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NMR NEWSLETTER

NO. 304

JANUARY 1984

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Should be Addressed to:

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

AUTHOR INDEX ------ TAMU NMR NEWSLETTER NO. 304, JANUARY 1984

DEADLINE DATES

No. 305 ------ 6 February 1984

No. 306 --------- 5 March 1984

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

DQ and COSY of a 22K Protein

Dear Prof. Shapiro,

your final ultimatum letter reached me again in the middle of a well deserved rest. In response, I am going to bore your readers with some 2D-NMR data.

The accompanying figure shows in the upper part a double quantum proton NMR spectrum of the aromatic part of the porcine adenylate kinase. The standard INADEQUATE pulse sequence and phase cycling scheme with superimposed CYCLOPS phasing and quadrature detection in both dimensions was used, the DQ diagonal runs from lower right to upper left. The connectivities found in this spectrum can also be seen in the COSY-spectrum (middle), which was aquired with the standard Jeener pulse sequence with CYCLOPS phase cycling.

In addition, a few more connectivities can clearly be resolved in the COSY-spectrum, resulting in a total of nine.

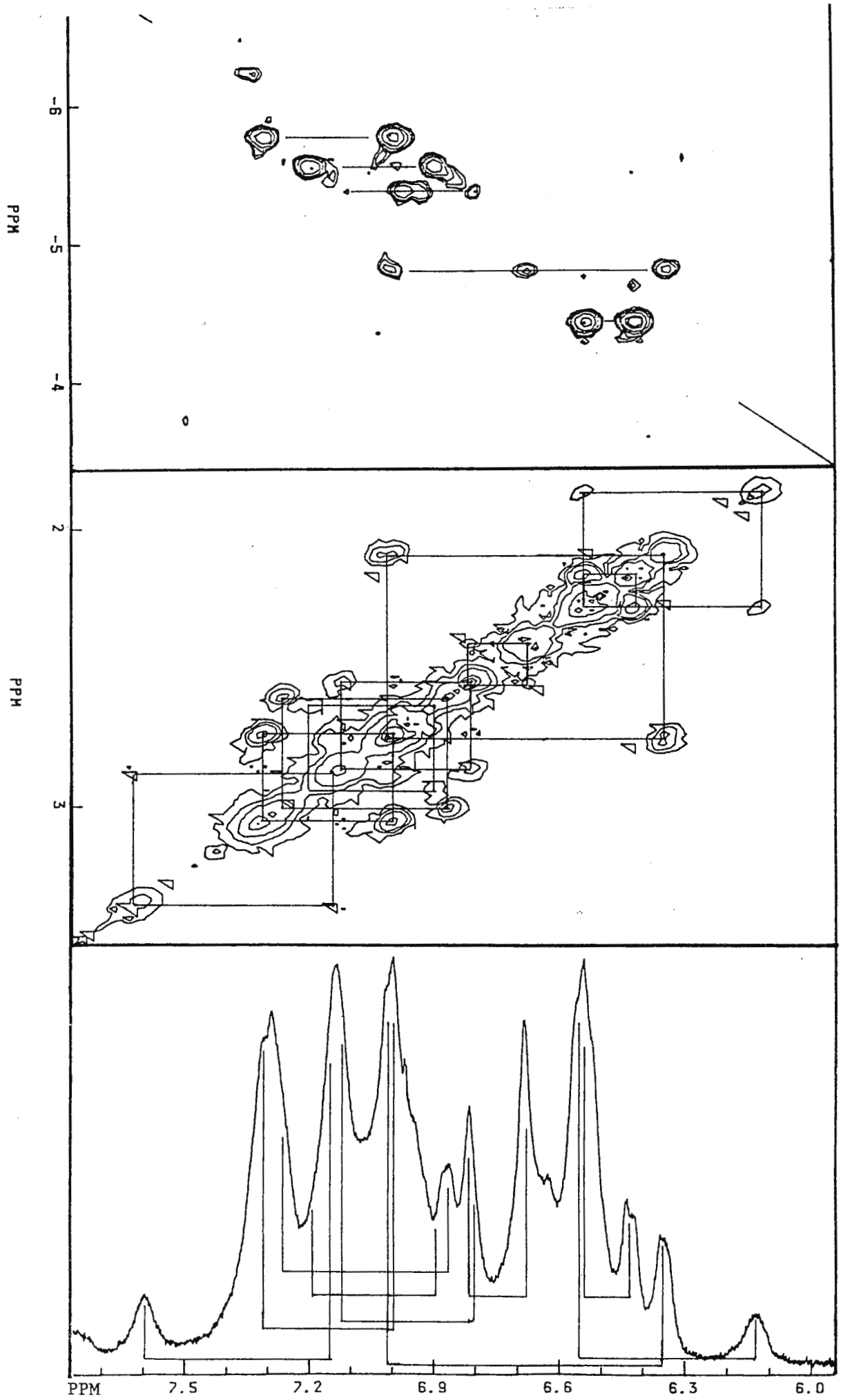
We are, as everybody may have guessed by now, basicly interested in a sound assignment of the seven tyrosyl spin systems in the spectrum. The outcome will be reported soon.

An additional remark: Both the DQ- and the COSY-spectrum were taken with the same sample and the same spectrometer settings, the J-coupling optimum for the DQ-spectrum selected to be 8 HZ. Both spectra took the same amount of spectrometer time (90 hours, 2 ml of a 2.5 mM solution, 10mm probe in a Bruker 360 CXP spectrometer).

Hoping desperately that this letter brings the TAMU-newsletters back on my desk

with best regards

Paul Rosch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Prof B I Shapiro
Texas A & M University
Dept of Chemistry
College Station
TEXAS 77843
U.S.A.

KR/GJT/MAT/JMM 23 AUG 83

Dear Professor Shapiro

SURFACTANT/ELECTROLYTE INTERACTIONS

In this letter we would like to report further progress in understanding the interactions between ions and surfactants using nmr quadruple splitting of sodium ions. It is well known that concentrated aqueous electrolyte solutions can "salt-out" or "salt-in" polymers and surfactants (cf. the Hoffmeister series for proteins). General thermodynamic considerations suggest that "salting-in" is due to positive adsorption of the electrolyte at the solute, while "salting-out" arised from negative adsorption (1). We have investigated the interactions of a "salting-in" electrolyte (NaSCN) and a "salting-out" electrolyte Na_2CO_3) with a commercial surfactant, dodecyl-dimethylamine oxide (C_{12-14} AO), using the hexagonal lyotropic mesophase. Since both the electrolytes are sodium salts, the major adsorption effects arise from the anions; never-the-less, ^{23}Na nmr can be used to study the interaction.

For lyotropic mesophases the measured sodium quadrupole splitting (Δ) is due to the fraction of ions in contact with the head goups (P_b), which have a splitting Δ_b. These "bound" ions are in fast exchange with ions well away from the surfactant for which $\Delta = 0$ (i.e. $\Delta = P_b \Delta_b$). The magnitude of Δ_b is dependent on the angle between the non-averaged electric field gradient at the sodium ion and the normal to the surfactant/water interface ($= \theta_{DM}$).

$$\Delta_b = \frac{3}{4} E_{Qb} S_b = \frac{3}{4} E_{Qb} \tfrac{1}{2} < 3 \cos^2\theta_{DM} - 1 > \quad \ldots\ldots\ldots\ldots (1)$$

(For a "powder" sample; E_Q is the quadrupole coupling constant, and S_b is the order parameter which depends on θ_{DM}). Ions which interact only with the head group/water surface have values of $\theta_{DM} \approx 0$, hence S_b is positive. When the ions penetrate between the head groups, θ_{DM} increases, and can become larger than the magic angle (54.74°), thus S_b can become negative (2). With weak non-specific, interactions between the

N8R300

head groups and ions (negative adsorption) only surface contact is expected - i.e. S_b positive. Strong anion adsorption (positive adsorption) should result in penetration of anions between head groups, which in turn, should lead to penetration of sodium ions into the head group region - i.e. S_b negative. Thus in surfactant/water/electrolyte mixtures we expect Δ_b to change sign as the electrolyte is changed from Na_2CO_3 to NaSCN.

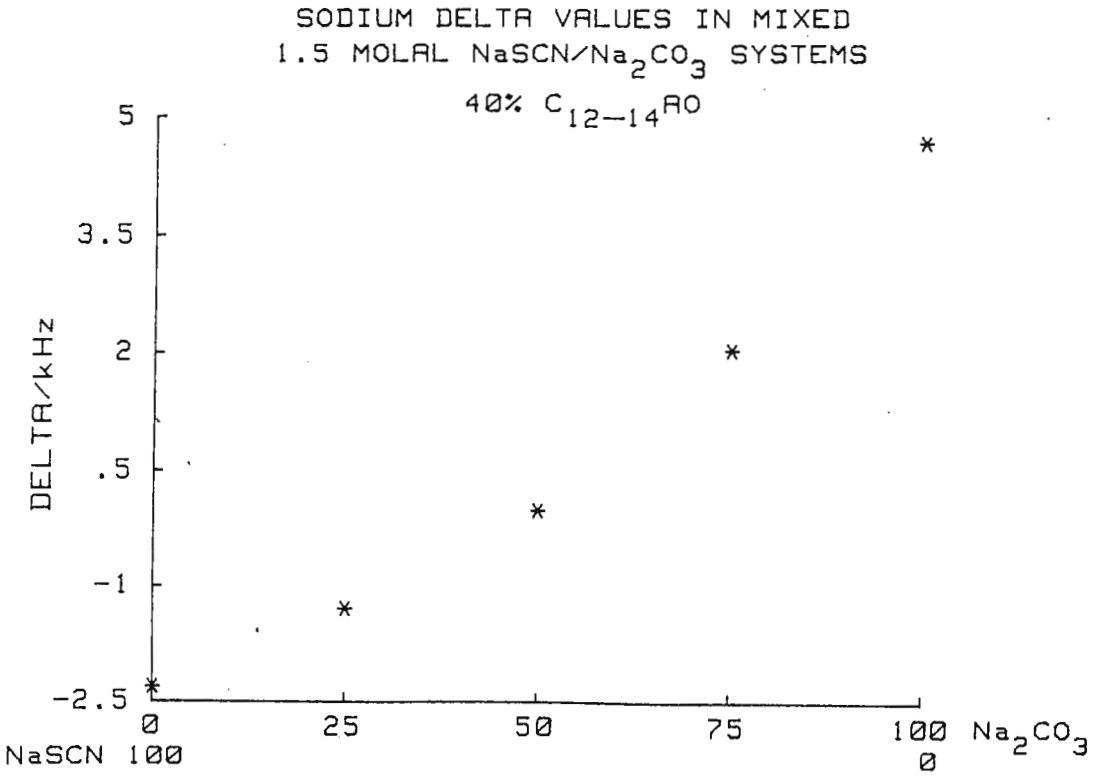
We have measured sodium Δ values for $C_{12}AO$/electrolyte mixtured covering a range of concentrations, and indeed, Δ values are of opposite sign for high concentrations of NaSCN and Na_2CO_3. This is illustrated in the attached figure, and supports the proposed mechanism for "salting-in"/ "salting-out" electrolyte effects. (Note that to facilitate comparison with lamellar phase data, the Δ values have been multipled by -1 to take into account the difference in sign of S_b between hexagonal and lamellar phases.) This work is currently being prepared for publication.

