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TABLE 1 DEUTERATED SOLVENTS

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D-11	Acetone- d_6	CD_3COCD_3	99.8%	1.1	-17	56	0.551 (32)
D-120	Acetone- d_6 + 1% TMS	CD_3COCD_3	99.8%	1.1	-17	56	0.551 (32)
D-13	Acetone- d_6	CD_3COCD_3	99.8%	0.87	-34	57	0.460 (20)
D-121	Acetone- d_6 + 1% TMS	CD_3COCD_3	99.8%				
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D-14							0.611
D-21							
D-122							
D-130							
D-28	Chloroform- d	$CDCl_3$	99.8%	1.50	-64	62	0.740 (20)
D-31	Chloroform- d + 1% TMS	$CDCl_3$	99.8%				

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FORTHCOMING NMR MEETINGS

Gordon Research Conference: Magnetic Resonance in Biology and Medicine, July 16-20, 1990; Tilton School, Tilton, NH. Chairman: R. G. Bryant. Contact: Dr. A. M. Cruickshank, Gordon Research Center, Univ. of Rhode Island, Kingston, RI 02881-0801.

Workshop of Special Topics in Medical Magnetic Resonance, sponsored by the Society of Magnetic Resonance in Medicine and the National Research Council of Canada, **July 23-27, 1990**; Whistler Mountain, BC, Canada. Contact: L. Forget - see Newsletter 374, 46.

Expanding Frontiers in Polypeptide and Protein Structural Research, sponsored by the National Research Council of Canada, **July 23-27, 1990**; Whistler Mountain, BC, Canada. Contact: L. Forget - see Newsletter 374, 46.

Tenth International Biophysics Conference, sponsored by the International Union of Pure and Applied Biophysics and the National Research Council of Canada, **July 29 - August 3, 1990**; Vancouver, BC, Canada. Contact: L. Forget - see Newsletter 374, 46.

Bat-Sheva Workshop on New Developments and Applications in NMR and ESR Spectroscopy, **October 14-24, 1990**, Israel; Contact: Dr. D. Goldfarb, The Weizmann Institute of Science, Rehovot, Israel. See Newsletter 377, 10.

Eastern Analytical Symposium, Garden State Convention Center, Somerset, NJ; NMR Symposia and Poster Sessions on **Nov. 13 and 14, 1990**; Contact D. C. Dalgarno or C. A. Evans, Schering-Plough Research, 60 Orange St., Bloomfield, NJ 07003; (201) 429-3957; FAX: (201) 429-3916.

Advanced Tomographic Imaging Methods for the Analysis of Materials, Symposium at the Fall Meeting of the Materials Research Society, Boston, Mass., **Nov. 26 - Dec. 1, 1990**; See Newsletter 378, 57.

Additional listings of meetings, etc., are invited.

Dr. B.L. Shapiro
966 Elsinore Court
Palo Alto, CA 94303, U.S.A.

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Sherbrooke (Québec) J1K 2R1

April 17, 1990.
(received 4/23/90)

¹H MREV-8 of Elastin

Dear Dr. Shapiro,

We have been studying domain structures and their mobilities in the insoluble protein Elastin with a variety of ¹H NMR spin dynamics experiments. Here we present our results of the lineshape as a function of the multipulse sequence MREV-8 cycle time (t_c). In Figure 1, a two-component lineshape is observed with a single 90° pulse while a one component lineshape (FWHH = 1.2 KHz) is obtained with MREV8, $t_c = 41.2 \mu s$. In Figure 2 we observe the qualitative change from a single to two-component lineshape (half-spectra are shown as data were acquired with on-resonance MREV-8 without quadrature) as a function of t_c . Adequate digital resolution was used throughout to detect the narrow component. We are presently trying to understand these results in terms of motional correlation time, the flip-flop rate of the rigid lattice, and the cycle time of the MREV-8 sequence.

Sincerely,

Serge Lacelle
André Saint-Jean

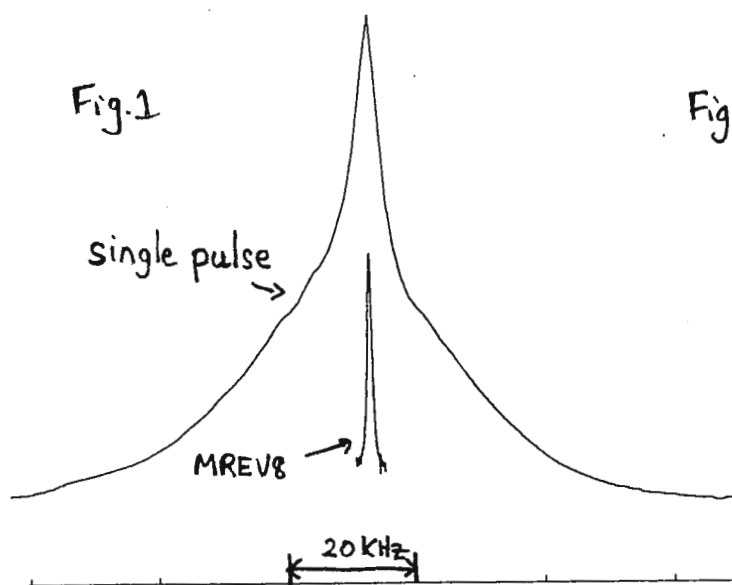
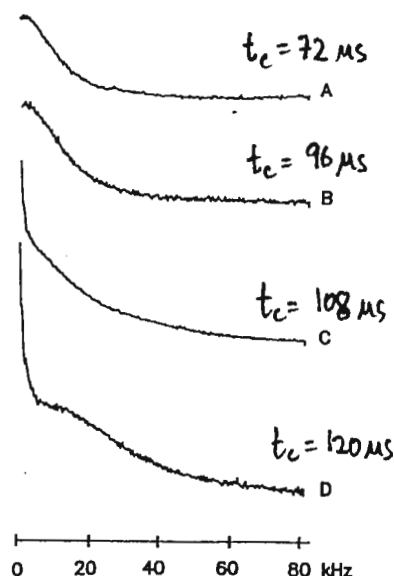


Fig.2



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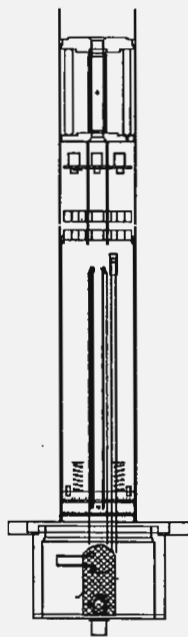
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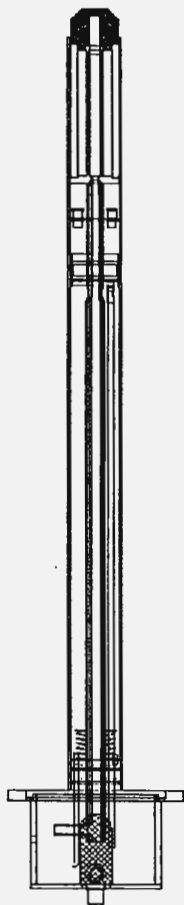
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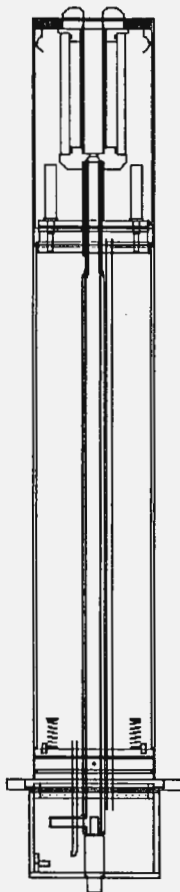
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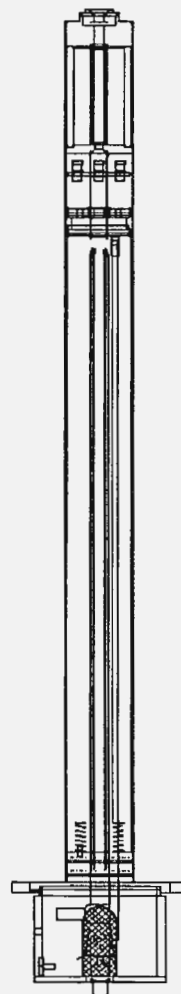
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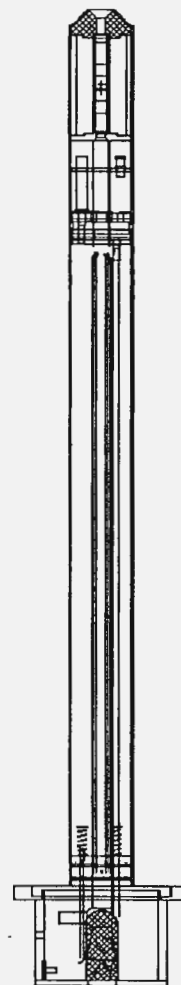
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Dr. B. SHAPIRO
TAMU NMR NEWSLETTER
966 Elsimore Court
PALO ALTO CA 94303 U.S.A.

Department of Organic Chemistry
and NMR Laboratory

(received 4/23/90)

P-31 Relaxation Rates Enhancement: an efficient test for the chemical stability of MRI contrast agents.

For several years, our lab has been involved in the characterization of MRI contrast agents. In an original protocol we developed (1), the dispersion profiles of proton water relaxation rates (NMRD) were recorded in different media: aqueous solutions at different pH and temperatures, aqueous solutions containing albumin, blood, serum,.... Since it has been proposed to include these contrast agents into liposomes targeting the liver, making them potential agents for spectroscopy, we decided to measure their influence on P-31 relaxation rates of phosphorylated metabolites (adenosine triphosphate ATP, inorganic phosphate Pi and, although absent in the liver, phosphocreatine PCr) in buffer solutions at physiological pH and 37 °C. Our results show that the macrocyclic DOTA-Gd induces a small and similar increase of the relaxation rates of all metabolites (fig 1). As expected, this increase is linearly dependent on the concentration of the paramagnetic center.

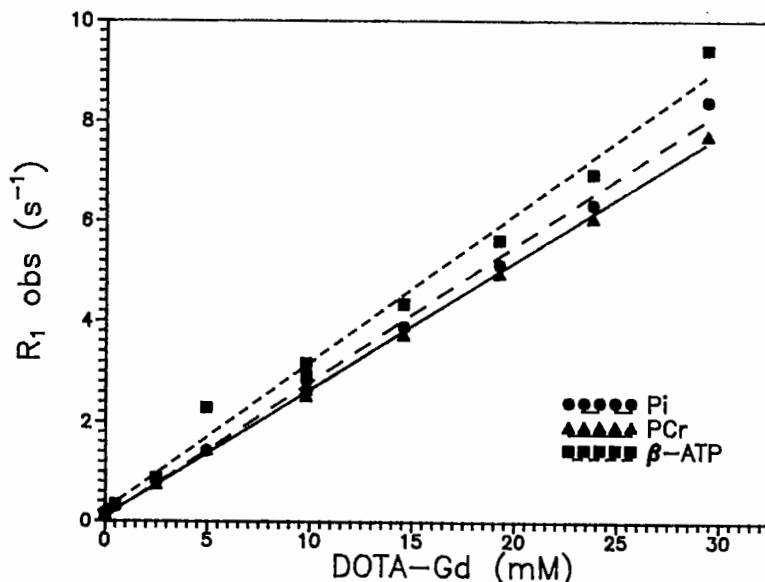


Fig 1: P-31 relaxation rates of PCr, Pi and ATP (20 mM) at pH 7.2, 37 °C vs DOTA-Gd concentration

DTPA-Gd and DTPA-BMA-Gd also induce a moderate and linear enhancement of PCr and Pi relaxation rates. However, the relaxation enhancement of ATP phosphate groups induced by these compounds is much more pronounced and his dependence on the concentration of contrast agent is no longer linear (fig 2 and 3).

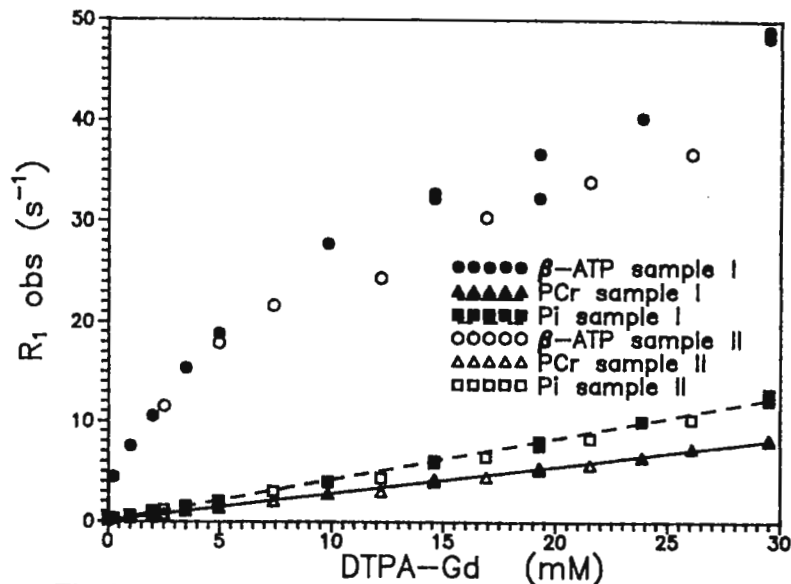


Fig 2: P-31 relaxation rates of Pi, PCr and ATP (20 mM) at pH 7.2, 37°C vs DTPA-Gd concentration.

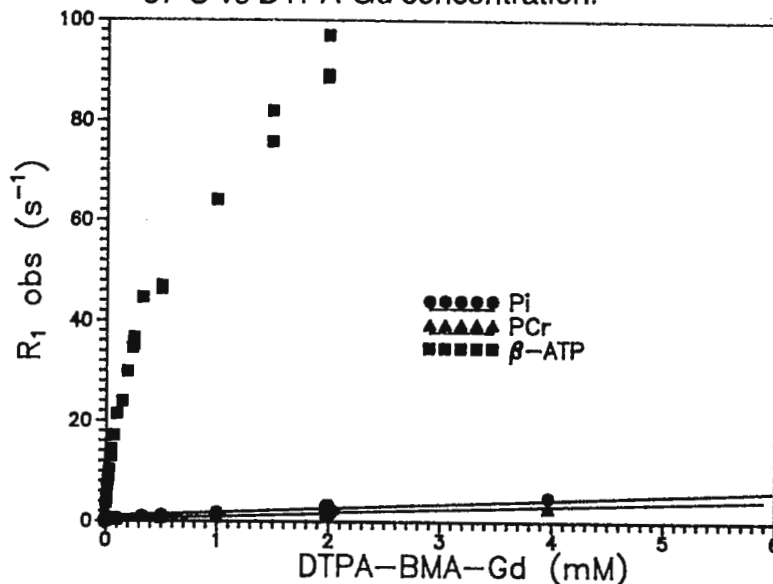


Fig 3: P-31 relaxation rates of Pi, PCr and ATP (20 mM) at pH 7.2, 37°C vs DTPA-BMA-Gd concentration.

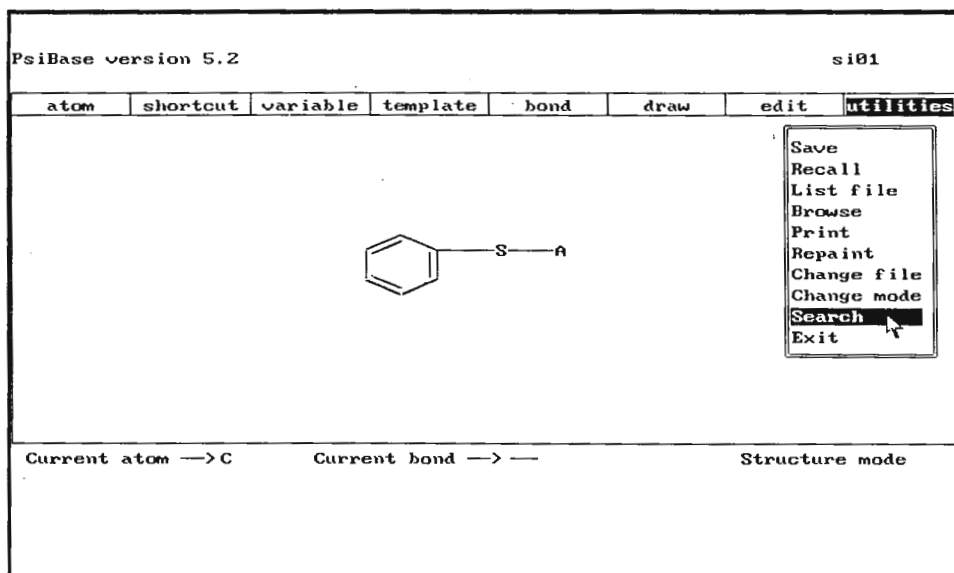
The linear enhancement of relaxation rates can be explained by outersphere interactions between the paramagnetic centers and the phosphorus nuclei whereas, the large and non linear increases are explained by an association of gadolinium ion with ATP. P-31 relaxation rates of ATP are thus good probes to assess the stability of paramagnetic chelates in solution and we think this method should be used as a complement of the original protocol(1) to test the stability of contrast agents in physiological medium.

Dr. R.N. MULLER

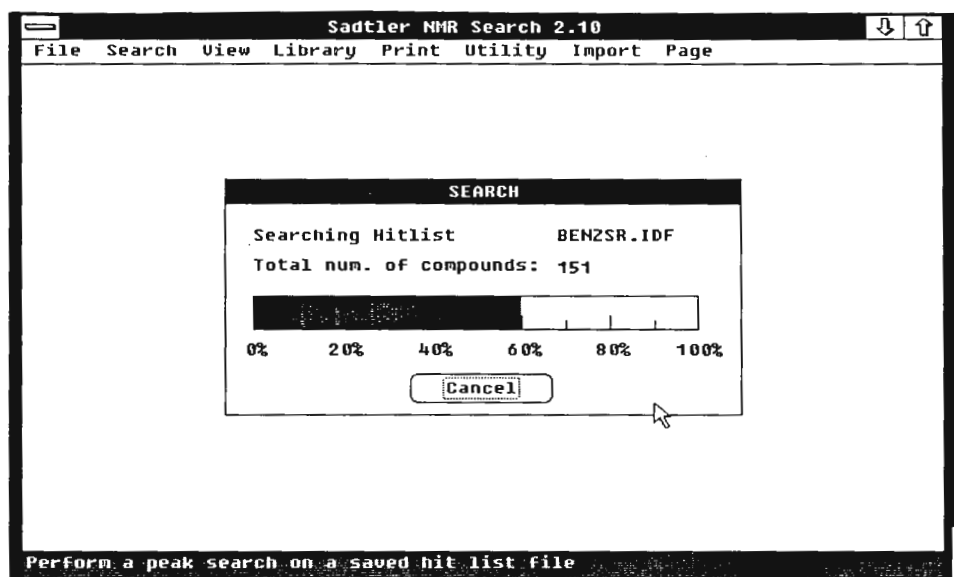
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(1) R.N. Muller, F. Maton and Y. Van Haverbeke Diagn. Imaging Int. Suppl. Nov 1988 62-63



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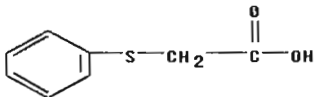
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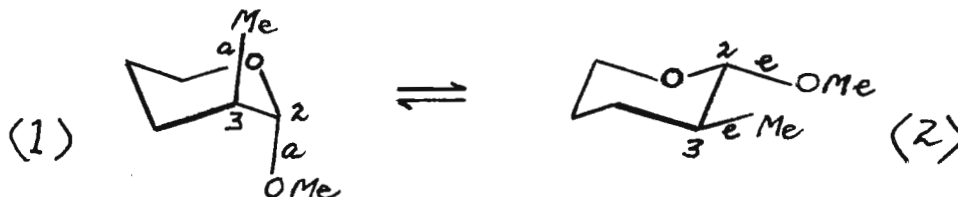
Dr. B.L. Shapiro,
TAMU **NMA** Newsletter
966, Elsinore Court,
Palo Alto, CA94303
U.S.A.

19th April 1990
(received 4/23/90)

Disappearing Conformations

Dear Dr. Shapiro,

The room temperature ^{13}C nmr spectrum of (1 \rightleftharpoons 2) showed the expected 7 lines. Significant broadening, followed by a sharpening, of all lines occurred as the temperature was lowered. However, at 162K only the 7 lines of (2) could be detected.



Now in 2-methoxytetrahydropyran, the proportion of favoured axial conformation is temperature-independent, since ΔH° is zero (ref.1.) and $\ln K = \Delta S^\circ/R$. In 3-methyltetrahydropyran, however, the proportion of strongly favoured (ref.2.) equatorial conformation should increase rapidly at low temperatures, since ΔS° is expected to be zero and $\ln K = -\Delta H^\circ/RT$. Although thermodynamic parameters are not expected to be additive in a 1,2-disubstituted ring, our inability to detect (1) at 162K is not surprising.

References

1. Tetrahedron, 1987, 43, 4699.
2. J.A.C.S., 1982, 104, 3635.

Yours sincerely,

H. Booth

H. Booth
S.A. Readshaw

Lehigh University



Department of Chemistry
telephone (215) 758-3470

Seeley G. Mudd Building 6
Bethlehem, Pennsylvania 18015

Dr. Bernard L. Shapiro
TAMU NMR News Letter
966 Elsinore Ct.
Palo Alto, CA 94303

The Relaxivity of Two Mn(III) Porphyrins
in Aqueous and Glycerol/Water Solutions

(received 5/7/90)

Dear Dr. Shapiro,

The presence of manganese (III) complexes of tetraphenylsulfonfyl porphyrin (MnTPPS₄) and deuteroporphyrin 2,4 disulfonic acid (MnD24DSA) increases the water proton relaxation rate ($1/T_{1P}$) in both aqueous and glycerol/water solutions. The water proton relaxation rate normalized to concentration of Mn(III) porphyrin is termed the relaxivity. For the work presented here, the concentration of both Mn(III) porphyrins was 1 mM, so the results can be compared directly.

The magnitude of the relaxivity at 90 MHz is dominated by two parameters contained in the Solomon-Bloembergen-Morgan (SBM) equations: τ_R , the rotational correlation time of the Mn(III) porphyrin, and r , the distance between the center of the Mn³⁺ ion and the bound water protons. This was inferred by extrapolating the NMRD profile for MnTPPS₄ to 90 MHz and is supported by the linearity of $1/T_{1P}$ with viscosity over absolute temperature (η/T) as shown in Figure 1a (also see Equation (2)). Here, the η/T ratio was changed by changing the temperature of the aqueous solutions.

