BRIAN SYKES

TEXAS A&M UNIVERSITY

NMR NEWSLETTER

No. 356

May 1988

Continued on page 76

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FORTHCOMING NMR MEETINGS

2nd European Congress on NMR in Medicine and Biology, June 23-25, 1988; Berlin, West Germany; contact Prof. R. Felix, Dept. of Radiology, Charlottenburg University Hospital, Spandauer Damn 130, D-1000 Berlin 19, West Germany.

30th Rocky Mountain Conference, July 31 - August 5, 1988; Radisson Hotel, Denver, Colorado; NMR and EPR symposia; contact M. M. Reddy, U.S. Geological Survey, 5293 Ward Rd., Arvada, CO 80002, (303) 236-3601.

XIII Intl. Conference on Magnetic Resonance in Biological Systems, Aug. 14-19, 1988; Madison, Wisconsin. See Newsletter 349, 60.

NATO Summer School: "A Methodological Approach to Multinuclear Magnetic Resonance in Liquids and Solids: Chemical Applications", August 22 - September 2, 1988; Maratea, Italy; See Newsletter 353, 76.

XXIV Ampere Congress on Magnetic Resonance and Related Phenomena, August 29 - September 3, 1988; Poznan, Poland; Dr. S. Hoffmann, Instytut Fizyki Molekularnej PAN, ul. Smoluchowskiego 17/19, 60-179 Poznan, Poland.

Teaching Course on Nuclear Magnetic Resonance, September 5 - 9, 1988; Trondheim, Norway; Ms. I. S. Gribbestad, The MR Center, N-7034, Trondheim, Norway.

European Workshop on Nuclear Magnetic Resonance: Seminar on Contrast in MRI and MRS, September 12 - 13, 1988; Trondheim, Norway; Ms. I. S. Gribbestad, The MR Center, N-7034, Trondheim, Norway.

27th Annual Eastern Analytical Symposium and Exposition, October 2 - 7, 1988; New York Hilton Hotel, New York; General Chairman - Dr. Harvey S. Gold, Polymer Products Dept., E256/308, E. I. du Pont de Nemours & Co., Wilmington, DE 19898; (302) 695-3669.

International Post-Graduate Course 'NMR in Agriculture, Plants and Products', October 3 - 15, 1988; Wageningen, The Netherlands; Dr. Ir. J. H. de Ru, Foundation for Post-Graduate Courses, Agricultural University, Hollanseweg 1, NL-6706 KN Wageningen, The Netherlands.

Additional listings of meetings, etc., are invited.

All Newsletter Correspondence Should Be Addressed To:

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

(415) 493-5971

DEADLINE DATES

No. 358 (July) ———— 17 June 1988
No. 359 (August) ———— 22 July 1988
No. 360 (September) —— 19 August 1988
No. 361 (October)——23 September 1988

The University of Rhode Island, Kingston, RI 02881-0801
Department of Chemistry, Pastore Chemical Laboratory (401) 792-2318

March 18, 1988
(received 3/26/88)

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

Dear Barry,

COMPARISON OF TRAF AND GAUSSIAN APODIZATION FUNCTIONS

Approximately one year ago we reported (1) an apodization function (TRAF) that may be useful for improving resolution and/or S/N in NMR spectroscopy. We have programmed this function in PASCAL on our Bruker AM-300 and have been using it for approximately 6 months. Unfortunately, our programming capability is such that we are unable to program this function in machine language or to employ the array processor, and multiplication of an FID by TRAF requires approximately one minute. Nevertheless, we have found it useful and would like to report one interesting finding; namely, how this function compares with the standard Gaussian under certain conditions.

On most occasions we noticed, as we previously reported (2), that the use of the TRAF function gave better results than the Gaussian. However, on some occasions it gave the same or similar results. Consequently we have decided to investigate the conditions under which the two functions are similar. Our investigation is not complete, but we would like to report some of our preliminary results.

We have found that for relatively long acquisition times (t_a) the results from the use of the two functions are comparable. For normal, or slightly shorter than normal times, the TRAF function is considerably better. It is important to note that in this context, acquisition times are not measured in seconds. Instead, they must be measured in number of T_2^*s; i.e., the acquisition time (in seconds) divided by T_2^*. Employing these units we mean that a "normal" acquisition time is 3. After this time, the FID has decayed to 5% of its initial amplitude, A_o.

The spectra below show the resonance of the isopropyl proton in menthol. Figures 1A and 1B are the real spectra obtained from the untreated FIDs, with t_a = 3 and 6, respectively. Figures 1C and 1D are the results after treatment with the TRAF function, and 1E and 1F are the results after treatment with the Gaussian function. Figure 2 is a plot of S/N vs. t_a for these spectra, as well as for some intermediate cases.

The results are explainable when one considers the shape of the resultant FID; i.e., the new amplitude of the FID after it has been multiplied by the apodization function. When the TRAF is used the product function is always odd — this is why it can increase the resolution while still attenuating the

FIGURE 1

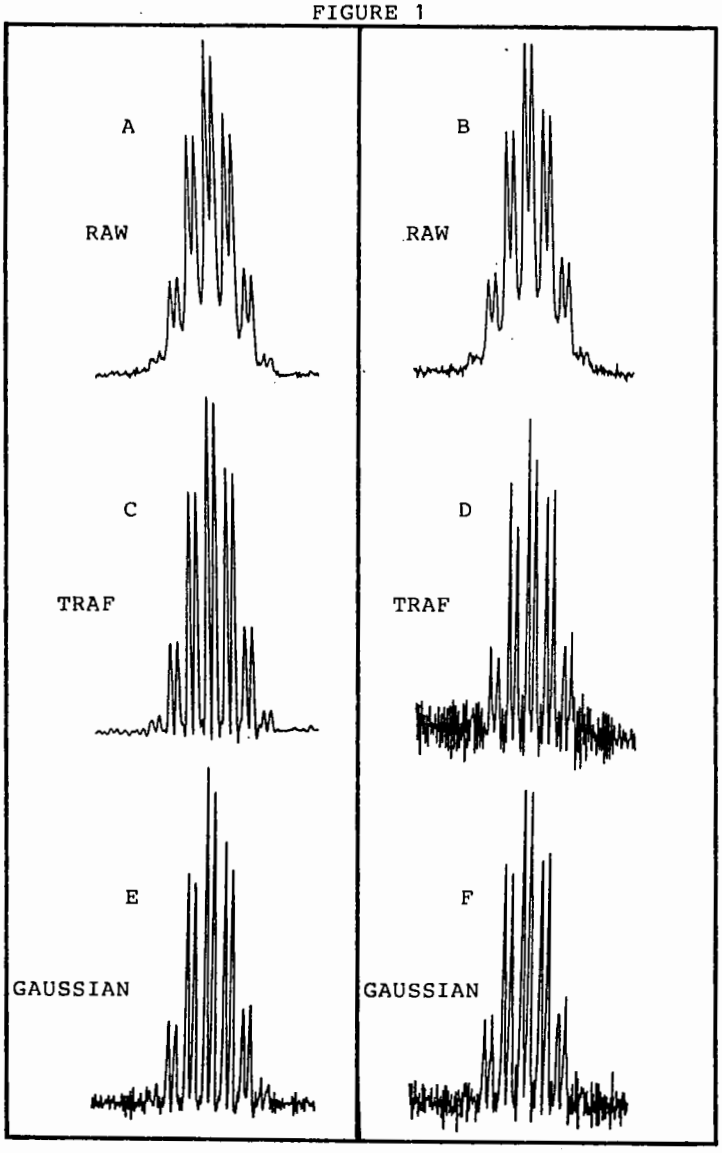
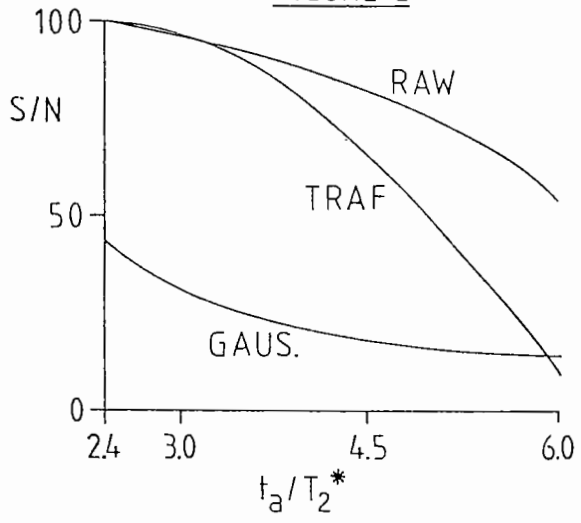


FIGURE 2



tail of the FID. As previously reported (1), only odd product functions will eliminate the side-bands that cause line-broadening in a phased real spectrum, and the TRAF function accomplishes this while simultaneously taking advantage of the matched filter concept for optimum S/N. However, to achieve this oddness, the TRAF function forces the product function to be 0.5 A_o at 0.5 t_a. The Gaussian, on the other hand, can produce a variety of shapes for the product function, because it has two adjustable parameters, and does not require oddness. But for the Gaussian to yield the same resolution as the TRAF, it must also produce an odd product function. This can be achieved with the two parameters, but the appropriate weighting for best S/N will not be accomplished, and the S/N will be inferior. This explains the results at $t_a = 3$.

When t_a is long (e.g., 6), the center of the collected FID is almost all noise, and the TRAF function multiplies this portion up to 0.5 A_o to produce oddness. This results in a drastically reduced S/N, which approaches or is worse than that obtained from the Gaussian when the two parameters are adjusted to yield the same resolution. This explains the results at $t_a = 6$.

Our conclusion is obvious. When high resolution is needed and S/N is an important consideration, do not acquire the FID beyond 3 or 4; the FID amplitude is only 0.02 A_o when $t_a = 4$. For more spectral data points, zero-filling should be used after multiplying by the TRAF function.

Please credit this to the account of Dan Traficante.

Sincerely,

Michael A. McGregor Daniel D. Traficante

MAM/DDT/rks

(1) D.D. Traficante and G.A. Nemeth, J. Magn. Reson. 71, 237 (1987).

(2) D.D. Traficante and D. Ziessow, J. Magn. Reson. 66, 182 (1986).

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POSTDOCTORAL POSITIONS in the area of biomolecular structure determination by high-field NMR spectroscopy. Current equipment includes two supercon NMR spectrometers (Bruker WH-400 and CXP-200) an iron magnet system (HX-90). A 600 MHz (AM-600) spectrometer and a 4.7T 15 cm imaging/spectroscopy magnet system will be installed during the summer at the NMR Facility. Computer programming experience is a definite asset. Interested candidates should send a resume and arrange for three letters of recommendation to reach Dr. N. Rama Krishna, Director, NMR Core Facility, CHS-B31, University of Alabama at Birmingham, Birmingham, AL 35294. EO/AAE

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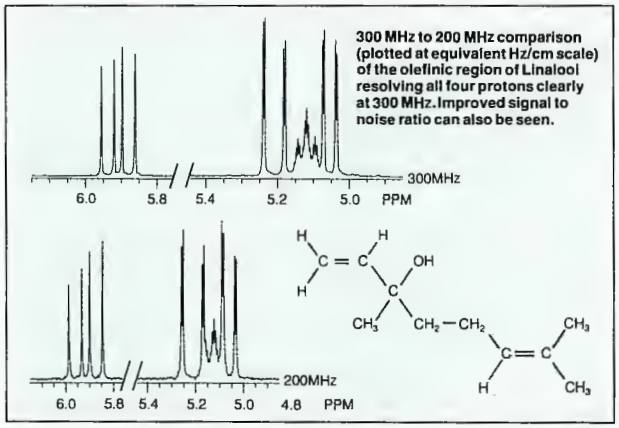

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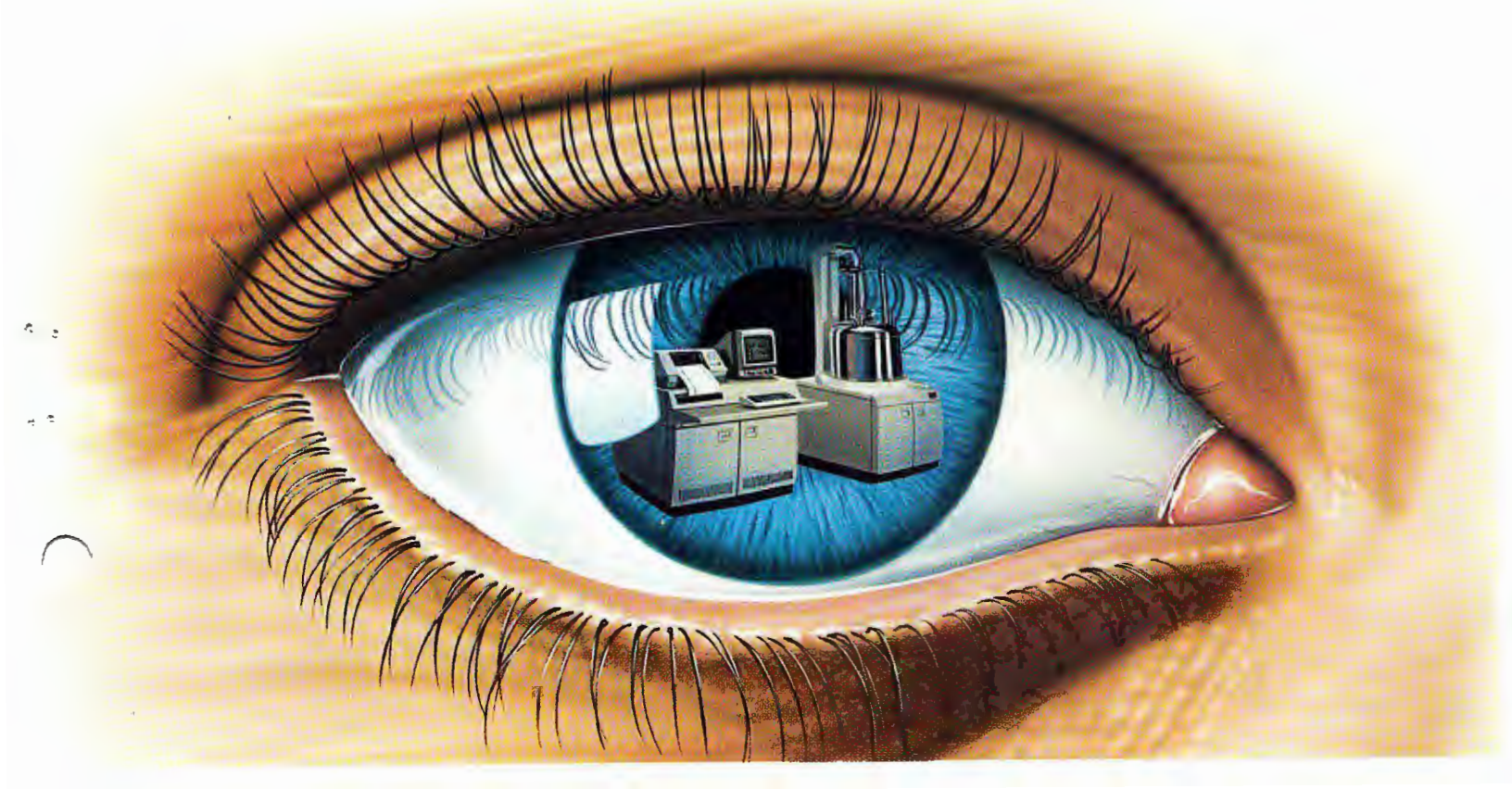

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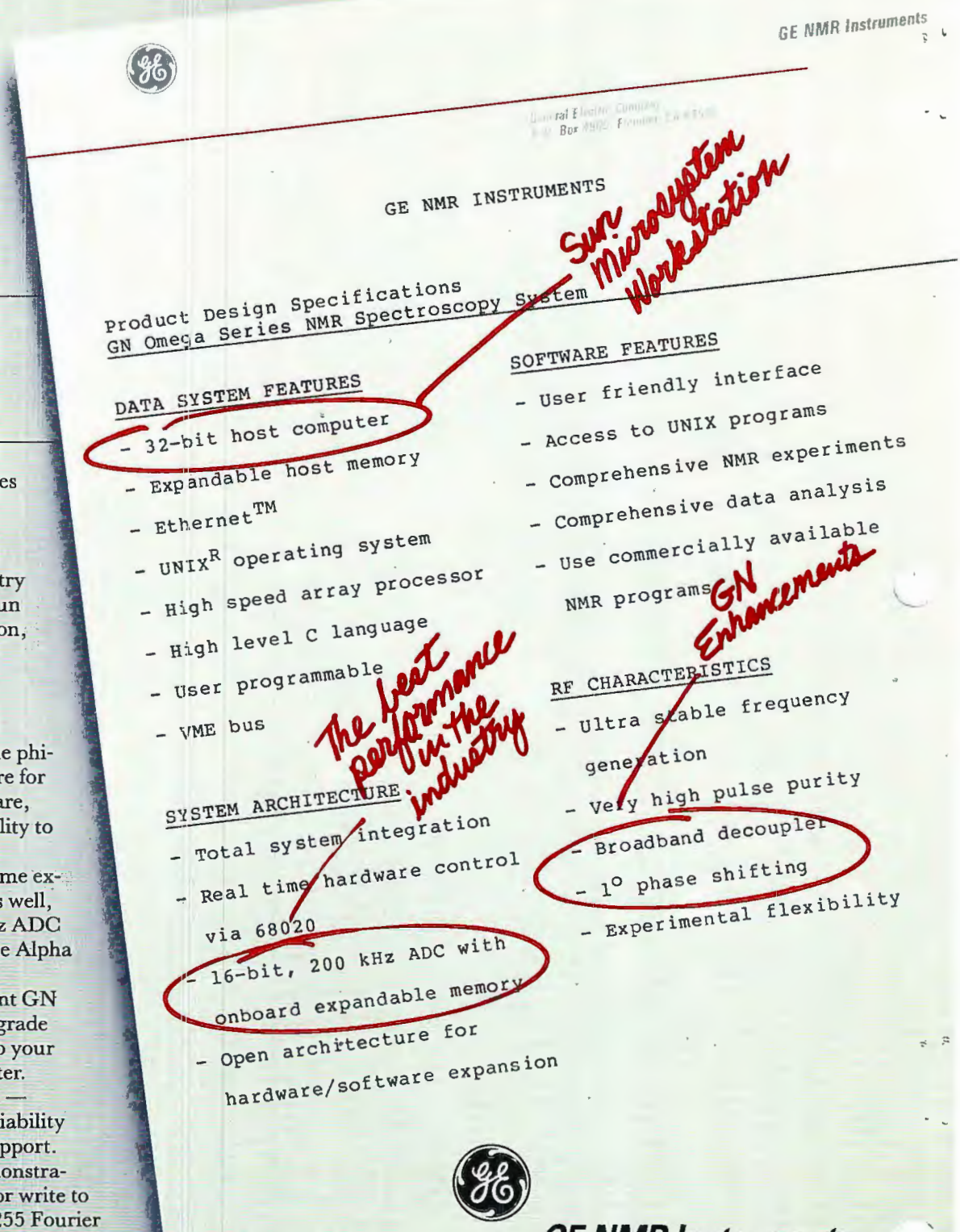

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GRUPPO MONTEDISON

MONTEDIPE

March 15, 1988
(received 3/28/88)

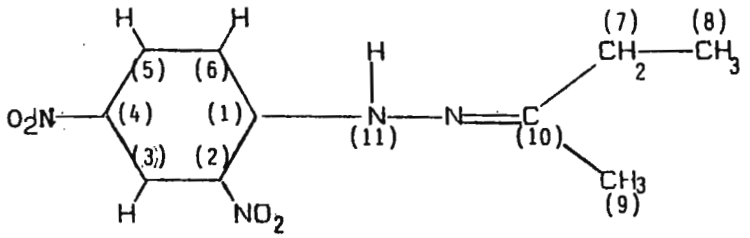
Professor BERNARD L. SHAPIRO
Editor Publisher
TAMU NMR NEWSLETTER
966 Elsinore Court
PALO ALTO California 94303
U.S.A.

MONTEDIPE/PM/CER
Stab.to Petrolchimico
Via della Chimica, 5
30175 PORTO MARGHERA VE
ITALY

Dear Professor Shapiro,

Title: Interpretation of ^{13}C and ^{1}H NMR spectra of 2,4-Dinitro phenylhydrazone of 2-Butanone and assignment of related syn-anti isomers in $CDCl_3$ solution.

In this letter, we report the full interpretation of the ^{13}C and ^{1}H NMR spectra of 2,4-dinitrophenylhydrazone of 2-butanone [1] in $CDCl_3$ solution, at room temperature. The ^{13}C chemical shifts and $J_{^{13}C-H}$ coupling constants for the aliphatic and aromatic parts of the molecule are reported in Table 1, compared with the experimental chemical shifts of 2-butanone (1) for the aliphatic moiety, while the assignment of the ^{13}C chemical shifts for the aromatic ring has been done on the basis of hetero-gated-decoupling and with the use of additivity effect relationships obtained on monosubstituted benzenes (2).



[I]

Table 1

^{13}C chemical shifts and $J_{^{13}C-^{1}H}$ coupling constants of 2-4-dinitro phenylhydrazone of 2-butanone, 0.53 M in $CDCl_3$, with reference to TMS.