Yours sincerely

K RENDALL G J T TIDDY M A TREVETHAN

(1) D G Hall, Trans Faraday Soc, 1971, 67, 2516; J C S Faraday Trans 2, 1974, 70, 1526

(2) G Lindblom, B Lindman and G J T Tiddy, J Amer Chem Soc, 1978, 100, 2299



304-5

Wageningen

Department of Molecular Physics

Your reference
Your letter of
Our reference 83/839 mh/mk
Date November, 30th, 1983
Enclosure(s)

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

Subject Selective excitation in high-resolution solid state NMR

Dear professor Shapiro,

In high-resolution NMR spectroscopy it has been demonstrated by Morris and Freeman (J. Magn. Reson. 29, 433-462, 1978), that NMR peaks in a spectrum can be selectively excited by the so-called DANTE pulse sequence.

Recently, we have carried out analogous experiments in solids, using magic-angle-spinning in combination with cross-polarization (CP). For selectively exciting a peak in a ^{13}C MAS NMR spectrum, the pulse scheme is given in Fig. 1. The difference with conventional CP-MAS pulse schemes is that after the contact pulse the ^{13}C magnetization is flipped back to the z-axis, after which a DANTE pulse sequence is applied. We have found that this pulse scheme produces appreciable better signal-to-noise ratios than direct excitation of the ^{13}C nuclei.

The results of the pulse scheme of Fig. 1 applied to crystalline glycine are shown in Fig. 2. In Fig. 2A the conventional ^{13}C CP-MAS NMR spectrum is shown. The centerband of the C = O peak is selectively irradiated in Fig. 2B, whereas in Fig. 2C the left spinning sideband is excited. In this case, the suppression of other peaks than the one excited is better than a factor of 10.

Currently, we are using the pulse scheme in our ^{13}C and ^{31}P MAS NMR investigations on virus systems.

Sincerely yours,

Marcus A. Hemminga Rolf M.J.N. Lamerichs P. Adrie de Jager

P.S. Please credit to professor T.J. Schaafsma

Agricultural University/Transitorium, De Dreijen 11/6703 BC Wageningen/The Netherlands/Tel. (08370) 82044/82634

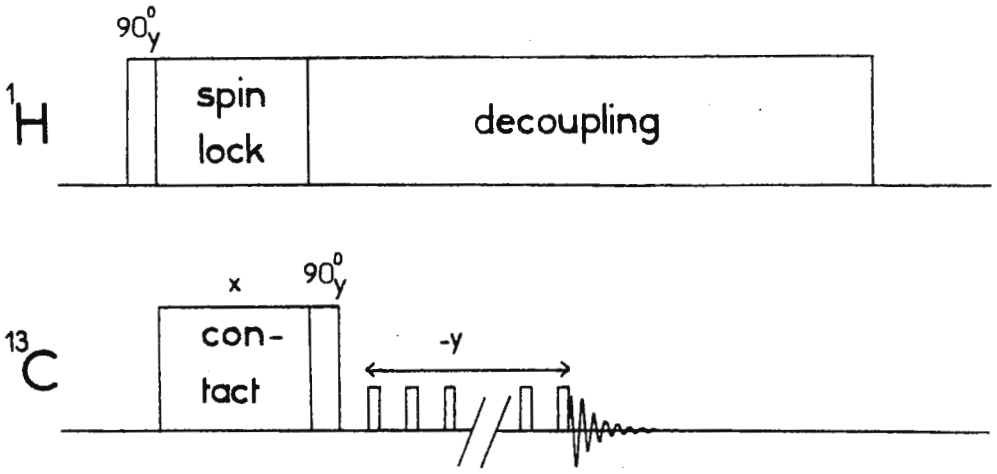


Fig. 1. Pulse scheme for selective excitation in CP-MAS ^{13}C NMR

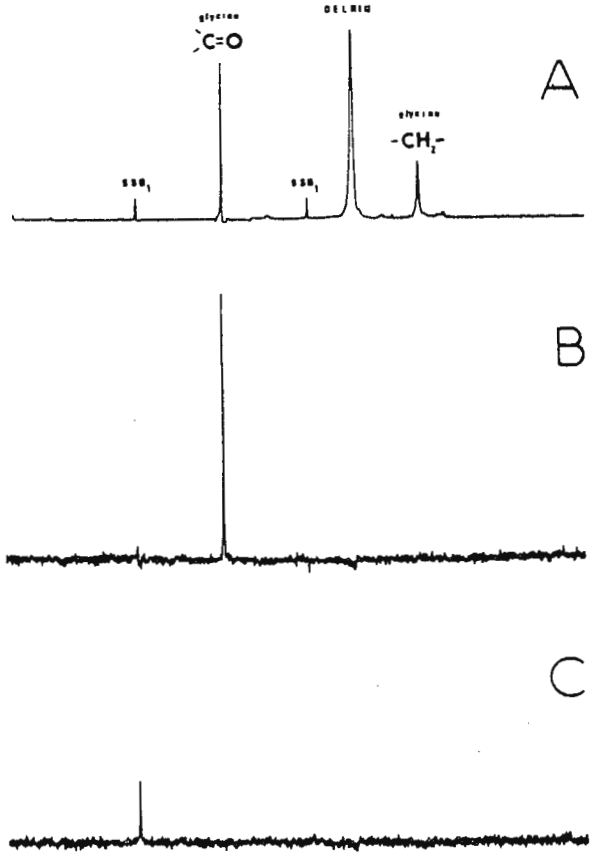


Fig. 2. 75 MHz CP-MAS ^{13}C NMR spectra of glycine in a DELRIN spinner measured on a CXP-300 NMR spectrometer.

A: Conventional CP-MAS spectrum.

B: C=O center band excited with the pulse scheme of Fig.1.

C: Left spinning sideband of C=O is excited.

Spectral width: 30 KHz.

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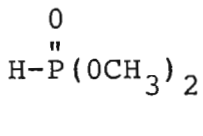
December 1, 1983

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

Subject: "Deuterium Isotope Effects on P-H and P-C Coupling Constants"

Dear Barry:

We have recently studied the NMR spectra of dimethyl phosphonate (I) and chloromethylphosphinic acid (II).



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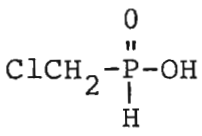


II

The P-H bonds in compounds I and II are readily exchanged with deuterium oxide to form P-D bonds. When a limiting amount of deuterium oxide is present, both deuterated and non-deuterated species are observed in the NMR spectra. The deuterium isotope effects on phosphorus and carbon shifts are well known. However, to our knowledge, the deuterium isotope effects on coupling constants have not been reported. The ^{31}P and ^{13}C NMR parameters of compounds I and II are listed in the attached table. As shown in the table, the deuterium isotope effects on P-H and P-C coupling constants are substantial.

Sincerely,

C. K. Tseng

L. L. Chang

mn
attachment

^{13}C and ^{31}P NMR Parameters*

	I (Protio)	I (Deuterio)	Δ(D-H)
$\delta^{31}P$	11.08	10.73	-0.35
$^{3}J(PH)$	11.92	11.87	-0.05

	II (Protio)	II (Deuterio)	Δ(D-H)
$\delta^{31}P$	27.01	26.61	-0.40
$^{2}J(PH)$	8.90	9.04	+0.14
$\delta^{13}C$	36.33	36.21	-0.12
$^{1}J(PC)$	97.96	98.65	+0.69

*The spectra were obtained on a Varian XL-200 NMR spectrometer and were taken at spectral widths of 3KHz for ^{13}C and 1.6 KHz for ^{31}P with 32K data points.

Eidgenössische
Technische Hochschule Zürich

Laboratorium
für Physikalische Chemie

Prof. Dr. R. R. Ernst
RIER/mü

CH-8092 Zürich
ETH-Zentrum
Durchwahlnummer 01 / 256 43 68
Telefonzentrale 01 / 256 22 11

November 29/1983

0430

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

Dear Barry,

UNIFORM EXCITATION OF QUADRUPOLE SPECTRA BY COMPOSITE PULSES

We have recently cooked up new composite pulses which give a homogeneous broadband excitation of spins I = 1 in solids in the presence of large quadrupole splittings. For an rf field corresponding to a 8 μs 90^0 pulse they produce a bandwidth approximately equivalent to that of a 0.5 μs 90^0 pulse. Incorporated in a quadrupole echo sequence, the new composite pulses are:

$$\text{I}: \quad 135^0_x \; 90^0_{-x} \; 45^0_x - t_1 - 135^0_y \; 90^0_{-y} \; 45^0_y - t_2$$

$$\text{II}: \quad 90^0_{-x} \; 180^0_x \; 90^0_{-x} \; 135^0_x \; 45^0_{-x} - t_1 - 90^0_{-y} \; 180^0_y \; 90^0_{-y} \; 135^0_y \; 45^0_{-y} - t_2 \; .$$