However, as glycerol was added to the solutions at a constant temperature of 20°C, the plots were no longer linear and MnD24DSA attained a higher relaxivity than MnTPPS₄ (Figure 1b). This somewhat surprising result may be described by considering one porphyrin in solution with the appropriate number of glycerol (N_G) and water (N_W) molecules around it to keep the concentrations the same as in the actual experiment. Assuming that there is no preference for a Mn(III) porphyrin to pick a glycerol or water OH group to bind in the two axial positions, three species are present: (1) the Mn(III) porphyrin with one water molecule and one glycerol molecule, (2) with two water molecules, and (3) with two glycerol molecules in the axial positions. Because the OH resonance consists of mainly water protons, we assume that only water containing species (1) and (2) contribute to the relaxivity and the respective probabilities for their occurrence are:

$$P_1 = E_1/\Omega ; \quad P_2 = E_2/\Omega \quad (1)$$

where $E_2 = N_W!/[2!(N_W - 2)!]$

$$E_1 = N_W!/[1!(N_W - 1)!] \times (3N_G)!/[1!(3N_G - 1)!]$$

$$\Omega = (N_W + 3N_G)!/[2!(N_W + 3N_G - 2)!]$$

The factor of three appears because glycerol has three OH groups. To describe the data in terms of the SBM equations, two different T_{1M} values must be considered corresponding to the two different species. The appropriate SBM equation for the present work is:

$$\begin{aligned} 1/T_{1P} = & P_2 [2/(N_W + 4N_G) (B/r_2)^6] (4\pi a_2^3/k) (\eta/T) \\ & + P_1 [1/(N_W + 4N_G) (B/r_1)^6] (4\pi a_1^3/k) (\eta/T) \end{aligned} \quad (2)$$

Here, the factor of four ensures that the concentration of Mn(III) porphyrin is the same as for the experimental solution by including the volume occupied by water and glycerol. The value of a_i ($i=1,2$) reflects the hydrodynamic radius of the appropriate Mn(III) porphyrin species₃ and is contained in the Debye-Stokes-Einstein expression where $\tau_R = 4 \pi \eta a^3 / 3kT$. The ratios a_i^3/r_i^6 are obtained from a least-squares procedure, and the best fit lines are shown in Figure 1b. Note that for aqueous solutions containing no glycerol, $P_1 = 0$ and $P_2 = 1$ because only water molecules are available as axial ligands and only the ratios a_2^3/r_2^6 are obtained from the slopes of the lines in Figure 1a.

In the glycerol/water solutions the a_2^3/r_2^6 ratios corresponding to two axially bound water molecules are very close to those obtained from aqueous solutions where only species (2) was present. We take this as having identified a Mn(III) porphyrin species with two axially bound water molecules. The relative magnitudes of a_1^3/r_1^6 are responsible for MnD24DSA attaining a higher relaxivity than MnTPPS₄. For this species, the presence of the bound glycerol molecule may affect the relaxation of the bound water molecule by changing the Mn³⁺-water proton bond distance (r_1) or by changing the electronic environment of the Mn³⁺ ion. Because the water and glycerol OH protons are in rapid exchange, we cannot assign definite physical meaning to the a_1^3/r_1^6 ratio at this time.

Ken

Kenneth E. Kellar

Natalie

Natalie Foster

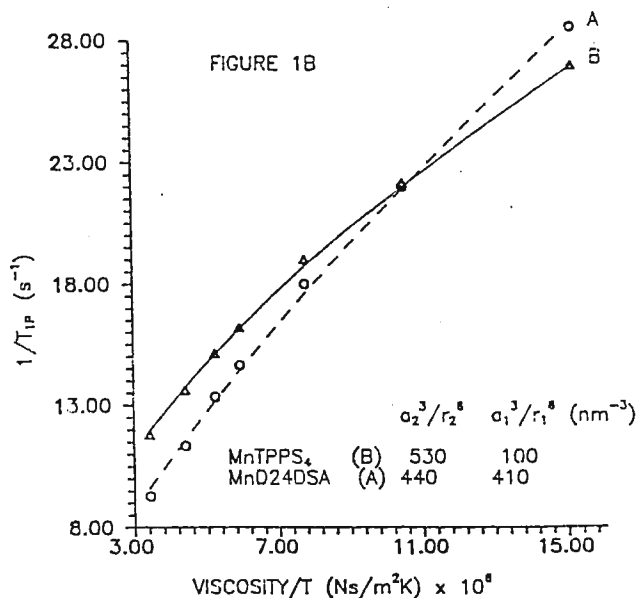
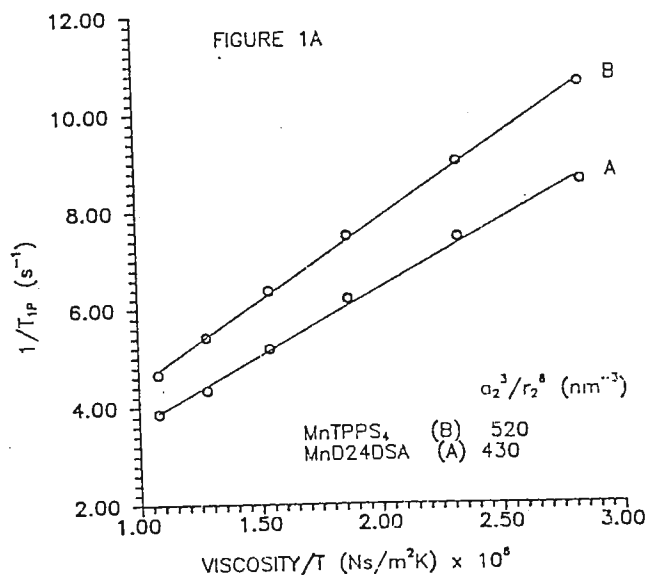
Jim

James E. Roberts

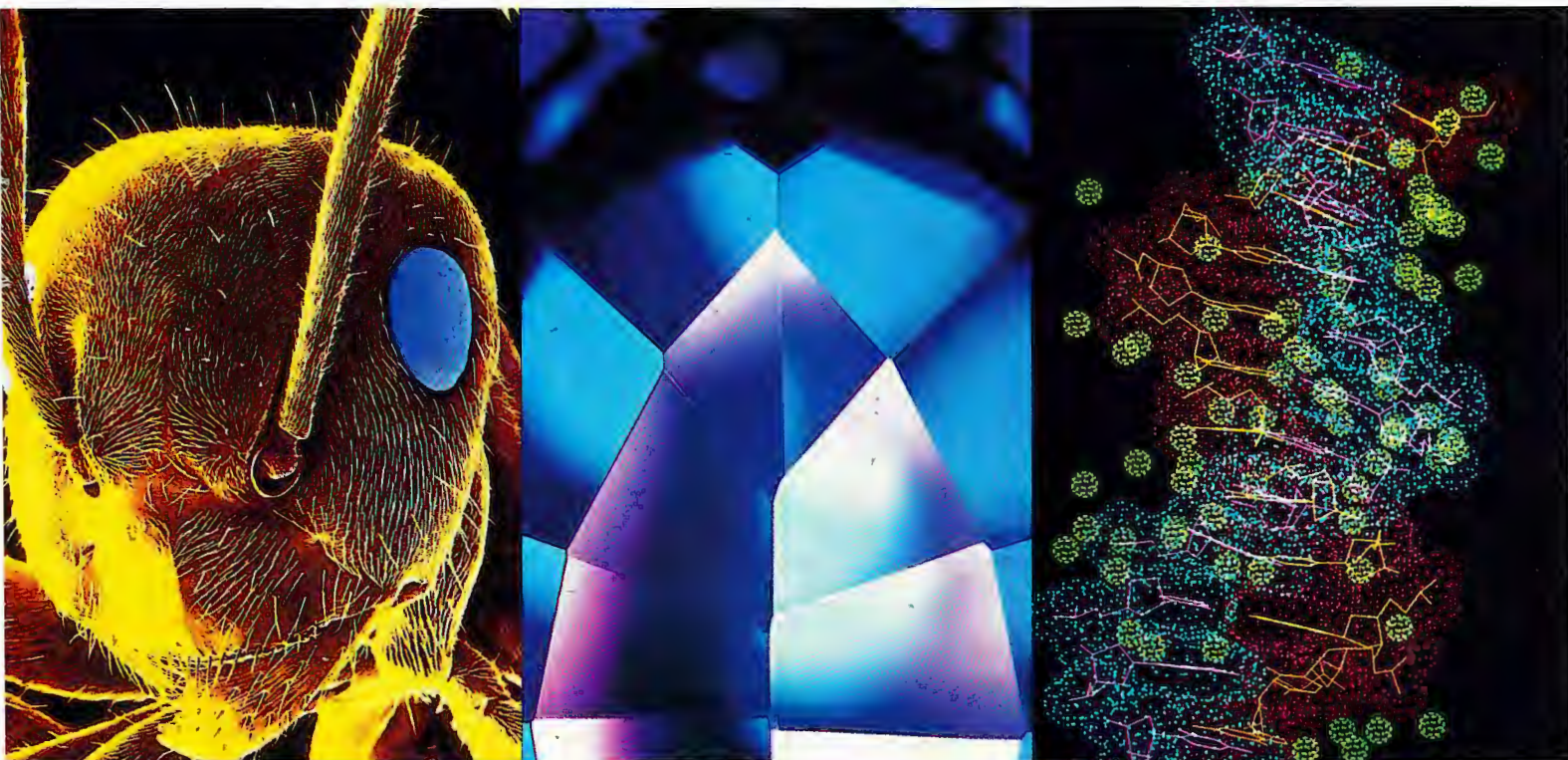
Bill

William R. Anderson

1. S.H. Koenig, R.D. Brown III, and M. Spiller, Magn. Reson. Med. 4, 252 (1987).



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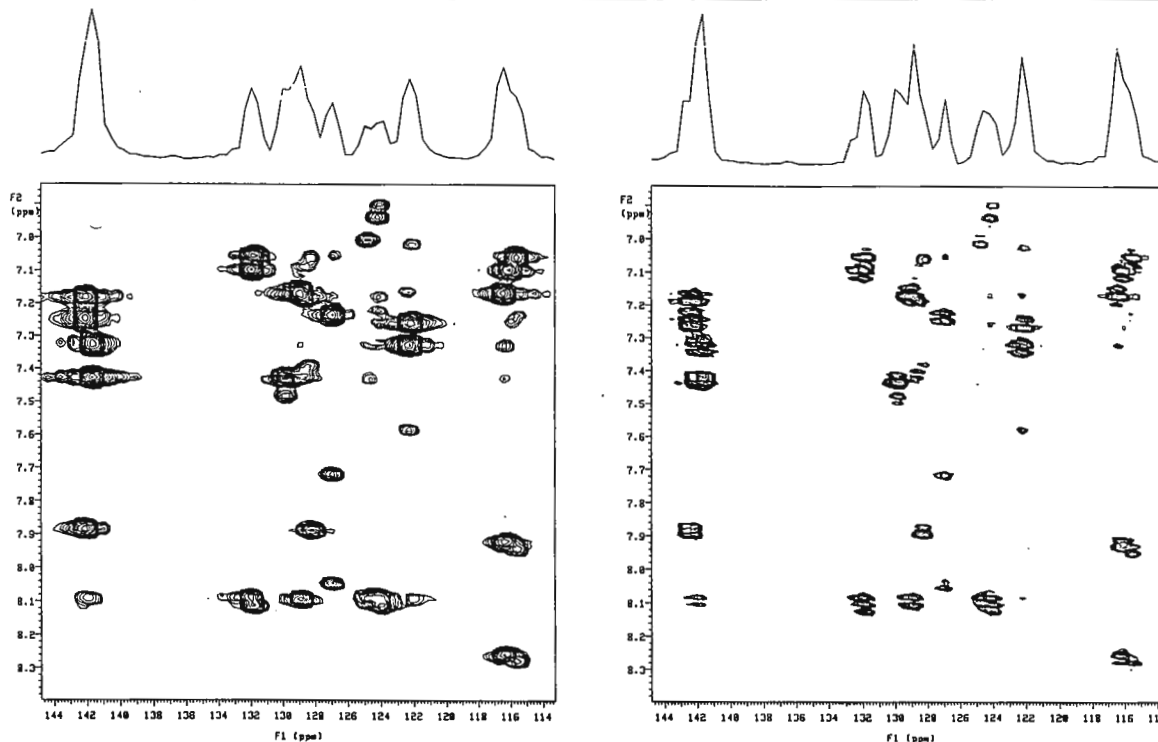
NMR WITH A FUTURE

varian 

MIXED MODE PHASING IN 2D: MAGNITUDE/ABSORPTION IN HMBC

The long (50 msec) fixed delays present in the HMBC pulse sequence allow homonuclear J modulation which cannot be refocused, and this leads to mixed phase multiplets when the t2 data is transformed. One way around this is to display and process the data in magnitude mode, constructing the interferogram from positive-only signals. Since the maximum evolution time in this experiment is very short, J modulation has little time to have an effect, and data in t1 can be transformed and displayed in absorption mode. UNITY software has mixed-mode processing as a standard feature.

This allows the operator full flexibility in choosing the type of data processing and presentation appropriate for any pulse sequence. The data below show a comparison of phase modes for a strychnine HMBC data set. The left expansion shows the traditional magnitude-only display, while the one at the right shows the same data processed with magnitude mode in F2 and absorption mode in F1. The better lineshape and resolution is evident. Sensitivity is improved as well, since weighting functions such as sinebell or pseudoecho are not used to force an absorption-like lineshape in magnitude mode.



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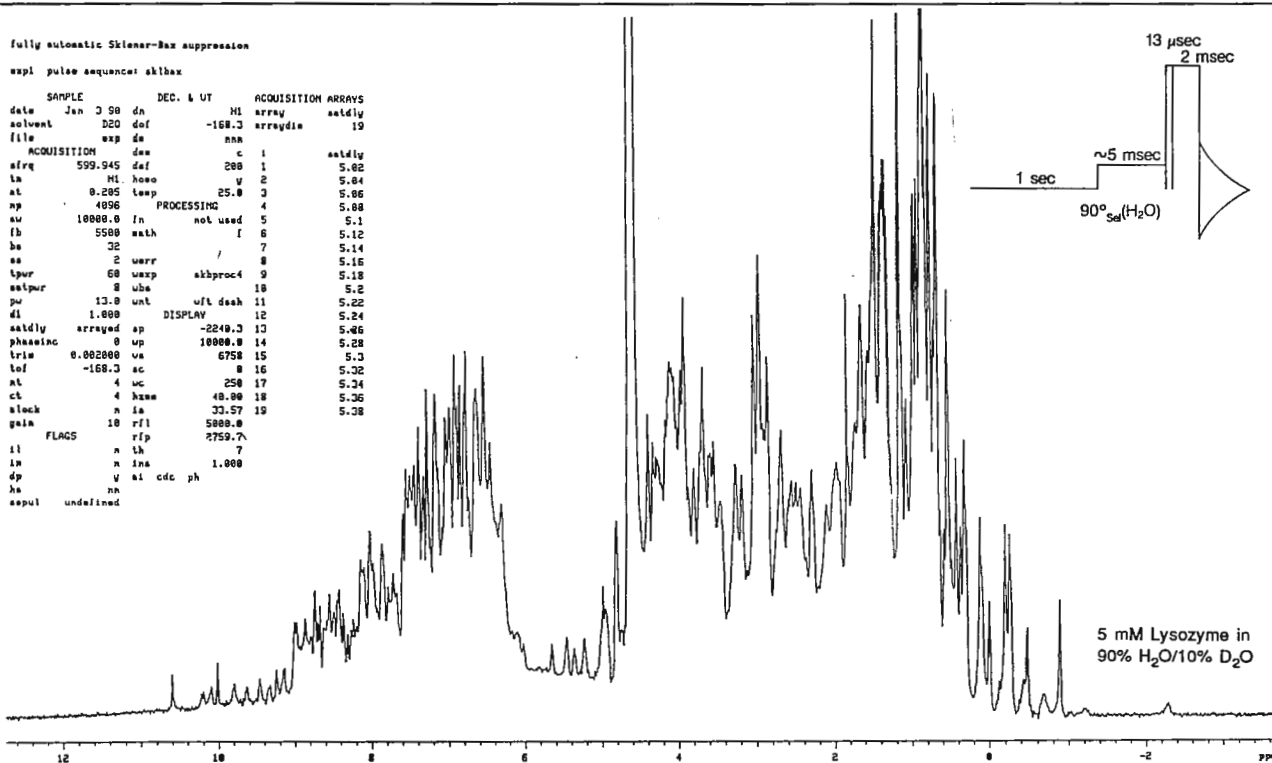
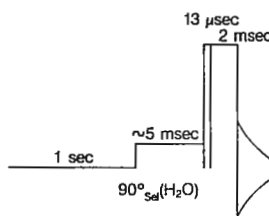
Widespread use of valuable techniques is sometimes hindered by its "non ease of use". Through the use of "arrays", MAGICAL (Varian's NMR Expert System), and user-written menu "buttons", the above experiment can be made automatic. One click on a button and the proper conditions can be iteratively determined by the spectrometer in a series of experiments where the soft pulse is successively refined in accuracy down to the 20 usec range. Analysis macros are automatically run after each experiment to determine the best water suppression and

based on these results the next experiment is set up. The whole procedure takes only a few minutes and the soft pulse is then determined to an accuracy of 0.5%. This 600 MHz spectrum was plotted as presented by the final analysis macro. It chose the best value for the soft pulse and displayed the associated spectrum, only adjusting the frequency-independent phase correction. The parameter table shows the final array of soft-pulse (in msec) values for these four-transient runs.

fully automatic Sklenar-Bax suppression

expt pulse sequence: sklbax

SAMPLE		DEC. & UT		ACQUISITION ARRAYS	
date	Jan 3 90	dn		MI	array
solvent	D2O	dof	-168.3	array	array
file	exp de	nan		array	19
ACQUISITION		PROCESsing		DISPLAY	
freq	599.945	del	200	1	5.02
la	MI	homo	v	2	5.04
at	9.295	loop	25.0	3	5.06
ap	4096			4	5.08
av	10000.0	in	not used	5	5.1
fb	5500	math	f	6	5.12
be	32			7	5.14
ee	2	uerr		8	5.16
tpur	60	wexp	skbproc4	9	5.18
estpur	9	uba		10	5.2
pu	13.0	unt	ufl dash	11	5.22
di	1.000			12	5.24
array	arrayed	ap	-2249.3	13	5.46
phaseinc	0	up	10000.0	14	5.28
tris	0.002000	va	6758	15	5.3
tof	-168.3	ac	8	16	5.32
at	4	uc	250	17	5.34
ct	4	hsum	40.00	18	5.36
alock	n	ia	33.57	19	5.38
gain	10	rfl	5000.0		
il	FLAGS	rfl	2759.7		
in	n	th	7		
ip	n	ina	1.000		
dp	y	ti	cdc ph		
he	nn				
aspl	undefined				



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NMR Applications Laboratory

May 7, 1990

(received 5/8/90)

Dr. Barry Shapiro
966 Elsinor Court
Palo Alto, CA 94303

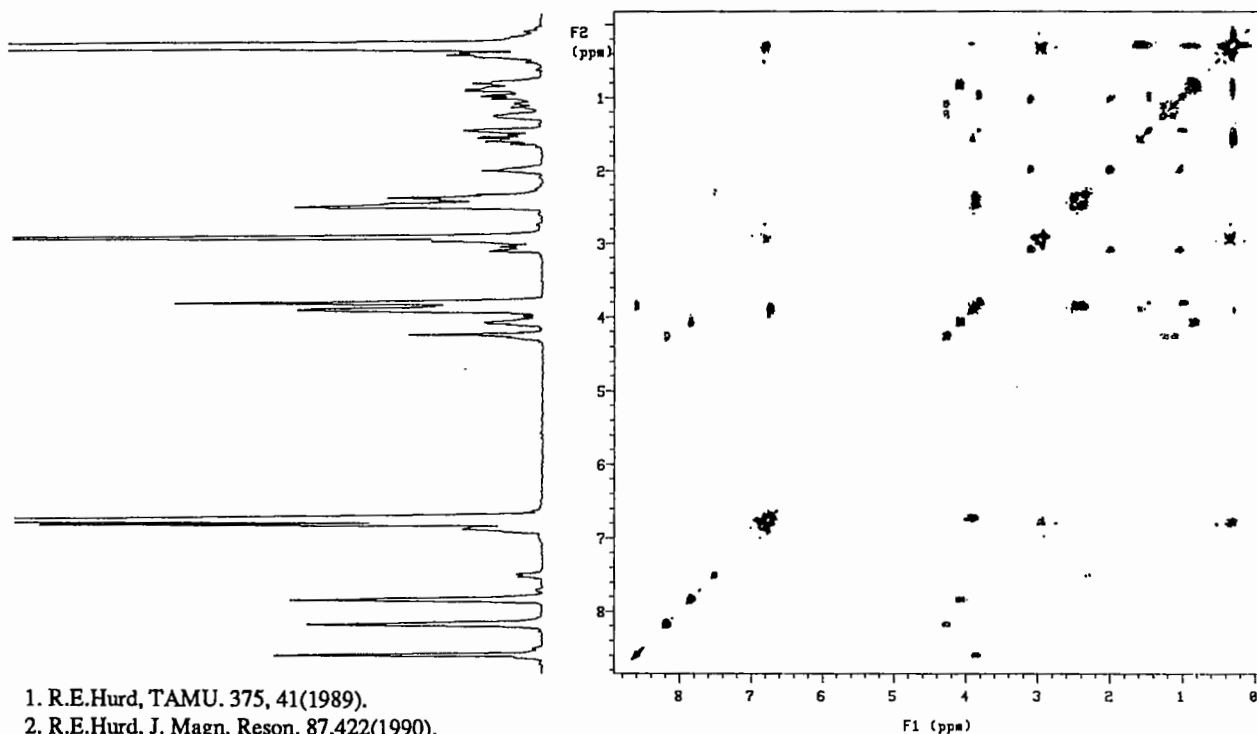
Pulsed Field Gradient Spectroscopy / BURP (excuse me!)

Dear Barry,

PEGS

There is a lot of interest now in the use of pulsed field gradients for applications other than imaging. This is, in part, because newer instruments have integrated this capability into the spectrometer system. Recent reports (1,2) have illustrated some of the applications. While having these capabilities available from manufacturers is relatively recent, it should be pointed out that these applications have been reported (3) a number of years ago, in particular the use of pulsed field gradients for F1 quadrature without phase-cycling, higher-order quantum selection in single transients, and solvent suppression. In this 1985 paper, Barker and Freeman described the use of pulsed field gradients (without "actively shielded gradients or gradient compensation) on an XL-200 that was six years old at that time!

