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FORTHCOMING NMR MEETINGS

- XIII Intl. Conference on Magnetic Resonance in Biological Systems, Aug. 14-19, 1988; Madison, Wisconsin. See Newsletter 349, 60.
- NATO Summer School: "A Methodological Approach to Multinuclear Magnetic Resonance in Liquids and Solids: Chemical Applications", August 22 - September 2, 1988; Maratea, Italy; See Newsletter 353, 76.
- XXIV Ampere Congress on Magnetic Resonance and Related Phenomena, August 29 - September 3, 1988; Poznan, Poland; Dr. S. Hoffmann, Instytut Fizyki Molekularnej PAN, ul. Smoluchowskiego 17/19, 60-179 Poznan, Poland.
- Teaching Course on Nuclear Magnetic Resonance, September 5 - 9, 1988; Trondheim, Norway; Ms. I. S. Gribbestad, The MR Center, N-7034, Trondheim, Norway.
- European Workshop on Nuclear Magnetic Resonance: Seminar on Contrast in MRI and MRS, September 12 - 13, 1988; Trondheim, Norway; Ms. I. S. Gribbestad, The MR Center, N-7034, Trondheim, Norway.
- American Chemical Society, 196th National Meeting, September 25 - 30, 1988; Los Angeles, CA; Includes several symposia on NMR; Contact B. R. Hodsdon, 1155 16th Street NW, Washington, CD 20036, (202)872-4396.
- 27th Annual Eastern Analytical Symposium and Exposition, October 2 - 7, 1988; New York Hilton Hotel, New York; General Chairman - Dr. Harvey S. Gold, Polymer Products Dept., E256/308, E. I. du Pont de Nemours & Co., Wilmington, DE 19898; (302) 695-3669.
- International Post-Graduate Course 'NMR in Agriculture, Plants and Products', October 3 - 15, 1988; Wageningen, The Netherlands; Dr. Ir. J. H. de Ru, Foundation for Post-Graduate Courses, Agricultural University, Hollansegweg 1, NL-6706 KN Wageningen, The Netherlands.

Additional listings of meetings, etc., are invited.

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. 360 (September) — 19 August 1988
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No. 362 (November) — 21 October 1988
No. 363 (December) — 18 November 1988

BIRKBECK COLLEGE
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Department of Chemistry
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28 April 1988 (received 5/31/88)

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

Dear Professor Shapiro,

QUADRUPOLEAR INTERACTIONS IN THE DIPOLAR NMR SPECTRA OF PLASTIC CRYSTALS

In the solid state, dilute spins such as ^{13}C can be studied by spinning the sample at the magic angle (MAS) which averages the broad anisotropic interactions to leave a narrow "liquid-like" spectrum. This experiment is not always ideal for studies of molecular rotation in solids because magic-angle spinning also averages the intrinsic motion of the molecules:- a sharp spectrum is observed but the effect you want to study is destroyed.

The individual molecular rotations can be returned to the narrowed MAS spectrum by selective substitution with quadrupolar spins. In this case the angular dependence of the interactions changes and there is no longer any unique magic angle. For ^{13}C bonded deuterium the spectrum is split with each carbon resonance broadened into a quadrupolar powder pattern. The theory of these effects has been discussed by several authors.

An interesting example of the effect is shown in the Figure of the ^{13}C CP-MAS nmr spectrum of $[(\text{C}_6\text{D}_6)\text{Fe}(\text{C}_5\text{H}_5)] [\text{AsF}_6]$, (I). At high temperatures the benzene carbons spin couple with the three spin states of deuterium to give three sharp isotropic signals, indicating that all the alpha phase molecules must exhibit fast isotropic rotation. At lower temperatures the individual resonances are broadened into different overlapping powder patterns which can be assigned to different modes of anisotropic rotation. In I the beta phase motion consists of external end-over-end rotations of the whole molecule combined with internal rotations of the benzene ring, whilst in the gamma phase all the external rotations are quenched and only the ring rotations remain.

Yours sincerely

David B. Davies

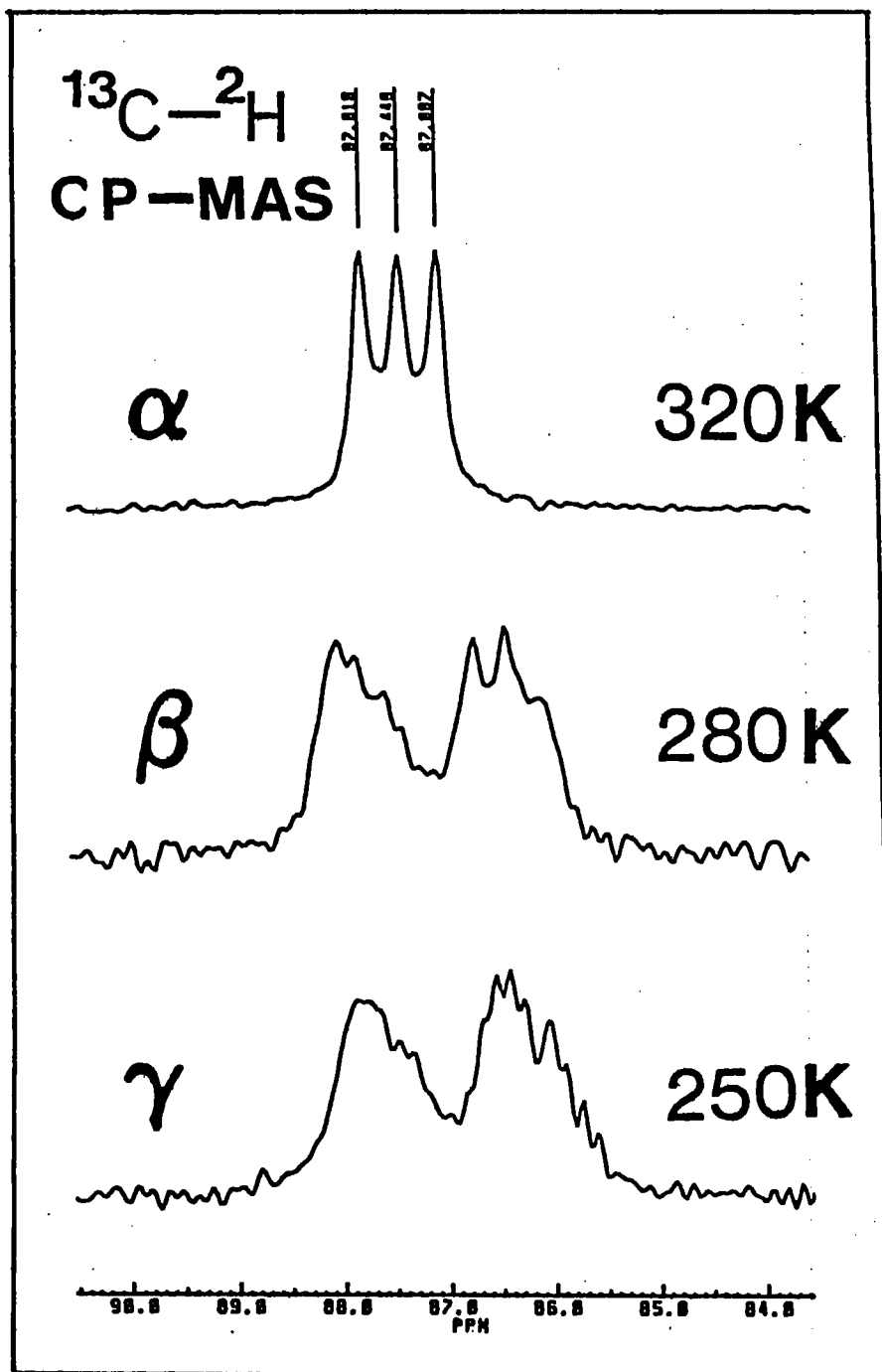
D B Davies

I Sayer

I Sayer

¹ Opella et al *Phil Trans Roy Soc. Lond.* A299, 665 (1981)
Bohm et al *J. Mag Res* 55, 197 (1983)

Figure: Benzene region of the ^{13}C CP-MAS nmr spectrum of I. The motion of the deuterio-benzene ring can be analysed from the residual powder patterns which are unaveraged by MAS.





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

May 30, 1988
(received 6/10/88)

Dear Barry,

After having received several reminders from you, Sture Forsén asked me, under threat of corporal punishment, to report somewhat on the NMR-activities going on at the Divisions of Physical Chemistry in Lund.

For this contribution, we thought it appropriate to report on some of the work going on in the area of surface chemistry here in Lund. For some time we have been studying viscoelastic micellar solutions formed by some cationic surfactants when the "common" inorganic counter-ion, such as bromine, is replaced with organic counter-ions, such as the salicylate-ion.

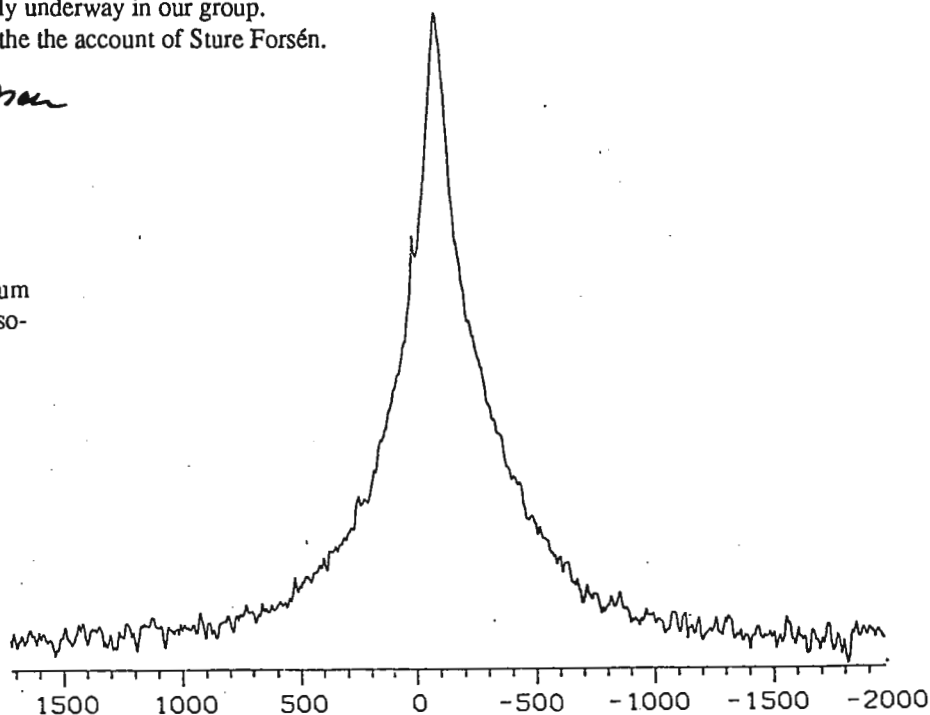
Shown in fig. 1 is the ^2H NMR-spectrum, taken at 26°C on a 8.5 T spectrometer, of a 0.2 wt % hexadecyltrimethylammonium salicylate solution, where the protons on the three methyl-groups on the nitrogen have been replaced with deuterons. Clearly, the NMR-bandform is non-Lorentzian. Moreover, there is more intensity in the wings of the spectrum than expected for a Lorentzian band, implying that the observed band-shape is in fact a sum of Lorentzians. Now, these viscoelastic solutions are composed of large micellar aggregates with varying size, and it is the tumbling, and in particular the end-over-end tumbling, of these aggregates that determines the bandwidth. Thus, for a situation where the exchange rate of surfactants between the different micellar aggregates is slower than the difference in R_2 between the micelles, a spectrum which is a sum of Lorentzians would result. This is clearly the case in the system presently under study. Since the rate of the end-over-end tumbling, and thus the bandwidth, is related to the length of the micelle it is possible to test various suggested distribution functions for the polydispersity in size of micellar aggregates against the observed bandshape in fig. 1.

Work along these lines is presently underway in our group.

Please credit this contribution to the the account of Sture Forsén.

Olle Söderman
Olle Söderman Ulf Olsson

Fig 1. A ^2H NMR spectrum
of a viscoelastic micellar so-
lution.



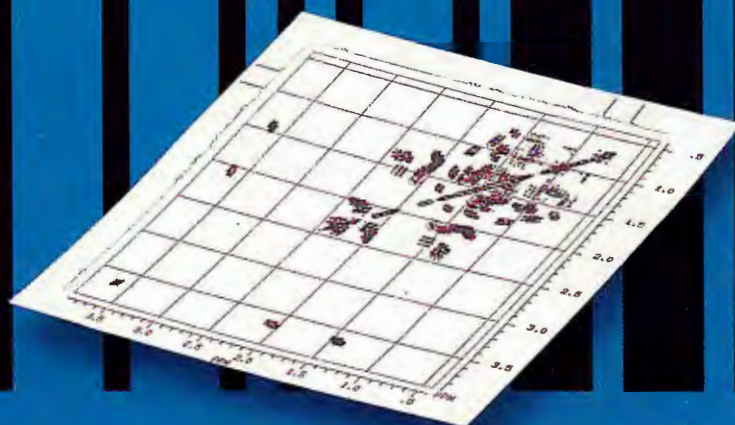
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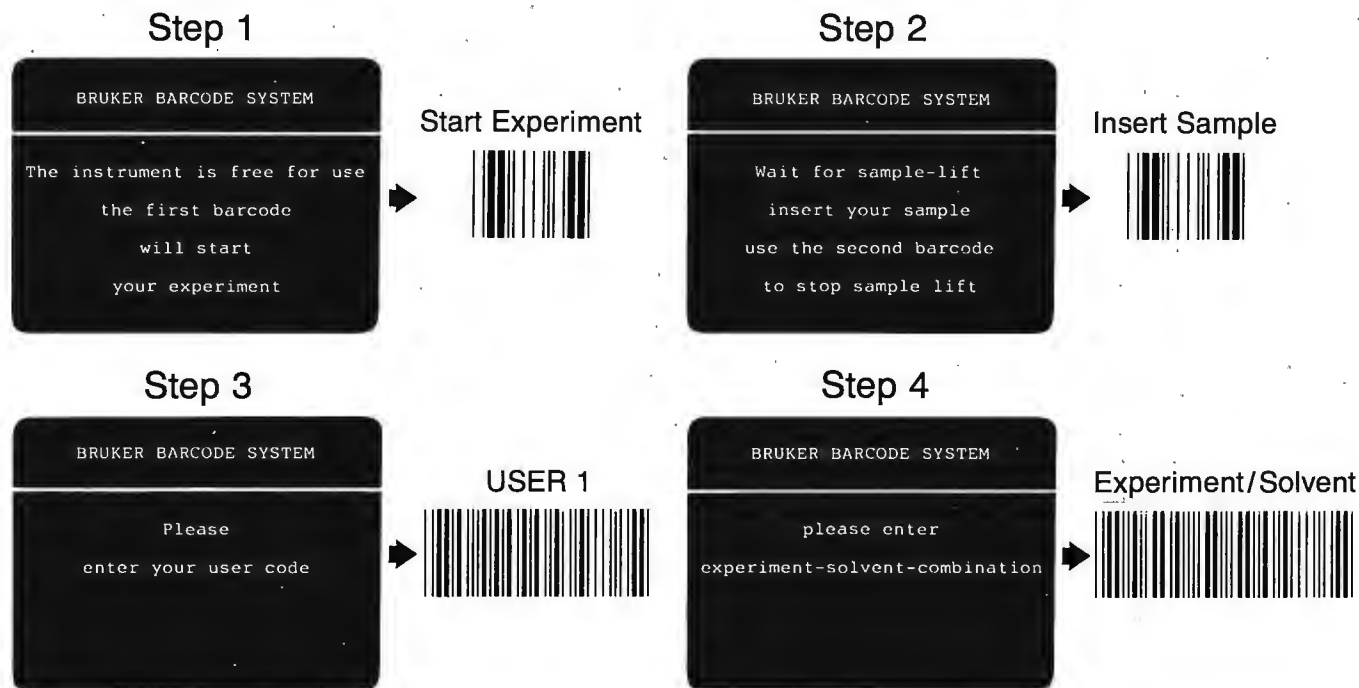
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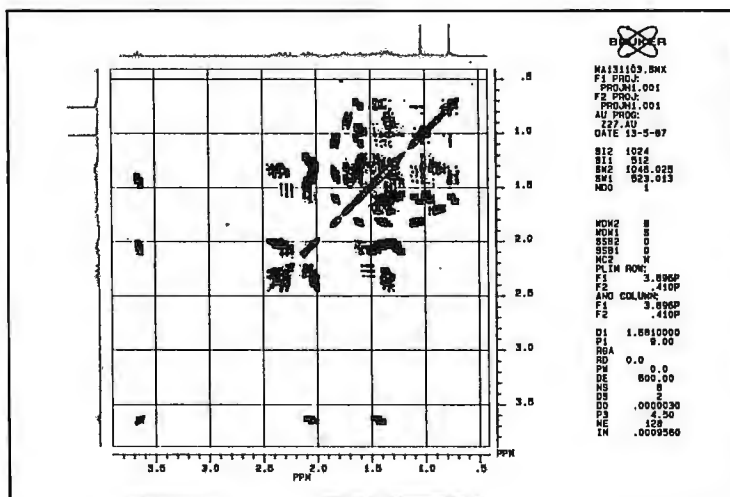


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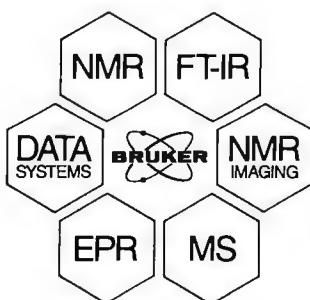
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Department of Physical Chemistry
Professor Peter Stilbs

358-7

Date

Stockholm, June 17, 1988
(received 6/28/88)

Prof. B L Shapiro
TAMU NMR Newsletter
966 Elsinore Court
PALO ALTO, CA 94303

U S A

"NMR1/NMR2 data and IMAGE processing on VAXstation 3200"

Dear Barry,

Thank you for your "gentle reminder". We are gradually getting in order at this site after the remodelling. One remaining problem appears to be that the roof paneling (10 mm gypsum plates on a metal frame) collapses at times, falling down, damaging instrumentation and causing a big mess. So far no persons have been hurt.