The observant reader will doubtless have noticed immediately that if all pulse durations are multiplied by 2 then each composite pulse is the same as a segment out of a WALTZ decoupling cycle (Shaka et al., J.M.R. 53, 313 (1983)). This is no coincidence since these sequences were constructed by recognizing a formal similarity between the problem of broadband inversion in spins 1/2 and broadband excitation in spin 1 systems. We show below computer simulations and experimental results for a conventional quadrupole echo sequence (a)

$$90^0_x - t_1 - 90^0_y - t_2$$

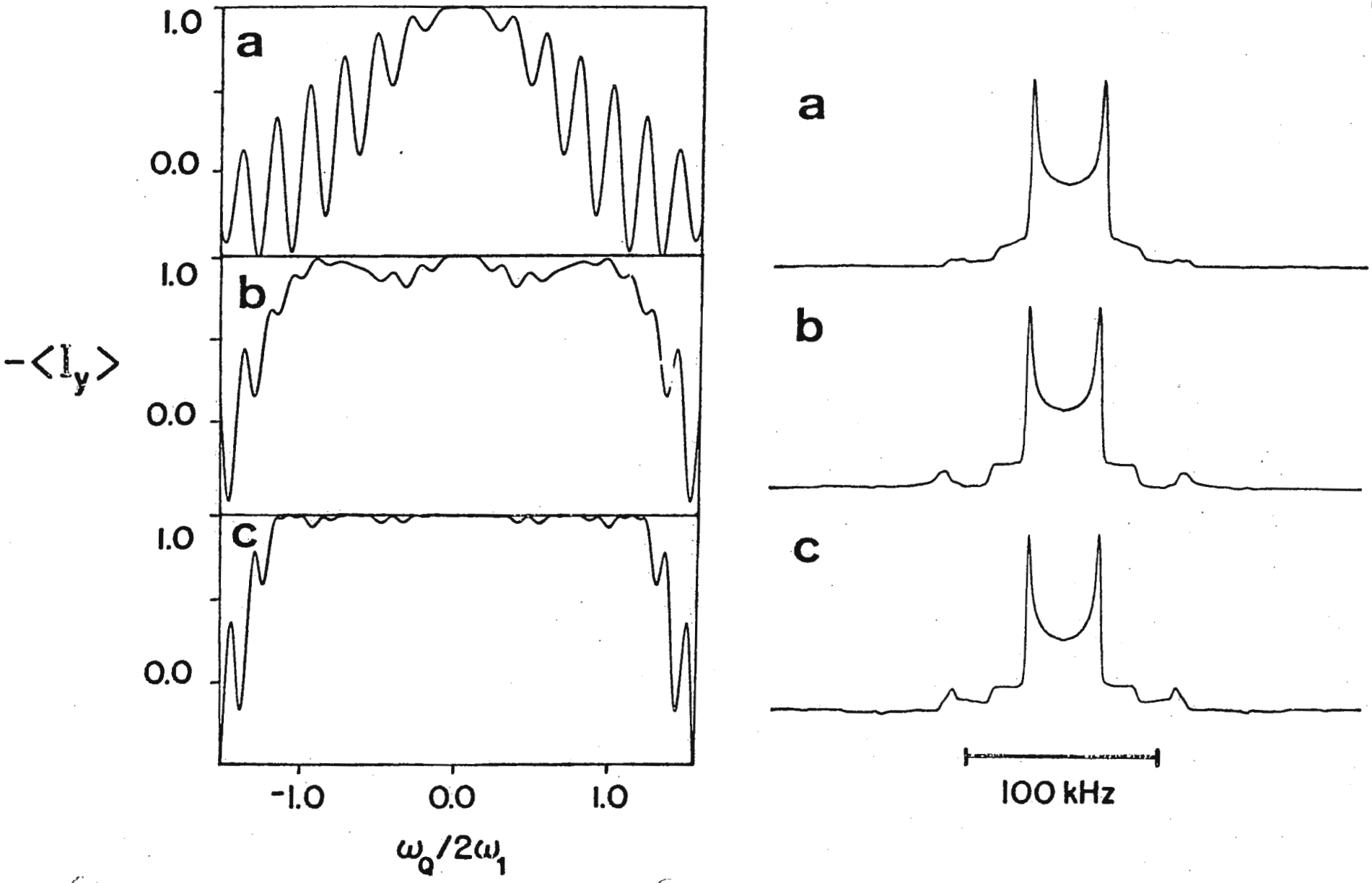
and the two composite pulse cycles (b and c). The simulations (left) plot echo signal against quadrupole interactions: The experimental results (right) are for deuterated PMMA using a 8 μs 90^{o} pulse length, and show the expected increase in bandwidths on using composite pulses. A paper with the theoretical treatment has been submitted for publication.

Sincerely yours,

Malcolm Levitt

Dieter Suter

Richard R. Ernst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a
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b
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c
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$-\langle I_y \rangle$
-1.0
0.0
1.0
$\omega_Q/2\omega_1$
a
b
c
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THE ROCKEFELLER UNIVERSITY
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21 December 1983

Dr. Bernard L. Shapiro
Texas A & M University

N-15 Studies of Gramicidin S in Water

Dear Dr. Shapiro:

Methods (e.g. INEPT)[1] for enhancing the signals from insensitive nuclei by spin polarization transfer from protons has opened up a wide range of problems to study by ^{15}N NMR that would have required unacceptable amounts of time by more conventional methods. For ^{15}N there is a theoretical increase of a factor of 10 in the intensity of the signal relative to that with no NOE and we routinely achieve a factor of 8.5 on our NT-300 wide-bore system using a 20mm ^{15}N probe. The INEPT method also takes favorable advantage of the generally shorter ^{1}H T_1's relative to the ^{15}N T_1's. Further it eliminates the possibility of signal nulling from partial NOE for the ^{15}N. These factors are particularly favorable for our research in the structure and interactions of the peptide backbone by ^{15}N NMR. We are particularly interested in studying peptides in water, a more biologically relevant solvent than many, but in which solubility is sometimes low. One molecule that is of interest for understanding the effects of structural and solvent interactions is the cyclic decapeptide gramicidin S_1 (GS), pictured in figure 1. Its structure is understood and is solvent independent, but GS is only slightly soluble (about 10^{-2}M) in water. Nonetheless using the INEPT method it is possible to obtain a spectrum such as the one in fig. 1A in about 90 min from a 10 ml sample in a 20mm tube. To assign this spectrum we used a homegrown modification of the INEPT pulse sequence[2] which allows for suppression of a nitrogen line in the spectrum by the application of a selective 180° pulse to one of the proton ^{15}N satellites. This can differentiate nitrogens that are coupled to protons with ^{1}H chemical shifts differences of only a few Hz. Examples of the application of this method are shown in figure 1B and C. It should be noted that the proline nitrogen is not observed in this experiment because of the absence of a directly coupled proton. Using this approach we have reexamined the previously reported spectra for gramicidin S in organic solvents[3] as well and measured the temperature coefficients of the protonated nitrogens in water and organic solvents. From these data we have been able to demonstrate long range perturbations in the ^{15}N spectrum transmitted via an internal hydrogen bond across a total of six bonds. Details of this work will appear in the Journal of the American Chemical Society.[4]

An increasingly important factor as more ^{15}N data becomes available and as more detailed analysis of chemical shifts are undertaken (e.g. temperature effects) is referencing the ^{15}N spectrum. Sensitivity and compatibility considerations preclude a good internal reference and external referencing suffers from factors such as the need for bulk susceptibility corrections. Our solution to this problem has been to use a ^{1}H reference compound. On most modern NMR spectrometers it is relatively easy to measure the proton spectrum of an ^{15}N sample through the decoupler coils of the ^{15}N probe. We have carefully measured the absolute frequencies of TMS protons and CH_3NO_2 nitrogen in a sample of CH_3NO_2 and 1% TMS locked externally. Using the value of 380.23 ppm for the shift of $CH_3{}^{15}NO_2$ relative to NH_3 liquid,[5] we have derived the following equation for the nitrogen shift scale relative to NH_3 based on the frequency of the TMS resonance

$$f_o^{^{15}N} = 30,405,530.9 + r * (f_{TMS}^{^{1}H} - 300,066,985.6)$$

where f^{1H}_{TMS} is the TMS frequency of the sample under study and r is the ratio of the TMS frequency in CH_3NO_2 to the NH_3 position derived from that sample, 0.10132914444, which should be constant for all spectrometers. The other frequencies are for our particular spectrometer, being the derived NH_3 frequency and that of TMS in CH_3NO_2. They can easily be scaled to other systems by measuring the absolute frequency of TMS at 1% in CH_3NO_2.[4]

Sincerely,

David H. Live

1. Morris, G. A.; Freeman, R. 1979 JACS 101, 760-762.
2. Davis, D. G.; Live, D. H.; Agosta, W. C.; Cowburn, D. 1983 JMR 53, 350-354.
3. Hawkes, G. W.; Randall, E. N.; Hall, W. E.; Convert, O. Biopolymers 1980, 17, 1815.
4. Live, D. H.; Davis, D. G.; Agosta, W. C.; Cowburn, D. 1984 JACS (May?)
5. Levy, G. C. and Lichter, R. L. "Nitrogen-15 Nuclear Magnetic Resonance Spectroscopy" J. Wiley and Sons, New York, 1979.

P.S. Please credit to David Cowburn's account.

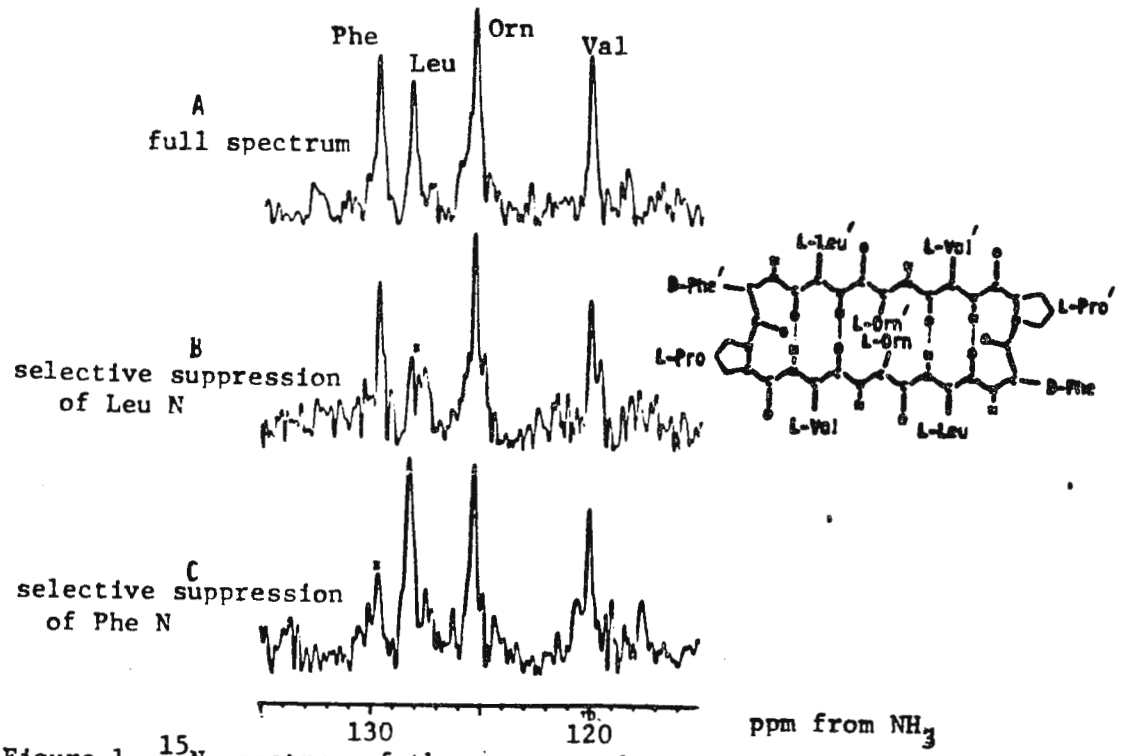

Figure 1. ^{15}N spectrum of the protonated nitrogens of gramicidin S in water

BRANDEIS UNIVERSITY
WALTHAM, MASSACHUSETTS 02254

GRADUATE DEPARTMENT OF BIOCHEMISTRY

December 5, 1983

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

How to make a 500 MHZ Spectrometer

Dear Barry:

We've recently installed an Oxford 500 small bore magnet on our homemade former 270 system. The probe was made by Cryomagnet Systems (Indianapolis) and is driven by a 3W ENI amplifier followed by a ~15W Motorola MHW-710-3 to give a ~22 μ sec 90° proton pulse. Janel supplied a 1.5 db noise figure preamplifier (their measurement) and performance seems similar to the best commercial systems though we've not made quantitative comparisons. The probe has an extra crossed coil for ^{15}N or ^{13}C irradiation (200 μsec 90° pulse ^{15}N from a 10 W ENI), and D lock is via the proton coil. Sara built a 500 MHZ PIN diode switch and Hoult-Richards switch (J. Mag. Res. 22, 561) like the one on the 270, and replaced the first local oscillator frequency of 160 MHZ (converting 270 to 110 MHZ) with 390 MHZ generated from our 120 MHZ clock using predominantly commercial components. The deuteron lock first local oscillator of ~35 MHZ (converting from ~41 MHZ to 6.15 MHZ) was replaced by a ~70 MHZ International crystal oscillator modified to a VCXO and phase locked to our clock more or less as we described previously (See p. 236, NMR and Biochemistry, Opella and Lu eds.; published by Dekker). Naturally we transferred our idiot-proof sample raiser-lowerer to the 500 (see same article) since we can't afford to have probes repaired.

The only unexpected problem is that when we apply a spoil we have to follow it by a ~30 msec recovery time, whereas previously at 270, 3 msec sufficed. Probably this is due to eddy currents in the apparently pure copper shield that Oxford has inside the shim coils. The magnet was delivered just before Labor Day and gave research quality spectra the following weekend; we would have gotten it going sooner except that our air conditioner (and consequently some electronics) failed just then. For our normal ±5° room temperature variation shim is plenty stable enough for tRNA work, and we have achieved the expected 0.5 Hz spinning linewidth, but have not done challenging high resolution work on smaller molecules. We're now replacing our old Nova computer with a system based on an Intel 8086-87 processor, and would like to hear from anyone who has programs for such a system or wants them.

Parts cost for this conversion was ~$200K for magnet, shim, and probes; ~$1K for proton conversion; ~$200 for lock conversion.

Advertisement We will be getting rid of our 90 MHZ Bruker WH90 in about 5 months. BNC 12 computer (similar to Nicolet 1080), low density Diablo disc, homemade timer, Haskris chiller, no terminals or printer, needs a PTS synthesizer for ^{13}C, ^{19}F, ^{31}P, but has probes and boxes for these. Our plans are indefinite. It is much modified and would require several hrs/week of attention by a highly competent engineer. The magnet is in good shape. The system was the second WH90 in the U.S., delivered 1973. Anyone interested should contact us.

Sincerely,

Sara Kunz Al Redfield

Sara Kunz and Alfred Redfield

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Rome, December 16th 1983

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

Detection and assignment of the phosphodiesters in ^{31}P NMR of the pathogenic microorganism Candida albicans.

Dear Professor Shapiro,

Candida albicans is a microorganism whose importance as opportunistic agent of disease is steadily increasing. It is characterized by its capacity to grow in two different forms i.e. the Y (yeast) form, consisting of single cells producing by budding, and the M (mycelial) form, consisting of a true hypha growing by the process of apical extension. This second form of growth seems to be endowed with greater pathogenicity potential (1).

We have recently analyzed the 31-P NMR spectra of the two forms of growth and showed modulations in some intracellular metabolites and products during the Y→M transition (2). In all cases, the spectra presented conspicuous, unidentified signals in the "phosphodiester" region, banding in the frequency range of 0.0 to -3.0 ppm with respect to external 85% H_3PO_4 (Figure 1), which were significantly modulated during growth and morphogenesis of the microorganism. We have further studied this aspect by use of purified standard compounds and an integrated biochemical approach. Therefore, we have shown the following:

1. The signal at -0.3 ppm originates from glycero-phosphorylcholine (GPC), a compound whose physiological role is very much debated. It is of interest that this compound is also significantly modulated in cellular systems other than C. albicans, in particular tumors, hemopoietic cells and red muscles (3).

2. The signals centered around -2.0 ppm is, with all reasonable evidence, originated or at least contributed by cell wall phosphomannan. Major evidence for this was derived from a comparison in the spectra of cellular extract and a purified phosphomannan antigen from both yeast and mycelial forms.

Fig.1 reports a ^{31}P NMR spectrum, obtained with a Varian XL-100 spectrometer operating at 40.5 MHz, at 4°C, of the hyphal form of C.albicans grown in glucose-proline-saline medium. The arrows point to GPC and phosphomannan signals.
Future studies using non-invasive, non-distructive approach of the type shown here may contribute to the elucidation of the metabolic significance

of the glycerol-phosphorylated compound. They may also shed some light on the phosphorylated immunogenic determinants of fungal cell wall.

Sincerely yours,

F.Podo G.Carpinelli M.Di Vito
A.Torosantucci L. Angiolella A. Cassone

(Work supported by grants from CNR-TBMS contract n. 83.00571.57 and CNR-CMI contract n. 83.00262.96).

References

1. F.C. Odds. Leicester; University Press.(1979).
2. A. Cassone, G. Carpinelli, L. Angiolella, G. Maddaluno and F.Podo. J. Gen. Microbiol. 129, 1569-1575 (1983).
3. T.J. Brady et al. Proceedings of an International Symp. on Nuclear Magnetic Resonance Imaging. Bowman Gray School of Medicine, Winston-Salem, North Caroline, October 1-3,1981.
4. G.Carpinelli, G.Maddaluno, F. Podo, E. Proietti, L. Santurbano, F. Belardelli. Second Annual Meeting Society of Magnetic Resonance in Medicine, San Francisco, August 16-19, 1983. (Abstracts, p. 77).

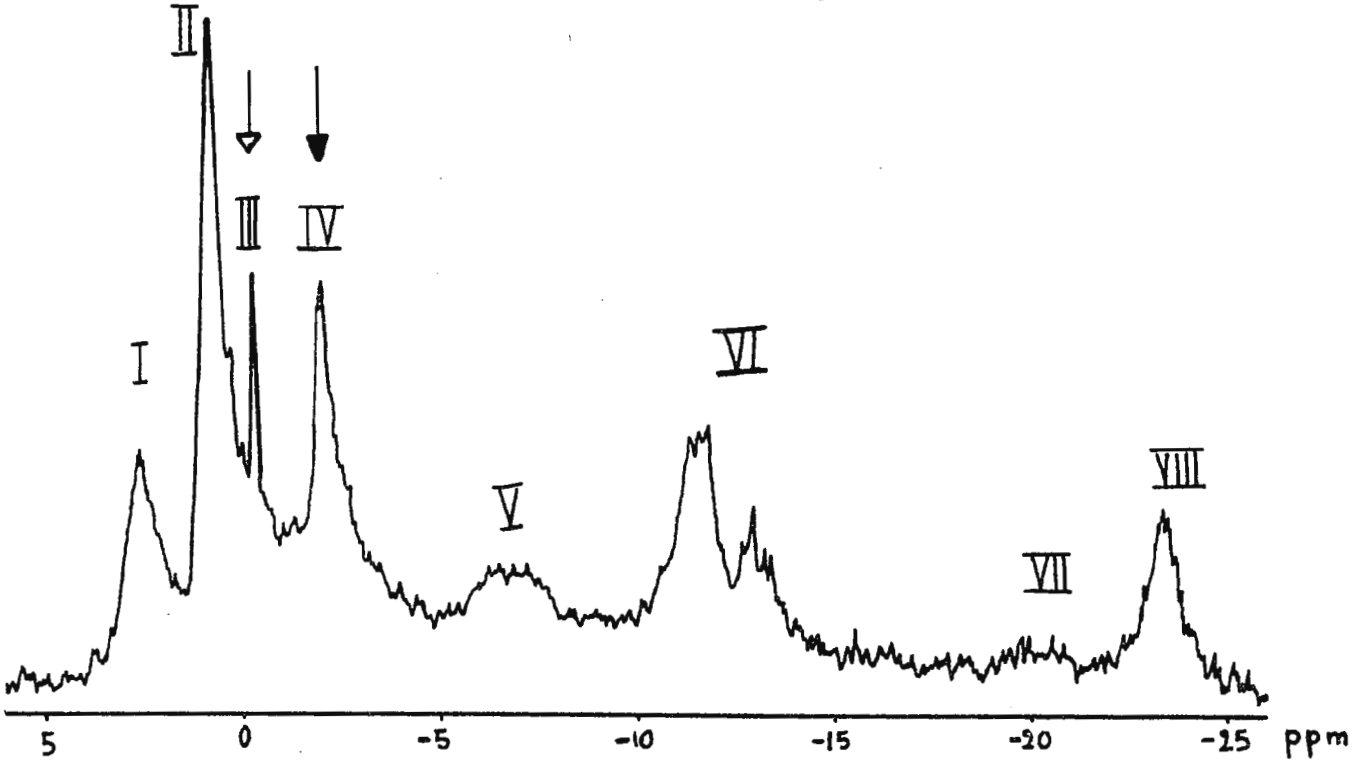


Figure 1

I= sugar phosphates; II= inorganic phosphate(s); III (white-head arrowed)= GPC; IV (black-head arrowed)= phosphomannan; V= γ-ATP and β-ADP; VI = α-ATP, α-ADP, NAD and other nucleotide-sugar derivatives; VII= β-ATP; VIII= long-chain poly-phosphates.

