TEXAS A&M UNIVERSITY

NMR NEWSLETTER

NO. 333

JUNE 1986

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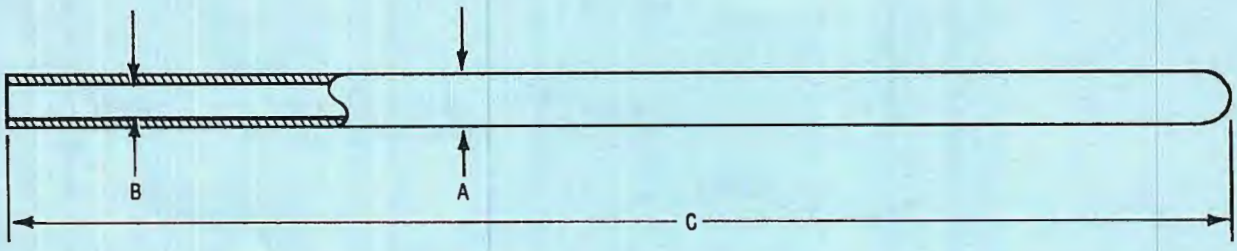

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540-PPT	Royal Imperial					0.001″	0.0005″
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TEXAS A&M UNIVERSITY
NMR
NEWSLETTER

AUTHOR INDEX -------- TAMU NMR NEWSLETTER, NO. 333, JUNE 1986

ADVERTISERS

FORTHCOMING NMR MEETINGS (Additional listings are solicited)

28th Rocky Mountain Conference - August 3-7, 1986; Radisson Hotel; Denver, Colorado; Conference Chairman: R. Barkley, CIRES, University of Colorado, Boulder, Colorado 80309, (303) 492-1158. Abstract Deadline: March 21, 1986. NMR Chairmen: J. Haw, Dept. of Chemistry, Texas A&M University, College Station, Texas 77843, (409) 845-1966, and F. Miknis, Western Research Institute, Box 3395, University Station, Laramie, Wyoming 82071, (307) 721-2307.

Gordon Research Conference on "Magnetic Resonance in Biology and Medicine" - August 4-8, 1986; Tilton School, Tilton, New Hampshire; Chairman: Paul D. Ellis, (University of South Carolina); Attendance applications to Dr. Alexander M. Cruickshank, Director; Gordon Research Conferences; Gordon Research Center; University of Rhode Island; Kingston, Rhode Island 02881-0801; (401) 783-4011 or (401) 783-3372; Office - Summer Schedule: Colby-Sawyer College; New London, New Hampshire 03257; (603) 526-2870; Poster Session Chairman: Professor N. Dennis Chasteen; Department of Chemistry; University of New Hampshire; Durham, New Hampshire 03824.

XXIII Congress Ampere on Magnetic Resonance - September 15-19, 1986; Rome, Italy; XXIII Congress Ampere, Dipartimento di Fisica, Universita de Roma, "La Sapienza," P. le Aldo Moro 5, I-00185 Roma, Italy.

Federation of Analytical Chemistry and Spectroscopy Societies (FACSS XIII) - September 28-October 3, 1986; St. Louis, Missouri; Program Manager: Dr. Sydney Fleming, FACSS (Titles), 24 Crestfield Road, Wilmington, Delaware 19810.

1986 Eastern Analytical Symposium - October 20-24, 1986; Hilton Hotel, New York; see Newsletter No. 329, p. 23 and Newsletter No. 325, p. 27.

All Newsletter Correspondence
Should be Addressed to:

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

DEADLINE DATES

No. 335 (August) --------- 25 July 1986

No. 336 (September) ---- 26 August 1986

University of Alberta
Edmonton
Canada T6G 2H7

Department of Biochemistry
474 Medical Sciences Building, Telephone (403) 432-5460

Professor Bernard L. Shapiro
Texas A&M University
Department of Chemistry
College Station, Texas 77843-3255

"MacNMR" Programs Available.

Dear Barry:

In an attempt to produce an educational 'tool' to help the NMR spectroscopist understand all the new pulse sequences, we have developed a Macintosh computer program which shows the response of the magnetization vectors during any series of pulses or precession periods. The vector display is based upon a density matrix calculation. In keeping with Mac philosophy we have, of course, called these programs MacNMR. We have a homonuclear AX and heteronuclear AX spin system simulation available.

Two of the screens displayed by this program are shown in the attached Figure 1. The top screen is the main screen and shows the menu for pulse or precession selection, a partial summary of the pulse sequence, the density matrix in diagramatic form, and the magnetization vectors. The bottom window shows the complete summary of the pulse sequence and the density matrix in numeric terms. The menubar across the top of these windows allows the user to enter parameters, enter arbitrary pulse or precessions, phase cycling, relaxation, printing the screen, and an online help facility. This figure shows the result of a N-15 / H-1 INEPT pulse sequence. Notice the ratio of intensities of the N-15 lines is -11/9.

We are prepared to distribute these programs to interested users. If you want a copy of both versions, please send $20 to the above address to handle costs.

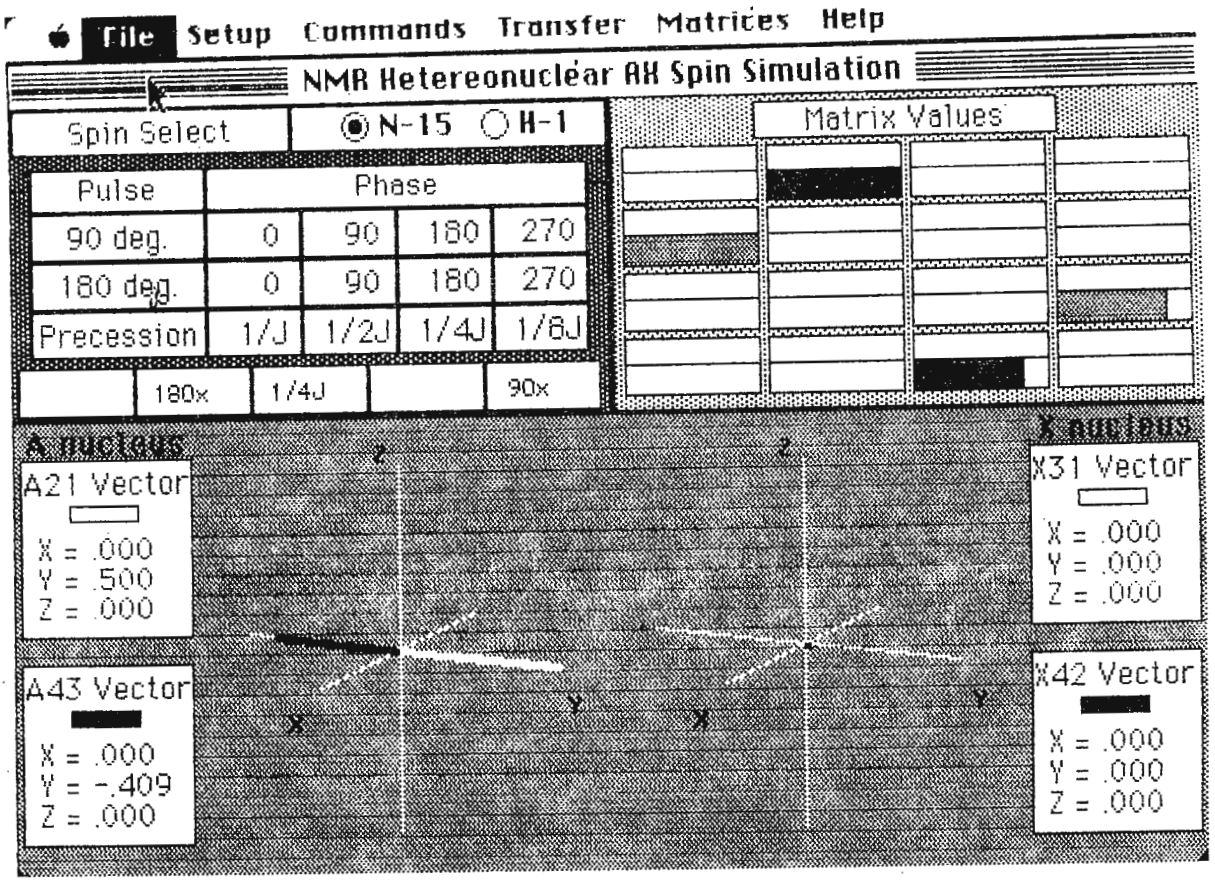
Best regards

Brian

Brian Sykes

Robert Boyko

Robert Boyko


File Setup Commands Transfer Matrices Help
NMR Hetereonuclear AX Spin Simulation
Spin Select
N-15
H-1
Matrix Values
Pulse
Phase
90 deg.
0
90
180
270
180 deg.
0
90
180
270
Precession
1/J
1/2J
1/4J
1/8J
180x
1/4J
90x
A nucleus
X nucleus
A21 Vector
X = .000
Y = .500
Z = .000
A43 Vector
X = .000
Y = -.409
Z = .000
X31 Vector
X = .000
Y = .000
Z = .000
X42 Vector
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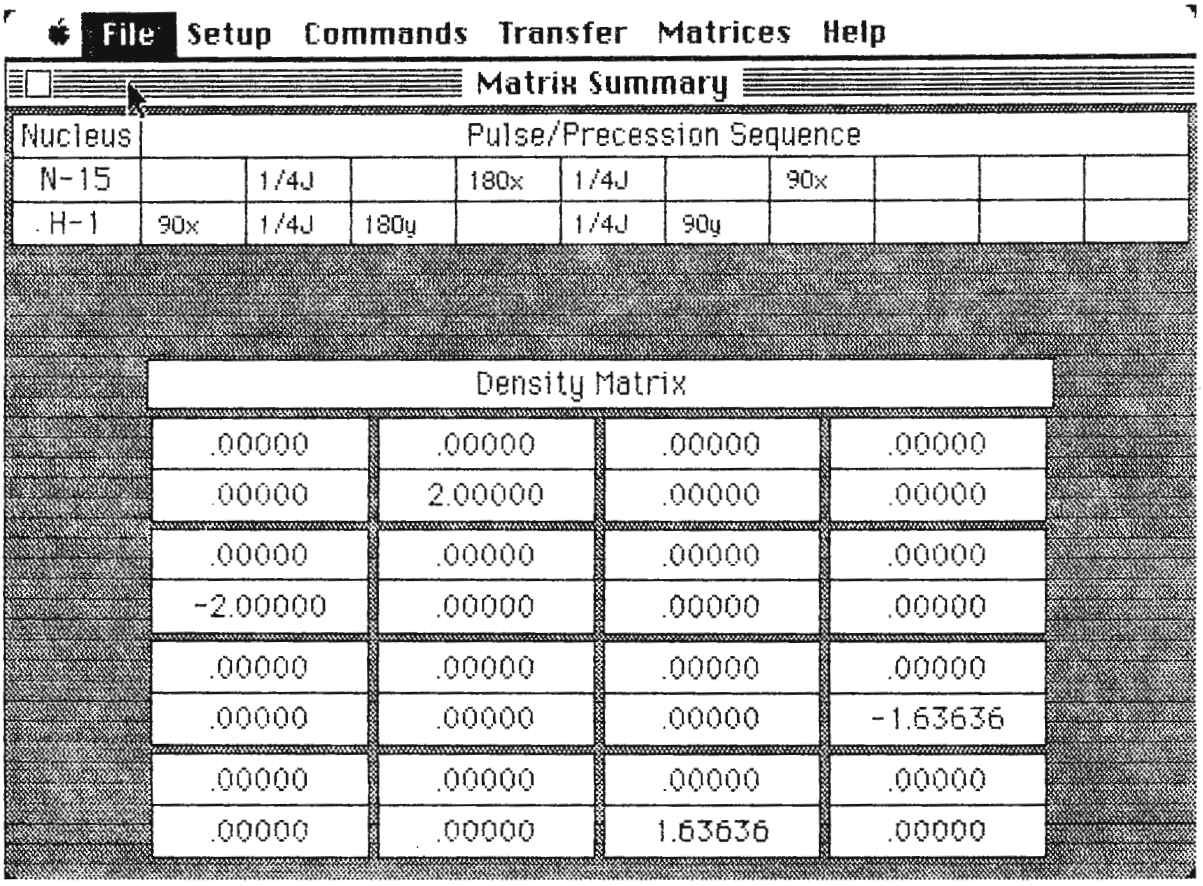
Y = .000
Z = .000
Z
Y
X
Z
Y
X


File Setup Commands Transfer Matrices Help
Matrix Summary
Nucleus
Pulse/Precession Sequence
N-15
1/4J
180x
1/4J
90x
H-1
90x
1/4J
180y
1/4J
90y
Density Matrix
.00000 .00000 .00000 .00000
.00000 2.00000 .00000 .00000
.00000 .00000 .00000 .00000
-2.00000 .00000 .00000 .00000
.00000 .00000 .00000 .00000
.00000 .00000 .00000 -1.63636
.00000 .00000 .00000 .00000
.00000 .00000 1.63636 .00000

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B L Shapiro
Texas A & M University
Department of Chemistry
College Station
Texas 77843 3255
USA

21st March 1986

NMR Imaging in Inhomogeneous B_o-Magnetic Fields

This first contribution from the Fens at Cambridge contains some local news, plus some results obtained by Tim Norwood whilst he was working for his MSc thesis in my Vancouver Laboratory.

Since 1980 our interest in combining NMR-spectroscopy with NMR-imaging has alerted us to the technical problems, and the associated costs, involved in obtaining large volumes of high homogeneity B_o field and has prompted us to consider a number of different approaches. One of these is based on the well known fact that the line-widths of zero-quantum coherences are independent of B_o inhomogeneity.

A zero-quantum (ZQ)-resolved image can be displayed by superimposition of the image of a species on top of it s ZQ-coherence. However, this may give rise to two problems: first, the multiplet structure of the ZQ-resonance will distort the corresponding image; second, the images from adjacent ZQ coherences may overlap.

The image shown in A, which is from six tubes containing a mixture of ethanol and 2-propanol, exhibits both these problems. As shown in B, the first problem can be eliminated by performing a broad band decoupled ZQ imaging experiment, so that discrete image components can be seen. However, the two images still overlap; fortunately it is a trivial matter to edit the ZQ responses such that separate images can be obtained for ethanol (Fig. C) and 2-propanol (Fig. D).

At long last I am making progress in establishing my new Department and I wonder if I might take this opportunity to draw attention to the fact that I shall shortly be advertising two academic posts (Assistant Prof = lecturer) plus an electronics engineer; all these posts are already funded and I hope that the appointments will be made as rapidly as possible. I already have four PhD studentships here plus a technician and other graduate fellowships will be avaialble for October 1st 1986; some Postdoctoral Fellowships will follow. Interested persons should write to me at Cambridge.

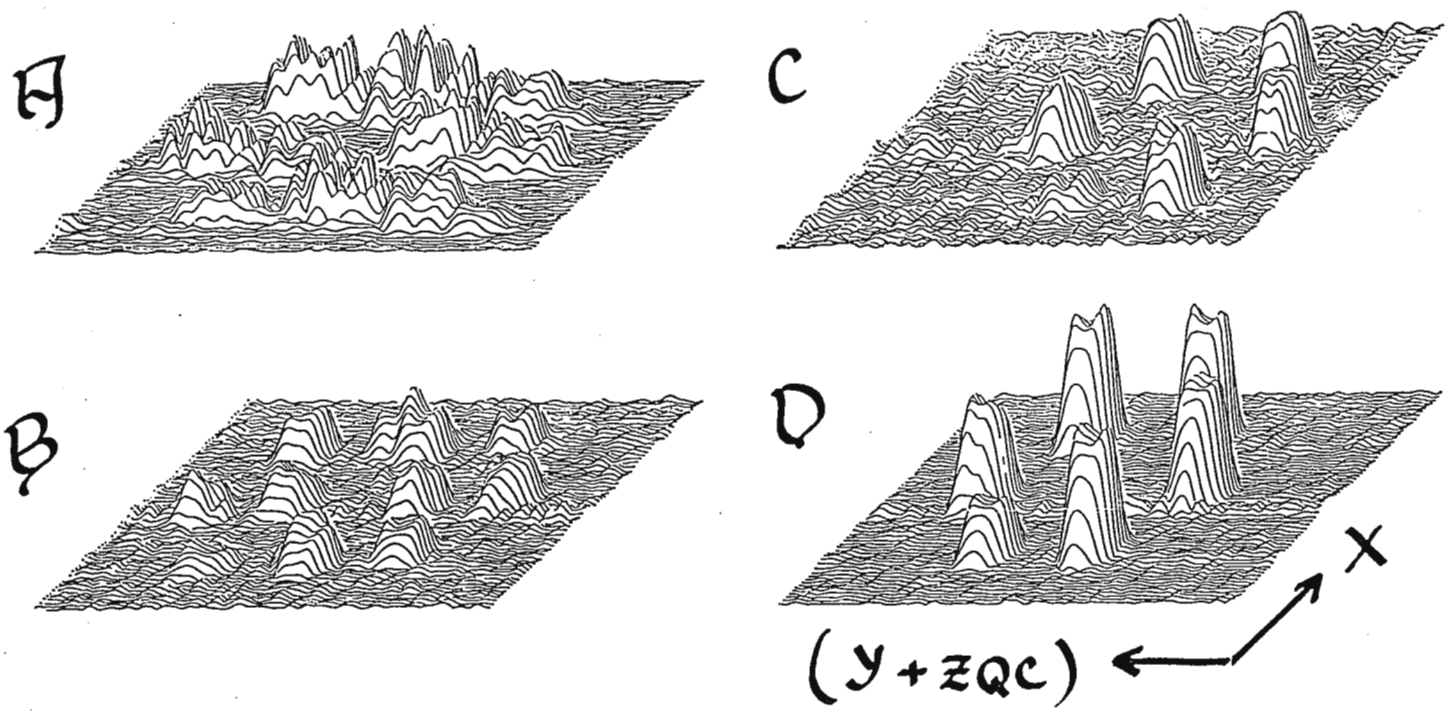
Since I have not closed down my Vancouver Laboratory it is not yet appropriate for me to write a "farewell" letter. Suffice it to state for the present that my Canadian students have behaved magnificently, as have my colleagues and the Canadian funding agencies. It would be unfair to take up valuable space here to give details either of the facilities available at Cambridge or my plans, but I would be very glad to provide information for anyone who is interested.

With all best wishes

Tim

L D Hall

T J Norwood



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(617) 667-9580

April 28, 1986

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

RE: Position Avaiable

Dear Dr. Shapiro,

We have an immediate opening in our NMR Software Development department for a person to develop acquisition and processing software for our NMR Spectrometers and Data Stations. We are seeking someone with an advanced degree in chemistry and a strong computer background. Assembly language experience is necessary and high level language (preferably Pascal or C) experience is a plus. Those interested should send their curicula vitae to my attention at the above address.

Sincerely,

Charles Thibault, Ph.D.
Software Coordinator

CTH/ec

THE J. HILLIS MILLER HEALTH CENTER
UNIVERSITY OF FLORIDA
GAINESVILLE, FL 32610

NMR Resource
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E.R. Andrew
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L.F. Brateman

College of Medicine
Department of Radiology
Box J-374
(904) 395-0293

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

April 10, 1986

Short title: S/N in imaging

Dear Professor Shapiro:

Optimizing the signal-to-noise ratio (S/N) of an NMR image can be a tricky procedure. One is faced with a number of compromise choices such as resolution, repetition rate, echo time, etc. We have been investigating one aspect of this problem and would like to discuss this with your readers.

A naive reading of Abragam (1) would lead one to assume that the noise in an image would be proportional to $\sqrt{4kTR\Delta\nu}$, where R is the shunt resistance of a tuned circuit and $\Delta\nu$ is the bandwidth of the receiver. The results for a test of this assumption are shown in Figure 1. This represents the S/N in a one-dimensional projection image measured in the frequency spectrum. The 'bandwidth' in this case is the spectral width with matched audio filtration. The applied gradient and spatial resolution were held constant as the 'bandwidth' varied. This results in a constant frequency resolution in the resulting spectrum. Within experimental error the S/N is constant. Upon reflection, one can see that this is the expected result. The noise voltage described above would be that measured at the terminals of the receiver at an instance in time. The noise apparent in the projected image has been frequency analyzed by the Fourier transformation process into narrow frequency bandwidths centered about each data point. Since the frequency resolution (and hence frequency bandwidth per data point) is constant, changing the spectral width should not change the frequency components of the noise in a fixed region of the spectrum.

The implication of the above result is that the S/N will be dependent on the frequency bandwidth per data point and not the width of the observed spectrum. This has been tested and the results are shown in Figure 2. The three curves represent three values of the spatial resolutions. The S/N has the expected linear dependence on the spatial resolution. All the curves have a slope of $-1/2$ which indicated that the S/N will have the following dependence on the bandwidth per data point, $\Delta\nu_f$.

$$S/N \sim (\Delta\nu_f)^{-1/2}$$

Upon reflection one can see that this has important implications for imaging at high magnetic fields where chemical shift effects begin to appear. To obtain the best possible S/N in an image one should not try to overcome chemical shift effects with gradients, but use chemical shift as additional information.

We find ourselves in the unusual situation of having more positions opened than personnel to fill them. We would like to encourage any persons with an interest in imaging, localized spectroscopy and high-resolution spectroscopy research to apply to the address above. We have positions for graduate students (~2), a spectroscopist and biochemist.