We have used the external (compensated) gradient set available with UNITY microimaging hardware to explore the utility of PFGS in multidimensional liquids applications. This setup allows the use of a standard liquids probe that simply slides up inside the external gradient set within the normal room temperature shim set in a widebore magnet on a UNITY 300. Once the gradient compensation has been done the probe may be used at any future time without redetermining the compensation time constants. In this case, a standard 5mm $^1\text{H}/^{19}\text{F}$ probe was used. We found that use of the Z-gradient only was adequate for proper coherence pathway selection. The spectrum below shows a double-quantum filtered COSY spectrum of gramicidin-S in DMSO- d_6 . Only one transient per fid was accumulated for these phase-sensitive data.



1. R.E.Hurd, TAMU. 375, 41(1989).
2. R.E.Hurd, J. Magn. Reson. 87,422(1990).
3. P. Barker and R. Freeman, J. Magn. Reson. 64,334(1985).

BURP! (That was delicious data!)

The same waveform generator that we use for shaping gradients in microimaging may also serve for selective RF excitation. From the days of "tailored excitation" (4), through DANTE (5) and multiple applications of amplitude-modulated pulses, the literature has been replete with new pulses to try. One of the most recent of these was described at April's ENC in Asilomar by Geen and Freeman-- BURP (Band-selective Uniform excitation Radiofrequency Pulse). This, along with variants such as UBURP and EBURP, are the product of iterative numerical analysis to achieve a pulse shape of desired excitation profile(6). For the most general applications, pulses such as these will exploit both phase and amplitude modulation to achieve a desired performance level.

In the spectra below, various degrees of band selectivity are illustrated. All these spectra were taken in one experiment wherein both the width and amplitude of the "tophat" pulse were specified for each spectrum using a double(linked) array (widths ranged from 2.1 to 122 ms for the lower to upper spectra). All spectra have the same phase corrections. The absence of large frequency-dependent phase corrections means that this pulse can serve as one of the pulses in a region-selective 2D experiment without the phase roll problems in phased mode (or sensitivity loss in magnitude mode) associated with gaussian pulses.

4. B.L. Tomlinson and H.D.W. Hill, J. Chem. Phys. 59,1775(1973).

5. G.A. Morris and R. Freeman, J. Magn. Reson. 29, 433(1978).

6. H. Geen and R. Freeman, J. Magn. Reson. 87, 415(1990).

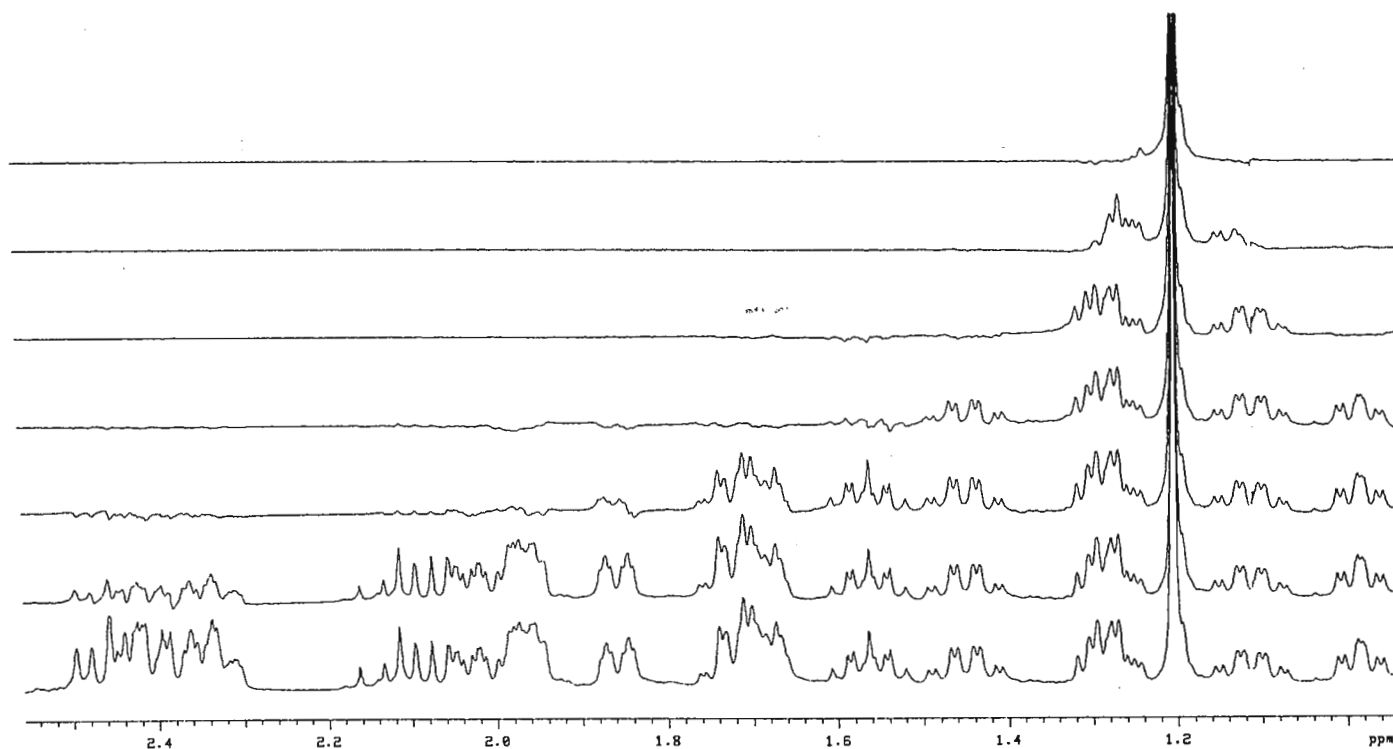
Sincerely Yours,


George Gray


Evan Williams


Paul Keifer

P.S. Although you don't spend much time nowadays at a spectrometer, I understand that you are now required to do considerable water suppression! Cheer up, into each life a little rain must fall--eventually!



THE UPJOHN COMPANY

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TELEPHONE (616) 323-4000

(received 5/14/90)

Dr. B.L. Shapiro
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto, CA 94303

Protein NMR Data Analysis Resource

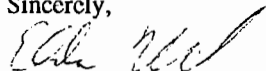
Dear Dr. Shapiro

Correlating the results from protein NMR experiments with the structure, dynamics, and chemistry of the protein involves looking at the data and the protein from several different angles. This often requires the use of information located in various articles and reviews. I have made an attempt to collect some of this information and to make it easily accessible through a computer program. The software has three primary functions: 1) Display a protein sequence and provide crude tools for the analysis of the primary structure (locate and color amino acids, hydrophobicity profiles, helical wheel plots, theoretical charge vs pH plots, locate unique, repeat, and non-unique peptides). 2) Provide rapid access to often used protein NMR data (coupling constant vs dihedral angle plots, random coil amino acid ¹H, ¹³C, and ¹⁵N chemical shift tables, low resolution amino acid COSY, RELAY, and HOHAHA coupling patterns, and NOE intensity vs secondary structure tables, calculated proton-proton distances from PDB data files). 3) Provide utilities for the entry of protein NMR data (chemical shifts currently, NOE, coupling constant, T1, and other data in the future) so the data can be organized and kept in an easily accessible location during the execution of an NMR protein project.

The software is now relatively stable and I would be glad to provide copies to anyone interested. The code is written in Microsoft QuickBasic (nothing state of the art) and is divided among several modules. A menu interface is used to control the program. Hardware required includes an IBM compatible microcomputer equipped with a hard disk and a VGA monitor. The software, data files and accompanying database including the sequences and proton chemical shifts reported for >30 proteins requires four 3 1/2" high density (1.44 MB) diskettes or four 5 1/4" (1.2 MB) floppies. If any of your readers would like a copy of the software, all I ask is that they send four of the appropriate disk media. In return I will provide a manual for the program, the executable form of the software, the source code, and the current data files. As the program evolves, I will attempt to inform users of major updates.

Please credit the account of Paul Fagerness for this contribution.

Sincerely,



Eldon Ulrich
Control Division
M/S 4834-259-12
The Upjohn Co.
Kalamazoo, MI 49001



THE UNIVERSITY OF ADELAIDE

BOX 498, G.P.O., ADELAIDE, SOUTH AUSTRALIA 5001

Dr B. L. Shapiro,
TAMU NMR Newsletter,
966 Elsinore Court,
Palo Alto, CA 94303,
USA

1 May, 1990
(received 5/10/90)

TITLE : The Case of the "Disappearing" Coupling Constant

Dear Dr. Shapiro,

During recent studies, in collaboration with Kevin Wainwright and Rebecca Keough of Flinders University, into heavy metal/pendant arm macrocyclic ligand complexes, we doubly ^{13}C labelled the ethylene linkage in the hydroxy-ethyl arms of the ligand, A, to follow an intramolecular exchange process in the Cd^{2+} complex. We were surprised when the ^{13}C spectrum revealed only one signal at room temperature - we were expecting an environmentally averaged AB quartet.

On lowering the temperature, the single signal split into two AB quartets (see Figure), indicating that there were two environments for the pendant arm of the macrocycle, in slow exchange.

This observation is consistent with the reversal of the relative chemical shifts of the $\text{N}-\text{CH}_2$ and CH_2-OH carbons on exchanging between bidentate and monodentate environments, resulting in a "singlet" (i.e. AB quartet with a very small δ_{AB}) under fast exchange conditions.

Regards,

A. Hounslow

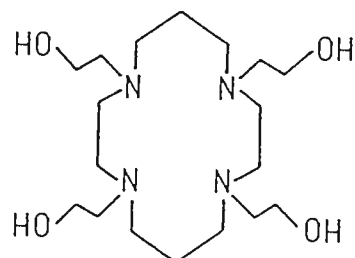
A. M. Hounslow

S. F. Lincoln

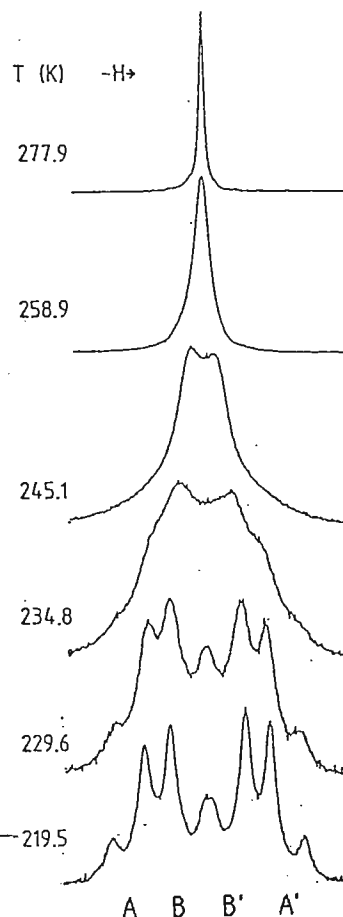
S. F. Lincoln

P. Clarke

P. Clarke



A



Figure

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Power Supply	TECHRON Model 7570

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Synthesizer	PTS-500, Computer Controlled
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Phase, Variable Steps	0.1 Degree Resolution
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Power Control	0 - 100 %
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Bore With RT Shims And Gradients (cm)	26.5	26	33	15	26.5	31	15
Helium Evaporation (ml/hr)	50	50	50	50	55	60	50
Nitrogen Evaporation (ml/hr)	400	400	400	400	450	500	400
Half Length (mm)	350	275	570	396	460	735	575
5 Gauss Line — Radial From Center (m)	3.4	3.2	4.7	3.7	5.3	6.4	5.1
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DER
TECHNISCHEN UNIVERSITÄT MÜNCHEN

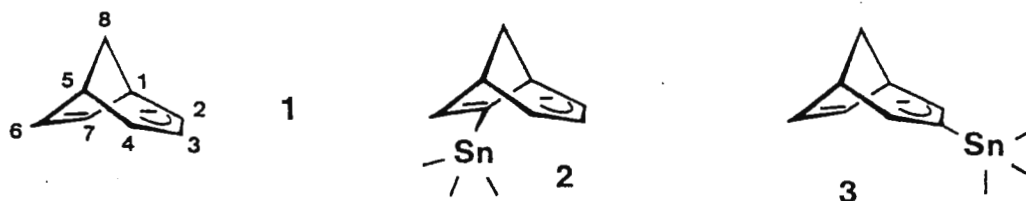
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May 3, 1990
(received 5/9/90)

Dr. B. L. Shapiro
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto, California 94303
U. S. A.

$J(^{119}\text{Sn}-^7\text{Li})$ and $\delta(^7\text{Li})$ of sandwiched carbanions

Dear Dr. Shapiro,
organic chemists are fascinated by the bicyclo[3.2.1]octa-2,6-dienylanion (1) because of its conceivable homoaromaticity. When we became interested in 1 and its story our more practical question was: in how far may 1 replace the cyclopentadienyl anion? After having established the solid state structure of **1Li(TMEDA)** we set out to study 1 in solution.

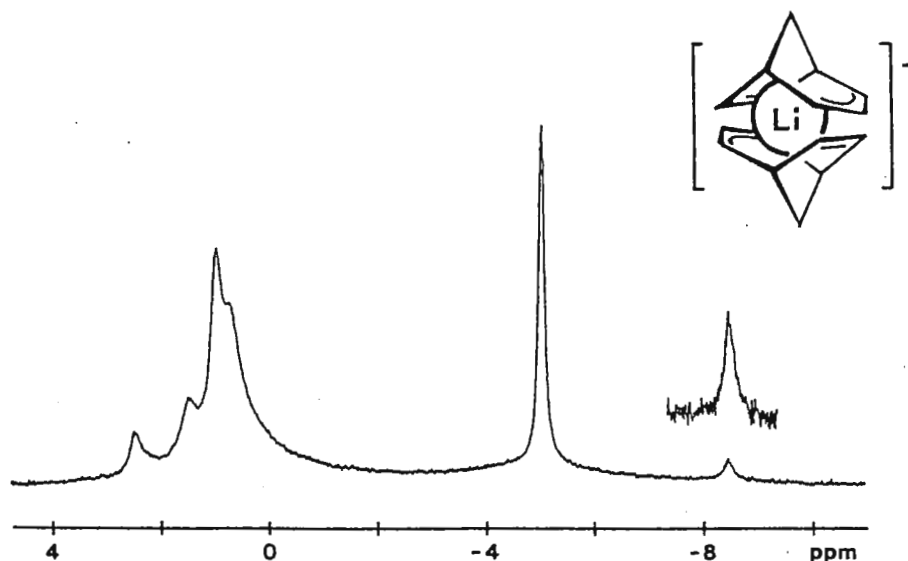


First temperature dependent ^{13}C NMR spectroscopy revealed that **1Li** exists in a contact-ion-pair/solvent-separated-ion-pair equilibrium. The most pronounced change with temperature was found for the $\delta(\text{C6/7})$ -values. We take this as strong evidence that lithium experiences a strong interaction not only with the allyl but also with the olefin part of 1.

When 1 was substituted the CIP/SSIP equilibrium changed; for instance, the CIP side of the equilibrium was favoured for **2Li**. A particularly useful probe for the metal-carbanion interaction was ^{119}Sn NMR. Thus at low temperature we observed a splitting of the tin resonance of **3Li** with a 1:1:1:1 pattern (approximately) and a spacing of 6.2 Hz. Obviously, this is a $^{119}\text{Sn}-^7\text{Li}$ coupling which does not seem to have been reported earlier.

A mixture of **2Li** and its 6-stannyl isomer gave two sets of ^{13}C NMR signals. This points to the formation of diastereoisomers in which two carbanions and Li^+ form a triple ion. An independent proof was obtained from the ^7Li NMR spectrum of **1Li** at -126°C which is shown below. There the resonance at -5.0 ppm belongs to the monomeric CIP and that at -8.5 ppm to the triple ion sandwich also shown in the figure. It is gratifying that the latter shift corresponds to the range which has been established by Fraenkel and Hallden-Abberton [1] and which has been found for lithium cyclopentadienyls by Paquette, Bauer, Sivik, Buhl and

v. Schleyer [2]. Hence, from the NMR point of view 1 may be regarded as a strongly disturbed cyclopentadienyl.



Details of this work will be published in Z. Naturforsch., B: Anorg. Chem. Org. Chem. to honor Prof. H. P. Fritz, a former member of the TAMU NMR Newsletter family and one of the editors of Z. Naturforsch., on the occasion of his 60th birthday. By the way, the journal publishes an increasing number of articles in English to meet the requirements of more foreign chemists.

Yours sincerely,

F. H. Köhler
(F. H. Köhler)

N. Hertkorn
(N. Hertkorn)

[1] J. Am. Chem. Soc. 103(1981)5667. [2] Ibid. in press.

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WINTON HILL TECHNICAL CENTER
6071 CENTER HILL ROAD, CINCINNATI, OHIO 45224-1703

March 27, 1990
(received 4/30/90)

Dr. Bernard L. Shapiro
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto, Ca. 94303

Subject: In Vivo NMR STUDY OF MICROORGANISMS UNDER OSMOTIC STRESS.

Dear Dr. Shapiro:

We have recently begun examining viable bacterial cells in aqueous media using proton nmr. Our subject bacterium is the foodborne disease microorganism Listeria monocytogenes. L. monocytogenes has the ability to withstand harsh environmental conditions such as extremes of pH and temperature; however little is known with regard to its reaction to osmotic stress. Many bacterial species under this type of stress accumulate intracellular "osmoprotectants" such as potassium ions, amino acids, and fully methylated amino acid derivatives called betaines. Our quest is to examine Listeria in vivo and identify differences that occur between osmotically stressed and unstressed cells. As of this writing, we have successfully used proton NMR to identify the presence of glycine betaine in viable whole cells. In addition, we are using magnetic resonance to compare the amount of this betaine produced in Listeria cells subjected to a range of inhibitory water activities (a_w). Since this was our first serious attempt at merging the mysteries of microbiology with the bedrock world of nmr, we thought we would share some of our preliminary learnings with your readers.

First, we were helped in our efforts by using a presaturation pulse sequence written for our QE-300 by Ms. S. Lee of Procter & Gamble's Miami Valley Laboratories. As shown following, in QE pulse dialect, it contains four composite 90 degree pulses (x,y,-x,-y) in addition to decoupler presaturation. Although not overly sophisticated, this sequence worked extremely well for our "100%" H₂O samples, and did not promote the "nightmare" phasing problems associated with some solvent suppression sequences.

COMPOSITE 90 DEGREE PULSE WITH DECOUPLER PRESATURATION

#1: D6,N,CO,A	P2 = Exact 90 degree flip angle
#2: D4,A	D4 = 5 ms
#3: P2/0,A	D6 = 1.1 sec
#4: P2/1,A	
#5: P2/2,A	
#6: P2/3,A	QPD On, Y=4
#7: A,A	AB On
#8: D2,X	

L1 adequate to saturate water peak (2850 - 3050)

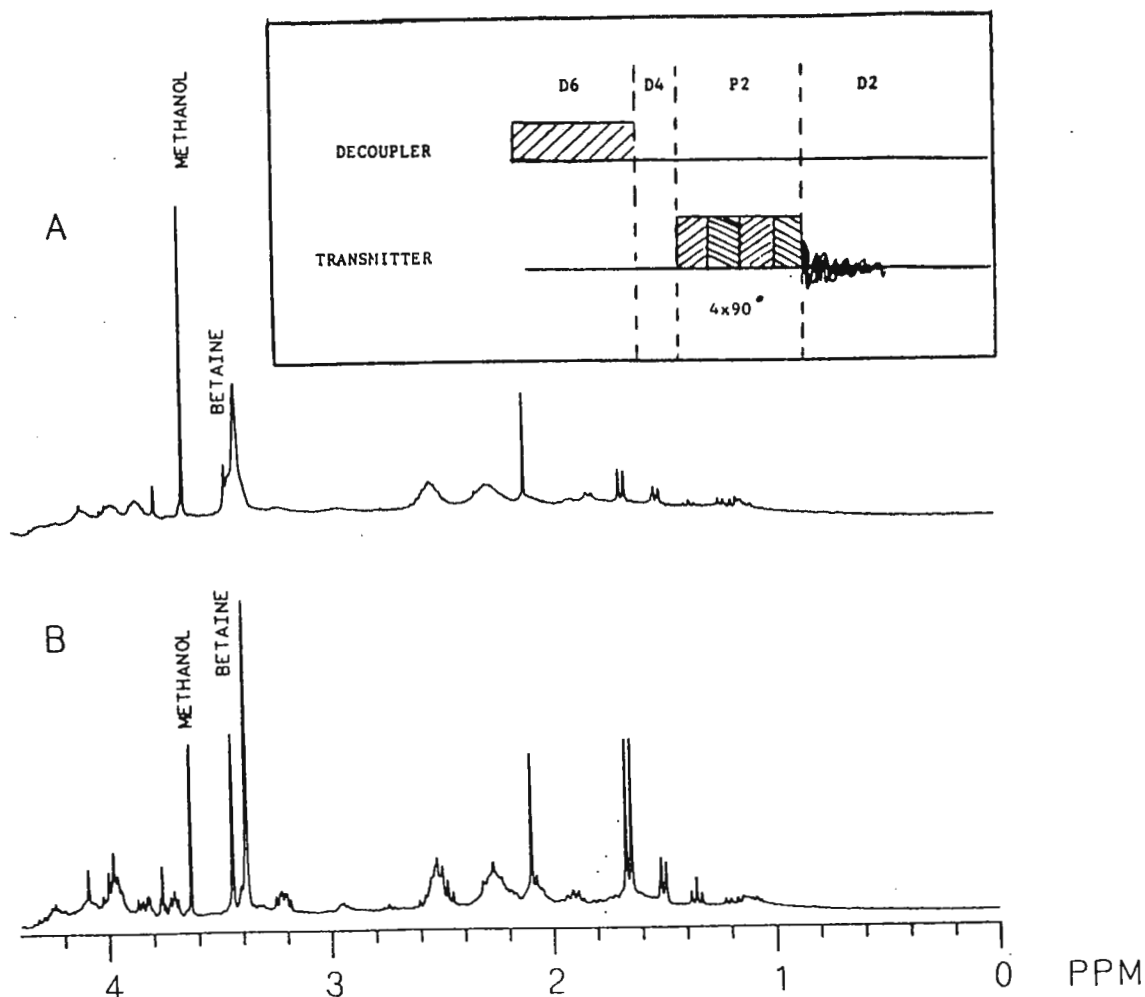
Second, in order to quantitate the growth (or decrease) in the organic solutes observed, and provide a deuterium lock, we introduced a sealed 1mm O.D. capillary containing methanol in D_2O . The D_2O provided a very stable, shimmable lock signal, while the methyl proton resonance of the methanol fortuitously came "in the clear" regardless of the water activity (a_w) employed. For comparative purposes, sample spectra of Listeria cells at $a_w = 0.95$ are shown in Figures A and B. Figure A was recorded shortly after the cells were stressed. Figure B was obtained for the same sample several days later. Both spectra result from 240 acquisitions representing a total experiment time of approximately 20 minutes. The glycine betaine methyl peak as well as the external methanol reference are labeled in both. Note the increase in the betaine resonance with time. We have measured the concentration of the glycine betaine pool in this sample at 0.07mg/ml. It is obvious from these spectra that a number of other things are happening in these cells... our work has just begun.