ABBOTT

Pharmaceutical Products Division

Abbott Laboratories
North Chicago, Illinois 60064

December 27, 1983

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

Dear Dr. Shapiro:

Use of a Chemical Shift Correlation Map (CSCM)
for Carbon Assignments of Erythromycin A in $CDCl_3$

In some of the early work on the C-13 assignments of erythromycin A in $CDCl_3$[1] there was some uncertainty for the assignment of carbons 2, 8, and 10 of the macrolide ring. By obtaining a CSCM plot on our NMC 360 MHz spectrometer (Figure 1) it was possible to assign these carbons unambiguously.

In Figure 1 the proton region between 3.40 and 2.55 ppm is shown with the proton attached to 2, 8, and 10 carbon identified. These protons were assigned using a COSY plot. With the CSCM plot it can be shown that the C-13 resonance at 44.9 ppm is C2, the resonance at 45.1 ppm is C8 and the resonance at 37.9 ppm is C10.

Sincerely,

Merrill Nuss

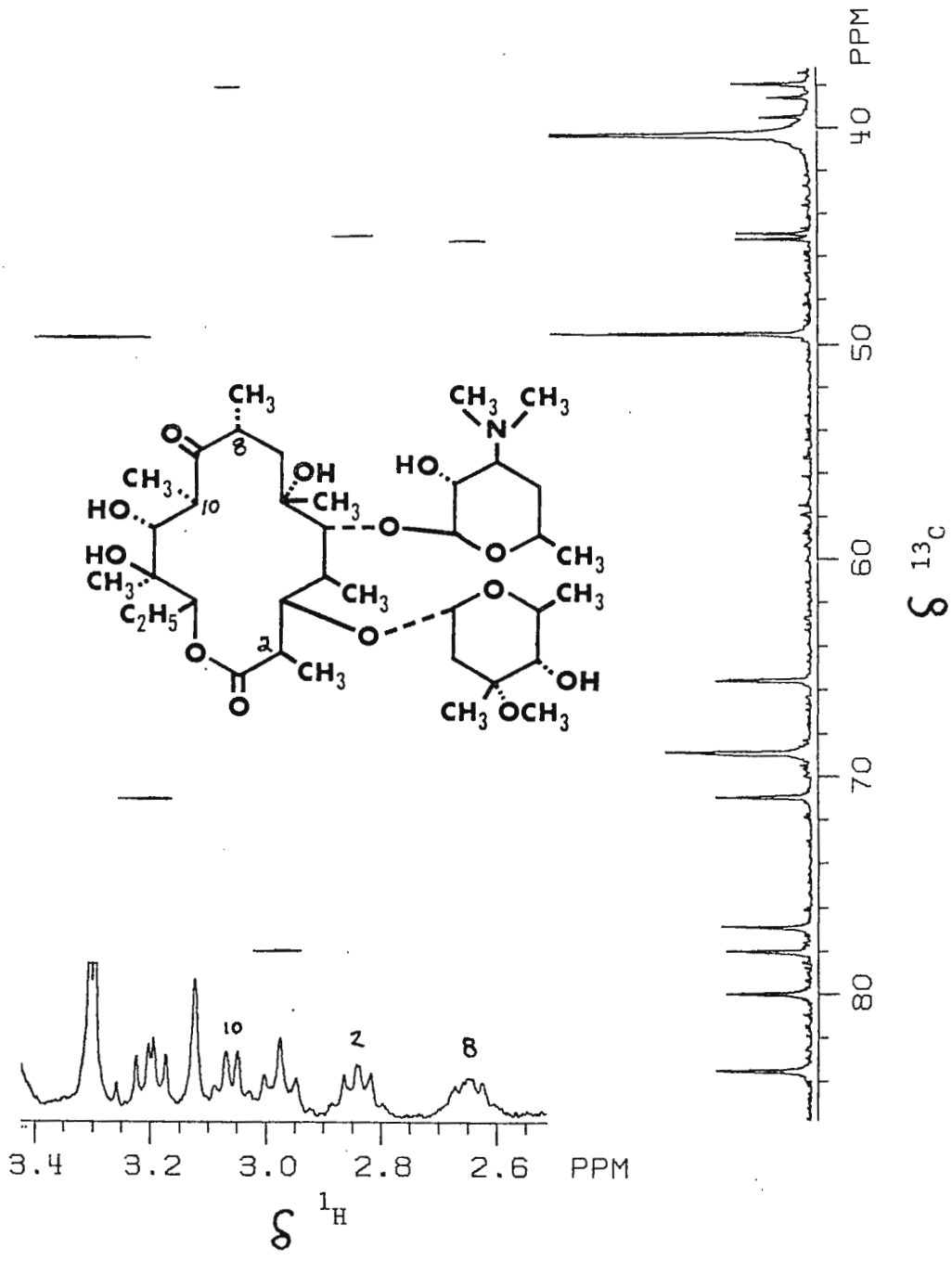
Merrill Nuss

Les Freiberg

Les Freiberg

[1]Omura, et. al., Tet. Let., No. 34, 2939 (1975).

Figure 1
CSCM Contour Plot of erythromycin A in $CDCl_3$



The Florida State University
Tallahassee, Florida 32306
Department of Chemistry

December 27, 1983

Professor Barry Shapiro
Texas A & M University NMR Newsletter
Department of Chemistry
Texas A & M University
College Station, TX 77843

Dear Barry:

Technetium NMR Monitor for Reaction Kinetics

An important question concerning the reduction of the pertechnetate ion has been raised in a DOE Radioactive Waste Isolation Project. It is assumed that basalt can convert soluble TcO_4^- into insoluble TcO_2. For ease of study in this system, it is important to determine whether hydrazine reduction can be used to model the slow and sometimes uncertain reaction found with natural basalt. The problem has arisen from the experimental results that have been found for the reduction. From all appearances, the technetium dioxide is far more soluble than previous data would imply. (The solubility is rather vaguely and variously reported to be in the range of 10^{-12} to 10^{-8} M.) Thus it is important to determine more about the nature of the reduction process. Specifically, does it go to completion?

Technetium nuclear magnetic resonance is an excellent tool for these studies if the conditions are favorable. Technetium is the fourth most sensitive of all nuclei but it has a magnetic moment of 9/2 which presents a potential problem. The only time that one can be certain that the lines will be sharp is when the compound has a symmetric field as found in T_d tetrahedral or O_h octahedral complexes (all peripheral groups the same). The pertechnetate ion satisfies these requirements quite well. Another serious problem to the general application of technetium NMR is that of the paramagnetism of reduced compounds. Most lower oxidation states will yield an atom with unpaired electrons until organometallic compounds are reached with their oxidation states of +1 to -1.

In spite of these limitations, it is possible to do excellent work by following the disappearance of the pertechnetate ion in a chemical reaction. Samples containing about 0.1 mg of Tc in 1 ml of solution yield sufficient NMR signals for studying reaction kinetics.

When the concentration of the hydrazine is considerably greater than that of the pertechnetate, the reduction is too fast for this technique to follow with ease. However, when the concentration of technetium is 0.008 M and the hydrazine 0.010 M or 0.0050 M in 0.1 $NaHCO_3$, then the reduction proceeds at a rate that has a half-life in the range of 5 to 10 minutes. It

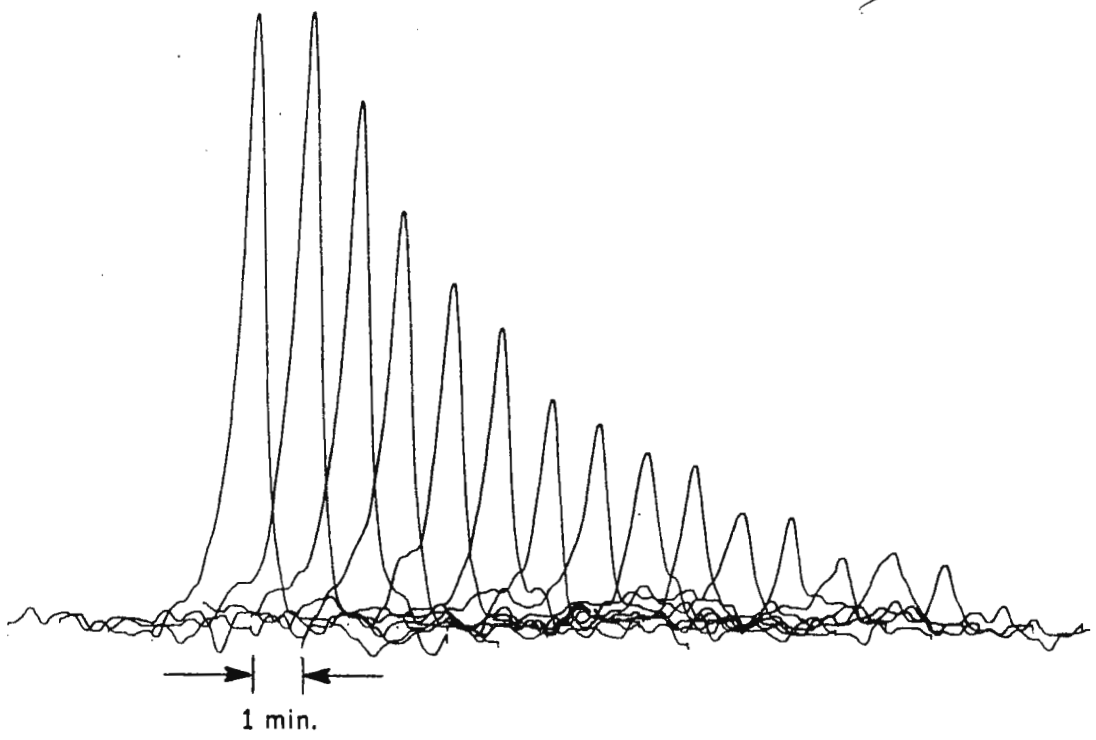
appears that the reduction goes to completion during the experimental period. Not surprisingly, the lower the concentration of hydrazine, the slower the rate of reduction. A representative set of data is shown in the Figure. (The lower apparent technetium concentration in the first determination has been found in each of the data sets obtained thus far with no adequate explanation forthcoming.)

