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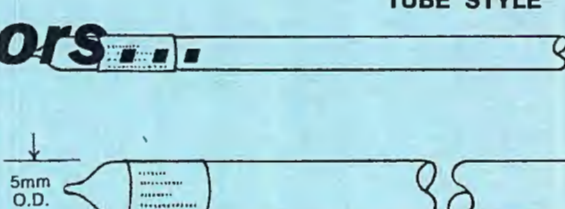
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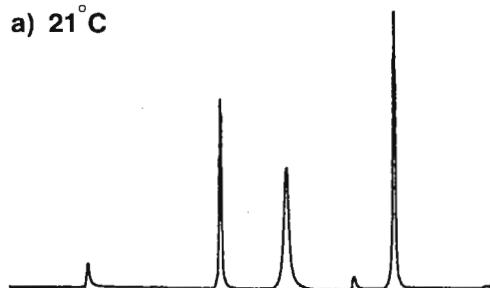
Dr. Bernard L. Shapiro  
 Department of Chemistry  
 Texas A&M University  
 College Station, Texas 77843 U.S.A.

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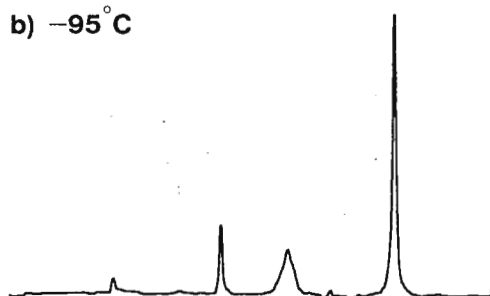
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# Variable Temperature CP-MAS with the GX Series FT NMR Spectrometers

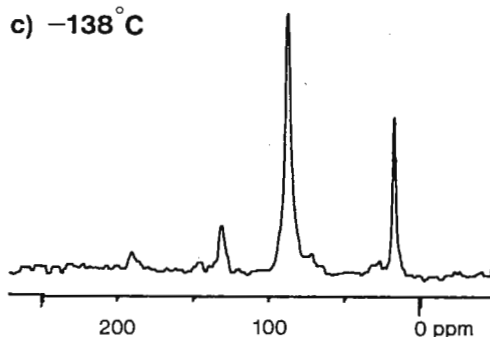
a) 21°C



b) -95°C



c) -138°C



<sup>13</sup>C (50.1 MHz) VT/MAS spectra of hexamethylbenzene. a) and c) <sup>1</sup>H-<sup>13</sup>C cross polarization. b) Bloch decay. The peak at ~90ppm is due to the Delrin rotor.

# JEOL

235 Birchwood Ave., Cranford, NJ 07016  
 (201) 272-8820



May 22, 1983

 $^{13}\text{C}^2\text{H}$  Polarization Transfer Studies

Dear Barry,

Previously we reported the utility of  $^{13}\text{C}^2\text{H}$  INEPT<sup>1,2</sup>. In our continued studies, we have observed  $^{13}\text{C}^2\text{H}$  DEPT and investigated the mechanistic aspects of polarization transfer from a nucleus of spin 1 to determine the optimal experimental conditions. INEPT<sup>3</sup> and DEPT<sup>4</sup> were originally intended to enhance the intensity of an insensitive observe spin by tapping a reservoir spin with a high  $\gamma$ . However, they can be used to provide spectral selectivity by transferring polarization from the spin of an atom (otherwise rare) synthetically attached to an atom whose NMR signal is desired. In this manner, we have selectively observed the spectra of deuterated carbons while suppressing resonances of other carbons by at least four orders of magnitude<sup>1,2</sup>.

Using a reservoir spin with  $I > 1/2$ , such as  $^2\text{H}$ , results in two significant differences from the original polarization transfer experiment. Polarization transfer from a spin where  $I = 1/2$  with a lower  $\gamma$  would be expected to diminish, not enhance, the intensity of the observe spin by a factor of  $\gamma_I/\gamma_S$ . However, since  $^2\text{H}$  has  $I = 1$ , there exists a greater energy difference between the levels effective in polarization transfer and thus a greater population difference to tap. Thus for a single pulse experiment as a result of the equilibrium condition, the coupled  $^{13}\text{C}^2\text{H}$  INEPT signal is only slightly less intense than that of the normal  $^1\text{H}$ -coupled  $^{13}\text{C}$  ( $4\gamma_D/3\gamma_C$ ). The  $^{13}\text{C}^2\text{H}$  polarization transfer experiment does enhance the observe signal with multiple acquisition, since the repetition rate depends on the recovery of the  $^2\text{H}$  (not  $^{13}\text{C}$ ) magnetization. Due to its quadrupolar nature,  $^2\text{H}$  generally has relaxation rates an order of magnitude faster than  $^{13}\text{C}$  allowing for a 10 times faster pulsing rate.

The greater spin also affects the refocussing of the observe spin by the pulse sequence. The timing of the polarization transfer is the same with either  $^1\text{H}$  or  $^2\text{H}$  as the reservoir, since in this period, the  $^{13}\text{C}$  spin acts on either spin system identically to align them antiparallel. However, since  $^2\text{H}$  has a higher spin than  $^1\text{H}$ , carbons coupled with deuterium have greater spectral multiplicity and, therefore, faster precessional rates in terms of the coupling constant during the refocussing period. As can be seen in Figure 1, this requires shorter refocussing delays (in inverse  $J$  units<sup>5</sup>) in the INEPT sequence and Figure 2 smaller  $\Theta$  in the DEPT sequence than in the corresponding  $^{13}\text{C}^1\text{H}$  polarization transfer experiments. Optimum values for  $\text{CD}$ ,  $\text{CD}_2$ , and  $\text{CD}_3$  as well as best compromise values are given in Table I. Greater detail is available in a forthcoming article<sup>6</sup>.

Peter L. Rinaldi  
Assistant Professor

Nicholas J. Baldwin  
Research Associate

- 1) P.L.Rinaldi and N.J.Baldwin, TAMU-NMR Newsletter, 288, 43(1982)
- 2) P.L.Rinaldi and N.J.Baldwin, J. Am. Chem. Soc., 104, 5791(1982)
- 3) G.A.Morris and R.J.Freeman, J. Am. Chem. Soc., 101, 760(1979)
- 4) D.M.Doddrell, D.T.Pegg and M.R.Bendall, J. Mag. Reson., 48, 323(1982)
- 5) Although not shorter in time, since  $J_{\text{CD}} \approx 1/7 J_{\text{CH}}$
- 6) P.L.Rinaldi and N.J.Baldwin, J. Am. Chem. Soc., submitted

Table 1. Delay times and pulse widths for CD polarization transfer.

Experiment	Optimum Values				
	CD	CD <sub>2</sub>	CD <sub>3</sub>	CD <sub>n</sub>	$\tau$
INEPT ( $\Delta$ )	$1/8J$	$3/40J$	$1/16J$	$3/40J$	$1/4J$
DEPT ( $\Theta$ )	$\pi/4$	$3\pi/20$	$\pi/8$	$3\pi/20$	$1/8J$

Figure 1

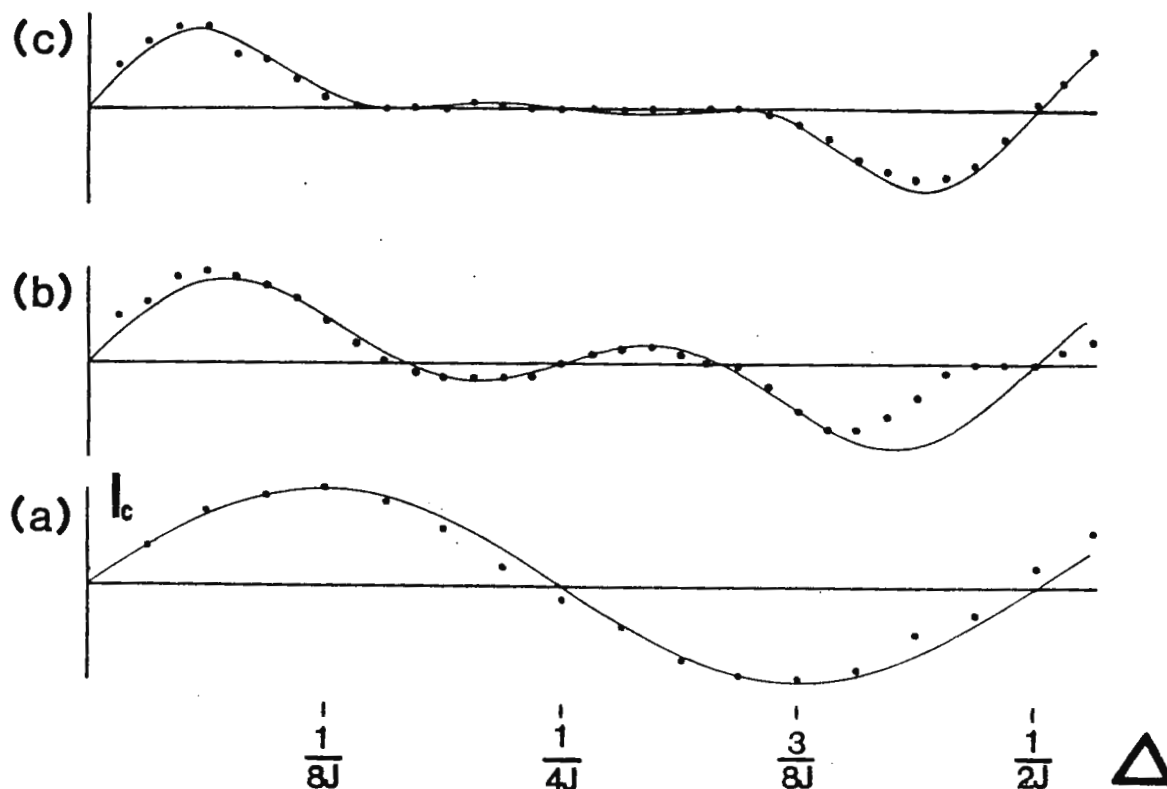


Figure 2

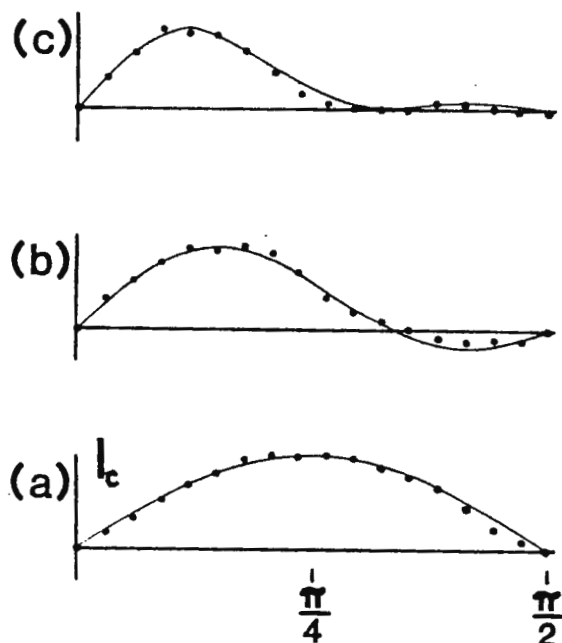


Figure 1. Carbon intensity plotted as a function of the final delay ( $\Delta$ ) in  $^{13}\text{C}\{^2\text{H}\}$  INEPT spectra of (a) benzene- $\text{d}_6$  (CD), (b) methylene dichloride- $\text{d}_2$  ( $\text{CD}_2$ ) and (c) acetone- $\text{d}_6$  ( $\text{CD}_3$ ). All spectra were taken at 50 MHz with  $^2\text{H}$  pulsed and decoupled at 30 MHz; (—) calculated, (·) observed.

Figure 2. Carbon intensity plotted as a function of the final decoupler pulse width ( $\Theta$ ) in  $^{13}\text{C}\{^2\text{H}\}$  DEPT spectra of the same species as in Figure 1. Theoretical curves are sums of weighted contributions of individual components of multiplets modulating the observe signal. From these, the equality  $\Theta = \pi J \Delta$  is noted as for other systems by D.T. Pegg et al, *J. Magn. Reson.*, **51**, 264(1983).





# The Guelph-Waterloo Centre for Graduate Work in Chemistry

Guelph Campus, Department of Chemistry, University of Guelph, Guelph, Ontario, N1G 2W1 519/824-4120

May 31, 1983

Dr. Barry Shapiro  
Texas A and M University NMR Newsletter  
Department of Chemistry  
Texas A and M University  
College Station, Texas 77843  
U.S.A.

Title: Solenoidal 20 mm probe for  $^{25}\text{Mg}$  observation on a Bruker WH-400

Dear Barry,

As you can see from the title, we have been interested in determining whether a solenoidal coil tuned to observe  $^{25}\text{Mg}$  at 24.477 MHz would offer any signal to noise advantages over our conventional, commercial 10 mm broadband probe. Our Bruker WH-400, which was installed three years ago, is equipped with the older design (i.e. non-digital) probes. On our instrument  $^{25}\text{Mg}$  is observed on a mid range broadband probe (13.5-55 MHz) which has an active volume of ca 2 mls. We have built a 20 mm solenoidal fixed frequency probe for our narrow bore magnet (ca 1.5 inch gap). This probe has a cylindrical sample container machined out of plexiglass which contains ca 3 mls of solution. We shimmed this probe on the FID of  $\text{H}_2\text{O}$  using the method described by Joe Ackerman and coworkers (J.Mag.Res. 42, 498 (1981)). We typically stopped shimming when the linewidth of the  $\text{H}_2\text{O}$  resonance was just less than twenty Hz. A comparison between the signal to noise obtained on this probe and on our commercial probe is shown on the accompanying figure. We find that there is about a factor of 2.3 in S/N between the two. Clearly some part of this difference can be ascribed to the larger active volume in the solenoidal coil. We feel that even though we can optimize our solenoidal design even further to give us better filling factors, our initial results are very encouraging.