δ(ppm)	$^{1}J_{C-H}$ (Hz)	Multiplicity	Assignment	2-butanone (1) δ(ppm)
9.49	126.5	Quartet	C_8 anti	
				7.40
10.59	127.5	"	C_8 syn	
15.79	126.5	"	C_9 anti	
				28.9
22.94	126.0	"	C_9 syn	
24.05	129.8	Triplet	C_7 anti	
				36.40
32.43	129.8	"	C_7 syn	

Aromatic part

δ(ppm)		$^{1}J_{C-H}$	$^{2}J_{C-H}$	$^{3}J_{C-H}$(Hz)	Multiplicity	Assignment
116.53	117.0*	177.0	3.0	/	Doublet	C_6
123.48	119,8	165.0	/	4.5	Doublet	C_3
129.07	139.6	/	4.5	7.5	Multiplet	C_4
129.92	130.4	169.5	<3	5.4	Doublet	C_5
137.65	136.1	/	/	/	Unresolved multiplet	C_2
145.38	142.6	/	3	6	Multiplet	C_1
		$J\ ^{13}C-NH = 4.5$				
159.29	/	/	/	/	Unresolved multiplet	C_{10} syn
160.33	/	/	/	/	Unresolved multiplet	C_{10} anti

* ^{13}C chemical shifts calculated on the basis of additivity relationships (2).

The table 2 reports the ^{1}H NMR parameters of the molecule [1]

Table 2

^{1}H chemical shifts and coupling constants of 2-4.dinitro phenylhydrazone of 2-butanone, 0.24 M in $CDCl_3$, with reference to TMS.

δ(ppm)	Assignment
1.218	H_8 syn
1.253	H_8 anti
2.070	H_9 syn
2.150	H_9 anti
2.455	H_7 anti
2.473	H_7 syn

$J_{H_7-H_8}$ syn = $J_{H_7-H_8}$ anti $\simeq$ 7.50 Hz

Aromatic part

δ(ppm)	J_{H-H} (Hz)		Assignment
7.960		*J_{H6-H11} =-0.02±0.01	H_6
8.280	J_{H5-H6}= 9.63±0.01	*J_{H5-H11} = 0.45±0.01	H_5
9.100	J_{H3-H5}= 2.58±0.01	J_{H3-H6} =-0.09±0.01	H_3
11.033			H_{11} syn
11.140			H_{11} anti

* The couplings are equal in both syn and anti isomers.

The relative abundance of the anti conformer obtained from ^{13}C, ^{1}H, and HPLC data is 19%, 18,9% and 18% respectively and agrees well with that reported by Karabatsos et al., 20% observing the ^{1}H aliphatic mojety NMR spectrum of the molecule. [1]

Sincerely yours

F. Coletta* F. Gottardi* Francesca Gottardi

G. Gurato C. Valentini**

* Departement of Physical Chemistry of the University, Via Loredan 2 - PADOVA

** Laboratorio Chimico delle Dogane - VENEZIA

References

(1) G.E. Hawkes, S.K. Herwig, J.D. Roberts. J. Org. Chem. 39 1017 (1974)
(2) A. ur Rahman - Nuclear Mag. Resonance pg. 160 - Springer N.Y. (1986)
(3) G.J. Karabatsos, J.D. Graham, F.M. Vane. J. Am. Chem. Soc. 84, 753 (1962)

UNIVERSITÉ DE LAUSANNE

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Dr. Bernard L. Shapiro
966 Elsinore Court
Palo Alto, CA 94303
USA

March 15th 1988
(received 3/24/88)

Dear Dr. Shapiro,

For some obscure reason, our group seems to be constantly preoccupied with experiments that employ pulses with small flip angles. We've advocated the use of such pulses in COSY, in NOESY, and in several other applications. The latest attempt to rewrite the history of NMR with small pulses aims at multiple-quantum excitation. The best known way of exciting such coherences (best known to liquid NMR people anyway) employs a [90-½τ-180-½τ-90] sandwich with a duration that must be matched to the (unknown) size of the coupling constants (ideally, $\tau = (2J)^{-1}$ for two-spin-½ systems). Of course, this requirement cannot be met if there is a spread of coupling constants, and the efficiency of multiple-quantum excitation is a rather complicated function of these couplings in larger spin systems.

We've recently tried to circumvent these matching problems by using sequences that do not contain any fixed delays. The simplest of these sequences may be written

$$90^\circ - \tfrac{1}{2}t_1 - 30^\circ - \tfrac{1}{2}t_1 - 90^\circ - t_2$$

where the 30° pulse in the middle of the evolution period converts some of the single-quantum coherence excited by the first pulse into double-quantum coherence, which can be selected by suitable phase-cycling.

As one might expect, the sensitivity is poorish, typically one-third of the conventional experiment in favourable cases. The spectra appear exceedingly complicated, since they contain twice as many multiplets as normal double-quantum spectra. One does actually get a small compensation for the trouble: the efficiency of the excitation is quite homogeneous, and hence the amplitude of the signals is uniform in so far as antiphase multiplet components do not mutually cancel.

This technique would have been published long ago had we been able to agree on a name. Should the method be called ½(1+2)-quantum spectroscopy? Would Alex Pines approve of fractional quantum numbers?

Yours truly,

Dominique Limat Stephen Wimperis Geoffrey Bodenhausen

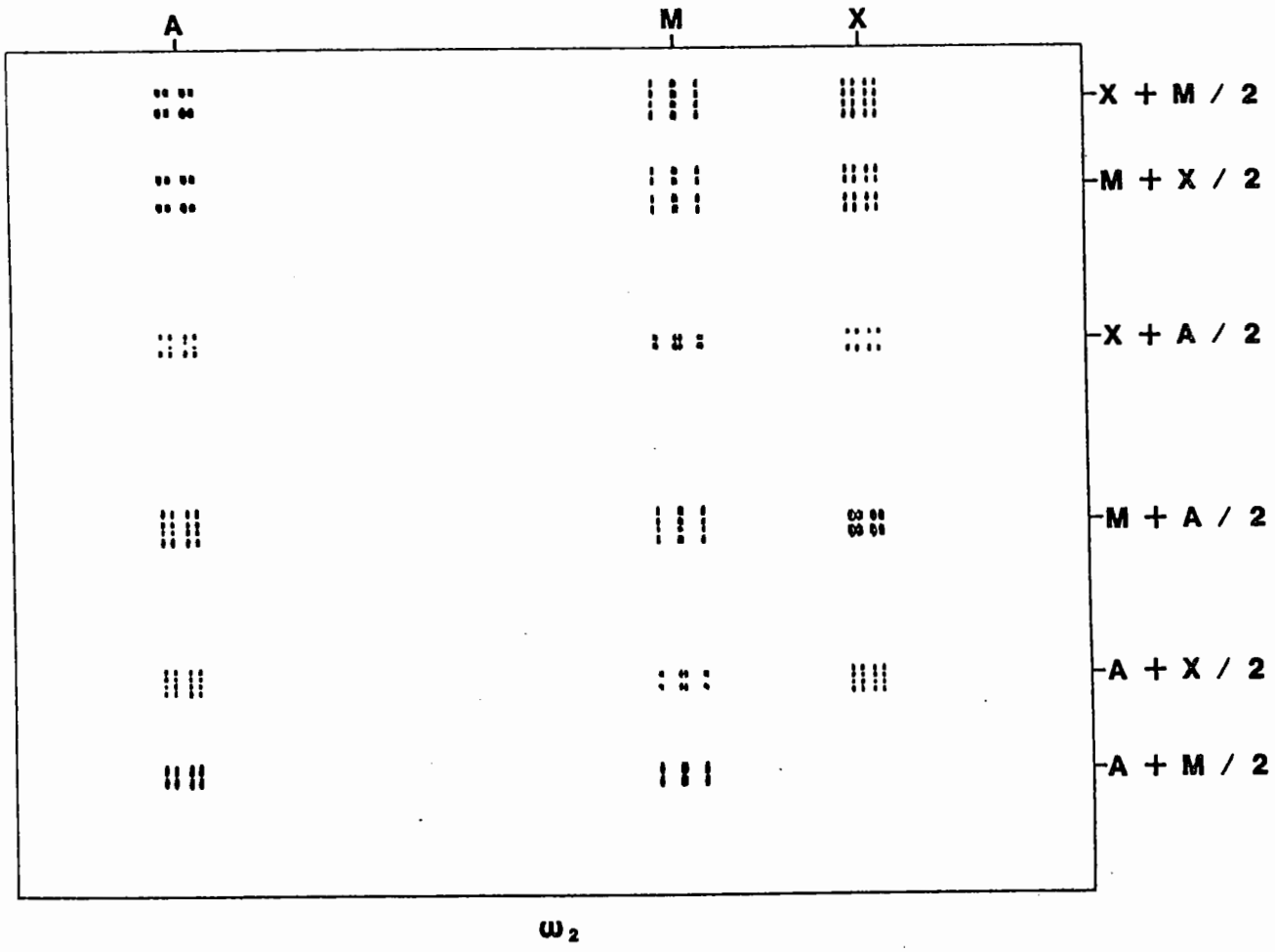

Figure 1. Two-dimensional spectrum of the AMX system of 2,3-dibromopropanoic acid, recorded with the sequence 90° - $\frac{1}{2}t_1$ - 30° - $\frac{1}{2}t_1$ - 90° - t_2 on a Bruker AM 400 spectrometer. The spectral widths are 400 and 500 Hz in the ω_1 and ω_2 dimensions respectively. The maximum spread in the ω_1 dimension can be 1.5 times the spectral width in ω_2. The effective chemical shifts in ω_1 correspond to averages of single- and double-quantum precession frequencies. The spectrum comprises 12 direct connectivity and 6 remote connectivity multiplets. Although no prior knowledge about the coupling constants has been used when setting up the experiment, all signals have uniform intensities except for remote multiplets which are in antiphase with respect to effective couplings that are vanishingly small compared to the linewidth.

THE UNIVERSITY OF SYDNEY

DEPARTMENT OF BIOCHEMISTRY

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21 March 1988
(received 3/28/88)

Prof. B.L. Shapiro
966 Elsinore Court
PALO ALTO CA 94303
USA

'Overdetermined' 1-D NMR exchange Analaysis

Dear Barry,

In the Feb '87 (No. 341) TAMU Newsletter, Wade and Johnston described a simple new method for measuring rate constants in chemically exchanging systems. The procedure uses the pulse sequence, $\pi/2$-t_1-$\pi/2$-t_m-$\pi/2$- observe (t_2). For a 2-site reaction scheme a fully 'relaxed spectrum' and four others are recorded; the latter being obtained with two different values of t_1 each with t_m=0 and one other non-zero value. In general, to analyse an n-site scheme 2n+1 spectra are required. However, we reached an 'impasse' on several occasions when trying to run the new method to analyse the kinetics of porcine kidney mutarotase catalyzing the anomerization of [1, ^{13}C]D-glucose. The main problem was a negative eigenvalue in the 'back transformation' analysis [1]; and since the theory requires that the natural logarithm should be taken of this, no progress could be made. This stimulated us to modify the previous procedure.

We now aquire m>2n spectra; i.e., we use more different t_1 values. Data analysis is naturally more complicated since the problem is now 'overspecified'. The previously square matrices are now rectangular; but a reasonably standard matrix manipulation procedure, called QR decomposition [2], leads to an upper triangular matrix from which it is possible to solve for the required 'exponential matrix'. The rest of the analysis follows the standard 'back transformation' [1]. A bonus from the analysis is an estimate of the variances of the rate constants.

One way of viewing the original analysis is that it involves fitting an exponential function with only one ordinate value (at t=0) and one other coordinate value. Our procedure over specifies the fitting function with more data points and thus, (a) it is less sensitive to noisy data and (b) it enables estimates of uncertainties of the parameters.

The figure shows a series of ^{13}C NMR spectra (16 transients, 16 k points, spectral width 2400 Hz, inter-transient delay 10s) obtained using our Varian XL/VXR 400 spectrometer, at 162 MHz with [1, ^{13}C]D-glucose (27.2 mM) and mutarotase (Sigma, 0.42 mg/ml) at pH 7.4 and 37°C. Figure A is the 'fully relaxed' spectrum showing the β- and α-anomer peaks of the labelled glucose. The frequency difference between the peaks ($\Delta\nu$) was 383.9 Hz. The upper spectra, B-E, were acquired with t_m=0 and t_1 equal to $1/16\Delta\nu$, $3/16\Delta\nu$, $5/16\Delta\nu$ and $7/16\Delta\nu$ respectively. The corresponding lower spectra were acquired with the same parameters except t_m=1s.

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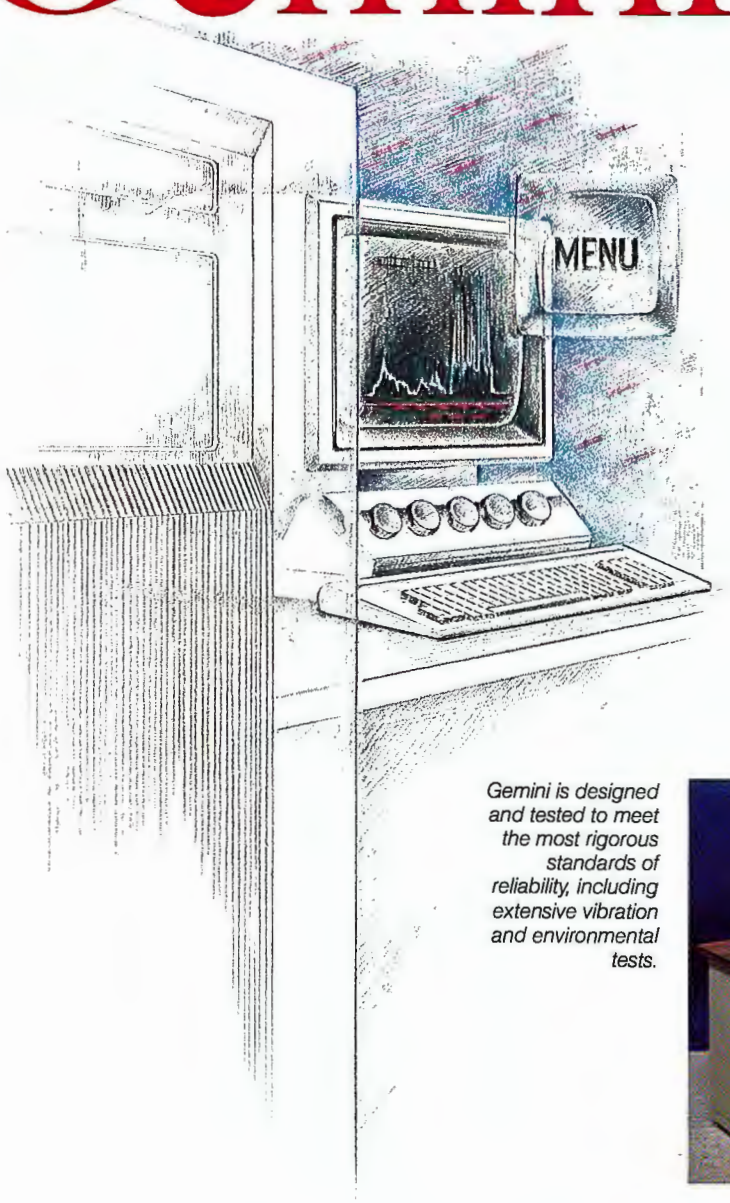

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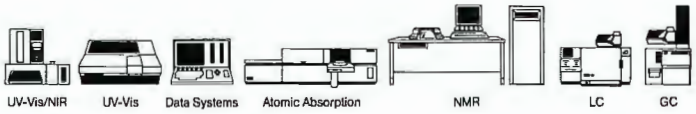

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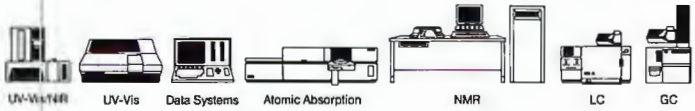

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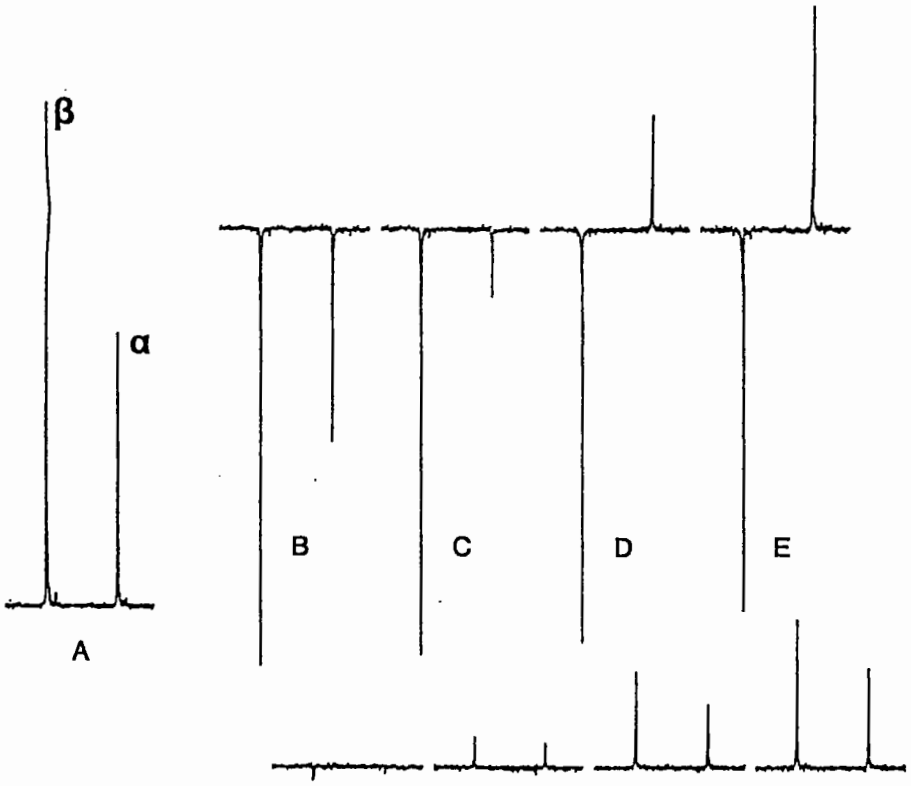
MAG-4615/338

We can illustrate the previously mentioned 'impasse' by applying the Wade-Johnston analysis to the spectral pairs B and D; this yields a negative eigenvalue, and therefore we can not obtain an estimate of the exchange rate constants. However, use of all the spectra with our new procedure gave the values $k_{\alpha\rightarrow\beta}$= 2.356±0.017 s^{-1} and $k_{\beta\rightarrow\alpha}$= 1.321±0.008 s^{-1} which were reasonably close to the values obtained from saturation transfer, *viz* 1.94 s^{-1} and 1.13 s^{-1}.

We have just submitted for publication a paper on the new procedure.



[1] P.W. KUCHEL, B.T. BULLIMAN, B.E. CHAPMAN, AND G.L. MENDZ, J. Magn. Reson. **76**, 136 (1988).

[2] G.H. GOLUB AND C.F. VAN LOAN, *in* "Matrix Computations", chap. 6, North Oxford Academic, Oxford, 1983.

Yours sincerely,

PHILIP W. KUCHEL BRIAN T. BULLIMAN BOGDAN E. CHAPMAN

:AD

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

February 16, 1988
(received 3/22/88)

Title: NOESY in water for short mixing times

Dear Professor Shapiro,

The application of sequential assignment strategies to proteins of molecular weight greater than 18000 is associated with several problems. One is the long correlation time, which necessitates a mixing time for a NOESY experiment shorter than 80 ms. This is difficult to achieve experimentally, because the two main approaches used to record NOESY spectra in water may no longer be adequate: first, presaturation of the water resonace may be precluded by rapid NH exchange, and second, the application of a variation of the NOESY sequence in which the last pulse is replaced by an excitation scheme that avoids exciting the water (e.g. a 1-1 pulse) is not applicable due to poor suppression in a quarter of the individual scans arising from radiation damping.

We found that a NOESY sequence in which the first two pulses are substituted by a combination of hard and soft pulses (1) leads to good results, even close to the water resonance. This is especially true, when Gaussian shaped soft pulses are used. The spectra obtained with the basic sequence $90_{(-x)sel}$ - 90_x - t_1 - 90_x - $90_{(-x)sel}$ - τ_{mix} - 90_x - Δ - 90_{-x} - t_2 (there is phase-cycling, of course) resemble those obtained with preirradition: in the region comprising the cross peaks between NH- and α-protons, only a small strip of +/- 25 Hz centered at the water resonance (ω_1=4.78) ppm is lost. The fact, that this strip is very narrow can be appreciated by comparing the two spectra in Fig. 1. The NOESY spectrum obtained with soft pulses is shown in A; the results of the same sequence without soft pulses is shown in B. The spectrum in B was recorded with a mixing time of 120 ms, which was the shortest possible to achieve on our spectrometer, while for B 70 ms was used.

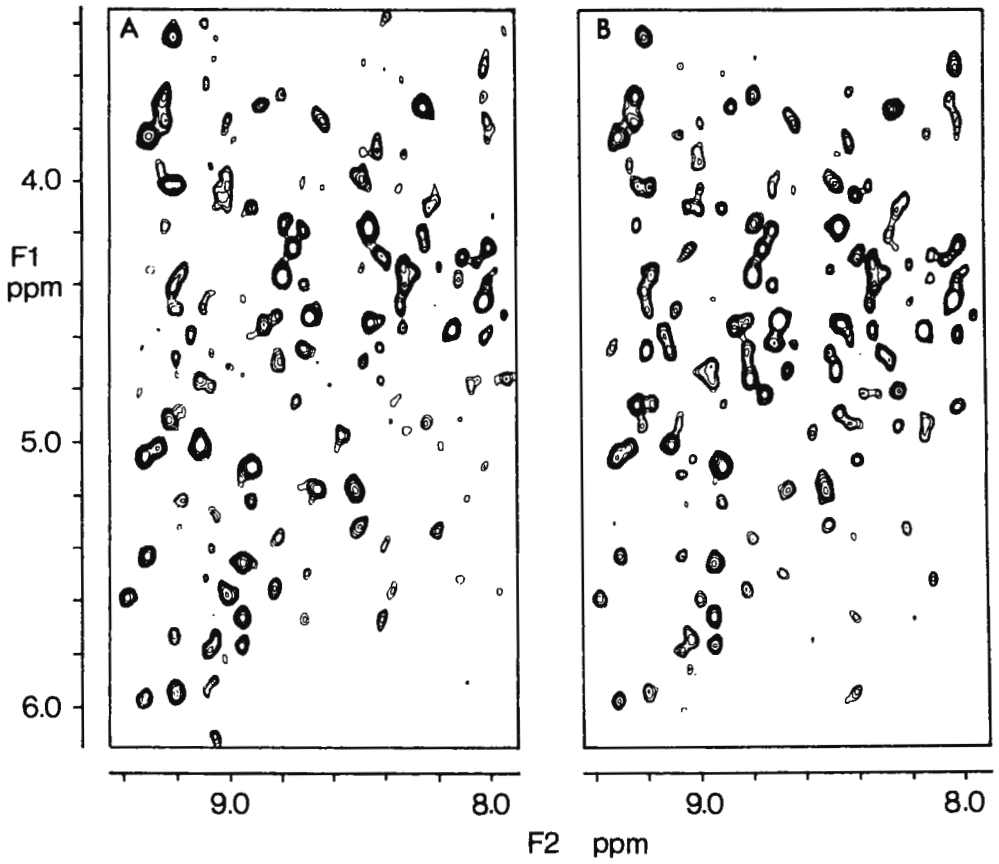

Fig. 1 (A) Soft-hard NOESY (mixing time 70 ms) and (B) NOESY with a Jump -Return 'read' pulse (mixing time 120 ms) of 1.5 mM interleukin-1α in 90% H_2O / 10% D_2O , 200 mM sodium phosphate, pH 5.0, T=20°C, 500 MHz. The length of the soft Gaussian pulses were 20ms.