(1) A. Abragam, "The Principles of Nuclear Magnetism," Oxford University Press, 1961, p.83

Sincerely yours,

T.H. Mareci, Sune Dønstrup and J.R. Fitzsimmons

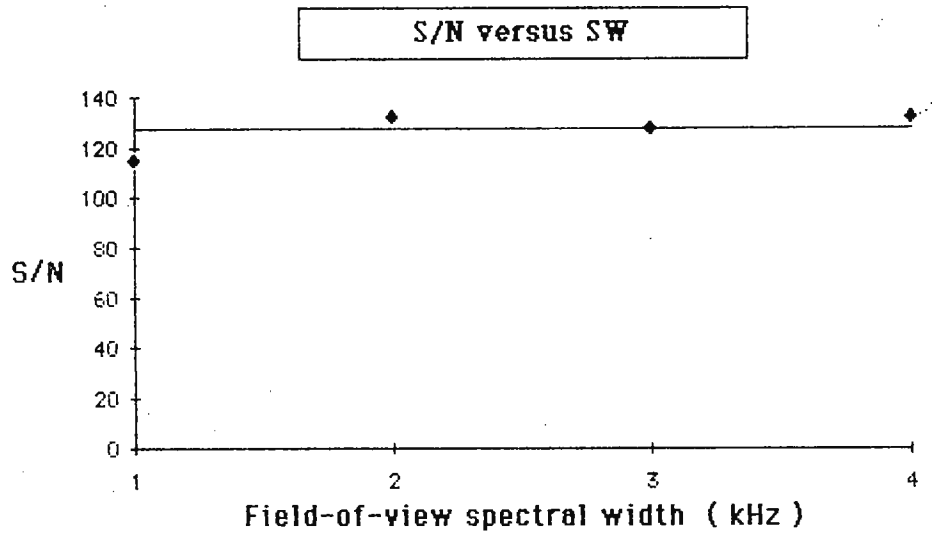


Figure 1

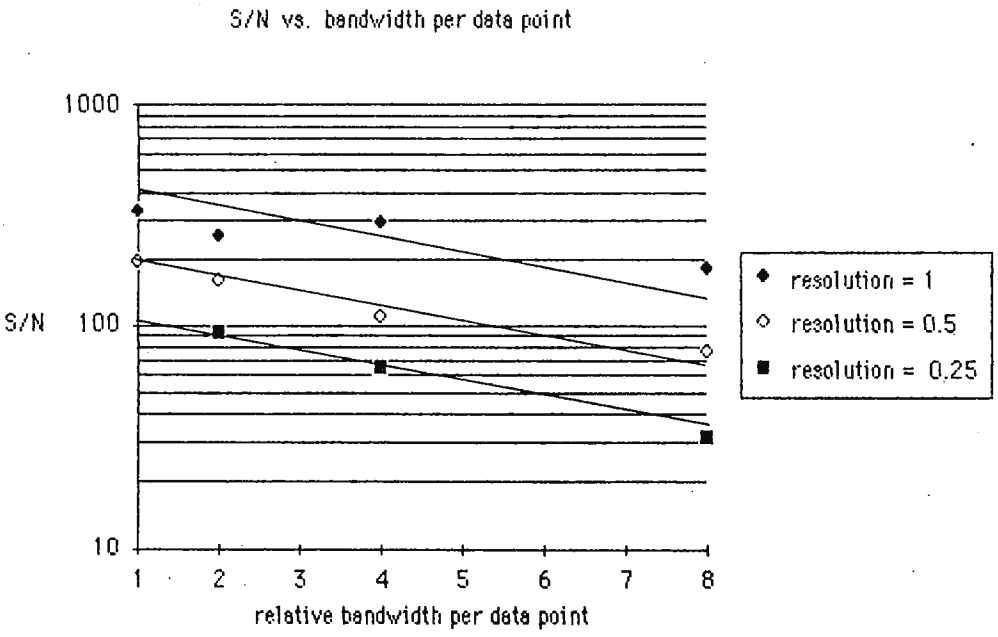


Figure 2

UNIVERSITE DES SCIENCES ET TECHNIQUES DU LANGUEDOC

GROUPE DE DYNAMIQUE DES PHASES CONDENSEES
Laboratoire Associé au C.N.R.S. N° 233

Montpellier, April 5 , 1986

Professeur Bernard L. Shapiro
Department of Chemistry
Texas A and M University
College Station, TX 77843
U.S.A.

Effect of cellular death and dehydration on NMR relaxation rates of guinea pig lymphocytes

Dear Professor Shapiro,

The user of an imaging system based on nuclear magnetic resonance (NMR) has to know tissue NMR characteristics, thus being able to predict the contrast between organs of interest in the image, or between healthy and pathological structures. One way to determine these parameters is to directly measure them from the imaging system itself ; but the "two points" technique most commonly used to that purpose is too much inaccurate (50 to 100 % inaccuracy)[1] thus leading to erroneous predictions. Another way is to make accurate determinations from ex vivo anatomical pieces of organs, into an NMR spectrometer ; the question is, in this case, to estimate the variations suspected from post mortem effects and survival properties of biological organs.

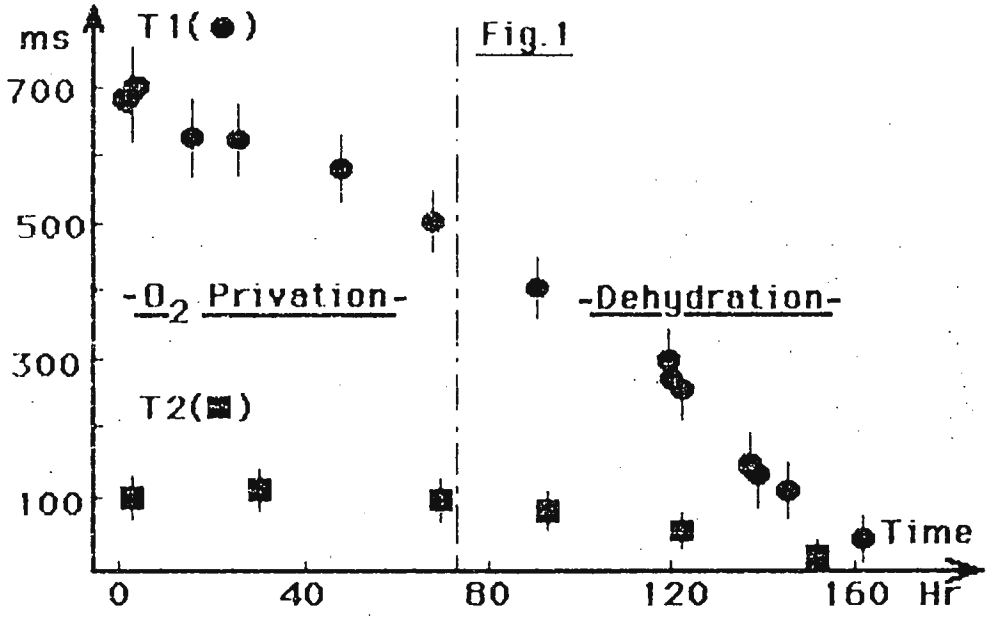
We thus investigated the post mortem behaviour of a cellular pellet of alive lymphocytes by measuring the NMR ^1H relaxation times T1 and T2, considering separately the effects of cellular death by privation of oxygen and metabolites, and the ones of dehydration of the pellet in ambient air. The results obtained from different experiments are shown in fig. 1, where the time scale represents the delay after cell collecting and pellet formation. The decrease of T1 and T2 relaxation times under dehydration in ambient air seems to be independant on the occurrence or not of the cellular death.

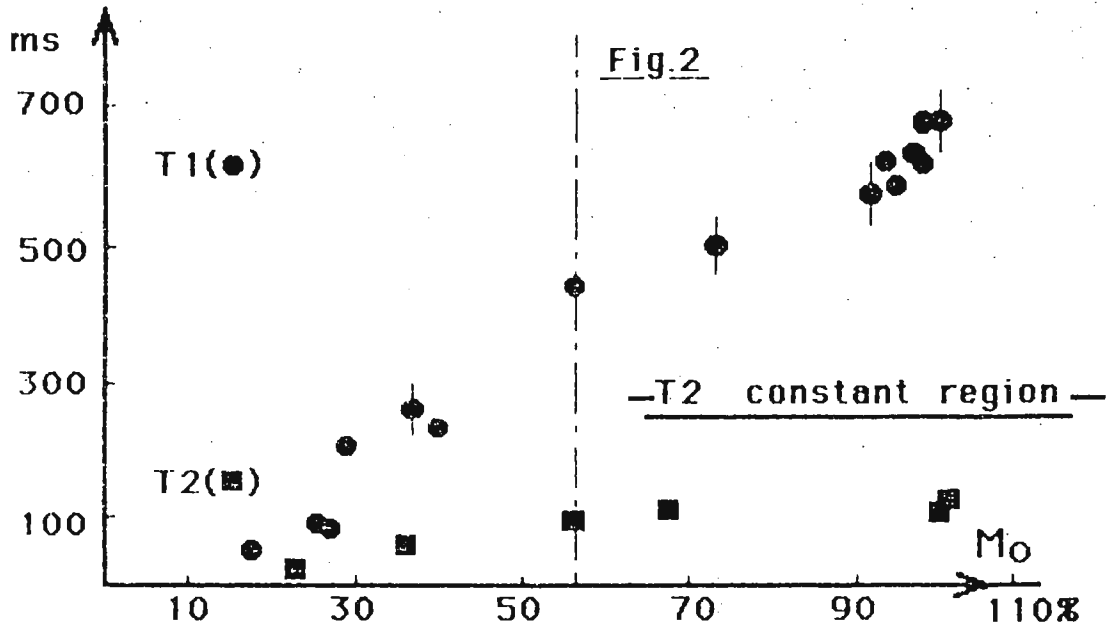
The decrease of T1 during cellular death by O_2 privation is significantly different, T2 being constant.

The processes affecting T1 and T2 during dehydration may be linked to disappearance of free water from the cells, but are not yet completely understood for cellular death by O_2 privation .

In the first 30 hours, under O_2 privation, the total variation of T1 is less than 15 % in that model [2], corresponding to the minimum of magnitude of T1 standard deviation when measured into an imager.

We conclude from these experiments that under given conditions of experience


ms
T1(●)
Fig.1
700
500
-O2 Privation-
-Dehydration-
300
T2(■)
100
Time
0
40
80
120
160 Hr


ms
Fig.2
700
T1(●)
500
300
-T2 constant region-
T2(■)
100
Mo
10
30
50
70
90
110%

(delay after excision...) one may use T1 determinations from ex vivo organs with sufficient accuracy, thus leading to predictions good enough for image contrast on an NMR Imager system.

The fig. 2 shows the almost linear relationship between T1, T2 and the water content [2,3,4] estimated from the maximum magnetization M_0. When the cellular pellet was apparently dried, (after 162 hours) T1 was constant at about 40 ±10 msec, corresponding to a water content of about 17 %. This fraction of water may correspond to bound water, and from Fullerton et al [5] we could expect for this fraction a T1 for bound water fraction of about 50 msec at 15 MHz, not so far from our results...

We are developping in Montpellier a fruitful cooperation between NMR physicists and chemists in the Sciences School and Medical Biophysicists and Radiologists at Medical School and Hospital, so promoting the necessary interdisciplinary cooperation needed for this delicate technique.

Sincerely,

Michel Zanca,
MD, Biophysicist

Patrick Bernier
PhD , NMR Physicist

(1) G.M. Glazer et al, Radiology , **155**, 2 (1985)419-420
(2) J. de Certaines, J.Biophys. Med. Nucl. , **5** (1981), 107-116
(3) R.Ader and J.S. Cohen, J. of Magn. Res. , **34** (1979), 349-356
(4) P. Mansfield and P.G. Morris, "NMR imaging in Biomedicine", (1982) Ac. Press, Chap. 2, p. 10-29
(5) G.D. Fullerton, I.L. Cameron and V.A.Ord, Radiology , **151** (1984),135-138

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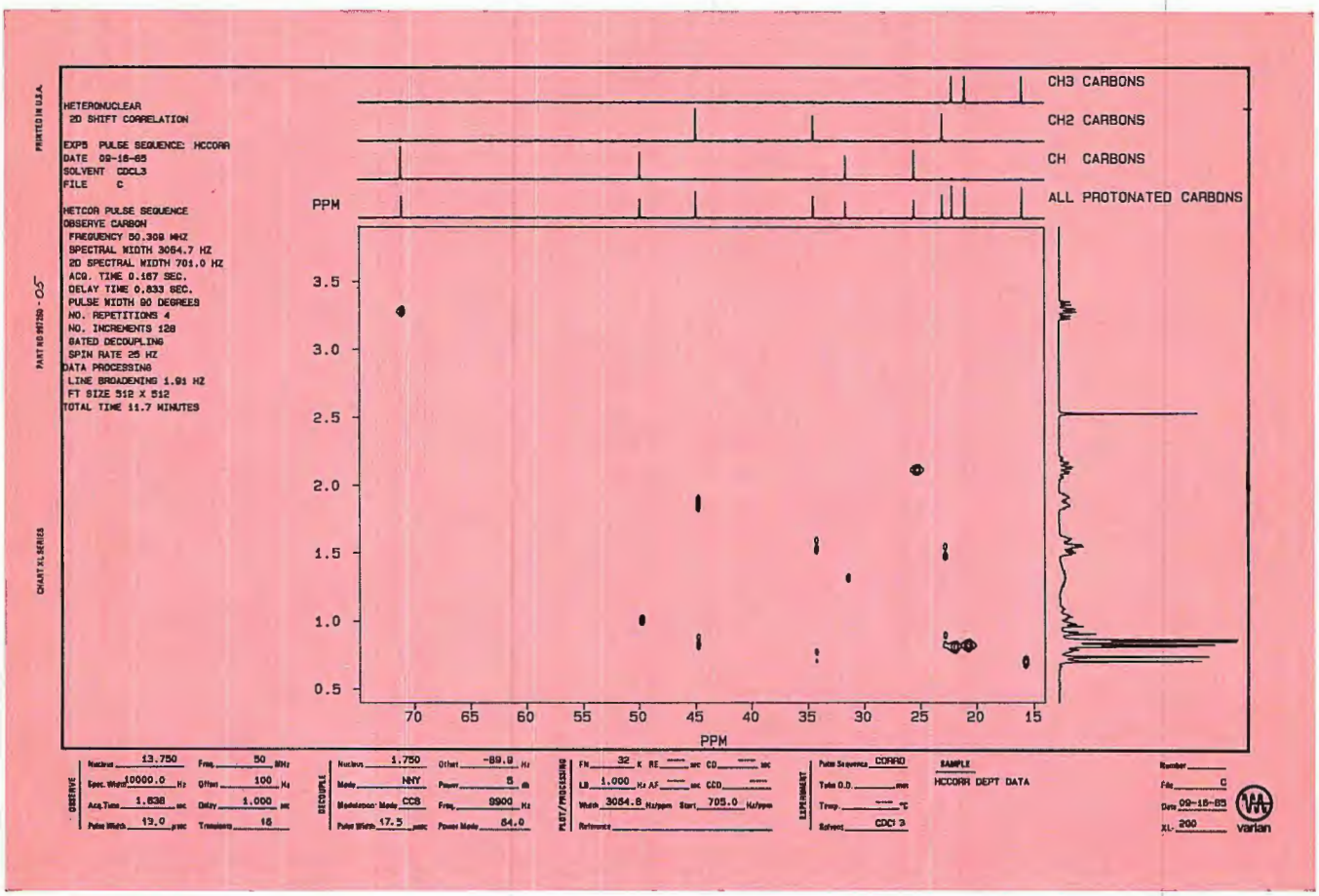

AUTOMATED HETERONUCLEAR CORRELATION

MAGICAL provides not just automation, but intelligent automation. Here, an automated series of proton, carbon, DEPTGL, and heteronuclear chemical shift correlation spectra were performed on a sample of menthol, all by typing *HCCORR*. Each of these spectra was optimized by MAGICAL. The proton spectrum was checked at the end to ensure that it had sufficient signal-to-noise; if it didn't, the experiment would have been rerun.

For the carbon experiment, where sensitivity is more of a problem, the signal-to-noise ratio was checked periodically during the course of the experiment, and the acquisition terminated when a preselected signal-to-noise ratio was reached. Using this number of scans as a guide, conditions were then chosen for the DEPTGL experiment, and that series of spectra was then acquired and automatically combined (spectral editing) to produce subspectra containing, respectively, protonated carbons, CH carbons, CH_2 carbons, and CH_3 carbons.

With 1D spectral information in hand, MAGICAL next determined the minimum spectral width for the heteronuclear correlation experiment for both protons and carbons (using protonated carbons only, as determined from the DEPTGL experiment). The rest of the 2D parameters were also optimized based on the 1D experiments, and the 2D experiment was then acquired, processed, scaled, and plotted. From start to finish, the entire series of experiments required only 22 minutes. This time will, of course, vary from sample to sample, because the MAGICAL analysis is not "canned," but adapts to the requirements of each particular sample.

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Boston, Massachusetts 02114

NMR LABORATORY
(617) 726-3081

May 15, 1986

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

Minimal Sampling

Dear Barry,

Our minimal sampling procedure (1) is directed toward the goal of shortening a multidimensional data acquisition to the minimum possible time by employing a linear algebraic solution to the task of spectral analysis. The algorithm is useful when the spectral frequencies are all known, and one desires only to measure amplitudes. Spectral frequencies may be complex to allow for Lorentzian lineshapes, or arbitrary lineshapes may be employed. In many cases of practical interest (for example, the t_1 time domain in 2D spectroscopy or the chemical shift dimension in chemical shift imaging) the possible spectral frequencies are known or can be measured in advance.

Briefly, the algorithm assumes only that the signal s be regarded as a linear superposition of basis signals f_k: $s(t_j) = \Sigma_k a_k f_k(t_j)$ where the index j runs over the realtime or nonrealtime samples of the signal at arbitrary (and not necessarily equally spaced) times t_j. Spectral analysis is accomplished by direct algebraic solution of this set of simultaneous equations, and not by a statistical approach such as in, for example, maximum entropy or linear prediction.

The advantage of this algorithm is that the number of signal samples is precisely equal to the number of basis signals, which is ordinarily much less than the number of signal samples required for a useful analysis by Fourier transform.

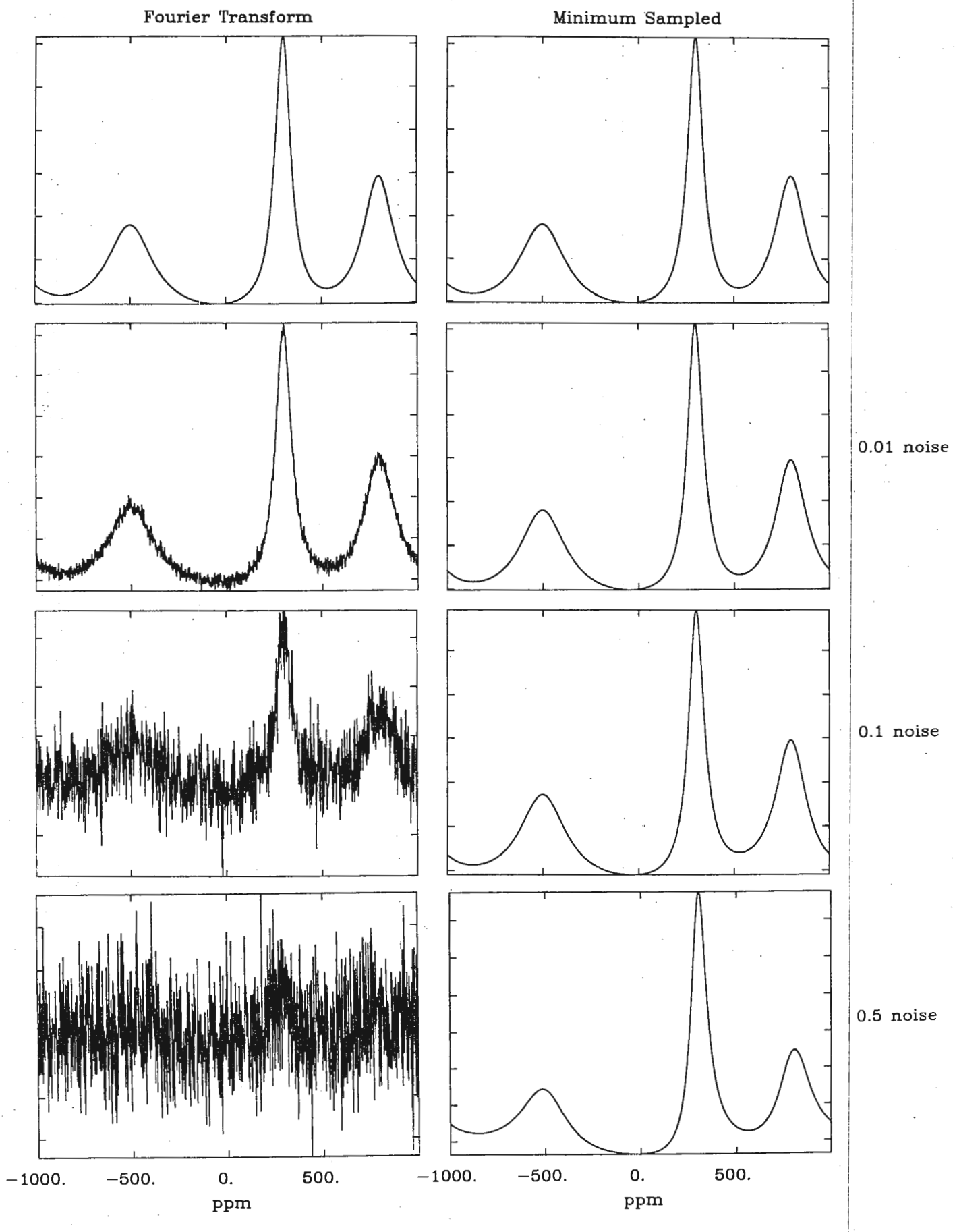
The figure displays the performance on synthetic signals containing three FID's each of unit amplitude. Progressive amounts of white noise (uniformly distributed on the interval [-0.5, +0.5] and scaled by the indicated amount) are added to the FID's, which are subsequently Fourier transformed or analyzed by minimal sampling. The algorithm requires in this case just three points on the FID and provides three amplitudes which are used to draw the right hand spectra for convenient visual comparison.

Sincerely,

Jerry Ackerman

Jerome L. Ackerman

(1) Society of Magnetic Resonance in Medicine, 4th Annual Meeting, London, August 1985.


Fourier Transform
Minimum Sampled
0.01 noise
0.1 noise
0.5 noise
−1000.
−500.
0.
500.
ppm
−1000.
−500.
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ppm

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May 19, 1986

QUADRATURE DETECTION IN 2D DOUBLE QUANTUM COHERENCE SPECTROSCOPY USING 60-DEGREE PHASE SHIFTS

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

Dear Barry;

We have been operating our Bruker AM-500 for over a year, and with it we have solved a number of interesting structural problems using 2-D NMR. In particular, we have performed a number of 2-D INADEQUATE experiments using either our 5 mm dual C/H probe or our 10 mm broadband probe.

The final read pulse in the INADEQUATE sequence relies on flip-angle effects to give quadrature detection in F_1; however, we decided to take advantage of our digital phase shifter, and by phase cycling the final read pulse in 60 degree increments, we are able to achieve quadrature detection in F_1 with a 90 degree read pulse. The 90-τ-180-τ-90 sequence used to create DQ coherence is cycled using 90 degree phase shifts as is the receiver phase which, along with the read pulse phase cycling, gives a 96 step sequence. Piveteau, et al.,[1] have proposed a similar scheme in which all of the pulses are cycled in 60 degree steps, but we have not yet had a chance to try their sequence.

The sesquiterpene shown in Figure 1 supplied by Professor Ray Doskotch of the College of Pharmacy at OSU provided a good opportunity to check out the pulse sequence and the spectrometer performance as a 26KHz spectral window was needed to observe all the signals.