We will be happy to supply any details of our design and circuit diagrams to anyone who is interested.

Sincerely yours,

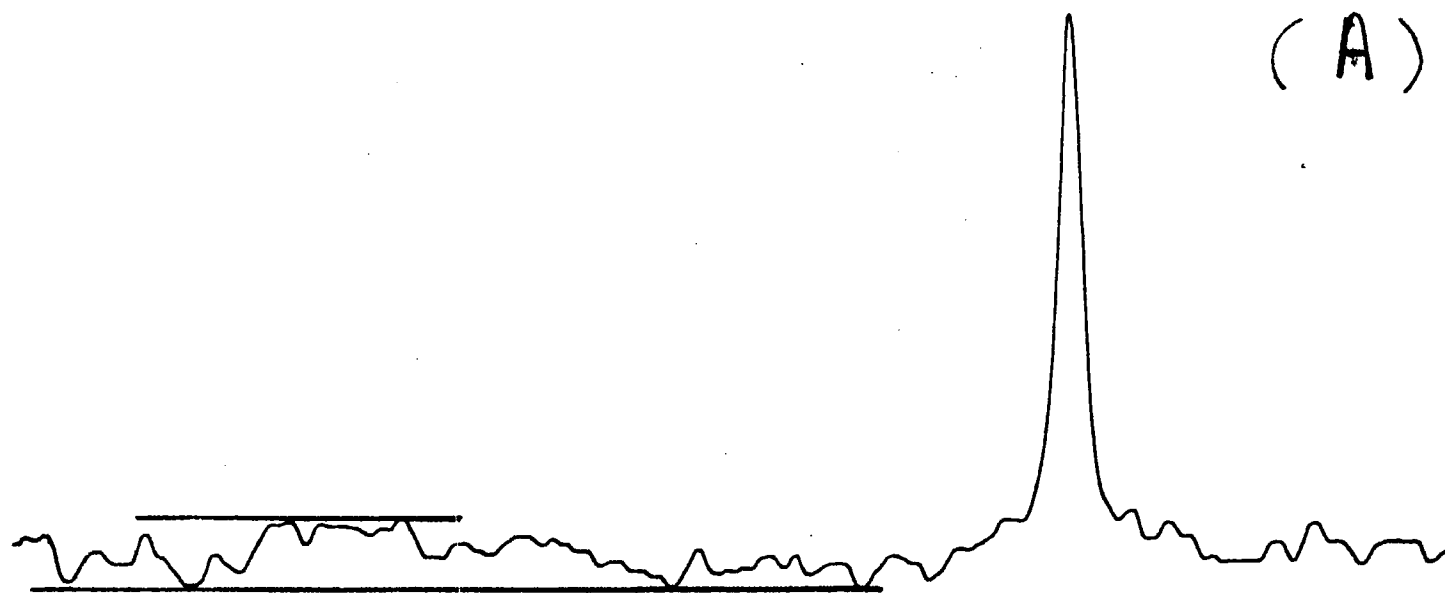
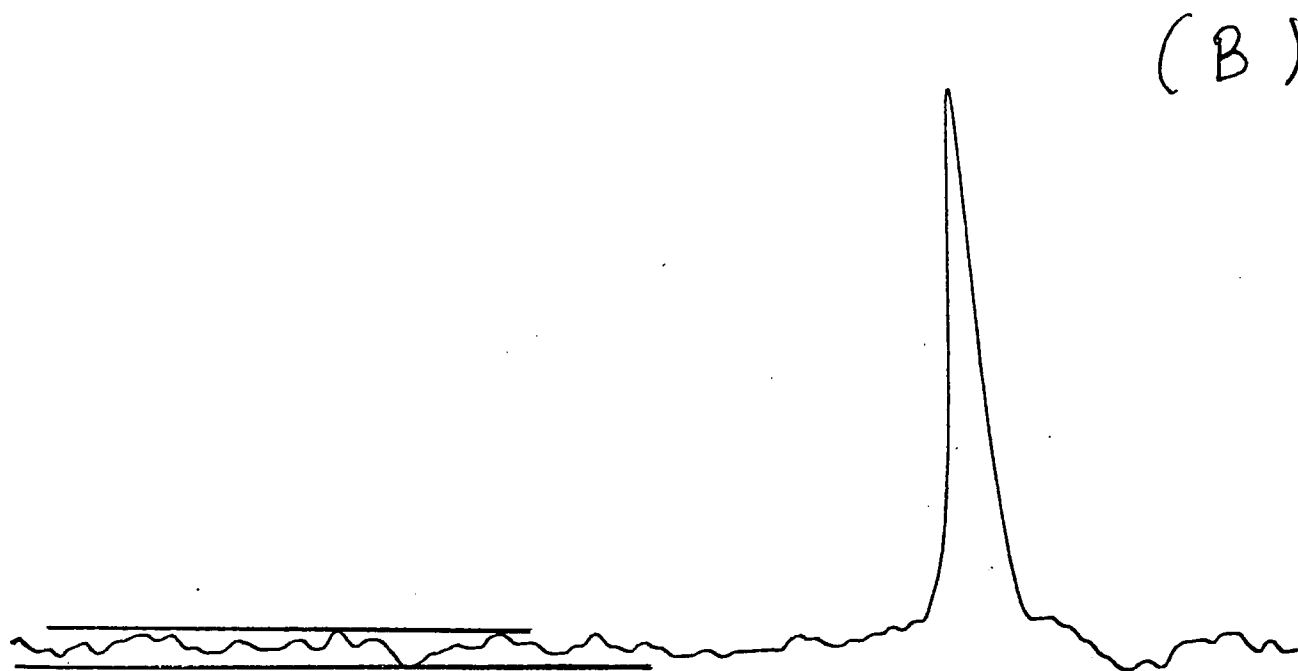
R.E. Lenkinski  
Manager

I.J. McLennan



/la  
Encl.

SOUTHWESTERN ONTARIO NMR CENTRE



$^{25}\text{Mg}$  NMR SPECTRA OBTAINED ON THE SAME SOLUTION  
OF  $\text{MgCl}_2$  USING (A) THE BRUKER COMMERCIAL PROBE  
AND (B) OUR HOME BUILT SOLENOIDAL PROBE.

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Theix, le

Références à rappeler :

Prof. B.L. SHAPIRO  
 Departement of Chemistry  
 College of Sciences  
 Texas University  
 College Station TX 77843

U.S.A.

Collagen hydratation  
 Studies of structural transitions

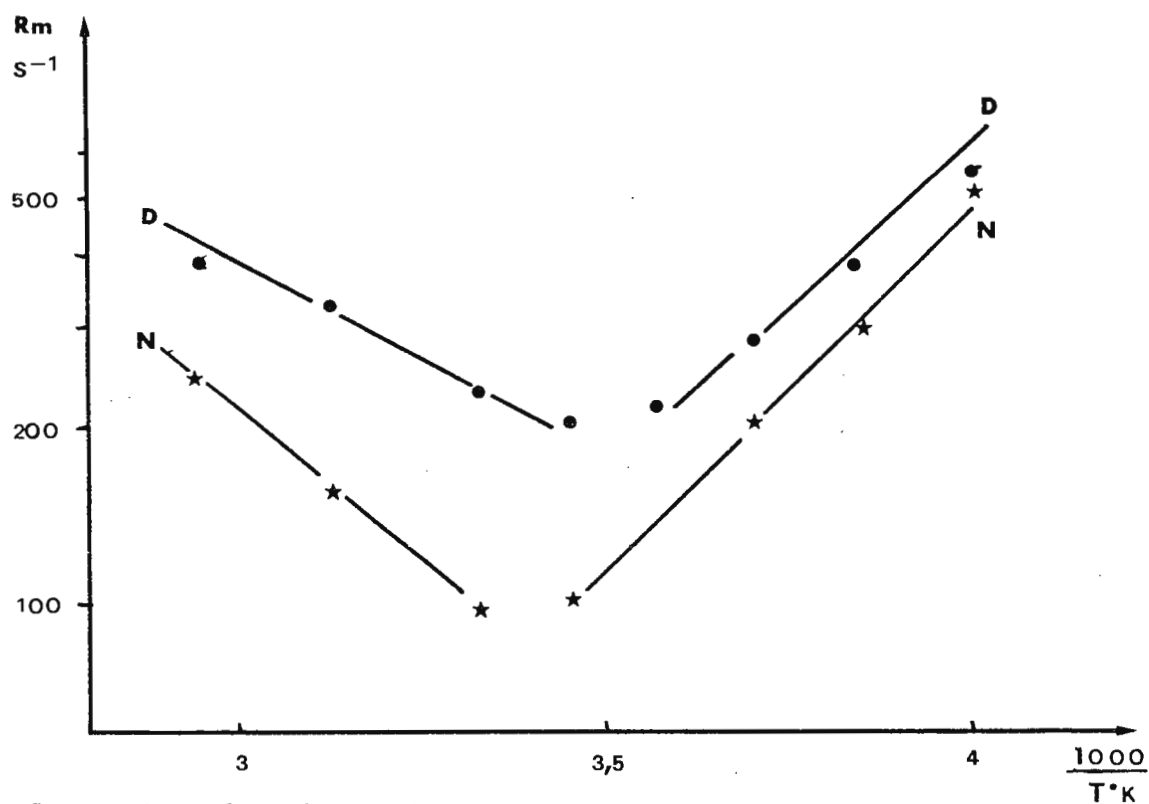
In our study of water collagen system, the thermal degradation has also been attempted. An increase was observed in the relaxation rates ( $R_1 = T_1^{-1}$ ) of the water deuterons upon the transition from native collagen fibers to gels of denatured collagen. This increase in  $R_1$  upon denaturation is irreversible. The "bound" water model, however, cannot explain the irreversibility of the denaturation effect. That stands in contrast to the renaturation experiments measured by optical rotation. According to Woessner and Snowden, an order along and close to the polymeric surface can provide a long correlation time. The denaturation of collagen might reflect the transition from a system containing the bundles of parallel tropocollagen molecules to a system containing the random coils of the denatured  $\alpha$ -chains.

The magnetization exchange rate  $\tau_2^{-1}$  - across the phase boundary from the bound water protons to collagen peripheral protons - is the dominant contribution to the cross relaxation rate -  $R_m \simeq A\tau_2^{-1}$  - at temperature  $T > 280^\circ\text{K}$ .

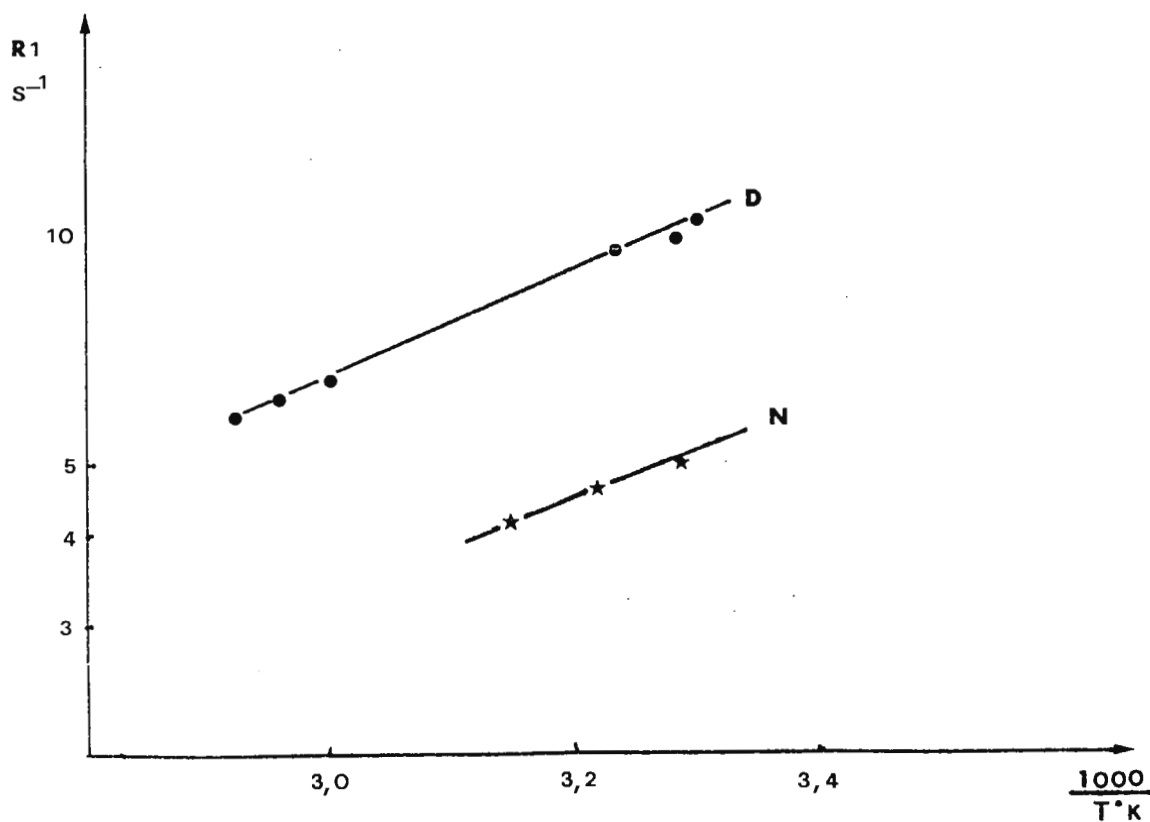
For the denatured sample, the cross relaxation rate is higher than that of the native collagen sample. It means that  $\tau_2$  becomes smaller. It's in good agreement with the long correlation time along the polymeric surface.