The biggest worry with the conclusion that complete reduction has occurred concerns the effect of the product TcO_2 on the spectrum of the remaining TcO_4^-. The technetium is in the +4 oxidation state. This would mean a d^3 ion which would yield a paramagnetic compound unless there is some solid state pairing of spins. Neither peak broadening or shifting is seen. The peaks remain sharp at 3 to 4 Hz at half-height. As a final confirmation that there was no effect of product on the spectrum of the reactant, the following experiment was done. A solution of pertechnetate containing excess hydrazine was allowed to stand until the technetium signal was totally gone. Then the solution was spiked with another quantity of pertechnetate and the NMR spectrum determined quickly. The technetium signal reappeared at the proper chemical shift and in about the intensity expected. No effect on the product was evident.

These initial investigations have shown that technetium NMR can be very useful in following the course of a chemical reaction at least from the standpoint of the disappearance of reactant. Further studies pertaining to technetium uptake by the environment are currently being pursued.

Sincerely,

R. J. Clark Richard Rosanske T. E. Gedris



304-23 **CSIRO**

Physical Technology Unit
Ryde, NSW

A Unit of the Institute of Energy and Earth Resources

338 Blaxland Road, Ryde. NSW 2112
Telephone (02) 80 0211 Sydney. Telex 70827

PH/BAR438/E
WAB/de

14 December 1983

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

Pulsed Proton NMR Transients in Coal Chars Containing Free Radicals

Dear Professor Shapiro,

Some of the highly-aromatic chars remaining after pyrolysis of certain organic constituents of bituminous coal contain large concentrations of unpaired electrons. These electrons exist predominantly as resonance-stabilised free radicals which are formed during the pyrolytic decomposition of the coal. The pulsed proton NMR signals produced at room temperature by these chars are substantially affected by strong proton-unpaired electron interactions. In fact, we have direct evidence from our data of a significant fraction of the protons so strongly coupled to unpaired electrons that they are "NMR invisible" even with ~1.5μs 90^0 r.f. pulses. Although the NMR signals, therefore, do not represent all of the protons in the samples, we demonstrate below that at least some of the protons which are observed in our experiments experience an appreciable unlike-spin interaction which in turn causes problems in the analysis and interpretation of transverse relaxation data.

The proton FID is a rapidly-decaying, essentially exponential signal with a time constant ~5μs. The exponential shape, as distinct from the Gaussian decay typical of rigid solids, may be due to fast relaxation of the electronic spins. Because a significant portion of the FID is obscured by the receiver dead time (~4μs), we tried using the solid echo stimulated by the 90^0_x-τ-90^0_y pulse sequence with τ = 6μs to represent the transverse relaxation. This technique works well for samples in which proton-unpaired electron interactions are not dominant - the echo peak height is a satisfactory measure of the number of protons and the subsequent decay may be Fourier transformed in order to obtain the second moment of the absorption lineshape. Although the chars in question produce a sharp, well-defined solid echo (Figure 1), theory[1] predicts that strong unlike-spin dipolar coupling appreciably attenuates the peak echo signal even at short pulse spacings because dephasing due to such interactions is not refocussed by the second 90^0 pulse. The observed τ dependence of the echo peak height (Figure 2) suggests that substantial attenuation occurs even at τ = 5 or 6μs, the smallest value for which the echo peak is not obscured by the receiver recovery.

PH/BAR438/E

In order to obtain a signal which is a better approximation to the FID, the magnitude of the transient produced by the 90^0_x-τ-90^0_x sequence (which is entirely due to unlike-spin interactions) (Figure 1) was added to the solid echo signal. The basis for this procedure is that the leading proton-electron cross-coupling terms in the power series expansions (as functions of τ and time) for these two transient signals are equal in magnitude but opposite in sign [1,2] and therefore cancel when the signals are added. The peak height of the resultant signal (Figure 1) is much larger than that of the solid echo (Figure 1) because it is attenuated much less at short τ by the effects of proton-unpaired electron interactions (Figure 2). The second moment of the Fourier transform also is larger than that for the solid echo as would be expected by the inclusion of contributions from more strongly coupled protons which do not produce a well-defined solid echo.

Work currently in progress is aimed at obtaining meaningful values from these and other transient signals for the like- and unlike-spin contributions to the second moment of the observable protons.

Yours sincerely,

Leo J. Lynch Wesley A. Barton

1. Mansfield P. Phys. Rev. 1965, 137, A961.

2. Siegle G. Zeit. Naturforsch. 1968, 23a, 556.

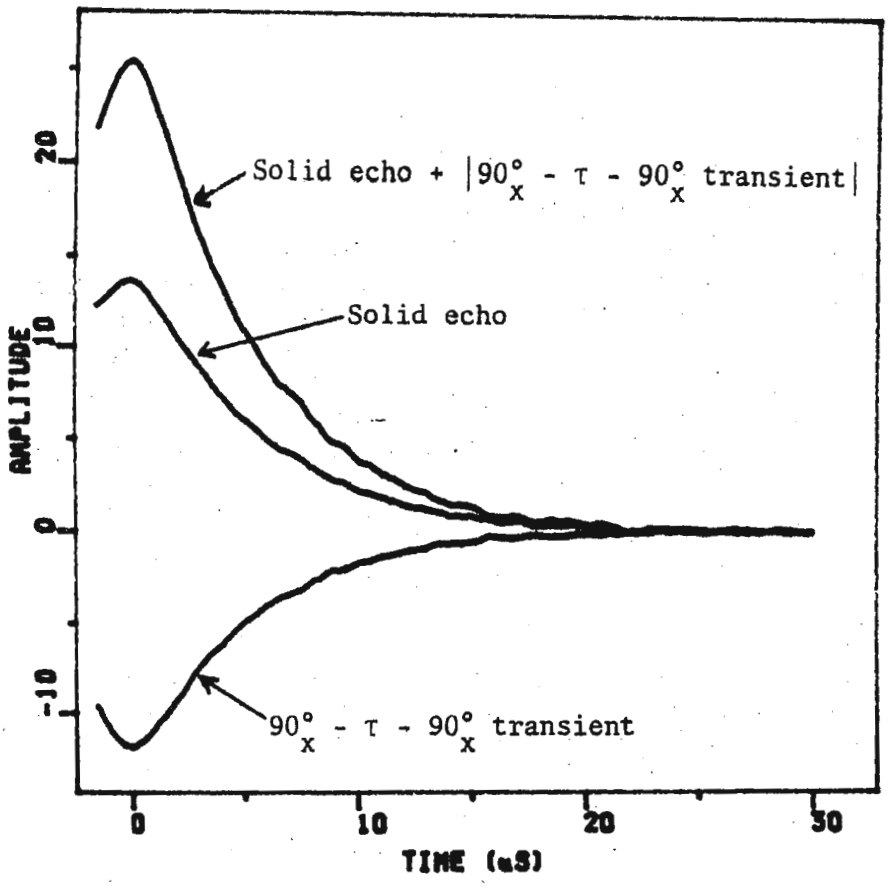


FIGURE 1:

Solid echo, $90^\circ_x - \tau - 90^\circ_x$ transient and the sum of their magnitudes for $\tau = 7\mu s$.

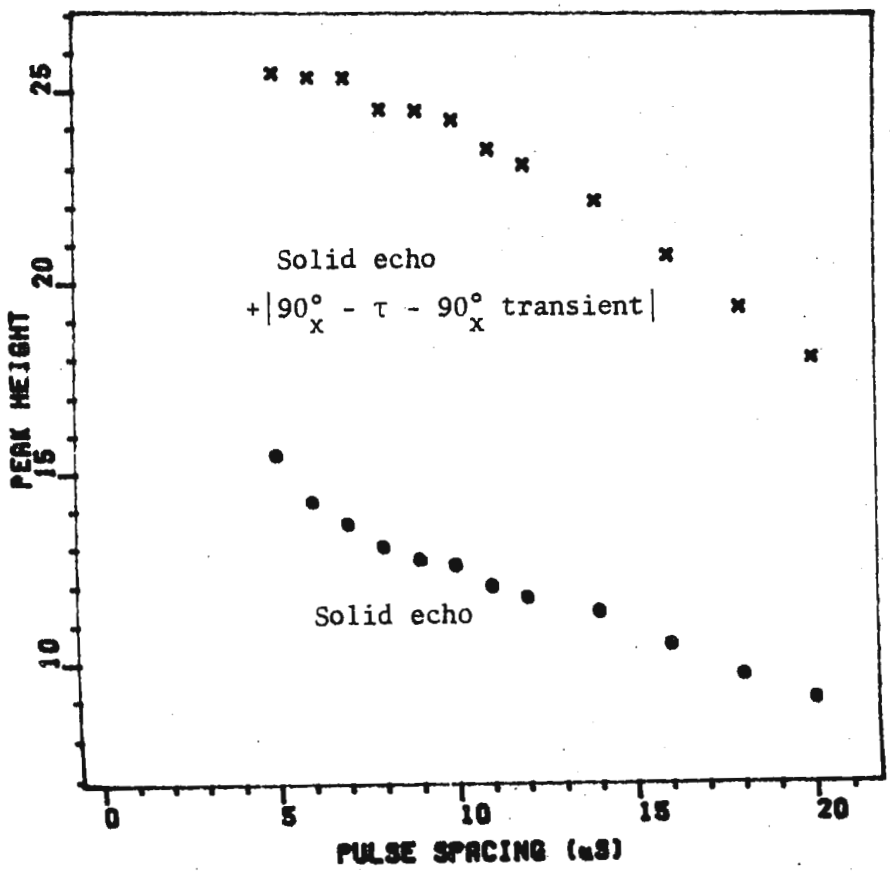


FIGURE 2:

Attenuation of the peak heights of the solid echo and the solid echo + $|90^\circ_x - \tau - 90^\circ_x$ transient| with increasing pulse spacing

