information may be obtained by writing to me at the above address. The final date for receipt of abstracts is October 1, 1972. The meeting will be held at the Cleveland Convention Center, Cleveland, Ohio, U.S.A., March 5-9, 1973.

H. L. Retcofsky

DIVISION OF CHEMISTRY AND PHARMACY  
UNIVERSITY OF MUNICH  
8000 MUNICH 2

INSTITUTE OF ORGANIC CHEMISTRY  
KARLSTRASSE 23

GERHARD BINSCH  
PROFESSOR OF THEORETICAL ORGANIC CHEMISTRY

20 August 1972

Professor Bernard L. Shapiro  
Department of Chemistry  
Texas A + M University  
College Station, Texas 77843

Three Postdoctoral Positions Available

Dear Barry:

Please permit me to (ab)use your Newsletter for this announcement.

1. Organic Quantum Chemist. Choice of projects: (a) Cluster expansions and natural orbitals; applications to organic photochemistry. (b) Semi-classical trajectory calculations on model hypersurfaces. (c) Intramolecular perturbation theory. Desired background: Quantum theory, numerical methods, a wizard in computer programming.

2. Pure NMR Spectroscopist. Choice of projects: (a) Theoretical and experimental relaxation studies on coupled proton systems. (b) Automated spectral analysis using total band shape. Desired background: Theory of nmr, computer programming, some knowledge of electronics. Plus for (a): Familiarity with density matrix methods. Plus for (b): Some experience with numerical methods, assembler language, construction of interfaces.

3. Applied NMR Spectroscopist. Choice of projects: (a) Conformational analysis of side chains in amino acids and polypeptides. (b) Correlated motions in model compounds. (c) Variable-temperature fluorine nmr. Desired background: experience in variable-temperature nmr and organic synthesis.

Some experience as indicated above is desirable, but not indispensable. Applicants with a variety of backgrounds will be considered. The essential ingredients are intelligence, superior motivation, scientific stamina and, for foreigners, the personal self-confidence to easily cope with the challenge of an unfamiliar environment.

The appointments will encompass a minor amount of auxiliary teaching duties. Applicants must be prepared to make a serious effort to pick up some German during the first weeks of residence.

The monthly salary is in the range of DM 1800 to 2300, depending on a number of factors. The positions can be filled immediately or later. The appointments would normally be for one year, but extensions are possible.

Individuals interested in applying should provide me with the usual personal and professional data and simultaneously arrange for three letters of recommendation to be sent to me.

Sincerely yours,

*Gerhard*  
Gerhard Binsch



Department of Chemistry • TEL. (603) 646-2501

August 31, 1972

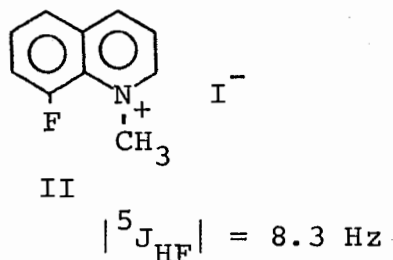
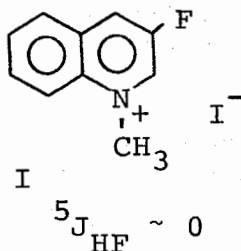
Dr. Bernard L. Shapiro  
Department of Chemistry  
Texas A&M University  
College Station, Texas 77843

Through-Space H-F Coupling

Dear Dr. Shapiro:

In connection with our interest in long-range H-F coupling<sup>1</sup> we have prepared and examined the proton nmr spectra of some fluorinated N-alkylquinolinium salts.

Thus, while I shows a sharp methyl singlet at 4.7 ppm (D<sub>2</sub>O; 60 MHz) II shows a doublet at 4.75 ppm, assigned to the methyl protons which are presumably split by the fluorine atom five bonds away,  $^5J_{\text{HF}} = 8.3 \text{ Hz}$  (D<sub>2</sub>O; 60 MHz).



Similarly, the spectrum of N-ethyl-8-fluoroquinolinium iodide shows further splitting of the ethyl group,  $^5J_{\text{HF}} = 2-3 \text{ Hz}$  and  $^6J_{\text{HF}} = 1-2 \text{ Hz}$ .