J.P. RENOU





Temperature dependance of water proton cross relaxation rate for collagen samples (16 mg H<sub>2</sub>O/g collagen) N : Native D : Denatured



Temperature dependence of water deuteron relaxation rate for collagen samples (1 g H<sub>2</sub>O/g collagen). N : Native D : Denatured

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June 1, 1983

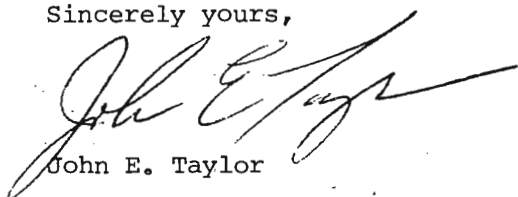
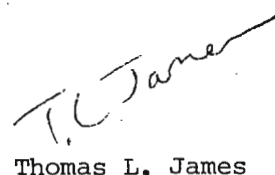
Professor B.L. Shapiro  
Department of Chemistry  
Texas A & M University  
College Station, Texas 77843Re: DNA Internal Dynamics from  $^{15}\text{N}$  NMR Relaxation

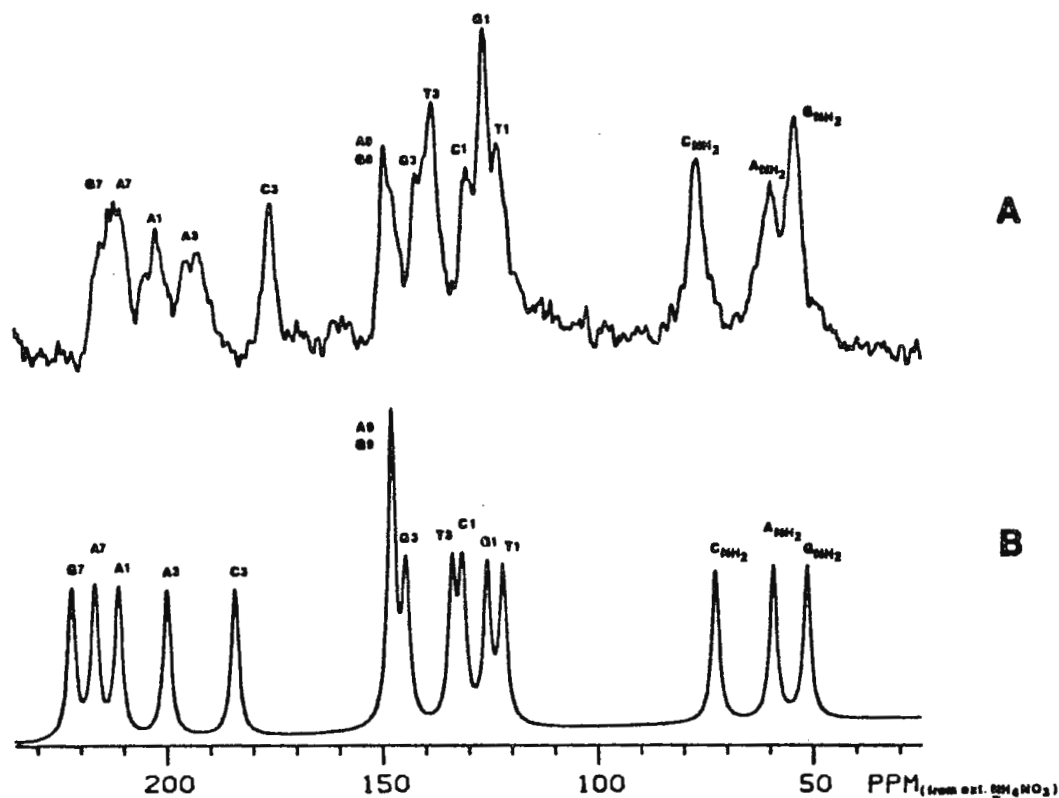
Dear Professor Shapiro:

We have extended our initial observations of the  $^{15}\text{N}$  spectrum of DNA (1-3) to include relaxation measurements of each resonance at 3 field strengths (2.35, 5.64, 11.75 T). Using standard bond lengths and angles (4), we have calculated the spectral density functions and relaxation parameters ( $T_1$ , NOE, and  $T_2$ ) (5) for isotropic and anisotropic reorientations with free, restricted, and jump internal motions. We find that isotropic models as a group and wobbles on a cone are inappropriate for short DNA fragments. Modeling our 200 +/- 50 base pair fragment as an anisotropic cylinder with internal motions has, however, provided the amplitude and frequency range for the base dynamics as they occur in these nucleic acid fragments.

We subsequently explored the dynamic motions of DNA by a lattice formalism which allows one to "jump" between varying structures of nucleic acids (6). The structures utilized were the result of molecular mechanics studies on poly(dAdT), tetranucleotides, and duodecamer structures (7). The lattice formalism precludes the superposition of geometric constructs on a molecule with many degrees of freedom and our initial success in delineating motions difficult to describe by geometric boundary formalisms attests to this. We find that helical twisting motions are inadequate alone but that base tilting motions will account for the  $^{15}\text{N}$  data. We feel therefore that the combined use of multiple frequency data and lattice model calculations will open a new window into the complexities of nucleic acid dynamics.

Sincerely yours,

  
John E. Taylor  
Joe W. Keepers  
Thomas L. James



- (A)  $^{15}\text{N}$  NMR spectrum (24.4 MHz) obtained at 20°C of  $^{15}\text{N}$ -labeled DNA (27 mg/ml) in 40 mM cacodylate, pH 7.2, and 40 mM NaCl; 10,000 transients were accumulated with 700 pulses using an acquisition time of 0.65 sec., a pulse recycle time of 3 sec., and broadband proton decoupling on only during signal acquisition (10 Hz line-broadening applied).
- (B) Simulated  $^{15}\text{N}$  spectrum of DNA composed from individual nucleotide spectra.

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3. Taylor, J.E., Keepers, J.W., James, T.L., (1982) Int'l Symposium on Structures and Dynamics of Nucleic Acids and Proteins-Collected Abstracts, La Jolla, CA.
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5. Keepers, J.W. and James, T.L., (1982) *JACS*, **104**, 929.
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(b) Kollman, P.A., Keepers, J.W., and Weiner, P., (1982) *Biopolymers*, **21**, 2345.

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June 8, 1983

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

DEPT(1) on the Bruker WH-200

Dear Barry:

Recently we tried some  $^{13}\text{C}$  spectral editing on our Bruker WH-200 with DEPT using 2-butanol (90% in ~10%  $\text{CDCl}_3$ ). Spectrum B was taken to enhance only CH resonances with the microprogram:

1. ZE
2. D1 S1 D0
3. (P1 PH1 D2):D P7 PH7
4. (P2 PH2 D2):D P5 PH5
5. (P3 PH3 D2):D P6 PH6
6. G0 = 2 BB
7. D0

} "Seq. A"

8. EXIT (We include P7 for Boltzmann suppression.)

To enhance only  $\text{CH}_2$  resonances with one microprogram, two "Seq.A"s were separated with an NM (negate memory) command with P4 replacing P3 in the second "Seq. A" resulting in spectrum C. In the second "Seq. A" the 'H decoupler was apparently pulsing in the noise modulation mode, thus PH1-PH3 has no meaning. To cure this problem, we simply add a "dummy" D3 S2 CW command between the two "Seq. A"s resulting in spectrum D.

Sincerely,

*Tom Nakashima*

Tom Nakashima

*Ted McClung*  
Ted McClung

TN:sjm

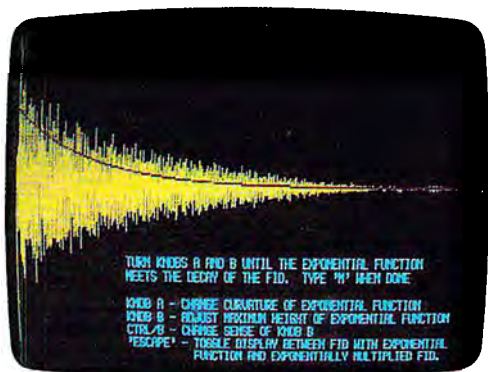
1. D.M. Doddrell, D.T. Pegg and M.R. Bendal, J. Mag. Res., 48, 323 (1982).



(cont. p. 12)



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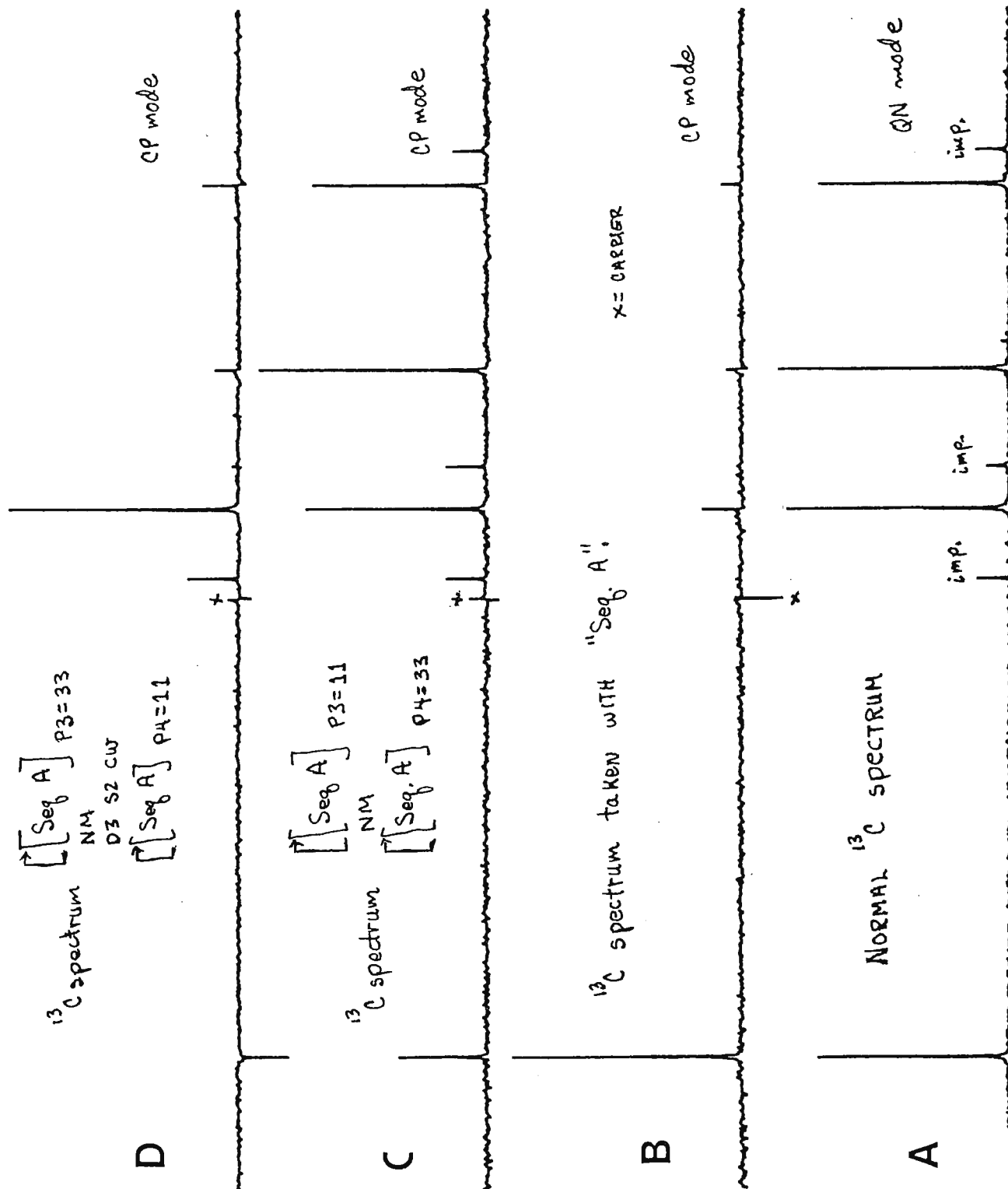
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Peter Stilbs  
Fysikalisk-Kemiska Institutionen  
Uppsala Universitet  
Box 532  
S-75121 Uppsala , Sverige

Tel: \*\*\*46-18-173646

Uppsala 15 June 1983

Professor B.L. Shapiro  
Department of Chemistry  
Texas AM University  
College of Science  
College Station  
Texas 77843  
U.S.A.

" Multinuclear Superiority "

Dear Professor Shapiro :

Thank you for your blue-green reminder. In response , I will report some recent results , achieved on our new multinuclear setup on the FX-100.

We have now three weapons ; fully multinuclear , first-strike Fourier transform Pulsed-Gradient Self-Diffusion capability with high sensitivity , fully stable long-term data acquisition with internal or external lock, automated fully component-resolved proton and carbon-13 self-diffusion coefficient determination capability , simultaneous spin-lattice and self-diffusion determination.....

Sorry , i'll start again.

Amongst our weaponry, are now such diverse elements as : Fourier transform self-diffusion capability on all nuclei , high-sensitivity long-term data acquisition capability , a fanatical devotion to Hahn , Stejskal and Tanner .....

No , it's no good . I wish to apologize to M. Palin , J. Cleese , E. Idle, T. Jones, G. Chapman and Cardinal Richelieu for the misuse of their production "Another Monty Python Record" , (Charisma CAS-1049 (1971)) as a basis for this letter.

And now for something completely the same ; a stacked plot on a Lithium-7-based, self-diffusion measurement on a dilute protein solution in heavy water is illustrated in Figure 1. The measurements were made overnight at a 1 millimolar lithium ion concentration. Lithium ions bind to the protein ; the extent of this binding is reflected in their time-averaged self-diffusion coefficient .