Yours sincerely,

P. Driscoll H. Oschkinat M. Clore A.Gronenborn

References

1. Sklenár, V., Tschudin, R., and Bax, A., J. Magn. Reson. **75**, 352 (1987)

DEPARTMENT OF HEALTH & HUMAN SERVICES

Public Health Service

National Institutes of Health
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March 17, 1988
(received 3/25/88)

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

NOESY Characterization of Hemin Derivatives

Dear Barry:

To model radical-mediated reactions which can alter the activity of heme proteins, we have developed a system containing reduced myoglobin and bromotrichloromethane. HPLC of the reaction mixture provided three products which retained the Soret band near 400 nm characteristic of iron porphyrins and whose FAB mass spectra contained MH^+ ions of m/z 661, 753 and 851 daltons. (The MH^+ of hemin, 4, is m/z 617). ^1H spectra of the purified modified hemins in pyridine solution after reduction by stannous chloride show the characteristic downfield peaks of the meso protons, the four methyl peaks, the multiplets of the propionic side chains, and peaks corresponding to a single vinyl group. Evidently a vinyl residue in each compound has been modified by the substitution of a carboxylic acid moiety, 1, the addition of two CCl_3 moieties, 2, and the addition of a CCl_3OH moiety, 3.

NOESY spectra serve very nicely to characterize these compounds. For example, the spectrum of the material of m/z 661, 1, shows that the meso proton at 9.94 ppm interacts with the methyl groups at 3.68 and 3.49 ppm and identifies it as the δ proton; the two methyls are evidently on rings I and IV, although not differentiated by this observation. The proton at 10.34 interacts with the propionic side chain, and is the γ proton. The interaction observed of the vinyl proton at 7.51 and the methyl at 3.69 demonstrates that these groups are on ring I.

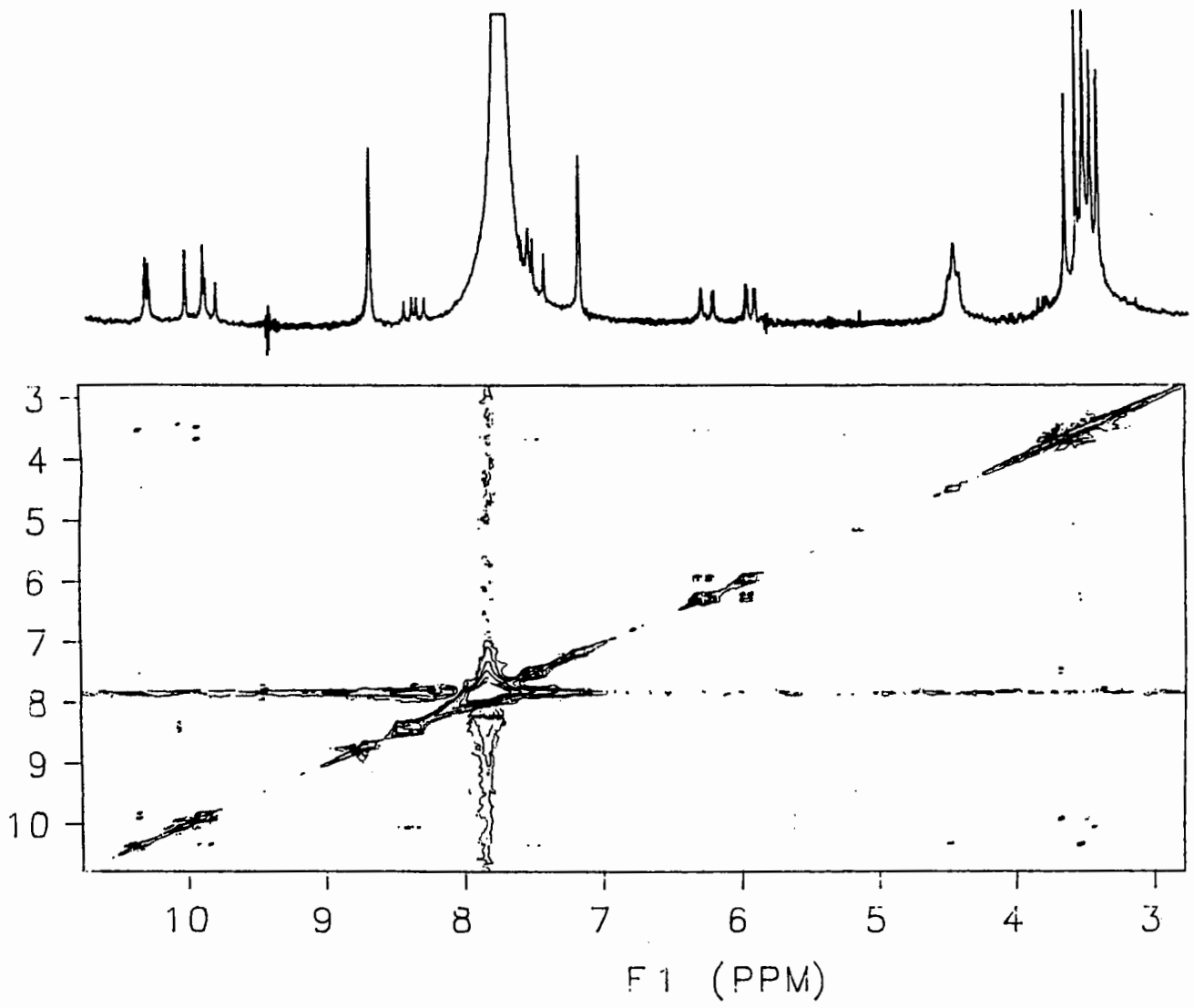
Similarly, spectra of the other two compounds show that it is the vinyl group on ring I which is altered in each case. This somewhat unexpected observation has prompted a good deal of speculation around here concerning the mechanism of formation of these products.

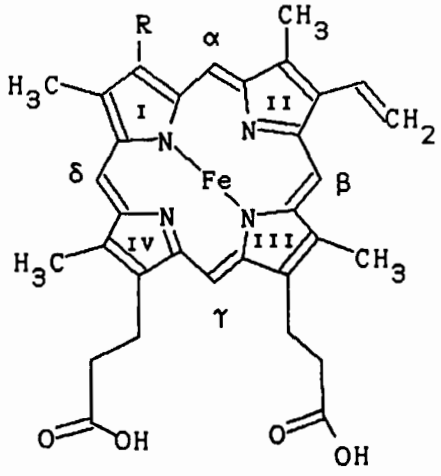
Cordially,

Bob Yoichi Osawa Lance Pohl

R.J. Highet
Laboratory of Chemistry

Yoichi Osawa Lance Pohl
Laboratory of Chemical Pharmacology





1. $CH{=}CHCO_2H$
2. $CH(CCl_3)CH_2CCl_3$
3. $CH(OH)CH_2CCl_3$
4. $CH{=}CH_2$

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MARBURG, DEN 30.3.88 (received 4/8/88)

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

SELINQUATE, a Frequency Selective Version of INADEQUATE

Dear Professor Shapiro,

Frequency selective pulses are currently the name of the game [1]. We designed a new pulse sequence [2], which makes 2D-INADEQUATE obsolete, by replacing the double quantum transfer pulse in the standard INADEQUATE technique [3] with a frequency selective pulse. The advantages of this new variant are: (i) To obtain a complete connectivity matrix only ^{13}C-NMR spectra corresponding to the number of different carbon atoms in a molecule have to be obtained. In practice, where often the assignment of only a few carbon atoms is uncertain, it is sufficient to obtain the connectivity of these atoms with our method in a few hours. (ii) In contrast to all 2D methods C,C spin coupling constants can be obtained in high digital resolution. (iii) Our method gives excellent results for long range spin coupling constants.

The pulse diagram is given below and spectra of n-propanol are shown. The experimental conditions were:
5 mm nmr tube with 20% solution of n-propanol in $CDCL_3$; Bruker AM-400 with Aspect 3000 and SEU (selective excitation unit) P3 Gauß-pulse, 20 ms, SW 12000, SI 64k, recycle time 5s, 512 scans. Trace b: P3 on resonance with C-1, only doublet at C-2 appears. Trace c: P3 on resonance with C-2, both doublets at C-1 and C-3 appear. Trace d: P3 on resonance with C-3, only doublet at C-2 appears.

Sincerely yours

[1] H. Kessler, H. Oschkinat, C. Griesinger, W. Bermel, J. Magn. Reson. 70, 106 (1986).
[2] S. Berger, submitted for publication.
[3] A. Bax, R. Freeman, S. P. Kempsell, J. Amer. Chem. Soc. 102, 4849 (1980).

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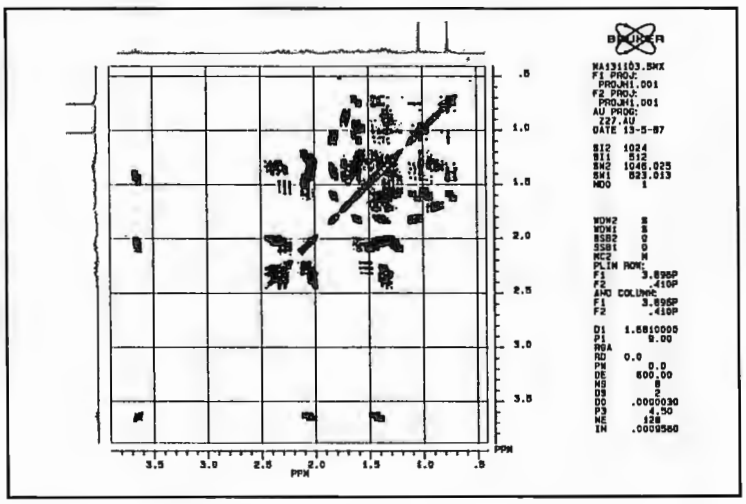

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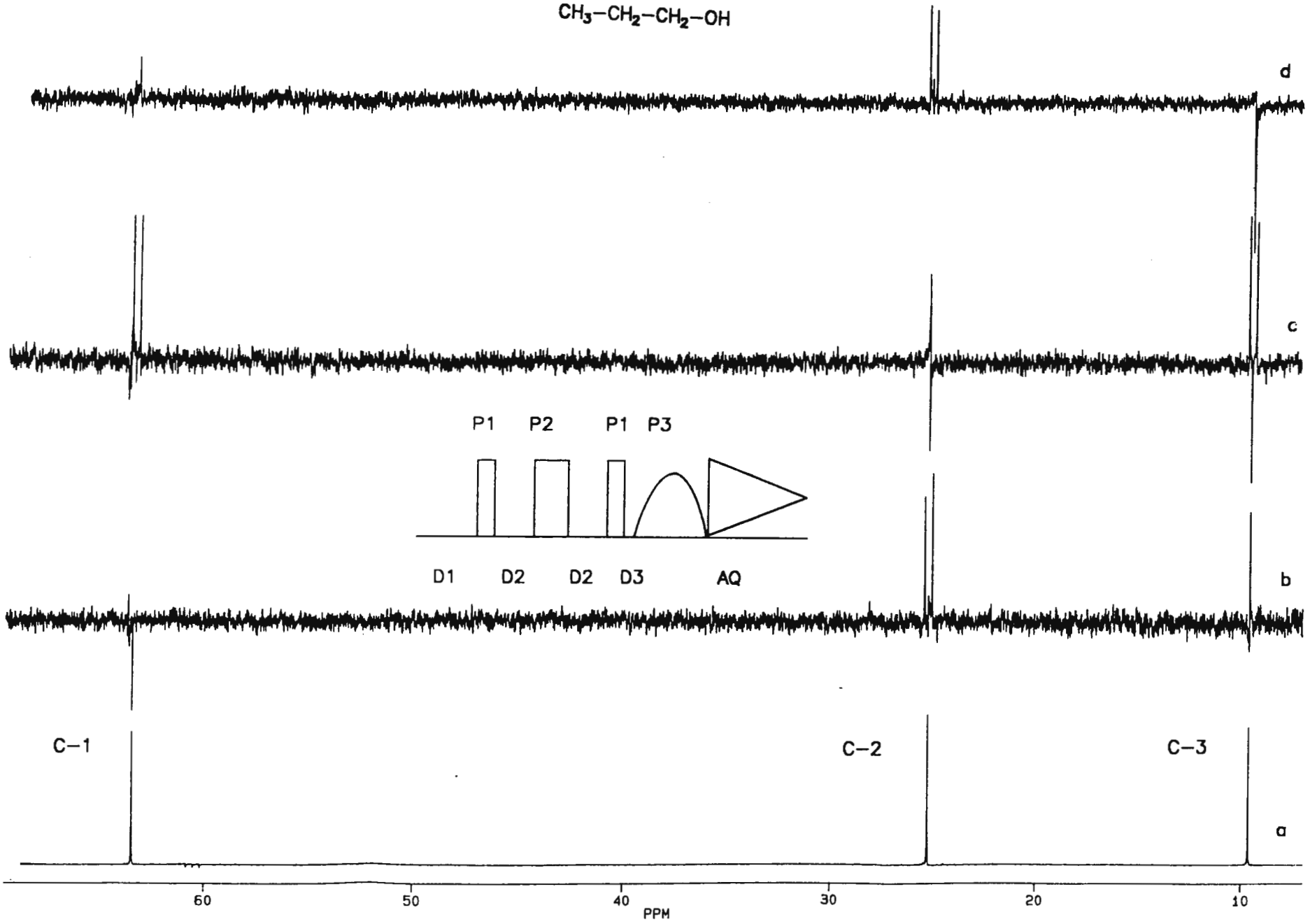
CH3–CH2–CH2–OH
d
c
P1
P2
P1
P3
D1
D2
D2
D3
AQ
b
C–1
C–2
C–3
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Prof. Dr. B. L. Shapiro
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto
California 94303
U.S.A.

uw kenmerk	uw brief van	ons kenmerk	datum	bijlage(n)
			March 30, 1988 (received 4/15/88)	

^{95}Mo quadrupole coupling in Mesitylene Molybdenum tricarbonyl.

Dear Barry,

NMR of liquid systems that are partially aligned by a static electric field can yield valuable information about anisotropic spin interactions. In case of polar molecules the alignment is a result of the coupling between the dipole moment and the electric field, which is chosen parallel to the magnetic field. Spectra of quadrupolar nuclei attached to such an oriented molecule will consist of 2I lines with an equal separation, given by:

$$\Delta\vartheta = \frac{3}{2I(2I-1)} \frac{eQ}{h} V_{zz} S_{zz} \qquad (1)$$

I is the spin of the nucleus, Q its quadrupole moment and V_{zz} the component of the field gradient tensor along the dipole moment. The averaged orientation is represented by S_{zz}:

$$S_{zz} = \langle \frac{3}{2}\cos^2\Theta - \frac{1}{2} \rangle_E = \frac{1}{15}\left[\frac{p_{eff} E_d}{kT}\right]^2 \qquad (2)$$

Θ is the angle between the magnetic field and the dipole moment, p_{eff} the effective dipole moment in solution and E_d the internal electric field [1]. For dipolar molecules, typical values of S_{zz} are 10^{-3} - 10^{-4}. An application of the method described is the determination of nuclear quadrupole coupling tensors. This will be demonstrated for ^{95}Mo (I = 5/2) in Mesitylene Molybdenum tricarbonyl (fig. 1). For this type of experiment a so–called " reference nucleus " is needed, with a known quadrupole coupling tensor and of course attached to the same molecule. A very suitable nucleus for this purpose is deuterium (I = 1). When the molecule is oriented in the electric field, the ^{95}Mo resonance will split into a quintet and the ^{2}H resonance into a doublet (fig. 2). The ratio of the observed splittings gives a direct relationship between the components of the quadrupole coupling tensors along the dipole moment for both nuclei:

$$\frac{\Delta\vartheta(^{95}\mathrm{Mo})}{\Delta\vartheta(^{2}\mathrm{H})} = \frac{1}{10} \frac{(eQ/h)\, V_{zz}(^{95}\mathrm{Mo})}{(eQ/h)\, V_{zz}(^{2}\mathrm{H})} \qquad (3)$$

It is obvious from the molecular symmetry that the principal axis system of the ^{95}Mo quadrupole coupling tensor coincides with the molecular axis system. Because the molecule has effective axial symmetry along the z–axis, the principal components $V_{x"x"}$ and $V_{y"y"}$ are equal.

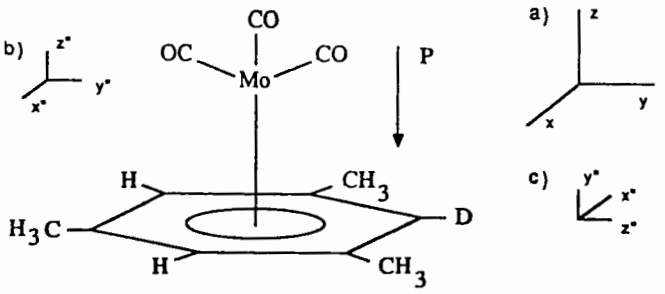


figure 1. Mesitylene-d_1 Molybdenum tricarbonyl,with a) molecular axis system, b) principal axis system of the ^{95}Mo field gradient tensor, c) principal axis system of the ^{2}H field gradient tensor.

The experiments were performed with a solution of mono-deutero-Mesitylene Molybdenum tricarbonyl in benzene (0.07 M). Using a quadrupole coupling constant of 186 ± 6 kHz (e^2qQ/h) and an asymmetry parameter η of 0.05 ± 0.02 for deuterium [2] (fig.1; eq = $V_{z''z''}$, $\eta = (V_{x''x''} - V_{y''y''})/V_{z''z''}$), the principal components of the ^{95}Mo quadrupole coupling tensor were calculated to be: (eQ/h) $V_{z''z''} = \pm (947 \pm 60)$ kHz, (eQ/h) $V_{x''x''}$ = (eQ/h) $V_{y''y''} = \mp (474 \pm 30)$ kHz.

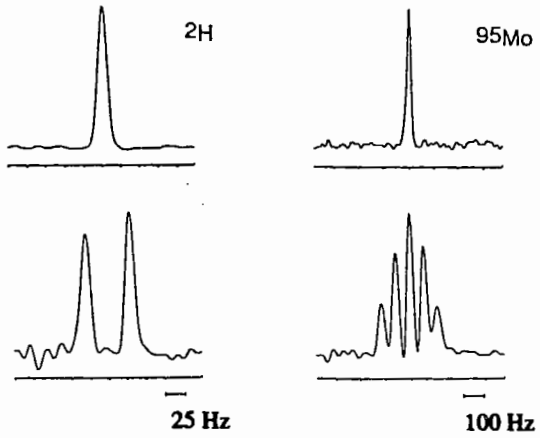


figure 2. NMR spectra of deuterium (left) and molybdenum-95 (right) without an electric field (top) and with an applied electric field of 1.0×10^7 V/m (bottom).

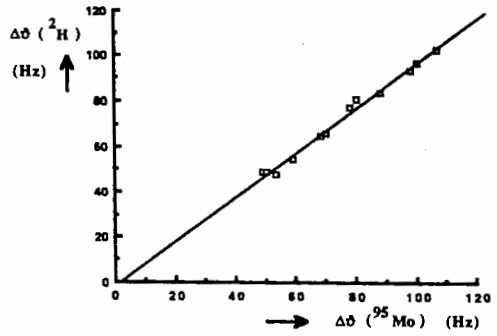


figure 3. ^{2}H line splittings vs. ^{95}Mo line splittings at several electric field strenghts.

Sincerely Yours,

L. Huis P. J. W. Pouwels C. MacLean

References.

[1] C. J. F. Böttcher and P. Bordewijk, *Theory of Electric Polarization* (Elsevier, Amsterdam, 1978).

[2] A. Loewenstein, *Advances in Nuclear Quadrupole Resonance* (HeydenLondon,1983), Vol. 5, p. 53; J. P. Jacobsen and E. J. Pedersen, J. Magn. Reson. **44**, 101(1981); J. P. Jacobsen and K. Schaumburg, J. Mol. Struct. **58**, 37 (1980), and references mentioned in these papers.