Interestingly, the methyl-fluorine internuclear distance and observed H-F splitting are about the same for II and for 4-fluoro-5-methylfluorene,<sup>1</sup> where in the latter compound the H and F are separated by six bonds. This further supports the notion of a "through-space" contribution to the coupling in compounds of this type.

These spectra were run on our recently-acquired Perkin-Elmer R-24 spectrometer since our Varian HA-60IL is undergoing extensive modification.

Sincerely yours,

*Gordon W. Gribble Keith Haden*  
Gordon W. Gribble      Keith Haden  
Assistant Professor  
of Chemistry

1. G.W. Gribble and J.R. Douglas, Jr., J. Amer. Chem. Soc., **92**, 5764 (1970).





# THE UNIVERSITY OF GEORGIA

DEPARTMENT OF CHEMISTRY • ATHENS, GEORGIA 30601 • 404-542-2626

August 24, 1972

Professor B. L. Shapiro  
Department of Chemistry  
Texas A and M University  
College Station, Texas 77843

Dear Barry:

## An Inexpensive Spectrum Plotter

In the analysis of complex spectra, a calculated spectrum is required in order to make line assignments. Furthermore, it is desirable to obtain a plot of the calculated spectrum so that a visual check of intensities can be made. Depending upon the particular computing facility, the above procedure usually requires punching input parameters on cards for the trial run, submitting the program, waiting anywhere from a few minutes to a day for the results, and finally, somehow obtaining a plot of the spectrum.

Since most computing systems utilize terminals nowadays, we have bypassed the "waiting period" involved in the above procedure by using a Hewlett-Packard Model 7200 Graphic Plotter interfaced to a standard ASR 33 terminal to obtain the calculated spectrum along with a plot of the spectrum. The program which is stored on a data cell of the CDC 6400 consists of the calculation part of LAOCOON II plus our scaling and plotting routine. All that is required for obtaining a calculated spectrum plus plot is calling the program out of storage and entering the input parameters. This entire procedure usually takes less than two minutes. At present, we are using a stick plot. However, there is no reason why a lineshape factor could not be incorporated so that a line plot would be obtained. This would be particularly convenient for programs used to calculate DNMR spectra. I'll be happy to supply details to anyone interested. Also, Professor Ed Janzen is using this setup to obtain plots of esr spectra.

## A Request

We are interested in adding other nuclei capabilities to our HR-60 spectrometer. Specifically, we would like 24.2 and 15.0 MHz plus perhaps the V-4210A variable frequency rf unit with matching probes. If you have any of the above as excess equipment and wish to part with it for a reasonable price, please contact me at your earliest convenience.

Sincerely yours,

Richard H. Cox  
Assistant Professor

RHC:mjd



# XIth EUROPEAN CONGRESS ON MOLECULAR SPECTROSCOPY

## XI ЕВРОПЕЙСКИЙ КОНГРЕСС ПО МОЛЕКУЛЯРНОЙ СПЕКТРОСКОПИИ

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8 th August 197 2

Professor Bernard L. Shapiro  
Department of Chemistry  
Texas A & M University  
College Station  
Texas 77843 USA

Dear Professor Shapiro,

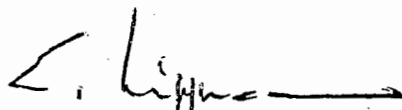
This time I would like to draw attention to our plan to organize the 11th European Congress on Molecular Spectroscopy in Tallinn next year, from May 28 to June 1, 1973. Two large topics are going to be on the agenda - optical spectroscopy of molecular crystals and all possible aspects of magnetic resonance of nuclei other than  $^1\text{H}$  and  $^{19}\text{F}$ . Although the word heteronuclei is admittedly ambiguous and perhaps sometimes misleading, we have formulated our topics as follows:

1. Fourier transform and pulse methods in NMR - meaning all work done with the Fourier transform technique as long as it is methodically new and opens up new possibilities for research, even though in proton or fluorine resonance. Work on biopolymers and other big molecules, such as steroids would be especially welcome. In the relaxation field all studies of nuclei other than  $^1\text{H}$  and  $^{19}\text{F}$  are welcome and proton relaxation too if the molecules studied are really large or somehow especially interesting.
2. Heteronuclear resonance of liquids - meaning all chemical and physical applications of the NMR spectra of all nuclei other

than  $^1\text{H}$  and  $^{19}\text{F}$ . Chemical shifts and the spin-spin coupling constants, relaxation, chemical polarization, conformation and internal rotation studies, studies of reaction mechanisms and pathways, etc. is all included. We hope to show that the tide has already turned and the "other" nuclei are the really important ones, leaving the protons (and sometimes even fluorines) to the peripheral position that befits their chemical nature.