In April we got the first Vaxstation 3200 to be installed in Sweden. Apparently it was also the first one on which NMR1/NMR2 was installed. Performance is far superior to the GPX model and comparable to the Sun 3/160. Also it could be incorporated in our VAX LAVC, providing good general BATCH processing capacity. Only a few technical problems have appeared. The hardware was purchased from DEC Sweden and the software from NMRI, so the system was not tested before delivery. Some plotting commands had logical bugs, most of which have been fixed. It is also a great relief to have a FAX machine when sorting out problems from overseas. NMRI support has worked fine, even including a personal visit. Our FAX number (if you care to call us) is (+46 8 7908207).

We recently got partial delivery of Bruker Ethernet, to be interfaced with NMR1/NMR2 at the VAX. It will be installed shortly. Unfortunately Bruker forgot to enclose the BRUKNET software for the VAX. Apparently, present operation of BRUKNET is not straightforward, with some rather severe restrictions as to write operations on the VAX. We hope to have a progress report later this year.

Yours sincerely

Peter Stilbs
Peter Stilbs



Georgia State University

University Plaza • Atlanta, Georgia 30303

Department of Chemistry

May 25, 1988

(received 6/2/88)

Bernard L. Shapiro, Ph.D.
966 Elsinore Court
Palo Alto, CA 94303

Dear Dr. Shapiro:

We often need to titrate solutions in NMR tubes. Usually this is accomplished by measuring the pH directly in the NMR tube with a narrow pH electrode. Recently, however, we have been running titrations on air-sensitive samples. The titrant can be injected through a septum without exposing the sample to air, but the tube cannot be opened for insertion of the pH electrode.

We are turning, therefore, to using pH indicator dyes. To an NMR tube, we add a dye with a pK_a near that of the pK_a of interest in the sample. The dye concentration is chosen to give an easily measurable absorbance in the NMR tube (path length approximately 5 mm). As we run the titration, we calculate the pH of the solution from the optical spectrum.

Optical spectra are taken directly in the NMR tube (Dixon, D.W.; Woehler, S. Rev. Sci. Inst., in press; Traylor, T.G.; Chang, C.K.; Geibel, J.; Berzinis, A.; Mincey, T.; Cannon, J. J. Am. Chem. Soc. 1979, 101, 6716-6731). Useful hints for reproducible results are: a) ensure that the slit width of the optical spectrometer is less than the diameter of the NMR tube (i.e. < 2 mm for a 5 mm NMR tube), b) build a holder which positions the sample reproducibly (we use an aluminum block drilled to accommodate the NMR tube) and c) always turn the same side of the NMR tube toward the beam. We have not found it necessary to mount lenses to focus the light in the UV/visible spectrometer.

The pH can be obtained easily if the ratio $[A^-]/[HA]$ is known ($pH = pK_a + \log([A^-]/[HA])$). This, in turn, is obtained in the standard way as $(Abs - Abs_{HA})/(Abs - Abs_{A^-})$ where Abs is the absorbance of the dye in the solution of interest, Abs_{HA} is the absorbance of the protonated species and Abs_{A^-} is the absorbance of the deprotonated species.

The Figure illustrates an experiment using pyridine as the base and bromocresol green ($\epsilon = 40,100$ at 614 nm) as the dye. The plot shows the chemical shift of the resonances of pyridine as a function of "pD", i.e. the pD calculated after each addition

of titrant assuming that the $pK_a(D_2O)$ of bromocresol green is 5.18. The latter value was measured in a separate experiment in D_2O with the pH meter calibrated against H_2O buffers.

Some experiments will not tolerate the addition of a dye. But for many experiments in which the NMR tube cannot be opened, this technique provides a useful solution to the measurement of pH in NMR tubes.

Sincerely yours,

Dabney K White Dixon

Dabney White Dixon

Scott E Woehler

Scott E. Woehler

Louisa Amis

Louisa Amis

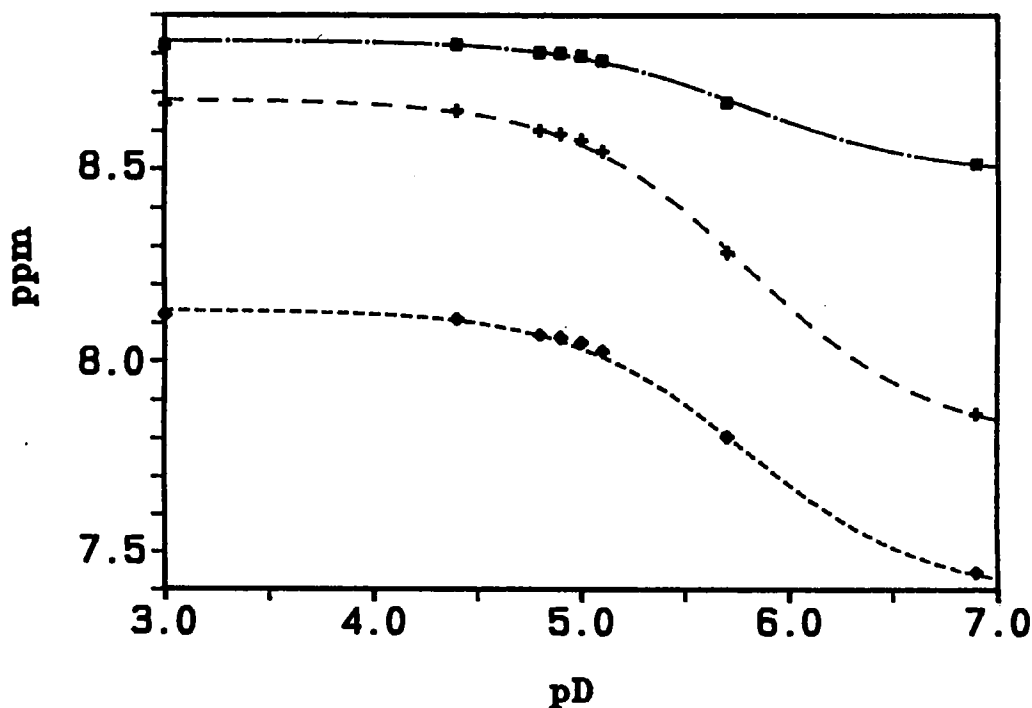


Figure. The chemical shifts of the ortho (■), meta (◆) and para (+) protons of pyridine as a function of pD.

Department of Chemistry and Biochemistry

Campus Box 215
Boulder, Colorado 80309-0215
U.S.A.Dr. B.L. Shapiro
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto, CA 94303

BEWARE OF TILTING MAGNETS

June 15, 1988 (received 6/13/88)

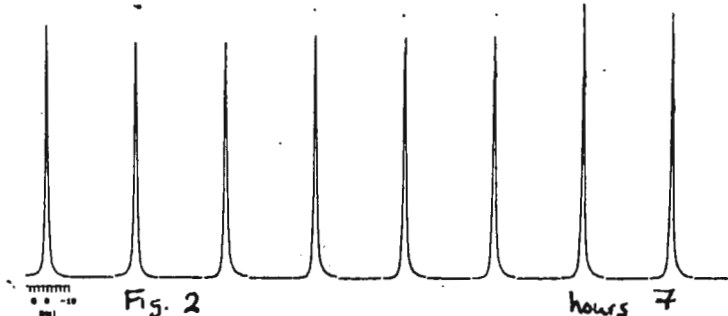
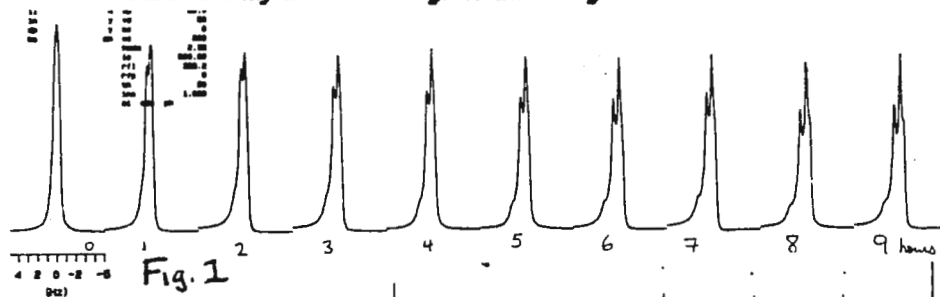
Dear Barry:

I have recently been involved in the installation and check out for two Varian VXR-500 spectrometers, one at Rutgers University and one at my new home in the Department of Chemistry and Biochemistry at the University of Colorado at Boulder. The Rutgers instrument was installed in August 1987 and exhibited some major lineshape problems for the next few months which had both me and Varian extremely concerned. The symptoms of this problem were that the lineshape would degrade dramatically after a few hours. Figure 1 shows the plot of an overnight run of chloroform where the linewidth started around 0.7 Hz with a lock level of 92%. After 9 hours the lineshape is terrible and the lock level decreased to 64%. This was an "average" overnight run and some were much worse. Needless to say Varian and I spent a great deal of effort trying to find the source of the problem and almost every part of the spectrometer and magnet was considered as a possible suspect. After many, many tests and lots of frustration, Varian suggested that the source of the problem could be tilting of the magnet, so we ran the same test after deflating the air shocks used for vibration isolation and an example of this is shown in Figure 2. Here we see that the linewidth varied from 1.0 ± 0.2 Hz overnight and the lock level was essentially constant. Thus it was clear the our problem had to do with the air shocks and Varian informed me that their in house tests showed that tilting of the magnet by even 0.5 degree had a significant effect on lineshape. Since there were no major vibration problems at Rutgers the instrument has been left on the ground and the degradation of lineshape problem has completely disappeared.

A Gemini-300 and VXR-500 were installed at Colorado in April 1988 and fairly large incoherent peaks at ± 30 Hz were found on both instruments. This problem was subsequently found to arise from vibrations cause by air supply motors in the building. There were 7% 30 Hz sidebands for one scan on the 500 which were completely eliminated by inflating the vibration isolators. Luckily these shocks do not seem to be leaking and so we are able to get excellent long term lineshape stability on the 500. We plan to tie the three air shocks together so that they will not cause the magnet to tilt in case one does start to leak. We are presently putting the Colorado instrument through all its paces and so far have been extremely pleased with its performance.

Please note my address change to the one given in the above letterhead.

Sincerely,

Dr. Arthur Pardi
(303) 492-6263

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83-30013	D-Glucose- ¹³ C ₆	1g	325.		
	30 atom % ¹³ C	10g	3000.		
		25g	7125. [†]		
83-61007	High-protein algal biomass	1g	250.		
	90 + atom % ¹³ C	10g	2000.		
	ca. 70% crude protein	25g	4750.		
		50g	8750. [†]		
Typical approximate amino acid analysis (%)					
Ala 6	Cys 1	His 1	Lys 4	Pro 3	Trp 1
Arg 6	Glu 9	Ile 4	Met 2	Ser 4	Tyr 5
Asp 6	Gly 3	Leu 6	Phe 4	Thr 4	Val 6

Isotec Inc. also has stock of a variety of specifically ^{13}C -labelled amino acids as well as

83-30000	D-Glucose-1- ^{13}C 99 atom % ^{13}C	1g	325.
		5g	900.
		10g	1400.
		25g	3250. [†]

*Prices are FOB Miamisburg, Ohio for delivery in North America; please request prices for delivery to the other continents.

[†]Please request prices for quantities that you do not see listed.

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Abbott Laboratories
Abbott Park, Illinois 60064

May 31, 1988 (received 6/22/88)

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

Dear Barry,

PULSE T1 MEASUREMENTS ON A T60: EQUIPMENT FOR SALE

We have been evaluating some compounds as contrast reagents by determining the T1 of water in mouse livers on our NT360 with some encouraging results. Unfortunately, the lowest field strength magnet for pulse experiments is 300 MHz. There is a T60 close to our GN300, 20 feet from the probe interface module on the GN300 to the T60 console. By using the GN300 as our RF source and the magnet and probe of the T60, we were able to run pulse T1 studies at a field strength that approximates current imaging systems, 80 MHz and lower.

The setup for this type of experiment is very simple. A cable (RG 8/U) was run from the low band port of the probe interface module on the GN300 to the A2 port on the T60. Both the transmitter and receiver modules of the T60 were disconnected. See T60 Functional diagram No. 87-121-701. After determining the 90 degree pulse on the T60 probe, the experiment was run using the normal T1IR experiment on the GN300.

A comparison of the T1 values of normal mouse livers is given below.

Mouse Liver	T1 OF WATER IN MOUSE LIVERS	
	360 MHz	60 MHz
#1	1.11	0.48, 0.47
#2	0.97	0.42
#3	0.98	0.44
#4	1.03	0.43, 0.45

NT360 CONSOLE AND EXTRA NIC-1280 COMPUTER

We are purchasing a Nalorac console for imaging small animals such as mice and rats on our 360 widebore magnet as well as running the normal 1D and 2D high-resolution experiments. We hope to have the system in place at Abbott by the end of 1988. To make room for the new console, we plan to sell our NT360 console and an extra 1280 that was originally used as part of a remote data work station. We are keeping the magnet, RT shims and the He and N2 monitors. If anyone is interested in purchasing part or all the electronics, he can either call Jim Carolan at Nalorac (415)229-3501 or Merrill Nuss at Abbott (312)937-2085. The NT360 console was purchased in 1982 and is still used daily for 1D and 2D experiments.

Sincerely,

Merrill *Pete* *Dave* *Dave* *Pat*
Merrill Nuss Peter Fruehan Dave Johnson Dave Betebener Pat Carney

School of Chemistry
Leeds
LS2 9JT
Telephone 431751

May 26, 1988
(received 6/3/88)

The Amazing Tilted Lozenges

Dear Professor Shapiro,

Although we do not wallow extensively in the mysteries of radiofrequency rotating frames and other related matters which might involve us trying to do hard sums beyond our intellect, we nevertheless make great use of multielement¹ n.m.r. in our synthetic polyhedral boron chemistry.

We have been doing some collaborative n.m.r. work with Dr. T.R. Spalding and his group at University College, Cork, Eire, on twelve-vertex *closo*-1,2-chalcogenaplatinaboranes. In this we find [¹H-¹H]-COSY spectroscopy very useful, although, with the borane-cluster ¹H resonances, simultaneous [¹¹B] decoupling must be applied to change the ¹H spectra from what is often an unsightly agglomerate of broad overlapping partially-collapsed 1:1:1:1 quartets to what is in principle a neat row of nicely separated singlets that are generally a few Hertz wide due to the ³J(¹H-B-B-¹H) and related interproton couplings that permit the [¹H-¹H]-COSY experiment to work.

The top trace in the figure shows such a ¹H spectrum for [2,2-(PEt₃)₂-*closo*-2,1-PtTeB₁₀H₁₀]. This is a subtraction of a ¹H-{¹¹B(off-resonance)} spectrum from a ¹H-{¹¹B(on-resonance)} spectrum, a procedure that we have found useful to eliminate all the lines from the ¹H spectrum that are not coupled to ¹¹B. Some of the borane ¹H peaks have distinguishable satellite features (but not on the scale displayed in the figure) due to ⁿJ(¹⁹⁵Pt-¹H) where *n* = 2, 3, 4, and in the corresponding [¹H-¹H]-COSY spectrum we were pleased to see that some of the cross peaks involving these satellited lines took the form of tilted lozenges rather than approximating to more circularly symmetrical blobs. This is because only either a) high-frequency/low-frequency and low/high frequency or b) high/high and low/low correlations are observed, depending on the relative signs of the two ⁿJ(¹⁹⁵Pt-¹H) couplings. A tilt of the lozenge to the right indicates that they are of like sign, and a tilt to the left that they are of opposite sign.

We have also experienced the amazing tilted lozenges in some [¹¹B-¹¹B]-COSY work on platinaborane-type species. However, we have not seen many of these, since only ¹J(¹⁹⁵Pt-¹¹B), which can take values of up to a few hundred Hertz in compounds so far examined, is ever of sufficient magnitude to be seen above the linewidths of the ¹¹B resonances, which are often themselves a few hundred Hertz wide in metallaboranes with bulky ligands on the metal centres. All ¹J(¹⁹⁵Pt-¹¹B) that we have been able to compare so far have been of the same sign, presumably positive.

¹We use the term "multielement", because we consider all our n.m.r. experiments multinuclear until manufacturers improve instrument sensitivity by 10²⁰ or so.