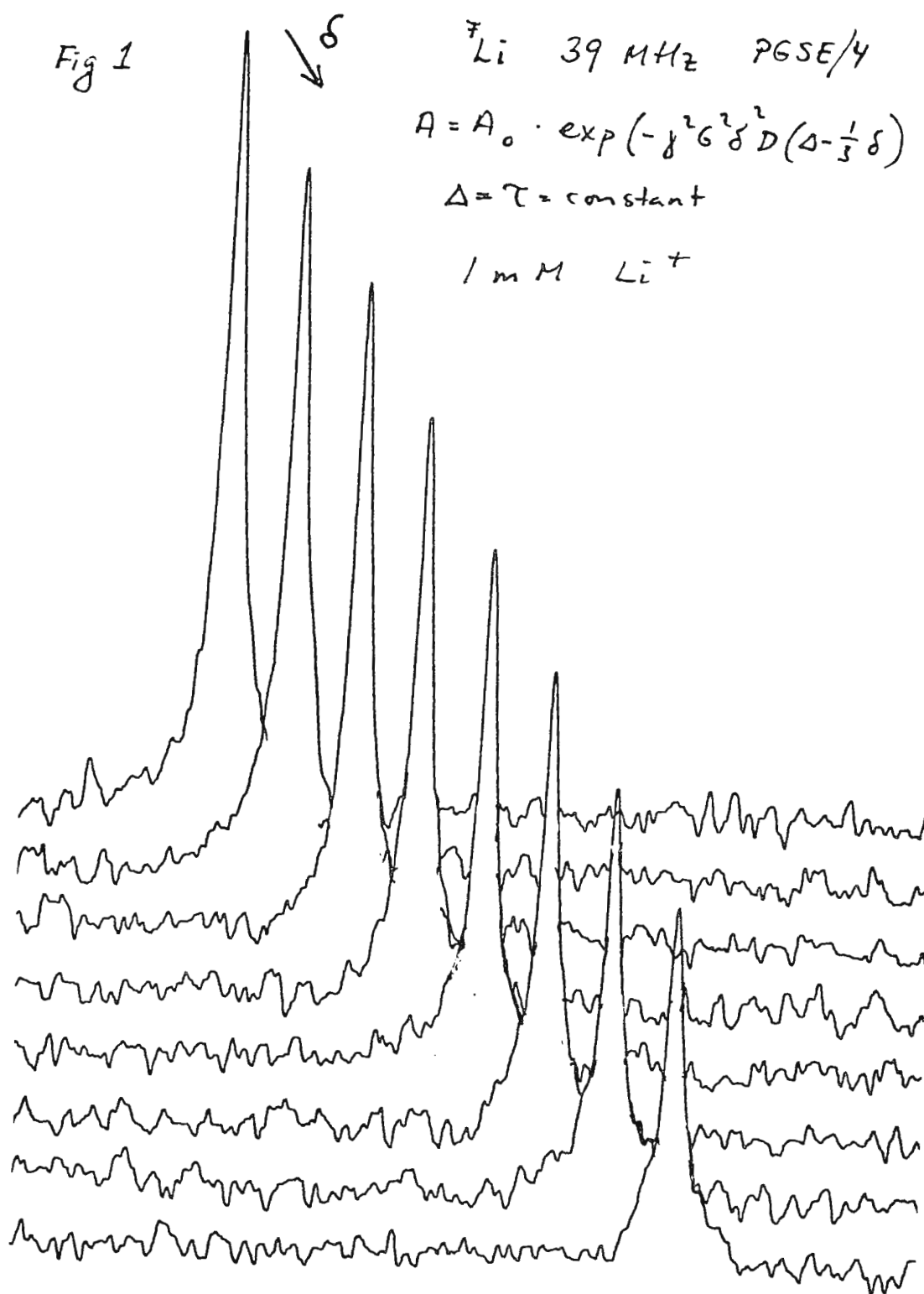
We hope to provide a more coherent report on the progress of this and similar measurements in the near future.

Yours sincerely,

*Peter Stilbs*

Peter Stilbs





**Standard Oil Company (Indiana)**

Amoco Research Center  
Post Office Box 400  
Naperville, Illinois 60566  
312-420-5111

June 17, 1983

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

Dear Barry:

Multinuclear FTMAS on a High Resolution Spectrometer

Last year we added a broadband Nicolet MAS probe to our wide bore multinuclear NIC-200 spectrometer in order to study zeolites. The  $^{29}\text{Si}$  and  $^{27}\text{Al}$  spectra of a NaX zeolite shown in Figures 1 and 2 illustrate the type of spectra we have been able to achieve with our system even though we are only using the rf power amplifiers that came as standard equipment on our high resolution NIC-200. Of course, the requirements to observe Si and Al are not very demanding. To further test the probe, which has a specified tuning range of 30-80 MHz, we selected CsCl and  $\text{Na}_3\text{PO}_4$  because  $^{133}\text{Cs}$  has a resonance frequency of 26.2 MHz and phosphorus has a resonance frequency of 81 MHz. We easily detected both of these resonances as shown in Figures 3 and 4. The  $^{133}\text{Cs}$  was a bit difficult because of its very long  $T_1$ , which we estimated by progressive saturation to be about 600 s, but its high sensitivity allowed it to be seen in a single scan.


Because of the relatively low rf power of a high resolution spectrometer,  $90^\circ$  pulses tend to be too long to insure uniform excitation of broad resonance lines. For example, the central transition of the  $^{11}\text{B}$  resonance of  $\text{B}_2\text{O}_3$  has a linewidth of about 25 KHz at 64.2 MHz. Our spectrometer has a  $90^\circ$  pulsewidth of 20  $\mu\text{s}$  at this frequency. Therefore a 7  $\mu\text{s}$  ( $30^\circ$ ) pulse was used to obtain the static spectrum shown in Figure 5.

In order to increase both the signal to noise and frequency range of our probe, we replaced the standard 3-turn coil for our 10-mm spinner with a 7-turn coil. At silicon frequencies this resulted in a two-fold increase in sensitivity. At the same time, we were able to observe the  $^{91}\text{Zr}$  resonance of  $\text{ZrPO}_4$  at 18.7 MHz as is shown in Figure 6.

Sincerely,

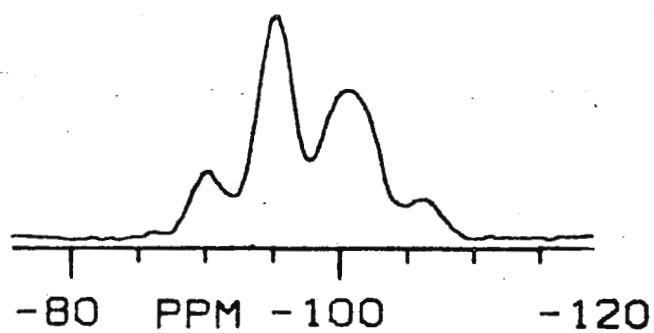
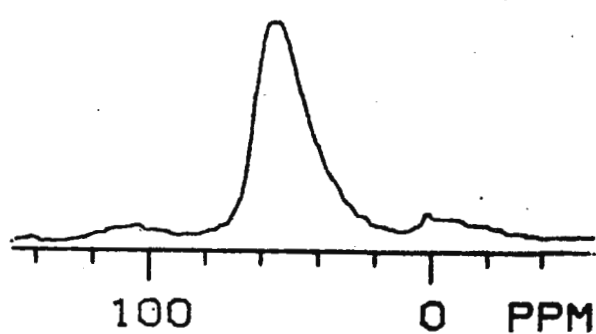
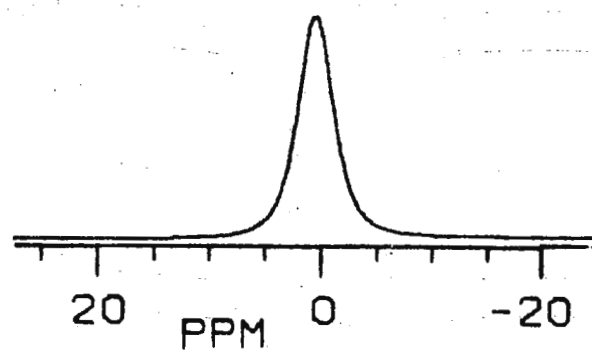
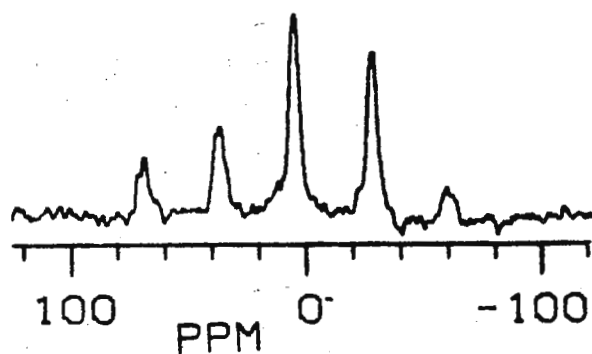
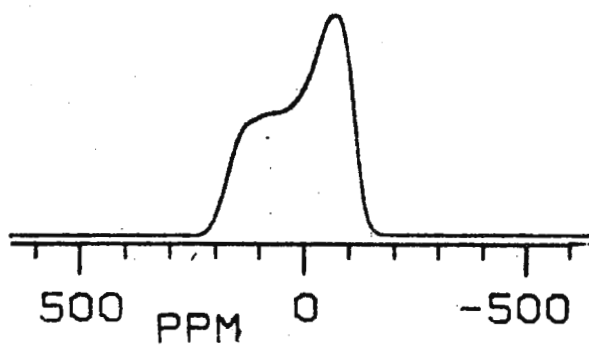
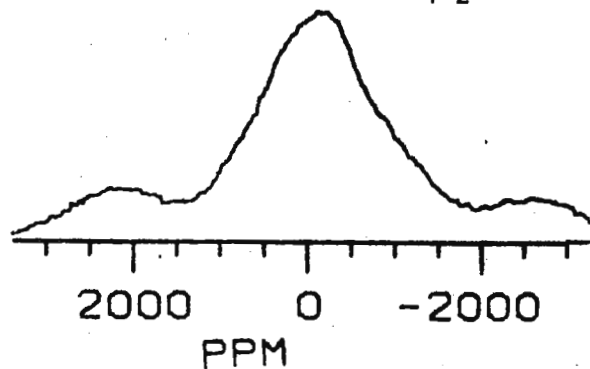


G. J. Ray, B-5  
(312) 420-5217



R. L. Hemmig, F-9  
(312) 420-5154

jh  
Attachment

Fig 1  $^{29}\text{Si}$  MAS (Zeolite)Fig 2  $^{27}\text{Al}$  MAS (Zeolite)Fig 3  $^{133}\text{Cs}$  MAS ( $\text{CsCl}$ )Fig 4  $^{31}\text{P}$  MAS ( $\text{Na}_3\text{PO}_4$ )Fig 5  $^{11}\text{B}$  Static ( $\text{B}_2\text{O}_3$ )Fig 6  $^{91}\text{Zr}$  ( $\text{Zr}(\text{HPO}_4)_2$ )



Carleton University  
Ottawa, Canada K1S 5B6

June 9, 1983.

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

Title:  $^{13}\text{C}$  NMR In Vivo Detection of Cryoprotectants and Lipids  
In Overwintering Larvae

Dear Barry:

One of the facts of life in this part of Canada (as you know) is the long and frigid winter. Despite temperatures of  $-30^{\circ}\text{C}$  or lower, however, many insect larvae survive and proceed to pupation in the spring. Recently we have been collaborating with Professor Ken Storey of Carleton's Institute of Biochemistry regarding in vivo NMR studies of some overwintering species, specifically the 3rd instar larvae of the goldenrod gall fly, Eurosta solidaginis.

The attached  $^{13}\text{C}$  spectrum represents about 10 minutes work on the XL-200. The 10 mm tube contained about 50 live larvae gently deposited to a depth of about 3 cm. It is clear that  $^{13}\text{C}$  NMR represents a rapid qualitative screening tool for carbonaceous components in these species.

Storey's group have analyzed the larvae and find that cold acclimation causes rapid build up of the cryoprotectant polyols glycerol and sorbitol to levels near 250 mM. Resonances in the range of 65-76 ppm are primarily due to these components.

Also, high concentrations of triglycerides are evident from the  $^{13}\text{C}$  spectrum. A spectrum of a chloroform soluble larval extract confirms this, and there appears to be only one  $\text{C}=\text{C}$  in the fatty acid chain, indicating the likely presence of oleic and/or palmitoleic acid esters.

Best regards,

mcs

Sincerely,

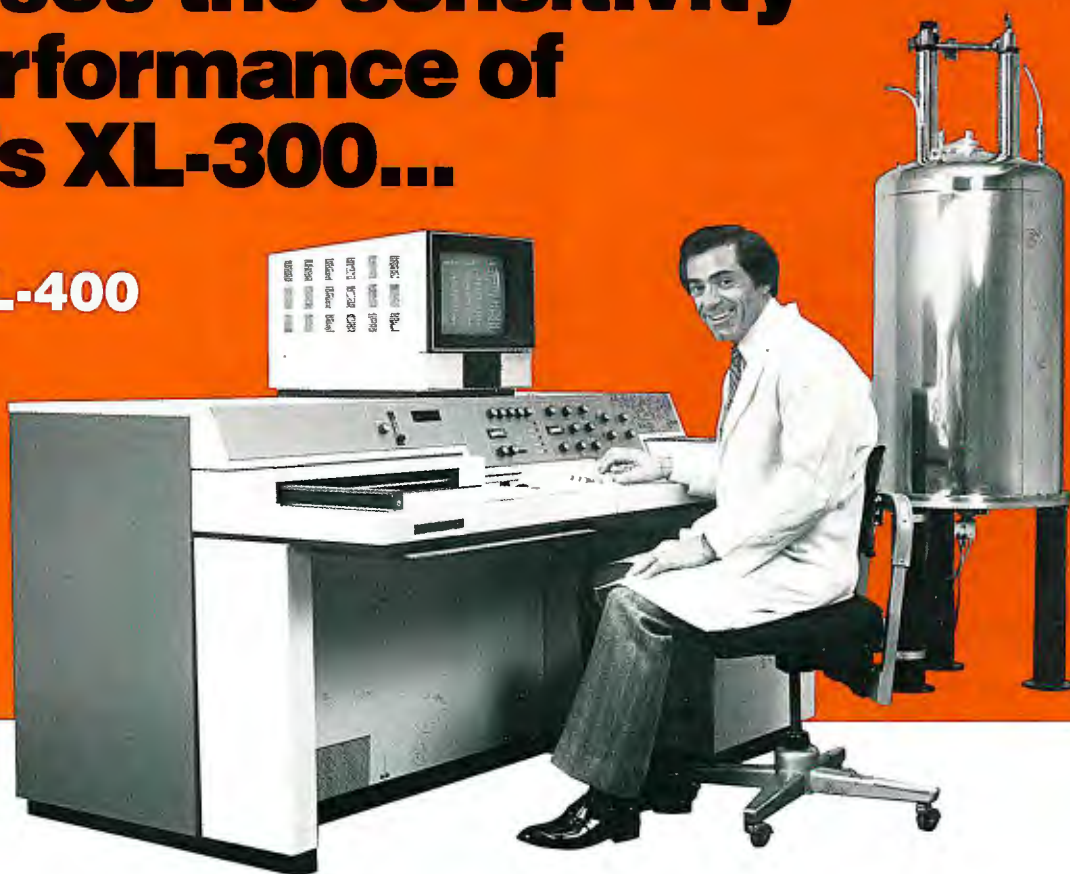
G.W. Buchanan, Professor of Chemistry.