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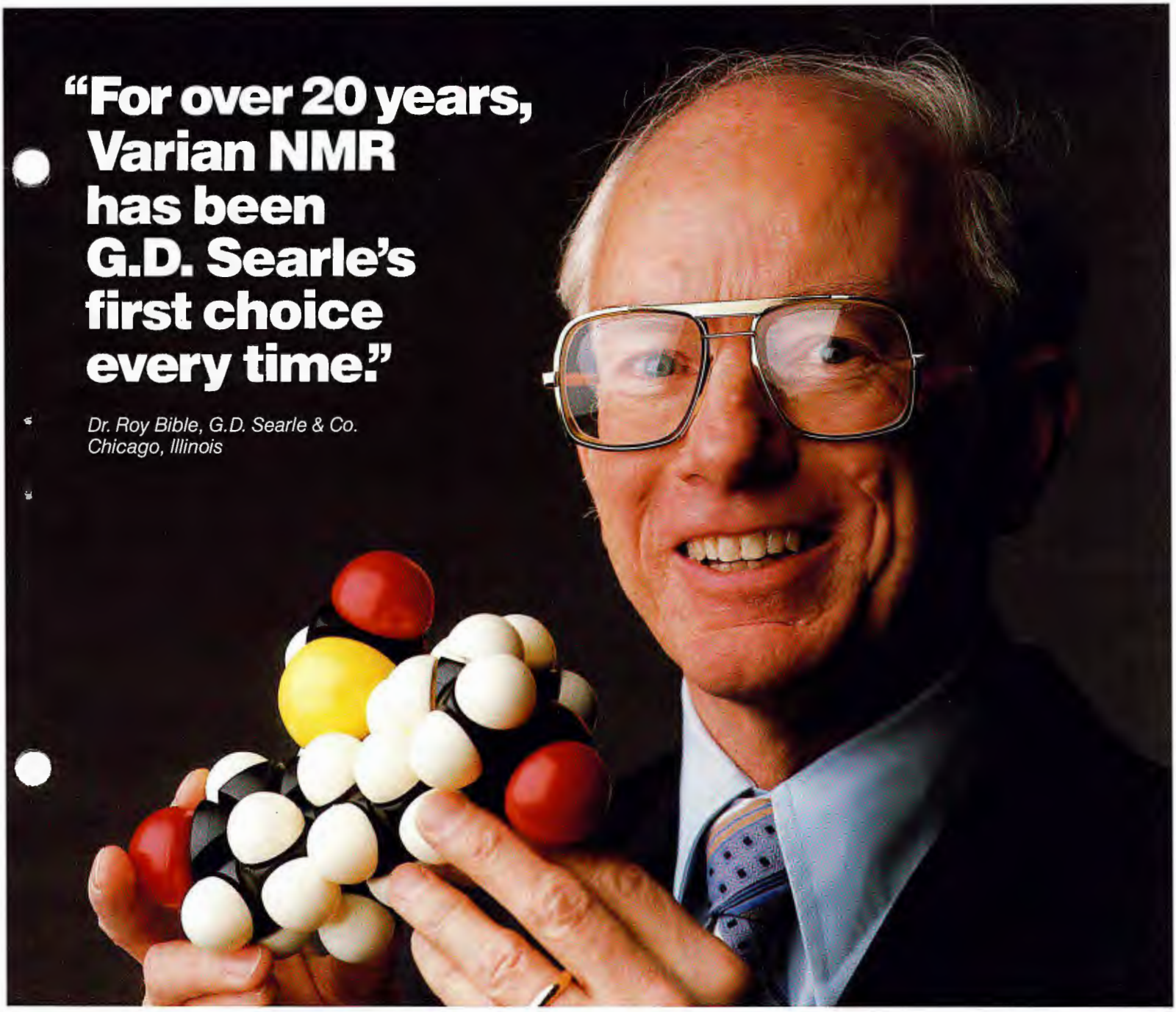
Dr. Roy Bible, G.D. Searle & Co.
Chicago, Illinois



Dr. Bible, Research Fellow and Manager of Searle's Physical Methodology Department, has published two books on nuclear magnetic resonance and is the co-author of an ACS audiovisual course on the interpretation of NMR spectra. He is shown here with a molecular model of Searle's aldosterone antagonist, spironolactone.

Here's why Dr. Roy Bible has depended on Varian NMR to solve G.D. Searle's chemical structure problems. "Our task in the Physical Methodology Lab," says Dr. Bible, "is to produce accurate information on an uninterrupted basis for Searle R&D scientists. We need reliable, yet sophisticated instruments to meet that responsibility. In NMR, the record speaks for itself: we've purchased nine Varian instruments since 1961."

Look at Varian's record. "Overall, Varian's performance, support and service have been excellent in helping us solve problems related to the synthesis and development of new chemical compounds. For example, we now routinely run 2D NMR on Varian's XL-200 supercon system to determine the structure and conformation of potential pharmaceutical products. Since 1961, we've run more than 250,000 NMR spectra at Searle, all on Varian instruments."

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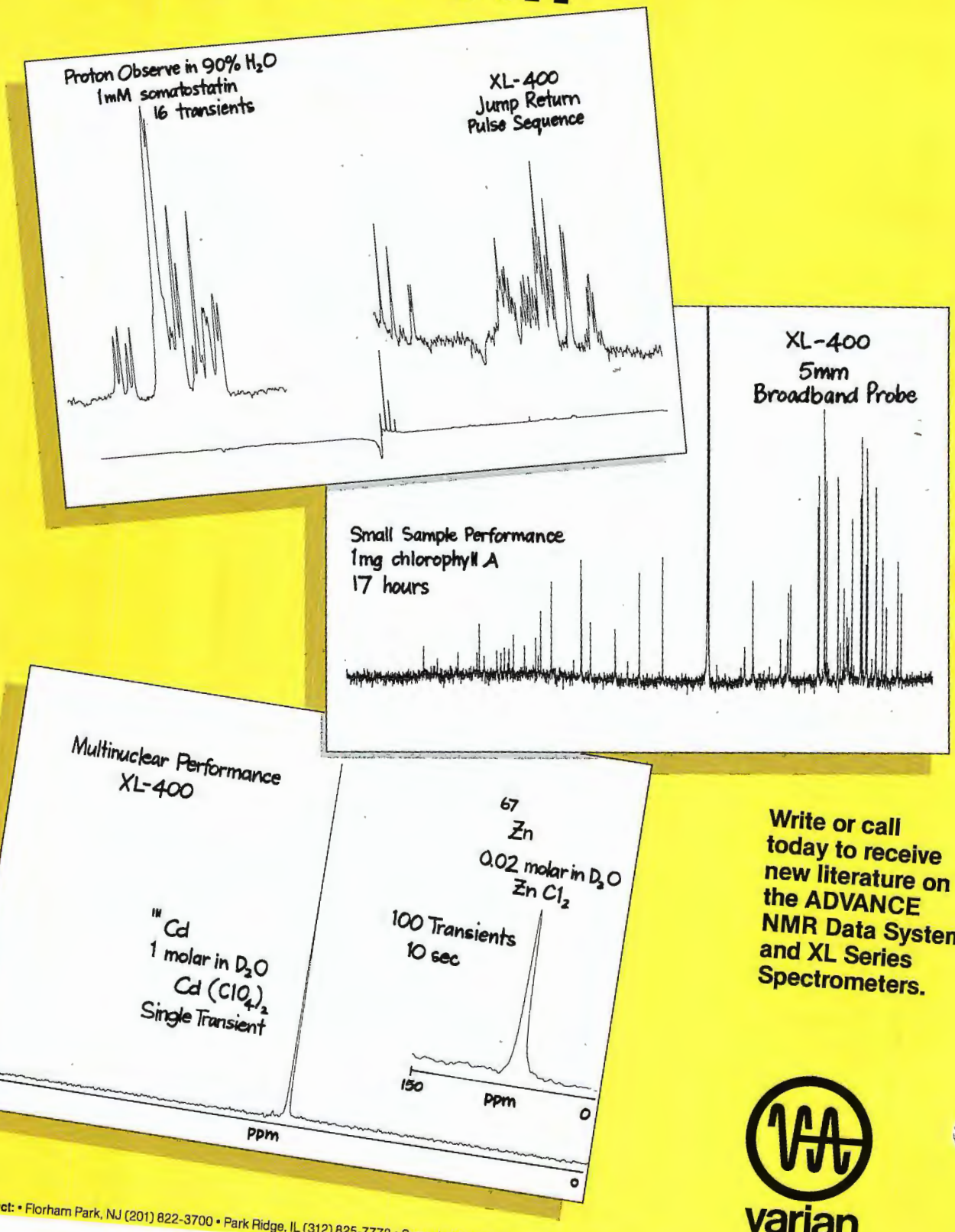

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ISTITUTO SUPERIORE DI SANITA'
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Laboratorio di Biologia Cellulare

Rome, December 16th 1983

TELEGRAMMI ISTISAN - ROMA
TELEX RMOTI ISTISAN

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

Proceedings of the EEC Workshop
on "Identification and Characterization
of Biological Tissues by NMR"
(Rome, May 1983)

Dear Prof. Shapiro,

A Workshop on "Identification and Characterization of Biological Tissues by NMR" was held at the Istituto Superiore di Sanità, Rome (May 18-20, 1983) on behalf of the Working Group on Biomedical Engineering of the Committee on Medical and Public Health Research of the European Economic Community. This Meeting, one of the first of this kind held in Europe, was inspired by the following main objectives: critical review of the physical and physiological background to tissue characterization by NMR; discussion of the most significant research problems involved with the determination of NMR relaxation behaviour in tissues, their biophysical interpretation and diagnostic implications; consideration of NMR methodologies involving the resonance of other nuclei, relevant to tissue characterization; identification of appropriate standardization and calibration methodologies; proposals for coordinated research activities in a joint European cooperation in this field.

The Programme of the Workshop was prepared by Prof. J.S. Orr (London), Prof. K.H. Schmidt (Tübingen), Ir. A.L. Luiten (Eindhoven) and myself. Main Contributors were: C.J.G. Bakker (Utrecht); G.J. Béné (Génève); W.M. Bovée (Delft); J. Chambron (Strasbourg); A.R. Cowen (Leeds); J.D. De Certaines (Rennes); W. Derbyshire (Sunderland); R. Mathur-De Vré (Bruxelles); M. Foster (Aberdeen); A. Haase (Göttingen); J.M. Lhoste (Orsay); A. Luiten (Eindhoven); B. Maraviglia (Rome); K.J. Olsen (Herlev, Copenhagen); J.S. Orr (London); F. Podo (Rome); G. Radda (Oxford); J. Reisse (Bruxelles); K.H. Schmidt (Tübingen); A.L. Segre (Montelibretti, Rome); P. Styles (Oxford); D.G. Taylor (Guildford); M. Villa (Pavia).

The full Proceedings of the Workshop are now in publication in a special issue of the Annali dell'Istituto Superiore di Sanità printed by the Istituto Poligrafico e Zecca dello Stato. The Publisher will print only a limited number of extra-copies, besides those reserved for the Annali's mailing list (subscribers and exchanges). The number of extra-copies to be printed out will depend upon the requests of Colleagues or Institutions active in this field. Those who are interested in the proceedings can (without obligation) send me a note, so as they can receive further information on the modalities of acquisition.

yours sincerely,

Franca Podo

(Dr. Franca Podo)

AMHERST COLLEGE
AMHERST • MASSACHUSETTS • 01002

Department of Chemistry
Telephone 413-542-2342

December 28, 1983

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

^{31}P Spectra of Bilayers on an FX-100?