We have recently submitted aspects of this work for consideration for publication in *J. Chem. Soc., Dalton Trans.*

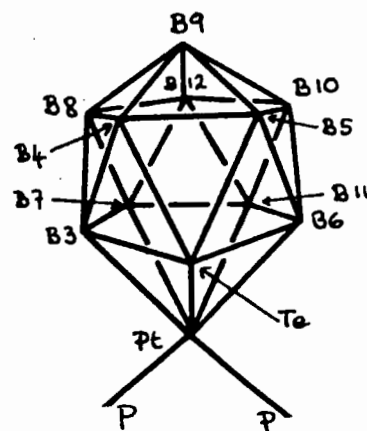
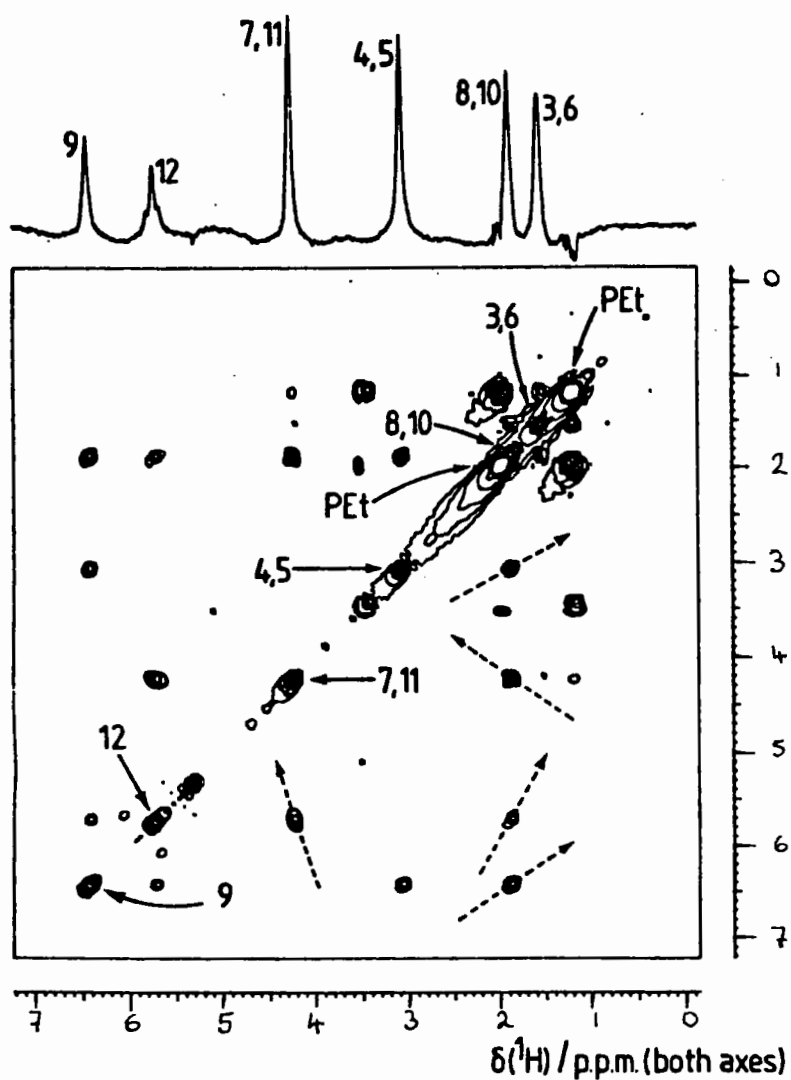
Sincerely,

John D. Kennedy

John D. Kennedy

Xavier Fontaine

Xavier L.R. Fontaine





UNIVERSITY OF
STOCKHOLM
Physical Chemistry

Professor Bernard L. Shapiro
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto, CA 94303, USA

Stockholm, June 17, 1988 (received 6/27/88)

Carbon-13 Relaxation in Sucrose outside of Extreme Narrowing

Dear Dr Shapiro,

Few years ago, McCain and Markley (1) reported measurements of carbon-13 spin-lattice relaxation times and NOEs for sucrose in aqueous solutions as a function of temperature and magnetic field. We have recently extended their work towards longer rotational correlation times, far beyond the extreme narrowing range. This has been accomplished by using a 2:1 solvent mixture of D₂O and DMSO-d₆. The mixture is at room temperature about four times more viscous than water; in addition, it has a freezing point of about -65°C. We performed T₁ and NOE measurements on a 0.5 M sample in the temperature range -15°C to +30°C, at 9.4 and 6.3 Tesla. The relaxation times for the eight non-equivalent CH carbons in the molecule were in all measurements very close to each other, indicating isotropic motion. The average CH T₁ and NOE results are summarized in Fig. 1 and 2, respectively.

The data are interpreted in terms of the following model. The usual expressions for the relaxation rate and the NOE for a molecule undergoing isotropic rotational diffusion (1) are used. The rotational correlation time is assumed to follow a two-parameter, Arrhenius-type (what else at this address!) relation. The interaction strength (the dipole coupling constant, DCC) determining the amplitude of the spectral densities is the third parameter of the model. The results of the least squares fit are shown as solid curves in the figures. The Arrhenius activation energy for the reorientation of sucrose comes out as 37 +/- 2 kJ mol⁻¹, which is high but not unreasonable, and the DCC as 130 +/- 1 kHz. The DCC is about 5% lower than the value recommended by Söderman (2). A similar reduction of the DCC was also reported by McCain and Markley (1).

Yours sincerely

Jozef Kowalewski

Helena Kovacs

REFERENCES

1. D.C. McCain and J.L. Markley, J. Am. Chem. Soc., 108, 4259 (1986).
2. O. Söderman, J. Magn. Res., 68, 296 (1986).

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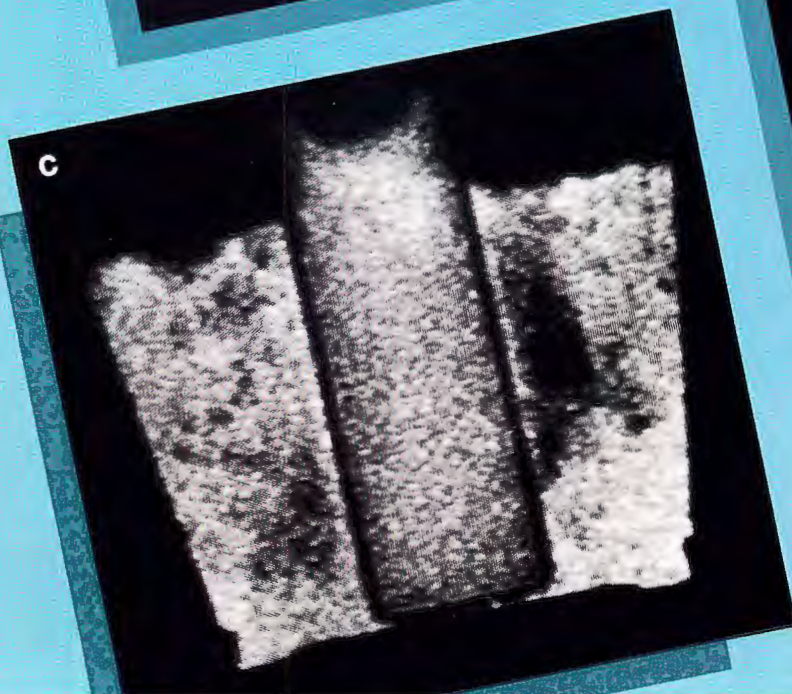
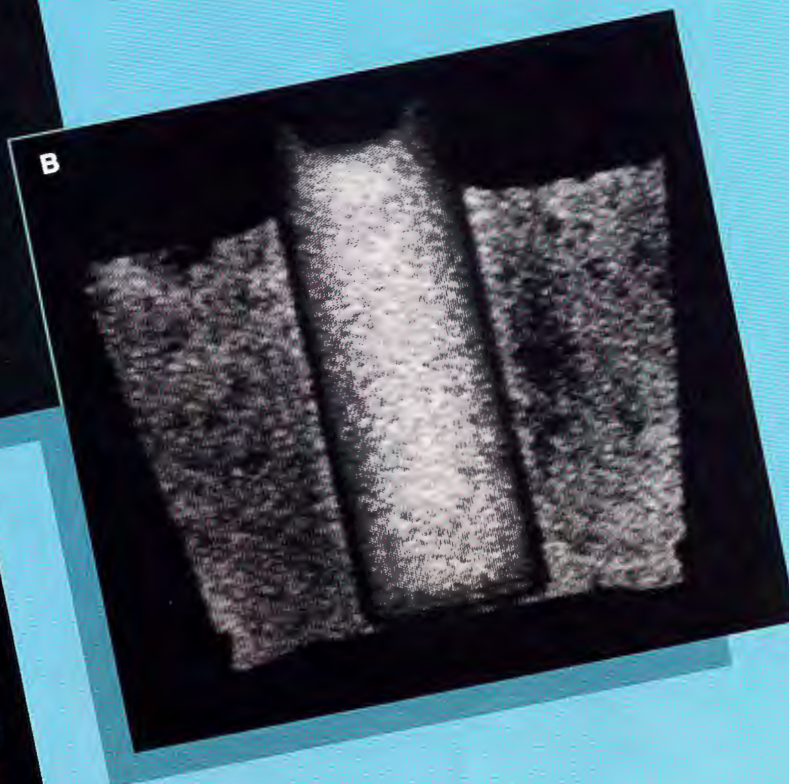
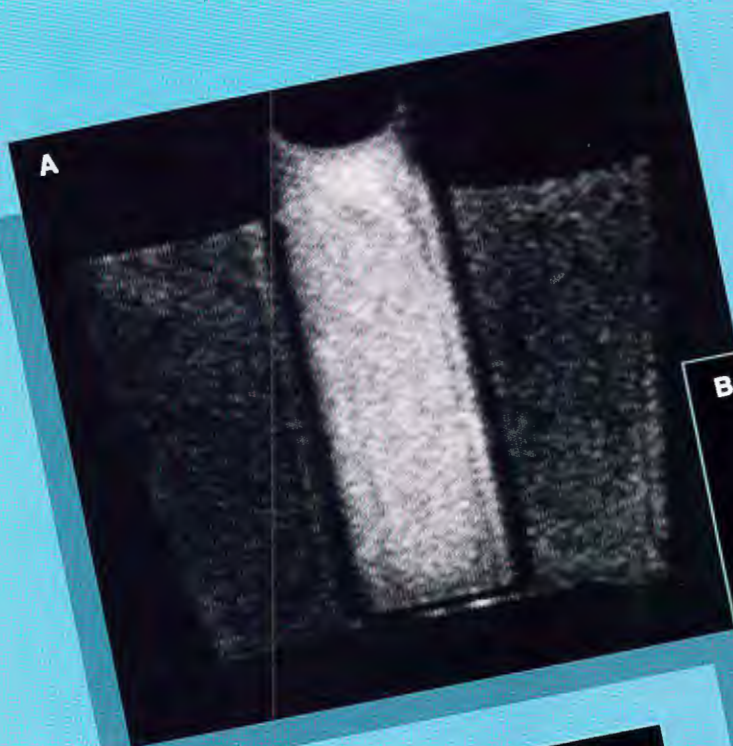
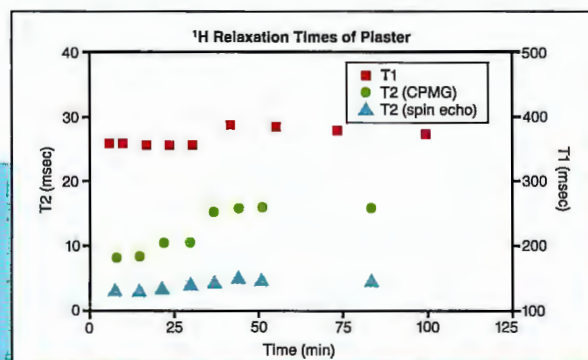
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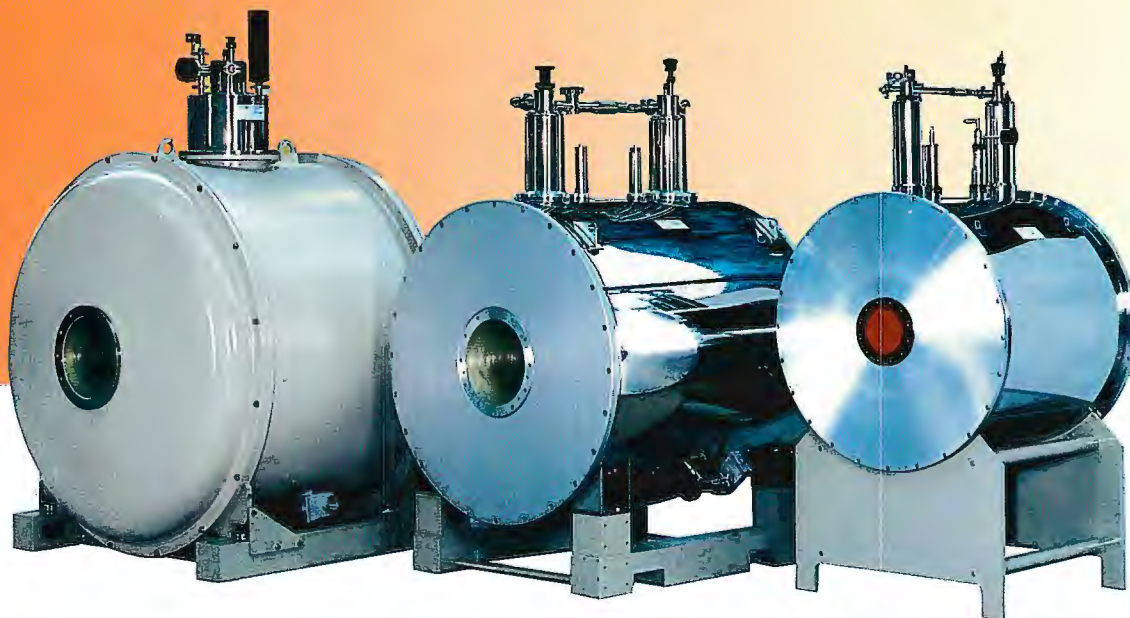
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300/180	7.05T	183 mm	125 mm	4.0 G/cm	80 mm DSV ± 6 ppm	35 mm DSV 0.1 ppm	5.60 m	4.45 m
200/330	4.7T	330 mm	254 mm	2.3 G/cm	140 mm DSV ± 5 ppm	70 mm DSV 0.1 ppm	6.95 m	5.60 m
200/400	4.7T	400 mm	324 mm	1.8 G/cm	140 mm DSV ± 4 ppm	80 mm DSV 0.1 ppm	8.50 m	6.75 m
85/310	2.0T	310 mm	225 mm	3.0 G/cm	100 mm DSV ± 5 ppm	70 mm DSV 0.1 ppm	4.50 m	3.63 m

DSV = Diameter Spherical Volume
HHLW = Half-Height Line Width
PPM = Parts Per Million



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200/400, 200/330, and 300/180 magnets for the NMR Imaging Spectrometer System.
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600 East Mermaid Lane
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19118

June 22, 1988
(received 6/28/88)

Professor B. L. Shapiro
Texas A&M NMR Newsletter
966 Elsinore Court
Palo Alto, CA 94303

TITLE: Optimized Conditions for Obtaining ^{31}P Spectra of N_2 Fixing
Soybean Nodules.

Dear Barry:

As part of our ongoing research effort into the study of metabolism and stress responses associated with plant tissue function, we have embarked on *in vivo* studies of nitrogen fixing soybean nodules.

Initial attempts at obtaining 161.7 MHz ^{31}P spectra of the attached (35-40 days old) nodules suspended in water in a 10 mm tube without air bubbling gave the result seen in Fig. 1A. This spectrum is similar to that from detached nodules reported by Mitsumori et al.¹ (Fig. 1B). However, both spectra do not provide sufficient resolution or show high enough nucleotide levels to be useful for metabolic studies. With gas bubbling there is a slight increase in nucleotide levels (NTP, UDPG). However, the gas bubbles also destroy a good bit of the homogeneity, making the spectral linewidths very broad.

To alleviate these problems, we have devised a system whereby the detached and intact nodules can be constantly perfused (45 ml/min) with an oxygenated buffered glucose solution. The spectrum seen in Fig. 1C shows a considerable increase in S/N resulting from the rapid perfusion of the intact nodules. If the nodules are split in half prior to the perfusion, efficient gas and nutrient penetration produces highly energized nodules which exhibit well resolved spectra as shown in Fig. 1D. In this example, we were able to obtain separation of the three monoester resonances, glucose-6-phosphate, fructose 1,6 diphosphate and choline phosphate. In addition, we have resolved a new phosphodiester resonance "x" which appears to be associated with the nitrogen fixing bacteroids in the symbiotic matrix.

It is clear that the attainment of high resolution is often the name of the game if one is to make some progress in *in vivo* studies.