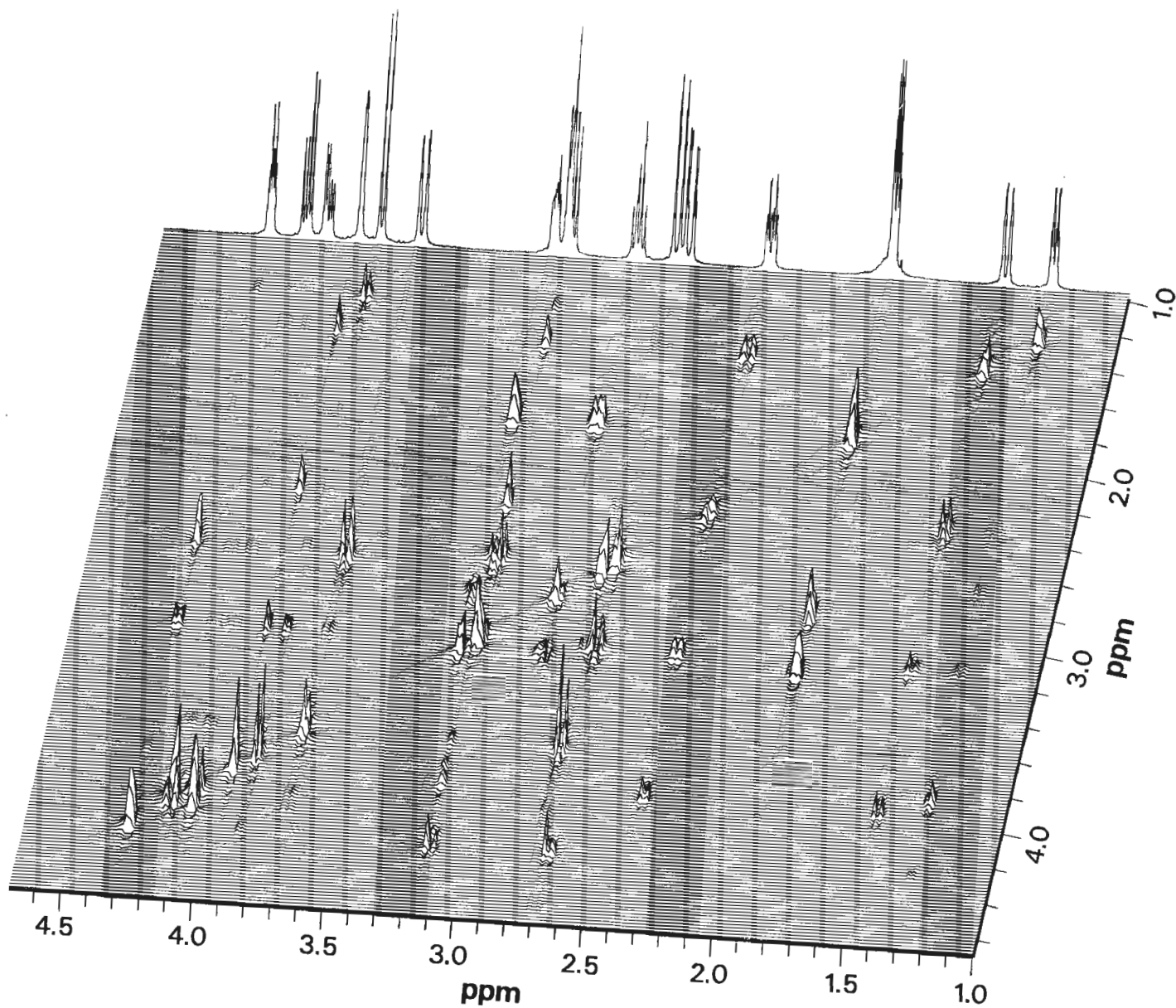
Please credit S. Lee's account.

Sincerely,

J. D. Wendel
J. D. Wendel

D. R. Gardner
D. R. Gardner



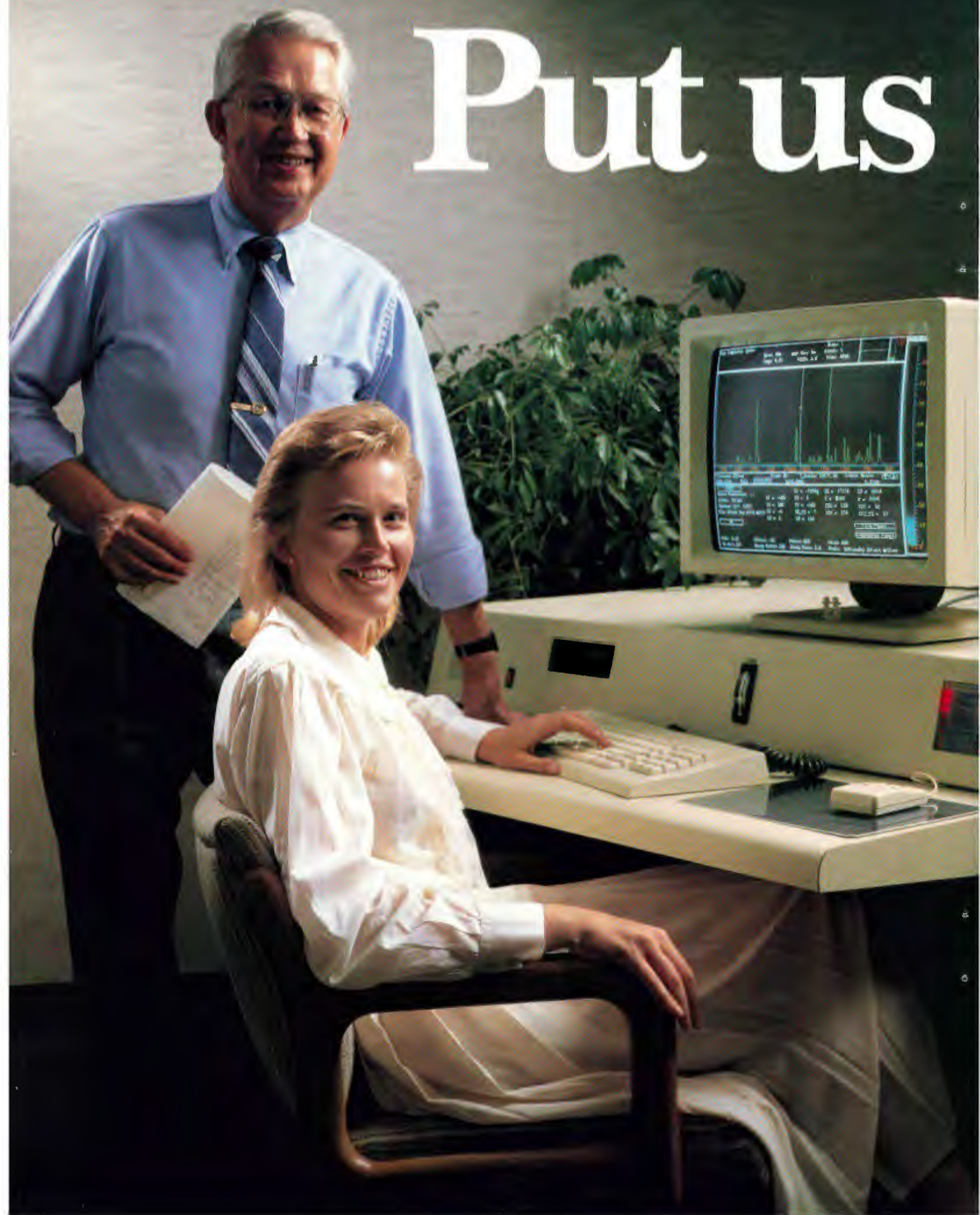


HOHAHA of Strychnine on an Omega 600



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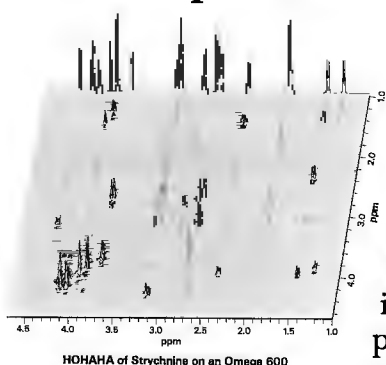


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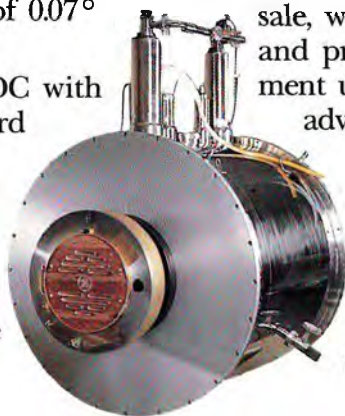
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Dynamic range vs. spectral width; spectral width vs. digital resolution. Trade-offs have been required due to NMR system hardware limitations. With the Omega™ Data system's Alpha HDR digitizer, no trade-offs are necessary. As shown in Figure 2 with a 16-bit dynamic range, 200 KHz spectral width, 64-bit complex acquisition word size, and up to 32 MBytes (4 MWords complex) of on-board acquisition memory available, the spectrometer is no longer the limiting factor when designing the most demanding experiments. Other outstanding features of the Alpha HDR include variable dwell periods, phase shifts of each sampled data point as small as 0.05 degrees, and segmentation of the digitizer memory into as many as 64K blocks. These features further distinguish the GN-series spectrometer equipped with the Omega Data System as the leader in NMR technology.

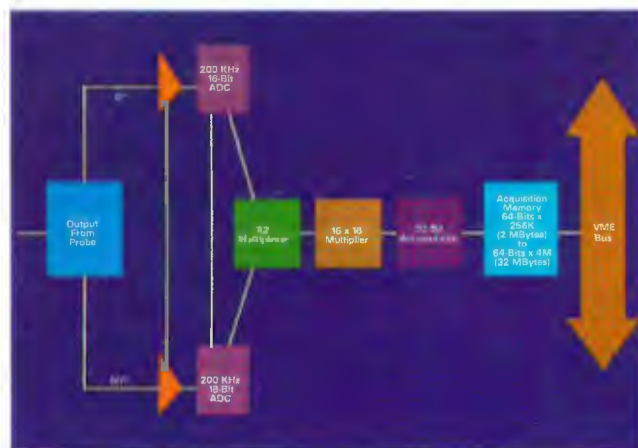


Fig. 1
The Alpha HDR digitizer.

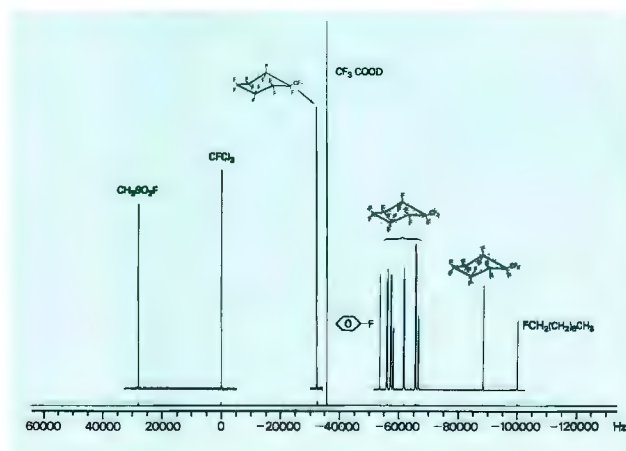
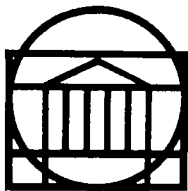


Fig. 2
200 KHz spectral width ^{19}F spectrum acquired on a GN-500 Omega System. Note the extremely flat baseline obtained with the Alpha HDR.



GE NMR Instruments

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UNIVERSITY OF VIRGINIA
DEPARTMENT OF CHEMISTRY
McCORMICK ROAD
CHARLOTTESVILLE, VIRGINIA 22901

April 30, 1990 (received 5/7/90)

Dr. Bernard L. Shapiro
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto, California 94303

Addition of CPMAS Capability to NT-360 NB Spectrometer

Dear Dr. Shapiro:

We have recently added a high resolution solids capability to our Nicolet NT-360 spectrometer with narrow bore magnet. When carrying out this project, we avoided any major construction and obtained the main items necessary for cross polarization and magic angle spinning (CPMAS) from commercial sources. The only construction/modification we performed was to make some cables and alter connections in the NT H-1 decoupler and broadband observe units. New items included a variable temperature MAS probe, 500 watt 8-130 MHz rf amp, and duplexing circuit, all from Doty Scientific. Also, a Tecmag Deokit 2A decoupler controller and Henry Radio 1 kW 360 MHz rf amp were obtained.

The Nicolet probe interface was replaced by a Doty duplexing circuit which is capable of handling higher power. The rf output of the NT observe unit was attenuated 8 db, the final NT pulse rf amp was bypassed, and the rf was fed to the Doty amp. The observe rf was gated in the observe unit, and a gate for the Doty amp was derived from observe unit gate. The output of the Doty amp was put into the duplexing circuit.

The precision and speed of the rf phase and amplitude control in the NT H-1 decoupler were insufficient for cross polarization. Therefore, we obtained the Deokit 2A for H-1 rf control. Modification of the NT decoupler and incorporation of the Deokit 2A was done in the following way. All rf gates in the NT decoupler were removed, as was the phase shifter. The output of the buffer amplifier was attenuated 12 db so that its amplitude was 1 volt and was put into the Deokit 2A. Lines for rf amplitude and phase control were removed from the NT decoupler and put into the Deokit 2A. The rf output of the Deokit 2A was routed to the final NT decoupler amplifier. The rf output of the NT decoupler was put into the Henry amp, and the Henry output was put into the Doty probe. The probe uses a maximum of 40 watts of H-1 rf power, so an amplifier with a considerably smaller maximum output could be used in this application.

C-13 CPMAS spectra of solids have been obtained by using rotating frame field strengths of 40 KHz for cross polarization and 50 KHz for H-1 decoupling. Also we are interested in biological membranes and have obtained C-13 MAS spectra of hydrated dispersions of phospholipid and cholesterol. The spectra are very similar to those obtained by Oldfield et al. Biochem 26 (1987) 6919. The spectrometer can be switched between the solids configuration and most solutions configurations in five minutes. A switch between a solutions configuration utilizing H-1 decouple and observe rf and the solids configuration takes about 15 minutes. We would be happy to discuss the above with anyone who is interested. We are grateful for the assistance received from Doty Scientific, Henry Radio, and Tecmag.

Jeff Ellena
Jeff Ellena
(804) 924-3163

Laurie Kelsh
Laurie Kelsh

M. J. Grubb
Marvin Grubb

Dave Caffso
Dave Caffso



THE UNIVERSITY OF MANITOBA

DEPARTMENT OF CHEMISTRY

Winnipeg, Manitoba
Canada R3T 2N2(204) 474-9321
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May 4, 1990 (received 5/15/90)

Small, positive and rational.

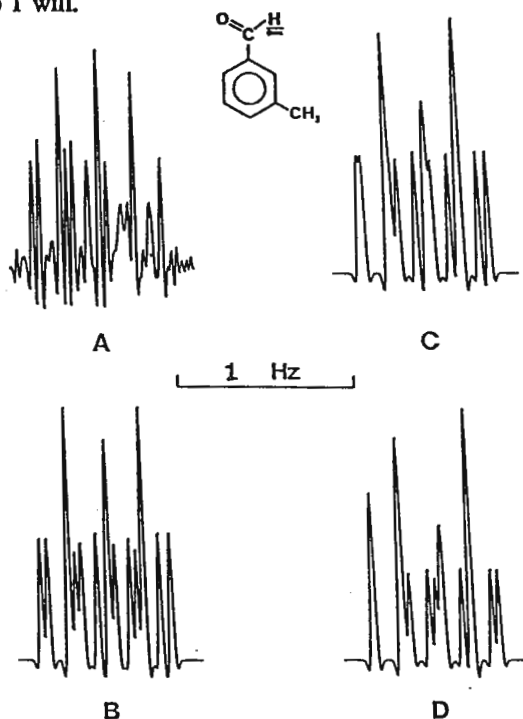
Dr. B.L. Shapiro,
TAMU Newsletter,
950 Elsinore Court,
Palo Alto, CA 94303.
U.S.A.

Dear Barry:

The pursuit of small, long-range coupling constants provides entertainment and information, of course, but also some knowledge. The aldehydic proton nmr spectrum of 3-methylbenzaldehyde at 300 MHz (acquired by Kerry Cox for a dilute solution in acetone- d_6), shown as A, is an example. The spectrum has been over-massaged, but demonstrates splittings of as little as 0.04 Hz or less. The aldehydic proton is coupled to all the ring protons (not to the methyl protons); the second-order nature of the (whole) spectrum entails that none of the splittings equals a coupling constant. However, as a consequence the sign and magnitude of the small coupling constant over six formal bonds, $^6J(\text{H,CHO})=^6J$, follows: in B, the simulated spectrum has 6J as +0.014 Hz, in C it has -0.014 Hz and D has 6J as zero. Does it matter that 6J is indeed positive? Well, yes.

Normally, coupling constants of this kind, involving a proton on the sidechain, are negative and depend on the degree to which the alpha C-H bond twists away from the aromatic plane (conformational implications). A number of INDO MO FPT computations of the kind described by Reino Laatikainen (Can.J.Chem. 64, 1859 (1986)) suggest that it's the $2p_z$ orbital on the oxygen atom of the carbonyl group which plays havoc with the 'normal' transmission of nuclear spin state information. We have hit on a simple empirical model of the effect of meta substituents on 6J (Can.J.Chem. 67, 827 (1989)); it predicts +0.012 Hz for 6J in the present molecule and invokes a valence bond structure of benzaldehyde (ionic at the carbonyl site), a structure first touted by Don Eaton and coworkers in their studies of contact shifts. I could maunder on at length, but you would no doubt say stop STOP! So I will.

Best wishes from


Ted Schaefer.


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Dr. B. L. Shapiro

TAMU NMR Newsletter

966 Elsinore Court, Palo Alto, CA 94303

RE: Lineshape analysis software for a Macintosh computer

DEPARTMENT OF CHEMICAL ENGINEERING
BERKELEY, CALIFORNIA 94720-9989

Dear Dr. Shapiro:

Over the past few years, tremendous changes have occurred in the personal computing environment. While the speed and functionality has increased, the quality of the software (in both ease of use and quality of output) has improved dramatically. LaserWriter (and usually POSTSCRIPT) output has become the norm, with complete control over fonts and point sizes in charts and graphs an expected capability. The quality of everything from lab reports to journal articles has improved while the time and effort to achieve this level has decreased. The recent hardware improvements allow this same standard of desktop computing to be brought to bear on the computing-intensive field of nonlinear curve fitting. Our goal is to develop various lineshape fitting algorithms to apply to spectra within the Macintosh environment with the same ease and power we find in other software applications. With the recent introduction of the Mac IIx (at about 12 Mips, the same as a Sparcstation) the Mac can finally be counted on as a full purpose workstation without sacrificing performance (actually, most of our analyses work quite well on a more affordable Mac SE/30).

Lineshape analysis provides useful information in all areas of spectroscopy, and has a great utility in solid state NMR. Computer programs for fitting data with common functions such as a Gaussian, Lorentzian, chemical shift power patterns, and combinations of these have been available for specialized applications on standard platforms (like a VAX) for some time. As the IBM PC became commonplace, and more powerful, these specialized and limited applications became available on that platform as well. Many a researcher will commonly go through the process of gathering data on specialized computer data stations, work it up there (on a VAX perhaps), transfer it to a PC for other customized manipulations, then put it on a Macintosh for ease in document preparation.

As mentioned in a previous contribution to this newsletter (no. 362-7) we have been using ASYST programs on a PC for much of our data analysis. However, much of this functionality is being supplanted by C code on the Macintosh to better mesh with our overall computing environment. We have also made use of *Mathematica* (from Wolfram Research) to aid in the development of these programs (as mentioned in our newsletter no. 371-13) but due to the current unavailability of a compiled version of *Mathematica* we have ported fitting routines to C. Although these routines are now fast, they lack some functionality while running in a sparse programming environment. We would like to include our C-code within a more flexible environment. MacGraphX, a new graphing application from Bravo Technologies, has the versatility to read both our PC generated raw data files as well as those manipulated in our ASYST programs on an IBM PC. It can also import files directly from our Mac II based spectrometer, reading the data directly out of Tecmag's MacNMR files (once the proper file specifications are added). By including our fitting program as a code resource to MacGraphX we gain the functionality and benefits of a supported "point and click" environment which we feel is quite beneficial for science and engineering applications. The development platform of Think C 4.0 on the Mac is a useful and powerful tool for programming on the Macintosh. When used in conjunction with MacGraphX, we have found the combination to be an effective tool for data analysis: allowing flexible and quick curve fitting in a format which allows control over final data output not easily obtained in other programs and practically impossible in a completely "home-built" application (like our ASYST programs).

We have developed data templates to allow the automatic importing of data from our home-built PC-based spectrometer as well as our Mac-based Tecmag machine. By applying standard least-squares analysis to our desired nonlinear functions we have recently applied some sums of Gaussian fits on the Mac. This code has been made available to commercial developers and academic users and we are optimistic in seeing a proliferation of inexpensive nonlinear curve fitting commercially available in the near future. We are continuing our efforts to fit anisotropic chemical shifts and quadrupolar lineshapes (as in the spin one powder pattern work in newsletter no. 317-13) in this manner.