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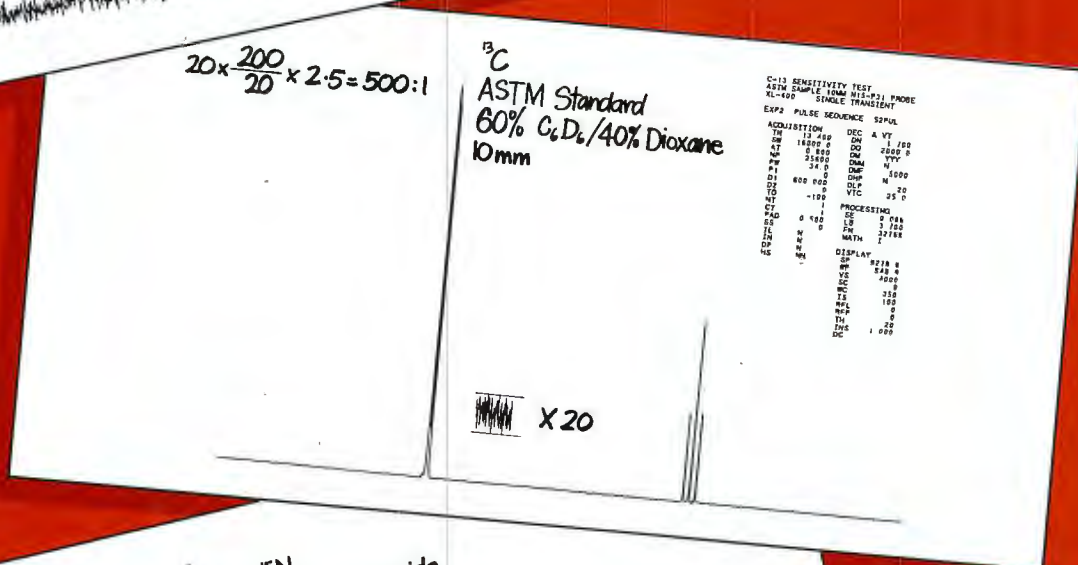
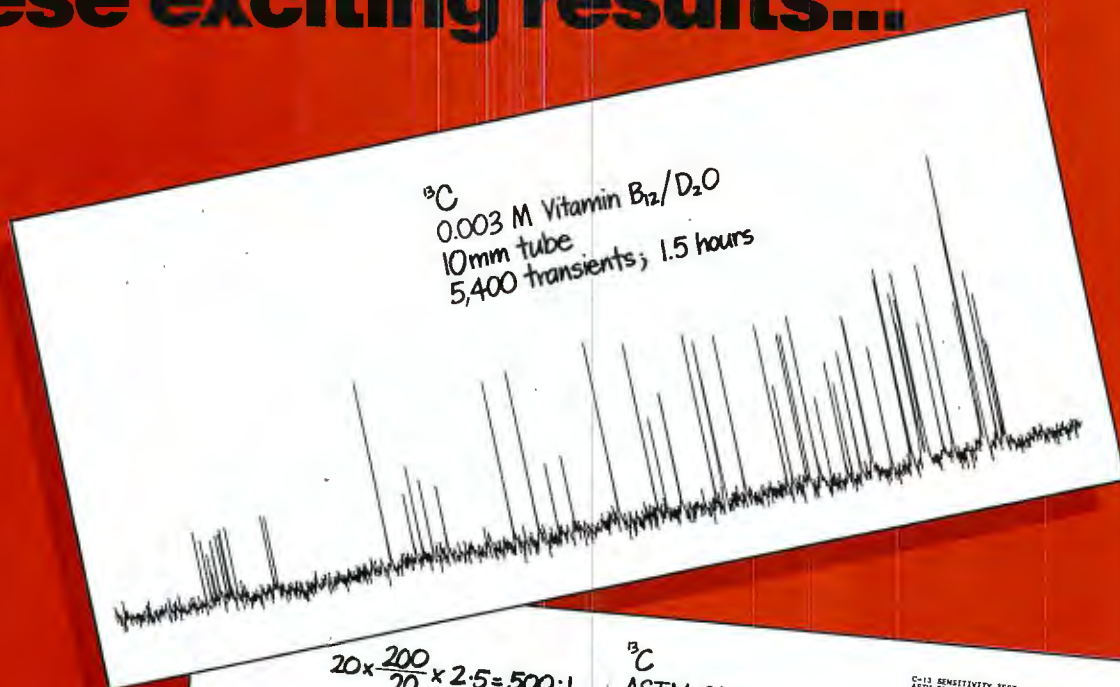
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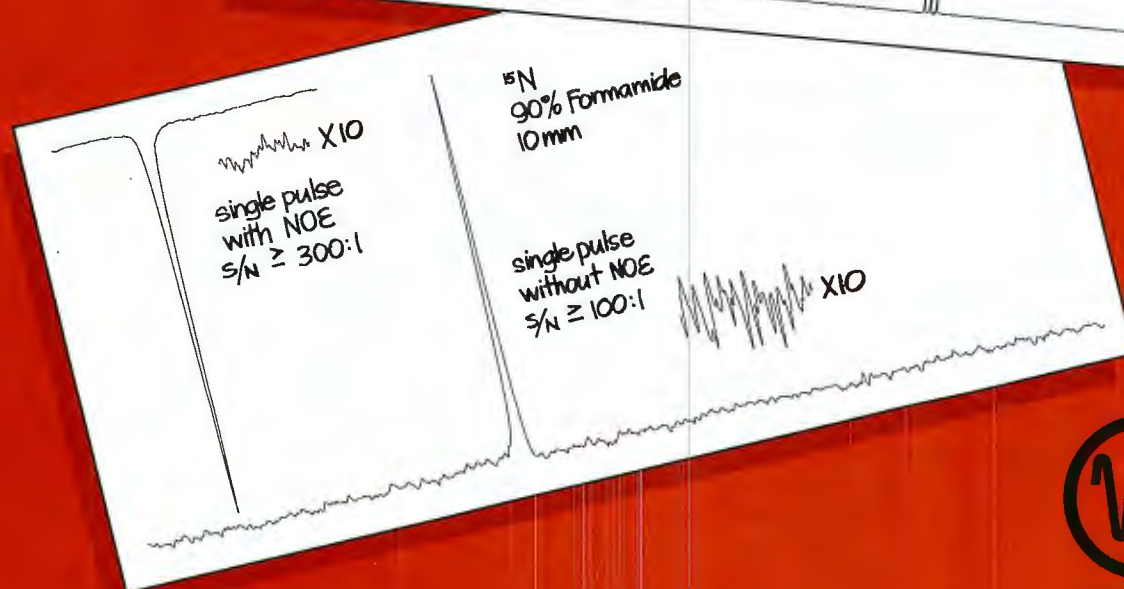


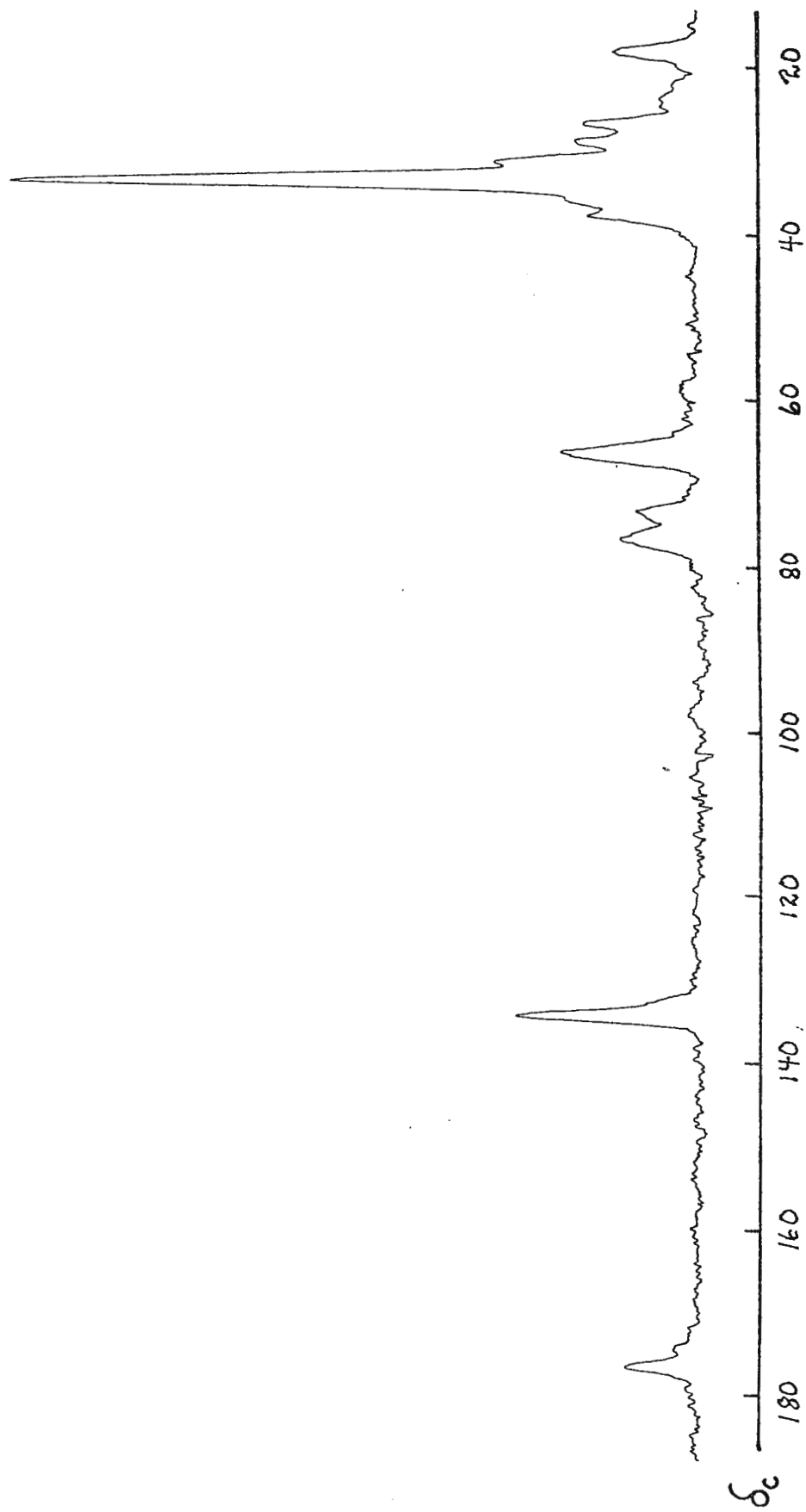
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$^{13}\text{C}$  (400 scans) Eurosta solidaginis larvae





UNIVERSITY OF GRONINGEN LABORATORY OF PHYSICAL CHEMISTRY  
NIJENBORGH 16, 9747 AG GRONINGEN, THE NETHERLANDS

Tel.: 050 -117087

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

GRONINGEN, 20 June 1983

Dear Barry,

Sequential NMR Assignments for a 14 base pair DNA duplex

During the last few years 2D NMR methods have caused a breakthrough in the study of biological macromolecules. Thus, for small proteins as Wüthrich has shown, it is now possible to make individual resonance assignments for 80-90% of the protons solely by NMR methods. In addition, mainly from 2D NOE, information on secondary and tertiary protein structure becomes available so that it will not be for long that an "NMR structure" of a protein can be established.

We have recently proposed a sequential assignment method for nucleic acids (JACS 105, 2914 (1983)). The method is based on 2D NOE spectra and works for right-handed helical nucleic acids of moderate size (say, up to 20 base pairs). Sequence information is present in 2D NOE spectra because cross-relaxation networks extend through the whole molecule, one for each strand of a duplex, involving the (deoxy)ribose and base protons.

An example is shown in the Figure, which shows part of a 500 MHz 2D NOE spectrum of a 14 base pair lac operator DNA fragment. Cross-peaks that link base protons (purine H8 and pyrimidine H6) with ribose H1' protons occur in region a and with H2' and H2'' protons in region b. A base proton makes a "contact" not only with the sugar protons of the same nucleotide but also, in a right-handed helix, with those of the neighbouring one at the 3'-phosphate. The assignment procedure amounts then to find the connectivities between cross-peaks in region a (base to H1'), which are reflected in region b (base to H2' and H2''). The positions in this latter region are in turn reflected in region c (H1' to H2' and H2'' in the same deoxyribose unit). In addition other cross-relaxation networks exist involving cytosine H5 and thymine methyl protons, so that a consistent set of assignments can be found as shown in the Figure for the upper strand of the 14 bp duplex. Thus, we have assignments for all purine H8, pyrimidine H6 and H5, thymine methyl, ribose H1', H2' and H2'' and in addition a number of H3' and H4' protons. Not bad for a non-symmetrical 14 bp DNA duplex (28 nucleotides!).