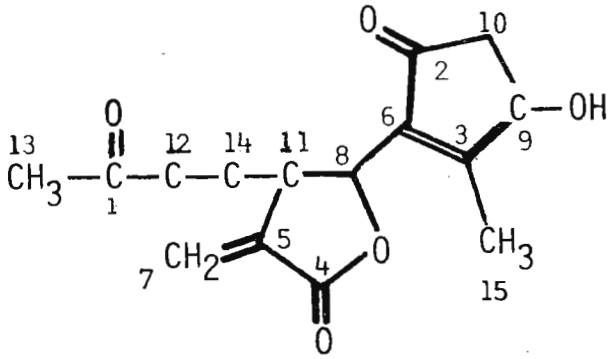

Figure 1

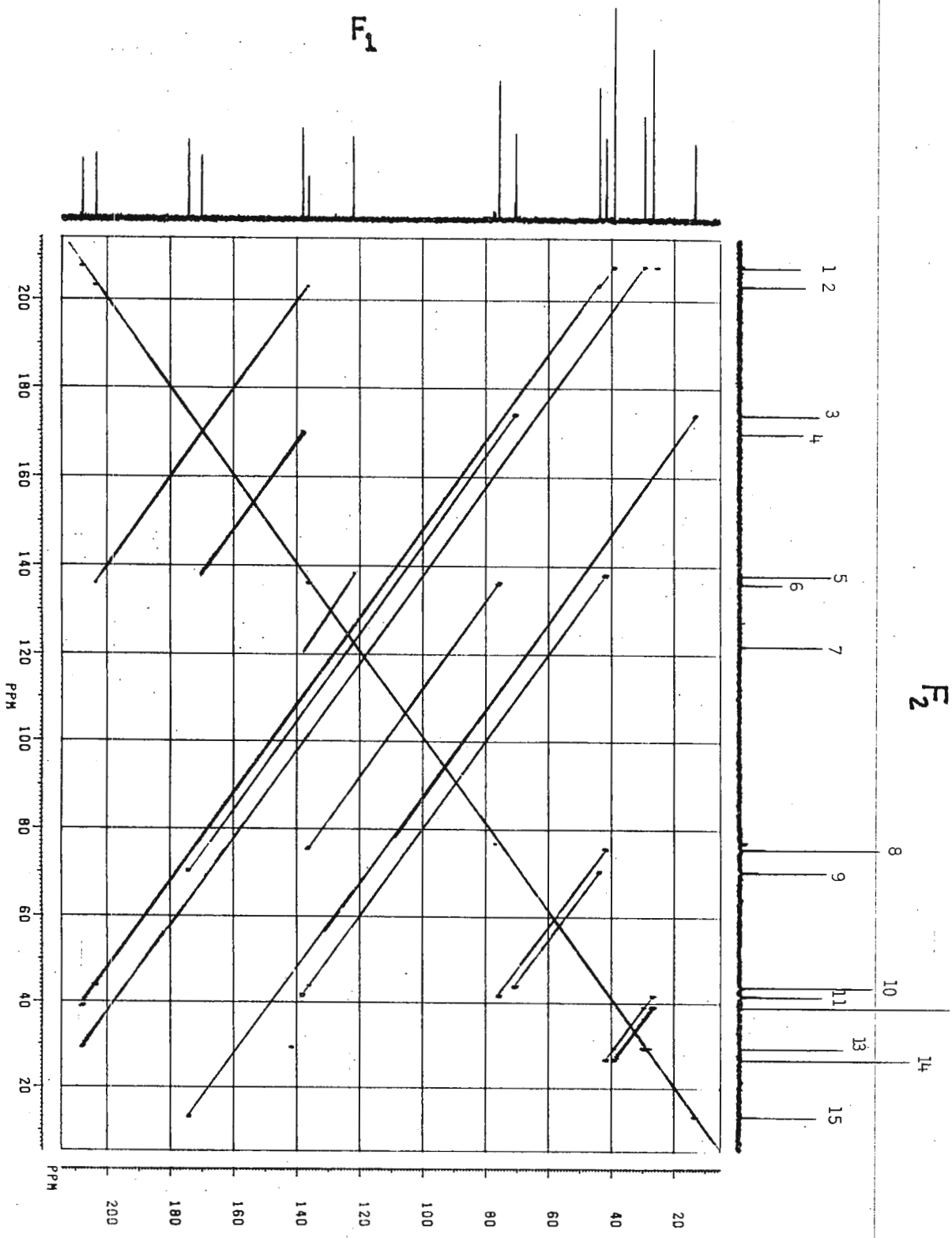

Figure 2. 2-D Double-Quantum contour plot.
1-D spectrum plotted above with assignments.

A contour plot of the entire 2-D spectrum is shown in Figure 2. In this example, we used a second t_1 delay as suggested by Turner[2] to decrease the spectral window in F_1. The only interaction not observed was between the two quaternary carbons C_3 and C_6. Two other pairs of signal C_4-C_5 and C_5-C_7 had a signal on only one side of the diagonal through the spectrum. It is interesting to note that across both of the keto-carbonyls, two bond couplings are observed (C_{12}-C_{13} and C_6-C_{10}).

We are using a similar scheme with 45 degree phase shifts to give F_1 quad detection for 2-D triple-quantum coherence experiments.[3]

Please credit this to Alan Marshall's account.

Sincerely,

Chuck

Charles E. Cottrell
NMR Spectroscopist

1. D. Piveteau, M. A. Delsuc, and F. Y. Lallemand, J. Magn. Res., 63, 255 (1985).

2. D. L. Turner, Molec. Phys., 44, 1051 (1981).

3. C. E. Cottrell, J. M. Beale, P. J. Keller, and H. G. Floss, 27th ENC, Baltimore, MD, 13-17 April, 1986, Abstract A9.

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WESTERN RESEARCH

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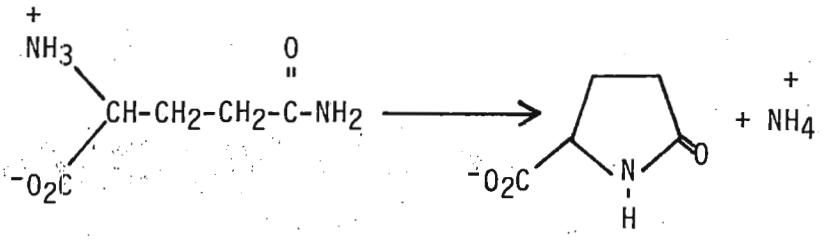
May 23, 1986

Monitoring Tomato Processing
by Carbon-13 NMR.

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

Dear Barry:

We have been interested in using C-13 NMR spectroscopy to characterize the tomato processing steps. Figure 1-A shows a 10-60 ppm expansion of the C-13 NMR spectrum after the initial step of the processing and Figure 1-B after the final step. Comparing spectra 1-A to 1-B, the decrease in intensity of all resonances assigned to Glutamine and the increase in Pyroglutamate can be clearly observed.



The production of Pyroglutamate at the expense of Glutamine as a function of each step of tomato processing is shown in Figure 2. The data suggests that considerable cyclization of the Glutamine is occurring in step 4 of the processing.

Sincerely,

C. K. Tseng D. O. Adams D. J. Ashworth R. Falkenberg

mn 6:38

FIGURE 1. Carbon-13 NMR spectra of tomato water soluble organic components (A) after initial stage of processing and (B) after final stage of processing. ALA - Alanine; GABA - Gamma-aminobutyrate; PG - Pyroglutamate; GLN - Glutamine; GLU - Glutamate; ASN - Asparagine; ASP - Aspartate; MAL - Malate; CIT - Citrate. Arrowhead in 1-B denotes the absence of Glutamine resonances.

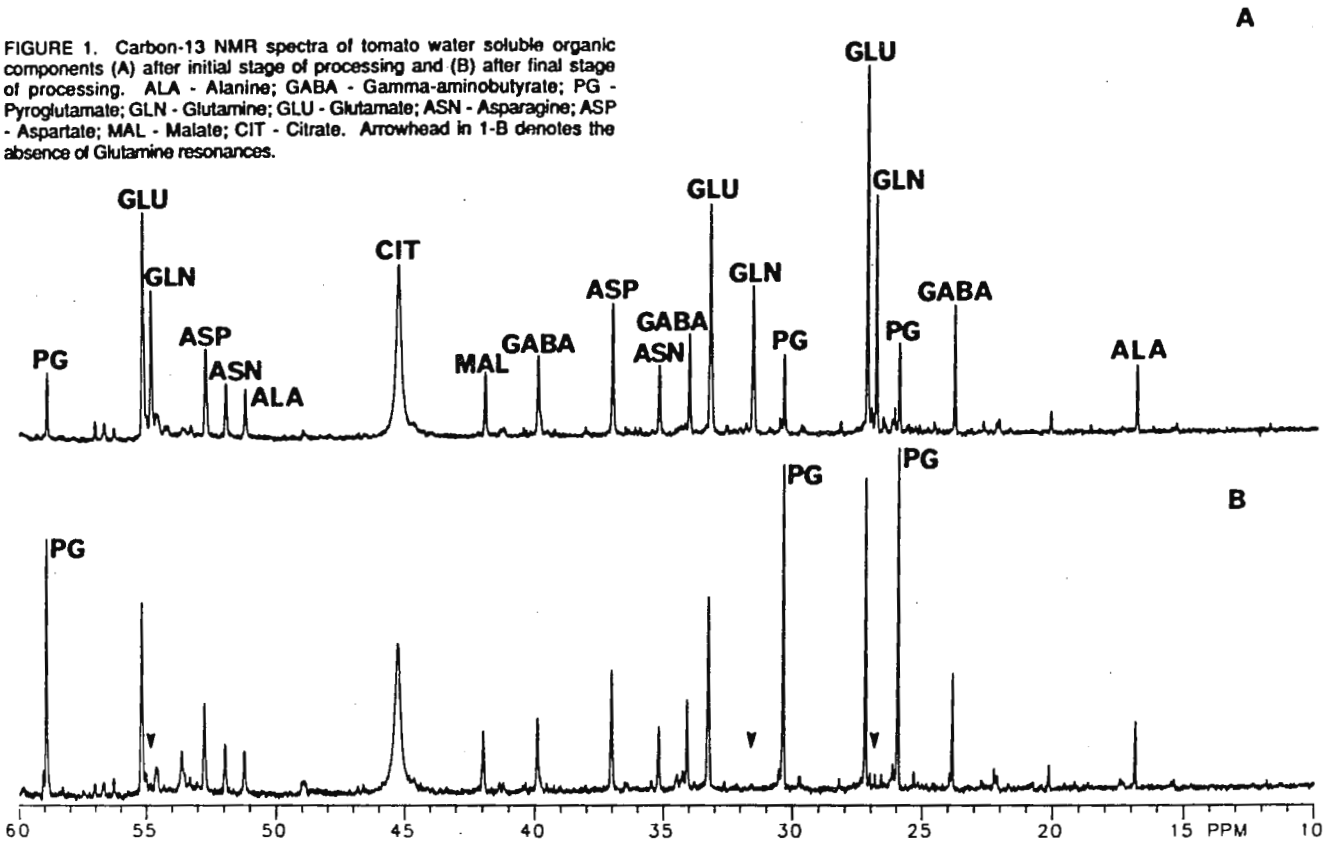
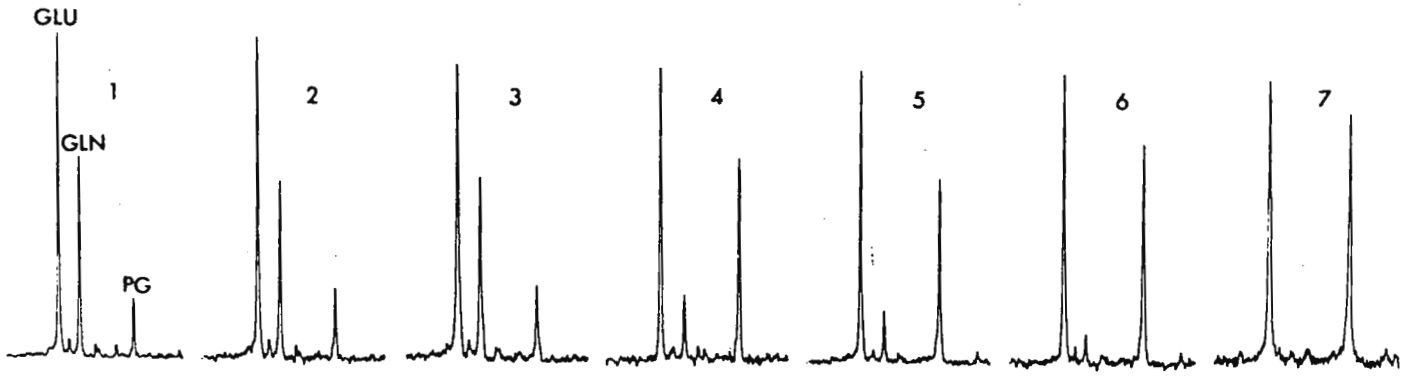


Figure 2. 25-28 ppm C-13 NMR spectral expansions of each stage of tomato processing showing the conversion of Glutamine to Pyroglutamate. GLU - Glutamate; GLN - Glutamine; PG - Pyroglutamate.



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BMNA/86/7

20th March 1986

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

Dear Dr. Shapiro

Prototropism in Aryl-deazapurines

We have recently concluded a study of protonation and tautomerism in 1-deaza and 3-deazapurine using ^{1}H, ^{13}C and ^{15}N NMR (Mag.Reson.in Chem.,24, 55, 1986) but this was only the prelude to studying compounds of more interest pharmaceutically. These included the compound series I. In the parent heterocycles, we found the tautomeric equilibria to be fast on the NMR timescale but in I (R=OMe) with the 2'-OMe substituent also present this is slowed down so as to enable the observation of the tautomers. Analysis of the ^{13}C shifts, the use of model compounds, and the investigation of long-range ^{13}C-^{1}H coupling constants has enabled us to assign the resonances of the tautomers. In this series we observe all situations from slow exchange, through intermediate rate broadening to fast exchange according to the nature of the substituent R. For I(R=OMe) as the free base in dmso-d_6 only two species are observed consistent with hydrgen bond formation and tautomerism occuring in a concerted fashion. We are currently attempting to get large amounts of data together for publication purposes, but the ^{13}C shift data for I(R=OMe) are shown in the Table, together with assignments for each tautomer.

Yours sincerely

John

Jan

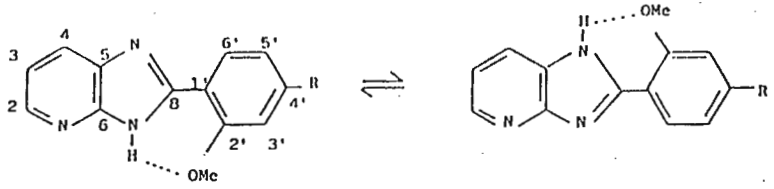
J.C. Lindon

J.M. Williams

Department of Physical Chemistry

^{13}C NMR chemical shifts for R=OMe (ppm)

I:



major

minor

Proportion	~ 65%	~ 35%
C2	143.6	142.8
C3	117.2	117.8
C4	119.3	125.3
C5	126.7	134.7
C6	155.6	148.9
C8	151.4	150.6
C1'	110.2	110.7
C2'	162.6	162.6
C3'	98.6	98.4
C4'	158.6	158.6
C5'	106.5	106.2
C6'	131.5	131.0

University of Durham

Department of Chemistry

Science Laboratories, South Road, DURHAM,
GREAT BRITAIN, DH1 3LE
Telephone: Durham (0385) 64971

Professor of Chemistry:
Robin K. Harris

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

24th April, 1986.

Dear Barry,

POSTDOCTORAL POSITION AVAILABLE

I have an opening in my group for one or two years for a postdoctoral fellow to work on solid-state NMR, both high-resolution (MAS) and broadline. The area of interest is catalyst systems, and the research involves, inter alia, metal resonances (e.g. ^{51}V, ^{113}Cd, ^{119}Sn) of metal oxides and cluster compounds. Examination of adsorbed species by ^{13}C and/or ^{1}H NMR is also of interest. The salary is on the usual U.K. Research Assistant 1A scale. The position is available as from 1 October 1986. Liaison with a research group at the University of Warwick, active in the catalyst area is involved. Applicants should send me a C.V. and give the names and addresses of two referees.

Best wishes,

Robin

333-22

University of Delaware

DEPARTMENT OF CHEMISTRY
NEWARK, DELAWARE 19716

May 6, 1986

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

Title: DISASTER OF THE WEEK!

Dear Barry:

At the recent ENC, the hordes were fascinated and confounded by the "mystery poster." Both of them will be pleased that we shall now come out of the closet and reveal the answers. One of us (RC) frequently observes and terrorizes graduate students who are operating the departmental FT NMR spectrometers. In doing so, he observed some interesting screw-ups and, as a user education effort, began posting them as the "disaster of the week." Those of your readers who supervise service facilities may find this an interesting way to educate their users. A related set of problems for CW-NMR was published in American Laboratory (1).

The "answers" for spectra shown on the following pages are:
(1) The operator found that the peaks were more intense when out-of-phase than when in phase. Insufficient data points.
(2) The receiver gain is set too high.
(3) Quadrature images: the phase detectors need adjusting.
(4) User typed FT twice.
(5) Spectrometer not locked -- deuterium sweep was running.
(6) Splitting of TMS was due to maladjusted Z shim.

For those unfortunates who missed the poster, each "disaster" was accompanied by four semi-humorous to semi-serious multiple-choice answers.

Roger W. Crecely Cecil R. Dybowski Joseph H. Noggle

(1) A. Tchapla, G. Emptoz, A. Aspect and A. Nahon, American Laboratory, 17 (11) 1985, pp 38-49.

/vl

DISASTER OF THE WEEK!

1

The operator shimmed well, the resonances were narrow. However during phasing of the spectrum the methyl peak in ethyl benzene was more intense when the spectrum was out of phase. See "A" below. The acquistion parameters are listed below. The problem can be resolved by changing which of the following.

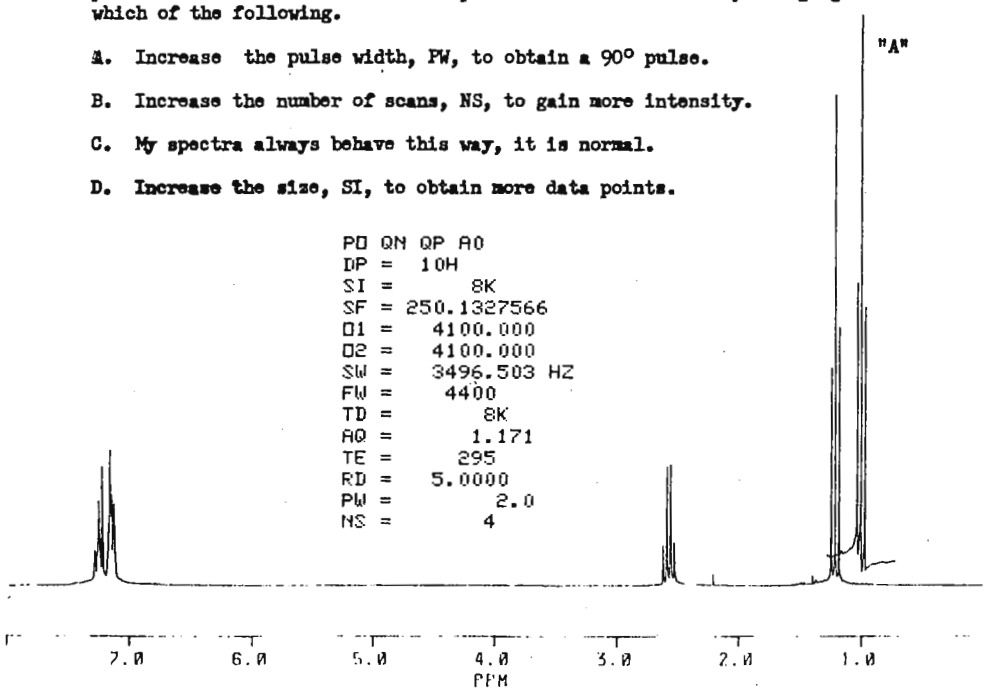
A. Increase the pulse width, PW, to obtain a 90° pulse.

B. Increase the number of scans, NS, to gain more intensity.

C. My spectra always behave this way, it is normal.

D. Increase the size, SI, to obtain more data points.

```
PD QN QP A0
DP =  10H
SI =      8K
SF = 250.1327566
O1 =   4100.000
O2 =   4100.000
SW =   3496.503 HZ
FW =    4400
TD =      8K
AQ =     1.171
TE =     295
RD =   5.0000
PW =       2.0
NS =       4
```



2

DISASTER OF THE WEEK!

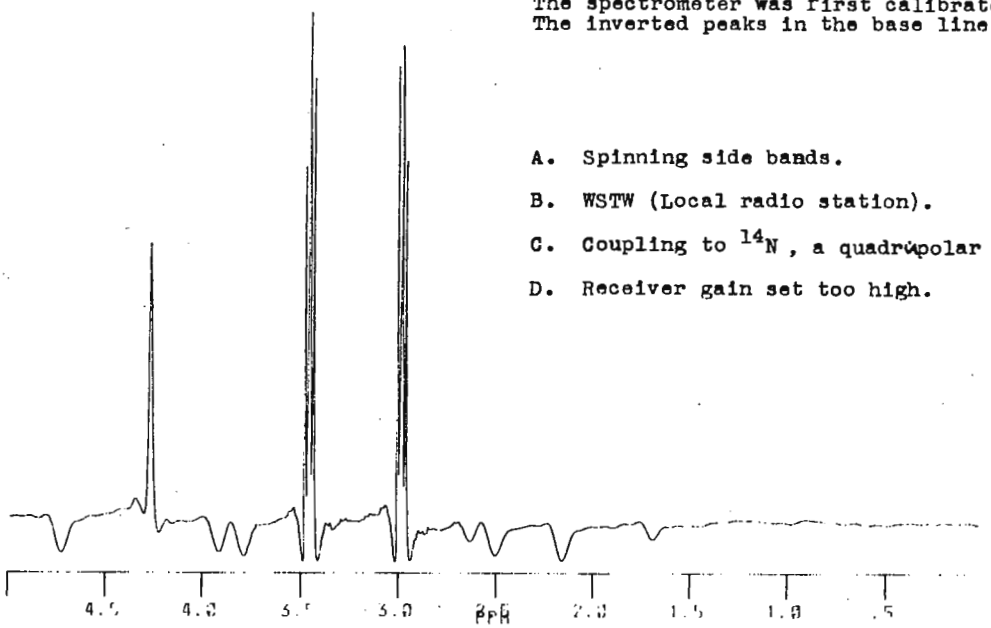
A ^{1}H NMR spectrum of $(HOCH_2CH_2)_4NNO_3$ in D_2O is shown below. The spectrometer was first calibrated using ODCB/TMS/Acetone-d_6. The inverted peaks in the base line are artifacts due to:

A. Spinning side bands.

B. WSTW (Local radio station).

C. Coupling to ^{14}N, a quadrupolar nucleus.

D. Receiver gain set too high.



3

DISASTER OF THE WEEK!