Gaussian lineshapes have been fitted to data on various machines. The results for a 512 point data set (shown in figure 1) range from just over 7 seconds on a Mac II to just about 4 seconds on a Mac IIci. The SE/30 averaged between these results. All results assume 8-bit color on the Mac II series and 2 bit black and white for the SE/30. All computers were operated with 8 megs of ram and from internal SCSI hard disks. These times include the time spent drawing-- both the original data set and the resultant fit-- to the screen (i.e. a 2k data set took about 29 seconds on a Mac II, 13 seconds of which was calculation; the same set took 13 seconds on a Mac IIci, 7 seconds for calculation). In black and white mode on the Mac II, the 2k set still took 13 seconds for the calculation, but the total time dropped to 21 seconds due to quicker screen redrawing. Obviously the larger screen and color capabilities of the Mac II series makes data processing easier, but the results on readily available Mac SE/30's are very acceptable. In figure 2 we have a screen shot of a ^1H powder pattern of the organic solid acetanilide, with probable H_2O contamination appearing as the central sharp peak. We fit this 216 point data set with the sum of one Gaussian and one

Lorentzian in less than five seconds on an SE/30. (The quality of fit is limited by the actual shape probably needing another broad Gaussian added in). Anyone interested in obtaining our preliminary C code can write to us at the address above. For information about MacGraphX contact Bravo Technologies at P.O. Box 10078 Berkeley CA 94709-0078, or call 1-415-841-8552. Although still in Beta testing, MacGraphX should be shipping soon and be readily available (distributed mainly through mail order like their other products, SPAMM and MacCalc, are). A version of this of type of simple Gaussian fit should ship for free with the product, as should simple templates for data input and other mathematical transforms.

Figure 1

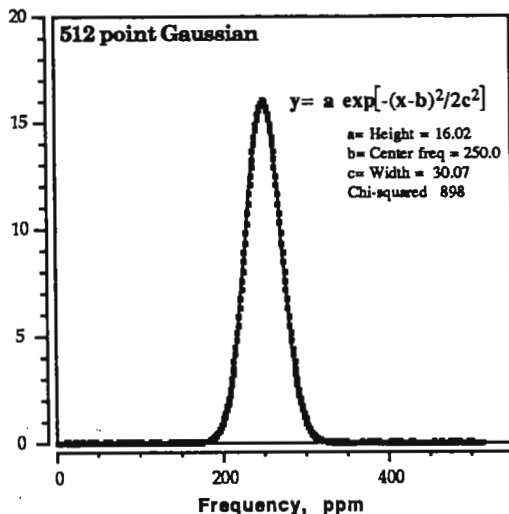
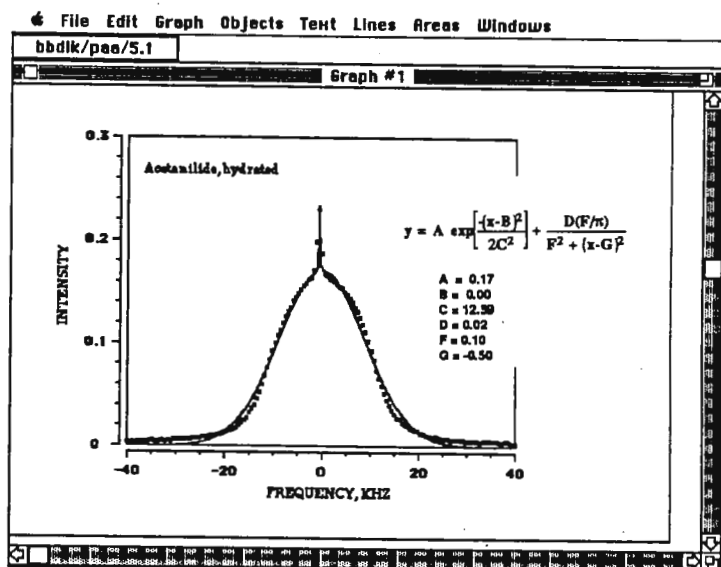


Figure 2



Sincerely,

Phillip A. Armstrong
Phillip A. Armstrong
Research Associate
Chemical Engineering

Bruce I. Berkoff
Bruce I. Berkoff
Research Associate
Biophysics

Jeffrey A. Reimer
Jeffrey A. Reimer
Associate Professor
Chemical Engineering

This contribution should be credited to J.R Havens of Raychem Corporation.

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(received 5/12/90)

Z•SPEC Four Nuclei Probe

FEATURES:

^1H , ^{19}F , ^{31}P and ^{13}C observe capability without retuning the probe. The four spectra shown on the back were obtained using this probe. The only thing the NMR operator does is change the observe frequency of the spectrometer. The probe contains no internal switches and thus cannot wear out from repeated observe nuclei changes.

APPLICATIONS:

The Z•SPEC Four Nuclei Probe is a great addition to any NMR lab requiring high efficiency of sample throughput. Laboratories with automatic sample changers or open access environments benefit from the increase in experimental flexibility.

TECHNICAL:

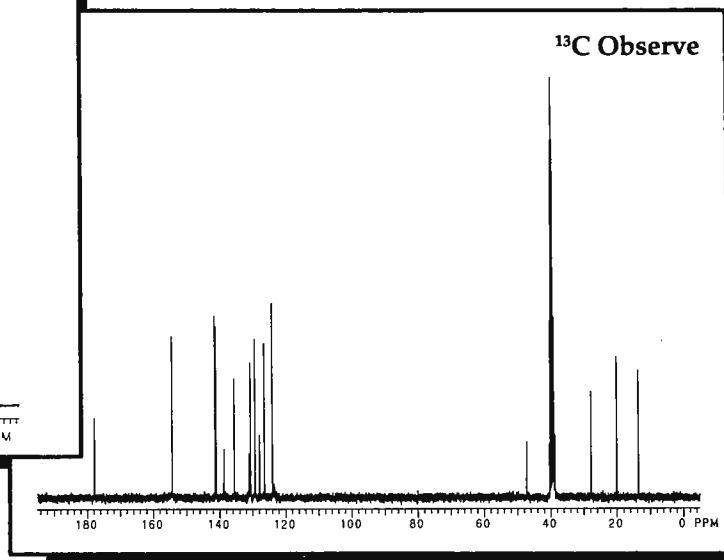
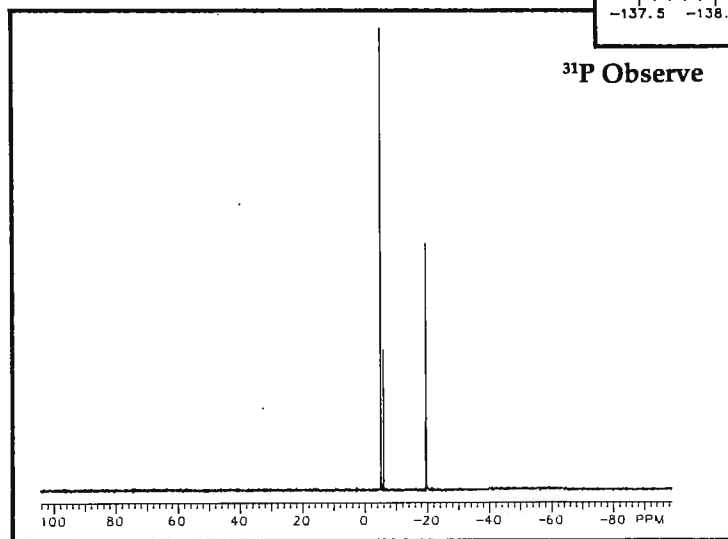
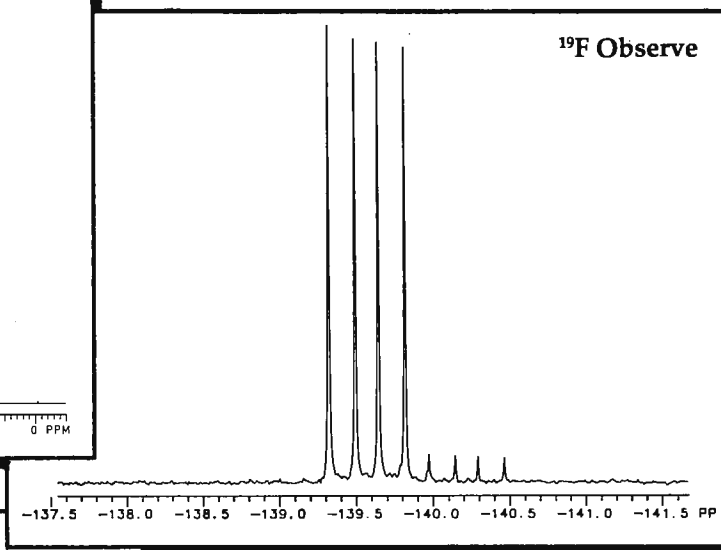
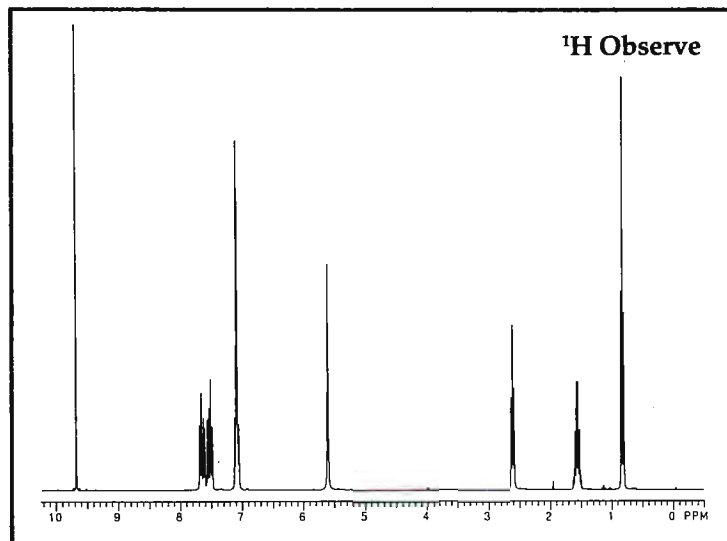
The Z•SPEC Four Nuclei Probe interfaces directly to any Varian 200, 300 or 400 MHz NMR Spectrometer. The probe is capable of observing any of the four nuclei without retuning the observe frequency or changing 1/4 wavelength cables.

For more information, please contact Toby Zens, Manager of the Z•SPEC Products Group.



NALORAC CRYOGENICS CORPORATION
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Tel: (415) 229-3501 • FAX (415) 229-1651

Z•SPEC Four Nuclei Probe Spectra*



* Spectra obtained with spectrometer operating in an automatic and unattended mode.

NCC

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TAMU NMR Newsletter

Policies and Practical Considerations

(Revised May 1990)

The TAMU NMR Newsletter (formerly the IIT NMR Newsletter, and originally the Mellon Institute NMR Newsletter) continues with the same name, under the aegis of Texas A&M University, although the undersigned Editor/Publisher now resides in California. The Newsletter, now in its thirty-second year of consecutive monthly publication, continues under the same general policies as in the past. All communication with the Newsletter must be directed to the address overleaf.

1. Policy:

The TAMU NMR Newsletter is a means for the rapid exchange of information among active workers in the field of NMR spectroscopy, as defined broadly, including imaging. As such, the Newsletter will serve its purpose best if the participants impart whatever they feel will be of interest to their colleagues, and inquire about whatever matters concern them.

Since the subscriber/participant clearly is the best judge of what he or she considers interesting, our first statement of policy is "We print anything." (This usually is followed by the mental reservation, "that won't land us in jail.") Virtually no editorial functions are performed, although on rare occasions there is the need to classify a contribution as 'not for credit'. I trust that the reasons for this policy are obvious.

The TAMU NMR Newsletter is not, and will not become, a journal. We merely reproduce and disseminate exactly what is sent in. Foreign participants should not feel obliged to render their contributions in English.

2. Public Quotation and Referencing:

Public quotation of Newsletter contents in print or in a formal talk at a meeting, etc., is expressly forbidden (except as follows), and reference to the TAMU NMR Newsletter by name in the scientific literature is never permissible. In order to quote results or use material from the Newsletter, it is necessary, in each individual case, to obtain the prior permission of the author in question and then to refer to the material quoted as a "Private Communication". If your copy of the Newsletter is shared with other readers, it is your obligation as the actual recipient of the Newsletter to see that these other readers of your copy are acquainted with, and abide by, these statements of policy.

3. Participation is the prime requisite for receiving the TAMU NMR Newsletter:

In order to receive the Newsletter, you must make at least occasional technical contributions to its contents.

We feel that we have to be quite rigorous in this regard, and the following schedule is in effect: Eight months after your last technical contribution you will receive a "Reminder" notice. If no technical contribution is then forthcoming, ten months after your previous contribution you will receive an "Ultimatum" notice, and then the next issue will be your last, absent a technical contribution. Subscription fees are not refunded in such cases. If you are dropped from the mailing list, you can be reinstated by submitting a contribution, and you will receive back issues (as available) and forthcoming issues at the rate of nine per contribution.

Frequent contributions are encouraged, but no "advance credit" can be obtained for them. In cases of joint authorship, either contributor, but not both, may be credited. Please indicate to whose account credit should be given. Please note that meeting announcements, as well as "Position Available," "Equipment Wanted" (or "For Sale"), etc., notices are very welcome, but only on a not-for-credit basis, i.e., such items do not substitute for a *bona fide* technical contribution. Similar considerations must occasionally be applied to a few (quasi-)technical items.

4. Finances:

The Newsletter is wholly self-supporting, and depends for its funds on advertising, donations, and individual subscriptions.

The Subscription fee for the October 1990 - September 1991 year has been set at US\$150.00, with a 50% academic or personal subscription discount. Subscriptions are available only for the twelve monthly issues which begin with the October issue and run through that of the following September. However, a subscription can be initiated at any time, and the issues back to the previous October will be provided as long as copies remain available.

Companies and other organizations are also invited to consider joining the list of Sponsors of the Newsletter. Sponsors' names appear in each month's Newsletter, and copies of the Newsletter are provided to all Sponsors. The continuation of

Continued

this non-commercial Newsletter depends significantly on the interest and generosity of our Sponsors, most of whom have been loyal supporters of this publication for many years. We will be happy to provide further details to anyone interested.

Another major, indeed most essential, source of funds for the Newsletter is Advertising. We earnestly encourage present and potential participants of the Newsletter to seek advertising from their companies. Our rates are very modest - please inquire for details.

5. Practical Considerations:

- a) All technical contributions to the TAMU NMR Newsletter will always be included in the next issue if received before the published deadline dates.
- b) Please provide short titles of all topics of your contributions, so as to ensure accuracy in the table of contents.
- c) Contributions should be on the *minimum* (NOTE!!) number of 8.5 x 11" (21 x 27.5 cm) pages, printed on one side only. Contributions may not exceed three pages without prior approval. Each page must have margins of at least 0.5 - 0.75" (1.3 - 2.0 cm) on all sides. Please observe these limits. Black ink for typing, drawings, etc., is essential. All drawings, figures, etc., should be mounted in place on the 8.5 x 11" pages. We are not equipped to handle pieces of paper larger than 8.5 x 11" (21 x 27.5 cm).

Foreign subscribers are reminded that regardless of the standard paper length you use, all material - letterhead, text, figures, addresses printed at the page bottom, everything - must not exceed 10" (ca. 25.3 cm) from top to bottom.

Significant savings of Newsletter pages and total space can be made by exercising close control over the formatting and type sizes of the contributions. Please consider the following:

i) For those with computers, try using a smaller type font. The body of this page is printed in 10 point type, which I believe is adequate for most purposes. Even 12 point is acceptable, I suppose. Those who are computerized can also employ non-integral spacing of lines so that sub- and superscripts don't collide with lines below and above.

ii) PLEASE avoid excessive margins. *Instruct your secretaries to avoid normal correspondence esthetics or practices, however time-honored or 'standard'!* This page has margins on both sides of 0.6" (ca. 1.55 cm), which is very adequate. Margins of the same size at the top and bottom are sufficient also, but don't worry if there is more space at the end of your document, for I can often use such spaces for notices, etc.

Also, please avoid large amounts of unused space at the top of letters. Give thought to the sizes of figures, drawings, etc., and please mount these so as to use the minimum space on the page.

iii) 'Position Available', 'Equipment Wanted', and Similar Notices. These are always welcome, without charge, but not for subscription credit, of course. Such notices will appear, however, *only* if received with these necessarily rigid constraints: a) Single spaced; b) both side margins 0.6 - 0.7" (1.5 - 1.7 cm.) - NOT WIDER; c) the minimum total height, please, but definitely no more than 4.5" (11.5 cm.) This will let me place such notices wherever a bit of space occurs.

iv) AVOID DOUBLE SPACING LIKE THE BLACK PLAGUE ! ! ! This is extremely wasteful of space. Even sans computer, small type and 1.5-line (if needed) spacing can be had with a little effort.

6. Suggestions: They are always welcome.


B. L. Shapiro

Address for all correspondence:

Dr. Bernard L. Shapiro
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto, California 94303
U.S.A.

Telephone: (415) 493-5971. Please confine telephone calls to the hours from 8:00AM - 10:00PM, *Pacific Coast Time*.

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Prof. Bernard L. Shapiro
 966 Elsinore Court
 Palo Alto, CA 94303
 U S A

(received 5/18/90)

HOW TO CONNECT AN OLD 1.41 T IRON MAGNET TO A MODERN SPECTROMETER

Dear Prof. Shapiro,

One of our major fields of interest is solvent exchange studies on paramagnetic systems. We were very happy to have an 18 year old Bruker WP-60 multinuclear FT spectrometer equipped with a 1.41 T iron magnet. Together with our modern spectrometers (having fields up to 9.4 T) we were able to perform frequency dependence studies by an up to six-fold change of field. The magnet of the WP-60 spectrometer still works well but the computer, which was still the old BNC-12, having 8k data memory and 20 bit word length, was completely obsolete and caused a lot of problems. Measurement of weak signals (^{17}O for example) was heavily restricted by memory overload and T_1 experiments were very difficult to perform.

To overcome these problems we arranged to use the 1.41 T (60 MHz ^1H) magnet with a new multinuclear Bruker AC-200. To be able to use our old probes with ^2D or ^{19}F lock we kept the old lock unit including the scope and the shim facilities from the WP-60 console and put it into a separate rack (Figure 1).

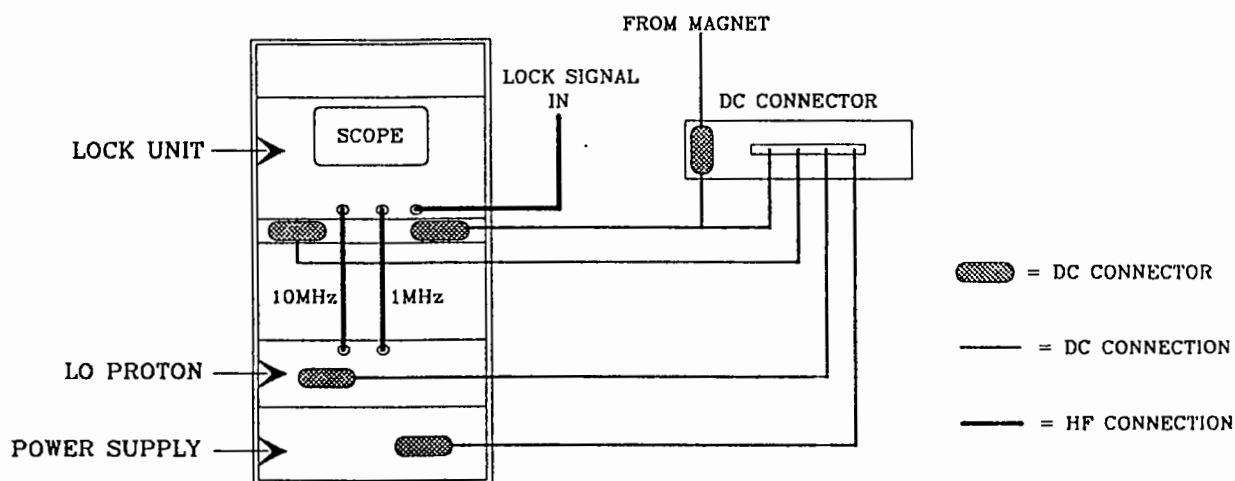


Figure 1: A schematic representation of the arrangement of units in the rack (viewed from the rear) and the electrical connections between them. The plate shown on the right is attached to the rear of the rack and has been displaced in the diagram for the sake of clarity.

To run this unit we also kept the LO proton and the power supply from the old apparatus. This is enough to lock the field with either a ^2D or a ^{19}F lock and to shim it. We have thrown away the rest of the console. If we set-up the AC-200 console to 60.013 MHz and connect the BB preamplifier to the ^1H probe of the old iron magnet (which is placed just to the left of the console), we can record spectra without any difficulty. In the same way we can set the AC-200 to any resonance frequency above 8 MHz and connect it to the BB probe of the iron magnet. T_1 measurements are now as easy as with a modern cryomagnet system despite of the fact that the sensitivity loss due to the lower field is enormous. Another positive effect is the higher sensitivity of modern preamplifiers and receivers. Figure 2 shows the increase in S/N of a factor of 4 by changing from the old to the new electronics in ^{17}O NMR.

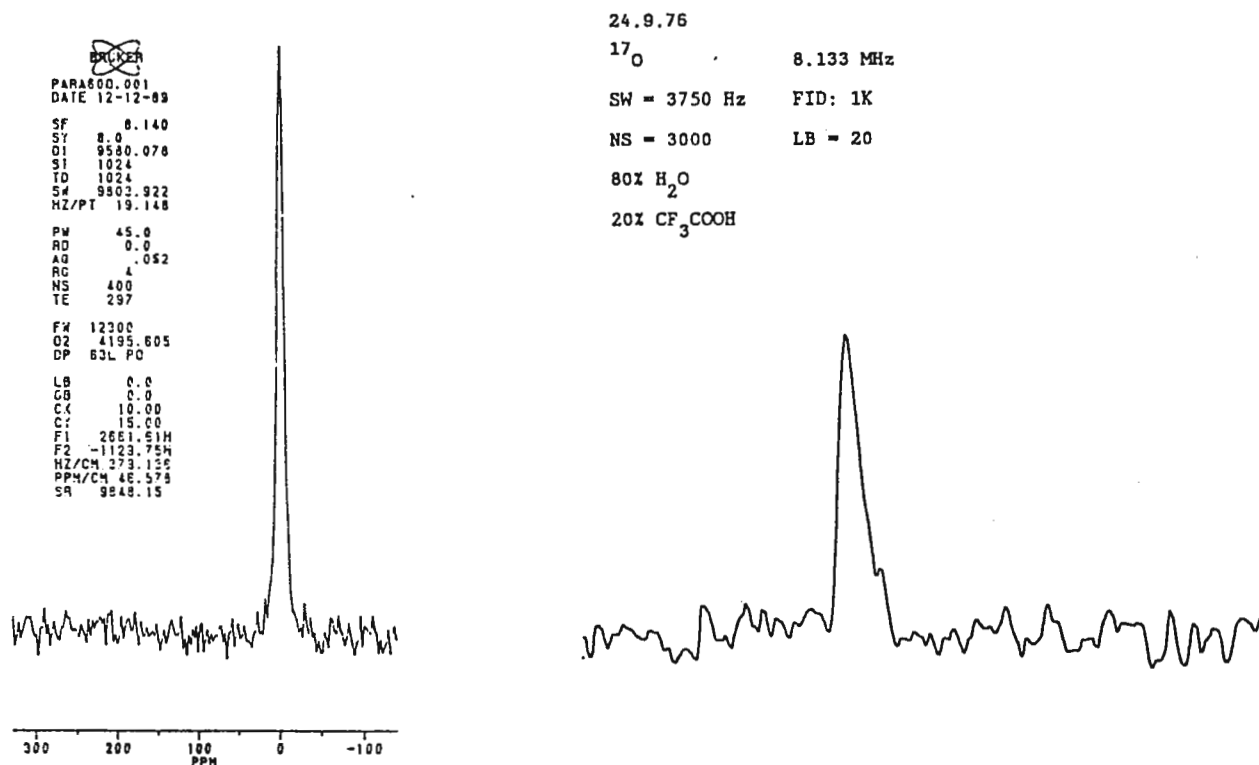


Figure 2: ^{17}O spectrum of tap water recorded using the AC-200 console (left) and the spectrum of an 80% water sample taken with the old WP-60 console (right).