We have our new 23-storey hotel building ready and even though it may be small by American standards we can provide good accomodation for all who are interested and write to our Secretary Dr. T. Saluvere using the address given in the letter-head. The second announcement will be distributed in October and the deadline for short summaries of papers is January 15, 1973. Even though the Congress is officially an European one, we do hope that nobody takes seriously such old-fashioned geography.

Sincerely,



Endel T. Lippmaa

Vice-Chairman of  
XI th ECMS

**ECMS88**  
**TALLINN 1973**

I am interested in the XI ECMS and would like to receive further information.

Full Name

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I plan to submit Paper

Tentative Title or Subject



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# JEOL

PS-100  
Console-  
Spectrum:  
25.1 MHz  
 $^{13}\text{C}$  Solid  
Natural  
Rubber



## Give your pulse a heart.

**For the kind of pulse required in tomorrow's NMR research, we have the perfect heart today. It's our PFT-100 Fourier Transform system. A system designed with incredible power, unusual versatility.**

The PFT-100 is capable of a 15  $\mu\text{sec.}$  pulse sufficient to produce a  $90^\circ$   $^{13}\text{C}$  magnetization vector tipping. Complete controls permit fine adjustments to regulate Pulse Width, Interval and Repetition. The system design allows you to select single or multiple pulse programs such as FT, DEFT, SEFT and more. Plug-in hard board program modules make it all possible.

The magnet and CW/FT probe is also at the state of the art. It features a dual cross coil detection system with Deuterium ( $^2\text{H}$ ) Internal lock. It has a hetero-nucleus External lock, as well. You choose a fixed or replaceable insert probe which permits 5, 8 and 10mm/OD variable temperature sample tubes.

There's more. Push button controls and the solid state design of the PS-100 CW/NMR. There is also complete compatibility with most existing FT data reduction systems. They combine to make it a total research NMR system.

### At the heart of NMR research.

JEOL provides worldwide service and knowledge that are unsurpassed in the NMR field. With our MH-60, C-60HL, MH-100 and the PFT-100, we offer a broader range of equipment and locations than any other firm.

Information and demonstrations are available through our offices in Tokyo, London, Paris and Sydney. As well as the newest laboratory in the United States devoted to NMR research at JEOL, 235 Birchwood Avenue, Cranford, N.J. 07016.



NMR Spectrometers/Mass Spectrometers/ESR Spectrometers/Laser Raman Spectrophotometers/Laboratory Computers/Scanning Electron Microscopes/Electron Microscopes/X-ray Diffractometers/Amino Acid Analyzers.



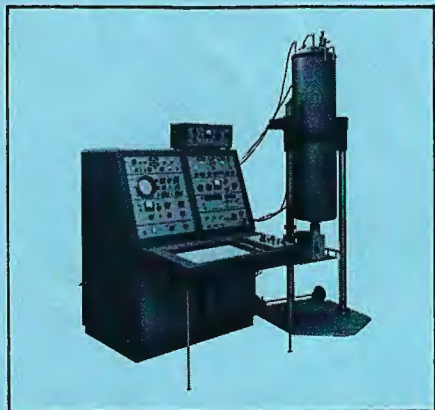
# When You Need an NMR System, See Varian First.

Varian, world leader in magnetic resonance spectroscopy, offers a complete selection of NMR spectrometer systems; from low cost systems designed to solve the typical problems of industrial and academic laboratories to the ultimate in research NMR systems.

Our basic systems are briefly described here. Ask us what's new for any one, since we're continually expanding the flexibility and research capability of NMR instrumentation.