Best regards,

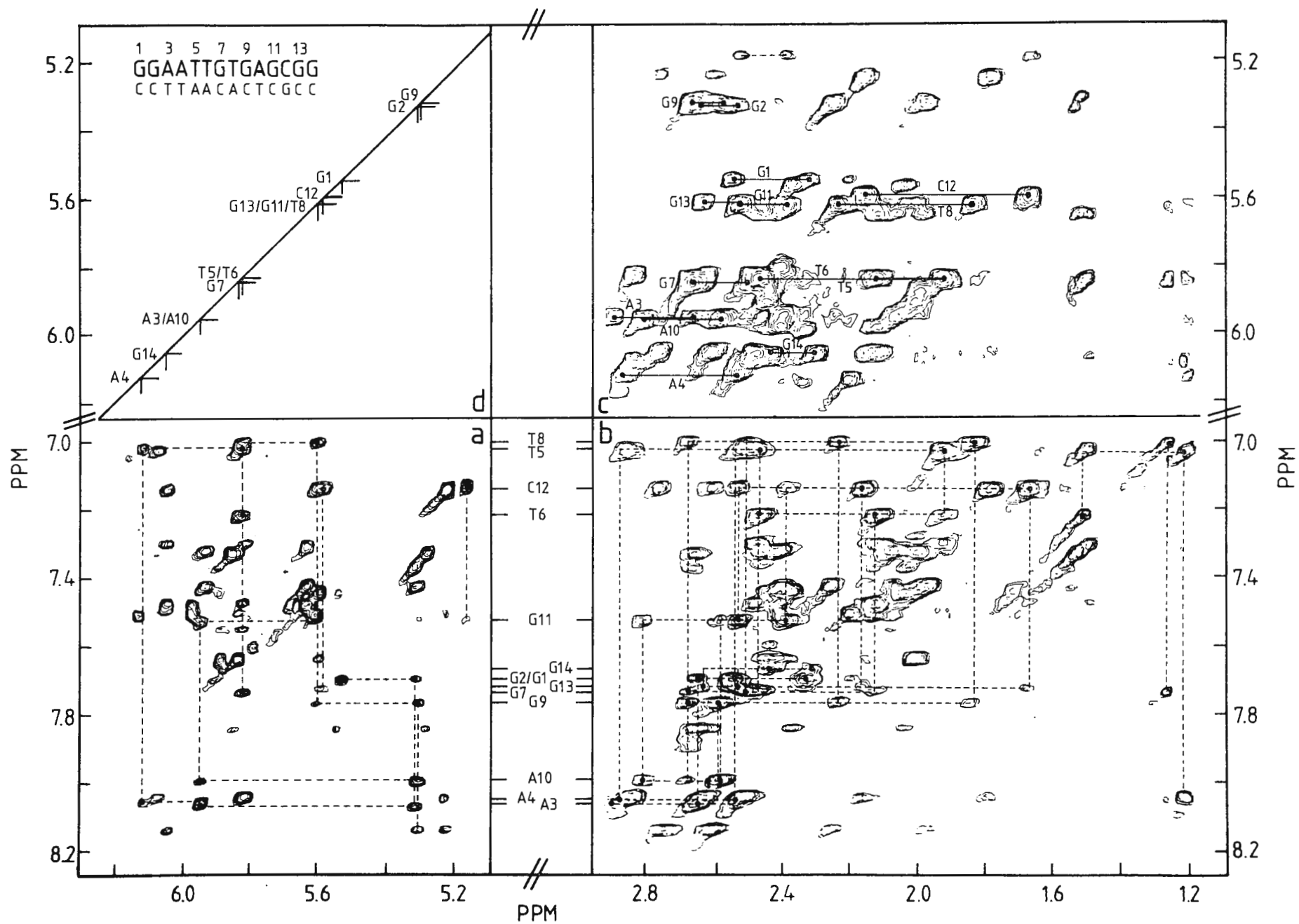
R. M. Scheek

R. Boelens

R. Kaptein

Please credit this contribution to the account of Dr. W. Weringa







UNIVERSITY OF MISSOURI-COLUMBIA

College of Arts and Science

Department of Chemistry

 123 Chemistry Building  
 Columbia, Missouri 65211  
 Telephone (314) 882-2439

June 20, 1983

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

Re: a) Chemical Shift  $^{13}\text{C}$ - $^1\text{H}$  Correlation Maps  
 b) Position Available

Dear Professor Shapiro:

Chemical shift  $^{13}\text{C}$ - $^1\text{H}$  correlation maps can be generated by a new pulse sequence:

$^1\text{H}$ :  $\pi/2(x)-t_1/2-\pi/2(y)-\tau-\pi(x)-\tau-\pi/2(y)-t_1/2-\tau-\pi/2(x)-\tau'$ -dec.  
 $^{13}\text{C}$ :  $-\pi(x)-$   $-\pi/2(x)-$  -acq.

where  $\tau=1/(2J_{\text{CH}})$  and  $\tau'\sim 0.3/J_{\text{CH}}$ . At the middle of the evolution time  $t_1$ , three  $^1\text{H}$  pulses have been added. Those protons which are directly attached to  $^{13}\text{C}$  nuclei (coupling  $J_{\text{CH}}$ ) are apparently not affected, while distant ones flip over, if two suppositions are valid:

- (i) one-bond coupling  $J_{\text{CH}}$  significantly exceeds all other couplings, and
- (ii) protons are weakly coupled.

Since the above pulse sequence selectively eliminates homonuclear ( $^1\text{H}$ - $^1\text{H}$ ) coupling from the  $F_1$ -dimension, correlation maps:

- (i) show better signal-to-noise ratios (see Figure 1)
- (ii) projection onto the  $F_1$ -axis gives rise to a "proton-decoupled" proton spectrum, and
- (iii) couplings between  $^1\text{H}$  and other nuclei ( $^{19}\text{F}$ ,  $^{31}\text{P}$ ,...) can be measured separately.

A communication about the new pulse sequence has been submitted for publication in J. Magn. Reson. Please credit this letter to the account of Professor T.C. Wong.

Best regards.

Sincerely yours,

V. Rutar

VR/sdI

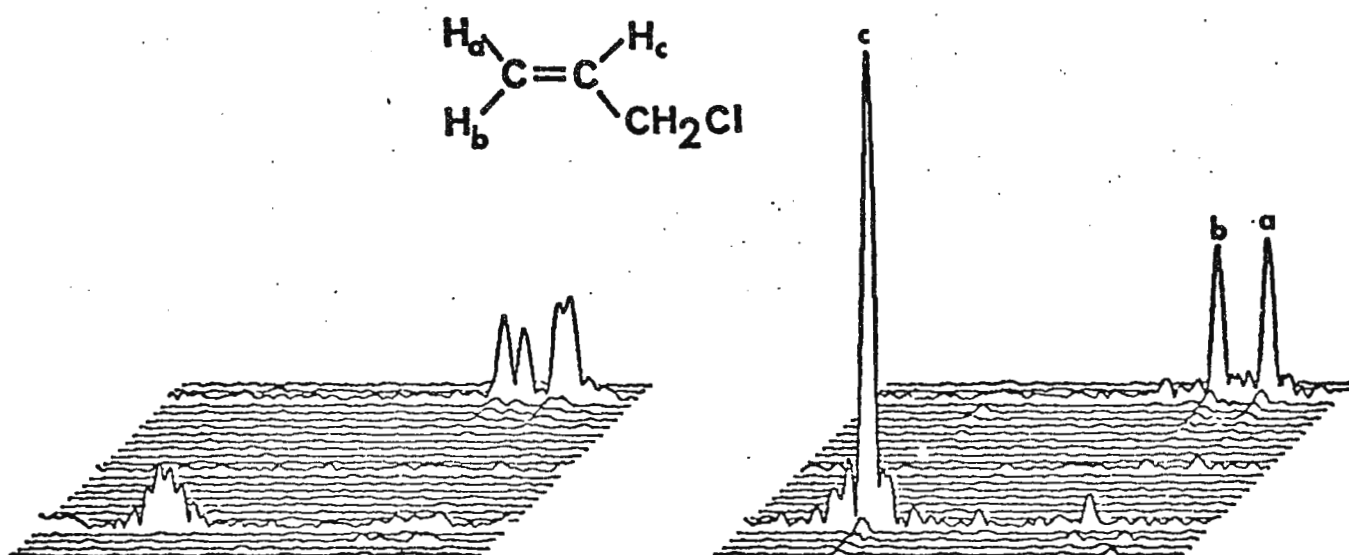


Fig. 1: Comparison between the low-field parts of the traditional (left) and new (right)  $^{13}\text{C}$ - $^1\text{H}$  chemical shift correlation maps. Since the modified pulse sequence eliminates proton multiplet structure from the  $F_1$ -dimension (horizontal), intensity is not dispersed and signal-to-noise ratio increases. Interpretation of spectra and  $^1\text{H}$  chemical shift determinations are also simplified.

P.S.: A NMR spectroscopist position is available at the University of Missouri, Columbia, Missouri (see attached).

**NMR SPECTROSCOPIST** The University of Missouri-Columbia is seeking an NMR Spectroscopist. The successful candidate will be expected (a) to assume responsibility for the operations of the campus NMR Facility, (b) to train and advise users on the use of the NMR spectrometers, (c) to initiate an independent or collaborative research program in the magnetic resonance area. The Facility currently has a Nicolet 300 MHz wide-bore multinuclear FT NMR, a home-built 180 MHz and a JEOL FX-90 proton/carbon spectrometers. Candidates should have a recent Ph.D. in chemistry, biochemistry, or physics with a strong background in modern FT NMR. Experience with electronics and computer systems is also desirable. Salary commensurate with qualification. Position available October 1983. Application should include resume and 2-3 letters of recommendation, and sent to: Professor T.C. Wong, Department of Chemistry, University of Missouri, Columbia, Mo. 65211. AA/EO Employer.

## UNIVERSITY OF CALIFORNIA, SAN DIEGO

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SANTA BARBARA • SANTA CRUZ

June 28, 1983

DEPARTMENT OF CHEMISTRY, B-014  
LA JOLLA, CALIFORNIA 92093Dr. B.L. Shapiro  
Department of Chemistry  
Texas A&M University  
College Station, TX 77843

## Quadrupolar Order with Dipolar Coupling

Dear Barry,

We have been making extensive use of the Jeener-Broekaert pulse sequence  $90_x - \tau - 45_y - t_1 - 45_y - \text{ACQ}$  (1) for purposes of measuring individual motional spectral densities of deuterium in liquid crystals (2). It is well known that for a single deuteron, proper choice of  $\tau$  leads to creation of a state of purely quadrupolar order, and that subsequent evolution consists of single exponential decay of  $\langle H_Q \rangle$  and single exponential recovery of  $\langle H_Z \rangle$ . Both relevant spectral densities are easily determined from the two relaxation rates.

Multideuteron systems, partially ordered such that both quadrupolar and dipolar splittings are resolved, are more complicated. Two reasonable conjectures about such systems lead to opposite conclusions. Conjecture A: quadrupolar and dipolar interactions both transform like second rank tensors, the distinction between quadrupolar and dipolar order is academic and the J.B. sequence should always "work" for determining two spectral density parameters. Conjecture B: since  $H_Q$  and  $H_D$  do not commute the state of purely quadrupolar order is not the same as that of dipolar order, and a small dipolar coupling between deuterons may open new relaxation channels much like scalar coupling does for sets of chemically shifted spins.

Peter Luyten, who recently returned to the Free University of Amsterdam after a year's visit in our laboratory, ran across this problem in connection with the relaxation behavior of the  $(o,m)$  deuterons of biphenyl in nematic and smectic solvents. In order to see whether either conjecture is close to physical reality we simulated the relaxation response of these deuterons to JB pulse sequences. Both the simulations and Peter's experimental results confirm that conjecture B is correct: the relaxation behavior is easy to analyze reliably only when the dipolar coupling is weak.

Sincerely,

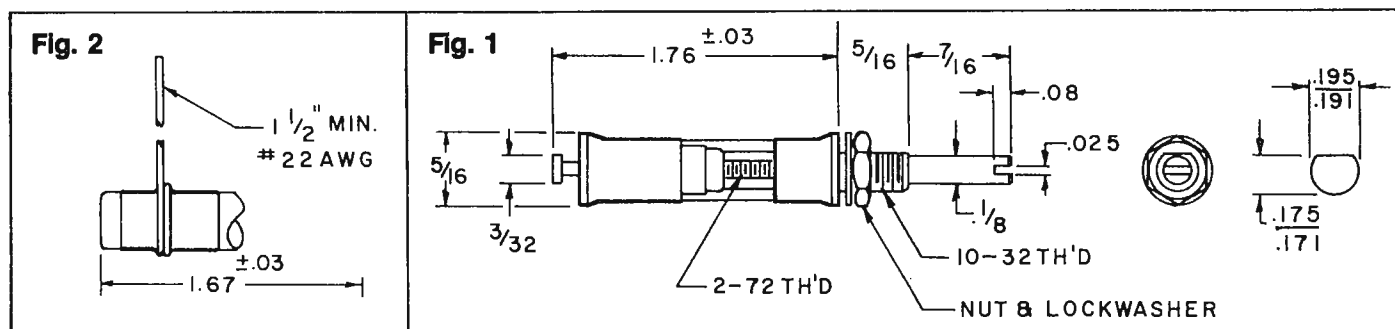
Robert L. Vold  
Regitze R. Vold  
Professors of Chemistry<sup>1</sup> J. Jeener and P. Broekaert, Phys. Rev. 157, 232 (1967).<sup>2</sup> W.H. Dickerson, R.R. Vold and R.L. Vold, J. Phys. Chem. 87, 166 (1983).

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NMQM22GE	Quartz	1	22	2000	2500	Fig. 2

Insulation Resistance:  $10^6$  megohms  
 Temperature Coefficient:  $-100$  to  $+500$  ppm/ $^{\circ}$ C  
 Temperature Range:  $-55^{\circ}$ C to  $+125^{\circ}$ C  
 Withstanding Voltage: Twice D.C.W.V.