D. H. Powell

Dr. D. Hugh Powell

L. Helm

Dr. Lothar Helm

Prof. A.E. Merbach



MR Resources, Inc.

158R Main Street, Gardner, MA 01440

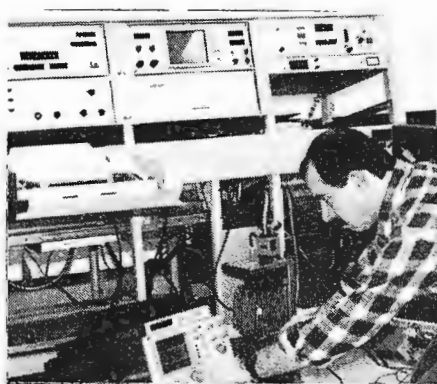
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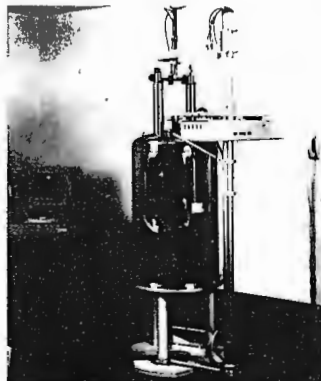
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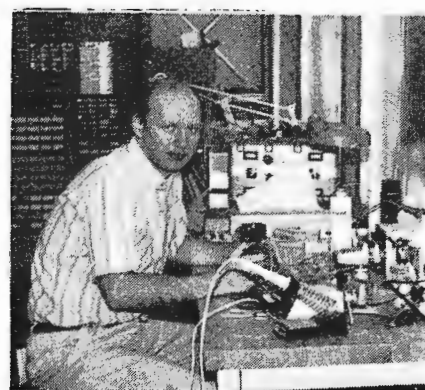
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May 4, 1990
(received 5/11/90)

Dr. Bernard L. Shapiro
Editor/Publisher
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto, California 94303

^{29}Si CP-MAS Contact Times

Dear Professor Shapiro:

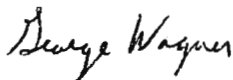
Optimum ^{13}C CP-MAS contact times (to yield maximum S/N) normally fall in the range of 1-10 msec. However, optimum ^{29}Si contact times can be considerably longer. To illustrate this, we show below plots of ^{29}Si intensity vs. contact time for two common ^{29}Si solid state NMR standards, the silicate Q_8M_8 and tetrakis-(trimethylsilyl)silane (TTMS) (Figure 1), and a completely siliceous synthetic zeolite (1) (Figure 2). All data were obtained at 7.0 T corresponding to a ^{29}Si frequency of 59.6 MHz.

As the plots demonstrate, maximum signal intensity is achieved using contact times of 30 msec for Q_8M_8 and 60 msec for TTMS. For the zeolite, maximum signals are obtained for the five resolved resonances using a contact time of ca. 90 msec. Note that when using such long contact times, one must be careful that the decoupler does not remain on too long. For our Chemagnetics probe, the maximum decoupler time is 200 msec which limits the acquisition time to about 110 msec for the zeolite samples.

The use of appropriately long ^{29}Si contact times is especially important when it comes to doing ^{29}Si 2D NMR of zeolites (2,3), where it is desirable to achieve maximum S/N in the shortest time. This has been a big help in our efforts to obtain ^{29}Si COSY spectra of the synthetic zeolite (^{29}Si present in natural abundance) without resorting to isotopic enrichment.

Please credit this contribution to the account of Dr. Vera V. Mainz.

Sincerely,



George W. Wagner



Hee K. Chae

- (1) H. K. Chae, G. W. Wagner, W. G. Klemperer, unpublished results.
- (2) C. A. Fyfe, H. Gies, Y. Feng, J. Chem. Soc., Chem Commun., (1989) 1240.
- (3) C. A. Fyfe, H. Gies, Y. Feng, J. Am. Chem Soc., **111** (1989) 7702.

Figure 1.

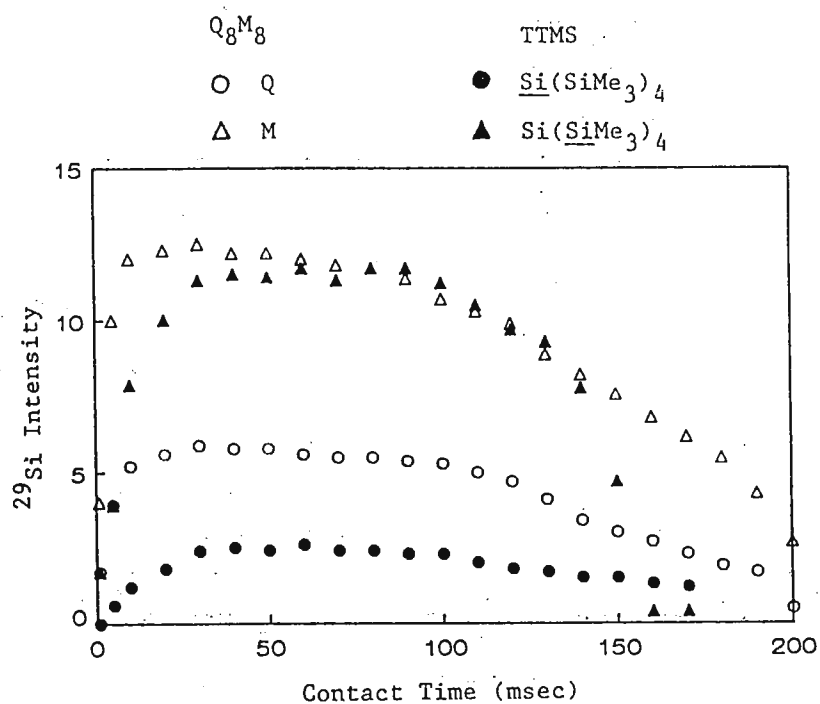
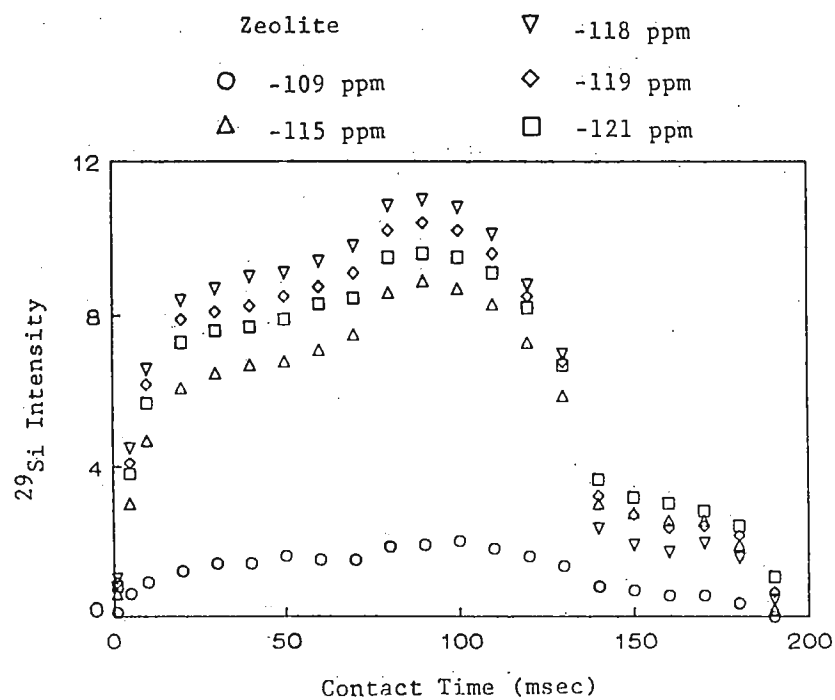


Figure 2.





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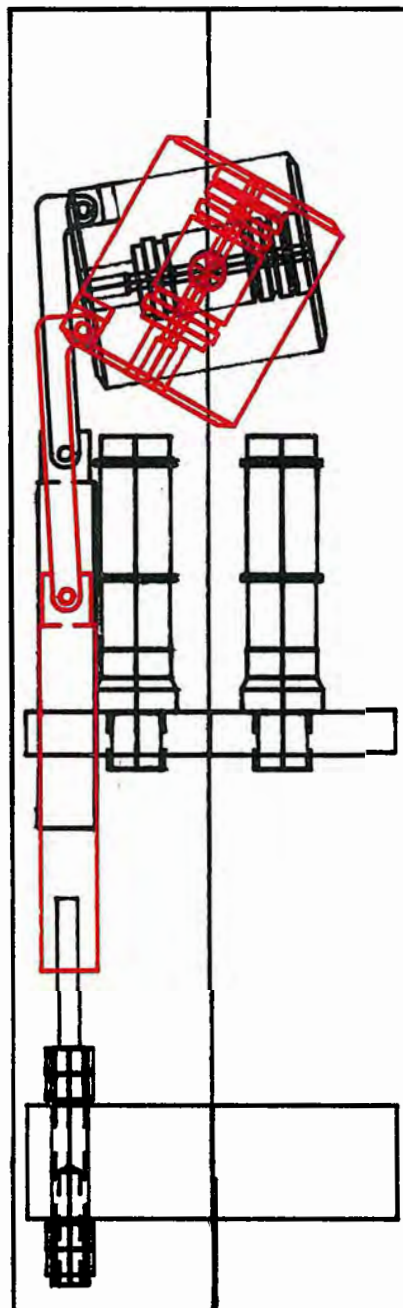
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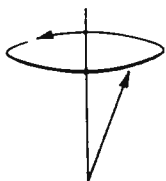
DAS Operation:

The menu-driven software is designed for clarity and ease of use. Angle change is initiated by a 50 μ s TTL pulse from the spectrometer. Less than 15 ms later the spin axis has stepped to the next angle in the list. The minimum time between initiating pulses is 40 ms. All angles are referenced to Magic Angle, which can be set precisely by standard NMR techniques. This angle calibration normally is required only once at initial installation.

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STANFORD UNIVERSITY
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SMRL OFFICE:
415/723-6270

May 2, 1990
(received 5/2/90)

Dr. Barry Shapiro
TAMU NMR Newsletter

Dear Barry,

Re: ^1H Detected ^{15}N - ^1H Correlations on a Bruker AM-500 Using an External X-Nucleus Decoupler Instead of Reverse Electronics

We are studying the structure and function of the *trp* repressor and, in order to assign the proton spectrum of this 25 KD protein, extensive isotopic labeling has been necessary. However, our older model Bruker AM-500 is not equipped with the reverse electronics that are normally used to implement the ^1H detected X-nucleus correlated or edited experiments. In order to perform these very useful experiments, we have used an FMR X-Nucleus Decoupler and a FMR 5mm ^1H (^{15}N - ^{13}C) RPT Probe. For the HMQC experiment the Bruker ^1H observe channel was used for proton pulses, the X-nucleus decoupler was used for X hard pulses as well as preprogrammed composite X-decoupling, and the Bruker proton decoupler was used for presaturation of the solvent. In order to be able to control the phase of the X pulses using Bruker phase programs in automation, the output of the Bruker PTS synthesizer was fed into a powered buffered splitter from which there are 3 outputs: one used for the normal proton transmitter, one used to generate a proton decoupler coherent with the transmitter, and one used as the input to the X-nucleus decoupler. This way the phase of the X pulse can be set during a 2 μs delay with the command **Dn:T PHn**. Figures 1 and 2 (on next page) show an ^1H - ^{15}N HMQC and ^1H - ^{15}N HMQC with NOE relay of ^{15}N -Leu labeled *trp* repressor, respectively. The samples were 2.4 mM in protein, 500 mM KCl, 50 mM Phosphate Buffer pH = 5.7, 90% H_2O , 10% D_2O and the spectra were run at 45°C. The ^1H - ^{15}N correlation peaks of all 19 leucines are labelled in Figure 1, and the NOE crosspeaks between leucine NHs and neighboring NOEs are shown in Figure 2.

Sincerely,

Cheryl H. Arrowsmith

(Credit to the account of Oleg Jardetzky)

LABORATORIES:
Bldg. 7-250, Rm. 101
415/723-4063
Bldg. 7-309, Rm. R322
415/725-1811

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Figure 1

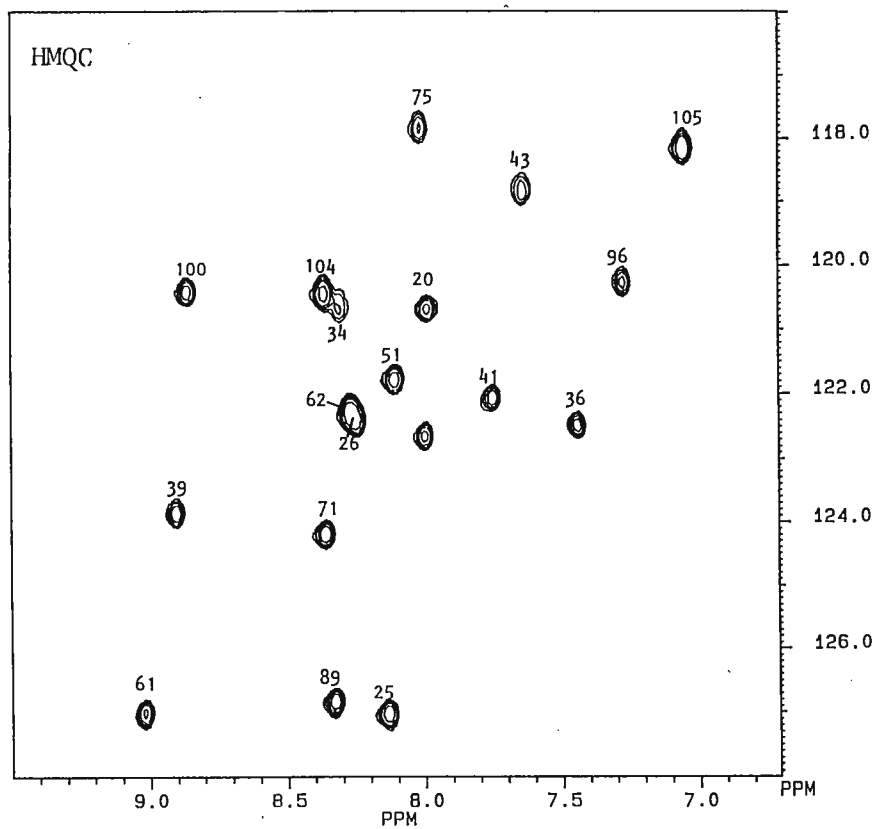
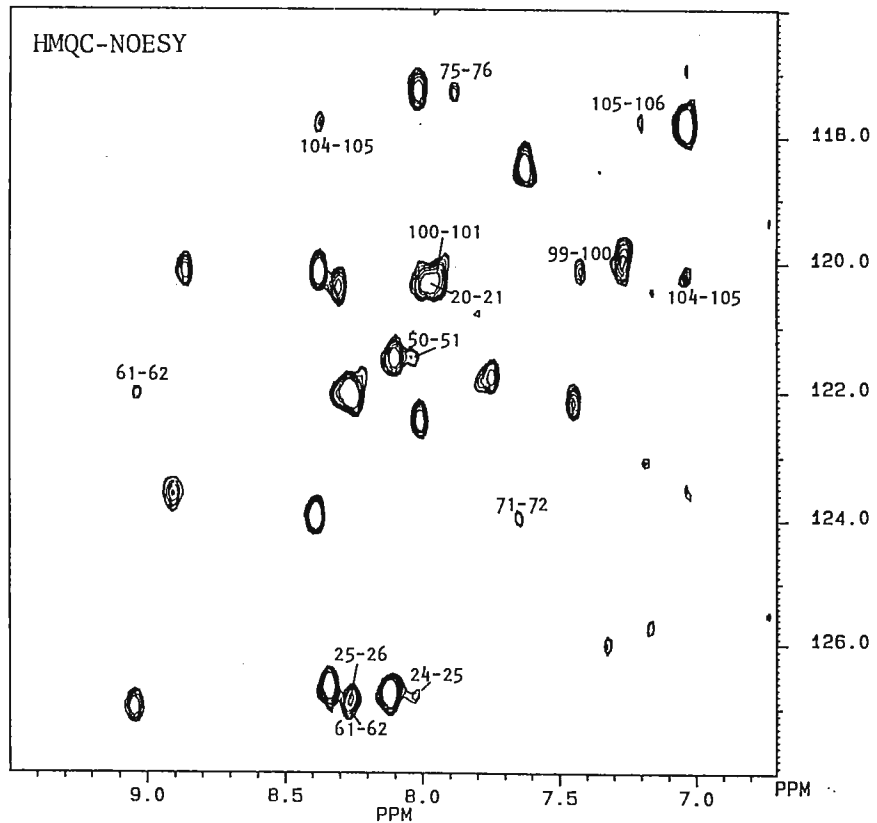
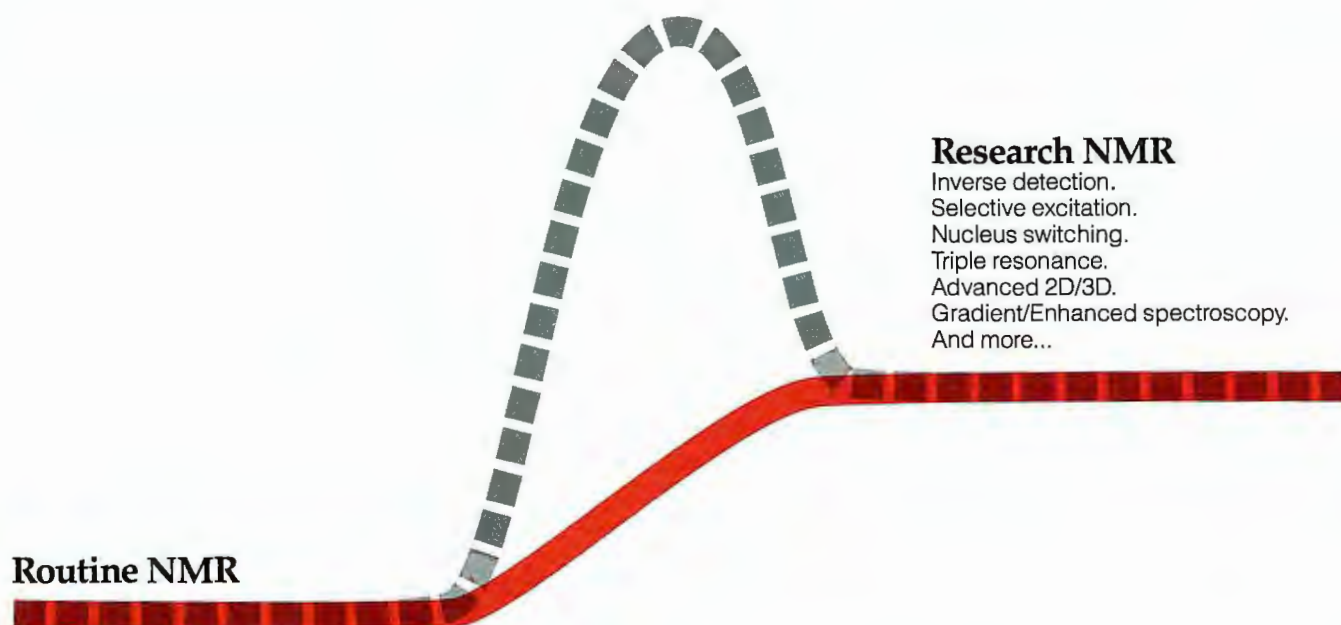


Figure 2



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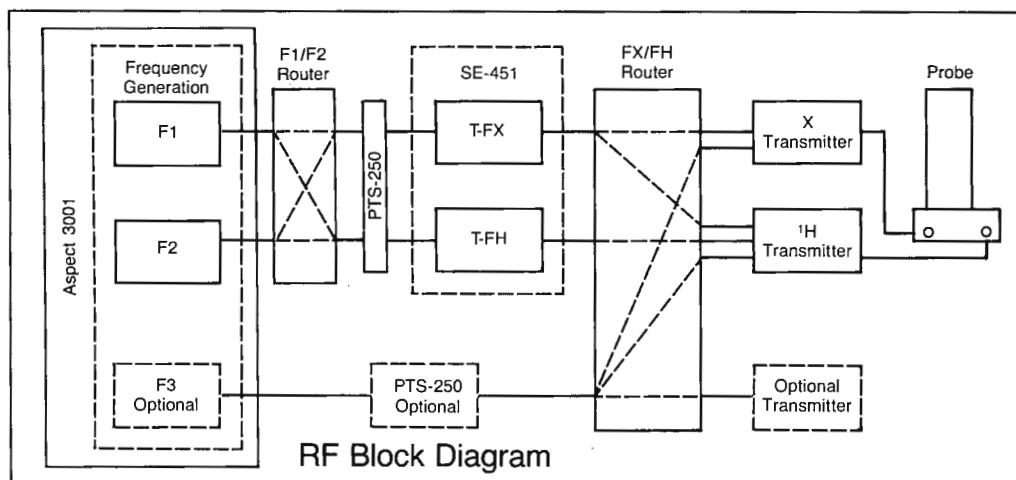
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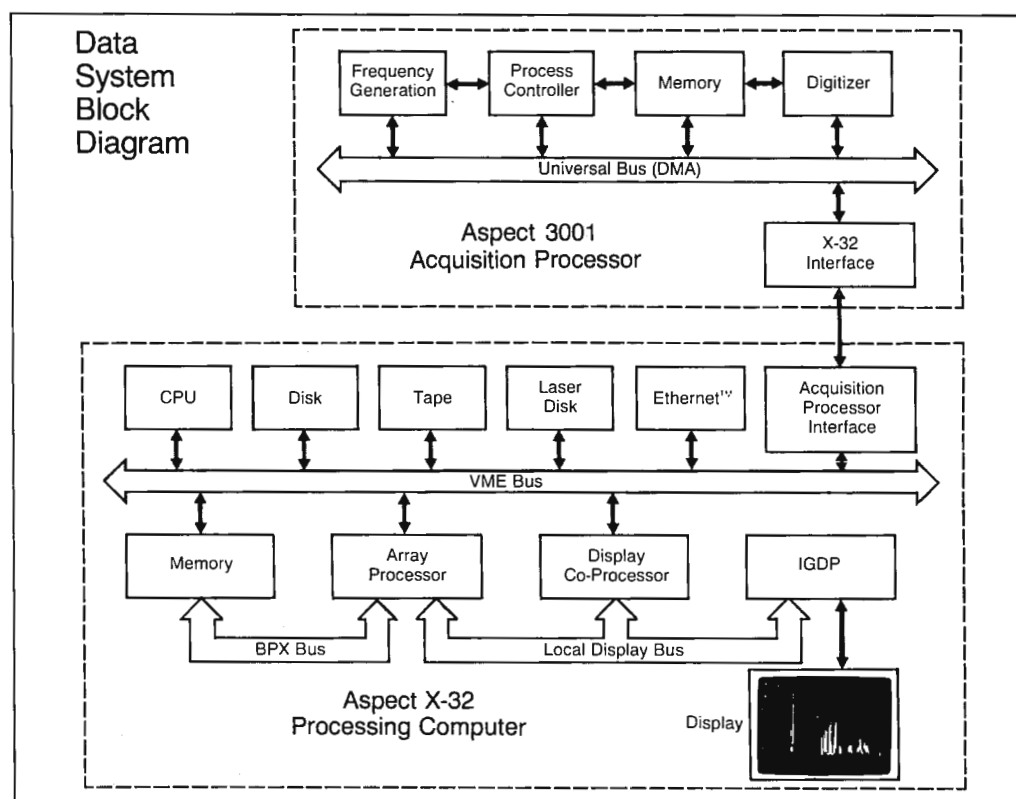
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NMR Technologies Inc.