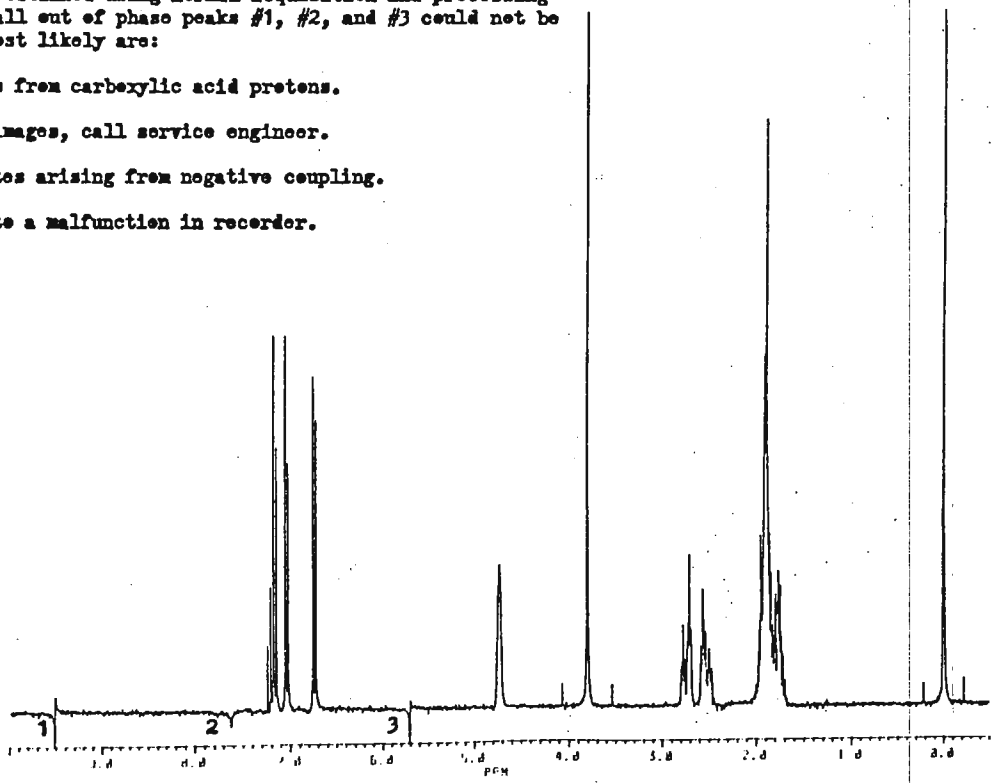
^1H spectrum was obtained using normal acquisition and processing parameters. Small out of phase peaks #1, #2, and #3 could not be phased. They most likely are:

A. Folded peaks from carboxylic acid protons.

B. Quadrature images, call service engineer.

C. J_{CH} satellites arising from negative coupling.

D. Spikes due to a malfunction in recorder.



4

DISASTER OF WEEK!

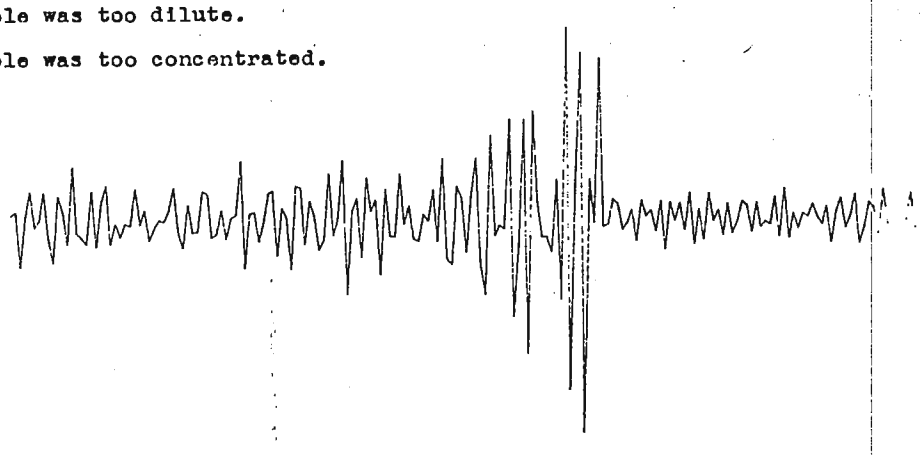
Obtained a normal ^1H spectrum of an organic compound. FID looked good, but after processing spectrum it looked like this. Most likely cause is:

A. Poor phasing

B. FT typed twice.

C. Sample was too dilute.

D. Sample was too concentrated.



5

DISASTER OF THE WEEK!

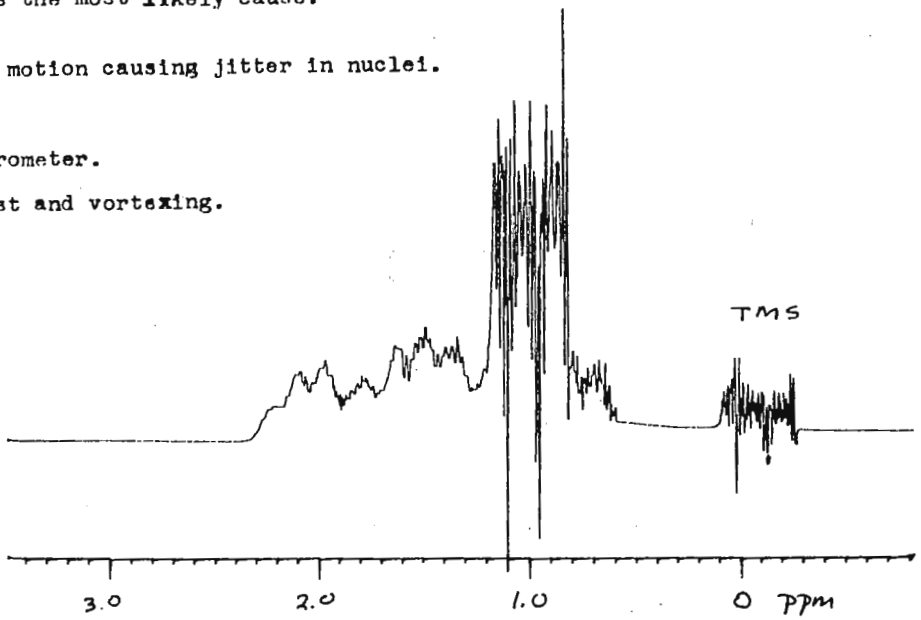
This ^{1}H spectrum of an alkyl organic compound/$DCCl_3$ was obtained on an FT NMR. The noise is not due to the spectrometer, but operator error. What is the most likely cause?

A. Rare case of Brownian motion causing jitter in nuclei.

B. Eccentric NMR tube.

C. Failure to lock spectrometer.

D. Sample spinning toofast and vortexing.



6

DISASTER OF THE WEEK!

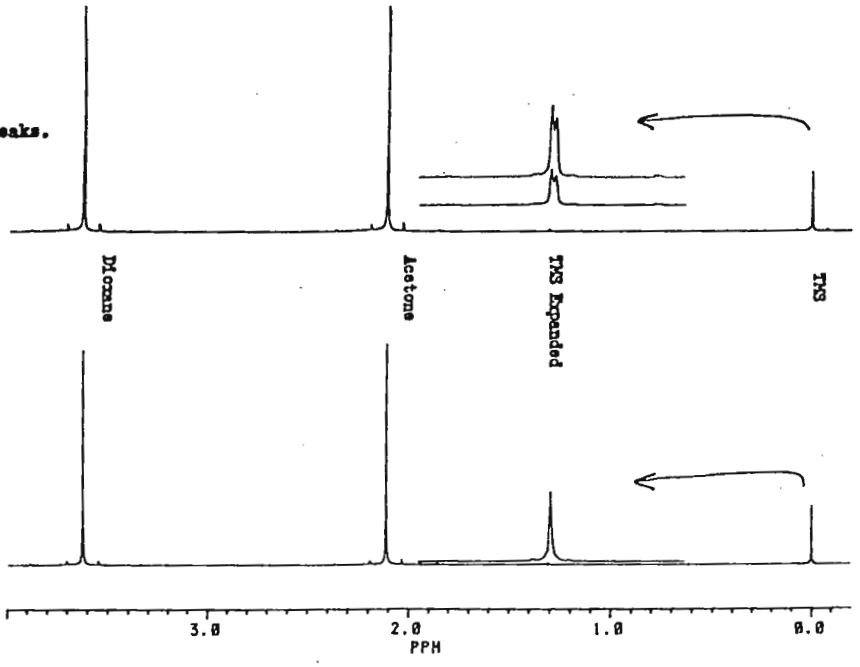
The reference sample from the R-12 was run on the AM-250. The top spectrum was obtained first, but every peak was split into a doublet. A small adjustment was made to obtain the bottom spectrum. What adjustment was made?

A. "Z" Shim used to improve homogenaity of field, H_o.

B. Spin rate increased from 15 to 25 Hz.

C. Lock gain was decreased to eliminate saturation of peaks.

D. Plot speed was increased to "Blur" doublets.



UNIVERSITÉ RENÉ DESCARTES (PARIS V)
UER DES SCIENCES PHARMACEUTIQUES ET BIOLOGIQUES

UNITÉ DE PHARMACOLOGIE MOLÉCULAIRE (INSERM U 266)
LABORATOIRE DES INTERACTIONS BIOLOGIQUES (CNRS UA 498)

LABORATOIRE DE CHIMIE ORGANIQUE

DIRECTEUR : PR BERNARD P. ROQUES

Paris, May 12th, 1986

Professor B.L. SHAPIRO
Department of Chemistry
Texas A & M Univertity
College Station
TEXAS 77843-3255.

^{31}P NMR studies of bisintercalating drugs complexed with autocomplementary oligonucleotides.

Dear Doctor Shapiro,

For any years we are investigating the interaction between bisintercalating drugs and DNA. Although important results on the intercalation mechanism can be obtained from proton NMR studies, these later are often time consuming. Since the DNA conformation is modified by interaction of various drugs as illustrated by the partial unwinding of specific regions of DNA, induced by intercalating agents, ^{31}P NMR spectroscopy could be powerful tool in the determination of the type of complexes formed.

One of the major problem occuring in such ^{31}P studies arised from the extreme broadening of the signals in the complexes hindering the assignment of these nuclei. The bisintercalating drug ditercalinium (1) forms with the mini helix $[d(CpGpCpG)]_2$ a 1:1 complex wich is in slow exchange on the NMR time scale with the free tetranucleotide. Owing to the unambiguous assignments of ^{31}P signals in the free helix the ^{31}P signals in the complexed form can be assigned by two dimensional homonuclear chemical exchange experiment (Figure 1).

The classical experiment can be greatly improved by using the phase sensitive mode with time proportional phase increment (TPPI) together with a composite pulse decoupling system (CPD in Bruker software) applied during the acquisition (Figure 2).

This method now ccurrently used in our laboratory allows to determine easily the site(s) of intercalation of mono or bis-intercalating drugs in autocomplementary oligonucleotides of various sequences.

(1) Roques, B.P., Pélaprat, D., Le Guen, I., Porcher, G., Gosse, C. and Le Pecq, J.B., (1979) BIochem. Pharm. 28, 1811-1815.

B.P. ROQUES

M. DELEPIERRE.

(continued on page 29)

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(continued from page 26)

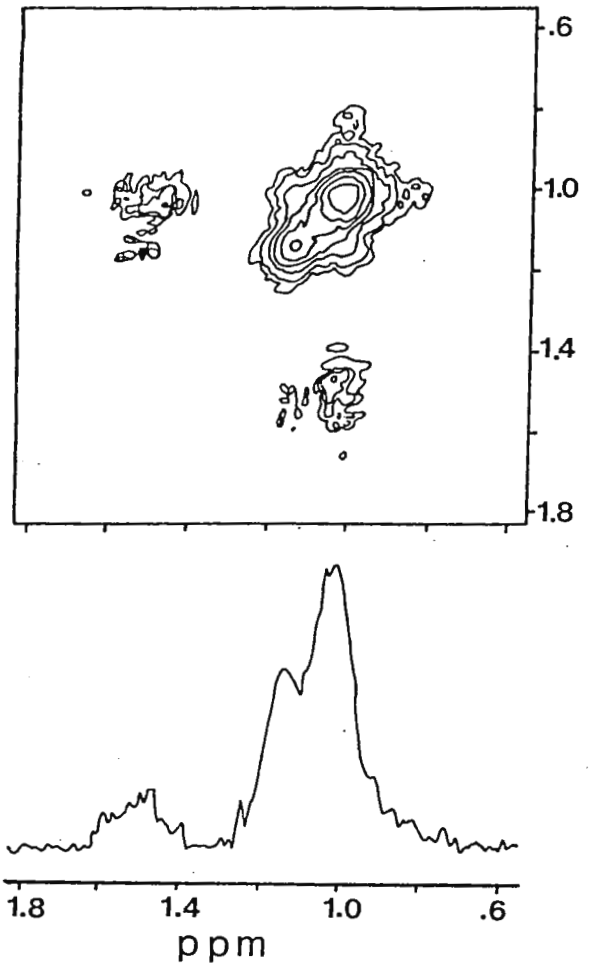


Figure 1.

^{31}P spectrum at 162 MHz of d(CpGpCpG) ditercalinium homonuclear two dimensional ^{31}P-^{31}P chemical exchange (mixing time 600 msec, recycle delay 4 seconds, 128 scans, with Gaussian multiplication in both dimensions, LB = 2).

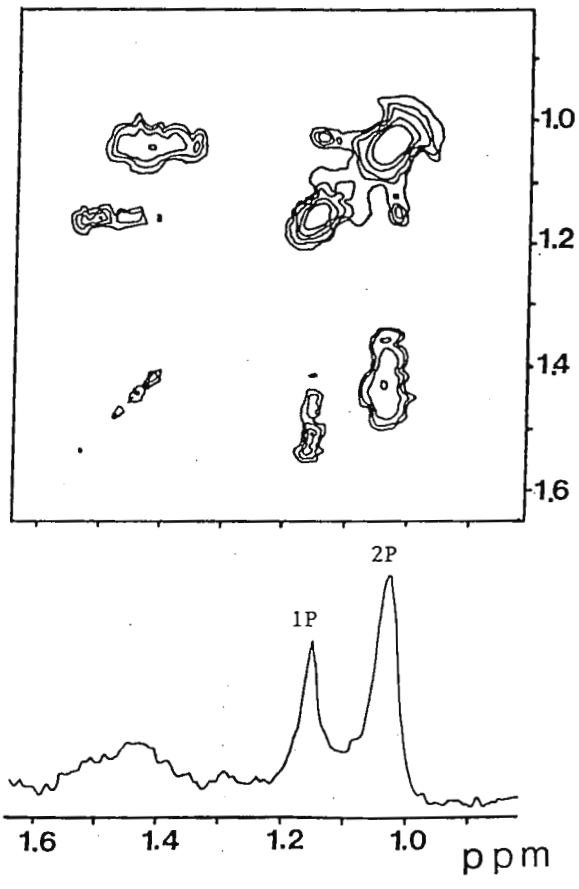


Figure 2.

^{31}P spectrum at 162 MHz of d(CpGpCpG) ditercalinium complexes phase sensitive homonuclear two dimensional ^{31}P-^{31}P chemical exchange with proton decoupling during acquisition (mixing time 600 msec, recycle delay 4 seconds, 128 scans, with Gaussian multiplication in both dimensions, LB = 2).

FOX CHASE
CANCER CENTER
INSTITUTE FOR CANCER RESEARCH ■ 7701 BURHOLME AVENUE ■ PHILADELPHIA, PENNSYLVANIA 19111
215/728-6900

May 27, 1986

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

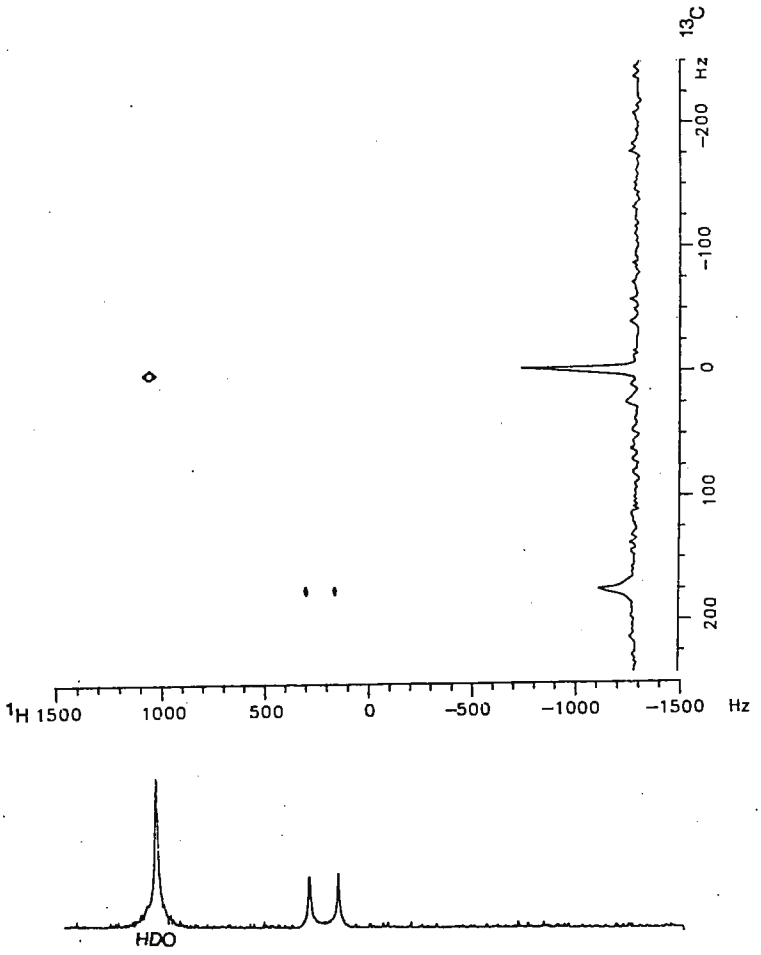
Re: HETERONUCLEAR PHASE SHIFTING ON A NT-300

Dear Professor Shapiro:

Recently we have modified our Nicolet NT-300 to allow ^{1}H detected heteronuclear multiple quantum experiments and submit a brief description of the modification. The broadband heteronuclear decoupler which we described (1) serves as the basis for the heteronuclear frequency. A phase shifter was added which allows 90 phase shifts over the frequency range available to the X nucleus channel (< 200 MHz); phase shifts are accessed through the external ports on the 293 pulse programmer. A 50 watt ENI amplifier serves as final amplification stage. We have used proton detection on the decoupler coil of a 10 mm broadband probe, and achieve a 30 µsec 90^{o} pulse for ^{13}C on the normal observe coil. The figure below shows a contour plot and projections of the first 2D ^{1}H - ^{13}C double quantum shift correlation experiment that we performed using a sample of 1 mM [methyl-^{13}C]methionine in 99% D_2O. The sequence used was:

$$90(^{1}H)_x - (1/2J) - 90(^{13}C)_\phi - t_{1/2} - 180(^{1}H)_x - t_{1/2} - 90(^{13}C)_x - \text{acquire } ^{1}H.$$

where ϕ and the receiver are cycled together through x,y,-x,-y, which yields a double quantum spectrum decoupled in the F1 dimension (2). In this experiment the suppression of uncoupled protons was perhaps 200 fold.



In the course of setting up the pulse program we came across a caveat with Nicolet phase cycling. Because we are accessing the X nucleus phase shifts from an external line from the 293 pulse programmer, we wrote our pulse program to explicitly include the 4 steps of phase cycling for the X nucleus, and so there were 4 acquisitions in the same program. It turns out that with more than 2 acquisitions in the same pulse program, the phase cycling for the observe frequency must be written differently from the normal technique: the phase description is appended to the first acquisition trigger but not to any others. The phase cycling is written as if each acquisition trigger has a phase description appended. When the phase designator is added to more than 2 acquisition triggers, the system refuses to phase cycle at all. Our thanks to the folks at GE for figuring out this undocumented feature.

1. R.W. Dykstra and G.D. Markham (1985) J. Magn. Res. 65, 149-155.
2. A. Bax, R.H. Griffey and B.L. Hawkins (1983) J. Magn. Res. 55, 301-315.

Sincerely,

Doug Markham Bob

George D. Markham - R. W. Dykstra

The University of New Mexico

UNM/LOS ALAMOS CENTER FOR NON-INVASIVE DIAGNOSIS
1101 Yale Blvd., N.E.
Albuquerque, NM 87131
Telephone: (505) 277-8512

April 15, 1986

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

"Inexpensive Gating of a GN-300 to the Cardiac Cycle"

Dear Barry:

We have been performing ^{1}H and ^{31}P NMR spectroscopy on myocardial tissue of rats in vivo and perfused Langendorff model hearts ex vivo. It has been known that the levels of ATP and phosphocreatine fluctuate within the cardiac cycle as monitored by ^{31}P NMR. We wished to extend these studies using ^{1}H NMR, to detect changes in the levels of other metabolites.

In our ex vivo heart, a balloon filled with deuterium oxide is inserted into the left ventricle. The balloon is connected to a pressure transducer, which generates the electrical signal passing on to our Beckman model RD dynagraph. Following amplification to 20-30 mV by the dynagraph, we steal the signal and monitor it on our Tektronix model 2465 oscilloscope. The scope can be triggered on the rise or fall of the pressure wave from the heart. This scope also has a 5V TTL logic level output tht goes high with the trigger. We route this to the external input line (Pin 4) on the 293B that controls a conditional line in the pulse programmer.

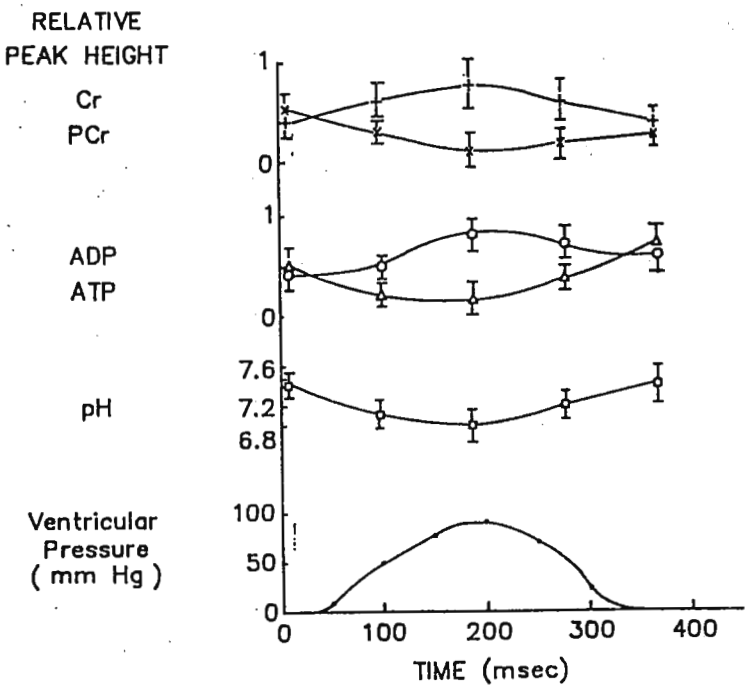
The pulse sequence is written so that the spectrometer repeats a short delay of 2 msec until the gating on the pressure wave triggers the conditional jump to the next event in the pulse sequence. Following the acquisition of data, the pulse sequence returns to the conditional delay and waits for another heart beat. The acquisition time is a fraction of the total cycle length. The region of the cardiac cycle which is sampled is adjusted with a variable delay time, which is the period of the pulse sequence before the excitation pulse.