Sincerely,

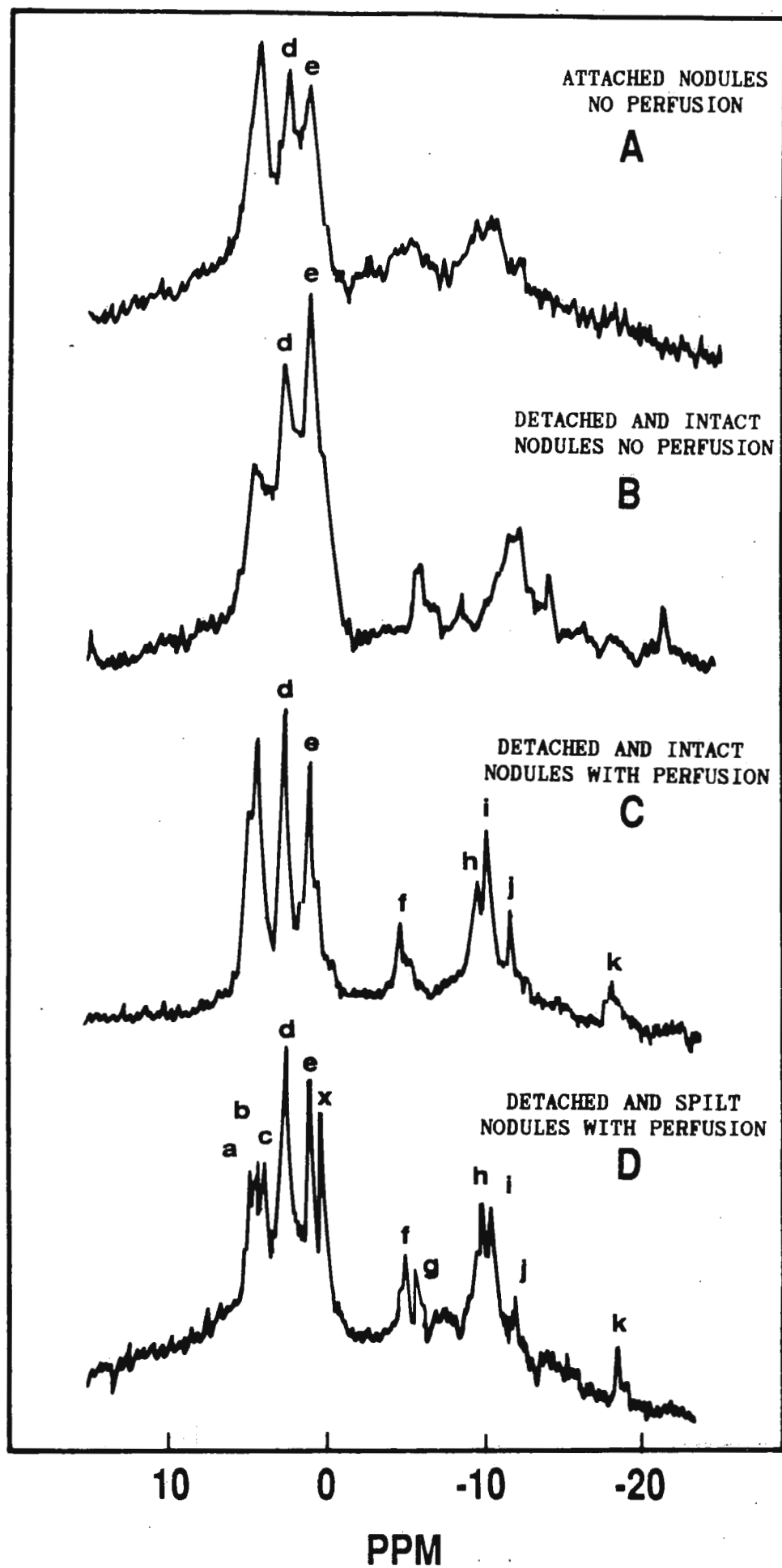
PHILIP E. PFEIFFER

DOMINIQUE ROLIN

RICHARD T. BOSWELL

Reference

1. Mitsumori, F., Yoneyama, T. and Osamu, I. Plant Science 38:87-92 (1985).

Fig.1: IN VIVO ^{31}P NMR SPECTRA OF SOYBEAN ROOT NODULES.

NMR SPECTROSCOPIST. Spectral Data Services, Inc. has an opening for an NMR spectroscopist. Duties will include operation and maintenance of spectrometers, experimental design, interpretation of results, and updating and improving NMR hardware and software. Applicants should have a degree in physical science and experience in NMR spectroscopy. Polymer or zeolite background preferred. Excellent communication skills and experience with rf electronics is desirable. Salary will be contingent on experience. Interested applicants should send resumes to: Gary L. Turner, President, Spectral Data Services, Inc., 818 Pioneer, Champaign, IL 61820, or call (217)352-7084.

Post-Doctoral Positions Available

I currently have openings for two post-doctoral positions. One is for our studies of the DNA repair pathway and the occurrence of multiple conformations in DNA duplexes (JACS 109, 7217; 110, 1620; 110, 2690). The other is for our staphylococcal nuclease project which is aimed at comparing the structures and activities of wild type and mutant proteins (Biochemistry 26, 6278 and about the current issue). (Both positions will include activity in NMR and molecular dynamics.) We have lots of NMR spectrometer time and a SUN 3/280 for off-line data processing. Computational and graphics facilities include an E&S PS350, Silicon Graphics 4DGT70, a number of microvaxes and access to supercomputers. Please contact Philip H. Bolton, Department of Chemistry, Wesleyan University, Middletown, CT, 06457.

POSITIONS AVAILABLE FOR POSTDOCTORAL FELLOWSHIPS AND JUNIOR FACULTY POSITIONS AT UNIVERSITY OF CALIFORNIA, SAN FRANCISCO

The Magnetic Resonance Unit at the Veterans Administration Medical Center/University of California, San Francisco has several positions available. Activities in this program include: Development and improvement of magnetic resonance localization techniques for *in vivo* MR spectroscopy using computer simulations and computer pulse optimization. In particular, spectroscopic imaging techniques are being developed and compared with MR localization techniques. *In vivo* MR experiments on animals includes studies of cancers, cerebrovascular ischemia, and cardiac ischemia. Projects on human subjects include studies of muscle, heart, brain, liver, kidney, and cancer.

MR instrumentation includes a 2.0 Tesla whole body MRI/MRS system, an animal 2.0 Tesla MR spectroscopy system, and a (soon to be delivered) 7.0 Tesla MRI/MRS system with self-shielding gradient coils for small animals, perfused organs, and isolated cells.

Faculty include Gerald Matson, Ph.D., Andrew Maudsley, Ph.D., Donald Twieg, Ph.D., Gregory Karczmar, Ph.D., Hoby Hetherington, Ph.D., Albert Thomas, Ph.D., and Michael Weiner, M.D. Interested candidates should write to:

Dr. Michael W. Weiner, M.D.
Magnetic Resonance Unit (110)
VA Medical Center
4150 Clement Street
San Francisco, CA 94121

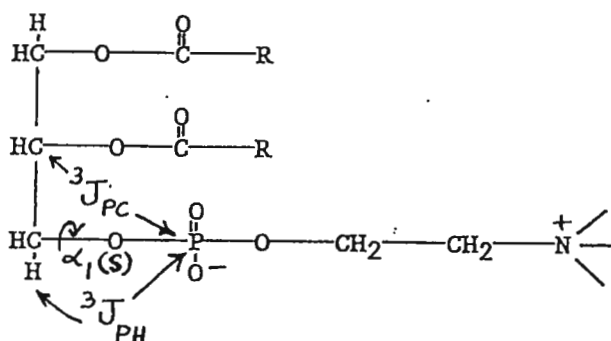
The Michael Faraday Laboratories
Department of Chemistry
815 753 1131

June 20, 1988 (received 6/23/88)

Professor Bernard Shapiro
966 Elsinore Court
Palo Alto, CA 94303Title: $^3J(\text{POCC})$ and $^3J(\text{POCH})$ in Phospholipids and Thiophospholipids

Dear Barry:

The oxygen and sulfur analogues of dioctanoylphosphatidylcholine exhibit some interesting physical and chemical differences. The critical micelle concentration for the sulfur analogue is lower than that of the oxygen analogue, meaning that it aggregates more easily in aqueous solution. Also, the enzyme *B. Cereus* phospholipase C hydrolyzes the sulfur analogue at a rate approximately 100 times slower than it hydrolyzes the oxygen analogue. The C-S or C-O bond is the site of hydrolysis for phospholipase C.



We have measured most of the vicinal coupling constants for these phospholipids (0.100 M in $\text{CDCl}_3/\text{CD}_3\text{OD}$ 2:1 v/v), hoping to discover conformational differences between the two analogues. The most interesting data to come out of the work is the large difference in the coupling constants shown in the figure. These coupling constants are related to the value of the dihedral angle α_1 . In the oxygen analogue, $^3J(\text{POCC}) = 7.89$ Hz. Using qualitatively Lankhorst's relationship (1) between $^3J(\text{POCC})$ and dihedral angle, and assuming a three-fold rotational barrier about the C-O bond, one comes up with a predominantly trans conformation about α_1 (P and C trans). This is expected on the basis of prior NMR and X-ray diffraction studies of phospholipids. However, for the sulfur analogue, $^3J(\text{PSCC}) =$

CSI 2T Applications

Shielded Gradients: Theory and Design

NMR imaging and localized spectroscopy depend on the use of pulsed magnetic field gradients. As these techniques have grown more complex, it has become apparent that eddy currents created in the magnet cryostat and other structures by pulsed gradients have become the chief limitation to many sophisticated applications.

Figure 1a illustrates the design problem for unshielded gradients. Figure 1b illustrates the shielded gradient arrangement. Figures 2a and 2b show the contours of constant flux for an unshielded and shielded Z gradient coil, respectively. This demonstrates that, for the shielded gradients, most of the flux has been kept away from the magnet bore.

The GE corporate Research and Development Center has designed and implemented two experimental shielded gradient designs for the CSI 2T system. Figure 3a is a photograph of one of these sets. Table 1 shows some of the properties of these gradients (Fig. 3b).

The dramatic reduction of eddy currents which can be made over the conventional, unshielded gradients is shown in Figures 4a, b. These graphs show frequency as a function of time following the application of a long, constant amplitude gradient pulse which is suddenly cut off. Soon after cut off, a 90° pulse is applied and the complex FID recorded. The instantaneous frequency is then obtained from the FID and normalized by dividing by the frequency offset at the sample during the gradient pulse.

Figure 4a shows a typical decay of extra magnetic fields in a CSI 2T instrument caused by eddy currents in the conventional, unshielded gradient set with compression.

Figure 4b shows the decay of the uncompensated shielded Z gradient and Figure 4c shows the Z gradient decay with compensation. Note that the time scale for 4b and 4c is five times shorter than that for the unshielded gradients.

References:

1. Mansfield P, Chapman B: *J Magn Reson.*, 1986, 66: 573-576; *J Phys.*, 1986, D 19, L239-31; *J. Phys. E.*, 1986, 19: 540-545; *J. Magn Reson.*, 1986, 72: 211-223. SMRM Fifth Annual Meeting, Abstracts Book, 1986, 455-6.
2. Roemer, P.B.; Edelstein, W.A.; and Hickey, J.S.: SMRM Fifth Annual Meeting, Abstracts Book, 1986, 1067-8.

Acknowledgements:

P. Roemer, W. Edelstein, and D. Elsner, GE corporate Research and Development Center

Fig. 1a.

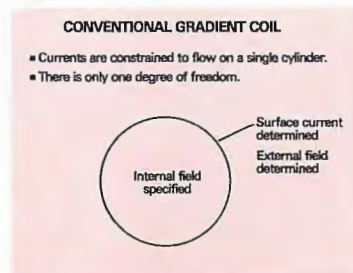


Fig. 1b.

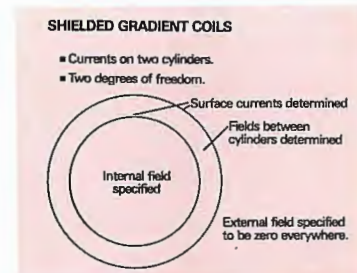


Fig. 1a—Design problem for unshielded gradients. The field inside the winding is specified to be a linear gradient and the current pattern on the cylinder is determined. **Fig. 1b**—Design arrangement for shielded gradients. The field inside the inner cylinder is specified to be a linear gradient and the field beyond the outer cylinder is specified to be close to zero. The current patterns on both inner and outer cylinders are then determined.

Fig. 2a.

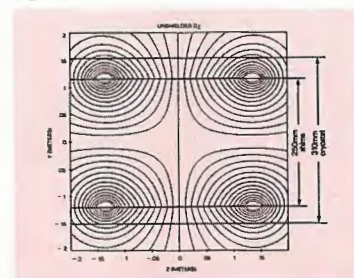
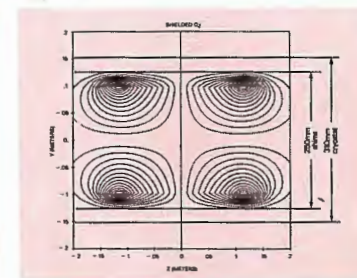


Fig. 2b.



Lines of constant flux for Z-gradient. Fig. 2a—Unshielded gradient. Note that flux lines extend well beyond the cryostat bore. **Fig. 2b**—Shielded gradient. Flux lines are kept within the outer gradient cylinder.

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Fig. 3a.



Fig. 3b.

SHIELDED GRADIENT PARAMETERS				
Gradient name	Gradient ID	Maximum RF coil ID	Gradient field at 70 A	Rise time to full current (70 A)
S150	6"	4.6"	10 G/cm	~100 μ s
S200	7.5"	6.25"	5 G/cm	~100 μ s

Fig. 3a—S200 shielded gradient set which produces 10 g/cm with 70 A and a rise time of 120 microseconds with an applied 120 V. Fig. 3b—Table showing some properties of two experimental shielded gradient designs.

Fig. 4a.

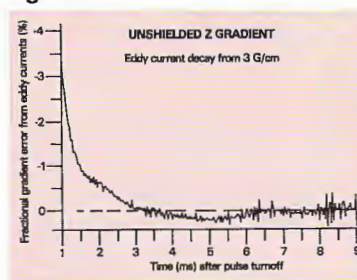


Fig. 4b.

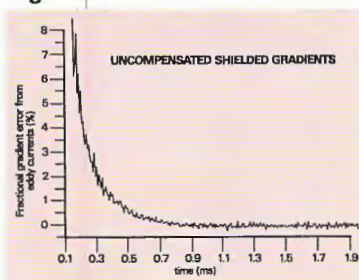
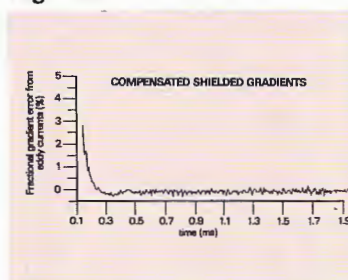


Fig. 4c.



Decay of field following application of square gradient pulse. Fig. 4a—Unshielded gradients. Fig. 4b—Shielded S150 gradient with no waveform compensation. Fig. 4c—Shielded S150 gradient with waveform compensation.

Note that the unshielded gradients started at 3 G/cm whereas the S150 gradient pulse had a strength of 9 G/cm.

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2.21 Hz. When sulfur replaces oxygen in the coupling path, one would expect the coupling transmission to increase, due to the decreased polarity. We conclude that the conformation about α_1 is considerably more gauche in the sulfur analogue.

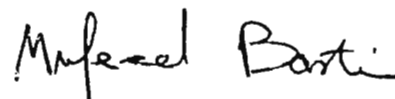
The $^3J(\text{POCH})$ coupling constant is 6.99 Hz in the oxygen analogue while $^3J(\text{PSCH}) = 14.72$ Hz in the sulfur analogue. The larger coupling constant for $^3J(\text{PSCH})$ reflects the expectation of a larger coupling through sulfur but it is also consistent with a greater percentage of gauche conformation about α_1 . Whether these conformational differences about α_1 are related to the different chemical and physical properties of the oxygen and sulfur analogue remains to be seen. A change in preferred conformation about α_1 will change the head group conformation relative to the acyl chains of the phospholipid.

Since Lankhorst's equations were derived primarily from oligoribonucleotide data, they cannot be used for these molecules, except in a qualitative manner. Other considerations are that the P-S-C bond angle is probably different from the P-O-C bond angle, and the bonds to sulfur will be longer than the bonds to oxygen; these factors will also affect the quantitative form of the Karplus equation. We would be interested in hearing from anyone who is developing Karplus-type relationships for phosphates or thiophosphates.

Sincerely,



Laurine A. LaPlanche



Mufeed M. Basti

- (1) P. P. Lankhorst, C. A. G. Haasnoot, C. Erkelens and C. Altona, J. Biomol. Struct. Dyn. 1, 1387 (1984).

Syracuse University has a STAR Technologies ST-100 100 MFLOP array processor for sale.

The 32-bit ST-100 is a high-performance array processor. It attaches to general-purpose computers for signal processing, image processing, simulation, geophysical applications, and general scientific computing. The ST-100 offered for sale is fully functional and eligible for hardware maintenance. It is equipped with 1.5 Megawords of 32 bit memory, an interface for a VAX Unibus and software for DEC VAX computer hosts, other interfaces are available from STAR. The ST-100 array processor software provides two levels of optimization. The Array Processor Control Language (APCL), a subset of ANSI FORTRAN 77 with language extensions, makes the ST-100 Array Processor easy to use and includes a large Application Support Library. Additionally, there is further optimization available from the Macro Assembly Language. The list price for this current-model system is over \$300,000. The ST-100 will be sold for the best offer received before September 1, 1988 (min. \$95,000). For further information contact either Prof. George C. Levy or Dr. Bill Curtiss, (315) 423-1021, Chemistry Department, Syracuse University, Syracuse, NY 13244-1200.

(received 6/15/88)

A multidimensional analysis of Low resolution pulsed NMR data for
the determination of ethanol in alcoholic beverages.