Dr. David E. Axelson, Ph. D.
President

Head Office & NMR Spectroscopy Division
Advanced Technology Centre,
9650 - 20 Avenue, Unit 132
Edmonton, Alberta, Canada T6N 1G1
Tel: (403) 462-2798 Fax: (403) 428-5376

Ravi Philari, P. Eng., MBA
Vice-President

Consulting Division
301-120 Holland Ave.,
Ottawa, Ontario
Canada K1Y 0X6
Tel: (613) 729-6030 Fax: (613) 729-3752

Dear Barry,

April 17, 1990
(received 4/30/90)

Polyethylene-Starch Composites

In many instances the characterization of filled polymers / composites can be conveniently pursued through the use of solution NMR, with the fillers themselves being fortuitously invisible and thus improving the resolution of the spectra obtained. In other cases the additives themselves can be accurately quantified at very low concentrations.

We therefore recently investigated the possibility of quantifying the amount of starch in polyethylene - starch blends through the use of C-13 solution NMR measurements (Bruker AC-F spectrometer, C-13 frequency 50.306 MHz, 16K data points, 5000 - 6000 transients, 120°C, 6s recycle delay) for comparison with a reasonably accurate FT-IR method. It was soon apparent that the starch component was not amenable to study by this method due to its particulate nature under these experimental conditions (Figure 1).

Solid state magic angle spinning spectra on the as - received film samples themselves were then attempted (Figure 2) (500 transients, 6s recycle delay, 1 ms contact time, 4.9 us 90° pulse width, 3 kHz MAS, 2K data points). Under these conditions the starch resonances were readily observed.

We are now systematically exploring the exact conditions for quantitative analysis over a very wide range of compositions (about 1 - 40 wt% starch).

Sincerely,


David Axelson
NMR Technologies Inc.



Aruna Sharma

Tony Tikuisis
Novacor Chemicals Ltd
Technical Centre
Calgary, Alberta

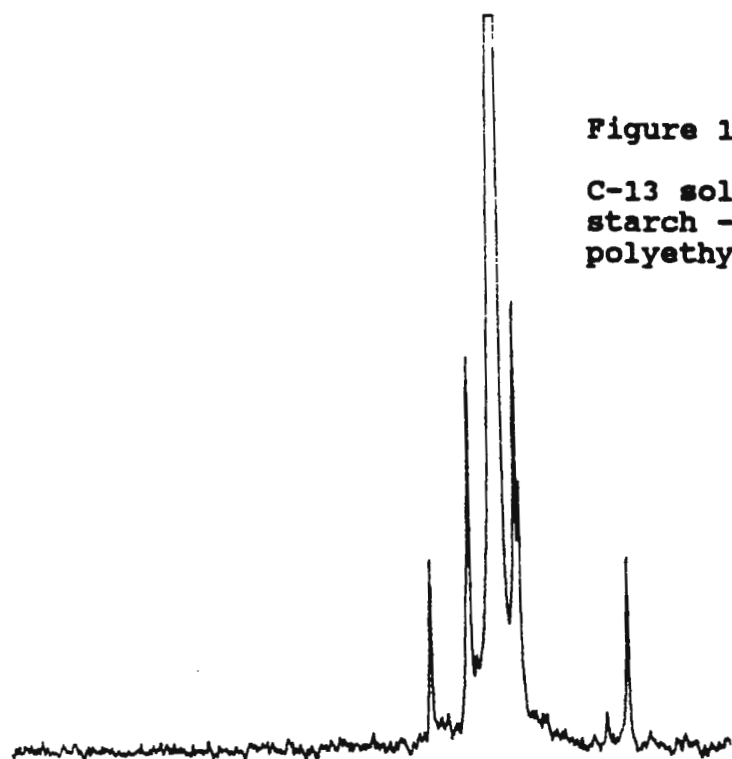


Figure 1

**C-13 solution NMR spectrum of
starch - filled low density
polyethylene.**

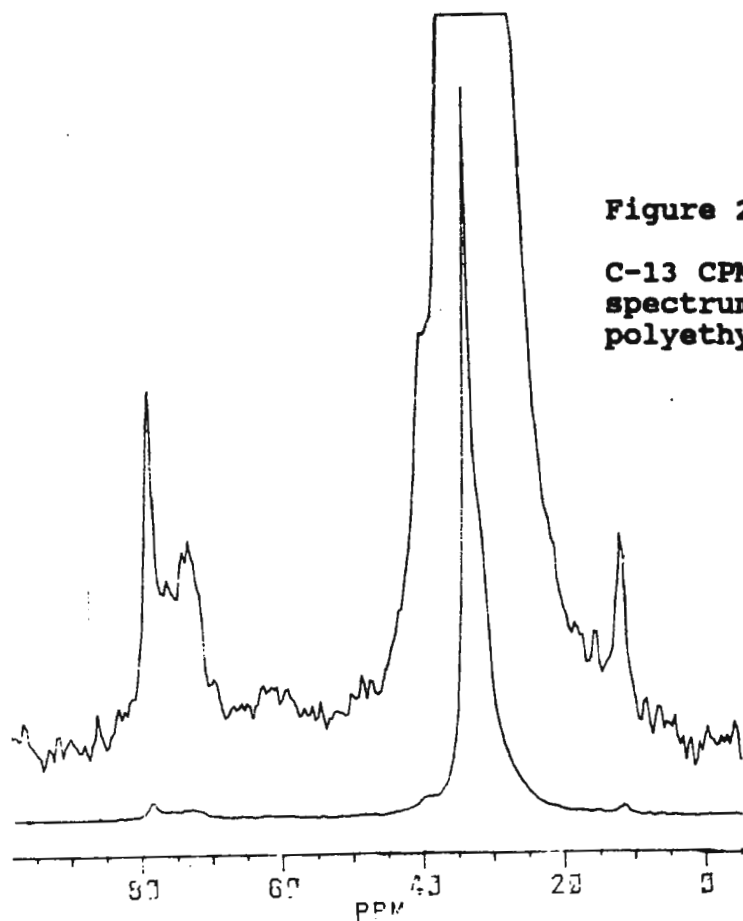


Figure 2

**C-13 CPMAS solid state NMR
spectrum of starch - filled
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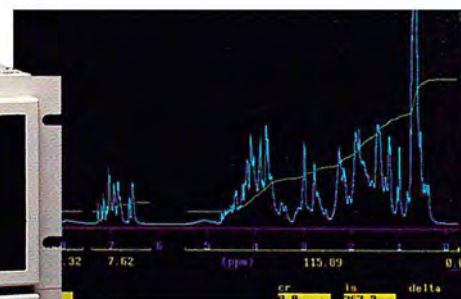
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3 May 1990 (received 5/9/90)

Dr. B. Shapiro,
966 Elsinore Court,
Palo Alto, CA 94303

Dear Dr. Shapiro,

We have been encouraging our biologically oriented colleagues to use NMR as a research tool. Since our AM400 has a standard bore magnet, there are limitations to what we can usefully do (and keep viable). Two systems have worked well, principally because they do not require continuous oxygenation.

The first of these arose from a collaboration with the Botany department. We were looking at extracts of New Zealand lichens to identify the major fungal metabolites. We tried intact lichens but, not surprisingly, the signals were poor. It was pointed out to the botanists that NMR samples need to be as macroscopically and microscopically homogeneous as possible. They had interests in other symbiotic organisms and one of these, a species of sea anemone with algal symbionts, has given excellent spectra.

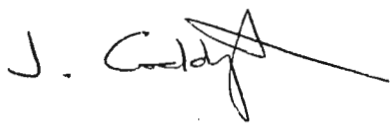
The *Anthopleura* anemones are quite small and we pack about 50 into a 10mm NMR tube. We can monitor their metabolic state by ^{31}P . They are unperturbed by small additions of D_2O to their sea water and show no signs of stress after 3 hours in the magnet. Many then consider the glass of the tube to be a substitute cockle shell, which is their normal anchorage. It is sometimes difficult to get them out, but when placed back in natural seawater they extend their tentacles and resume feeding behaviour. The ^{13}C spectra we obtain are well resolved and clearly show the large amounts of lipid present. There is a question as to whether the algae supply lipid or lipid precursors to the animal. We are conducting feeding experiments with $^{13}\text{CO}_2$ and monitoring uptake into the compounds visible by NMR. Preliminary results indicate that it is the animals that make their own lipid from smaller supplied metabolites.

The second system is involved in a study of the buffers used to bathe hearts for transplantation surgery. These organs are kept cold but usually not oxygenated. There is evidence that the addition of aspartate to the buffer solution improves post-surgical oxidative metabolism and function. The mechanism by which this protection of the myocardium occurs is not clear.

In collaboration with the Pathology department, we began a study of the intracellular pH and high energy phosphate levels in hearts stored for up to 24 hours. We were restricted by the diameter of the NMR tubes as to which animal models we could use. Mouse hearts are small enough but are very difficult to routinely prepare, particularly the insertion of a cannula into the aorta for perfusion by the cardioplegic solution. Rat hearts are marginally too big unless adolescent animals are used. These latter fit snugly into the tubes without damage and we have used these teenaged organs. The hearts are suspended from the aortic cannula by cotton string and are passively bathed by the selected buffer. We collect ^{31}P spectra at 4°C , taking care to let the signals relax, and get adequate S/N in about 6 minutes. We see the expected changes in levels of P_i , PCr and ATP as we monitor over 24 hours. Our results show that added aspartate does not act by maintaining the intracellular pH at normal or near normal values during storage. This behaviour is characteristic of all the rat hearts we used but initial work with mouse and other models is not so clear-cut.

Please credit this contribution to FRI, Rotorua.

Yours sincerely,



Jan Coddington

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THE UNIVERSITY OF MELBOURNE
Department of Organic Chemistry

Professor: D. W. Cameron
Telephone: (03) 344 6492
Professor B.L. Shapiro
966 Elsinore Court
PALO ALTO CA 94303
U.S.A.

Head: D. P. Kelly
Telephone: (03) 344 6464
20 April 1990
(received 4/26/90)

CSA of Carbocations at ~200K

Dear Barry,

Some time ago we reported on relaxation studies of the *t*-butyl cation generated in superacid, which showed a massive contribution by Chemical Shift Anisotropy to the ^{13}C relaxation rate of the cationic carbon at 213K and 5.87 tesla.¹

We have recently completed manuscripts (based on Ph.D. thesis of Dr Ralph Leslie) on the relaxation mechanisms of a range of carbocations² and on the interaction between cations and the medium.^{3,4}

Unlike the *t*-butyl cation 1b where the shielding anisotropy had to be obtained from the powder spectrum,^{1,5} sufficient data were available for the cationic carbons of the other cations 2-5 for us to calculate the shielding anisotropies ($\Delta\sigma$) from the rotational diffusion constants D_{\parallel} and D_{\perp} . These values are considerably larger than those observed for sp^2 carbons of neutral species and in some cases larger than those observed for linear sp carbons.

The ratio $T_1^{\text{OBS}}/T_1^{\text{CSA}}$ gives the fractional contribution to the relaxation of the cationic carbons, which is shown to be significant (11-25%) even at low field strengths, and major (42-71%) at higher field strengths. The remainder is considered to be due to dipole-dipole mechanisms (inter and intramolecular) but not to the spin-rotation mechanism.

I hope that this is an appropriate response to your HELP! notice!

Yours sincerely,

David

D.P. Kelly

Cation	B° (T)	$T_1^{\text{OBS}}/T_1^{\text{CSA}}$	$\Delta\sigma$ (ppm)	
1b	5.9 1.4	0.80 0.19	450	
2	5.9 2.3	0.40 0.11	380	
3	4.7 2.3	0.50 0.20	500	
4a	4.7 2.3	0.42 0.15	430	
4b	4.7 2.3	0.55 0.24	440	
5a	5.9 2.3	0.71 0.16	540	
5b	4.7 2.3	0.57 0.25	440	

1. Kelly D.P., Leslie D.R., Craig R.A. *J. Magn. Reson.* **1983**, 52, 480-491.
2. Kelly D.P., Leslie D.R. *Magn. Reson. in Chem.* in press.
3. Kelly D.P., Leslie D.R. *J. Am. Chem. Soc.* in press.
4. Kelly D.P., Leslie D.R. *J. Am. Chem. Soc.* submitted.
5. Yannoni C.S. *Acc. Chem. Res.* **1982**, 15, 201.

NMR Museum Collectors!?

In the process of moving our electronics shop we unearthed an NMR FIELD CONTROL, Model FC-501, manufactured by Harvey-Wells Electronics, Southbridge, MA. The Tulane property tag indicates that it was purchased in 1957. It has a genuine cathode ray tube, an ammeter, and about a dozen switches and dials. It is designed for rack mounting and it's about 9" high. It looks like it still has all of its tubes and other components. Call soon, before it heads to the city dump.

Andy Waterhouse, Dept. of Chemistry, Tulane University. 504-865-5573

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An immediate opening for an individual to conduct Bio-NMR studies is available in the pharmaceuticals division of CIBA-GEIGY Corp. Candidates should possess a Ph.D. in Biochemistry, Animal Pharmacology or a closely related discipline and should be fully conversant with the application of NMR theory and practice to the study of in-vitro and in-vivo biological systems. Experience in the isolation, preparation and evaluation of rodent hearts is essential. The individual is also responsible for surgical or non-invasive procedures leading to in-vivo NMR examination of rodents with surface coil probes. Familiarity with NMR1 (New Methods Research Software) is also desirable. Candidates should possess a high degree of innovative and practical skills and be willing to work closely with biologists, toxicologists and electrical engineers in a multidisciplinary environment.

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Probes: 5 mm ^1H , 5 mm ^1H (custom), 5 mm ^2H (custom), 12 mm ^{13}C , 12 mm ^{15}N (custom), 12 mm ^{19}F , 12 mm BB ^2H - ^{31}P , 20 mm BB ^{14}N - ^2H , 20 mm sideways spinning ^{13}C and ^{31}P .

The data station consists of 1280 processor with 128 k ram, teletype model 43, CDC Hawk dual disc drive and color raster display.

Miscellaneous: Hardware, complete schematic and manuals. All or any of the above is available at a fraction of the original price. For more information please contact: **GERRY MCQUAID, DEPARTMENT OF BIOCHEMISTRY, UNIVERSITY OF ALBERTA, PHONE: (403) 492-3006, FAX: (403) 492-0886.**

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82-00732	Acetone-d ₆	99.9	10 x 1 g 50 x 1 g 100 x 1 g 1 x 10 g 5 x 10 g 10 x 10 g	amp amp amp amp amp amp	16.00 75.00 145.00 16.00 75.00 145.00
82-00731	Acetone-d ₆ + 0.05% TMS (v/v)	99.9	1 x 25 g 5 x 25 g 10 x 25 g	scb scb scb	37.00 180.00 345.00
82-00733	Acetone-d ₆ "100%"	99.96	10 x 0.75mL	amp	55.00
82-05012	Acetonitrile-d ₃	99.8	10 x 1 g 50 x 1 g 1 x 10 g 5 x 10 g	amp amp amp amp	25.00 115.00 25.00 115.00
82-05014	Acetonitrile-d ₃ + 0.05% TMS (v/v)	99.8	1 x 10 g 5 x 10 g 1 x 25 g	scb scb amp	25.00 115.00 60.00
82-84077	Benzene-d ₆	99.6	10 x 1 g 50 x 1 g 100 x 1 g 1 x 10 g 5 x 10 g 10 x 10 g	amp amp amp amp amp amp	20.00 80.00 150.00 20.00 80.00 150.00

CAT. NO.	PRODUCT	ENRICHMENT (Min. Atom % D)	QUANTITY	PACKAGE	PRICE [†]
82-84036	Ethylbenzene-d ₁₀	98	1 x 1 g 1 x 10 g	amp amp	20.00 115.00
82-00061	Methanol-d ₄ (~0.7 atom % ¹³ C)	99.8	10 x 1 g 50 x 1 g 100 x 1 g 1 x 10 g 5 x 10 g 10 x 10 g	amp amp amp amp amp amp	45.00 180.00 345.00 45.00 180.00 345.00
82-00059	Methanol-d ₄ + 0.05% TMS (v/v) (~0.7 atom % ¹³ C)	99.8	1 x 10 g 5 x 10 g 10 x 10 g 1 x 25 g	scb scb scb scb	45.00 180.00 345.00 110.00
82-04099	Methylcyclohexane-d ₁₄	99.6	1 x 1 g 1 x 5 g	amp amp	35.00 120.00
82-86007	Nitrobenzene-d ₅	99.5	1 x 10 g 5 x 10 g	amp amp	35.00 150.00
82-70030	Nitric Acid-d (65% in D ₂ O)	99	1 x 10 g 1 x 25 g	scb scb	65.00* 135.00
82-70024	Potassium Deuterioxide (40% in D ₂ O)	99	1 x 25 g 1 x 50 g	pl scb pl scb	55.00* 95.00*
82-00064	iso-Propanol-d ₈	99	1 x 5 g 1 x 10 g	amp amp	44.00 75.00
82-70028	Sodium Deuterioxide (40% in D ₂ O)	99	1 x 50 g 1 x 100 g	pl scb pl scb	40.00* 75.00*
82-70800	Sulfuric Acid-d ₂ (96% in D ₂ O)	99	1 x 50 g 1 x 100 g	pl scb pl scb	45.00* 75.00*
80-07001	Tetramethylsilane (NMR Grade)		1 x 50g	septum vial	17.00
82-84081	Toluene-d ₈	99.6	10 x 1 g 50 x 1 g 1 x 10 g 5 x 10 g	amp amp amp amp	45.00 180.00 45.00 180.00
82-02510	Trifluoroacetic Acid-d	99.5	1 x 10 g 5 x 10 g 10 x 10 g 1 x 25 g	amp amp amp amp	19.00 80.00 115.00 40.00
82-84073	o-Xylene-d ₁₀	99	1 x 5 g	amp	44.00
82-84075	p-Xylene-d ₁₀	99	1 x 5 g	amp	44.00

* Price / grams solution

† Prices are FOB Miamisburg, OH for delivery in North America; request prices for delivery to the other continents.