SIEMENS

April 13, 1988
(received 4/14/88)

Human in vivo ^{13}C at 4T

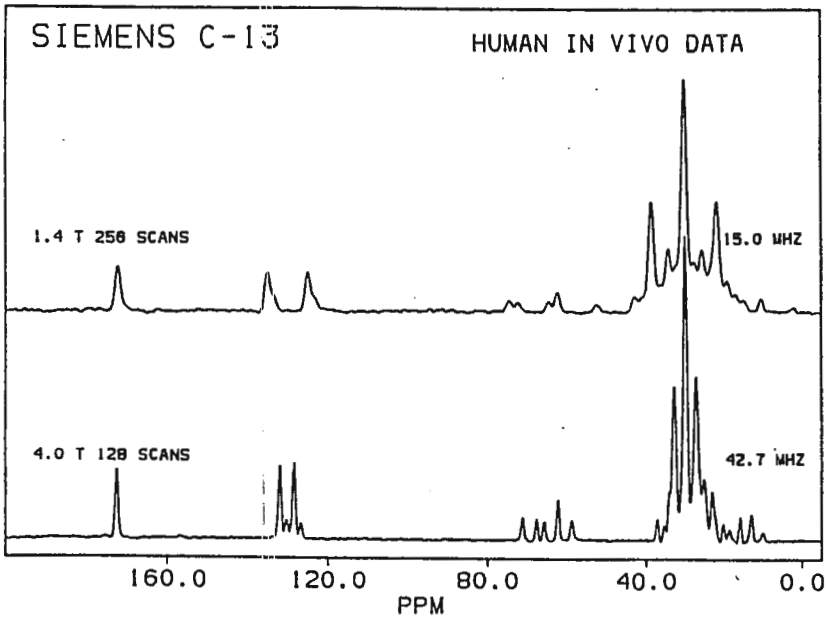
Dear Barry,

Since 4T whole body MR systems are now becoming commonplace, I thought that your readers would like to see some recent in vivo carbon data at 42 MHz. Our research high field system is built around a 4T 1.25 meter warm bore SCM designed and constructed at the Siemens Research Laboratories in Erlangen (FRG). The magnet is a 6 coil design with 2 SC shim coils and uses only helium cryocooling. A Linde-designed reliquifier maintains the 550 liter helium bath without the need for refilling. The 10.6 ton 2.8m long (2.2m OD) SCM stores 39 MJ at 4T with a current of 376A. Without RT shims the corrected homogeneity is $< \pm 2.5$ ppm over a 50 cm dsv. Shimmed NMR linewidths of better than 0.05 ppm can be achieved with 500 cc samples. Field drift is less than 3.6×10^{-8}/h. This magnet has been at field since January 87 (including the closed loop He system).

Human in vivo ^{1}H (170Mhz), ^{31}P, and ^{19}F spectroscopic studies have been performed on this system, as well as ^{1}H and ^{23}Na head imaging. Since ^{1}H decoupling for ^{13}C human in vivo spectroscopy presents some difficulty, particularly at higher fields, the ^{13}C experiments were thought to be of less utility than other nuclei. However, as one can see by the improvement in the ^{13}C resolution at 4T (see Figure) over similiar data taken at 1.4T (15Mhz), the need for ^{1}H decoupling in order to assign resonances is greatly reduced at 42 Mhz. The example is mainly lipid resonances from the calf. No localization technique except surface coil placement was used. The increase in S/N at 4T coupled with the excellent homogeneity of this whole body magnet means that techniques such as CSI can be used to localize ^{13}C spectra in reasonably small voxels for various human organs.

Sincerely,

Michael J. Albright
Dietmar Hentschel
Jurgen Vetter
Ralf Ladebeck



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		0.25g	350.
		0.5g	650.
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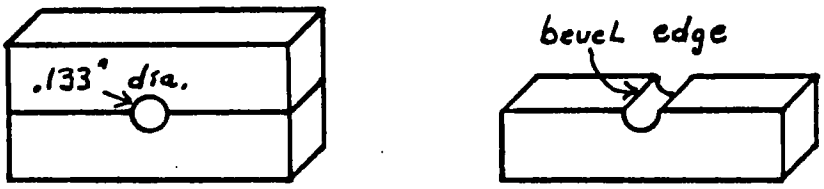
April 14, 1988
(received 4/18/88)

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

Dear Barry:

While modifying our Bruker AM-250 to enable operation in the reverse correlation mode, it became clear the main problem would be fabricating the RF cables. The cables are RG-188/U coaxial cable terminated with Burndy subminiture coaxial connectors. The cable and connectors are relatively inexpensive but the required crimping tool and the proper dies are well in excess of $1,000. All attempts to find a loaner tool or someone with a tool who could install the connectors failed. We were able, after some thought, to design a make-shift crimping tool which results in a fairly reasonable crimp.

The tool is easily made by clamping two pieces of 1/2" X 1/2" X 1" brass together and drilling a .113" hole centered at their mating surfaces with a # 33 drill as indicated below.



Then remove the two brass pieces from the vise and slighty bevel the inner edges of each piece along the hole with a file. This lessens the chance of the brass tool damaging the connector. To use the tool simply strip the cable and solder on the inner conductor then assemble the connector on the cable and place the two halves of the tool over the connector and compress in a vise to complete the crimp.

The connectors used:

Pin	RCDXK-1D28	Burndy Corp. Norwalk CT 06856
Socket	RMDXK-10D28	Burndy Corp. Norwalk CT 06856

Sincerely yours,

Dennis Warrenfeltz

Herman van Halbeek

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April 1988
(received 4/9/88)

Dr B Shapiro
966 Elsinore Court
Palo Alto
CA 94303
USA

Dear Barry

SATURATION TRANSFER WITH BROAD-LINE SPECTRA

Population-transfer methods, using the nmr of protons and other spin-½ nuclei, are of immense value for the study of kinetic processes. They could be equally useful with quadrupolar nuclei, such as ^{17}O. However, the relaxation times of these are such as to preclude DANTE-type saturation or 2D NOESY experiments. Indeed, one problem with such 2D experiments is that the available $\pi/2$ pulses are much too long to stimulate the entire spectrum.

I have found that this last problem can be inverted to yield an advantage. With a typical 10 mm aqueous ionic ^{17}O sample on our Bruker WH400, the π pulse is only effective over about ± 100 ppm. Now the ^{17}O nmr spectra of many polyanions covers a range of 1300 ppm, and most of the resonances fall into fairly well-separated groups. An example might be terminal Mo-O (700-900 δ), bridging Mo-O (300-550 ppm), and internal O, within 120 ppm of H_2O at δ = 0. Because of this convenient separation, one merely has to perform a π–τ–small flip angle experiment, where τ is either long, to create a standard spectrum, or else typically 3 ms, to allow some exchange to take place. The area to be inverted must be set at the centre of the spectrum. The experiment isn't quite quantitative, of course, but with suitable subtraction, perhaps using an unaffected peak as a reference, one can either remove the peaks not involved in exchange, or, alternatively, retain only these.

In the top example I have taken a relatively complicated sample containing a mixture of vanadomolybdates and isopolymolybdates, with modest ^{17}O enrichment. The spectrum from this is the lower trace. I had begun to suspect that the vanadomolybdates were kinetically inert whereas the isopolymolybdate oxygens were in exchange with solvent water. I therefore placed the water resonance at O1 for selective inversion. Sure enough, the spectrum that remains after subtraction of unaffected resonances (upper trace) is solely that of the two isopolymolydbates present at this pH, namely $[Mo_8O_{26}]^{4-}$ (the narrower lines), and a higher-Mo species spectrum of the non-exchanging species can be similarly obtained.

A counter-example is given in Figure 2. The species is $[Mo_7O_{24}]^{6-}$, where I had noticed two broader resonances, one from four terminal oxygens and one from four bridging oxygens. A dynamic process was indicated by the way these broadened further upon increasing the temperature. But were the oxygens exchanging with each other or with solvent water? I found that inverting the water peak had no effect on the rest of the spectrum, but that on inverting the bridging oxygens around 350 δ, the inversion was partly transferred to the broad component of the terminal Mo-O region, at ca 750 δ. This subtraction spectrum (blank minus inverted) is shown in the lower trace, and the conclusions are supported by the reverse experiment (upper trace).

The method clearly works well for ^{17}O nmr. In theory the inversion region could be made more precise by using, eg, a 3π pulse. However, in practice I have found that one cannot work successfully when the inverted and the observed peaks are separated by less than about 5 KHz. Perhaps even this limitation could be removed by shaped pulses, but, alas, I do not have the hardware for these.

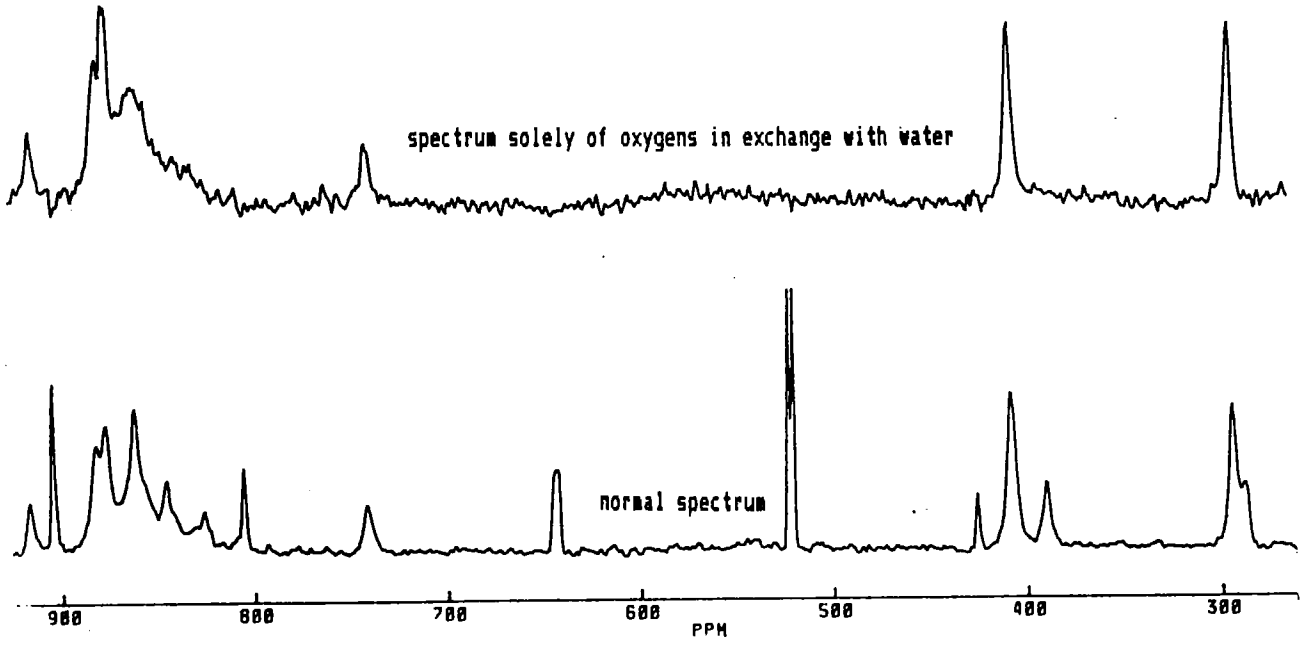
Yours sincerely

Oliver Howarth

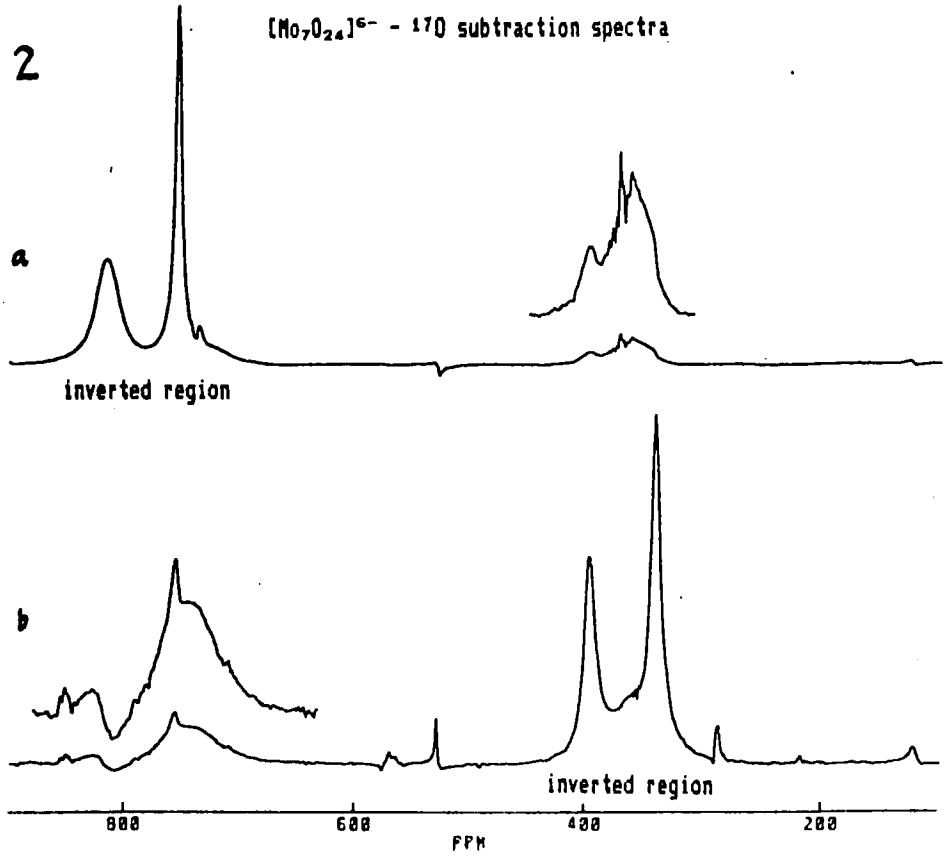
Oliver Howarth

1

VM09 SAMPLE PH 2.2 323K 17O WITH SUBTRACTION SPECTRUM



2



356-32

DEPARTMENT OF HEALTH & HUMAN SERVICES

Public Health Service
National Institutes of Health

National Institute of Diabetes and
Digestive and Kidney Diseases
Bethesda, Maryland 20892

Professor Bernard Shapiro
966 Elsinore Court
Palo Alto. Cal. 94303

15. April 1988
(received 4/19/88)

NT - Owners Beware:

Dear Barry

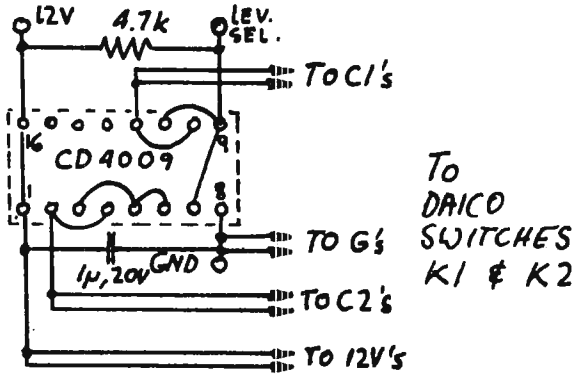
The NT series spectrometer spin decouplers have 2 possible power settings that can be selected by the L1 - L2 commands. The selection is executed by fast (10µs) pin diode switches, Daico model 100 C 1292 A. There is a potential problem in that circuit.

We experienced an extra few db of loss in one branch plus a distorted pulse shape, and traced it to one switch, so I intended to replace the switch. From the usual commercial sources these switches turned out to be very expensive. I therefore took a second look at the circuit and found that the switch was fine, but was not being driven properly. The switch driver, Dec Fast Relay Switcher, (drawing #107154B or 150430B) will only drive one side of the switches fully on, not the other.

The solution is to replace the whole switch driver circuit with one CD 4009 chip ($ 0.35). It is mounted on a piece of holy board that replaces the pc-board sandwiched between the switches. The schematic/layout appears below.

Sincerely
Rolf Tschudin

Rolf



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DEPARTMENT OF CHEMISTRY

March 29, 1988
(received 4/2/88)

"Selected Detection of Histidines in Proteins"

Dr. Bernard L. Shapiro
Editor/Publisher
TAMUNMR Newsletter
966 Elsinore Court
Palo Alto, CA 94303

Dear Barry:

Several years ago, J. Am. Chem. Soc. **106**, 4299 (1984), we showed that the histidine protons of RNase could be selected for by means of filtering the spectrum. When the pulse sequence 90°-t-180°-t is used and the results co-added for a proper group of t values the signals from the phenylaline, tyrosine and tryptophan multiplets disappear leaving only the signals from the C3H and C4H protons of histidine. We have recently been investigating staphylococcal nuclease and when we attempted the above procedure on SNase we kept missing a signal. SNase has four histidines and hence we expected eight signals total with four from C4H protons and four from C2H protons. For this and other reasons to be discussed elsewhere SNase was prepared with all of the ring protons of phenylalanine, tyrosine and tryptophan replaced with deuterium. These samples allow a very clean observation of only the histidine protons as indicated by the results in the figure. The top spectrum is of SNase is the absence of ligands and the bottom spectrum is for SNase in the presence of Ca^{2+} and pdTp with both samples at pD 7.8. All of the amide protons have been exchanged out. The spectra show that one of the histidines (H121) has a split peak even at high pH and in the presence of ligands. It seems that Fox and Dobson missed this effect in their previous studies of SNase since only the C2H protons are well resolved in the spectrum of fully protiated SNase and hence they could not tell what was happening with the C4H protons.

We have examined these samples at a variety of pH values and temperatures. The evidence is pretty clear that SNase exhibits multiple conformations over a very wide range of conditions. It is quite likely that histidine is particularly sensitive to conformational variability due to its pKa value and that multiple conformations would be detected for other proteins if they were examined in this fashion.

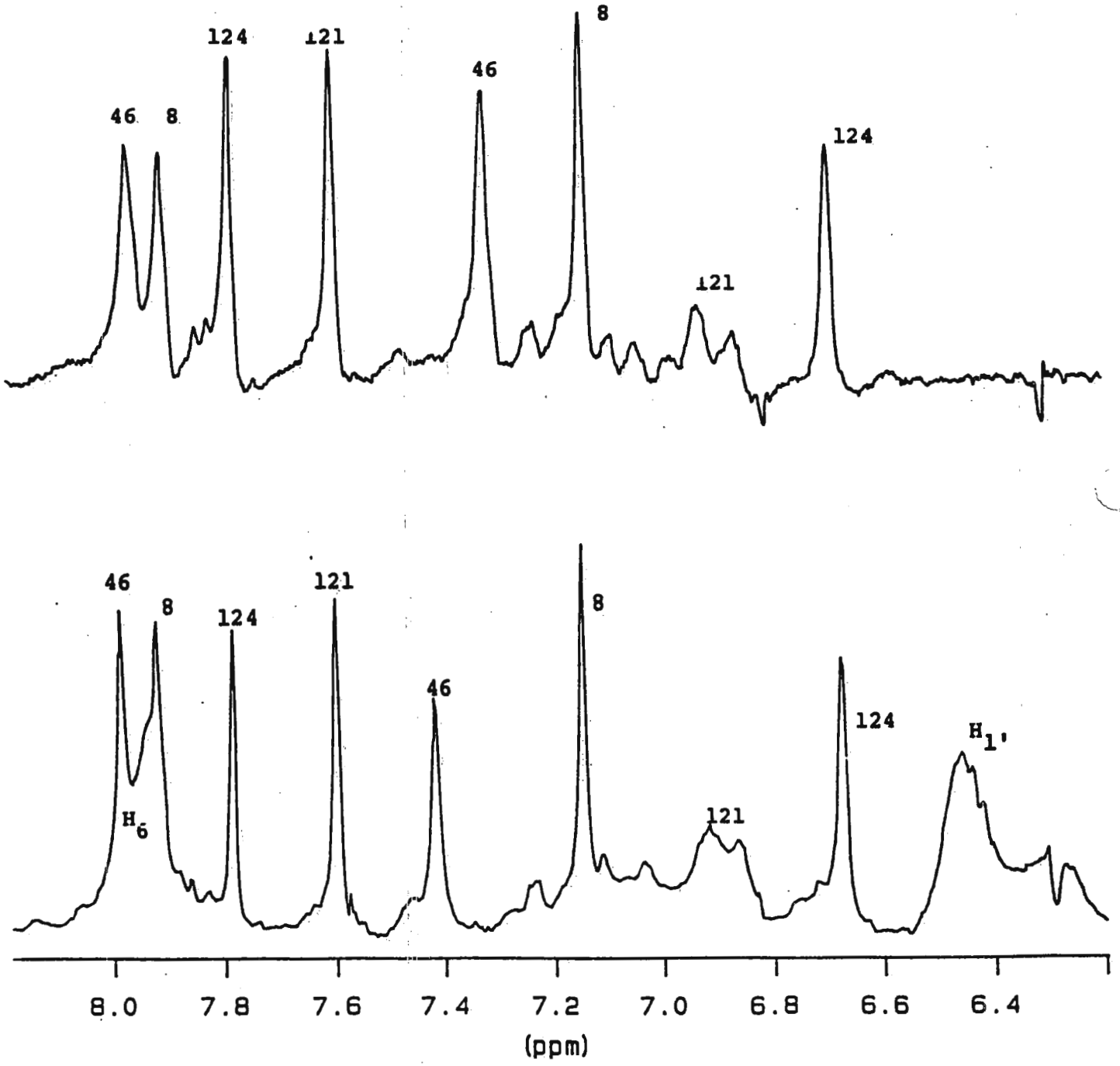
Sincerely,

Philip H. Bolton
Professor of Chemistry

PHB/jbh

SNase, His only

Figure 1.



National Institutes of Health
National Cancer Institute
Bethesda, Maryland 20892

Biotechnology Fellowship Available

A Biotechnology Fellowship is available in my laboratory in the National Cancer Institute for 2D NMR studies and conformational analysis of oligodeoxynucleotides and analogs (see, Biopolymers *26* 2041, 1987; PNAS *84* 7706, 1987). This is restricted to US citizens and is a new NIH training program to extend the application of biotechnology to cancer and AIDS research. Postdoctoral experience in the subject indicated will be given priority. We also have an Evans & Sutherland Molecular Display and a micro-VAX-II, and experience in handling these and the relevant programs will also be considered.