The same strategy is used <u>in vivo</u>, except that the EKG signal from across the chest of the rat is sent to the dynagraph and used to trigger the oscilloscope. The low level of the EKG signal makes good grounding of the spectrometer to the dynagraph and oscilloscope imperative.

As shown below, changes in ATP, PCr, and pH are observed through the cardiac cycle, as detected in fluctuations in low field ^{1}H signals from H8 of ATP, the $NHPO_4$ group of PCr, and H4 of B-carnosine present in myocardial tissue. We have also detected a small decrease in the level of lactate at systole.

Rich Griffey Charles Gasparovic John Frierson

Richard Griffey, Charles Gasparovic, John Frierson
Center for Non-Invasive Diagnosis
University of New Mexico
1201 Yale, NE
Albuquerque, NM 87131



DEPARTMENT OF HEALTH & HUMAN SERVICES

Public Health Service

National Institutes of Health
National Cancer Institute
Bethesda, Maryland 20892

May 12, 1986

Professor Bernard L. Shapiro
TAMU NMR Newsletter
Department of Chemistry
Texas A & M University
College Station, Texas 77843-3255

Metabolism of Perfused Drug-Sensitive and Resistant Breast Cancer Cells and Position Available

Dear Barry:

We have recently been investigating the metabolic properties of human breast cancer cells that are sensitive to chemotherapy (WT) and a line that has been selected for resistance to adriamycin (Adr^R). Cells are cast in agarose-gel threads (1) and continuosly perfused as we have described (2). By steady state ^{31}P NMR studies of the perfused cells we have detected higher levels of phosphocreatine and lower levels of phosphomonoesters, phosphodiesters, and diphosphodiesters (3). Characteristic differences in the levels of these phosphate compounds may become clinically useful in the detection of drug resistant tumor cells.

^{13}C NMR has been used to monitor the utilization of ^{13}C-glucose and the production of ^{13}C-lactate under various conditions. Under control conditions, no rate differences between WT and Adr^R cells were observed (Figure). However, in the presence of inhibitors or effectors of PFK activity, such as citrate and phorbol esters, the rates of Adr^R glucose uptake and lactate production increase, while those for WT cells decrease. These differences in both steady-state intracellular metabolite levels, and of glycolysis may reflect significant alterations in control of bioenergentic mechanisms between drug resistant and sensitive cells.

We have constructed a probe for *in vivo* studies on subcutaneous tumors in small animals. When the NIH NMR Research Center is completed next year we will extend these studies to the GE CSI systems. I have a Visiting Fellowship available for this work that is intended for a non-US citizen who has three years or less post-doctoral experience.

Sincerely,

Robbe C. Lyon

Jack S. Cohen

References:

1. D. Foxall, J.S. Cohen, and J. Mitchell, *Exptl. Cell Res.* **154**, 346 (1984).
2. R.C. Lyon, P. Faustino, and J.S. Cohen, *Magn. Res. Med.*, in press.
3. J.S. Cohen, R.C. Lyon, C. Chen, *et al.*, *Cancer Res.*, in press.

Figure: Level of ^{13}C-glucose and ^{13}C-lactate in the perfusate of WT and Adr^R (AR) cells at 37° following the addition of ^{13}C-glucose. % Intensity is relative to the initial spectral intensity of ^{13}C-glucose with the cells at 4°.

(continued on page 37)

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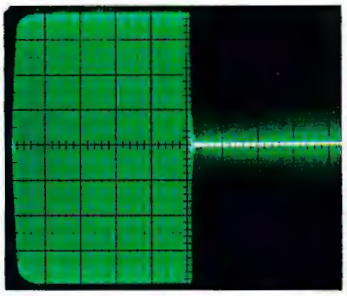

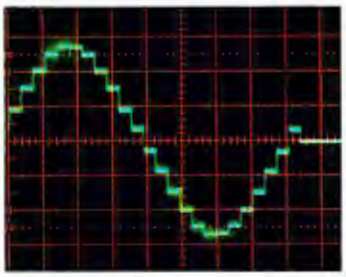

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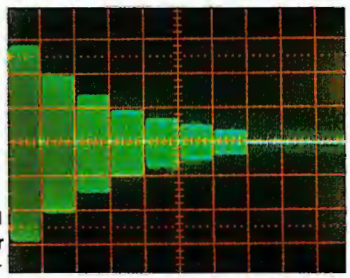
Shaped decoupler pulse. ►



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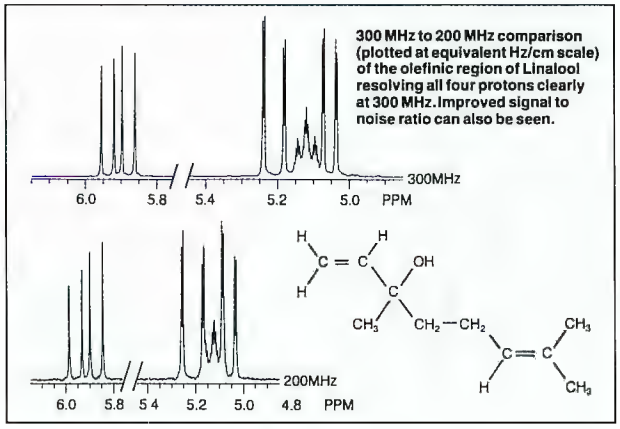

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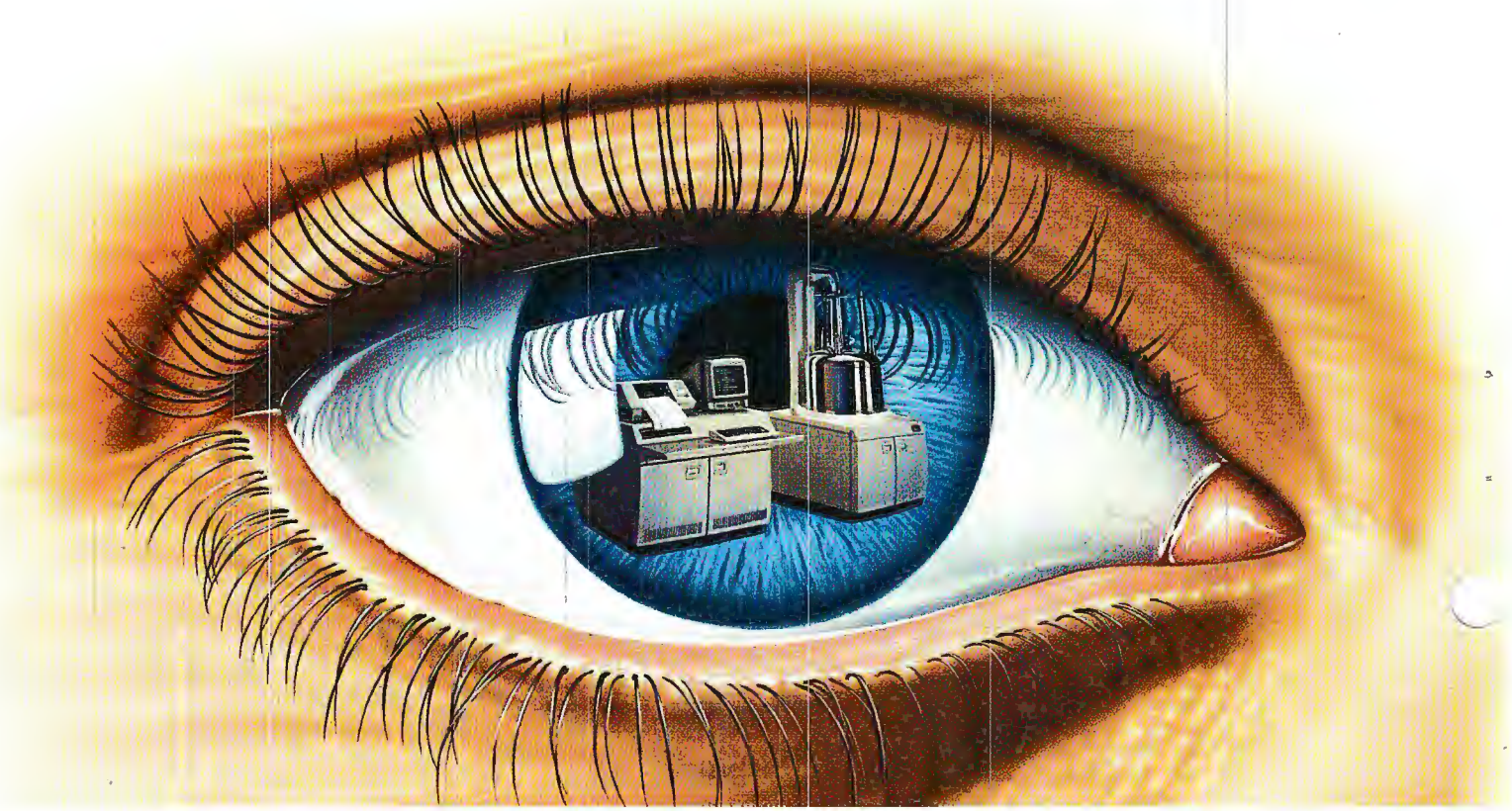

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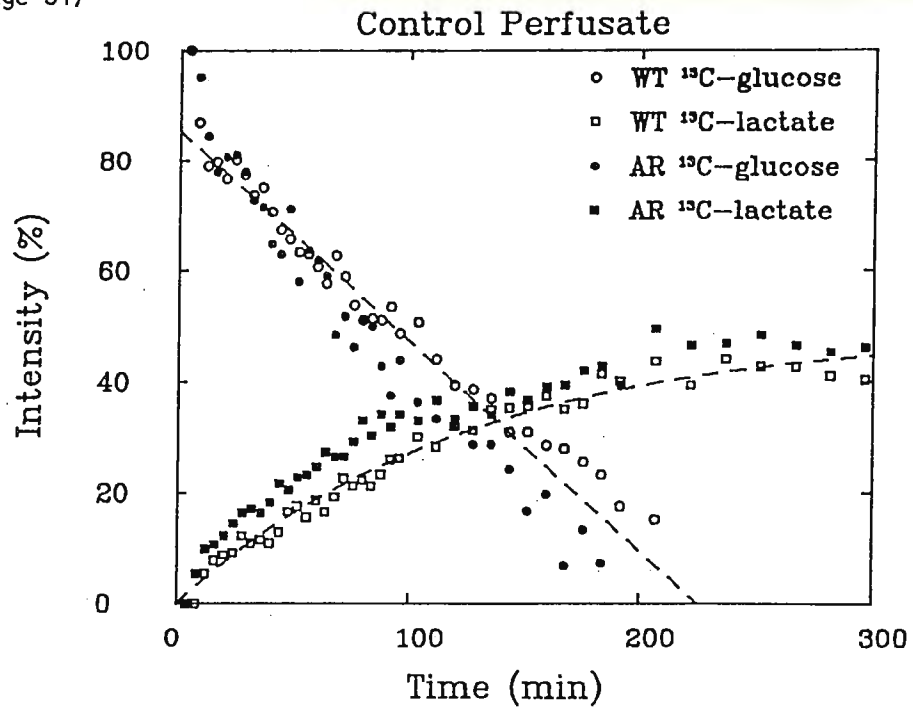

UNIVERSITY OF GRONINGEN
DEPARTMENT OF ORGANIC CHEMISTRY

NIJENBORGH 16
9747 AG GRONINGEN
THE NETHERLANDS

PHONE: 050-634235
TELEX: 53935 CHRUG NL

Equipment wanted: A 5 mm ^{13}C-probe for a Nicolet NT200

Our Nicolet NT200 NMR spectrometer, bought in 1981, is equiped with a Nalorac narrow bore magnet. We would like to buy a (second hand) 5 mm ^{13}C-probe (50.3 Mhz) in good shape and with a good signal/noise ratio. The external diameter of the probes used in our spectrometer is 37 mm and the length of the part that goes into the shaft of the magnet is approximately 465 mm. The probes are introduced from the lower side of the magnet. General Electric does not sell probes anymore for the type of magnet we have. We would therefore like to contact somebody who can offer us a probe as described.

Sincerely yours,

Wilhelm D. Weringa

Dr. W.D. Weringa

333-38

Eidgenössische
Technische Hochschule Zürich

Laboratorium
für Physikalische Chemie
Prof. Dr. R. R. Ernst

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May 21, 1986

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

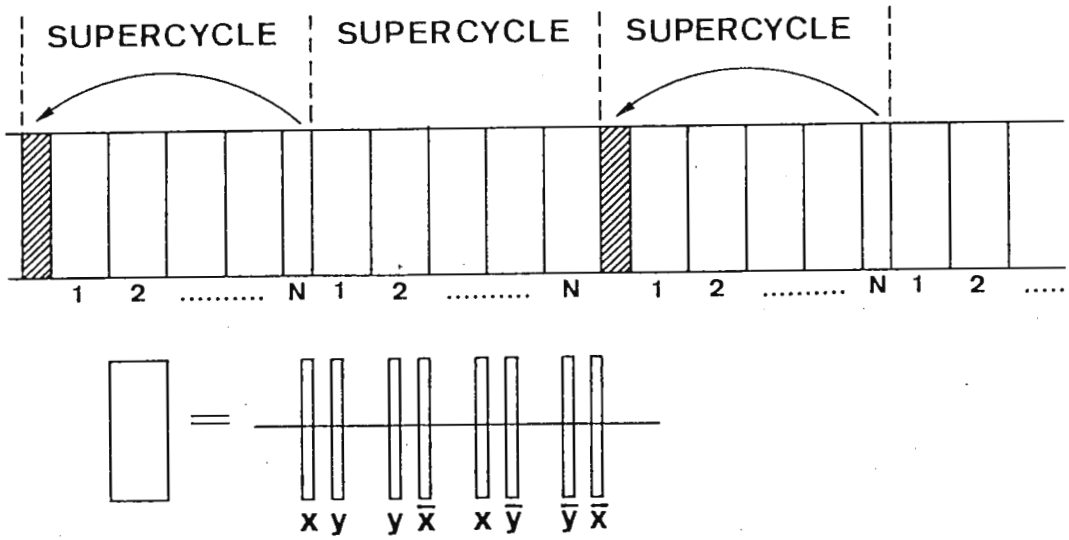
SELECTIVE IRRADIATION UNDER
MULTIPLE PULSE DIPOLAR DECOUPLING IN SOLIDS

Dear Barry,

In the course of our studies of heterogeneity of polymer blends we had a need for selective proton spin diffusion measurements. By selectively inverting or saturating one class of protons, it is possible to monitor the spin diffusion process to further protons. In order to obtain sufficient resolution for a selective irradiation experiment, multiple pulse line narrowing is a prerequisite. For this reason, we had to develop techniques for selective irradiation in the presence of a multiple pulse line narrowing sequence, such as MREV 8, and simultaneous magic angle sample spinning.

By introducing after every N-th multiple pulse cycle a very short additional pulse, it is possible to create a comb of sidebands, one of which can be used for selective irradiation under high resolution conditions. Instead of introducing additional pulses which destroy the cyclic properties of the pulse sequence, it proved convenient to permute a brief fragment of a pulse from the end of every second supercycle, consisting of N cycles, to its beginning, leading

to a modified decoupling pulse sequence of the form



This sequence was employed for selectively saturating the protons in one component of a polymer blend. This allowed us to determine the extent and composition of pure and mixed domains in mixtures of polystyrene and poly(vinyl methyl ether). A paper describing the technical details is in print in Journal of Magnetic Resonance while the polymer aspects will be described in a paper in print in Macromolecules.

Best regards.

Sincerely yours,

P. Caravatti Richard

Pablo Caravatti Richard R. Ernst

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May 22, 1986

Professor B.L.Shapiro
Editor, TAMU NMR Newsletter
Department of Chemistry
Texas A and M University
College Station, TX 77843-3255

Dear Prof. Shapiro,

Pattern Recognition Analysis of ^{1}H and ^{13}C NMR Data Of Crude Oils

During the period of the reconstruction of our NMR laboratory and the instalation of new AM-400 we had evaluated four years NMR experiments with different crude oils.

We solved by "Pattern Recognition Analysis" two questions:
a) finding groups of crude oils with the similar parameters;
b) determining of the number of parameters which are necessary for full description of measured crude oil samples.
This result had been obtained:
a) the group of all crude oils can be divided by this method into the group of pipe-line crude oils and the group of the others.
b) For NMR spectroscopists is mainly useful the second information - it was found that studied group of crude oils can be sufficiently chracterised by following five features - intensities of H_{α}, H_{γ}, C^{H}_{ar}, C_{alif} and the content of sulphur instead of originally used 12 NMR intensities (from ^{1}H and ^{13}C spectra), molecular weight measurement and total elemental analysis.
So the Pattern Recognition Analysis can help us to decrease the number of "noisy" information in some cases of semiquantitative NMR analysis of fossil fuel samples.

The Manuscript with the complete description of problem we hope to send into FUEL soon.

Sincerely

Milan Hájek

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Spurious Outputs: −75dBc

Phase Noise: −75dBc, (0.5Hz-15KHz)
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Range: 0.1-160MHz
Resolution: 0.1Hz-100KHz (opt.)
Switching: 5-20μs
Output: +3 to +13dBm: 50 ohm
Spurious Outputs: −75dB

Phase Noise: −63dBc, (0-15KHz)
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Switching: 5-20μs
Output: +3 to +13dBm: 50 ohm
Spurious Outputs: −70dB

Phase Noise: −63dBc, (0-15KHz)
Freq. St'd: Oven, TCXO, Ext.
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PTS 500

Range: 1-500MHz
Resolution: 0.1Hz-100KHz (opt.)
Switching: 5-20μs
Output: +3 to +13dBm: 50 ohm
Spurious Outputs: −70dB

Phase Noise: −63dBc, (0-15KHz)
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117871 GSP Moscow V-437
USSR

Professor B.L. Shapiro

Texas A & M University
Department of Chemistry
College Station, Texas 77843
USA

April 18, 1986

Title: T_1 Measurment by 2D-NMR

Dear Barry:

It is well recognized that nuclear spin-lattice relaxation time T_1 correlates with molecular spatial structure and dynamics. Application of conventional 1D-NMR techniques of T_1 measurment for complex compounds, for instance, biological macromolecules deals with problem of resonance interference. In this respect it is natural to employ the advantages of 2D-NMR spectroscopy for individual signal resolution.

One of the simplest 2D-NMR scheme for proton spin-lattice relaxation elucidation, which we use to call IR-COSY, combines common inversion recovery (IR) procedure and homonuclear J-correlated 2D spectrocsopy (COSY) by the nonselective pulse sequence:

$$180^\circ - \tau - 90^\circ - t_1 - 90^\circ - t_2.$$

The frequency domain IR-COSY spectrum looks similar to the usual COSY spectrum, but intensity of cross-peaks of the J-coupled A and X protons depends on τ, T_{1A}, T_{1X} and cross relaxation term ρ_{AX}, as can be shown by density operator calculation.

The IR-COSY technique was applied to 10 mM gramicidin A species 3 solution in dioxane-d_8 [FEBS Letters 165, 51 (1984)]. The set of six absolute mode IR-COSY spectra for τ = 0.3 μs, 0.1, 0.25, 0.5, 2.0 and 3.5 s was obtained in 26 h. Intensities $I(\tau)$ of 110 isolated cross-peaks, measured from corresponding cross-sections of the spectra, show single exponential dependence, as revealed by three parameter (I_∞, B, T_1) fit to the equation:

$$I(\tau) = I_\infty[1-B \exp(-\tau/T_1)].$$

Thus nonselective T_1 values of the gramicidin A individual protons were extracted. For the AX spin system the cross-peak with coordinates $\omega_1 = \Omega_A$ and $\omega_2 = \Omega_X$ gives T_{1A} value; whereas the cross-peak with $\omega_1 = \Omega_X$ and $\omega_2 = \Omega_A$ -- T_{1X} value.

Comparison of the obtained IR-COSY results with conventional IR measurments in 1D spectrum for 20 well resolved signals demonstrates perfect agreement (±5%).

Effect of TEMPO spin probe on T_1 values of this sample was also evaluated by IR-COSY procedure. The results are in full accord with the previously established molecular structure of the gramicidin A species 3 in dioxane solution as the left-handed, antiparallel double helix $\uparrow\downarrow\pi\pi^{5.6}_{LD}$.

Other procedures for T_1 and also T_2 measurment of individual resonances can be easily devised on the basis of homo- and heteronuclear 2D-NMR spectroscopy.

Sincerely yours

Vladimir Bystrov Alexandr Arseniev Alexandr Sobol

EMORY UNIVERSITY
Department of Chemistry

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April 29, 1986

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The Chemistry Department at Emory University would like to sell a Bruker WH-90 in working condition.

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Any interested parties should contact Dr. David Live at the above address.

UNIVERSITY OF GÖTEBORG
DEPARTMENT OF ORGANIC CHEMISTRY

Professor Bernard L. Shapiro
Texas A & M University
Department of Chemistry
College Station, Texas 77843-3255
USA

Suggested title: Swedish iron

Dear Professor Shapiro,

To initiate our subscription to TAMU, I would like to mention a few words about what is going on here in terms of ^{57}Fe NMR. We use our Varian XL-400, installed October -85, and a sideways, non-spinning, solenoid coil probe from Cryomagnet Systems Inc. to obtain ^{57}Fe NMR spectra of e.g. heme proteins. This can be achieved in a reasonable amount of time: MbCO, about an hour, $Hb(CO)_4$, an overnight run.

The interpretation of the ^{57}Fe chemical shift is, of course, of interest in mechanistic studies. We have run a few porfyrin complexes of the type Fe(protoporfyrin IX) (CO) (X), where X is a series of pyridine and imidazole derivatives.

Fig. 1 shows a plot of the chemical shift vs. pKa of the conjugate acid of the base (BH^+).