Using a Carr Purcell spin-echo sequence ($90^\circ x - t/2 - 180^\circ y - t/2 - \text{acquisition}$), the amplitude of the echo, $E(t)$, depends among others on the number of protons in the sample and on the modulations induced by coupling constants (J), that is to say on the chemical composition of the sample.

For an aqueous solution of ethanol or a sample containing a distillation residue (from wines or alcoholic beverages) and ethanol, we have measured the amplitude of the echo for different values of t .

Then, we applied two methods of multidimensional analysis on the data (27 different alcoholic solutions, 16 variables (t), and one more variable : the alcoholic degree ($\% \frac{V}{V}$)) :

- Using the multilinear regression methods, the first independent variable E (60 ms) allows to predict the alcoholic degree :

$$\text{alcoholic degree} = 113.45 - 34.2 E(60 \text{ ms}) \quad r = 0.96666$$

The residual standard deviation is 5.2.

If six independent variables are taken into account = E (60 ms), E (130 ms), E (0 ms), E (30 ms), E (70 ms) and E (20 ms), the residual standard deviation is 1.69 and the multiple correlation coefficient is 0.9972.

- Using the principal component regression (1), the results are improved.

The prediction equation obtained with two principal components (C_1 and C_3), for instance is :

$$\text{alcoholic degree} = 28.89 + 10.58 C_1 - 34.87 C_3 \quad r = 0.9934$$

With five components (C_1 , C_3 , C_5 , C_2 and C_4) it is improved again. On figure 2, we showed the predicted alcoholic degree versus the actual alcoholic degree

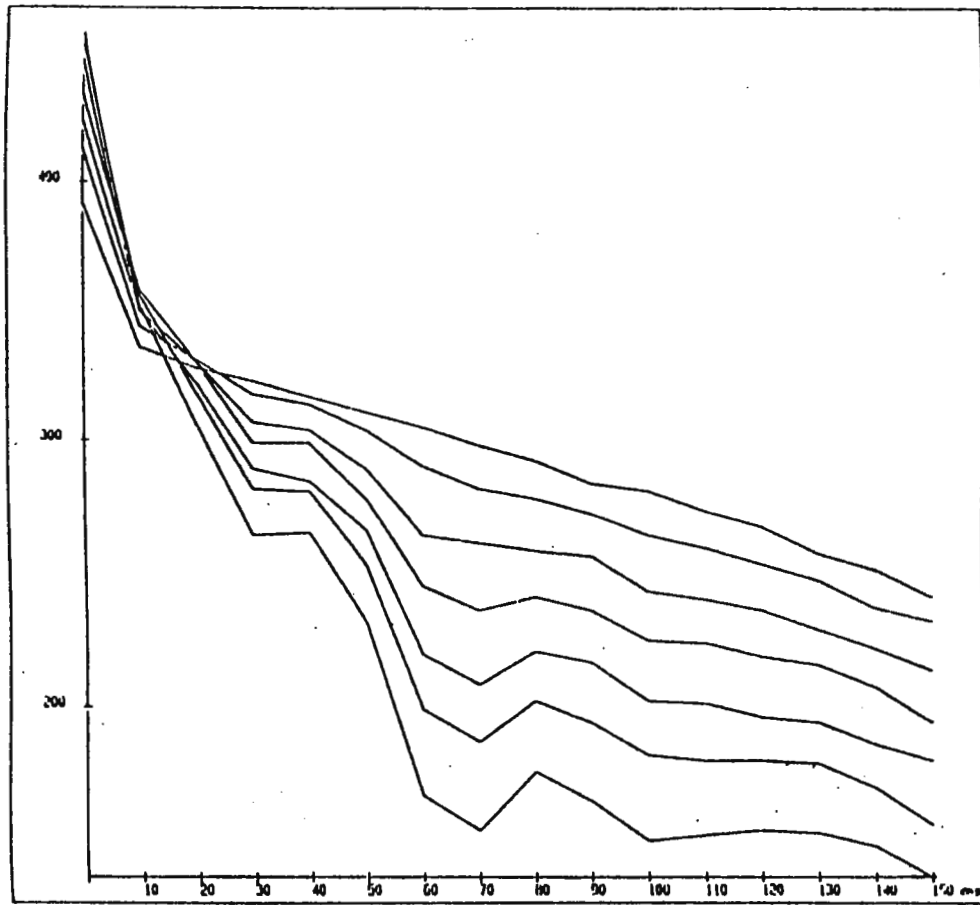


Figure 1 : a set of data from 7 samples giving the amplitude of the echo (X 10 mV) for different values of t (16 values from 0 to 150 ms)

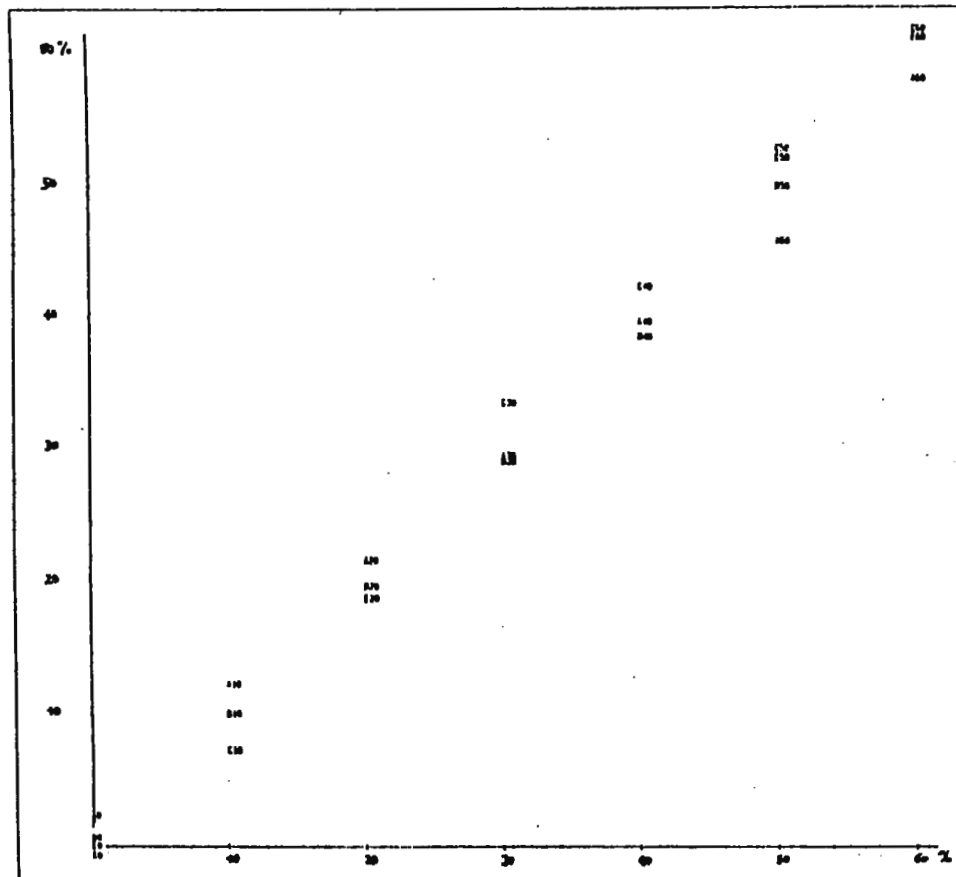



Figure 2 : the predicted values versus the actual values of alcoholic title (% v/v) for 23 samples


for 23 samples of alcoholic solutions.

These methods confirm the importance of the magnetisation at $t = 60$ ms which corresponds to the maximum of the J echo modulation of the echo at the refocusing time of $\frac{1}{2J} = 68$ ms.

We already used this modulation to determine the ethanol concentration in alcoholic beverages (2).


Dominique BERTRAND
(INRA)


Charles TELLIER


Marion GUILLOU-CHARPIN
(CEMAGREF)

Literature cited :

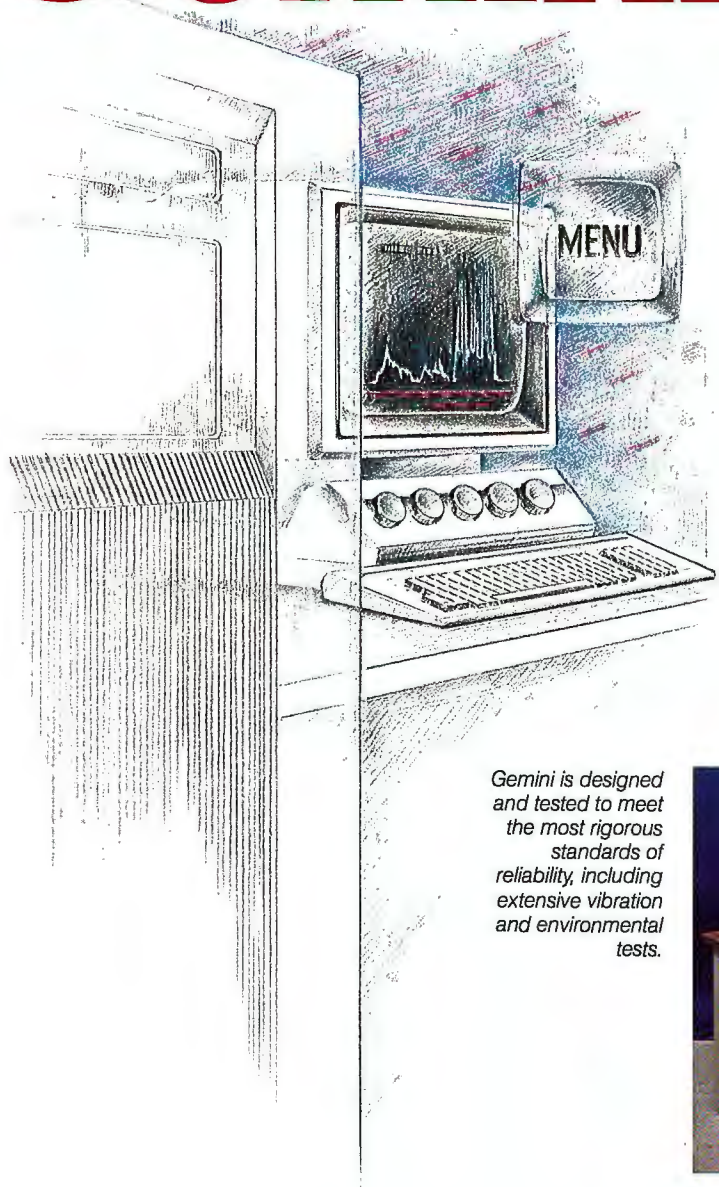
- (1) P. Dagnelie - Analyse statistique à plusieurs variables (Presses Agronomiques de Jemblaux - 1982)
- (2) M. Guillou - C. Tellier - Analytical Chemistry (in press).

NMR Spectroscopist at the Laboratory for Molecular Spectroscopy, School of Chemical Sciences, University of Illinois-Urbana. Requirements for this position include a PhD in chemistry, or extensive equivalent experience with state-of-the-art NMR spectroscopy; and the ability to work independently and collaboratively in a stimulating research environment. Experience in devising or implementing new FT-NMR experiments (e.g., 2D NMR, new pulse sequences), and a working knowledge of organic chemistry and/or biochemistry is also desired. Duties will include the operation of several high-field FT-NMR instruments including a GN500, the supervision of instrument operators, and the instruction of graduate students and postdoctoral fellows in NMR techniques. The successful applicant will have the opportunity to engage in NMR research independently or in collaboration with faculty of the School of Chemical Sciences.

The starting date is January 2, 1989 but other starting dates could be negotiated. This is a regular, full-time position. The starting salary is commensurate with experience. Submit resume and three letters of recommendation, preferably by October 14, 1988 to Dr. Vera V. Mainz, Department of Chemistry, Box 34 Noyes Laboratory, University of Illinois, 505 S. Mathews Ave., Urbana, IL 61801 (Tel: 217/244-0564). The University of Illinois is an affirmative action/equal opportunity employer.

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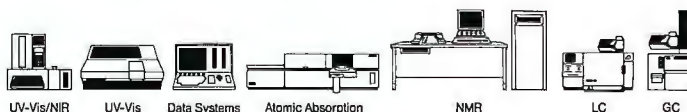
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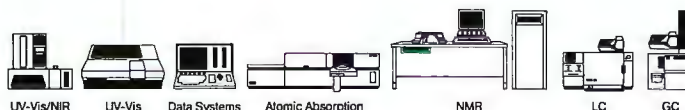
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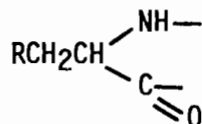
P.O. BOX 2000, RAHWAY, NEW JERSEY 07065-0900

June 15, 1988
(received 6/27/88)Professor B. L. Shapiro
Department of Chemistry
Texas A&M University
College Station, Texas 77843

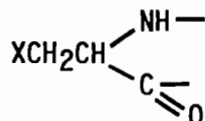
Dear Barry:

Semi-Broad Band Proton Homodecoupling in the 1D Mode

During the early stages of our peptide research at 300 MHz, the desirability of semi-broad band proton homodecoupling capability became apparent. Such a technique would simplify the primary task of peak assignments since it would provide us with a quick classification of residue types based upon the response of the α -hydrogens to irradiation blanketing the high field (ca 1-2 δ) and midfield (ca 2.5-3.5 δ) regions. α -hydrogens affected by the high field irradiation belong to the residue type:



where R represents one or more Sp^3 carbons. α -protons which are decoupled when the 2.5-3.5 δ region is irradiated belong to the class:



where X represents an Sp^2 carbon or sulfur. α -hydrogens unresponsive in both experiments limit the possibilities to serine, glycine and threonine.

In addition, irradiating the α -hydrogen region would simplify the midfield and high field regions by removing all α,β couplings. This would be of particular value in sorting out individual signals and geminal coupling constants in the usually congested 3 δ region. Although the methodology to be described was designed for peptides it, of course, has general applicability.

-2-

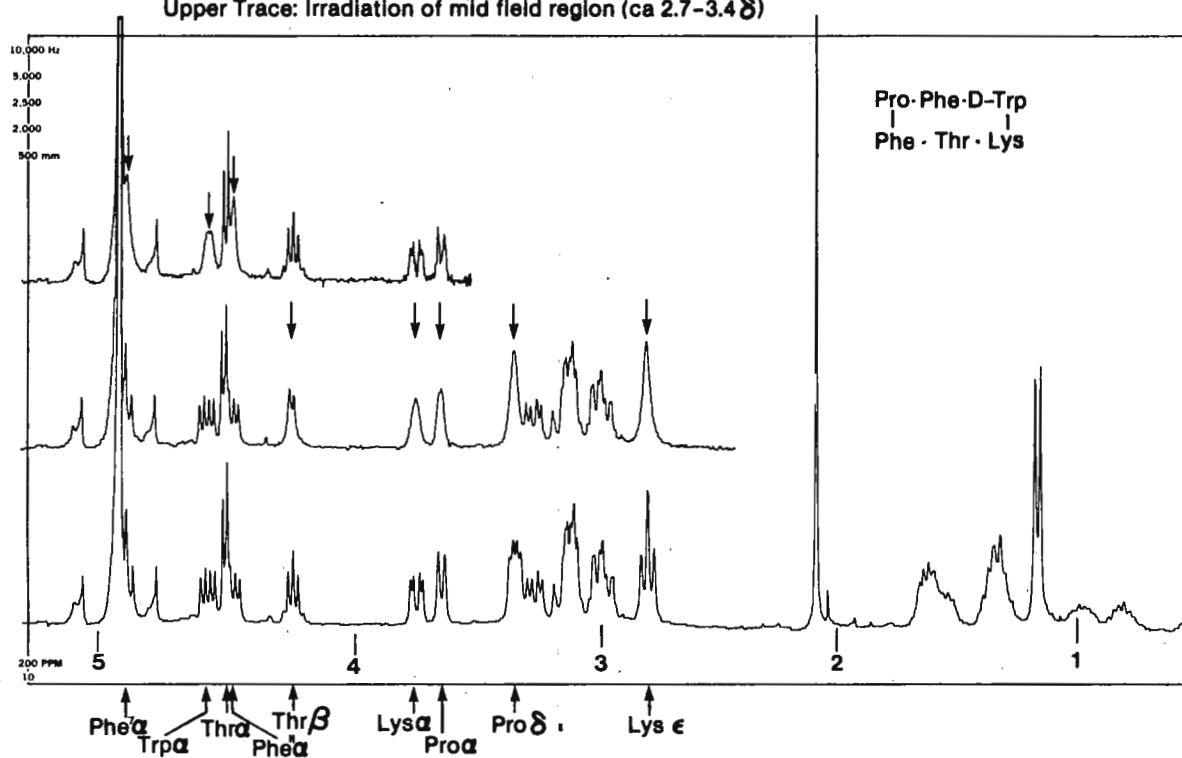
The utility of the semi-broad band techniques as a diagnostic aid is illustrated in figure 1 with one of our cyclic hexapeptides in the somatostatin series. The normal spectrum of the aliphatic region, aside from the lysine γ -CH₂ multiplets at 0.55 δ and 0.40 δ , is shown in the bottom trace. Irradiating the region between 0.5-1.5 δ (middle trace) identifies all protons having high field neighbors which includes in the present example Lys α H, Pro α H, and Thr β H as well as Lys ϵ -CH₂ and Pro δ -CH₂. Irradiating the 2.7-3.4 δ region identifies the α -protons of the aromatic residues. Note that the Thr α H is unaffected in both experiments.