Construction: Non-magnetic materials  
 Tuning Torque: 3 in. oz. max.  
 Life: Stop to Stop - 1000 min.  
 Resolution: Over 50 usable turns

- Notes:**
1. The embedded band glass (Fig. 1) has a metal end cap with a turret terminal rather than the rounded seal end of the others. (Fig. 2)
  2. No rating is given for RF voltage since we cannot test at high RF voltages at the use frequencies. However, the quartz dielectric should be by far the best, the annular glass next, and the embedded band style the worst since its inner dielectric is only 0.0035" thick.

Prices	Part No.	2-4	5-9	10-24	25-49	Consult factory for larger quantities
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	NMQM22GE	98.00	91.00	85.00	76.00	

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## Voltronics

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# Los Alamos

Los Alamos National Laboratory  
Los Alamos, New Mexico 87545

INC-4, Mail Stop C346  
INC-4:83-207

June 27, 1983

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

Dear Barry,

## 1. Rf Heating Effects

Control and measurement of temperature and temperature gradients within lossy samples is of major importance for NMR experiments on temperature sensitive samples or on materials undergoing phase transitions. We have used  $\text{Co(CN)}_6^{3-}$  (Levy, Bailey & Wright, J. Mag. Res. 37 353 (1980)) to estimate the rf heating and temperature gradients induced in lossy samples by proton decoupling in our Bruker WM 300 spectrometer, and have found much more substantial effects than generally admitted. Even low Rf decoupling power gave fairly large temperature gradients and heating effects within 3ml of an aqueous 0.2 M NaCl, 0.05 M  $\text{Co(CN)}_6^{3-}$  solution contained in a 10 mm sample tube, (Figure 1). By positioning a small microcell containing  $\text{Co(CN)}_6^{3-}$  at various heights in the sample tube we determined that the major direction of the temperature gradient was vertical (Figure 2) under both spinning and non-spinning conditions.

## 2. "Imaging" without a VAX and $\text{H}_0$ orientations.

We recently desired to orient the spinning axis of sideways spinning probes along either the x or y shim coil axis of our superconducting magnet. By placing two 8 mm sample tubes in a 20 mm tube and orienting the sample at specific angles in the shim tube, we were able to obtain a 2D "image" of the protons in the phantom (Figure 3) and determine the orientation of the shim coil axes. The necessary field gradients were applied by adjusting the x or y shim control to its limits.

On an even more nuts and bolts level, we recently learned that the magnetic field in our Bruker WM 300 magnet points down, whereas the field in our Oxford CXP 200 magnet points up; consequently our NMR laboratory is a permanent quadrupole.

JM.

Jim Brainard

Carl

Carl Storm

Enchi

Eiichi Fukushima

Best regards,

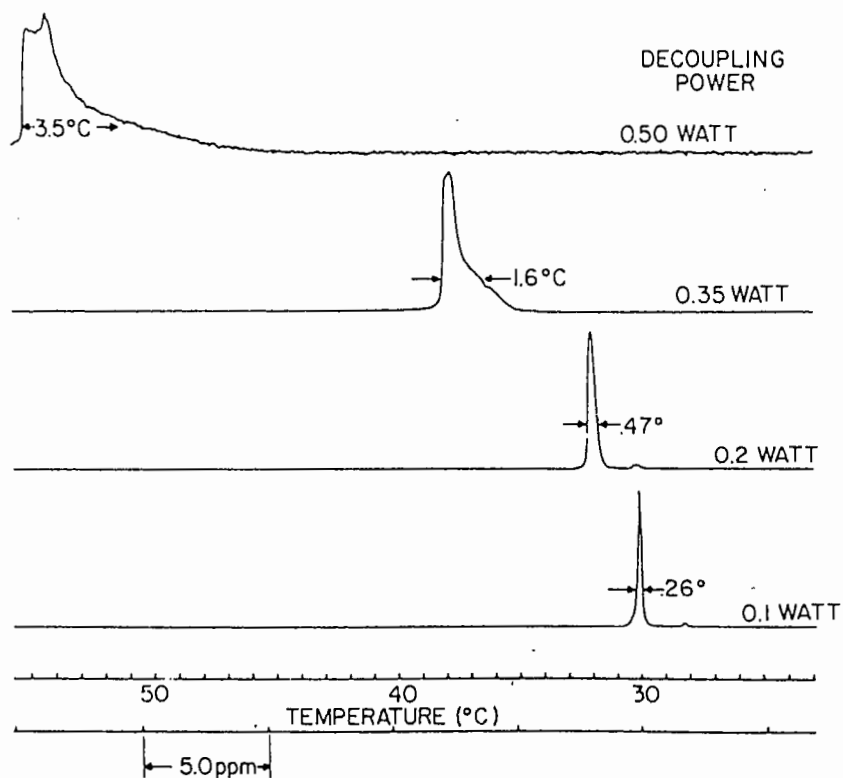
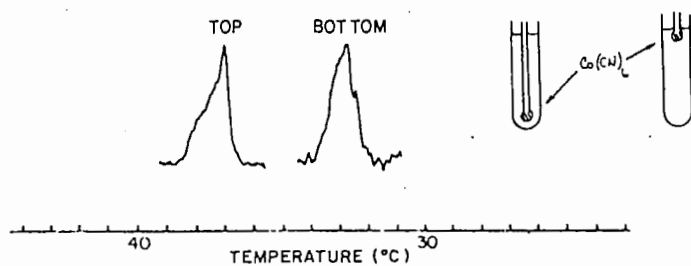
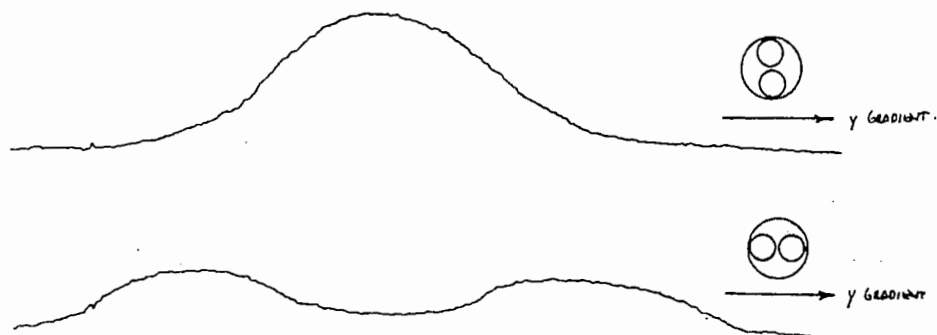
Bill

Bill Earl

Please credit this contribution to Carl Storm's subscription.

JRB/jk

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Figure 1.  $\text{Co}(\text{CN})_6$  in a 10mm sample tubeFigure 2.  $\text{Co}(\text{CN})_6$  in Microcell within a 10mm sample tubeFigure 3. 2-D "image" of two 8mm tubes filled with  $\text{H}_2\text{O}$



**CSIRO****Division of Fossil Fuels**

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MAW.jcm

A Division of the Institute of Energy and Earth Resources

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Telephone (02) 887 8666

Telex AA25817

9th June, 1983

Professor Barry L. Shapiro  
 Department of Chemistry  
 Texas A and M University  
 College Station  
 TEXAS 77843  
USA

Dear Barry,

GASPE AND INEPT FOR DEUTERATED STUDIES

As part of our investigation of the chemistry of coal liquefaction we needed to trace the incorporation of deuterium into liquid products. It was necessary to determine if one or two deuterium atoms are incorporated at  $\alpha$  and  $\beta$  sites in 4-methylpentan-2-one and tetralin. Both  $^1\text{H}$  INEPT and GASPE (J. Mag Res, 1981, 45, 8; Org. Mag. Res. 1981, 16, 111) sequences are useful for these purposes. For example, Figure 1 shows the INEPT spectrum of deuterio-4-methylpentan-2-one for a)  $\Delta = 2\text{J}^{-1}\text{s}$  and b)  $\Delta = 3(4\text{J})^{-1}\text{s}$  showing proportions of  $\text{CHD}$ ,  $\text{CH}_2\text{CHD}_2$  and  $\text{CH}_2\text{D}$  groups. Resonances are split because of the deuterium coupling. Likewise Figure 2 shows the GASPE spectrum of deuterio-4-methylpentan-2-one for  $\tau = \text{J}^{-1}$ ,  $\tau = (2\text{J})^{-1}\text{s}$  showing resonances from deuterated carbons. The inversion of phase, and coupling makes estimations of the amounts of deuterated analogues to be determined with ease.

The method should be useful for a wide range of organic compounds. The results will be published in Fuel later this year.