Dr. Jack S. Cohen, Biophysical Pharmacology Section
Bldg. 10/ room 6N105, NCI/NIH, Bethesda, MD 20892

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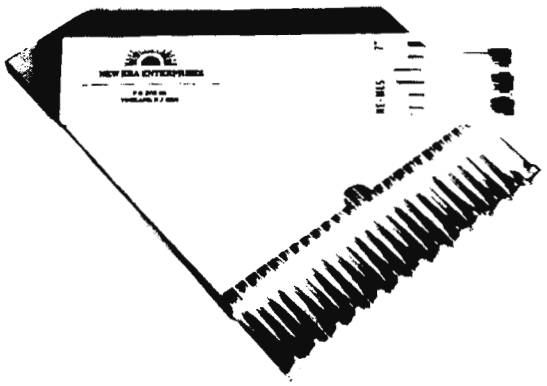

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April 14, 1988
(received 4/18/88)

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

Subject: Measuring The Q (Quality Factor) of NMR Probes.

Dear Professor Shapiro:

The continuing widespread interest in in-vivo NMR spectrosopy has led to the necessity of numerous laboratories to engage in probe design. In reviewing the reported performance of these "home built" probes we have observed literature Q, ("quality factor"), values ranging from 50 to 300. Clearly this wide range of values cannot be compared in any meaningful way. This is in part due to the fact that there are many ways to measure probe circuit Q.

A frequently used method suggested by J.Murphy-Boesch (1), based on reflected power, requires an oscilloscope, a sweeper, a directional coupler and a lot of eyeballing. Q is defined as the resonant frequency divided by the bandwidth measured at the half power points. (Since the circuit is matched to 50-ohms, the Q measured should be multiplied by two to obtain the free ringing Q of the coil.) It is difficult to accurately count graticules and the losses and reflections from the cables, connectors and the directional coupler are difficult to calibrate out. The measurements have to be estimated from a distorted picture. Data achieved in this manner, although, may be useful for day to day comparisons in one lab, is not necessarily accurate and cannot be compared from lab to lab.

We now report a more precise measurement of Q which can be done with a network analyzer, however, care must be taken to get repeatable, reliable data. In our lab, we are using a Hewlett Packard 8753A network analyzer and we would like to suggest a reliable method for measuring Q. Since the 8753A cannot measure Q directly on a one port device, (a problem you will quickly run into if you try following the manual procedure for Q measurements!), it is necessary to convert to Z parameters and measure the bandwidth at the 45 degree phase shift at the resonant frequency. It is also important to note that even when using a network analyzer, the instrument calibration is critical and the measured Q will vary with different calibration procedures. The following procedure was worked through with the help of HP engineers and P.Peppin at Yale. It seems to produce the most accurate reproducible results.

1. Choose resonant frequency as center and span about 20 MHz.
2. Do a full S11 one port calibration using HP type N50-ohm calibration kit. (Part#85032B-calibration without the HP standards is a source of error and can produce data which is not repeatable.)
3. The data should then be normalized by connection of a BNC short and displaying its phase response. Store the displayed data in memory and display data divided by memory.
4. Connect probe and tune and match using smith chart display.
5. Turn on Z parameter conversion and display the phase of Z:Refl.
6. Set marker on min/max and zero it (this should be your resonant frequency). Set width value at +/- 45 degrees and put widths on. The instrument calculates Q and displays it!

(1) J.Murphy-Boesch,in "Biomedical Magnetic Resonance"(t.l. James and Margulis,Eds) p.47, Radiology Research and Education Foundation, San Francisco,1984.

Measurement of Q on a surface coil probe gave different numbers depending on the method we used. Using reflected power the Q measured somewhere between 45-55 depending on cable lengths, connectors etc. The same probe measured on the network analyzer gave results ranging from 55-72 when a different calibration procedure used. Using the method described above we were able to get consistent Q values (within 5% error) on several probes. More accurate data may be achievable if a standard BNC calibration kit could be established.

The method reported here is to date the best we have found for obtaining reliable Q values. We recommend this procedure as a standard to those engaged in probe design so that meaningful comparisons can be made with Q values reported in different laboratories.

Sincerely,

Nina C. Gonnella

Dr. Nina C. Gonnella

Robin F. Silverman

Ms. Robin F. Silverman

Positions Available: Massachusetts General Hospital/Harvard Medical School

1) Manager, High Field Spectroscopic NMR Facility

We are seeking a person with a Ph.D. in Chemistry, Physics, Biology, or a related discipline, to supervise the operation of a new Bruker MSL 400 spectrometer to be used for chemical and *in-vivo* spectroscopy, solid-state spectroscopy, and imaging of biological and nonbiological specimens. The successful candidate should possess several years experience in the operation and maintenance of high field spectrometers, knowledge of state-of-the-art NMR techniques, good organizational and communications skills, as well as the ability to interact with a number of investigators sharing a wide variety of interests and to supervise a technician. We will also strongly encourage the Manager to develop an independent research program in addition to collaborating with other investigators.

2) Instrumentation Specialist

This individual must possess a Bachelor's or higher degree in engineering or physical science, as well as experience with instrumentation of the type found in NMR equipment. Responsibilities include overseeing maintenance of NMR instrumentation, upgrading these instruments, developing new instrumentation, supervision of a technician. Familiarily with analog, digital and RF electronics is essential; experience with computers a strong plus.

3) Postdoctoral Fellow: Solid-State Imaging of Biological and Nonbiological Materials

We are seeking a person to participate in our program in multinuclear imaging applications in polymers, advanced ceramic materials and bone. This individual will help in setting up high field imaging on a 6 T spectrometer, and will conduct imaging experiments on a number of instruments, including the MSL 400. The applicant should have a Ph.D. in Chemistry, Physics, or a related field, and must be comfortable with hardware development.

The full MGH NMR Facility comprises two clinical and one high field small bore NMR imagers, and two high field spectrometers. One 1.5 T clinical imager and one 4.7 T imager/spectrometer are now on order. About forty-five researchers are associated with the Facility on either a part-time or full-time basis. Applicants for any of the above positions should direct a C.V. and arrange for two letters of recommendation to be sent to Jerome L. Ackerman, Director of Spectroscopic NMR, Department of Radiology, NMR Facility, Baker-2, Massachusetts General Hospital, Boston, MA 02114 (phone 617-726-3083). The MGH is an equal opportunity/affirmative action employer, and especially encourages women and minorities to apply.

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Please reply to

Massachusetts General Hospital
Boston, Massachusetts 02114

NMR LABORATORY
(617) 726-3081

March 14, 1988 (received 4/2/88)

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

Imaging of Solids

Dear Barry:

In addition to working on things medical, we in the NMR Facility at the Massachusetts General Hospital are developing methods for the NMR imaging of solids. Aside from the very great potential that NMR imaging holds for contributing to the study of materials in general, we can justify such a research program in a hospital context on the basis of novel medically relevant applications (such as the imaging of the mineral phase of bone) and through "technology spinoffs" (for example, the development of very high performance field gradient hardware) of general interest to other members of the group.

In the area of the development of advanced ceramic materials, we have been collaborating with Bill Ellingson of Argonne National Laboratory and John Weyand of Alcoa to explore the use of NMR for the production of images showing the distribution of residual porosity in green (unfired) and partially sintered ceramic components, and the distribution of polymeric binder in green components. We map open porosity (that internal void space connected with the exterior of the specimen) by vacuum impregnating the test piece with a suitably chosen fluid (usually benzene doped with $Cr(acac)_3$); we define the NMR-derived open porosity in a region as the fraction of maximum signal intensity normalized to that of pure fluid. Figure 1. shows a point-density plot of an image of a green disk containing some point nonuniformities in the porosity, implying the presence of defects which could ultimately lead to failure of the finished component under service conditions. Image processing and analysis techniques may be effectively applied to such data to develop measures of quality of the specimen; one example is the spatial porosity distribution function derived from the intensity histogram of the image.

The binders in these green ceramics often have T_2's in the range of 1 msec, exceedingly short in the context of conventional hardware. Using a probe with high intensity, rapid switching gradients (built for us by Doty Scientific, Inc.), we have been able to image the polymeric binders with echo times TE between 600 μsec and 4.0 msec. We employ either short-TE versions of conventional (or projection reconstructed) two-dimensional

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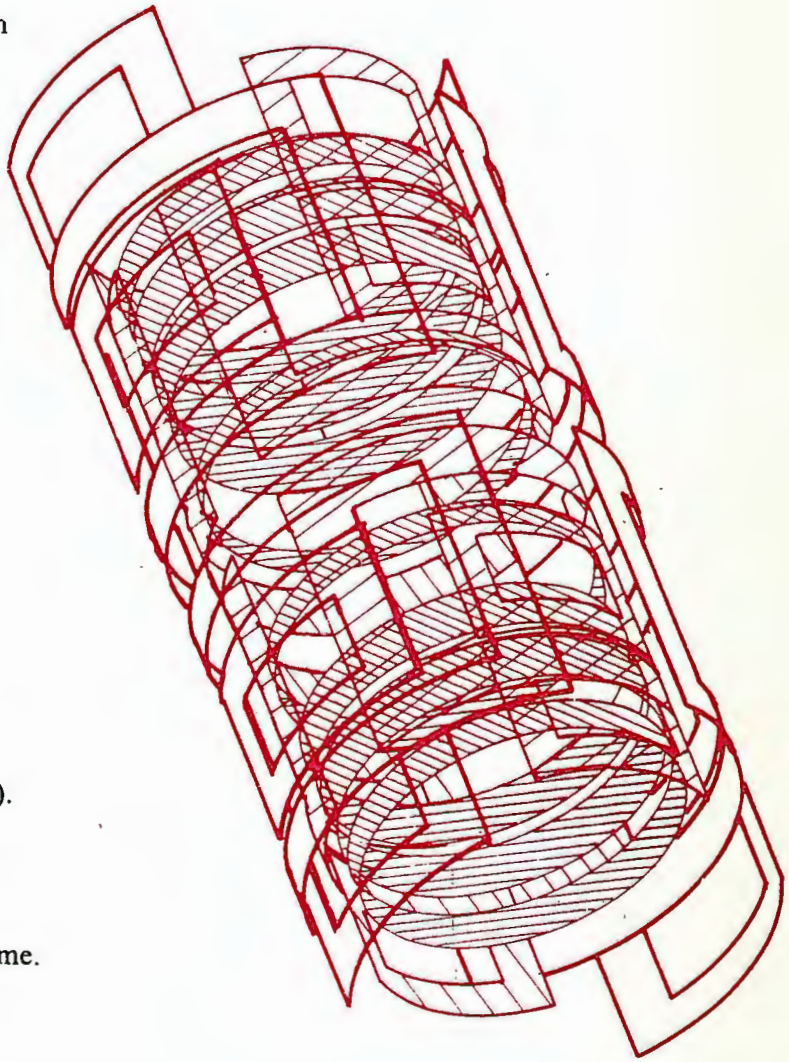

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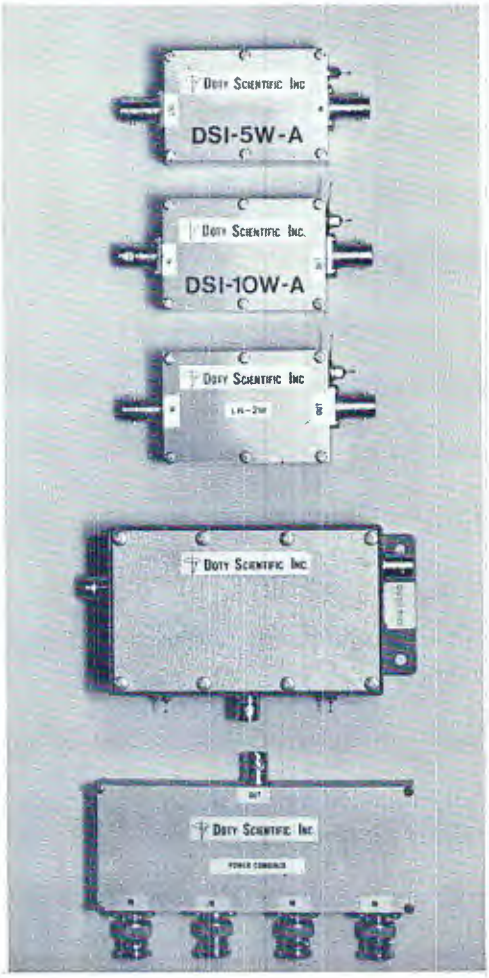

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spin-echo pulse sequences (without slice selection), or three-dimensional sequences (one spectral and two spatial dimensions) using only phase-encoding of spatial information. The last technique allows a clean separation of the intrinsic spectral lineshape (which degrades spatial resolution) from the spatial information in the data set; a disadvantage is that significantly more time may be required for the 3D acquisition even when a much smaller number of image pixels is acceptable. Figure 2. shows a cross-sectional image of the the polymer in a green test piece which is 15.5 weight percent binder.

These methods are applicable to a wide variety of materials with intermediate T_2. Examples are collagen and other "short T_2" proteins or cellulosic materials, many elastomers, the softer phases of composites, adsorbed or interstitial low molecular weight compounds, and the like.

Figure 1. Proton fluid-state NMR image of doped benzene in a green alumina compact; slice thickness about 2 mm; specimen diameter 25 mm; echo time 15 msec; imaging time about 7 min. The lighter regions (corresponding to lower signal intensity) have lower porosity than the rest of the specimen.

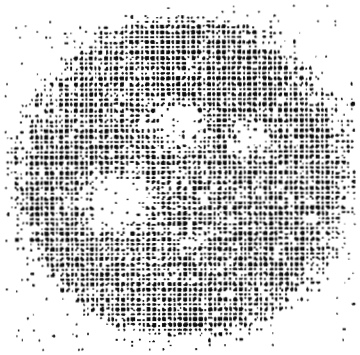

Figure 2. Proton solid-state NMR image of polymer in a test piece containing 15.5 weight percent binder. No slice selection was used. Echo time 2 msec; imaging time 7 min.

Sincerely,

Jerry Ackerman

Jerome L. Ackerman

Leo Garrido

Leoncio Garrido

THE CLEVELAND CLINIC FOUNDATION
9500 Euclid Avenue Cleveland, Ohio 44106

Thian Ng, Ph.D.
Head, MR Research and Development
Division of Radiology
216/444-8209, 216/444-8200

March 31, 1988
(received 4/16/88)

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

A chemical shift selective sequence for fat and water images/positions

Dear Dr. Shapiro:

As a part of the requirements to start our subscription of TAMU letters, we would like to briefly report the technical implementation of a chemical shift imaging sequence in our laboratory.

The pulse sequence is scehmatically presented in Fig. 1. It initiates with a train of spatially offset saturation pulses, to remove signal from flowing blood, and followed by a chemical shift selective $1\text{-}\bar{3}\text{-}3\text{-}\bar{1}$ composite pulse for either water or fat suppression. A slice selective 180° pulse is then applied at a selected delay TE and the spin echo is acquired. This sequence was used in lieu of a Dixon type sequence (in phase, out of phase) because it is capable of true fat and water discrimination with no ambiguity and with minimal preparation.

MRI was performed on a 1.4 Tesla Technicare whole-body imager and provided excellent water and fat only images as shown in Fig. 2. We have used this technique to study the plaque lesion of hypercholesterolemic dogs and rabbits. The image quality is excellent.

In addition, we are expanding our research effort in tumors and hypertension on humans and rodents, using both MRS and MRI in situ.

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edited by Joseph B. Lambert and Roberto Rittner

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A few Project Scientist positions requiring expertise in technical or biochemical aspects of MRS and MRI are currently available. Scientists who are interested, please send resume to Dr. Thian Ng or call 216-444-8209 for more information.

Sincerely,

Anthony Majors, BSc.

Thian C. Ng, Ph.D.

TCN:AM:lj

Flow Suppressed 1-$\bar{3}$-3-$\bar{1}$ Sequence

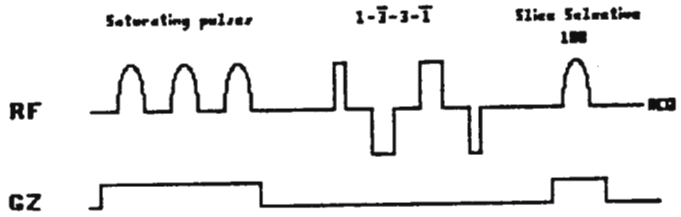

Fig. 1. Schematic diagram of the selective CSI sequence.

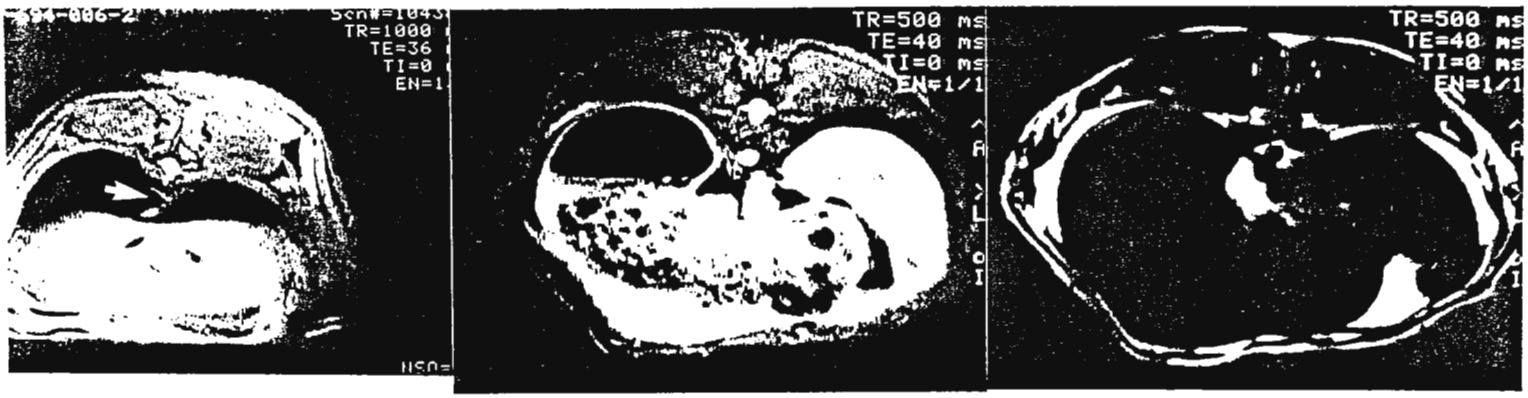

Fig. 2. Illustration of chemical shift MRI. (b) and (c) are the water and fat only images respectively taken from the same slide through the aorta of a hypercholesterolemic rabbit. (a) a water image with flow saturation pulse.

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April 14, 1988
(received 4/15/88)

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

C_α-H Proton Shift in Polypeptide Random Coil → Helix Transition

Dear Barry:

For my contribution I would like to recall an attempt I once made to repeat a published calculation[1] of this shift based on a magnetic anisotropy model from microwave data of formamide (Tigelaar & Flygare, or "T-F" model). For my calculation, the same molecular Zeeman parameters, α-helix conformation, and stereochemically allowed conformations of the random coil were used. However, the result was the prediction of a 0.20 ppm upfield shift for the random coil → α-helix conversion. This result is at variance with that calculated in Reference 1 (0.10 ppm downfield), but agrees with published experimental data.[2] A similar calculation based on the Zurcher anisotropy model for carbonyls[3] gave 0.19 ppm upfield shift for the coil → helix transition, while another calculation for an electric field model[4] showed comparatively little sensitivity to conformation (e.g., 0.02 ppm downfield for the transition). In the figure are my calculated isoshielding contours for L-amino acid $C_\alpha H$ shifts in ppm (downfield corresponds to negative-going values), following the IUPAC-IUB convention for conformation,[5] based on a right-handed α-helix reference (0 ppm) and the T-F model.

I am aware that the T-F model was unable to account for observed shifts in cyclosarcosyls,[6] and would be interested to hear of similar attempts to use it.

Sincerely,

D. M. Wilson

References

1. H. L. Tigelaar and W. H. Flygare, J. Amer. Chem. Soc. 94, 343 (1972).

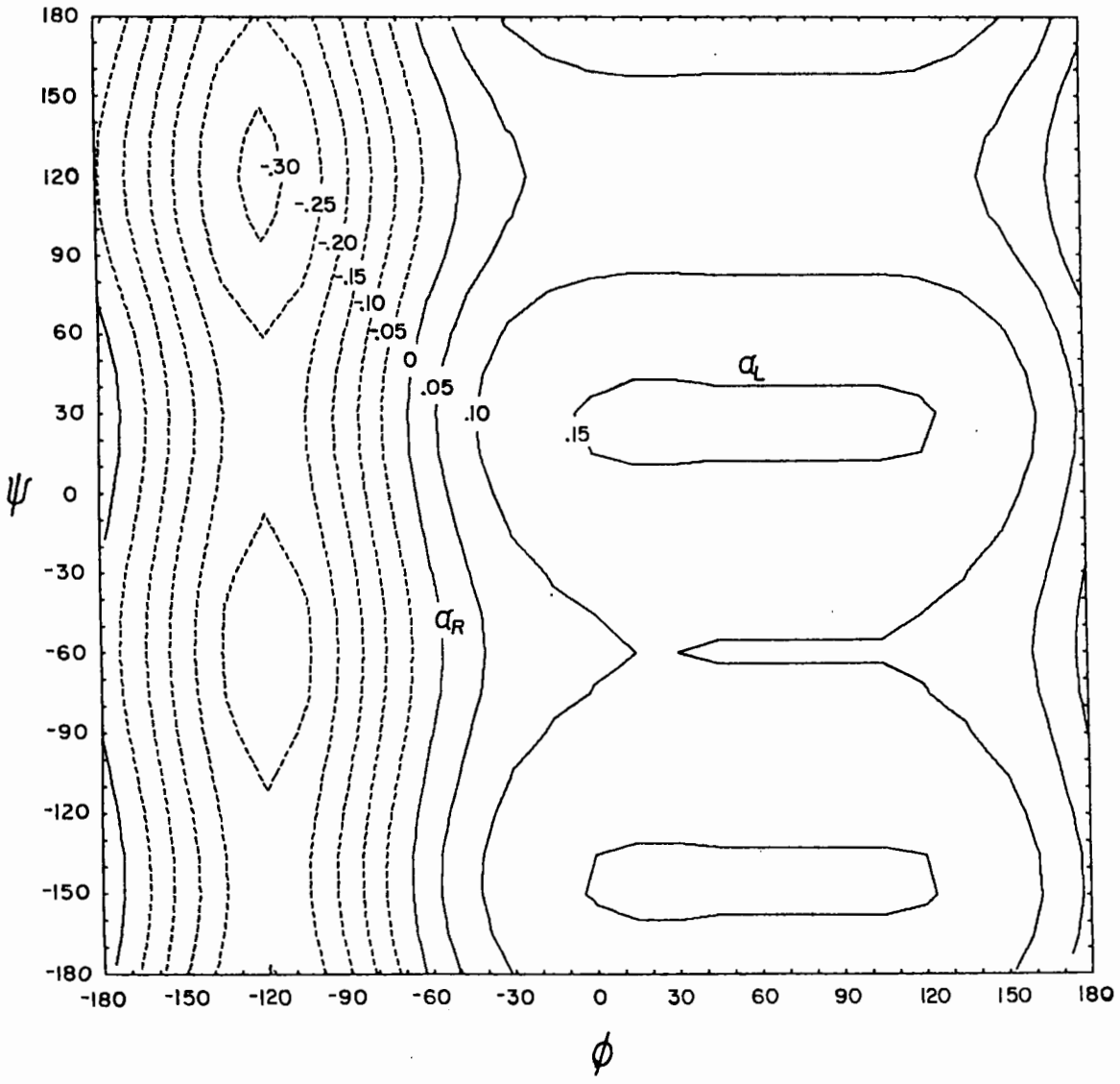
2. See References 1-6 in Reference 1; also M. Goodman et al., Proc. Nat. Acad. Sci. U.S.A. 70, 331 (1973).