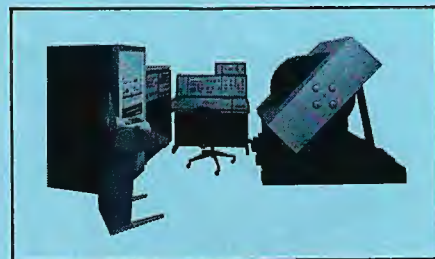
Locking flexibility for homo- or heteronuclear internal lock on  $^1\text{H}$ ,  $^{19}\text{F}$ , and  $^2\text{H}$ , and external lock on  $^1\text{H}$  and  $^{19}\text{F}$  are provided. Fourier transform operation,  $T_1$  measurements, homonuclear decoupling and solvent elimination techniques are also routine XL-100 operations. The XL-100 can be tailored to meet single purpose or multi-departmental NMR needs.

## HR-220 and HR-300



These offer the highest performance in commercially available high resolution NMR spectrometers. The HR-220 (220 MHz) and HR-300 (300 MHz) spectrometers are engineered for use in those applications requiring the highest magnetic field strengths such as liquid crystal, biological, biopolymer, and biochemical studies. Accessories are available for observation of  $^1\text{H}$ ,  $^{13}\text{C}$ ,  $^{19}\text{F}$ ,  $^{31}\text{P}$ ,  $^{11}\text{B}$ , and  $^2\text{H}$  in both the continuous wave and Fourier transform modes.

## XL-100



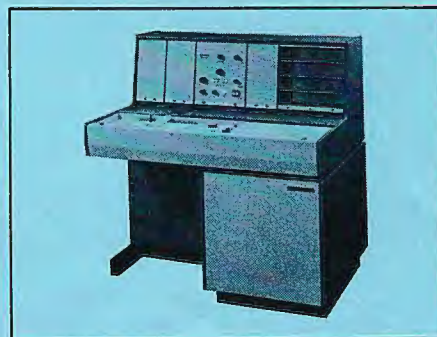
A pulsed-Fourier transform or frequency swept high resolution 23.5-kG spectrometer. It features observing capability from 7 to 100 MHz, and decoupling range from 2.9 to 58 and 94 to 100 MHz for continuous wave, noise decoupling or INDOR experiments.

## NV-14



A moderately priced 14-kG,  $^{13}\text{C}$  research spectrometer offering the highest performance per dollar of any NMR system now available. Accessories include Fourier transform for  $^{13}\text{C}$ , and other nuclei such as  $^1\text{H}$ ,  $^{19}\text{F}$  and  $^{31}\text{P}$ . It is a solid state, frequency swept spectrometer ideal for either high performance routine tasks, or for those research requirements demanding state-of-the-art capabilities along with versatile lock, spin decoupling, and variable temperature capability using 8mm sample tubes.

## System T-60



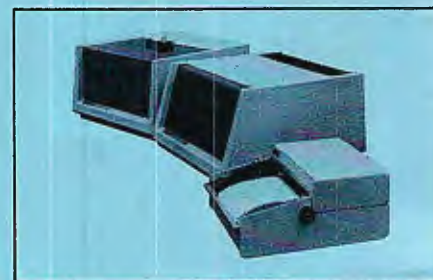
The most versatile instrument in the low-to-moderately priced end of Varian's NMR line, the T-60 features high performance and modular accessories. The basic instrument is excellent for routine applications. Accessories such as  $^{19}\text{F}$ ,  $^{31}\text{P}$ , variable temperature and spin decoupling expand its capability into many complex research applications.

## EM-360



A 60-MHz instrument which embodies Varian Anaspect's expertise in producing low cost spectrometers and Varian technical leadership in resonance spectroscopy. The result is an instrument which is far ahead of the field in combining economy and performance, flexibility and simplicity. The newest Varian NMR system, it's the low cost leader in the 60-MHz field, yet it delivers high priced performance. Guaranteed sensitivity of 18:1 signal-to-noise and 0.6-Hz resolution are performance features which suit this instrument to a wide variety of routine and research applications.

## EM-300



A rugged, low priced 30-MHz spectrometer giving excellent results with minimum effort. The EM-300 can easily handle routine proton applications. The instrument's ability to offset, expand or integrate have made it the preferred system in hundreds of teaching laboratories. Other owners have found the EM-300 to be just as useful in simple, routine control applications.

varian instruments



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