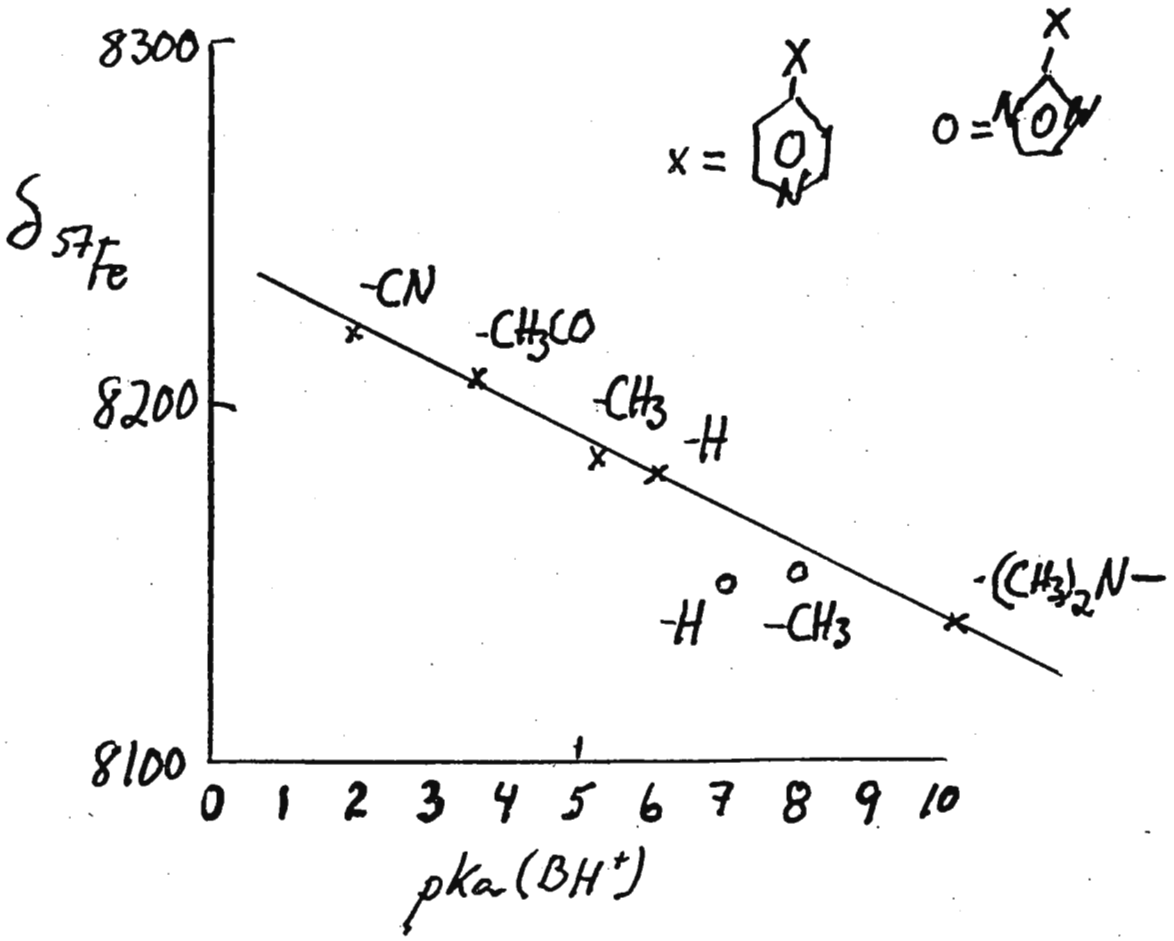
This is interesting because a plot of log K for the complex formation between a porfyrin and a base vs. pKa (BH^+) is also linear. In both plots the imidazoles deviate in much the same way from the pyridines.

Until the next pink slip!

Lars

Lars Baltzer

Postal address
Kemigården 3
S-412 96 GÖTEBORG
SWEDEN



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University of Illinois
at Urbana-Champaign

School of Chemical Sciences
505 South Mathews Avenue

Urbana
Illinois 61801

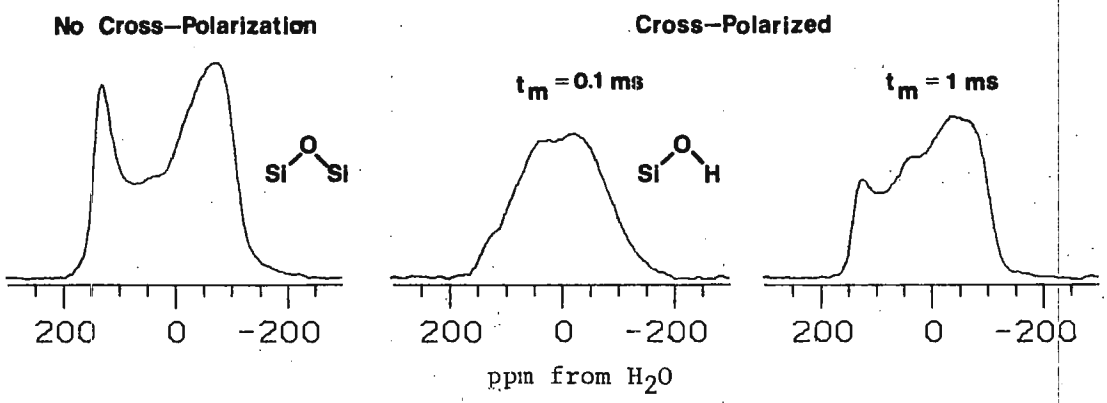
May 16, 1986

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

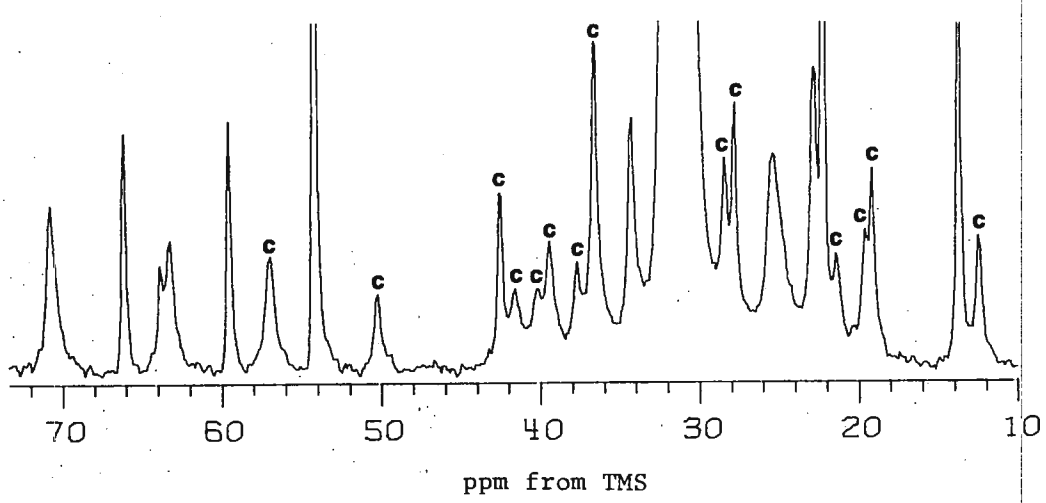
Dear Barry:

O-17 CP of Oxides; C-13 and ^{1}H MASS of Lipids

We've recently been investigating the utility of O-17 CP of ^{17}O-labeled oxides, using surface protons for cross polarization. We get S/N enhancements of up to a factor of ~7, as well as selectivity for hydroxyl groups, in for example, various silicas and aluminas. The following shows typical results for amorphous silica. We find T_{OH} = 35 μsec for the hydroxyl oxygens and T_{OH} = 850 μsec for the bridging oxygens, so that hydroxyl groups can be observed selectively using short mix times.



Second, we have been looking at lipids, this time using C-13 and ^{1}H MASS. We find we can readily observe cholesterol peaks in lecithin-cholesterol multilamellar dispersions, as shown below:



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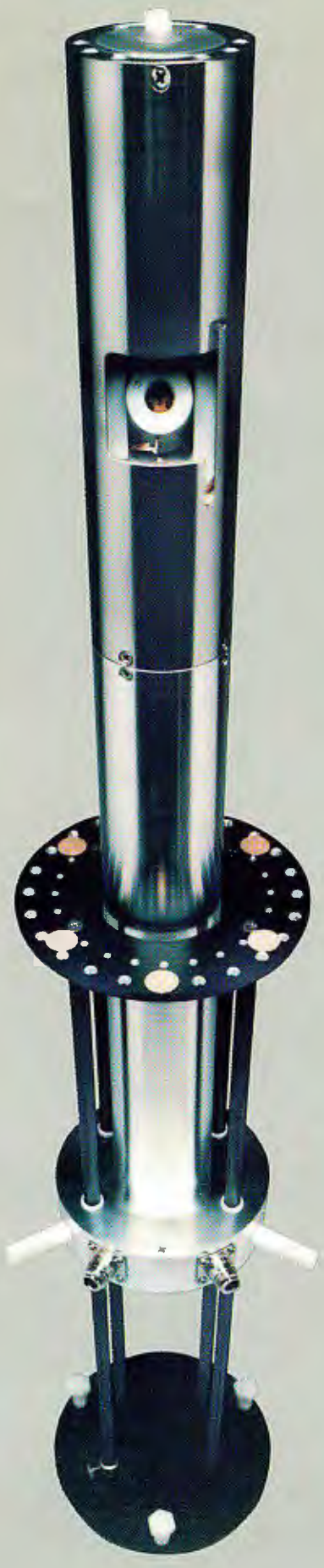

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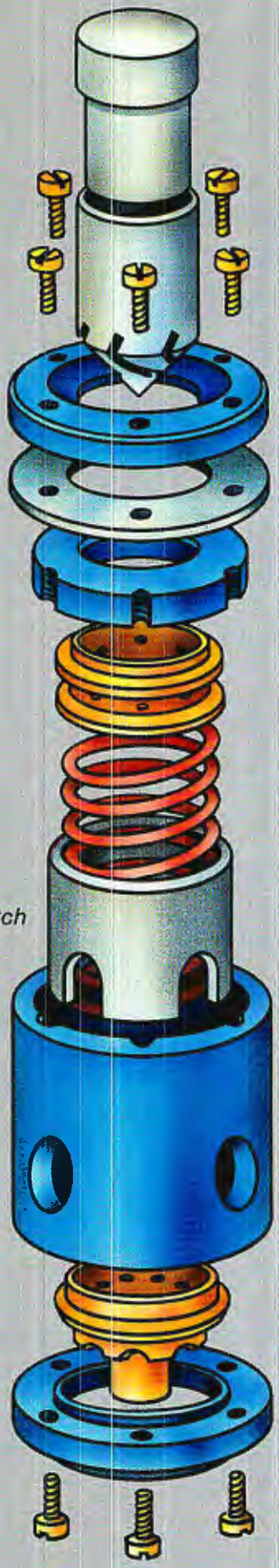

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M275C	20 gauss (3μsec)	6 KHz	4 KHz	only accepts Ceramic/KEL-F rotors
M395	10 gauss (6μsec)	5.5 KHz	N/A	triple bearing system for wide bore
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295C	9.5mm	7.9mm	0.78ml	CERAMIC	4 KHz @ 16 psi	highest sens., no C13 background
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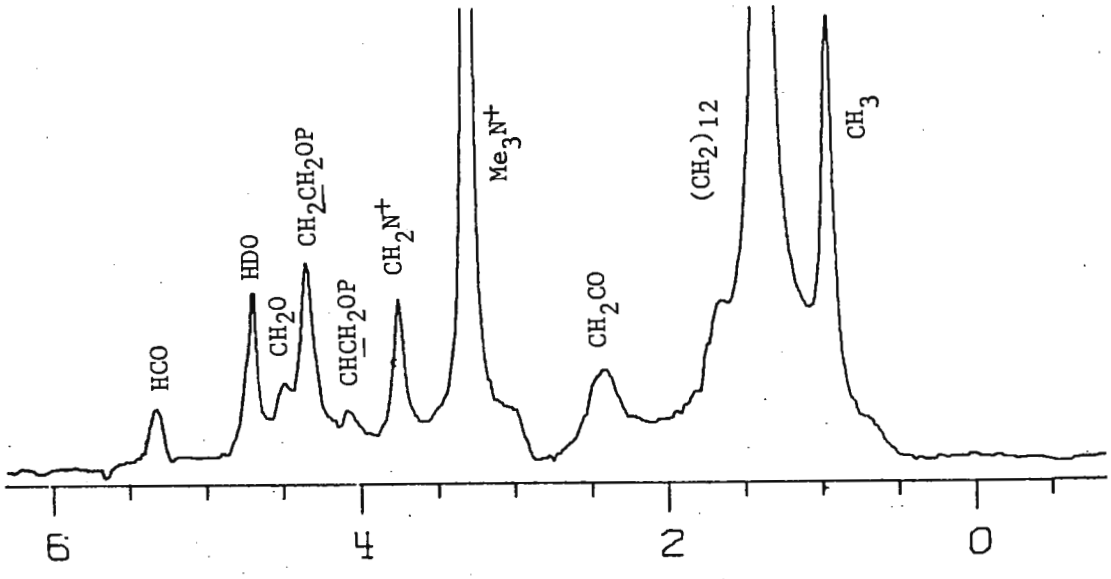
(continued from page 46)

In addition, we find it's possible to observe many cholesterol peaks in "intact" cell membranes, such as those of erythrocytes. We have also been impressed with the resolution of ^1H MASS spectra of lecithins at high field, as shown below:



Studies of other systems, and of biological membranes are in progress.

With Best Regards,

Yours sincerely,

Tom Walter — Jeff Forbes — Eric Oldfield

TW/JF/EO/kjm

PO Box No 90 Wilton
Middlesbrough Cleveland
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Dr B L Shapiro
Department of Chemistry
Texas A&M University
College Station
Texas 77843
USA

Your ref	Our ref	Tel ext	Date
	RT/RAH/PH	2487	14 May 1986

Dear Barry

^{13}C SOLIDS NMR STUDY OF DETERGENTS

The analysis of formulated detergents by solids NMR is not straightforward.

Jeol FX-200 QS spectra for two detergents, A and B, are shown in Figure 1. These have proved difficult spectra to obtain, due to experimental difficulties with KEL-F rotors (1). KEL-F rotors are preferable as they lack the significant background signal observed for Delrin rotors. In practice the KEL-F rotors deform in use, but only when the sample is a detergent powder, in all other cases (i.e. hundreds of catalyst and polymer samples) no deformation of the rotor occurs.

Detergent A has been analysed by wet chemistry and solution NMR and found to contain benzene sulphonate (35% w/w), alcohol ethoxylates (45% w/w) and TAED (tetra acetyl ethylene diamine). Comparing the solids spectrum with reference spectra shows features of the solids data are enhanced, especially the carbonyl and alkyl groups. It is a feature of cross polarisation experiments that the efficiency of cross polarisation is much higher for immobile components, this suggests at least some of the alkyl groups are strongly absorbed onto the builder along with the carbonyl groups of TAED. There are real indications of the ethylene oxide chain at 70 ppm, and of the benzene ring at 125 ppm. For the absorbed alkyl chains there are peaks present due to both the linear alkyl benzene sulphonate and the synprol ethoxylate.

The CP/MAS solids ^{13}C NMR spectrum of detergent B was performed using Delrin rotors, giving rise to the strong background signal at about 88 ppm. The determined components of B are alkyl benzene sulphonate (63% w/w), soap (3% w/w), and alkyl sulphate (35% w/w). Three sets of resonances are observed. Single peaks at 146.3 and 65.0 ppm and an alkyl group at 13-33 ppm. The alkyl group is linear, of the type seen for alkyl sulphates and also as the alkyl part of soaps. The peak at about 29 ppm which is characteristic of alkyl benzene sulphonates is absent, even though these form 63% w/w of the organic part of the sample. The peak at 65.0 ppm is probably due to the alkyl sulphate. The peak at 146 ppm is possibly the carbonyl of the soap, these last two peaks are possibly significantly shifted due to counter ion effects in the solid state. It should be noted that no signals due to the alkyl benzene sulphonate aromatic group are observed.

Fig. 1A

^{13}C CP/MAS solids NMR spectrum of detergent powder A

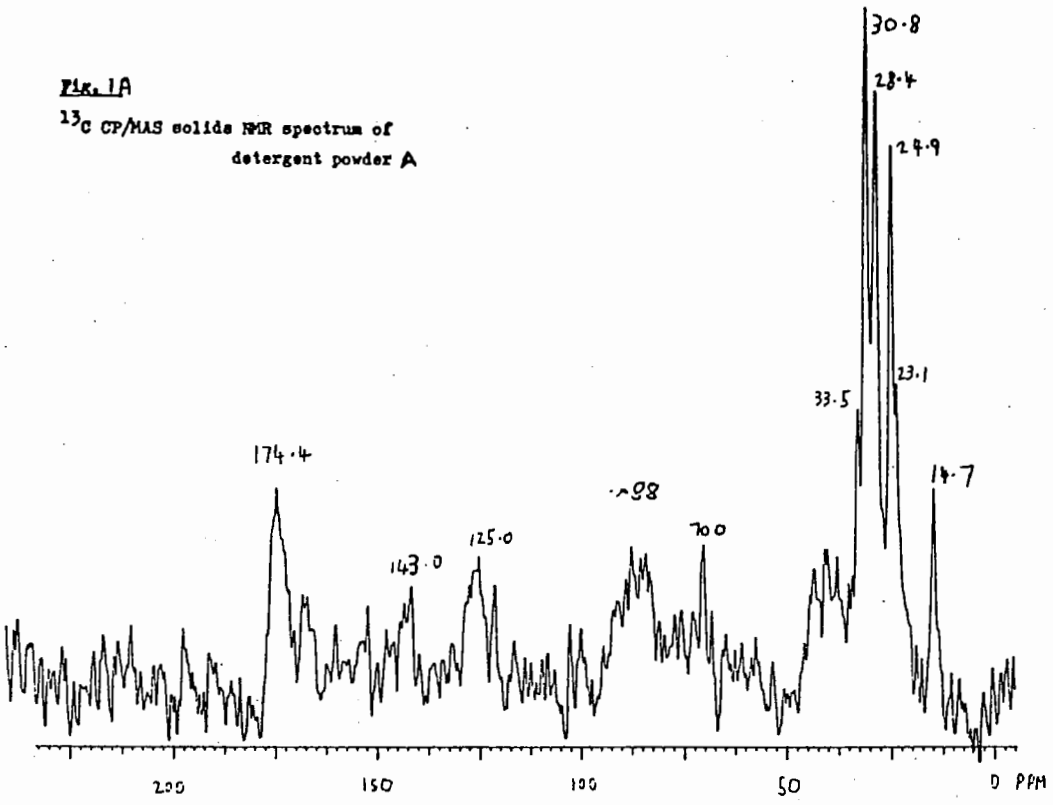
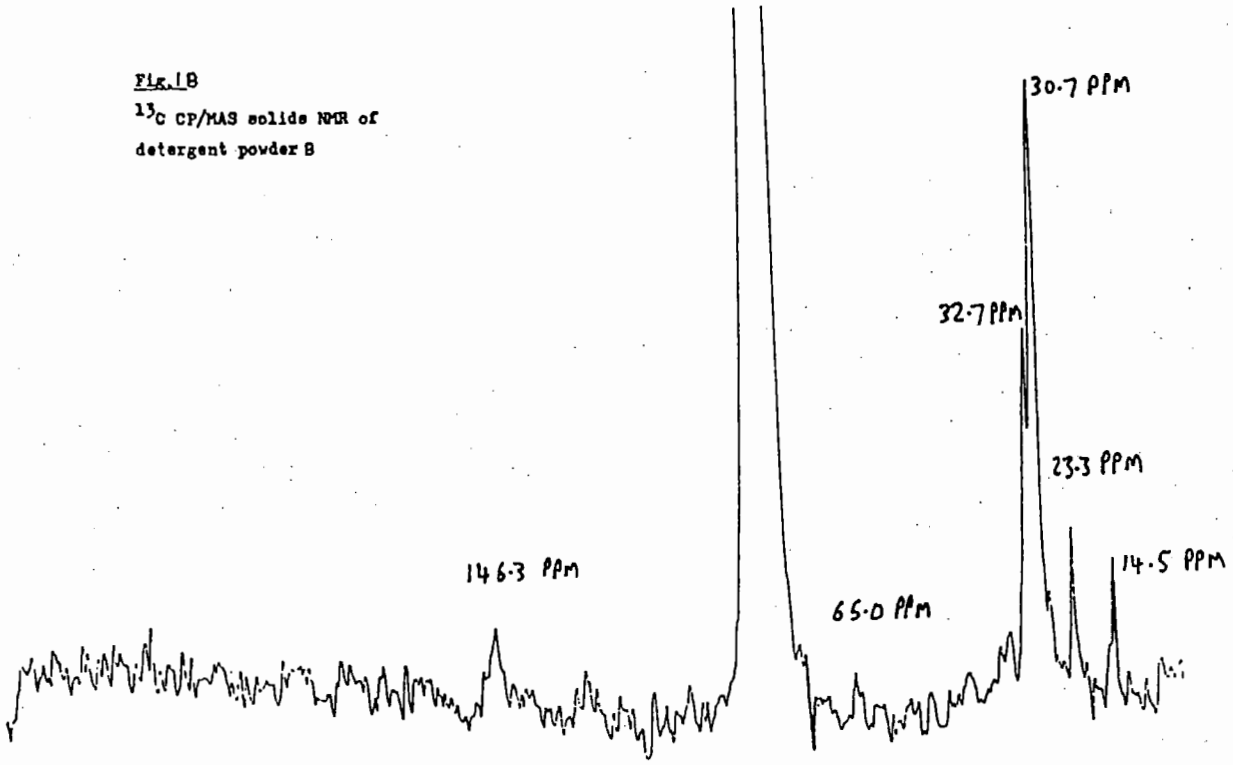


Fig. 1B

^{13}C CP/MAS solids NMR of detergent powder B



The above results suggest that the differential enhancements caused by the relative mobilities of the various 'solid' organic components will make the use of ^{13}C NMR as an analytical tool for detergent powders difficult, especially as some components are not observed at all. The information available on which parts of the component molecules are strongly absorbed to the builder could provide a powerful tool for understanding the processes by which the formulation was assembled. Proton MAS studies and relaxation time measurements of molecular mobility would also contribute to understanding this aspect of formulation.

REFERENCE

(1) R A Hearmon, J Magn Reson., 63 425 (1985).

Yours sincerely

R A. Hearmon

R A Hearmon
Analytical and Polymer Science Group
Research and Technology Department

OSU

The Ohio State University

Department of Chemistry
140 West 18th Avenue
Columbus, Ohio 43210-1173
Phone 614-422-2251

Professor Gideon Fraenkel
614 422-4210 office
614 422-4100 lab
TELEX 810 48217 15

May 15, 1986

Post-Doctoral Position

There is now a Postdoctoral position open in my group for someone who will work on structure and dynamic behavior of organolithium compounds. This involves synthesis of isotopically enriched, ^{6}Li and ^{13}C, organolithium compounds and their study using ^{13}C, ^{6}Li and ^{1}H NMR line-shape analysis. Several high field NMR instruments are used in this work - Brukers AM-500, CXP-300 and soon AM-250. Address inquiries to Professor Gideon Fraenkel at the above address.