3. R. F. Zurcher, Progress in NMR Spectroscopy, Vol. 2, J. W. Emsley et al., Eds., Pergamon Press, Oxford (1967), pp 205-257.

4. The electric field model was A. D. Buckingham's, Can. J. Chem. 38, 300 (1960); using a monopole distribution of Poland and Scheraga in Biochemistry 6, 3791 (1967); and dielectric constant $\varepsilon = 4$.

5. Biochemistry 9, 3471 (1970); this is different from the convention used in Reference 1.

6. N. J. Clayden and R. J. P. Williams, J. Magn. Res. 49, 383 (1982).



UNIVERSITY of PENNSYLVANIA

School of Medicine
Department of
Biochemistry and Biophysics
Philadelphia, PA 19104-6089

April 8, 1988
(received 4/15/88)

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

Coaxial Quadrature Hybrid

Dear Professor Shapiro:

I would like to communicate a simple, yet very useful circuit to be used with circular polarizing coils, which we use for head and body imaging.

The method of choice driving such a coil is a 3 dB directional coupler with 90^0 phase shift between its outputs. However, since the circuit should be mounted as near as possible to probe and preamp, i. e. on the magnet, no ferrites can be employed. For small-band needs coaxial circuits can be used instead. One of them has been described in (1)

We derived our design from the microwave branch-line coupler (2). It is probably the simplest design sufficient to do the job (see figure on next page). Four $\lambda/4$ lines implement all desired functions: (a) 3 dB power splitting, (b) $+90^0$ phase shift in transmit mode and (c) -90^0 shift in receive mode.

The coil is represented by its orthogonal ports X and Y, which both work as inputs and outputs. With a characteristic impedance of Zo for X, Y, transmitter (XMIT) and preamplifier (RCV), the coax-hybrid [1,2,3,4] consist of two $\lambda/4$ "thru"-lines of impedance Zo/sqrt(2) plus two $\lambda/4$ branch lines of impedance Zo.

We used for the branch lines RG-58U cable (ca. 52 ohms) and for the "thru"-lines pairs of RG-59U (75 ohms each) shunted in parallel. Based on a propagation speed of $v/c_0=0.65$ a first approximation for the cable lengths is: $l\,[cm] = \lambda/4 = 4875 / f\,[MHz]$.

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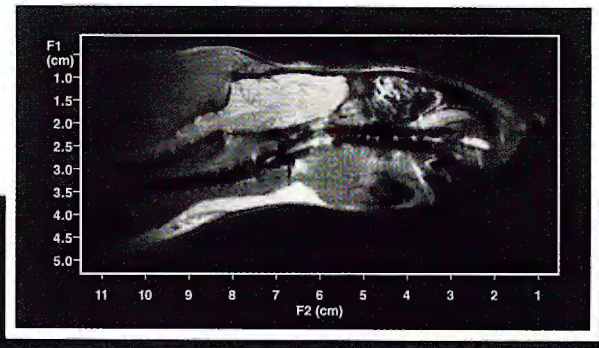

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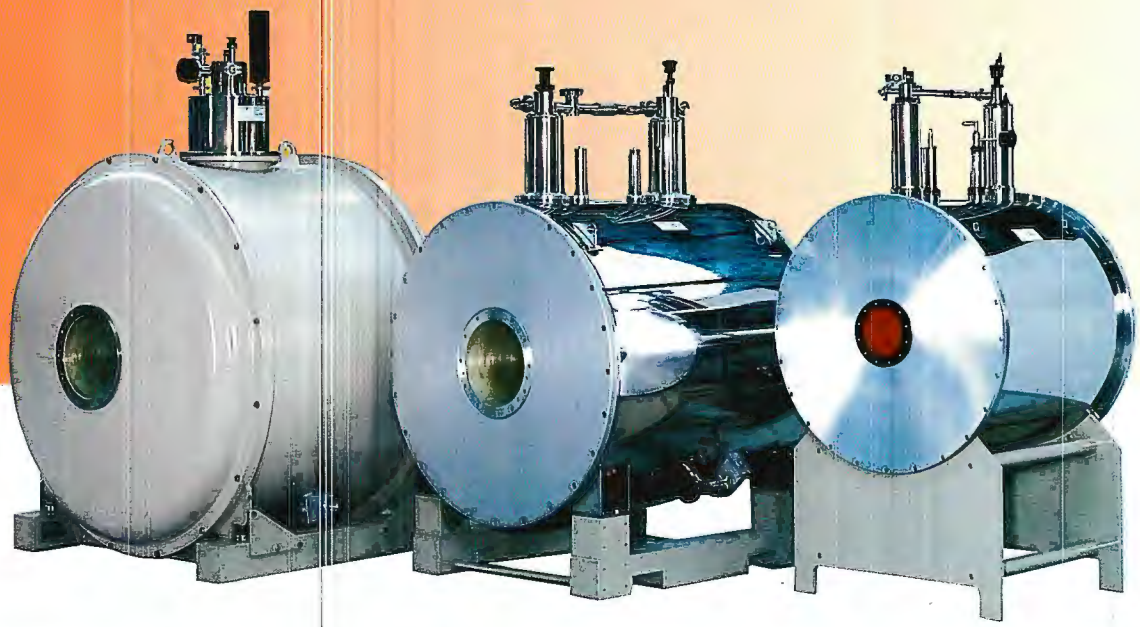
200/330	4.7T	330 mm	254 mm	2.3 G/cm	140 mm DSV ± 5 ppm	70 mm DSV 0.1 ppm	6.95 m	5.60 m
200/400	4.7T	400 mm	324 mm	1.8 G/cm	140 mm DSV ± 4 ppm	80 mm DSV 0.1 ppm	8.50 m	6.75 m
85/310	2.0T	310 mm	225 mm	3.0 G/cm	100 mm DSV ± 5 ppm	70 mm DSV 0.1 ppm	4.50 m	3.63 m

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



200/400, 200/330, and 300/180 magnets for the NMR Imaging Spectrometer System.
(Photos courtesy Oxford Instruments.)

Note: Equipment described is intended for investigational purposes, and is not approved by the FDA for clinical use.

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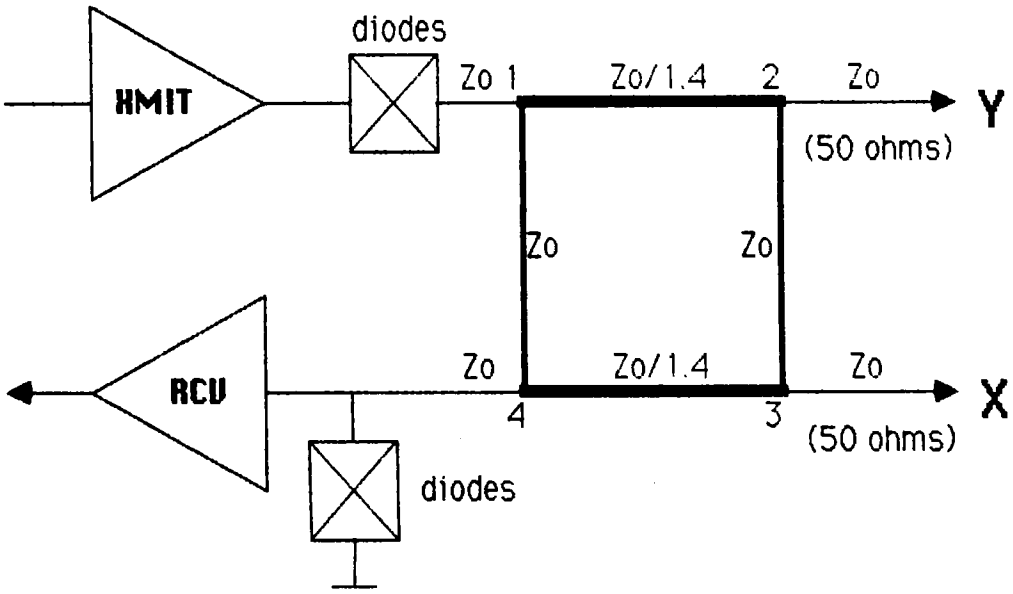

Figure: Coaxial Branch-line Hybrid.

The circuit is in use for about a year and has been successfully employed to drive coils with up to 1500 W RF power. Finally, here is the scattering matrix of the branch-line hybrid [1,2,3,4]:

$$\underline{S} = 1/\mathrm{sqrt}(2) * \begin{bmatrix} 0 & i & 1 & 0 \\ i & 0 & 0 & 1 \\ 1 & 0 & 0 & i \\ 0 & 1 & i & 0 \end{bmatrix}$$

Ref:
(1) Sank, V.J., Chen, C-N., Hoult,D.I., J. Magn. Reson. **69**, 236-242 (1986).
(2) Montgomery, C.G., Dicke, R.H., Purcell, E.M., "Principles of Microwave Circuits", MIT Radiation Laboratory Series, Boston Technical Lithographers Inc., 1947 (p. 308-311).

Sincerely,

Dipl.-Ing. Dr. techn. Manfred G. Prammer

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DEPARTMENT OF CHEMISTRY

SANTA CRUZ, CALIFORNIA 95064

April 4, 1988
(received 4/13/88)

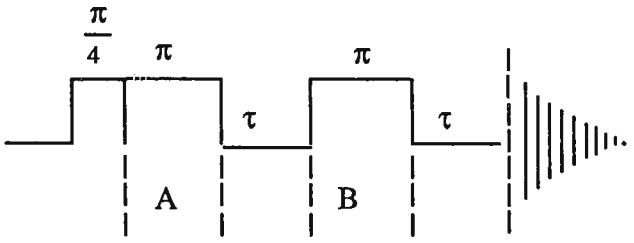
Professor B. Shapiro
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto, CA 94303

Title: PZAP

Dear Barry:

When using the standard Proton-Carbon or Broadband Probe to observe Fluorine-19, one must contend with a Fluorine background signal of about 200 ppm in width, due to the Teflon used in constructing the probe itself. A similar unwanted background can be observed when pulsing Carbon-13, due to Delrin and Teflon, and is especially pronounced when doing a high power standard one pulse experiment using the Magic Angle Solid's Probe.

Taking the basic concepts developed for the Attached Proton Test (Ref: S.L. Patt and J.N. Shoolery (1982), *J. Magn. Res.* **46**, 535-539) and the Hahn Spin-Echo T_2 measurement method (Ref: E.L. Hahn, *Phys Rev.* **80**, 580 (1950)), we have come up with an alternative to the standard pulse sequence for acquisition of the FID to eliminate the background signal for Fluorine-19 and Carbon-13.



Transmitter Phase A is shifted 180° after each acquisition and Transmitter B is correspondingly 180° out of phase with respect to Phase A. With this pulse sequence, the very broad lines associated with solids and viscous liquids are effectively spin-echoed out while the integrity of the spectrum is maintained. In order to achieve a phase corrected absorption spectrum with minimum anomalies at the baseline, this pulse sequence depends on a very accurate 180° pulse. The τ value is set to 400 micro seconds.

Except for small molecules, which seem to exhibit more efficient T_1's, the signal to noise of this pulse sequence is comparable when observing Carbon-13. However, we have not checked or tested absolute reproducibility except on concentrated samples, and would not recommend this sequence for normal Carbon-13 analysis. Where it does have an advantage is in monitoring known NMR signals without having to subtract out the background, which is most difficult with Fluorine-19 or when pulsing at high power with the Magic Angle Solid's Probe.

Using Nicolet /GE language, we wrote the pulse sequence called PZAP:

```
#1: D5
#2: P6,G
#3: PC4,G          XMTR PHASE: A
#4: D10,G
#5: PC4,G          XMTR PHASE: B
#6: D10,G
#7: A,G
#8: D6 JUMP TO #1

PHASE A = (S*2) + 2          MODULO: 4
PHASE B = 2*S                MODULO: 4
```

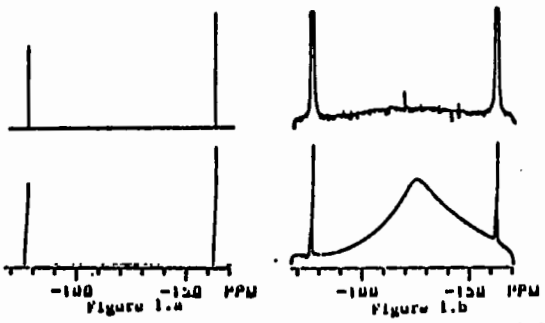


Figure 1.a Figure 1.b

Trifluoroacetic acid(-78ppm) & Hexafluorobenzene(-165ppm)

Figure 1.a,b: top: Fluorine-19 NMR Spectra using PZAP sequence
bottom: Standard pulse sequence for acquisition of FID

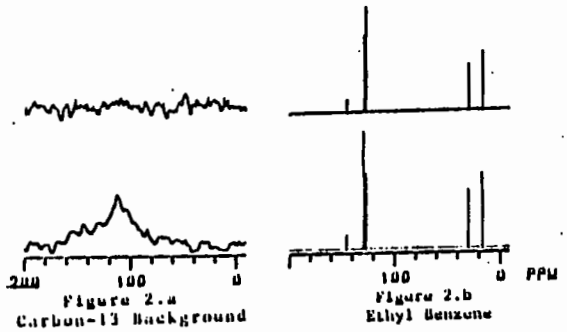


Figure 2.a Figure 2.b
Carbon-13 Background Ethyl Benzene

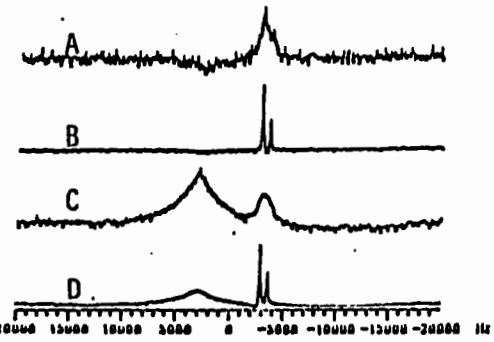


Figure3.A,B,C,D: Carbon-13 spectra of ADAMANTANE, decoupled(B,D)/non-decoupled(A,C)
top:A,B: PZAP pulse sequence
bottom:C,D: Standard pulse sequence for acquisition of FID

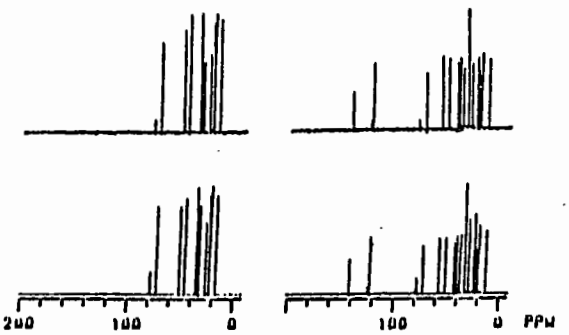


Figure2.c Figure 2.d
Menthol Cholesterol

Figure 2.a,b,c,d: top: Carbon-13 NMR spectra using PZAP sequence
bottom: Standard pulse sequence for acquisition of FID

Figure 1.b illustrates the magnitude of the Fluorine-19 background signal (bottom), and the elimination of the background (top)

Figure 2.a,b,c,d compares the PZAP sequence to the standard pulse sequence for acquisition of the FID. (a) is the Background, (b) Ethyl Benzene, (c) Menthol, (d) Cholesterol.

Figure 3.A,B,C,D: Decoupled and Non-decoupled spectra of Adamantane using Magic Angle Solid's Probe. Top A,B shows the elimination of the Carbon-13 background.

Sincerely,

Jim Loo

Duke University

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April 6, 1988
(received 4/12/88)

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

36" PLOTTERS AND ORCHIDS

Dear Professor Shapiro:

In the last few years, the spectrometer manufacturers have offered 36" plotters as an option for plotting NMR spectra in a "wallpaper" size. For example, 2D NMR spectra can be plotted on grids as large as 80 cm x 80 cm in size to allow discrimination of resonances that are less clear on smaller plot grids.

The Zeta 836 plotter installed on our GN-500 spectrometer 15 months ago works well. However, we recently encountered sporadic situations where the plotter's paper feed worked fine one day, and on a consecutive day it would run the same roll of paper off the roller sprockets, thereby generating a messy 2D plot and an unhappy researcher. Since loading a new roll gave smooth plots and a happy researcher, we initially wrote off the misbehaving roll as a "bad" roll, where the paper aligned nicely with the sprockets in the first part of the roll, but not in later parts of the roll. We eventually, however, realized that this plotter behavior correlated with the room environment and was due directly to the weather outside. More specifically, the level of paper hydration due to the relative humidity (RH) appeared to be responsible. On warmer rainy days, the plotter worked fine. On cold, dry days the 36" plotter paper shrunk sufficiently so that the plotter misbehaved. On days of extremely low humidity and low temperatures, we found that 36" paper shrinks by as much as 1/4", so that it no longer fits the plotter sprockets.

Fortunately Donald Mika, who came to the Duke facility from GE last year and Bruker (US and Europe) previously, grows orchids in his office and has a humidifier to hydrate his giant vanilla orchid. We therefore could rehydrate misbehaving 120 ft rolls of Zeta 36" paper by leaving them next to the orchids. Leaving a roll for a half day by the orchids did wonders for smooth plotting with the 836 plotter.

A check of the Zeta 836 and Zeta 8 manuals reveals optimal plotter operating conditions at +10 to +40°C (50 to 104°F) with RH 70% maximum at 40°C and 30% minimum at 10°C.* An inquiry to the paper manufacturer led to the following information from their quality control department: The 120' rolls of Zeta 836 paper work best at <u>RH 50</u> at typical ambient temperatures of 25°C. At <u>RH 70</u>, the paper expands by ~ 1/8". This is not a problem since a movable wheel on the Zeta allows for this expansion. At RH 30, one can expect

BOX 3711, DUKE UNVERSITY MEDICAL CENTER
DURHAM, NC 27710

the paper to contract by ~1/8". There is some play in the movable wheel to accommodate this contraction. At lower RH and lower temperatures, the contraction can be ~1/4", as we had already discovered. The movable wheel is now at an extreme position on the roller and the contraction is enough that the paper no longer fits.

The Zeta 836 paper measures to 37 3/16" in its horizontal span. Assuming a linear 1/8" expansion/contraction with a change of 20 in RH, this calculates to a change of about 0.0034" per linear inch in the translucent plotter paper (No. 204110). On the Zeta 8 with the smaller paper (No. 200902, 12 15/16" horizontal span), a change of 20 in RH should lead to only a 0.04" change in the paper span, which can be accommodated by the plotter wheels. One can therefore expect good behavior in plotting NMR spectra with the smaller Zeta 8 at most conditions.

Did you know that the vanilla orchid is the only edible member of the orchid family?

Sincerely,

Leonard Spicer Donald Hika Anthony Ribeiro

*NMR not necessarily being a weather-related science, we looked up Salisbury and Ross' classic text on Plant Physiology and found 100% RH defined as the vapor pressure above pure water at equilibrium in a closed container. Since vapor pressure approximately doubles for every 10°C increase in temperature, RH is highly temperature dependent. Warm air of course holds more water than cold air.

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DIVISION OF NUCLEAR MAGNETIC RESONANCE
215/728-3049

April 15, 1988
(received 4/20/88)

Dr. Bernard L. Shapiro, Editor
Texas A&M NMR Newsletter
966 Elsinore Court
Palo Alto, CA 94303

Title: **Probe Design for Cell and Tissue Studies**

Dear Dr. Shapiro:

We have recently spent some time redesigning our high-field (9. probes for cell and perfused tissue experiments. A critical pı been whether to use an axial or transverse orientation for our sample chambers, which reduces to the admittedly old problem o between saddle and solenoid type coil geometries. We thus com sensitivity of a single turn, 1:1 saddle coil of round conduct turn band coil of equal diameter (30 mm), made of copper strip length equal to 0.8 a diameter. Phosphorus spectra were acqui with a Bruker AM400 spectrometer (1200 scans, 1 sec repetitior width, and 60° pulse width for each probe). The cells (NIH 3' fibroblasts) were attached to microcarrier beads and superfus containing 50 mM concentration of phenyl phosphonate (PPA) as compound.