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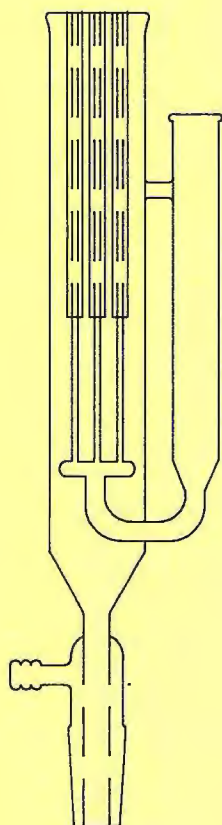
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Please allow us to bid on your annual NMR solvent requirements.**

CAT. NO.	PRODUCT	ENRICHMENT (Min. Atom % D)	QUANTITY	PACKAGE	PRICE [†]
82-04097	Benzene-d ₆ + 0.05% TMS (v/v)	99.6	1 x 25 g	scb	45.00
			5 x 25 g	scb	185.00
			1 x 100 g	scb	145.00
82-84078	Benzene-d ₆ "100%"	99.96	10 x 0.75 mL	amp	65.00
82-80554	Bromobenzene-d ₅	99.5	1 x 10 g	amp	50.00
			5 x 10 g	amp	210.00
82-80555	Bromoform-d	99.5	1 x 10 g	amp	30.00
			5 x 10 g	amp	125.00
82-80556	Chloroform-d	99.8	50 x 1 ml	amp	50.00
			1 x 100 g	scb	20.00
			5 x 100 g	scb	85.00
			10 x 100 g	scb	165.00
			1 kg	scb	155.00
82-00540	Chloroform-d + 0.05% TMS (v/v)	99.8	1 x 100 g	scb	20.00
			5 x 100 g	scb	85.00
			10 x 100 g	scb	165.00
82-80558	Chloroform-d "100%"	99.96	10 x 0.75 ml	amp	26.00
			1 x 10 ml	amp	22.00
82-04098	Cyclohexane-d ₁₂	99.6	1 x 1 g	amp	17.00
			5 x 1 g	amp	72.00
			1 x 10 g	amp	110.00
82-70005	Deuterium Bromide (48% in D ₂ O)	99	1 x 50 g	scb	90.00*
82-70007	Deuterium Chloride (35% in D ₂ O)	99.5	1 x 50 g	scb	50.00*
			1 x 100 g	scb	90.00*
82-70001	Deuterium Oxide	99.9	1 x 100 g	scb	45.00
			5 x 100 g	scb	210.00
			10 x 100 g	scb	400.00
			1 kg	pl scb	385.00
82-70901	Deuterium Oxide	99.8	min. 1 x 10 kg	plastic cont.	3500.00
82-70002	Deuterium Oxide "100%"	99.96	1 x 10 g	septum	15.00
			5 x 10 g	vial	50.00
			1 kg	pl scb	550.00
82-70003	Deuterium Oxide EXTRA	99.996	10 x 0.75 mL	amp	50.00
			1 x 10 g	amp	45.00
84-70001	Deuterium-depleted Water	< 5 x 10 ⁻⁵	1 x 25 g	scb	25.00
			4 x 25 g	scb	85.00
			10 x 25 g	scb	200.00
			20 x 25 g	scb	300.00
			1 kg	pl scb	500.00
82-00807	Dimethyl-d ₆ Sulfoxide	99.9	10 x 1 g	amp	14.00
			50 x 1 g	amp	65.00
			100 x 1 g	amp	125.00
			1 x 10 g	amp	14.00
			5 x 10 g	amp	65.00
			10 x 10 g	amp	125.00
82-00809	Dimethyl-d ₆ Sulfoxide	99.9	1 x 10 g	septum	14.00
			5 x 10 g	vial	65.00
			10 x 10 g		125.00
82-00813	Dimethyl-d ₆ Sulfoxide + 0.05% TMS (v/v) 99.9		1 x 25 g	scb	33.00
			5 x 25 g	scb	155.00
			1 x 100 g	scb	115.00

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SAN FRANCISCO, CALIFORNIA 94143

(received 4/25/90)

Dr. Bernard L. Shapiro
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto, CA ~~94030~~ 94303Spectral Editing of Heteronuclear Correlation Spectra
of Isotopically Enriched Proteins

Dear Dr. Shapiro:

The utility of ^{15}N - correlation methods in the assignment of proteins in the 10 to 20 kDa range is now well established.^{1,2,3,4} Recently we have isolated a sample of the soluble heme binding domain of rat hepatic cytochrome b_5 globally labeled with ^{15}N . We prepared the labeled protein biosynthetically by fermentation of a strain of E. coli transformed with a plasmid expressing the protein on a minimal medium containing $^{15}\text{NH}_4\text{Cl}$ as the sole source of nitrogen.⁵ We obtained the expression system for the protein from Prof. S. G. Sligar's laboratory at the University of Illinois. This expression system consisted of the synthetic gene coding for the published sequence of the 98 residue protein incorporated into a puc13 plasmid in an E. coli K-12 strain⁶.

We have employed a double-DEPT experiment for the acquisition of ^{15}N - proton correlation spectra of the labeled protein.⁷ In addition, we have developed HOHAHA and NOESY relayed double-DEPT experiments. Although somewhat less sensitive than heteronuclear multiple quantum correlation (HMQC) techniques^{8,9,10}, the double-DEPT experiment is of value for its selectivity. The double-DEPT procedure allows one to select for ^{15}N - proton correlations which arise from spin systems with a single proton attached to a ^{15}N nucleus. Despite the high intrinsic chemical shift dispersion of heteronuclei such as ^{15}N and ^{13}C , certain regions of heteronuclear correlation spectra of isotopically enriched proteins are sufficiently complex that effective spectral editing could be useful. For the case of the rat cytochrome b_5 only main chain amide proton - ^{15}N spin systems are observed. Side chain amide spin systems are largely suppressed. Although of limited utility in this particular application, in that side chain amide peaks can generally be distinguished by the presence of two peaks aligned along the same ^{15}N resonance, we believe this technique could be of much greater utility in editing crowded regions of ^{13}C - proton correlation spectra.

Shown below (Figure 1) are a double-DEPT correlation spectrum and a HMQC spectrum collected under identical conditions. Some regions containing additional peaks due to side chain amide protons found in the HMQC spectrum which are largely suppressed in the double-DEPT spectrum are circled on the HMQC spectrum. Shown in Figure 2 is a NOESY relayed double-DEPT ^{15}N - proton correlation spectrum of cytochrome b_5 . Some typical NOESY connectivity paths for some helical segments are indicated on the plot. The pulse sequences for the double-DEPT, HOHAHA and NOESY relayed double-DEPT are shown in Figure 3. Quadrature detection in ω_1 was achieved by time-proportional phase incrementation of the third 90 degree pulse on the heteronucleus. Complete phase expressions are listed in reference 7.

Yours sincerely,

R. D. Guiles

Vladimir J. Basus

Figure 1

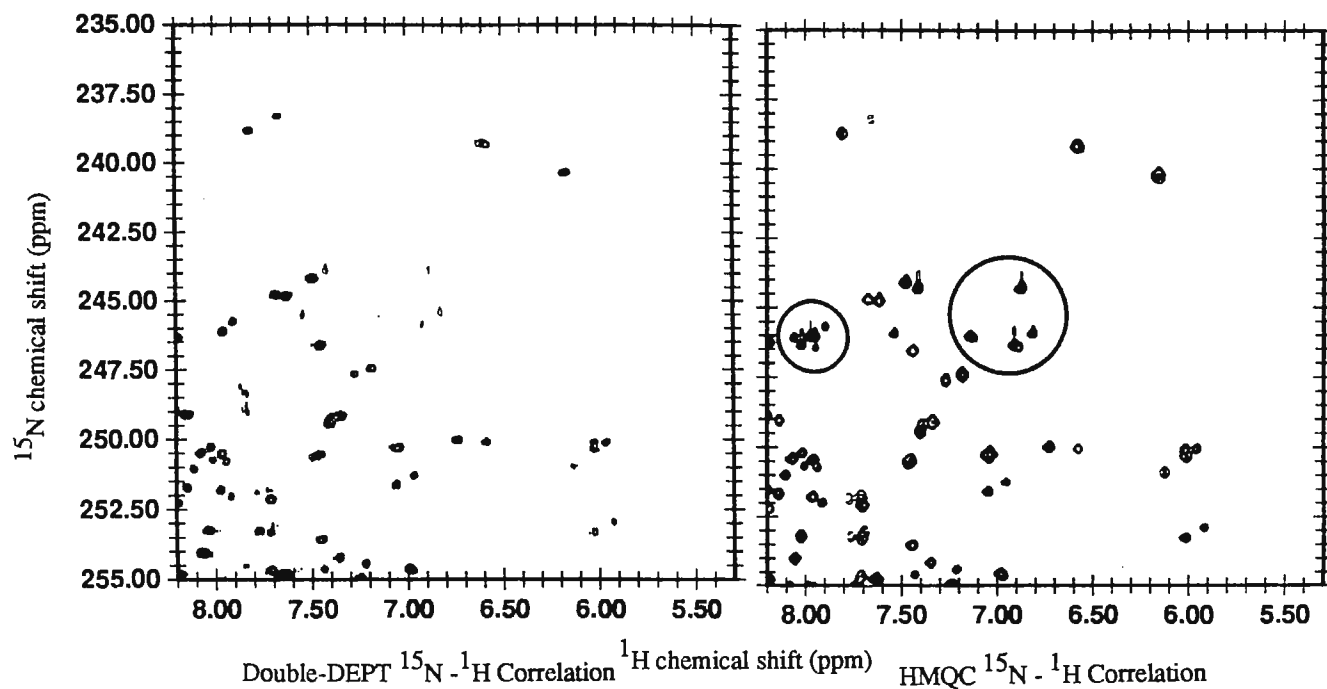
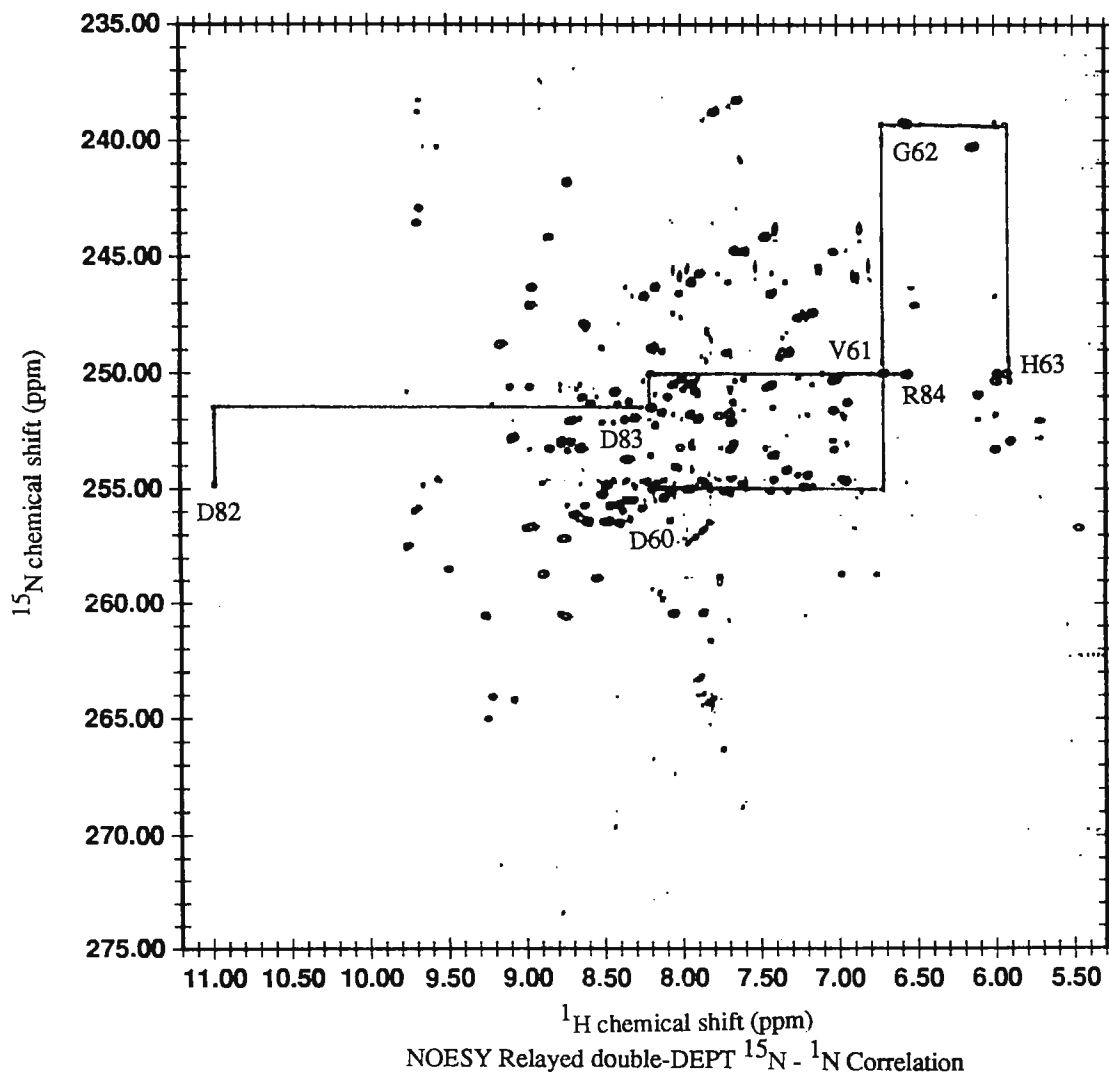


Figure 2



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Switching: 1-20 μ s

Output: +3 to +13dBm; 50ohm
Spurious Outputs: -75dB
Phase Noise: -63dBc (0.5Hz-15KHz)

Freq. St'd: OCXO, TCXO, Ext.
Interface: BCD par. or GPIB
Price: \$5,850.00*

PTS 250

Range: 1-250 MHz
Resolution: 0.1Hz-100KHz (opt)
Switching: 1-20 μ s

Output: +3 to +13dBm; 50ohm
Spurious Outputs: -70dB
Phase Noise: -63dBc (0.5Hz-15KHz)

Freq. St'd: OCXO, TCXO, Ext.
Interface: BCD par. or GPIB
Price: \$6,700.00*

PTS 300

Range: 0.1-300 MHz
Resolution: 1Hz
Switching: 1-20 μ s
Phase Continuous: 1Hz-100KHz steps

Output: +3 to +13dBm; 50ohm
Spurious Outputs: Type 1
-70/65 (typ/spec)
Phase Noise: -68dBc (0.5Hz-15KHz)

Freq. St'd: OCXO, TCXO, Ext.
Interface: BCD par. or GPIB
Price: Type 1 Type 2
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PTS 500

Range: 1-500 MHz
Resolution: 0.1Hz-100KHz (opt)
Switching: 1-20 μ s

Output: +3 to +13dBm; 50ohm
Spurious Outputs: -70dB
Phase Noise: -63dBc (0.5Hz-15KHz)

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NMR

NEWSLETTER

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Second Notice re 1990-91 Invoices and Change in Subscription Rates

Subscription renewal invoices for the October 1990 - September 1991 year will be mailed out during the last ten days of June. If you ought to receive such an invoice, and do not have it in your hands by July 6, please call or write me promptly. Payment of these invoices must be received by me no later than September 5, 1990 to ensure uninterrupted mailing of the Newsletter issues. Please do not delay execution of any necessary paperwork! Also, please be sure that the instructions on the invoice are followed precisely.

When you receive your invoice, you will find that it has become necessary to make a modest increase in the subscription rate. Note that this is the first such increase in three years. I regret that inflationary pressures in general, and a substantial incipient increase in the postage rates in particular, make the subscription rate increase necessary. Advertising rates will also increase modestly, and our loyal Sponsors will be asked to increase their contributions. The surcharge for Air Mail Printed Matter mailing will also need to be incremented.

The new subscription rate, effective 1 October 1990, will be US\$150.00 for the twelve monthly issues, postpaid. Personal or academic subscriptions will continue to be offered at a 50% discount, US\$75.00.

Thank you for your understanding and cooperation.

B. L. Shapiro
1 May 1990, 1 June 1990

All Newsletter Correspondence

Should Be Addressed To:

Dr. Bernard L. Shapiro
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto, CA 94303, U.S.A.

(415) 493-5971

DEADLINE DATES

No. 383 (August)----- 20 July 1990
No. 384 (September)----- 17 August 1990
No. 385 (October)-----14 September 1990
No. 386 (November)-----12 October 1990

CSI 2T Applications

Shielded Gradients and Localized Spectroscopy

Eddy current effects are the leading cause of errors and lack of consistent results in gradient localization methods. It is not surprising, then, that actively shielded gradients, which have dramatically reduced eddy currents, represent a significant technology advance for all forms of B_0 gradient volume localization and spectroscopic imaging methods. The fast rise time and high gradient strength characteristics of the coil used in these experiments are also important.

Even without pre-emphasis, shielded gradients recover fast enough to obtain spectroscopic information at 1 msec or less after a strong gradient has been turned off (Fig. 1).

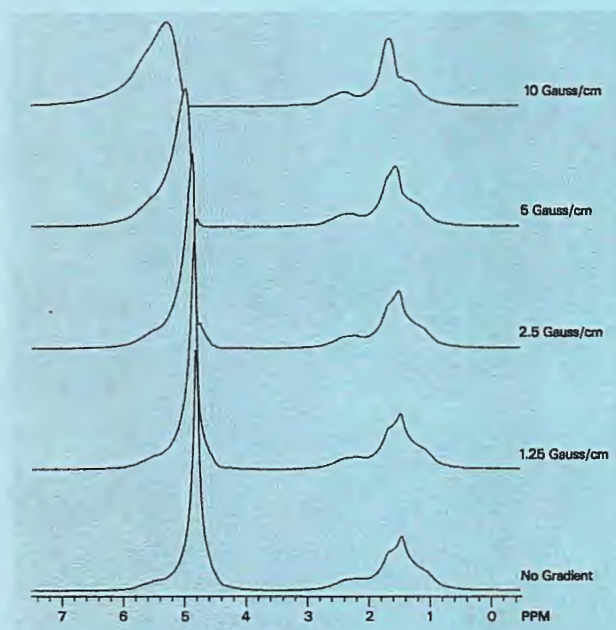


Fig. 1—Using an oil/water phantom, a 10 G/cm gradient will create a water frequency profile extending from 156 KHz to 280 KHz away from normal water resonance. Residual gradient effects of less than 0.01% (50 Hz at 10 G/cm) are observed in a spectrum acquired beginning 1 msec after a 20 msec gradient pulse.

As an example, a 4DFT spectroscopic imaging technique can resolve the four frequency domains that are associated with an NMR signal from an object: x-, y-, z-spatial coordinates and chemical shift δ . The above technique can be a practical alternative to single volume localized spectroscopy. This method allows phosphorous spectra to be obtained from well-defined regions as demonstrated in the following experiment, which was carried out on a GE CSI 2T system using high-strength, shielded gradient coils (Fig. 2). The phase-encode time is kept short (on the order of the dwell-time) to minimize phase-errors in the final spectra, as well as to avoid loss of signal due to T2 decay, which is significantly short in biological phosphates.

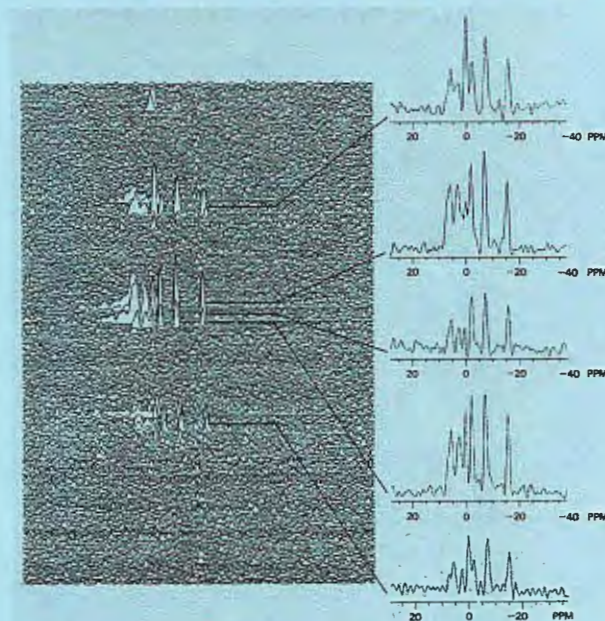


Fig. 2—Stacked plot showing 512 phosphorous spectra from 60 mm cubed region of a live rat. Each trace corresponds to 7.5 mm cubed region (voxel) from within the region of interest. The offset traces clearly show the achievable spectra and spatial resolution of the technique, as well as demonstrating localization of the liver phosphorous metabolites from that of overlying skeletal muscle. Total acquisition and processing time was two hours.



GE NMR Instruments

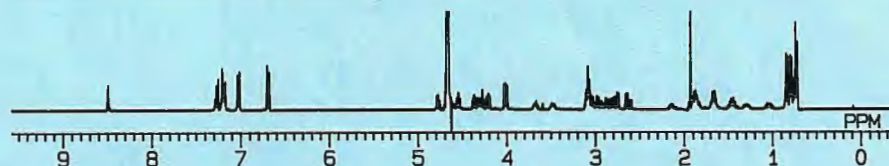
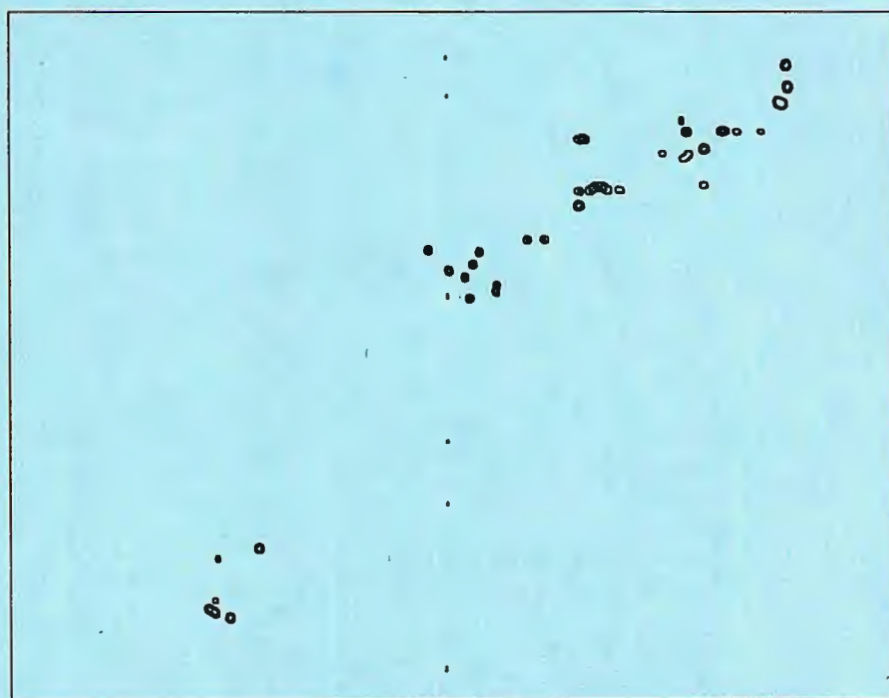
255 FOURIER AVENUE, FREMONT, CA 94539
(415) 683-4408, FAX (415) 490-6586

PRAUNHEIMER LANDSTRASSE 50, D-6 FRANKFURT 90
WEST GERMANY 4969 760 7431, TELEX 041 2002 GEG

AFTER THE WORK IS FINISHED...

THE OTHER CPF COMES OUT TO PLAY

When all of the day's production samples have been run and the piles of spectra plowed through, JEOL's CPF can quickly switch operation modes. This same highly automated machine which is used for all of the production work can produce very high quality research data for those non-routine problems that always seem to appear. In many cases all that is necessary to make the change is to log into the research account. This changes the CPF from an automated, limited instrument into a wide-open research-grade spectrometer. With the addition of the appropriate accessories the low cost CPF is capable of running the most sophisticated research type experiments including CP MAS solids. Now that the CPF is available at a field strength of 400 MHz in addition to the very popular 270 MHz, the high field research spectrometer has become very affordable. The above 400-CPF data is a reverse detection ^{13}C experiment run on Angiotensin-II. The instrument used for this experiment is a standard CPF with the addition of the optionally available reverse detection Broad Band probe. This data clearly shows that the routine need never be the only thing you can do.



For more information please contact:

JEOL

11 Dearborn Road, Peabody, MA 01960 (508) 535-5900 (Phone) (508) 535-7741 (FAX)