L-363,301: Semi Broad Band Decoupling

Bottom Trace: Normal spectrum

Middle Trace: Irradiation of high field region (ca 0.5-1.5 δ)

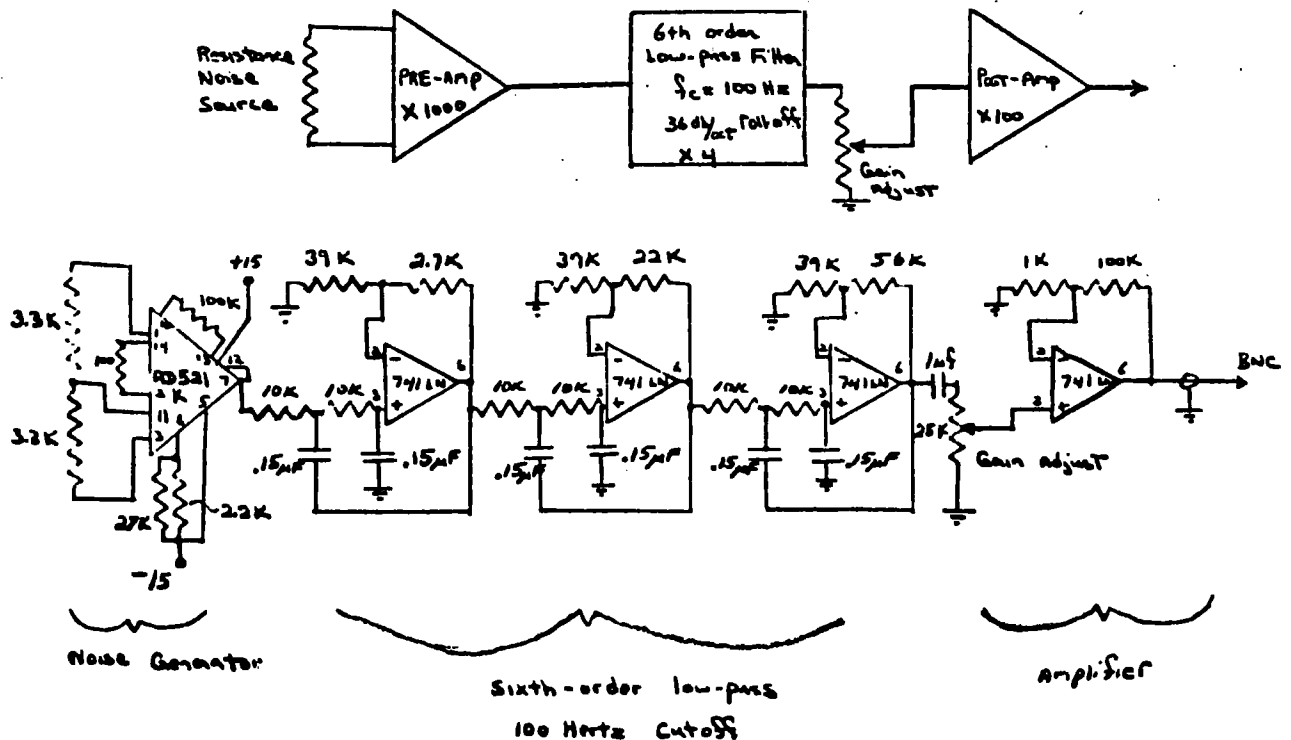
Upper Trace: Irradiation of mid field region (ca 2.7-3.4 δ)



-3-

Shown below is a schematic of the decoupler modulator. When used with our Varian SC-300 spectrometer, the output is connected to the phase modulator jack of the decoupler frequency synthesizer and produces a noise window approximately 200 Hz wide around the set decouple frequency.

When used with our XL-400, the output was connected to J3103 on the offset synthesizer and DMM set to E. In this case the window was about 100 Hz wide.



Sincerely,

Byron H. Arison
Animal Drug Metabolism

Byronne Arison

Herman Flynn
Animal Drug Metabolism

Herman Flynn

Gary S. Kath
Biophysics

Gay S Keth

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

(received 6/27/88)

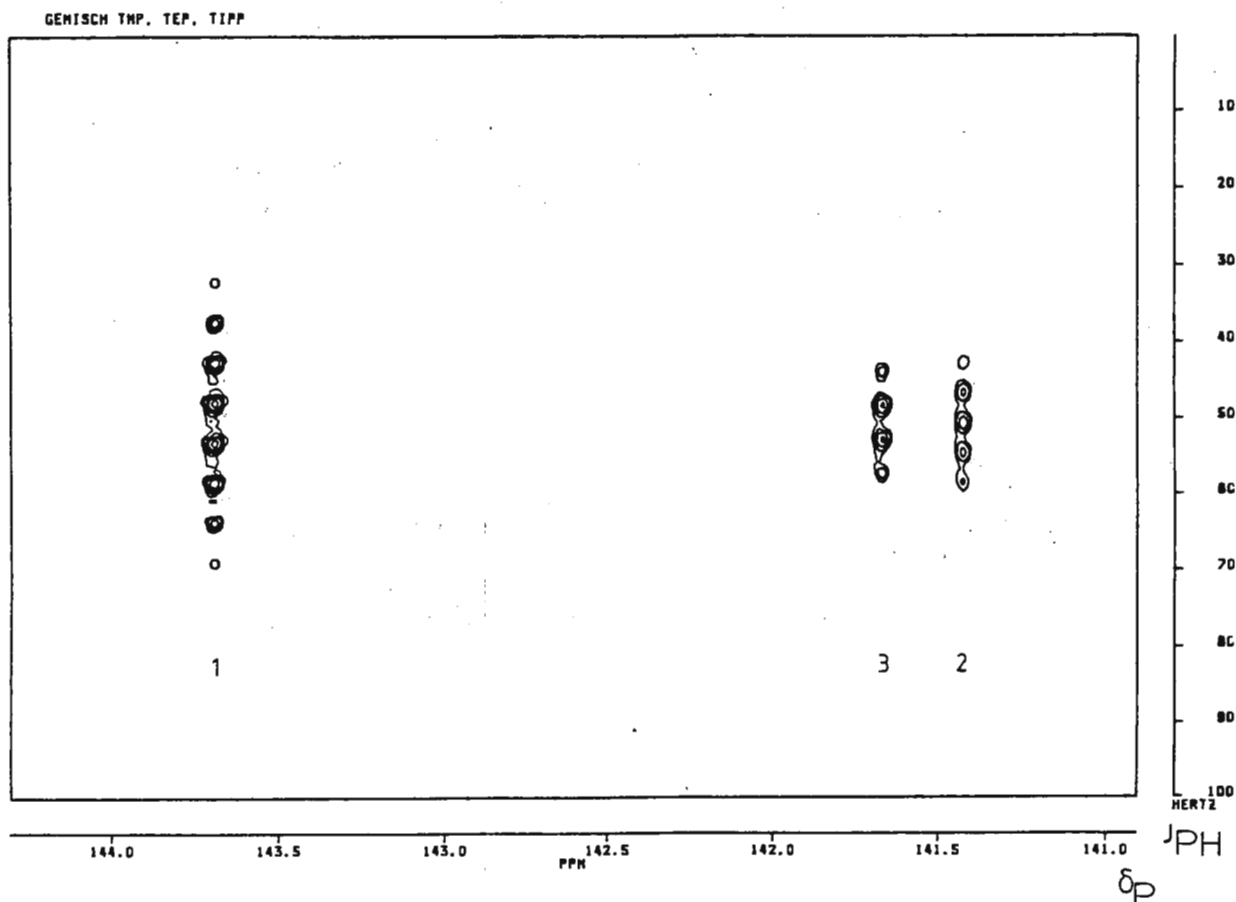
Title: P,H-J-resolved spectra

Dear Professor Shapiro!

I am on sabbatical leave in summer term 1988 and will be back in Düsseldorf at the beginning of September. This letter is a brief message to cover your reminder, which requests "practical contributions". M. Batz and several others from our group are looking into the analysis of reaction mixtures from synthetic work and into rotameric equilibria of phosphonic and phosphinic acids. Presently we are setting up the necessary facilities for P,C(H)-COSY technique after C. Brevard and A. Pagelot (not necessary to say: BRUKER SPECTROSPIN, Wissembourg) made excellent spectra from our compounds. We wish to acknowledge these studies prior to publication. Our routine instruments used for the analysis of mixtures and molecular structures are H,H-, H,H(P)-, C,H-, P,H- and P,P(H)-COSY spectra. Very recently we tried our hands on H,P-J-resolved spectra which might be helpful in certain situations yielding the number of protons coupling to phosphorus. An example is given for the analysis of an equimolar mixture of trialkylphosphites (RO)₃P: 1: R=CH₃, 2: R=C₂H₅, 3: R=iC₃H₇. A figure is attached showing the results from experiment done on our BRUKER AM200 spectrometer. Details are available on request.

Yours sincerely

Gerhard Hägele Michael Batz



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Advanced enhancement

1

Versatile Image Display



The versatility of NMRi's SpecStation™ environment provides flexible screen configurations to optimize display of intermediate processing results. Mouse-driven real-time inspection tools are supported to allow gray level manipulations which increase contrast and outline features of interest. The lower set of images at the left illustrates the effect of selective intensity range display, which highlights structures of uniform brightness. An example of gamma-curve remapping, which increases overall brightness, is applied to the rat torso image shown below.



2

Enhancing Structural Definition

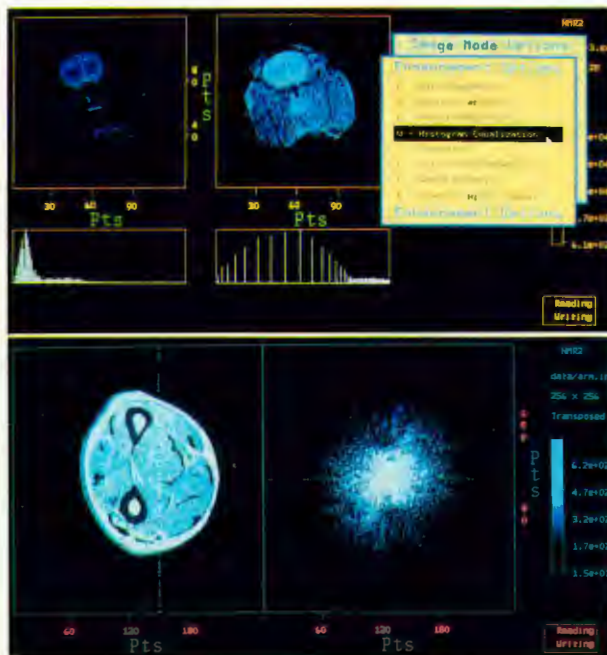


IMAGE includes a variety of contrast enhancement techniques to allow better perception of fine details and subtle changes in brightness. Options include:

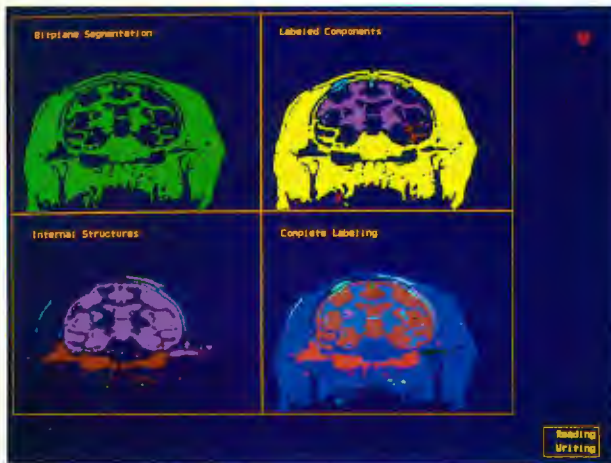
- Contour Extraction
- Region Emphasis
- S/N Ratio Improvement
- Adaptive Filtering
- Gray Level Morphology
- User-Defined Convolution Kernels
- Fast 2D Fourier Processing
- Histogram Equalization

The upper pair of images illustrates the source and result of contrast enhancement via histogram equalization. The lower pair shows a source image with its corresponding power spectrum; the vertical artifact in the original image is revealed as a horizontal structure in the power spectrum. Frequency processing of the power spectrum can effectively remove the artifact in the original image.

Advanced techniques for processing and measuring MR images.

3

Counting and Measuring Binary Patterns



An automated segmentation procedure based on the analysis of the intensities in a monkey brain image has produced the binary patterns shown here. A labeling procedure can be used to identify each connected component. A binary editing capability allows the user to point to the labeled structures and extract characteristic region parameters such as:

- Area, Perimeter
- Circular Shape Factor
- Length, Width
- Center of Gravity
- Fractal Dimension

4

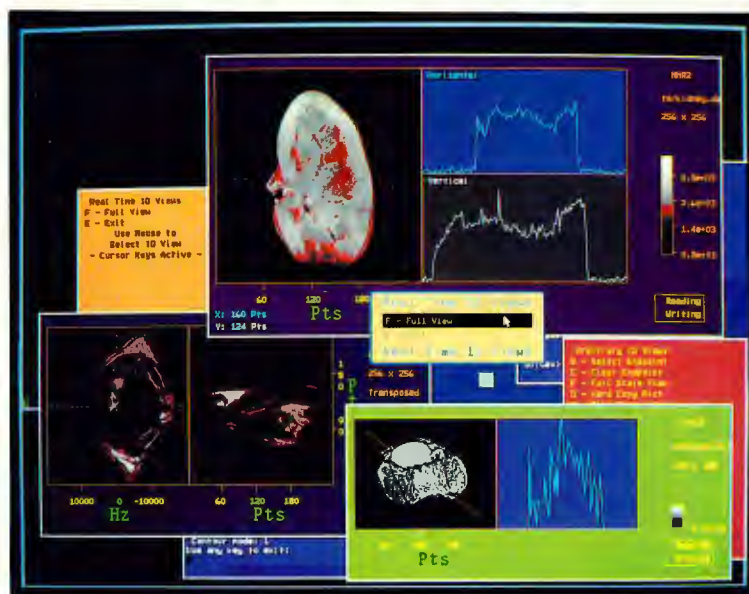
Advanced Restoration Methods



Several deconvolution techniques, based on the maximum entropy and other criteria are provided. Here, the original, leftmost images are enhanced through Gaussian deconvolution, to yield results with improved structural definition drawn in the right half of the display. Other enhancement techniques are available, including Principal Component Analysis for both noise reduction and composite filtering of images and 2D NMR spectra.

5

Gray Value Measurement



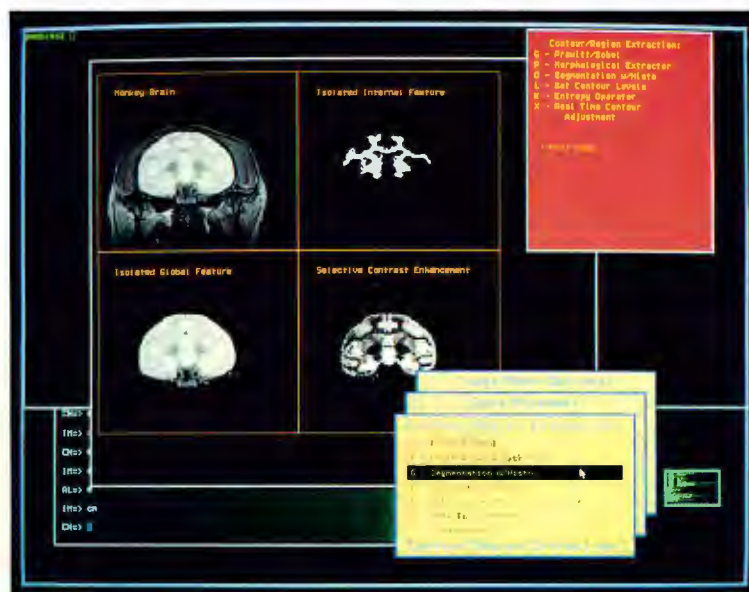
Real-time inspection and analysis of image intensities includes extraction and display of horizontal, vertical and arbitrarily oriented slices. Slices are selected graphically via mouse driven crosshairs. In addition, mouse-activated pop-up menus allow for fast and efficient use of these real-time tools as well as easy operation of all IMAGE facilities.

IMAGE measurement tools provide:

- Mean, Variance Measurement
- Histogram Analysis by Shape Modeling
- S/N Ratio Estimation
- Contrast Measurement
- Pixel Intensity Reporting
- Cross-sectional 1D Display
- Correlation Measurement

6

Region of Interest Selection and Processing



Powerful adaptive algorithms utilize regional processing to take advantage of local stationarity of image features. In this manner, filters can be automatically adjusted to yield optimal results for the areas of interest. In this example, the central surface of a monkey brain image has been selected and isolated for local contrast enhancement. Free form contour tracing via the mouse is also supported for interactive definition of regions of interest.

The LAB ONE™ NMR2/IMAGE program is an advanced, high performance research system that runs on UNIX/VMS workstations.

For further information contact:

nMRI:

New Methods Research, Inc.

719 East Genesee Street
Syracuse, New York 13210
(315) 424-0329 FAX: (315) 424-0356

Acknowledgements and Trademarks

Image data courtesy of S.U.N.Y. Upstate Medical Center at Syracuse, and GE NMR Instruments.