Our results, illustrated in the enclosed figure, reveal that the saddle coil (a) with its vertical geometry is better by about a factor of two in Bo homogeneity than the band coil (b) with its transverse geometry. Consequently, while the band coil yields a 40% greater integrated intensity, the saddle coil has better sensitivity in terms of the observable peak heights. The ratio of 1.4 in relative sensitivities between the band coil and the saddle coil (as measured by the integrated signal) is about 1/2 of the generally accepted factor of 2.6. This discrepancy cannot be explained entirely by differences in sample losses or Q. Since the latter factor of 2.6 was derived by Hoult and Richards [1] for coils having large number of turns, we have made some preliminary computations for coils of one and two turns. These calculations suggest that for these configurations the expected gain in sensitivity of the band coil over the saddle coil is close to what we have observed.

In conclusion, our analysis suggests that for high field vertical bore magnets the gain in sensistivity of the band coil over the solenoidal coil is relatively small. This small gain is more than offset however by difficulties in shimming of the band coil and consequently the saddle coil appears to be the configuration of choice for studies of perfused cells and tissues.

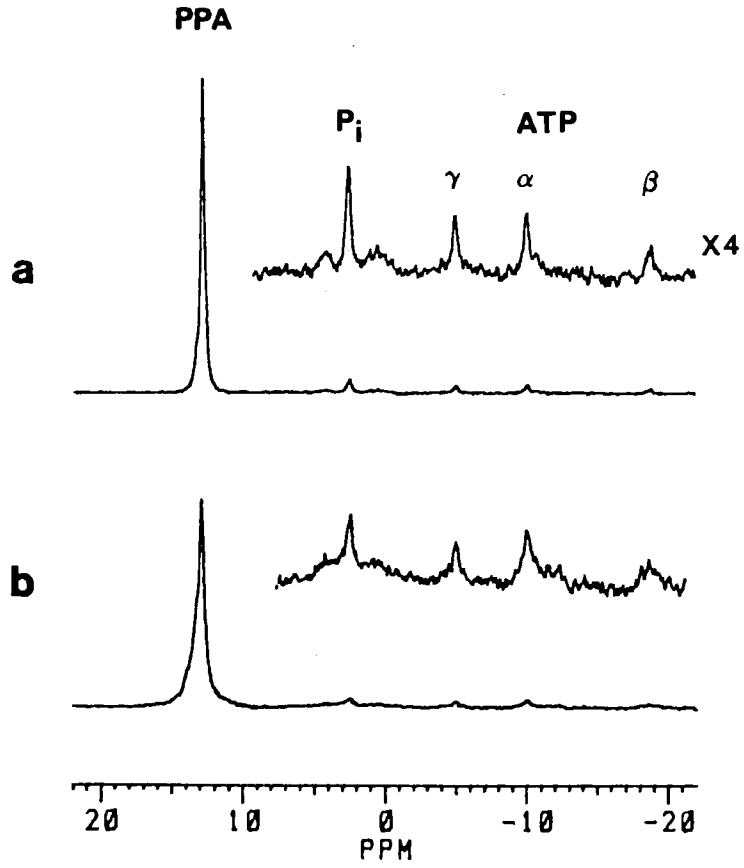
Please credit to Truman R. Brown's laboratory.

Sincerely,

Joseph Murphy-Boesch, Ph.D.

Benjamin S. Szwergold, Ph.D.

[1] Hoult, D.I. and R.E. Richards. J. Magn. Reson. 24:71-85, 1976.



356-60

Washington
WASHINGTON·UNIVERSITY·IN·ST·LOUIS

Department of Physics

March 28, 1988
(received 4/20/88)

Dr. Bernard L. Shapiro
TAMU NMR Newsletter
966 Elsimore Ct.
Palo Alto, CA 94303

Spurious Signal From Kel-F

Dear Professor Shapiro,

We are presently doing a study of some deuterated amorphous semiconductors, using deuterium magnetic resonance. In the course of this study we have found that quadrupole echo spectra often have been accompanied by a large spurious signal. After much trial and tribulation we discovered that the source of this signal was the Kel-F plastic that had been used as sample holders.

The signal existed and had the same characteristics at 30 MHz on a Bruker CXP 200 as it did at 46 MHz on a Chemagnetics CMX 300. It was found that the signal was many MHz broad, and the width of the spectra depended solely on the 90° pulse length and the digitization rate of the receiver. The signal could only be detected at temperatures below approximately 70 K and it had a T_1 on the order of 1 sec. Various tests led to the conclusion that the signal was not from deuterium, but was rather from ^{37}Cl and ^{35}Cl in the Kel-F. At 5 and 7 Tesla the Zeeman splittings are of the order of the quadrupolar couplings, so that the true Cl spectra are very broad; the observed narrower widths simply reflect instumental parameters.

Following are plots of 3 quadrupole echoes. Plot A is the signal seen from our a-Si(D,H) sample and Kel-F sample holder. Plot B is the Kel-F sample holder by itself and Plot C is an empty coil.

Sincerely,

Martin P. Volz R. E. Norberg Mark S. Conradi

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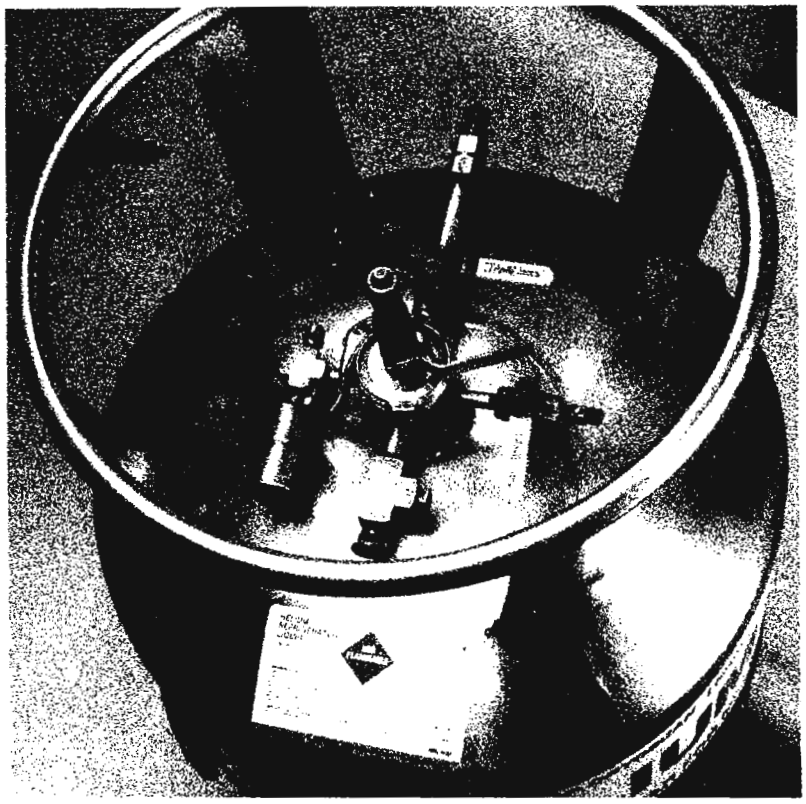

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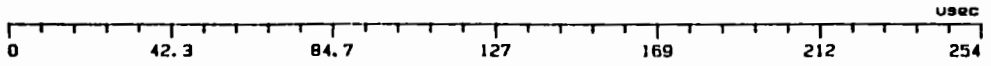
U.S.A.

Plot A

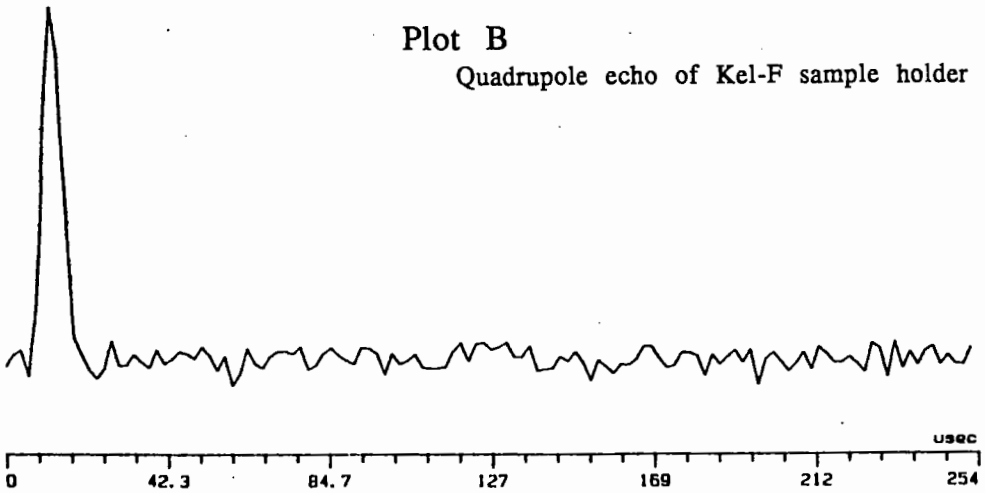
Quadrupole Echo of a-Si(D,H) and Kel-F sample holder

sw = 500 kHz
pw = 4 usec
tau = 40 usec
pd = 1 sec
T = 20 K



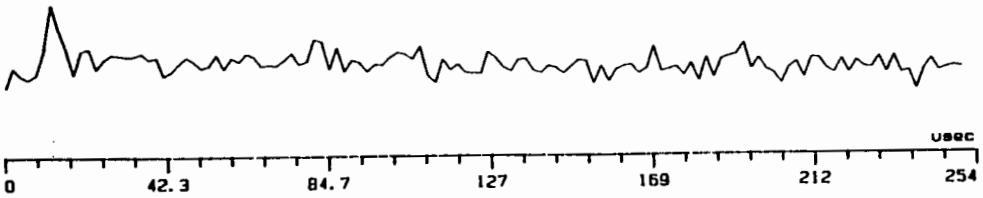
Plot B

Quadrupole echo of Kel-F sample holder



Plot C

Quadrupole echo of empty coil



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Division of Chemistry | Division de chimie

Ottawa, Canada
K1A 0R6

April 14, 1988
(received 4/21/88)

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

TITLE: Dynamic NMR: temperature calibration

Dear Dr. Shapiro:

In dynamic studies, we need to know accurately the temperature in the probe corresponding to the observed spectrum. While a 10° error on a coalescence temperature (T_c) would not affect greatly the $\Delta G^{\ddagger}_{T_c}$ value (error of 2KJ/mole on a 40 KJ/mole value at Tc = 200°K with $\Delta\upsilon$=75Hz) it could lead to an erroneous value of $\Delta S^{\ddagger}$ (error of 11 J/(°)/mole if $\Delta S^{\ddagger}$ = +20 J/(°)/mole in the above conditions).

We measured the temperature of a CD_2Cl_2/Acetone (80/20) mixture under typical experimental conditions, by means of a copper-constantan thermocouple positioned inside the 10 mm sample tube at the centre of the receiving and decoupling coils of a Bruker VSP 300 10 mm probe, without rotation of the tube or decoupling. The temperature inside the probe (cooled by cold N_2 gas flow) was controlled by a Bruker B-VT-1000 unit.

We can see that the error increases at lower temperatures and is about 10° at ~ 200°K. Fortunately, the temperature errors seem to follow a linear relationship.

The following table gives the temperature increase inside the same mixture when we switch the decoupling coil on during different kinds of decoupled ^{13}C pulse sequences:

DEPT(1)	WALTZ 16 with NOE(2)	Broad Band irradiation with NOE(3)
+ 0,6°±0,2	+2.2°±0.5	+4° ± 1

(1) Delay = 5s; Acquisition = .5s (dec. power = 20H; γB_2~4.000 Hz)
(2) decoupling power = 30 H continuously applied; γB_2~2.800 Hz)
(3) decoupling power = 20 H continuously applied; γB_2~4.000 Hz)

These results show that it is worth spending time calibrating the temperature of one's probe before starting NMR dynamic experiments!

Sincerely yours,

Claude Morat

Chris Ratcliffe

/pt

Canada

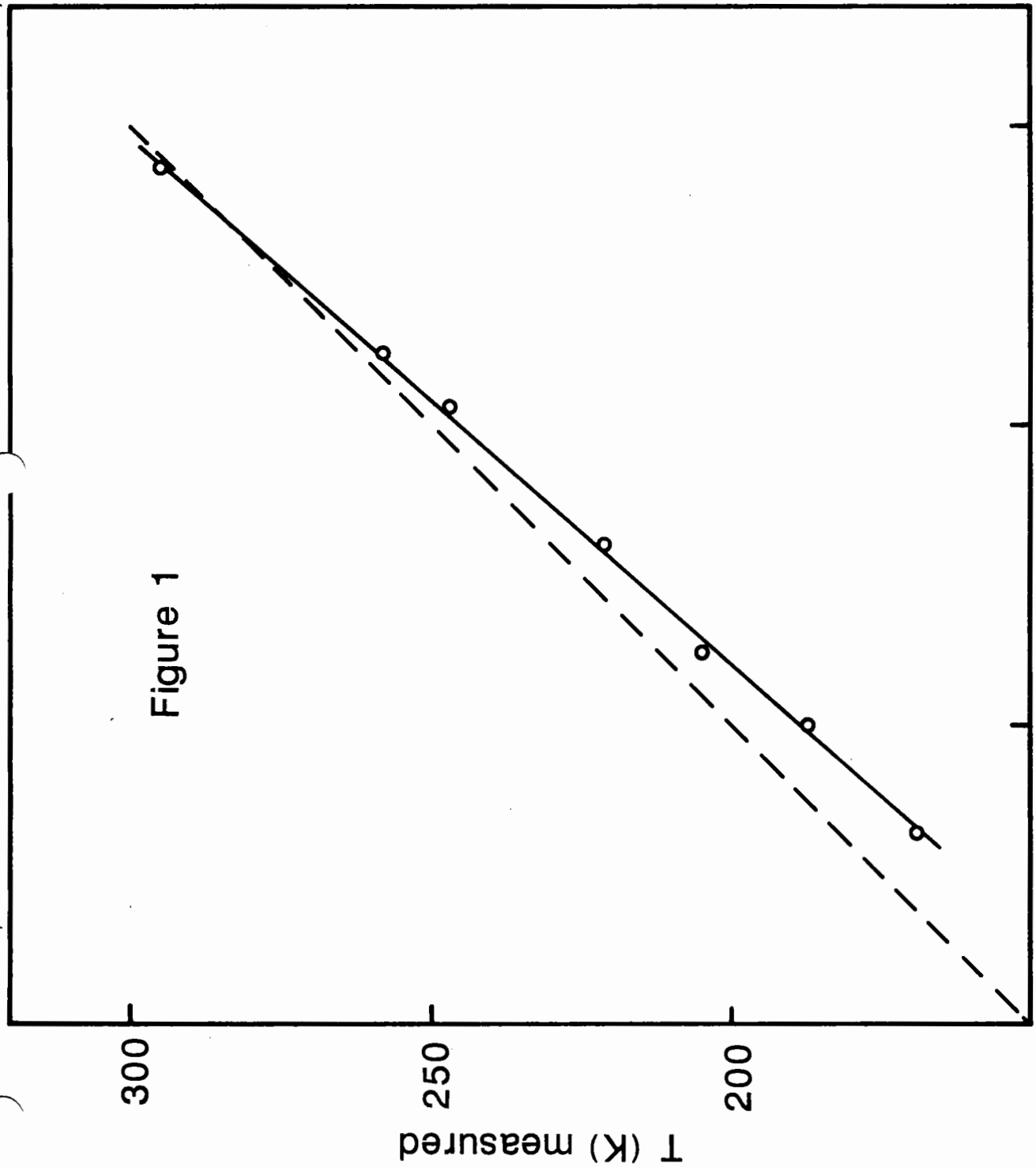

Figure 1

356-66

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POST DOCTORAL POSITION AVAILABLE

Dear Professor Shapiro,

We have a postdoctoral position likely from 1.7.88 - 31.12.89 available.

The duties of the position are:

- Development of a departemental NMR-net based on Bruker AC-300, Bruker AM-400, Bruker X32 and VAX 750. Knowledge of UNIX would be ideal.
- Development of Metal-NMR
- Application and Development of 2D-NMR methods.

Candidates with a Ph.D. in Organic/Inorganic or Physical Organic Chemistry and strong background in modern NMR should write to me and give two references.

Sincerely yours

POSTDOCTORAL POSITION available immediately for work on Electron Spin Resonance (ESR) studies of ionomeric membranes. Experience in ESR spectroscopy, computer simulations and polymers are highly desirable, in that order. The position is supported by an NSF grant and is available for one year initially, with possible extension.

Please send a curriculum vitae and the names of three references to:

Professor Shulamith Schlick
Department of Chemistry
University of Detroit
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or call (313) 927-1012 or 927-1258.

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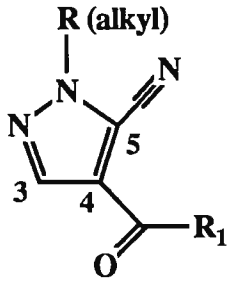
April 15, 1988
(received 4/21/88)

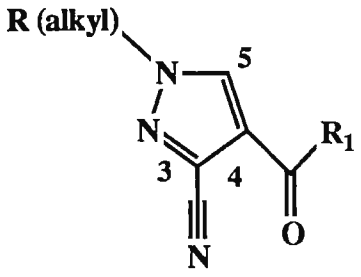
Dr. Bernard L. Shapiro, Editor
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto, California 94303

Dear Barry:

C-13/H-1 NMR OF 5-CN AND 3-CN PYRAZOLES

During the course of conducting an SAR study of selected pyrazoles for our pesticide program, numerous derivatives of both the 5-CN and 3-CN regioisomers were obtained. A quick inspection of the proton NMR spectra does not provide a reliable distinction between these isomers, so we explored the application of the relatively well documented method of solvent shift of the pyrazole ring proton and found an excellent correlation with C-13 nmr shifts. Thus, for our extensive series of pyrazoles H-5 (3-CN) experiences a downfield shift of 0.6 ppm (±0.2) in DMSO-d_6 relative to $CDCl_3$, while H-3 (5-CN) is comparatively insensitive to this solvent change. This was found to correlate well with C-13 NMR data, where C-3 (5-CN) appears at 137-140 ppm, while C-5 (3-CN) consistently resonates at 130-132 ppm regardless of R(alkyl) or R_1.





R_1 = OH, NH_2, OR (R = alkyl)

The structure of at least one compound was further verified by an X-ray crystal determination. We are confident that the C-13 method can be extended to fully substituted pyrazoles in the future. Please credit the account of Dr. D. Dorman.

Sincerely,

LILLY RESEARCH LABORATORIES

George E. Babbitt
Agricultural Organic Chemistry

Michael P. Lynch
Agricultural Organic Chemistry

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

April 4, 1988
(received 4/22/88)

Magic Angle Spinning of Fluoropolymers

Dear Barry,

This is in response to your gentle reminder and to forestall any thoughts of an ultimatum you might be contemplating... (We are all appeasers now). Please credit this contribution to the subscription of Eastman Kodak Company.

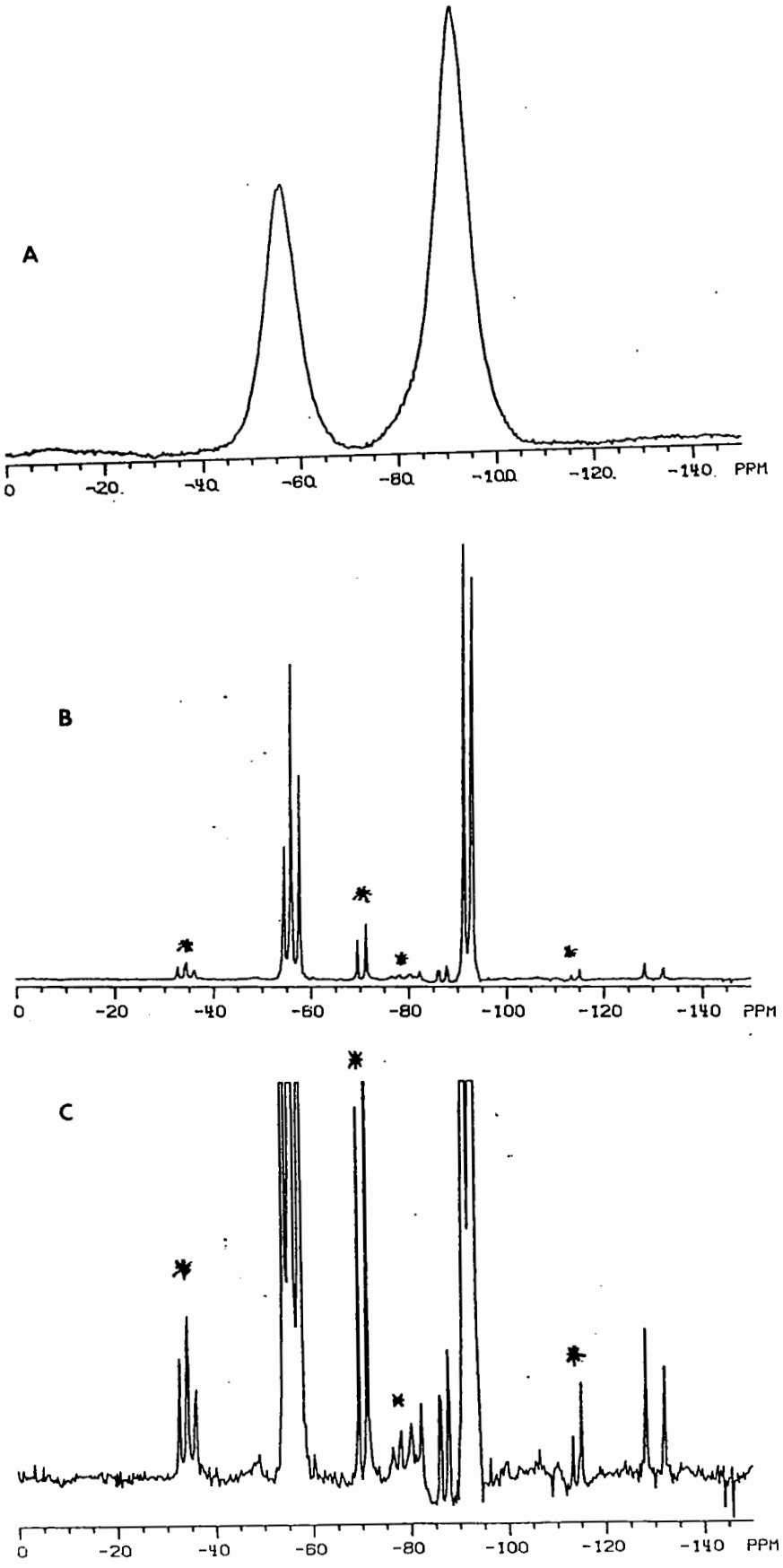
We have recently come across a class of fluoropolymers that contain long chains of -CF_2-O-CF_2-O-CF_2CF_2-O- in which the static ^{19}F linewidth is quite narrow (Fig. 1A). Magic angle spinning leads to excellent line narrowing and a surprisingly well-resolved spectrum with plenty of sequencing information (Fig. 1B). The "triplet" corresponds to -CF_2- in the three possible triad sequences and the "doublet" to ^{19}F in the two possible triad environments for -$C\underline{F}_2$-CF_2-. These assignments were suggested by Mr. W. C. Lenhart of Kodak's Analytical Technology Division. Spinning sidebands are indicated by *; the weaker peaks, seen more clearly in Fig. 3C, probably stem from end groups.