For Sale, Bruker HX-90 Magnet

We have for sale a Bruker HX-90 electromagnet, including cooling system, both in excellent condition. Interested parties should communicate with Carl Engelman at the Ohio State address.

Yours sincerely,

Gideon

Gideon Fraenkel
Professor of Chemistry

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May 12, 1986

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

Dear Dr. Shapiro:

Subject: Equipment Available.

We have a couple of old Varian DP-60 magnets (V4013A) and ancillary equipment which are available to anyone who would like them. These are 12 inch, high impedance magnets, one of which has not been operated for over 7 years, the other has not been used in about 3 years. The other equipment available is: two magnet power supplies (V2100B) for the above magnets one has been partially converted to transistors and the other has the old 304TL tubes, a Varian V3508 superstabilizer, and a Bruker BSN 15 proton external lock. These would probably be good replacement magnets for anyone currently using similar magnets. We cannot provide shipping but it will be very easy to load these on a truck in Los Alamos.

Anyone interested can telephone me at (505) 667-0773 or write to me at mail stop C345, Los Alamos National Lab.

Sincerely,

Bill.

William L. Earl.

HERCULES

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Research Center
Wilmington, DE 19899
(302) 995-3000

Equipment Wanted

We have a need for VT probes for Nicolet NT series top loading wide-bore (89 mm) magnets. We would appreciate hearing from anyone who wishes to dispose of unused or inoperable probes. Working probes tuned to 360 MHz are highly desirable but not necessary. Financial remunerations can be negotiated.

Please call me at (302)-995-3505 or Mark Sullivan at (302)-995-3269.

Yours very truly,

H. N. Cheng

333-56

NORTHWESTERN UNIVERSITY COLLEGE OF ARTS AND SCIENCES

Department of Chemistry

2145 Sheridan Road
Evanston, Illinois 60201

April 29, 1986

Prof. B. L. Shapiro
TAMU NMR Newsletter
Texas A & M University
College Station, TX 77843

^{13}C NMR of Oriental Lacquers

[or, 'Not Forever Amber']

Dear Barry:

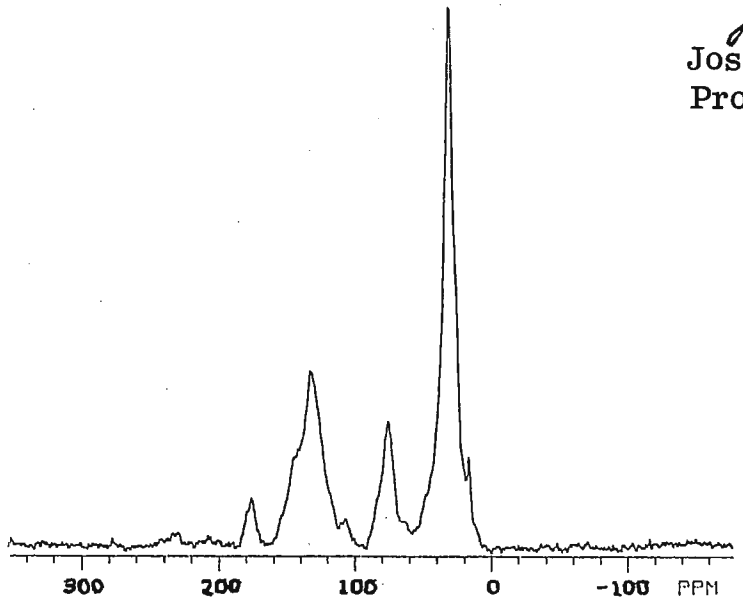
Several years ago we reported the first ^{13}C spectra of amber. Continuing our NMR examination of insoluble organics of interest to the art or archaeological world, we have now obtained ^{13}C spectra of a series of Oriental lacquers. These materials were supplied by Gary Carriveau of the National Gallery of Art, and the spectra were taken by Jim Frye at the Colorado State University Regional NMR Facility. Some of the spectra were obtained on as little as 70 mg of sample.

Lacquer is thought to be made primarily of a urushiol derivative, with various gummy additives; it is insoluble in almost everything but aqua regia. Urushiol is a 1,2-dihydroxy-substituted benzene, with an aliphatic 3 side chain. The spectra (see below for an example) show strong saturated and unsaturated peaks, and a weak carbonyl resonance. The peak at δ 90 suggests heteroatom-substituted carbons. In our preliminary survey, we have found that different lacquers vary in the relative amounts of the four peaks, providing a nice criterion for provenance or variety.

Sincerely,

Joseph B. Lambert
Professor of Chemistry

JBL:cs



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varian

April 25, 1986

Professor Barry L. Shapiro
Department of Chemistry
Texas A & M University
College Station, Texas 77843-3255

Subject: Problems with Editing DEPT Spectra?

Dear Barry:

Spectral editing using pulse sequences such as DEPT to generate CH-only, CH2-only and CH3- only spectra can present problems which are puzzling at first glance. The normal course of events (given a poorly-edited DEPT spectrum) is to suspect the calibration of the pulse widths, the steady-state of magnetizations and, of course, the quality of the software. However, even after careful calibrations the edited spectra may be poor. At this point eloquent arguments such as off-resonance effects, B1 inhomogeneities and multiple-guantum effects are brought into the picture. These are sufficient to complicate life enough so that most spectroscopists shrug their shoulders and assume that they are not current enough on density-matrix theory, rotation operators or the latest pulse-sequence improvements to understand the situation.

The actual reason for their problems may have a very traditional cause: temperature dependent chemical shifts! I have run into this problem a couple of times, and it is most insidious for the very type of experiment used to "set up" the spectrometer. We use a 50% solution of menthol as a convenient and rapid sample for testing the proper execution of experiments, particularly for different pulse sequences. The choice of this hydroxyl-bearing, hydrogen-bonding molecule is, in fact, most likely to bring out the above problem, since a change in temperature will cause a shift in the equilibrium involving hydrogen bond making and braking.

The first time I ran into this problem was when sample cooling air was inadvertantly left turned off after changing from solids to liquids. The change in temperature in going from continuous decoupling (used to run the normal C13 experiment) to about 50% duty cycle was enough to cause a shift in several of the carbons. Since the DEPT experiment was run with only 16 transients per value of the "theta" pulse, the sample cooled significantly during the collection of the four separate DEPT FID'S.

The second time I encountered this problem was on a higher--field spectrometer where I was using about 1 watt of power for decoupling. The heating from this rf was sufficient to cause the above problem when going to the DEPT experiment directly after running a continuously-decoupled experiment. Again, only 16 transients were accumulated per DEPT experiment. The raw spectra looked fine and the problem was only revealed in the editing process. Since only some of the carbons exhibited this temperature-dependent shift it is clearly impossible to shift the data before editing.

The moral of the story is to be sure that equilibrium conditions are attained before attempting any experiment which relies on adding or subtracting spectra--advice which we all have heard, but that which is not usually applied to spectral editing.

Sincerely,

George

George A. Gray
NMR Applications
Laboratory

333-58

Shell Development Company
A Division of Shell Oil Company

Westhollow Research Center
P. O. Box 1380
Houston, Texas 77001

April 18, 1986

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

Dear Professor Shapiro:

SUBJECT: IMPLEMENTATION OF HIGH FIELD NUCLEAR MAGNETIC RESONANCE IMAGING CAPABILITIES AT SHELL

The Magnetic Resonance Group at the Westhollow Research Center has recently implemented High Field Nuclear Magnetic Resonance Imaging (HFNMRI) capabilities.

The HFNMRI system consists of a CXP-200 console interfaced to an Oxford 200 MHz superconducting magnet. The console was recently upgraded to run with an Aspect-3000 computer equipped with an array processor and a new Z-18 pulse program controller board. The console is also equipped with a pulse shape modulator unit and an AR amplifier for slice selective RF excitation.

The 98 mm wide bore imaging magnet is equipped with a set of gradient coils driven by a preemphasis unit designed to minimize gradient switching times.

Both slotted resonator and crossed-ellipse coil design ^{1}H probes are available and can accommodate cylindrical samples up to 3 cm in diameter and 8 cm in length.

The spin warp Fourier imaging sequence shown in Figure 1 was implemented on the test phantom given in Figure 2. The phantom contained $CuSO_4$ doped water. The location of the four planes imaged using multislice acquisition was determined using a z-gradient profile. Slice thickness and in plane digital resolution were set at 1.3 mm and 150 μm, respectively. The sequence consisted of 256 phase encode steps with 32 s required for the 512x512 complex double Fourier transform. Finally, the spin echo and recycle delay times were set at 24.6 ms and 1 s, respectively.

Figures 3(a) and 3(c) are identical; both show the water present in the concentric 5 and 10 mm tubes. The black rings correspond to the 500 μm wall thickness of the inner 5 mm tube.

Figure 3(b) corresponds to a section through the slotted Teflon® spacer depicted in Figure 2. The central circular water signal is due to the inner 5 mm tube while the outer three regions are due to water in the 120° spaced slots in the Teflon plug.

Finally, Figure 3(d) displays the water signal from the outer 10 mm tube.

Future implementations will include multislice multiecho Fourier imaging as well as multislice multiecho back projection imaging schemes.

Please credit this letter to the account of Larry Sterna.

Sincerely yours,

Pierre N. Tutunjian
Associate Research Chemist
Analytical Department

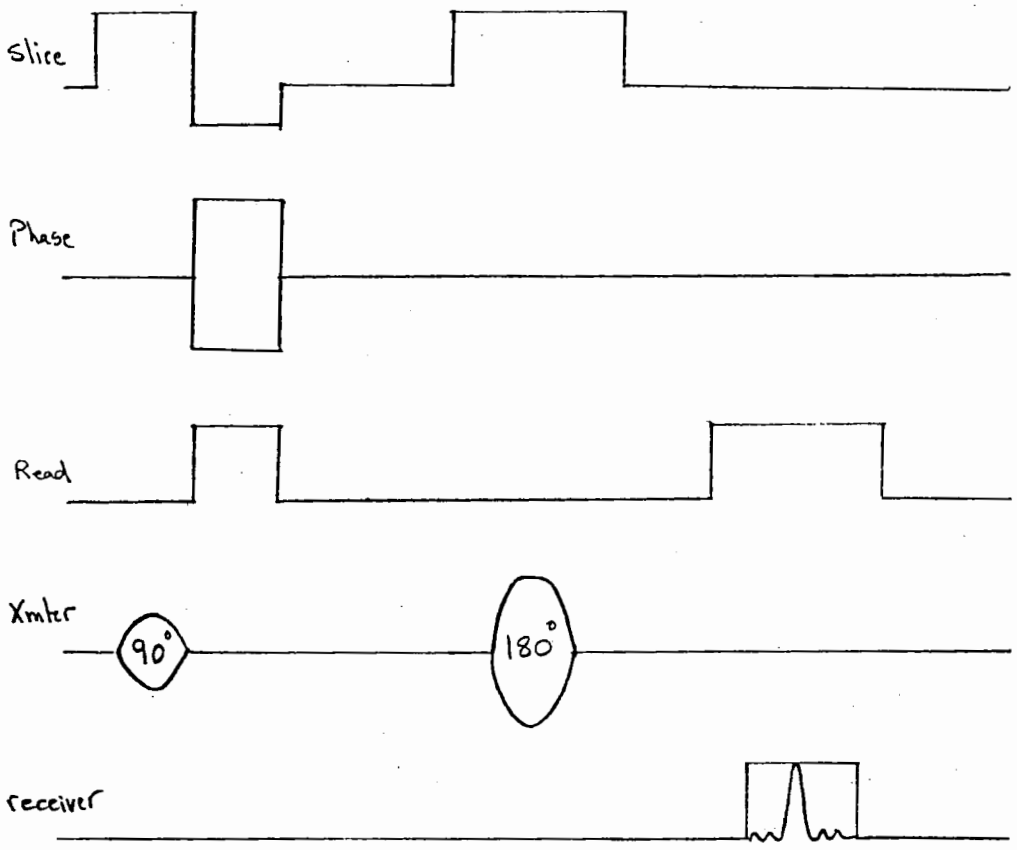

Figure 1

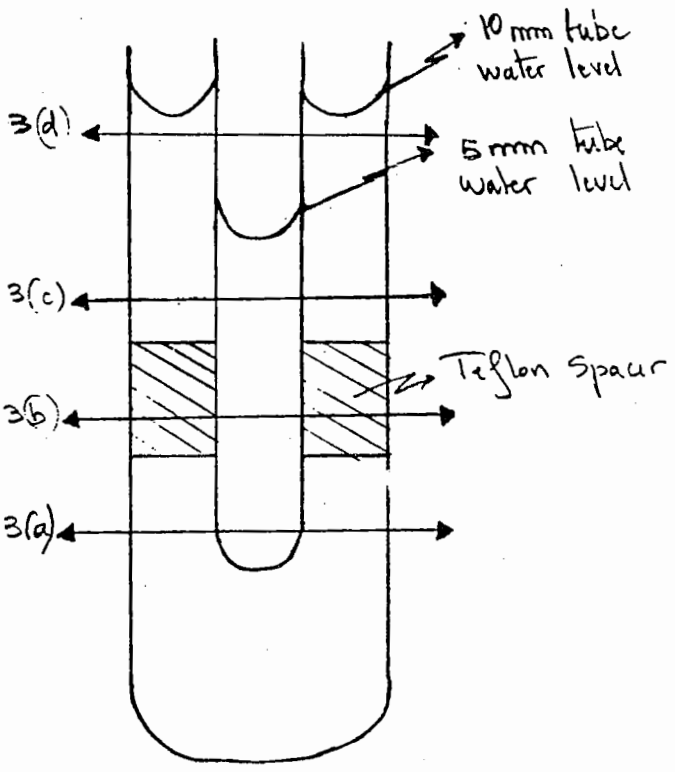

Figure 2

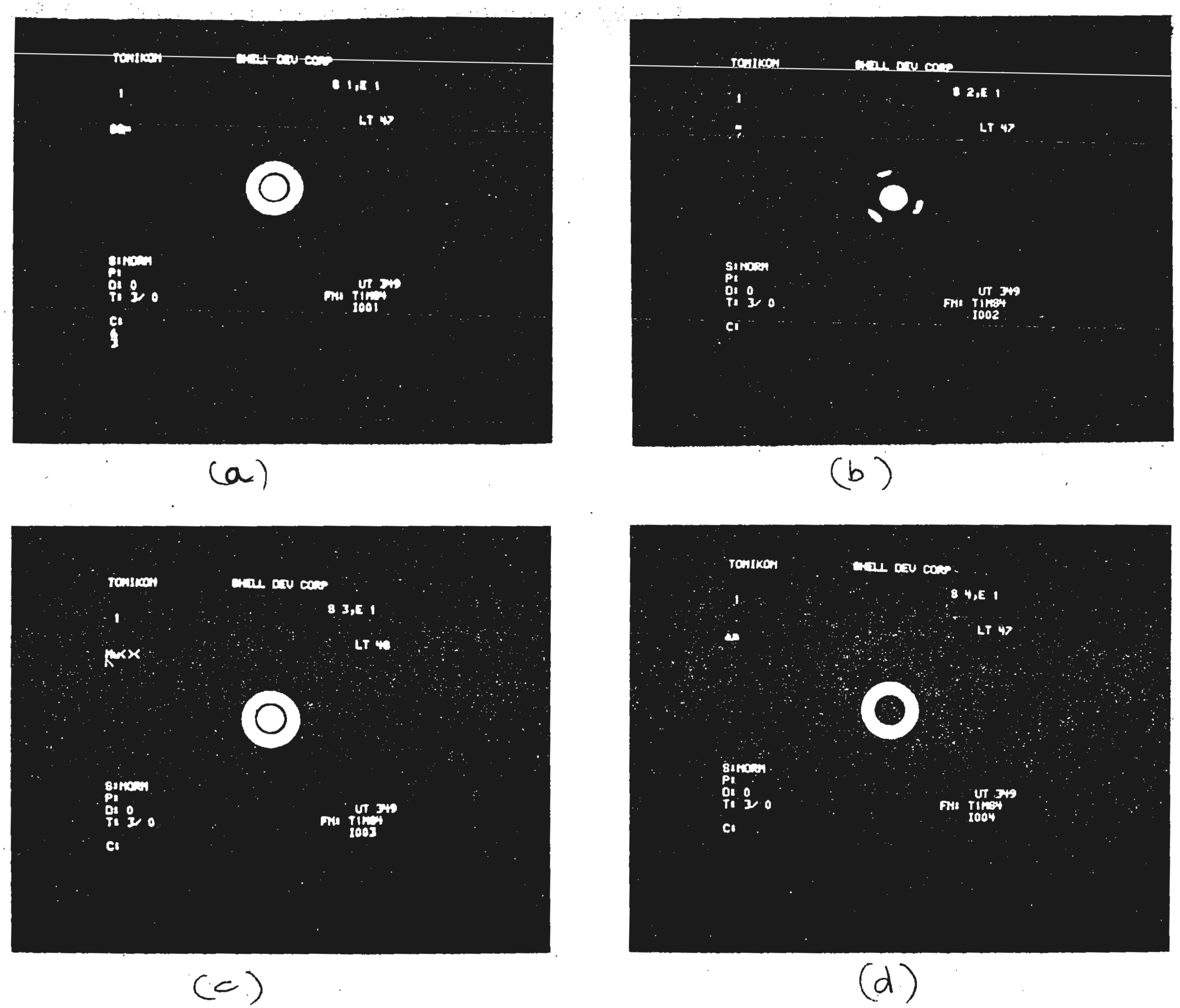
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Figure 3

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May 16, 1986

Professor B. L. Shapiro
Department of Chemistry
Texas A&M University
College Station, Texas 77843-3255

Dear Barry:

COMPUTER-AIDED SPECTRUM INTERPRETATION

We are just completing a series of three Pascal programs which we hope will alleviate some of the drudgery involved in the interpretation of NMR data. These programs are designed to be used on Aspect computers which use one of the three basic forms of DISNMR.

ListPPD:
=======

All NMR software packages contain a command which prints a "peak listing" consisting of the address, chemical shift (often in both Hz and ppm), and intensity or integration of each of the peaks of a spectrum. Bruker's DISNMR applications package contains a command PPD which creates such a peak listing, but sends it to a disk file rather than the printer. The disk file so created is not a text file, and therefore cannot be listed or edited in a convenient fashion. To overcome this problem we have written the Pascal program ListPPD, which is simply a modification of the Bruker program PEAKNORM. ListPPD requests the name of the file created with the PPD, normalizes the peak heights such that the most intense resonance is assigned an intensity of 100, and then provides the option of either typing the resulting table on the printer or writing an image of this table on the disk in the form of a text file.

DEPTInt:
=======

In our lab we routinely derive the multiplicities of carbon resonances from a comparison of the normal broad band decoupled C-13 NMR spectrum to DEPT spectra collected with the final editing proton pulse set at 135 and 90 degrees. This spectral comparison can be somewhat tedious for large molecules, even though the process itself is rather simple. DEPTInt was written to accelerate the process of interpreting DEPT spectra.

The program first reads the PPD file created from the broad band decoupled C-13 spectrum (called here the "zero spectrum"), looks for the solvent resonances and deletes them if found, and assigns all multiplicities to "?". At the end of this process the resulting table can be printed or written in text format to the disk.

Normally, however, one goes on to the next step of the analysis, which is to compare this table to that created by PPD for the DEPT spectrum in which the final editing proton pulse is set to 135 degrees (the "DEPT135 spectrum"). If a peak in the zero spectrum is missing from the DEPT135 spectrum, it is assigned the multiplicity "s"; if a peak is found to be inverted in the DEPT135 spectrum it is identified with the multiplicity "t"; and all upright peaks in the DEPT135 spectrum are assigned the multiplicity "e". The program reports how many peaks it found in the DEPT135 spectrum, and provides the user with the options of printing the table, writing it to disk, or going on to the next step.

In the final step of the analysis, the table created through the comparison above is in turn compared to the PPD file created for the DEPT spectrum with the final editing proton pulse set at 90 degrees (the "DEPT90 spectrum"). This comparison breaks the "e" multiplicity peaks into "d" and "q" multiplicities. Again, the program reports the number of peaks and provides options to print the results or store them in a disk file.

If the HEXInt program is to be used it is essential that the results of DEPTInt be written to a disk file in a text format. Hence, when DEPTInt is exited the program gives the user one last chance to write this file if he has forgotten.

HEXInt:
======

One of the most frequently used 2DNMR experiments here at Lilly is carbon-proton correlation, which for historical reasons I won't go into we call HETSEXBB. To facilitate the interpretation of the HETSEXBB spectrum we have written the Pascal program HEXInt.

HEXInt asks the user for the file name of the .SMX file containing the HETSEXBB spectrum to be interpreted. The program reads the 2D parameters it will need in the analysis, and also reads in and displays the F2 projection.

HEXInt uses the text file created by the program DEPTInt as a guide the interpretation of the HETSEXBB spectrum, and so in the

next step asks for the name of the file containing the table created by DEPTInt. The program reads this table and immediately starts the correlation process. If the DEPTInt table records a peak as being a singlet, HEXInt simply prints the carbon chemical shift and proceeds to the next peak. For all other multiplicities the program displays an expansion of the F2 projection with the peak under analysis marked by a red line, then reads and displays the vertical slice at this carbon chemical shift. For peaks with either "d" or "q" multiplicities the program simply finds the most intense peak in the slice, computes its proton chemical shift, and prints a line recording the multiplicity, and carbon and proton chemical shifts for this site. For methylene peaks, the program sends the slice spectrum to a crude home-made peak picker, which records the most intense peaks of this slice. If one peak is found to be much more intense than any of the others, the methylene group is assumed to bear protons which have chemical shift equivalence, and the chemical shift of this peak is computed and recorded. For other cases the program attempts a very simple AB-analysis of the spectrum, and returns the shifts of these two protons if successful.

In the final step of the program the user is asked for a file name for storage of the final table of data in a text format. This file can be transferred to other computers for inclusion into reports, or used in additional analysis of the NMR data.

These programs are undergoing testing in our laboratories, but we hope to have them ready to send them to ABACUS this fall.