Yours sincerely,

*Phil Collin**Michael Wilson*

P.J. Collin and M.A. Wilson  
Division of Fossil Fuels

FIGURE 1

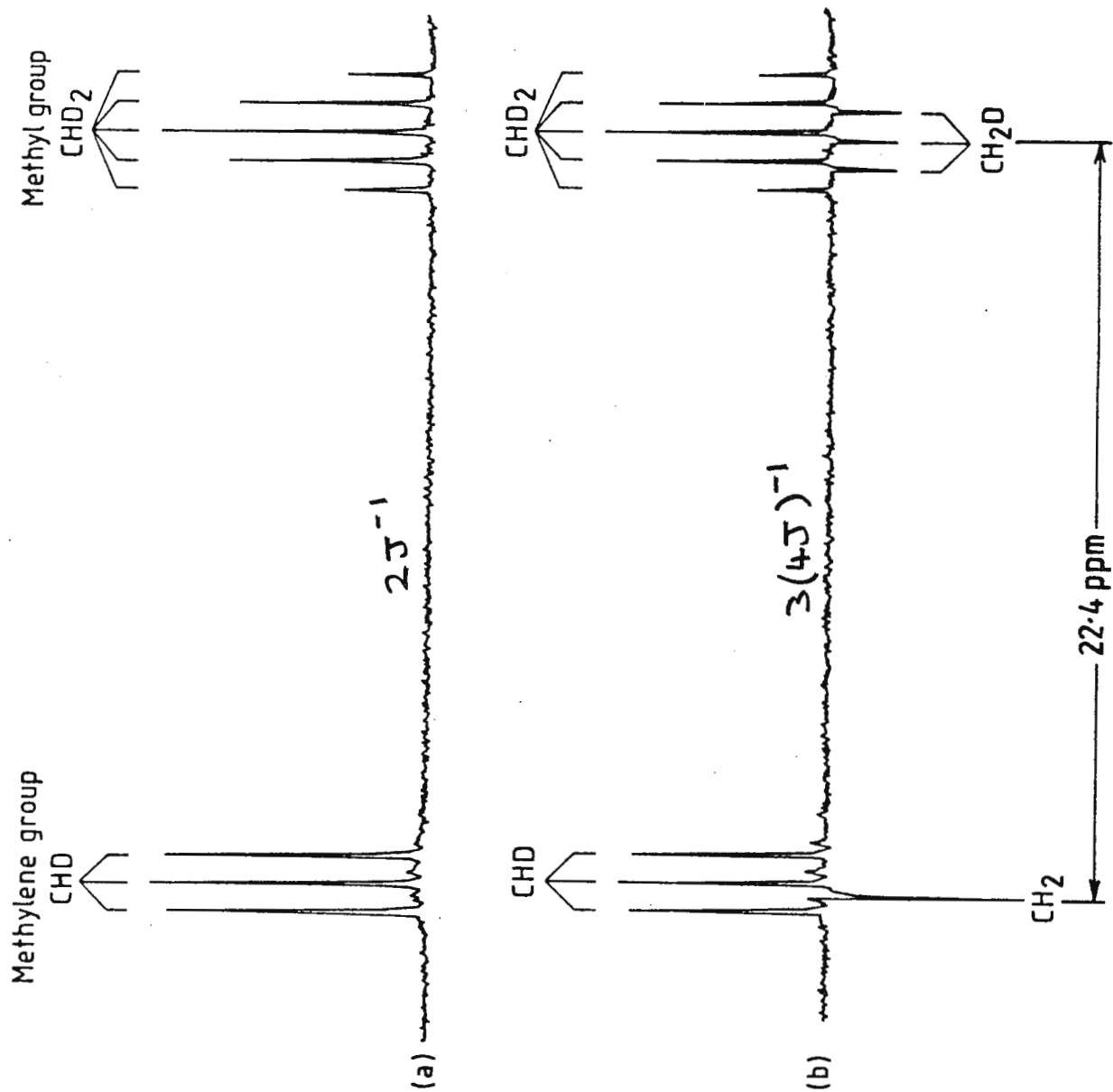


Fig. 1

FIGURE 2

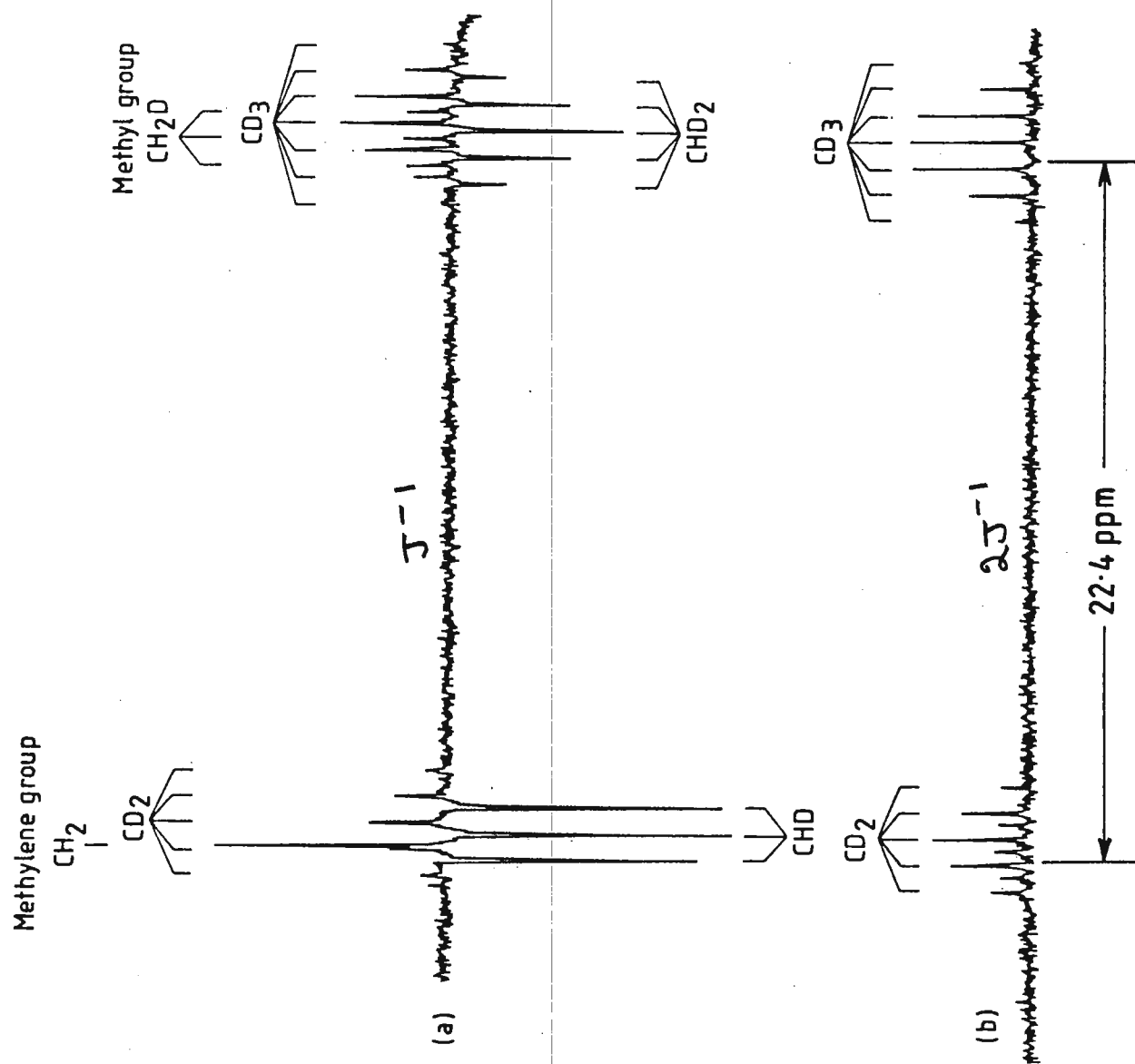
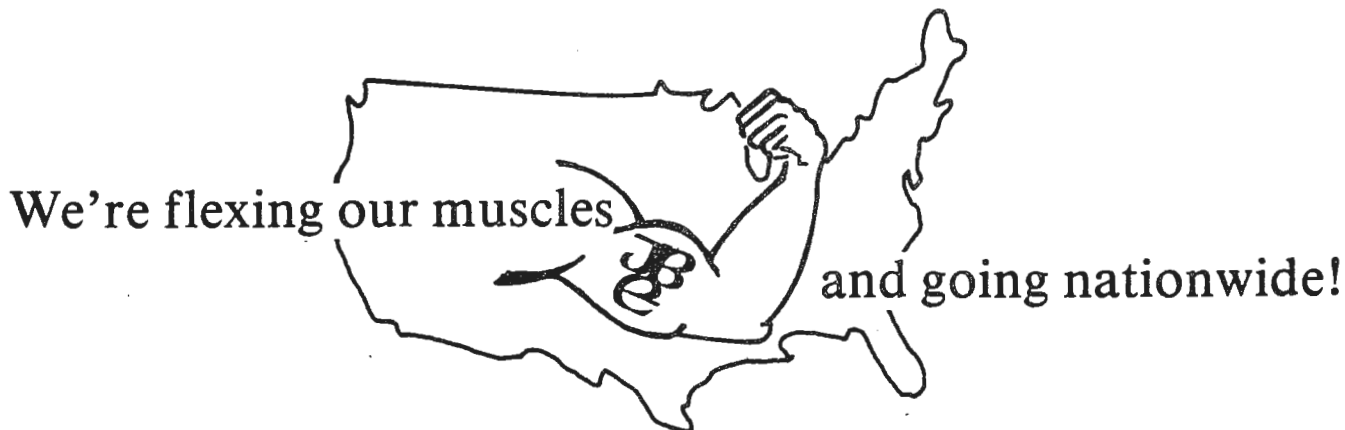


Fig. 2

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July 5, 1983

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

Dear Barry,

The Institute for Cancer Research has a position currently available for an instrumentation specialist. The primary duty will be the maintenance of high resolution NMR spectrometers and associated data systems as well as an EPR spectrometer. Proficiency in NMR electronics, hardware and software are essential. Familiarity with probe design, 2D-NMR and some computer programming experience is desirable. The Institute has a multinuclear Varian 60 MHz NMR spectrometer and a Nicolet 300 MHz NMR spectrometer and a Varian E-109 EPR spectrometer. If interested write to me at the above address or phone (215) 728-2727.

Sincerely,



Mildred Cohn

MC/msw

7 June 1983

Refer to: 83-211

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

Dear Dr. Shapiro:

A postdoctoral position exists within our research group for either a physical chemist or a physical organic chemist interested in the study of solids using NMR techniques. We are particularly interested in developing experimental techniques in multiple pulse and 2-D spectroscopy in solids in order to increase the understanding of the structure and dynamics of complex organic systems. Systems of interest range from simple organic molecules to complex geological polymers such as soil organic matter, shales, and coals.

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Sincerely yours,



Ronald J. Pugmire  
Professor of Fuels Engineering

RJP:bm



UNIVERSITY OF MISSOURI-COLUMBIA

College of Arts and Science

Department of Chemistry

123 Chemistry Building  
Columbia, Missouri 65211  
Telephone (314) 882-2439

June 30, 1983

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

Dear Dr. Shapiro:

NMR SPECTROSCOPIST

The University of Missouri, Columbia is seeking an NMR Spectroscopist. The successful candidate will be expected (a) to assume responsibility for the day to day operations of the campus NMR Facility, (b) to train and advise users on the use of the NMR spectrometers, (c) to collaborate with the research staff on problems in organic, inorganic, biochemistry, and biophysical chemistry, and/or to initiate an independent research program in the magnetic resonance area. The Facility currently has a Nicolet 300 MHz wide-bore multinuclear FT NMR, a home-built 180 MHz and a JEOL FX-90 proton/carbon FT NMR spectrometers. Candidates should have a recent Ph.D. in chemistry or physics with a strong background in modern FT NMR. Experience with electronics and computer systems is also desirable. Salary will be commensurate with qualification. The position will be available September 1983.

Application should include a resume and 2-3 letters of recommendation, and should be sent to: Professor T. C. Wong, Department of Chemistry, University of Missouri, Columbia, Missouri 65211. Persons who are interested can also reach me by phone at 314-882-7725 or 882-2439.

The University of Missouri is an Affirmative Action/Equal Opportunity Employer.

Sincerely yours,

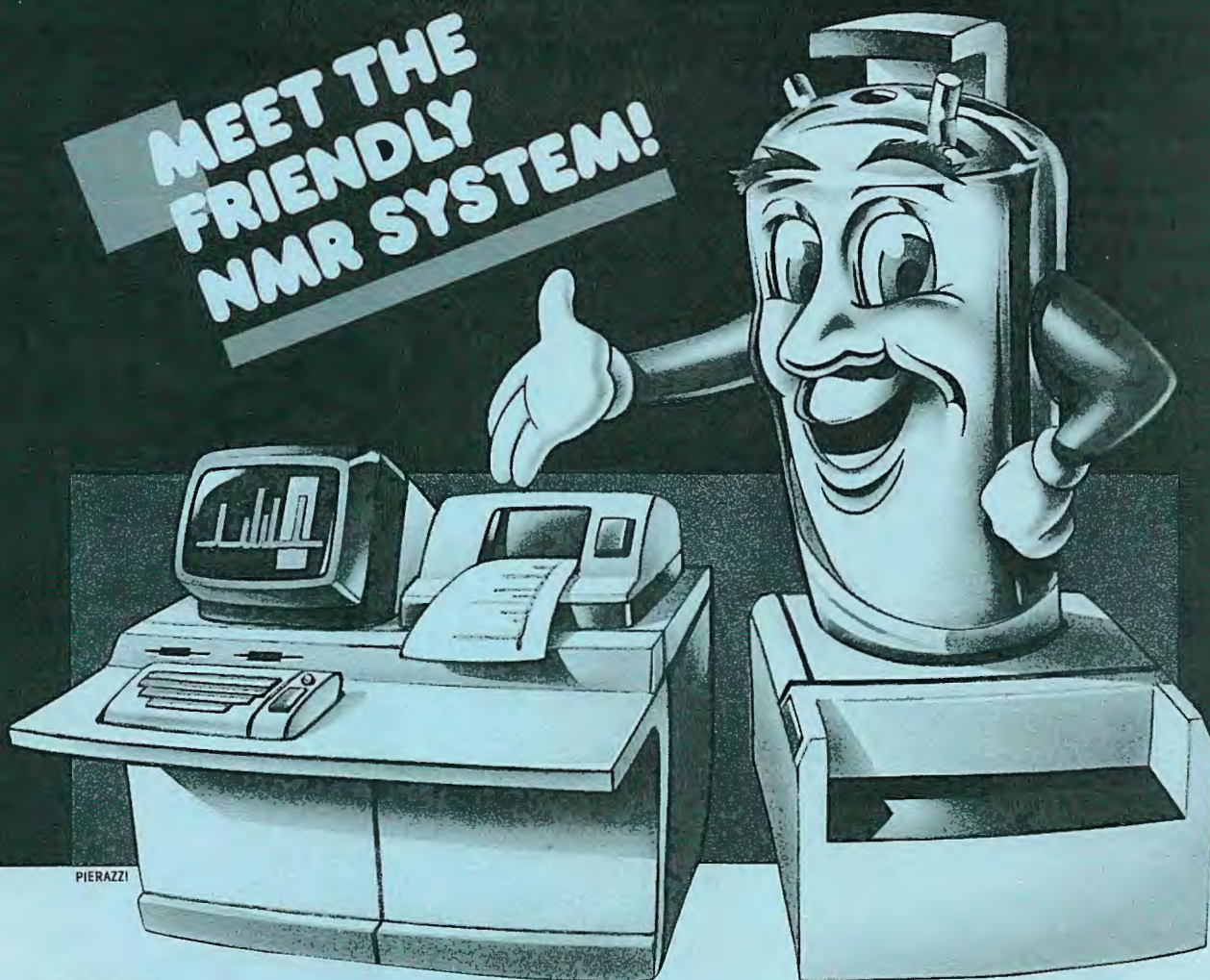
A handwritten signature in dark ink, appearing to read 'Tuck C. Wong'.

Tuck C. Wong  
Associate Professor and  
Director of NMR Facility

TCW/sdl



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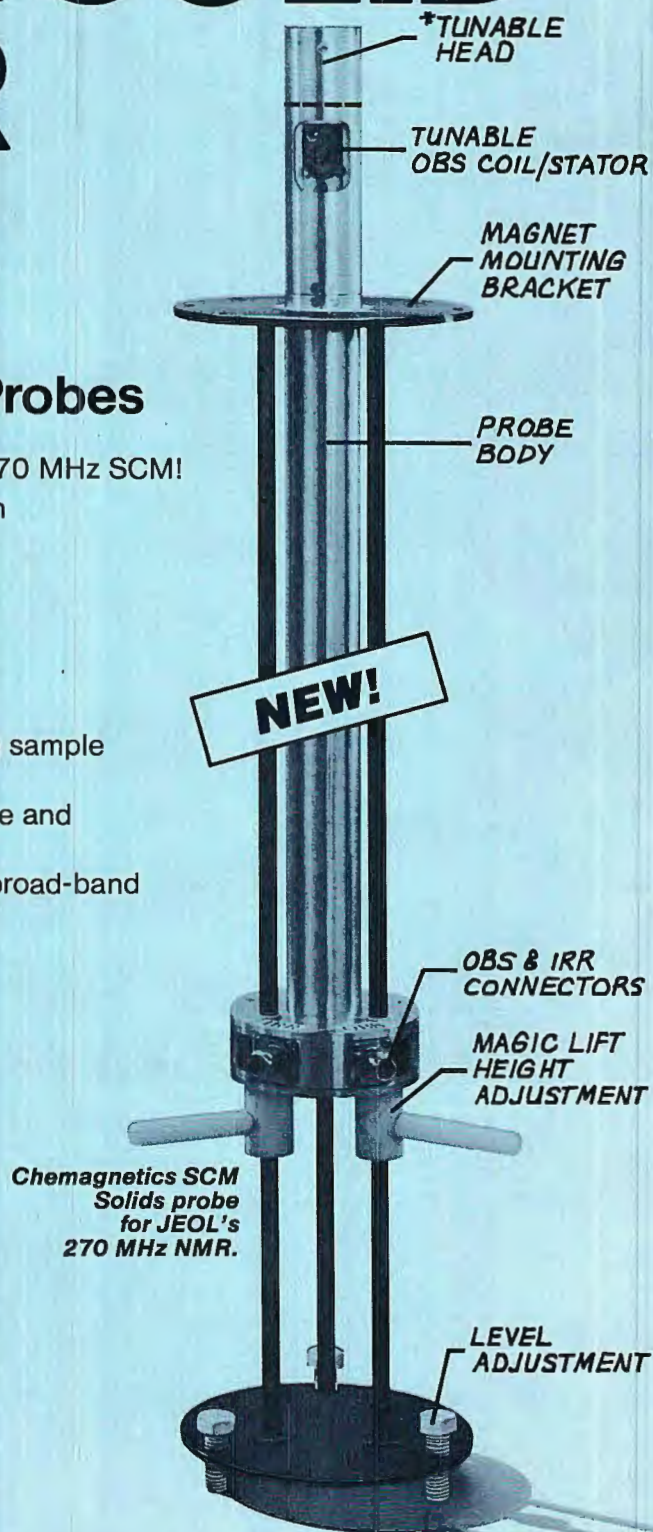
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