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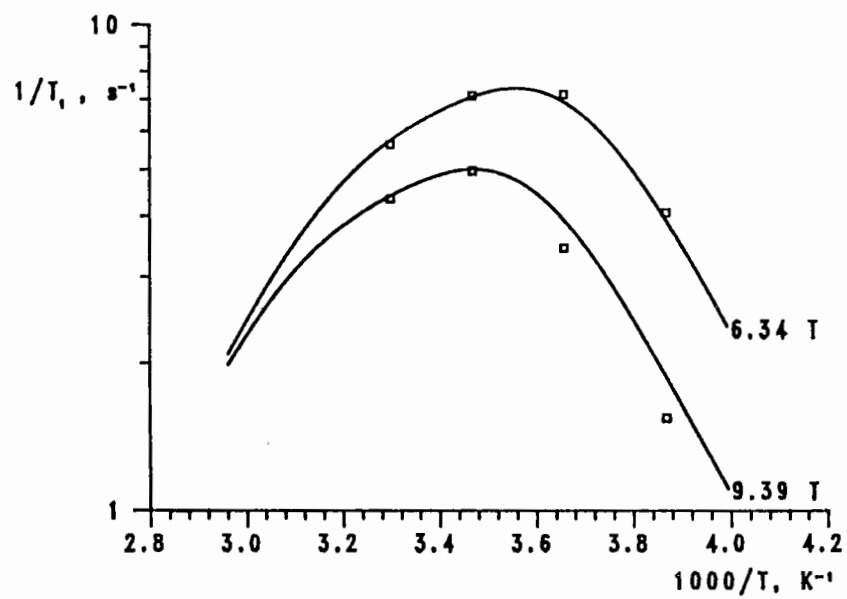


Fig. 1

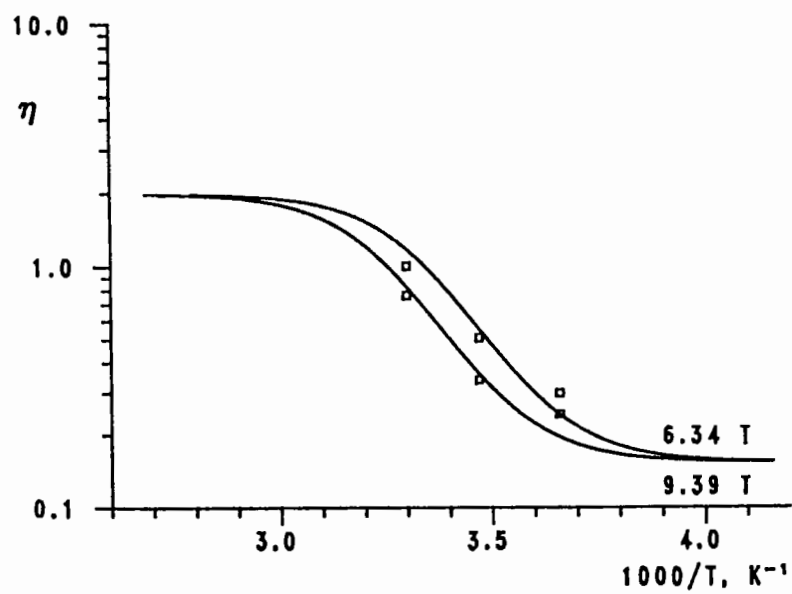


Fig. 2

**Universität Bern**

Institut für organische Chemie

CH-3012 Bern, Freiestrasse 3
Telefon 031 65431121.6.1988
(received 6/25/88)

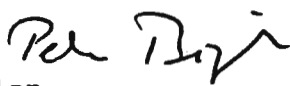
Dr. Bernard L. Shapiro
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto, CA 94303

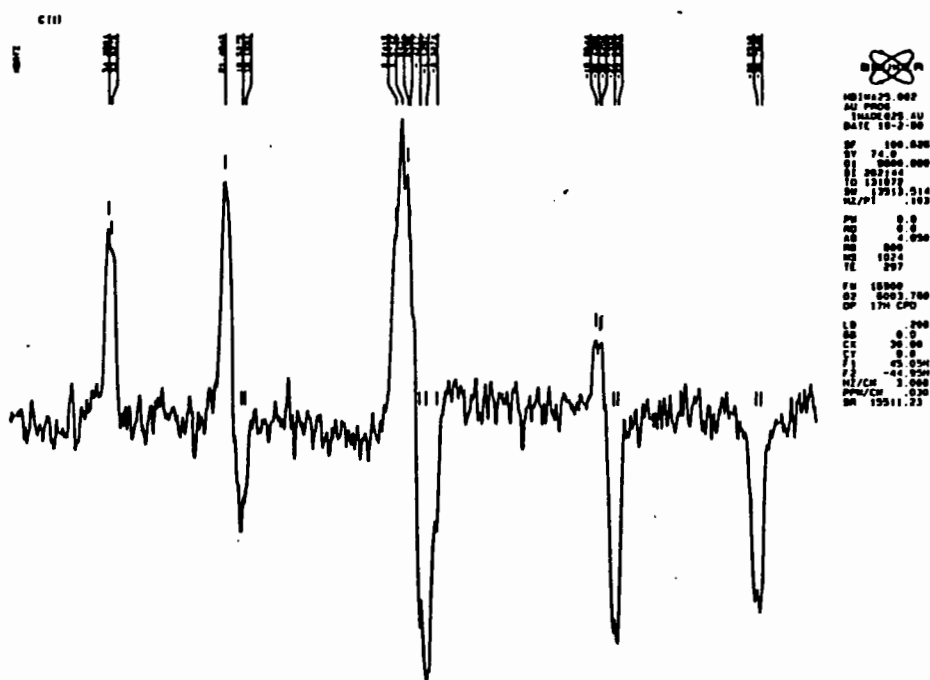
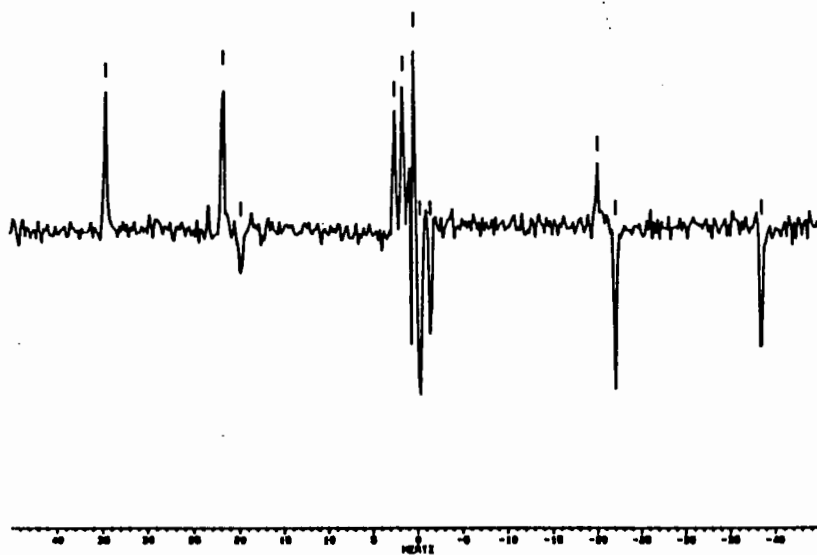
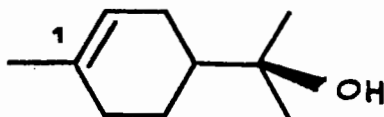
Dear Dr. Shapiro

Problems with the 1D INADEQUATE experiment

Interested in the evaluation of $^1J_{CC}$ - $^nJ_{CC}$ -data of terpenes we applied the 1D INADEQUATE pulsesequence. The unexpected broad lines and, more suspicious the strange doubling of most of the signals pointed to some instabilities either in the spectrometer or in the environmental conditions. Since the broadening is also highly dependent on the signal under investigation the sample itself could also be responsible for these unwanted effects (temperature dependent chemical shifts). We therefore applied the same pulsesequence again, but stored the data blockwise. Fourier transformation gave excellent results for all the blocks, with all the long range couplings resolved. By adding the separately processed spectra again a very poor result was achieved (Fig. 1a). On the dual display of our BRUKER system we noticed, that each of these spectra was shifted against the other by up to eight data points. Fig. 1b shows the result after addition of the individually shifted subspectra. No trends could be seen in going from the first to the last data block or in going from signals close to the carrier to more remote signals. The effects are highly signal specific. Fig. 1 shows the signal of carbon C-1 of α -Terpineol.

Sincerely


Peter Bigler



UNIVERSITÉ DE L'ÉTAT À MONS

FACULTÉ DE MÉDECINE
Avenue du Champ de Mars, 24

7000 MONS (Belgique)

Tél. 085/37.31.11.

Department of Organic Chemistry
and NMR Laboratory.

Two optimized DIGGER RF pulse shapes.

May 29, 1988. (received 6/9/88)

Dear Professor Shapiro,

The problem of tailoring RF pulses for selective excitations - well perceived by the NMR community - is particularly critical in imaging and localized spectroscopy. The latter discipline is indeed very demanding with respect to selectivity but offers very little since RF homogeneity and power are often quite limited.

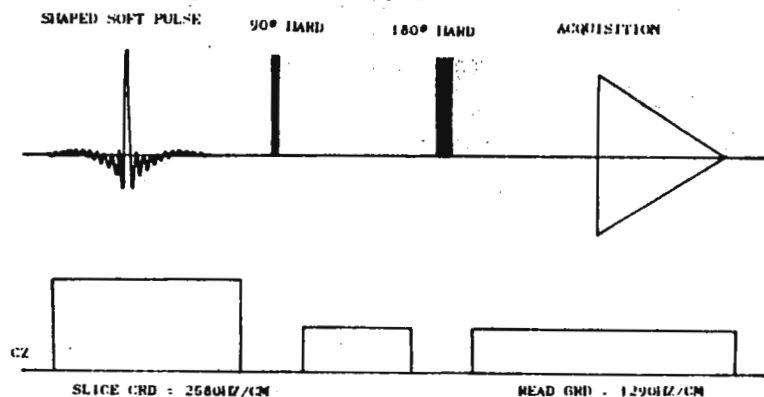
We have tested several approaches of this difficult problem of pulse shaping and we want present here some typical results of two optimization procedures applied to the DIGGER pulse sequence.

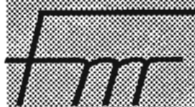
The first method (method A), proposed by Conolly and coll (1) gradually improves the RF pulse envelope by an iterative minimization of the difference between the ideal and the actual profiles. The second procedure (method B) is derived from the work published by Ngo and coll (2). It performs successive corrections by adding to the pulse shape the inverse FT of the difference between the desired and obtained profiles. Calculations by these two methods were performed with FORTRAN 77 routines on a IBM 9370 system.

Figures 1a, 1b, 2a and 2b show the shapes of the optimized RF pulses and the theoretical responses of the spin system calculated by the resolution of the Bloch equations. According to this theoretical approach, method B seems to be more efficient. It is worthwhile to mention that this method is also much faster (about one hour of cpu for 1000 calculated points vs approximately seven hours for 300 points with method A).

We experimentally checked the results of our simulations on a Bruker Biospec 24/40 system equipped with a 400 watts Intech RF amplifier and a 15 cm diameter saddle shape coil.

A water-filled cubic container (5 cm side) has been used as phantom *. The Fourier transform of the FIDs collected with the sequence shown hereunder and using the appropriate RF shapes gives the corresponding experimental slice profiles (figures 1c and 2c).





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Customized Spectrometer Analysis

If you are having problems getting good results with some of your NMR experiments, you would benefit from having a site visit to provide a detailed examination of your overall instrument performance. The nature of the analysis is customized to the needs of the individual researcher and can locate "weak links" in the spectrometer's performance. A systematic instrumental analysis can answer questions like:

- **Low signal to noise.** Is it Probe? Preamp? Observe receiver? Interference?
- **Poor 2DFT Performance.** Is it due to poor RF phase or amplitude stability? Floor vibrations? Sample temperature instability?
- **Poor Water Saturation.** Is it due to very low order lineshape? Poor RF homogeneity? Probe limitations?

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Nicolet NT Spectrometer Top Entry Probes

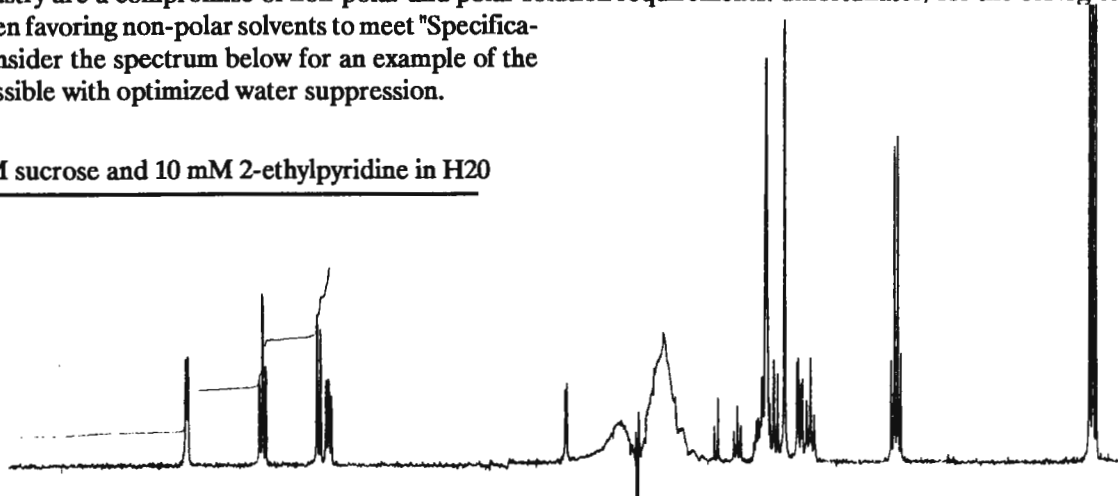
New Probe technology is available in top entry probes for the Nicolet NT Series spectrometers with much improved sensitivity. Ask for a quote for your specific needs. Some examples are:

NT 360 WB
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5mm ^1H { ^{200}P :1 0.1% Ethyl Benzene
 ^{31}P -2H } Reverse Detection Probe
175:1 0.1% Ethyl Benzene
12mm HB Probe
 ^{13}C 300:1 ASTM
 ^{31}P 300:1 1% TMPO

Biological Probes

When running typical biological samples, NMR probes need to be optimized differently than for non-polar organic solutions such as Ethyl Benzene. All probes intended for biological use (such as ^1H , ^{31}P and ^{13}C) need to be able to tune and match even with strongly ionic buffer solutions. ^1H probes must be able to deliver very low order lineshapes which allow water saturation experiments at decoupling power levels consistent with the observation of close in peaks. Typical probes in the industry are a compromise of non-polar and polar solution requirements, unfortunately for the biological spectroscopist, often favoring non-polar solvents to meet "Specifications". Consider the spectrum below for an example of the results possible with optimized water suppression.

10 mM sucrose and 10 mM 2-ethylpyridine in H₂O



5 and 10mm Reverse Detection probes

It is relatively straightforward to provide decent ^1H sensitivity in a RPT probe; the trick is to also give x-nucleus γH_3 values large enough so that the entire spectrum can be acquired in a single experiment. Our experience enables us to achieve the highest possible γH_3 without sacrificing overall sensitivity and lineshape. X-nucleus ranges of ^{31}P - ^{15}N , or ^{13}C - ^{15}N are available. Examples at 500MHz include:

sample	^1H EB sens	^1H p(90)	γH_3 (^{15}N)	γH_3 (^{13}C)
5 mm	350	9 μs	2kHz@10W	5kHz@10W
10 mm	600	18 μs	1kHz@10W	3kHz@10W

5,10,12,15,20mm Broadband probes

Tuning ranges of ^{31}P - ^{15}N and ^{15}N - ^{109}Ag are available. Other ranges are custom designs.

Surface Coils

The simple approach to surface coil design produces a circuit with excessive sensitivity to sample loading. Often it is not possible to match to all sorts of loads, leading to inefficient data collection. FMR can produce surface coils with much greater tolerance to different samples, either with adjustable tune and match for the greatest sensitivity, or with no adjustable components for the greatest ease of use. These latter surface coils may also be ordered in a hermetic design that may be autoclaved.