Sincerely,

LILLY RESEARCH LABORATORIES

Douglas E. Dorman, Ph.D.
Research Scientist
Physical Chemistry Research

DED:JCW

333-66

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POLYMER PRODUCTS DEPARTMENT
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April 3, 1986

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

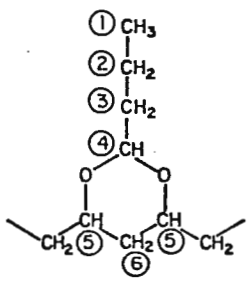
TWO-DIMENSIONAL J-RESOLVED NMR OF SYNTHETIC POLYMERS

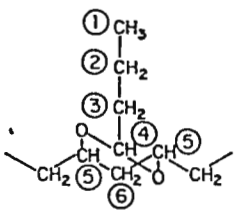
Proton NMR is a powerful tool for analysis of synthetic polymers since the chemical shifts and homonuclear coupling constants are sensitive to structural variables. However, the proton spectra of polymers are typically characterized by broad, ill-defined resonances due to spectral overlap. Consequently, the chemical shifts and coupling constants are difficult to extract from conventional proton spectra.

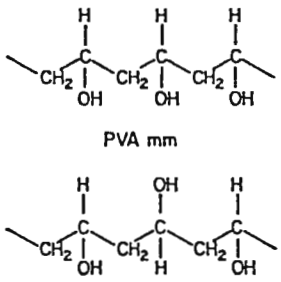
Two-dimensional J-resolved spectroscopy provides a means to extract these parameters since the chemical shift and J-coupling are separated along the two frequency axes. Application of this technique to synthetic polymers requires special considerations due to the short spin-spin relaxation times (T_2) typically associated with protons in synthetic polymers. If the T_2 is too short, the signal is lost during the evolution period of the 2D J-resolved experiment.

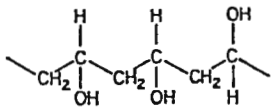
This is illustrated with poly(vinyl butyral) which is a copolymer of poly(vinyl alcohol) (PVA) and butyraldehyde rings as shown on the next page. The downfield region of the proton spectrum in DMSO at 100°C contains methine and hydroxyl protons in different environments. Although no splitting is observed in the normal spectrum for methine 4 in meso and racemic butyraldehyde rings, triplets are clearly observed (due to coupling to methylene 3) in the J-resolved spectrum. Furthermore, several multiplets with different chemical shifts but the same coupling constants are observed for each resonance due to sensitivity to longer sequences. The chemical shifts and coupling constants associated with methine 4 are measured easily from the 2D spectrum. However, the coupling patterns for PVA type methine protons are not observed. Other evidence indicated this is due to short T_2 relaxation

a) Typical structures associated with poly(vinyl butyral)

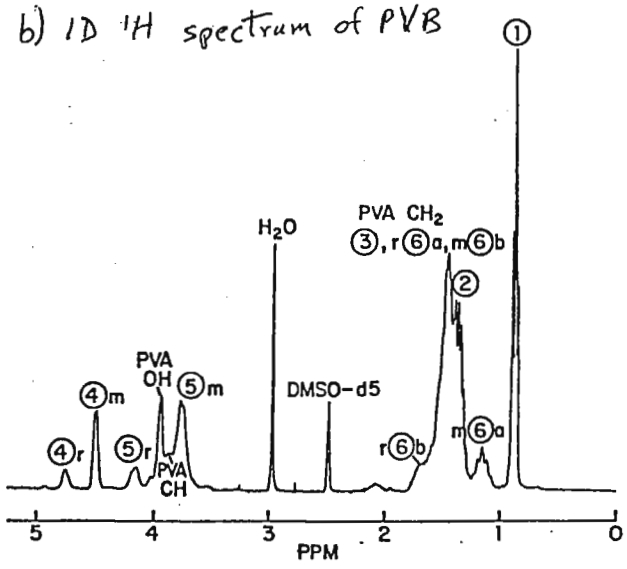




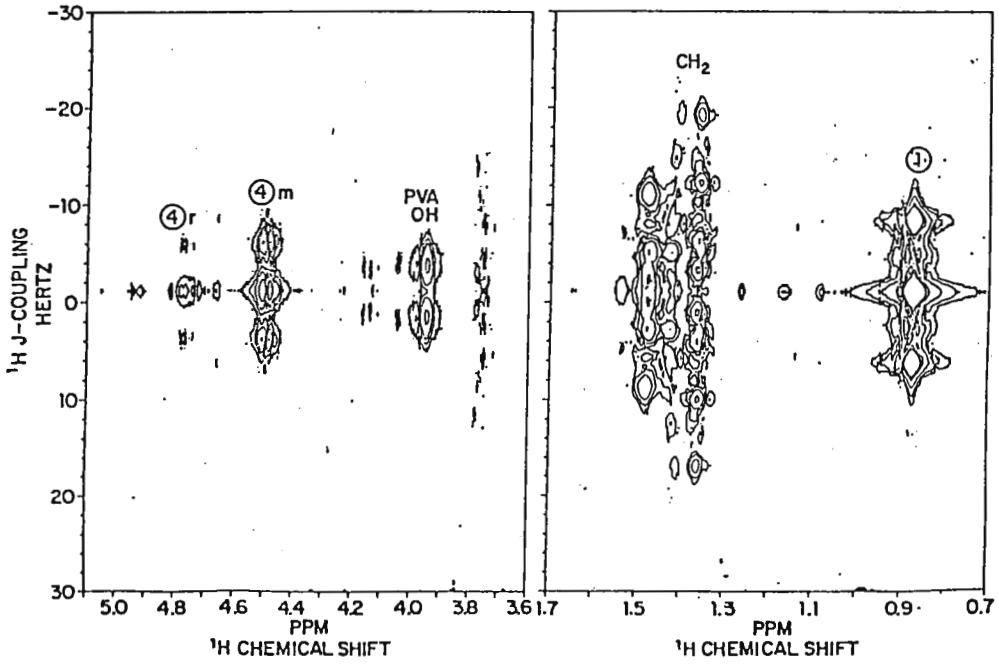




b) 1D ^1H spectrum of PVB



c) 2D J-resolved spectrum



times even at 100°C. However, this has some good consequences. The doublets observed in this region must correspond to PVA hydroxyl protons in different stereosequences. Hence, the absence of PVA methine multiplets allows the hydroxyl chemical shifts and coupling constants to be measured easily without interference from methine resonances. The methylene region is too complex to interpret in detail due to strong coupling effects. This example illustrates that 2D J-resolved spectroscopy is a powerful method for interpretation of polymers spectra, but it must be used with caution.

Martha D. Bruch

Martha D. Bruch

Warner-Lambert Company
Pharmaceutical Research Division
2800 Plymouth Road
Ann Arbor, Michigan 48105
313 996-7000

WARNER LAMBERT

We have an employment opportunity for a chemist at the B.S./M.S. level. The principal responsibilities for this position are in the field of NMR spectroscopy and involve (1) the operation and maintenance of a superconducting NMR system, (2) interpretation of spectral data, and (3) application of advanced NMR techniques. This position requires a knowledge of FT/NMR and organic chemistry. Experience with computers is also desirable.

The Pharmaceutical Research Division of Warner-Lambert/Parke-Davis is located in Ann Arbor, Michigan. Drug discovery and development are the primary focus of activity at this facility; which is located adjacent to the north campus area of the University of Michigan.

Please have interested candidates send their resume and list of references to:

Dr. Christopher Rithner
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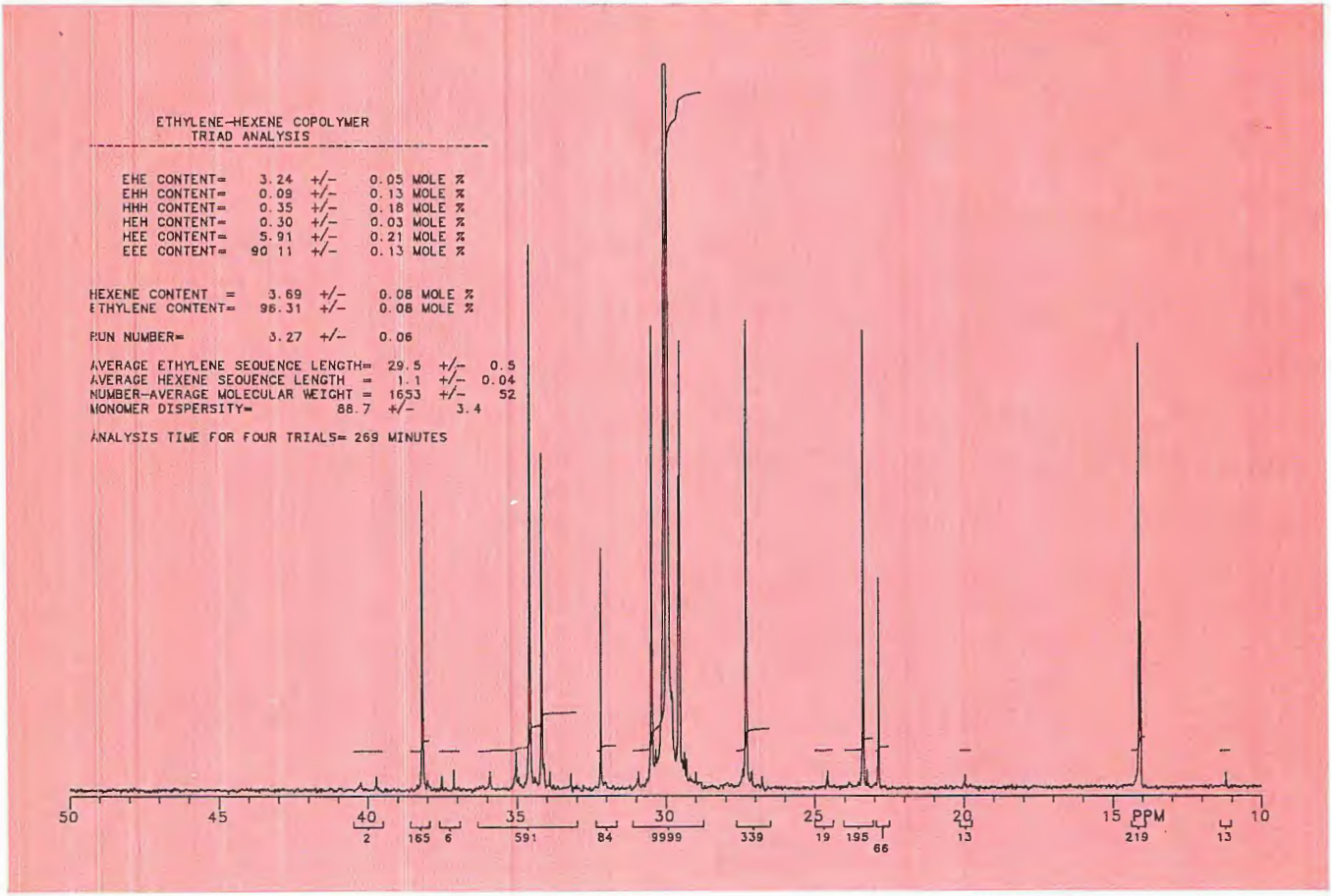

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UCSD MEDICAL CENTER

AMI.

May 28, 1986

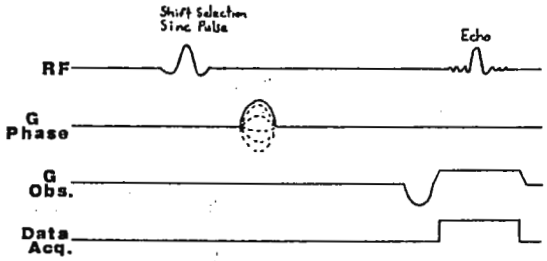
Dr. Barry L. Shapiro
Department of Chemistry
Texas A&M University
College Station, TX 77843

Chemical Shift Resolved Imaging

Dear Barry,

There has been a lot of interest in imaging nuclei other than protons. Unfortunately, most nuclei are too dilute and or insensitive to provide adequate signal to noise in an imaging experiment. One obvious candidate for multinuclear imaging is ^{19}F, with 83% sensitivity relative to protons. Sufficient amounts of ^{19}F can be introduced <u>in vivo</u> by injection, drinking or breathing of non-toxic perfluorocarbon "blood substitutes".

The imaging of perfluorocarbon distributions is complicated by large ^{19}F chemical shift dispersion. The large shifts result in unacceptable blurring of images obtained via ordinary ^{1}H imaging sequences. Our solution to this difficulty is to image each resolvable resonance separately using the shift selective pulse sequence shown below.



The selective pulse excites only the on resonance peak, since no slice select gradient is applied. This sequence is similar to many of the rapid imaging sequences available on commercial MRI units, in that reduced flip angles may be used to obtain rapid signal accumulation. The shift selective sequence produces images which are projections, much like an ordinary X-ray image. Partial volume effects due to the lack of a slice selection pulse are acceptable, since there is no signal from endogenous ^{19}F to complicate the image.

We have successfully used this sequence to image perfluoroctylbromide in the blood stream of rats and in the lungs of rabbits at 2.0T. We plan to use the same sequence to image each ^{19}F resonance in a multiplexed fashion, as soon as control of the spectrometer frequency becomes available in a compiler update. By performing multiplexed imaging (i.e., one resonance is acquired while the others relax), we hope to increase signal to noise dramatically.

Sincerely,

Joel

Joel F. Martin

Robert F. Mattrey

333-72

DEPARTMENT OF HEALTH & HUMAN SERVICES Public Health Service

National Institutes of Health
Bethesda, Maryland 20892
Building :1
Room :118
(301) 496- 2215

April 24, 1986

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

Dear Barry,

Please include the following announcement in your next newsletter:

Manager - In Vivo NMR Research Center

The National Institutes of Health is seeking a specialist in nuclear magnetic resonance to manage the operation and development of a new In Vivo NMR Research Center to be located on the NIH campus in Bethesda, Maryland. The new facility (designed, built and equipped by General Electric Medical Systems) will initially include three NMR imaging and spectroscopy systems: a 2.0 Tesla whole-body instrument, a 2.0 Tesla 45-cm bore instrument, and a 4.7 Tesla 40-cm bore instrument. The manager must be a person with advanced level training, preferably with a Ph.D. in chemistry or physics, and must have a strong background in NMR instrumentation and techniques. Hands-on experience with in vivo NMR research will be desirable.

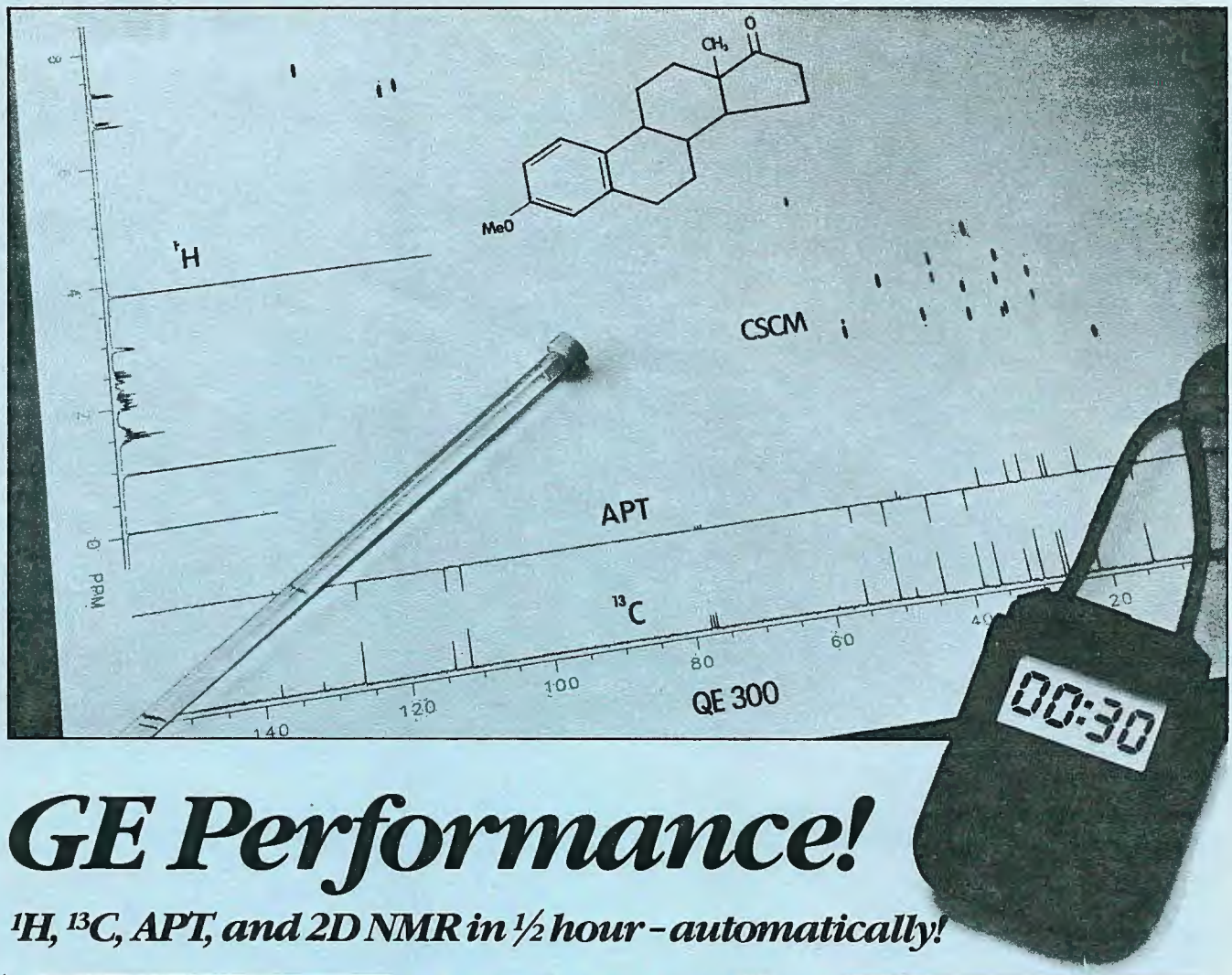
The Center, which will be housed in a new addition to the NIH Clinical Center, is designed to accommodate the instrumentation needs for NIH scientists who will come to the facility to perform their experiments. The principal responsibility for the manager will be to insure the smooth operation of the Center, including (a) oversight of the day-to-day operation of the facility, (b) coordination of instrument maintenance requirements (a full-time engineer will be on-site), and (c) ensuring the proper level of training is provided for all scientists utilizing the equipment. It is anticipated the manager will participate in the research activities of the Center, both by establishing collaborative research programs with NIH scientists and by pursuing individual research efforts.

Interested individuals should send a resume, a list of publications, and the names, addresses and phone numbers of at least three references as soon as possible to Dr. Cherie L. Fisk, Office of Research Services, Building 1, Room 13, National Institutes of Health, Bethesda, Maryland 20892. A starting date of October 1 is preferable; however, this is flexible depending upon availability of the candidate.

Sincerely,

Ted

Edwin D. Becker, Ph.D.
Associate Director
for Research Services



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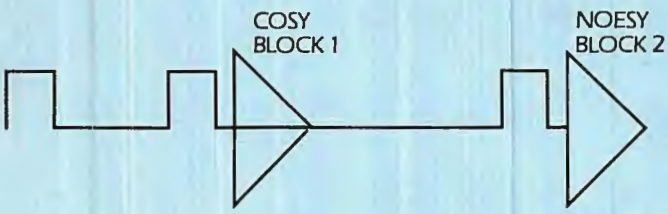
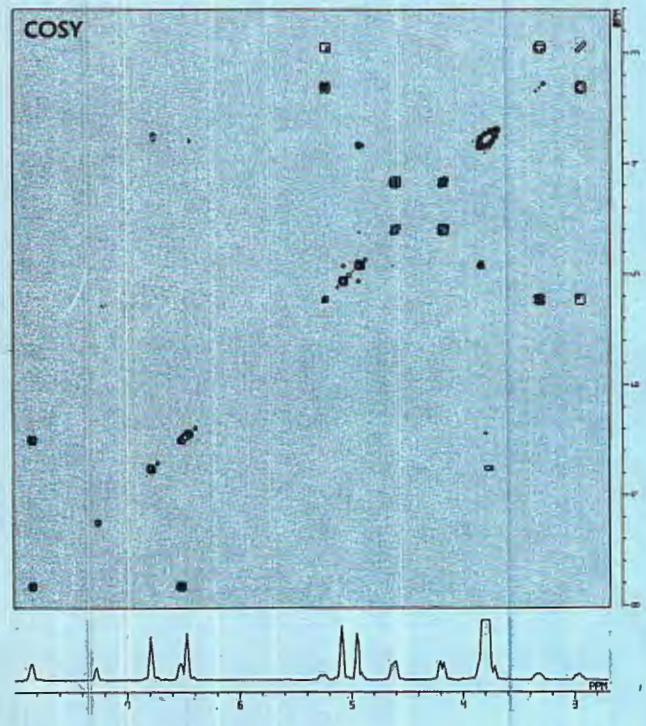
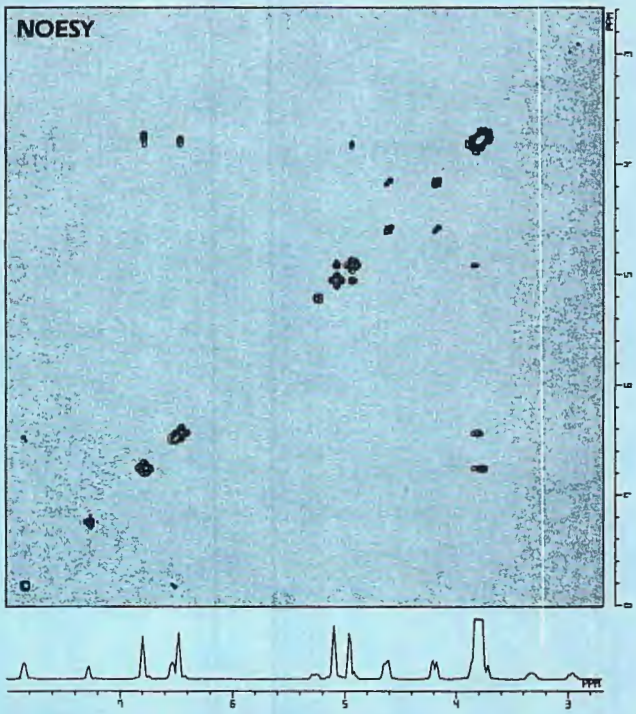


- Simultaneous acquisition of COSY and NOESY





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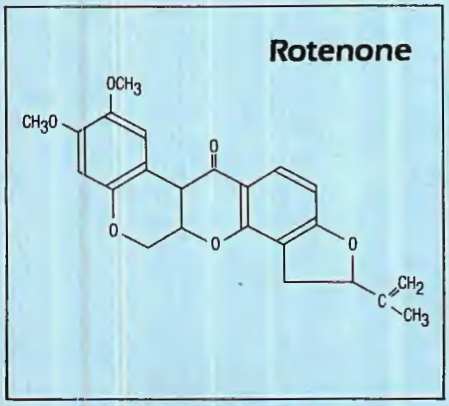
*COCONOSY (Haasnoot, et. al., J. Magn. Reson., 56,343 [1984])



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