Dear Barry:

Now that I'm back (sigh) from my leave year in Mary Roberts' lab at M.I.T., we've been busy isolating two enzymes that munch on triglycerides, and exploring the ^{13}C spectral properties of model mixtures for fat digestion. Our most surprising result, however, is the ^{31}P spectrum (Fig. 1) of a 1:1 bile salt-egg lecithin (BS-L) mixture which was obtained on our JEOL FX-100Q (high resolution, liquid state) spectrometer. Although we don't have enough decoupling power to take the lineshape seriously, an extended bilayer structure certainly seems to be present in these close-to-optically clear, dilute BS-L solutions. The formation of a variety of aggregates, also indicated by quasielastic light scattering and electron microscopy, is summarized schematically in Fig. 2. Sorting these out and examining their susceptibility to lipolytic enzymes should keep us out of trouble 'till ENC, at least.

Very truly yours,

Ruth

Ruth E. Stark
Assistant Professor
of Chemistry

RES/gtc

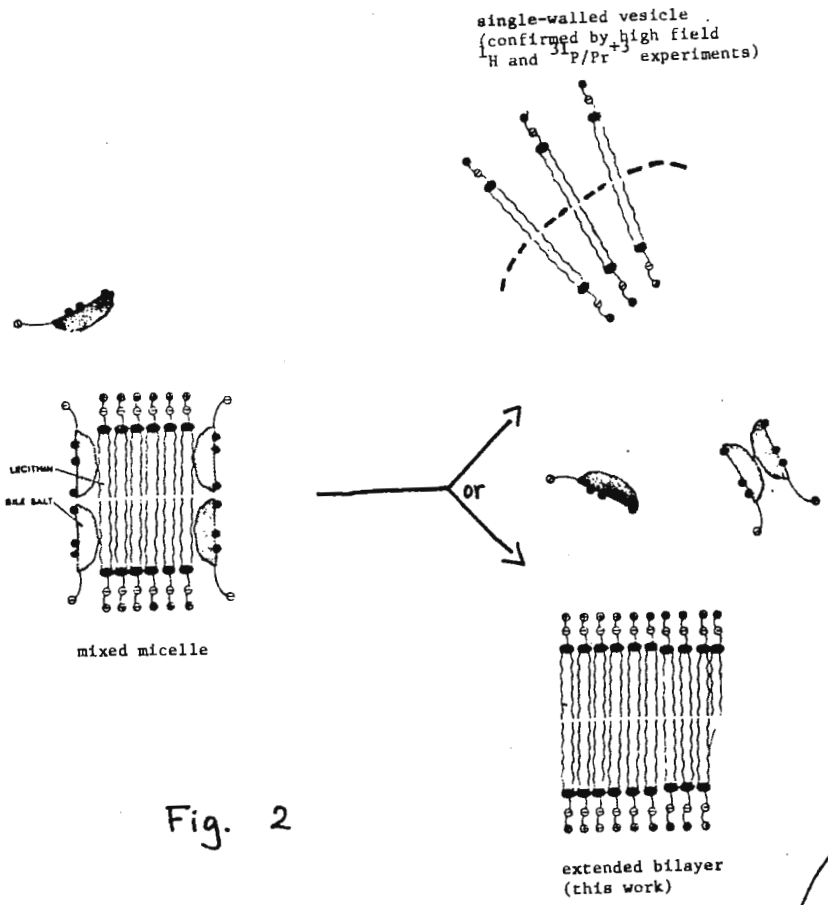

Fig. 2

^{31}P NMR
1:1 Taurocholate - Egg Lecithin
(1.7 mM each)
12 hr accumulation

Fig. 1

304-30

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DEPARTMENT OF CHEMISTRY
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813:974-2144
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December 5, 1983

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

Dear Barry:

For some years now, the program we wrote in your laboratory (LISA) some time ago has been available to people needing it as a FORTRAN listing. Numerous people have requested it and I have tried to be cooperative; however, the logistics in moving large amounts of paper and providing adequate documentation are often prohibitive.

A solution to the above problem is now available via microcomputers. The program is currently available in an APPLE II+ version and will soon be available in an IBM PC version. For the APPLE II+ it is necessary to have a disk drive (two are better) and 48K of memory. For the IBM PC two disk drives and 128K memory would be preferable.

Any person desiring copies of these programs is welcome to them provided he sends me a disk (double density, 5.25" diameter) and a stamped self-addressed envelope with adequate return postage to cover the cost of mailing a disk. At present only delivery of the APPLE II+ version can be guaranteed; the IBM PC version will not be available until after Feb. 1.

The program analyzes various fast-exchange equilibria and provides equilibrium constants, bound shifts, and statistical estimates of errors for these quantities.

Sincerely yours,

Milton D. Johnston, Jr.
Associate Professor of Chemistry

SUGGESTED TITLE: MICROLISA

Monsanto

PHYSICAL SCIENCES CENTER

Monsanto Company
800 N. Lindbergh Boulevard
St. Louis, Missouri 63166
Phone: (314) 694-1000

December 8, 1983

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

Dear Barry:

NMR STAFF POSITION AVAILABLE

A permanent position is available in the Physical Sciences Center of Monsanto Company (St. Louis, Missouri) for an rf and digital electronics engineer. The successful candidate will be a part of my NMR group (six permanent staff), and will report directly to Bob McKay. The main responsibilities of the job include maintenance and design of rf, microwave, and digital circuits used in our efforts to find new analytical applications of high-resolution solids nmr to chemical and biological systems.

Qualifications for the job could include extensive field experience and a modest formal training background; or an advanced degree in electronics and as little as two years' experience in the field. Experience with nmr spectrometers is desirable but not a pre-requisite. Commensurate with qualifications and experience, starting salary will reflect a competitive industrial scale.

Interested candidates should write directly to:

Mr. Michael J. Kunst, Personnel Manager
Monsanto Company
800 N. Lindbergh Blvd.
St. Louis, MO 63167

Sincerely,

Jake

Jacob Schaefer

JS:ct

University of East Anglia

Professor R.K. Harris

School of Chemical Sciences
University of East Anglia
Norwich, NR4 7TJ, ENGLAND

Telephone Norwich (0603) 56161
Telegrams UEANOR NORWICH

RKH/JH

8th December, 1983

Dear Barry,

Vacancy for a Senior Research Associate

This University has a Bruker CXP 200 spectrometer, with facilities for both high-resolution and high-power work on both solids and solutions. In connection with this system there is a vacancy for a post-doctoral Senior Research Associate (SRA) for one year (possibly more) as from 1.2.84. or thereabouts. The position will involve day-to-day responsibility for the CXP 200, with a small amount of service work for organic and inorganic chemists, but the major involvement will be with two research projects:-

1. The development and application of high-resolution ^{1}H (and ^{19}F) NMR of solids, using multiple-pulse (WAHUHA etc.) techniques with magic-angle rotation (MAR).

2. Multinuclear studies of quadrupolar nuclei in solution, with the emphasis on obtaining accurate representations of broad lines. Transition metals such as ^{59}Co and ^{63}Cu, together with ^{17}O and ^{33}S, are of particular interest.

The SRA would have access to our two home-built cross-polarization/MAR spectrometers for high-resolution ^{13}C studies on solids, as well as use of the CXP 200 for similar work on ^{29}Si and ^{31}P. The School also has, inter alia, two JEOL FX 100 spectrometers.

Salary will depend to some extent on experience but should commence at ca. £8000 p.a. with an increment in August 1984. Suitably qualified applicants who wish to spend a year (working hard) in the delightful historic environment of Norwich should write to me, sending a curriculum vitae, as soon as possible.

With best wishes,

Yours sincerely,

Robin Harris

ENC Inc. **Twenty-fifth Experimental Nuclear Magnetic Resonance Spectroscopy Conference**
Wilmington, Delaware, April 8-12, 1984

December 12, 1983

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

Dear Barry:

Please include the attached meeting announcement in TAMU NMR Letters.

> The 25th Experimental Nuclear Magnetic Resonance Spectroscopy Conference (ENC) will be held at the Radisson Hotel in Wilmington, Delaware, on April 8-12, 1984. Information for Registration will be sent to all previous ENC attendees (last five years) and is also available from the ENC Secretary, Dr. R. G. Bryant, Department of Chemistry, University of Minnesota, Minneapolis, MN 55455, 612/373-5575. The 26th ENC will be held on April 20-25, 1985, at Asilomar, California, and the 27th ENC is scheduled to be held April 13-17, 1986, at the Baltimore Hilton in Baltimore, Maryland.

Thank you.

Yours sincerely,

Frank

Frank A. L. Anet

FALA/cr

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12 December 1983

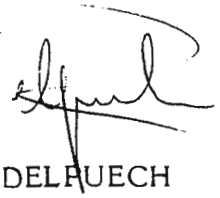
Professor Bernard L. Shapiro
Department of Chemistry
Texas A&M University
College Station, TX 77843

Postdoctoral Position
Available

Dear Professor Shapiro,

TAMU Newsletters readers may be interested by the following. A research position, sponsored by the Centre National de la Recherche Scientifique (CNRS) is available in the area of NMR spectroscopic characterization of coal and coal products (Bruker CXP-200 and AM-400). Some experience in the field of high-resolution NMR spectroscopy is required. Salaries : French Francs 8200 F per month (about $ 1000). The position may be immediatly occupied for one year. Applications with full curriculum vitae and two letters of recommendation should be sent to : Prof. J-J. Delpuech, Université de Nancy I, Faculté des Sciences, Laboratoire de Chimie Physique Organique, BP 239, 54506 VANDOEUVRE-LES-NANCY CEDEX, FRANCE.

Sincerely yours,



J.-J. DELPUECH

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(617) 667-9580

December 16, 1983

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

"Position Available"

Dear Dr. Shapiro:

We are expanding our staff and have positions for one or two Applications Chemists currently available. Required is profound experience in NMR as well as a genuine interest in the field of marketing and customer contact.

Please send resumes to my attention.

Sincerely,

Gerd Wolff
Vice President
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Los Alamos National Laboratory

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To apply, please submit resume and letters of recommendation in confidence to:

Madeline Lucas, DIV– 84-24
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