Imaging coils

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Single frequencies from 20 to 500 MHz in sizes from 1 cm to 30 cm. Examples are:

Size	Frequency	p(180)	power
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30 cm	85MHz	400	1 kW

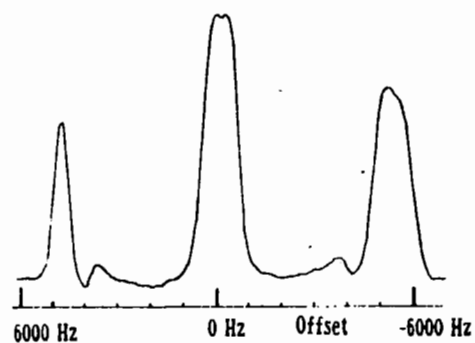
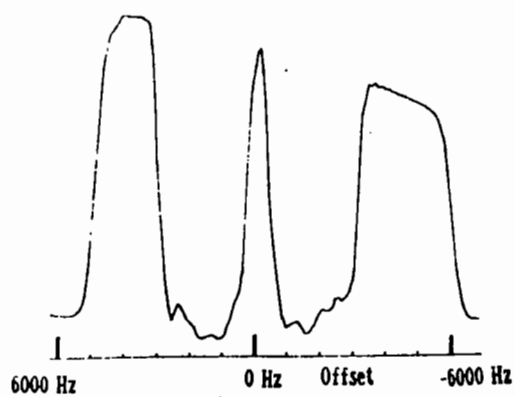
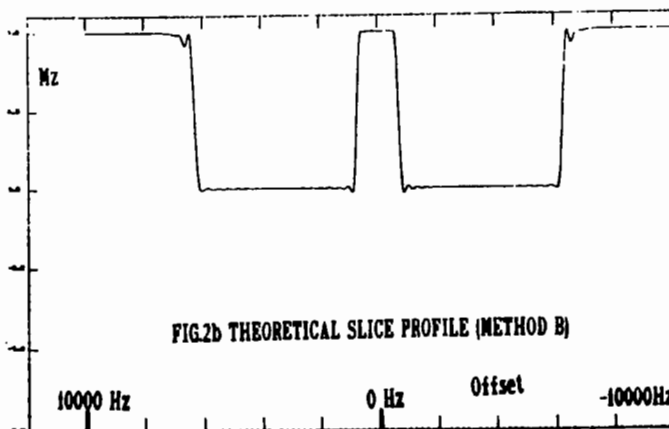
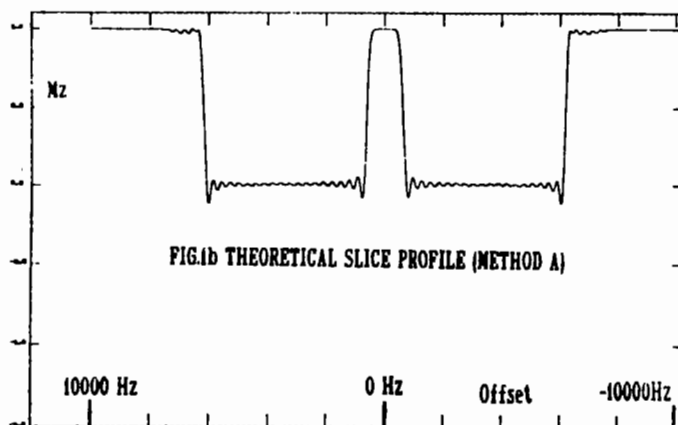
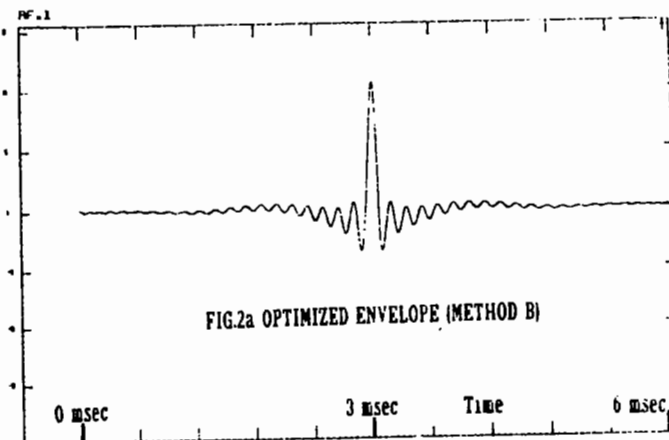
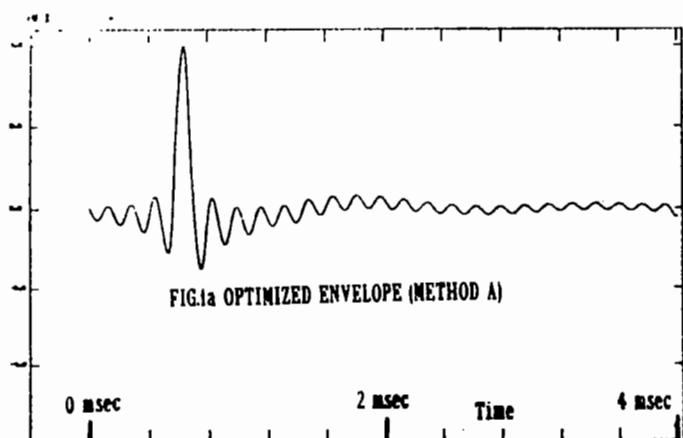


FIG.1c EXPERIMENTAL SLICE PROFILE (METHOD A)

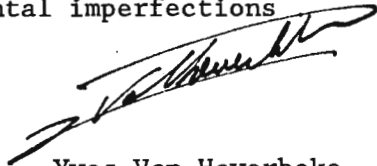
FIG.2c EXPERIMENTAL SLICE PROFILE (METHOD B)

consequence of a decrease of the frequency bandwidths. Although the quality of the experimental profiles is not as good as the calculated ones, they confirm the better efficiency of the RF profile optimized by method B. It is fair to mention that this type of shaping requires about 30% more power. Differences between calculated and observed profiles are to be attributed to hardware imperfections the consequences of which seem definitely larger than those coming from the non-linearity of the Bloch equations.

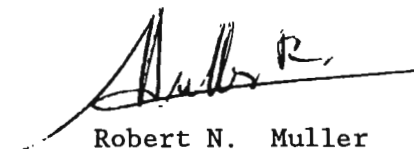
Further work must therefore take into account and try to compensate these instrumental imperfections



Alain Roch



Yves Van Haverbeke



Robert N. Muller

* Since our MSL 200-15 system is not yet equipped with the "5th channel", the experiments have been performed at the Bruker factory in Karlsruhe with the invaluable help of Drs Hoepfel and Kreibich to whom we are very grateful.

1) S. Conolly, D. Nishimura and A. Macovski, IEEE Trans. Med. Imaging, vol. MI-5, 106 (1986).

2) J. Ngo and P. G. Morris, Magn. Reson. Med., 5, 217 (1987).

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Professor B.L. Shapiro
TAMU NMR Newsletter
966 Elsinore Ct.
Palo Alto, CA 94303

(received 6/13/88)

Dear Barry:

When one encounters temperature dependence in ^{31}P NMR spectra of ligands on a metal cluster, one naturally supposes some fluxional process is operative, as is the case in several systems we have looked at in recent years. We report here an interesting alternative, which is simply that the chemically different ^{31}P nuclei in the compound have different temperature coefficients of their chemical shifts.

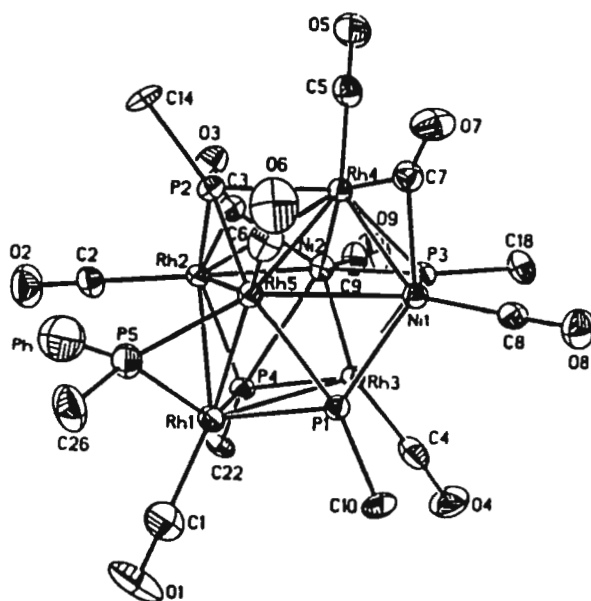
The solid state structure of **1** shows one μ_2 -phosphido (PHBu^t) and four phosphinidene ligands (PBu^t). Three phosphinidene ligands bridge four metal nuclei (μ_4), and one is triply-bridging (μ_3). The ^{31}P spectrum at 303 K shows only a single multiplet, at δ +572.6 ppm, which shows ^{31}P - ^{31}P and ^{31}P - ^{103}Rh couplings. Upon cooling, this multiplet separates without apparent broadening, into two multiplets in a 3:1 ratio. The resonance for the μ_3 -phosphinidene ligand moves upfield (~ 6 ppm) upon cooling from 273 to 203 K) with a temperature coefficient of ~ 0.085 ppm/K, while the resonance for the μ_4 -phosphinidene ligands shifts upfield by only ~ 1.8 ppm (~ 0.026 ppm/K) over the same temperature range. Unfortunately, the solvent of choice for this metal-cluster (CD_2Cl_2) prohibits us from warming above 323 K to see if the μ_3 resonance could be moved downfield from the μ_4 resonance.

It was mentioned above that the separation of resonances takes place without apparent broadening. Since the lines are intrinsically broad (5-20 Hz), it was difficult to be sure that no broadening had taken place. Moreover, since any fluxional process would probably be intramolecular, one would expect retention of spin-spin couplings. Consequently, it was not clear to us that some dynamic process was not occurring. To answer that question, we have repeated the temperature dependence study at two field strengths (8.5 and 11.7 T); a true exchange process should, of course, have a higher coalescence temperature at higher field. However, we have determined that this process is field independent as spectra taken at the same temperature at either field strength are superimposable from 203 to 323 K. We conclude that the cause of the separation of the peaks is as stated above, i.e., different thermal coefficients of the chemical shift, and not a dynamic process.

Please credit this note to the account of Tom Farrar.

B.A. Adams
R.E. DesEnfants

Bruce Adams
RE DesEnfants



$\text{Rh}_5\text{Ni}_2(\text{CO})_9(\mu_4\text{-PBu}^\pm)_3(\mu_3\text{-PBu}^\pm)(\mu_2\text{-PHBu}^\pm)$, I. (The tert-butyl methyl substituents and all hydrogens have been omitted for clarity.)

Newsletter Subscription Renewals

Once again, you are respectfully reminded (see Newsletter No. 356. p. 75) that effective for the October 1988 - September 1989 Newsletter year, the annual subscription rate for the TAMU NMR Newsletter will be US\$120.00. The new rate applies to all new and renewed subscriptions. The 50% academic/personal discounts will continue to be applicable. The new rate will continue to include mailing at Printed Matter (Third Class) rates. First class, Air Mail, or other special posting services are available, and I will be happy to provide firm quotations on request.

Invoices for the 1988-89 Newsletter year will be mailed out on July 7 to all subscribers other than those of Sponsor organizations and Advertisers. If you have not received an invoice by the time you read this notice, please let me know without delay. It will be greatly appreciated if you will see that these invoices are processed promptly, in order that the costs of operating the Newsletter can be 'contained'. Fiscal considerations require that subscription payment must be received (or clear indication given me that payment is in the works) by October 1, 1988 if your Newsletter mailings are to continue without interruption.

B.L.S.
1 July 1988

Policies and Practical Considerations

(Revised July 1988)

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 about to complete its thirtieth 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 is currently \$120.00 per year, with a 50% academic or personal discount being available. 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 quite essential, source of funds for the Newsletter is Advertising. We earnestly encourage present and potential participants of the Newsletter to seek advertising from their company. 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. There must be 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).

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. You 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.*

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.

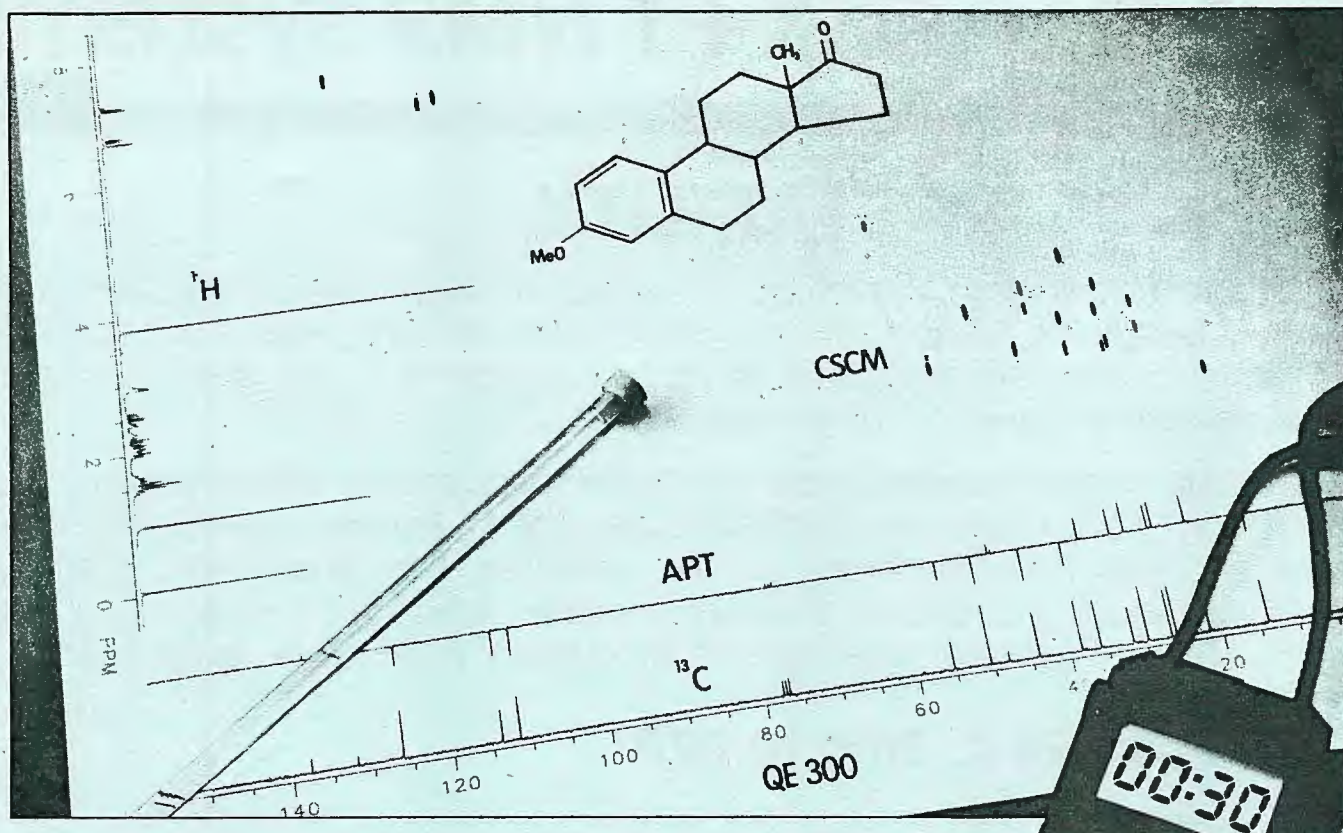
6. Suggestions: They are always welcome.


B. L. Shapiro

Address for all correspondence:

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TAMU NMR Newsletter
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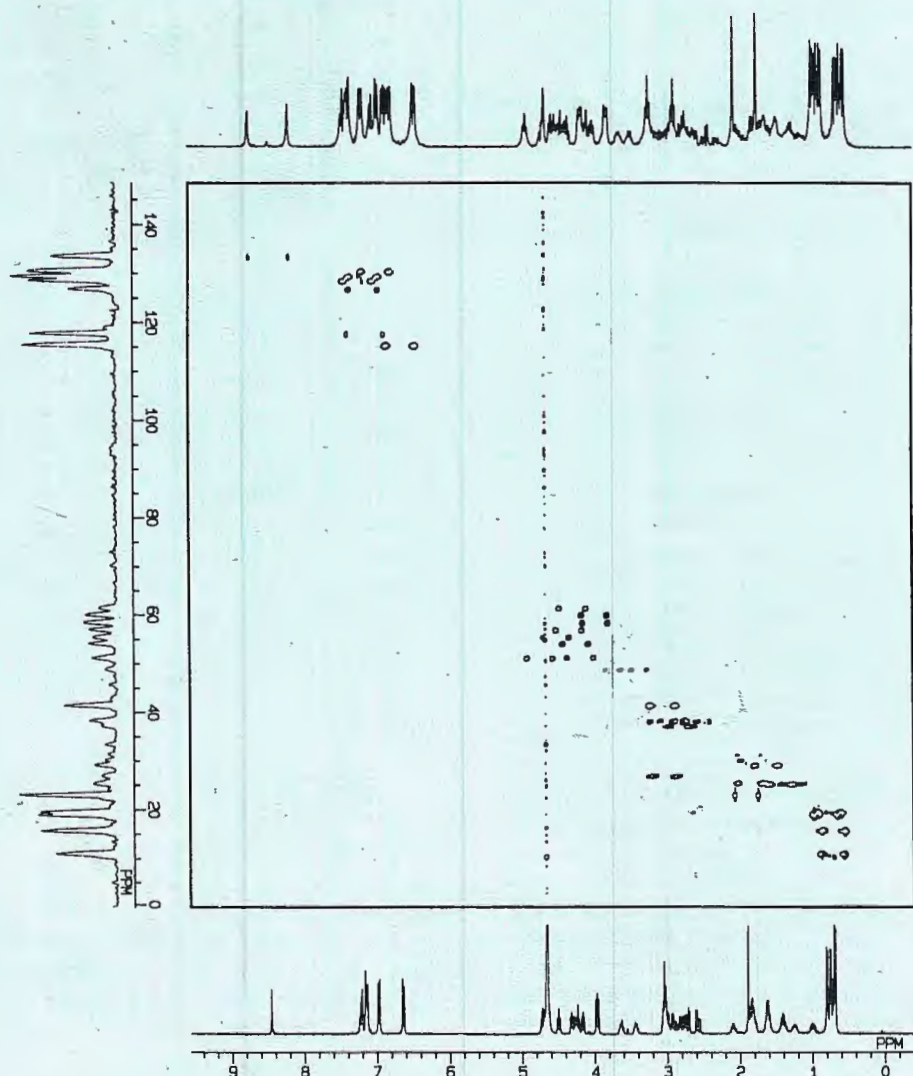
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