We have obtained high resolution ^{19}F MAS spectra from a large number of fluoropolymers, many of them not readily soluble in conventional solvents. We are now beginning to explore the utility of 2D NOE experiments in these solids. The spectra were obtained at 188.2 MHz using a Chemagnetics probe with the decoupler channel tuned to ^{19}F.

With best regards,

Nick

Nicholas Zumbulyadis
Corporate Research Laboratories


A
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-20.
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-80
-100
-120
-140
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C
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-60
-80
-100
-120
-140
PPM

Figure 1

Hoffmann-La Roche

Hoffmann-La Roche Inc.
340 Kingsland Street
Nutley, New Jersey 07110-1199

Direct Dial

Altering Overlap Patterns of Peptide Resonances by Changing The Solvent

March 23, 1988
(received 4/23/88)

Dear Barry:

One of the projects in our laboratory is the study of a series of peptide hormones in the size range of 25-35 residues. We are using solvents with various ratios of methanol/water to discern how these peptides fold, and to explore how the preferred structures might be stabilized by amino acid substitution and chemical modification. We have found that the structures of peptides of this size can be dramatically affected by the solvent, and that chemical shifts can be substantially altered. These changes in chemical shift often create differing patterns of overlap among samples of a given peptide. Changing solvents, therefore, can be useful for making assignments. The chemical shift differences are generally larger and more diverse than those observed following changes in temperature or pH.

Although overlap problems concerning J connectivity can usually be solved by performing additional experiments (relay-COSY, HOHAHA, DQ, TQ), we have not had much success in resolving NOE overlap with exotic experiments (eg. - relay-NOESY, DQ NOESY). In such a case, moving the resonances, by changing to a less structure-inducing solvent, and observing separated NOEs can at least suggest the NOE patterns present within the overlap in the stabilizing solvent. An example is cyclo$^{8-12}$[Asp^{8}, Ala^{15}]-GRF-(1-29)-NH_2. In 75% methanol, the segment Lys^{14}-Ala^{15}-Gln^{16} is within a series of residues exhibiting NH_i-NH_{i+1} NOEs. We assume that the Lys^{14}NH-Ala^{15}NH and Ala^{15}NH-Gln^{16}NH NOEs are both present and overlapped (Figure 1A). Although they are quantitatively useless, we would still like to know if both are really present. The results in 0% methanol suggest that they are (Figure 1B).

We have observed that differences due to solvent can sometimes be greater than those caused by amino acid substitutions. Side-chain protons appear to be affected to a lesser extent than the $C_\alpha H$ and NH protons. This is helpful because the latter are not easily matched by a direct comparison of the spectra (Figure 2).

Sincerely yours,

Dave Ross

David C. Fry, David N. Greeley, and Ross G. Pitcher
Department of Physical Chemistry

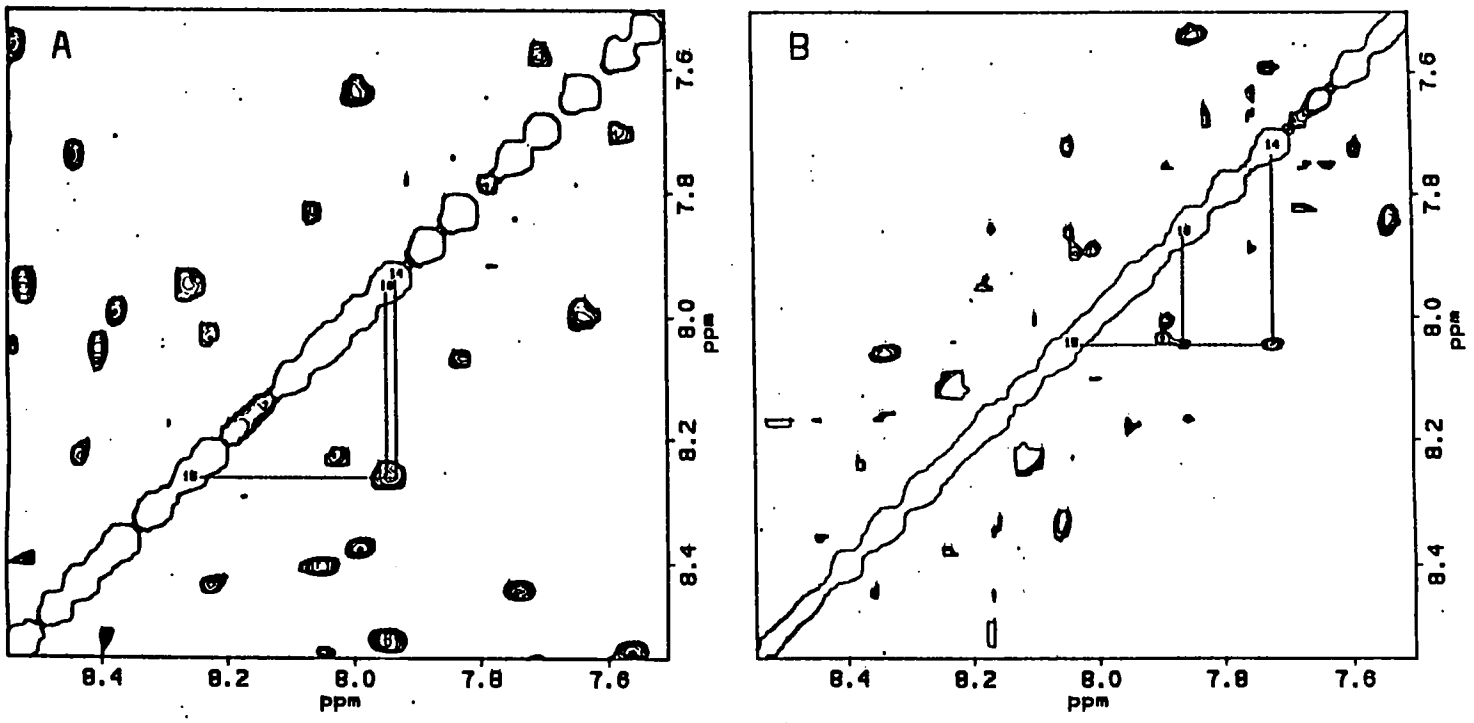

FIGURE 1: Part of the NH-NH region from the NOESY spectra of cyclo$^{8-12}$[Asp^{8}, Ala^{15}]-GRF-(1-29)-NH$_2$ in: (A) 75% CD_3OH/25% H_2O pH 3 22.5°C; (B) 0% CD_3OH/90% H_2O/10% D_2O pH 3 22.5°C. The mixing time was 0.2 s in each experiment. Dotted lines connect the NOEs: Leu^{14}NH-Ala^{15}NH-Gln^{16}NH.

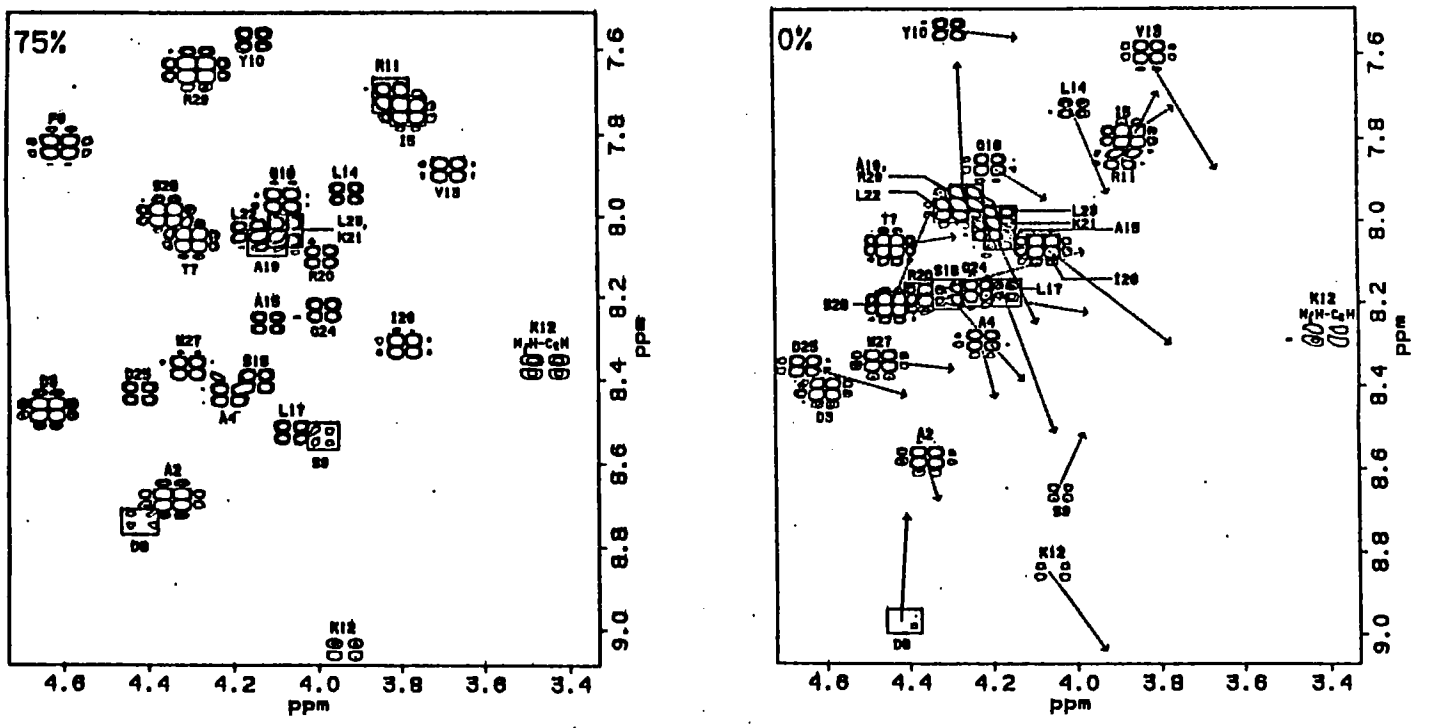

FIGURE 2: C_αH-NH "fingerprint" region from the COSY spectra of cyclo$^{8-12}$[Asp^{8}, Ala^{15}]-GRF-(1-29)-NH$_2$ in: (Left) 75% CD_3OH/25% H_2O pH 3 22.5°C; (Right) 0% CD_3OH/90% H_2O/10% D_2O pH 3 22.5°C. Arrows indicate chemical shift changes between the samples, from 0% to 75% methanol.

PENNSTATE

College of Medicine • University Hospital
The Milton S. Hershey Medical Center

Department of Radiology

P.O. Box 850
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(717) 531-6069

April 20, 1988 (received 4/22/88)

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

Dear Professor Shapiro:

Characterization of the Mitochondrial Matrix P-31 Metabolites

We have undertaken a study to characterize the intramitochondrial or matrix adenine nucleotide and inorganic phosphate P-31 NMR signals. Information about the distribution and NMR visibility of metabolites between the mitochondrial and cytosolic compartments within the cell would greatly aid our understanding of basic cellular processes and interpretation of in vivo and perfused organ P-31 NMR spectra.

We used a Bruker AM-400WB spectrometer equipped with a 20 mm C-13/P-31 double-tuned probe. Liver mitochondria were prepared from male Sprague Dawley rats after perfusion of the livers with ice-cold mannitol-sucrose medium containing EDTA. The mitochondria (30 mg/ml) were maintained at 8°C in a 15 mm NMR tube (fitted inside a 20 mm tube) in buffered KCl media containing CDTA with succinate and glutamate as respiratory substrates. At this low temperature, it was only necessary to insert the oxygen bubbler into the suspension between 7 minute periods of signal accumulation. This allowed for optimal shimming on the water proton FID which enabled us to accurately resolve the intra- and extramitochondrial inorganic phosphate and calculate the pH gradient (Figures 1,2). However, at more physiological temperatures constant oxygenation in the presence of fluorocarbons is recommended. It is interesting to note that the bubbling rate can be monitored by observing perturbations in the lock level if D_2O is contained in the outer 20 mm tube. An aqueous solution of methylenediphosphonic acid contained in a capillary was useful as an area and chemical shift standard. Due to the instability of the metabolites, T_1 values were computed from a modified progressive saturation experiment. Pairs of signal intensity ratios were obtained for the corresponding repetition times and the flip angle was optimized.

The effect of ionic environment was investigated by additions of Mg^{2+}, Ca^{2+} or Mn^{2+}. Figure 1 shows the effect of magnesium on the P-31 spectrum of ATP "loaded" mitochondria oxidizing succinate and glutamate. ATP and phosphate have been added as seen in 1A. $MgCl_2$ has subsequently been added in 1B, followed by the addition of excess CDTA in 1C. CDTA strongly chelates divalent cations which allows clear separation of the matrix from external β and γ ATP resonances.

The effect of mitochondrial inhibitors, oligomycin and carboxyatractyloside (CAT) are demonstrated in Figure 2. Oligomycin blocks mitochondrial ATP synthesis while CAT inhibits the adenine nucleotide translocase. ATP and phosphate have been added to the "loaded" preparation as seen in 2A. Oligomycin has subsequently been added in 2B (note the increase in ADP) while 2C shows the effect of both oligomycin and CAT. All spectra were recorded with 68° rf pulses at a 0.30 sec repetition rate and over 1200 accumulations.

We have concluded that the mitochondrial matrix metabolites are NMR visible at 8°C, consistent with parallel enzymatic assays. At 25°C it has been more difficult to quantitate inorganic phosphate, believed to be due to technical obstacles. The T_1 of the β-phosphate from matrix ATP is as short as 0.2 - 0.3 sec. Finally, saturation transfer experiments at elevated temperatures have demonstrated the feasibility of using this technique to measure fluxes with isolated mitochondria.
Detailed manuscripts are in preparation.

Sincerely,

Gerald D Williams

Susan M. Hutson* Gerald D. Williams+

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PEAK ASSIGNMENTS

1. MATRIX P_i
2. EXTERNAL P_i
3. MATRIX γATP + βADP
4. EXTERNAL γATP + βADP
5. MATRIX βATP
6. EXTERNAL βATP

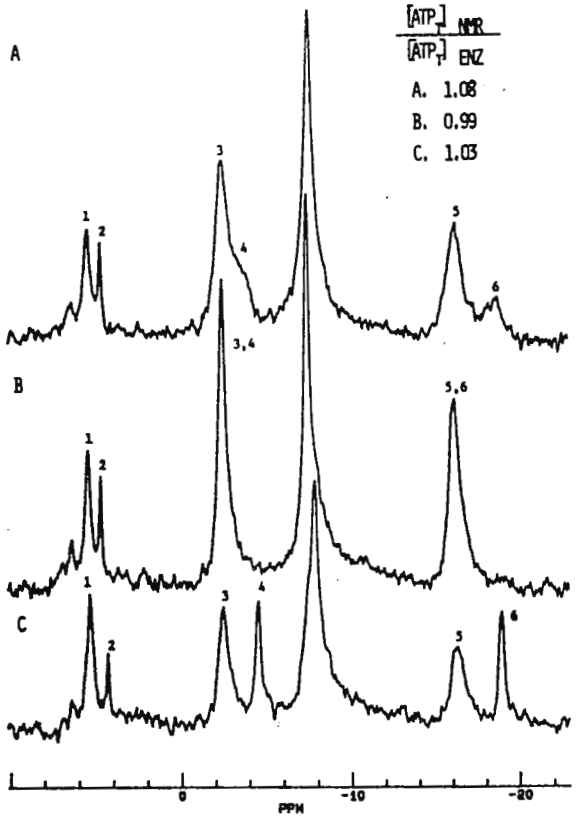

FIGURE 1.

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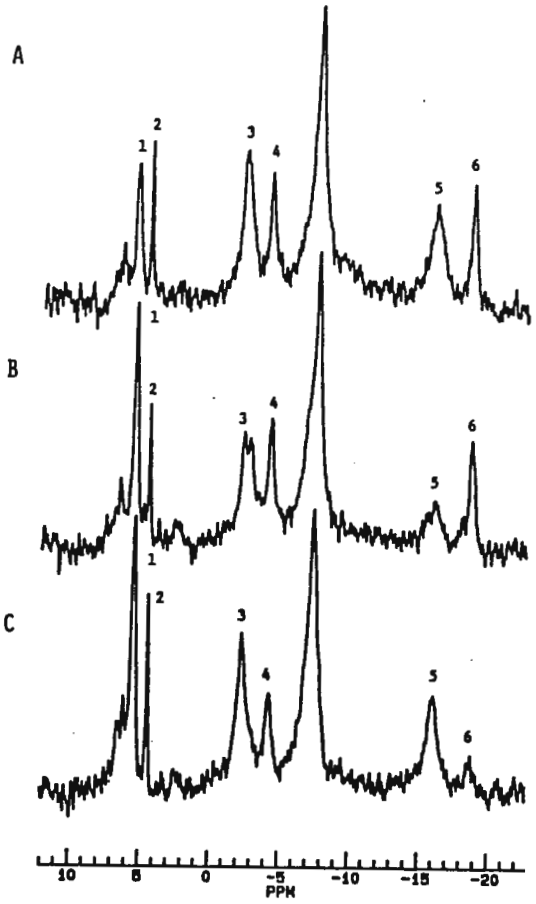

FIGURE 2.

THE UNIVERSITY OF SYDNEY

DEPARTMENT OF BIOCHEMISTRY

SYDNEY N.S.W. 2006
AUSTRALIA

TELEPHONE: (02) 692-2222
TELEX: UNISYD 26169

15 April 1988

UNIVERSITY OF SYDNEY, DEPARTMENT OF BIOCHEMISTRY

Lectureship in Biochemistry (Biochemical NMR spectroscopy)

Dear Barry,

We are currently advertising the above position for which duties will consist of education and research. Staff in our Department teach Biochemistry to students in Dentistry, Medicine, Science, Pharmacy and Veterinary Science. All academic staff are full members of the Faculties of Medicine and Science. Preference will be for someone with an active research committment to biochemical NMR spectroscopy; and other things being equal preference will be given to applicants studying macro-molecular structure (e.g., DNA/protein interaction). We have a Varian XL/VXR400 spectrometer and ADS4000 data station in the Department and a 600 MHz spectrometer will probably be purchased in 1989. The successful applicant will commence duties as soon as possible after January 1989. Appointments to Lectureships are usually probationary and the salary range is A$28,693-37,434. Applications quoting reference number, including a curriculum vitae, list of publications and names and addresses of three referees to the Registrar, University of Sydney, NSW, 2006 by 16 September 1988.

Yours sincerely,

Philip W. Kuchel

PHILIP W. KUCHEL,
Professor of Biochemistry

TAMU NMR Newsletter

Editor/Publisher: Bernard L. Shapiro
966 Elsinore Court, Palo Alto, CA 94303, U.S.A. (415) 493-5971

TAMU NMR Newsletter Subscription Rates to Increase.

For the first time in five years - since the October 1983 - September 1984 Newsletter year - it is necessary to raise the subscription rates.

The current subscription rate has been in effect since October 1983. The domestic letter rate then was 20 cents per oz.; it is now 25 cents, and rates for other classes of mail have increased at least comparably. During the last five years, the size of the average Newsletter issue has increased from 45 pages (average of the January-May issues) in 1983 to 82 pages in 1988. Printing, labor, supplies, and other costs have also risen substantially, of course.

Subscription rate rises would have occurred earlier and more frequently without the markedly increased revenues that have been derived from our loyal Sponsors and Advertisers. The part that these two strongly overlapping groups (*q.v.*) have played in keeping the Newsletter afloat cannot be overestimated. They deserve our best thanks and continuing support. However, additional advertising remains an important need, so please help, directly or indirectly, if you can.

To come to the point, then, please be advised that **effective for the October 1988 - September 1989 Newsletter year, the annual subscription rate for the TAMU NMR Newsletter will be US$120.00**. The 50% academic/personal discounts will continue to be applicable. The new rate will continue to include mailing at Printed Matter (Third Class) rates. First class, Air Mail, or other special posting services are available, and I will be happy to provide firm quotations on request.

Invoices for the 1988-89 Newsletter year will be mailed out approximately July 1. If you do not receive an invoice by, say, mid-July, please let me know. It will be greatly appreciated if you will see that these invoices are processed promptly, for fiscal considerations require that subscription funds must be received (or clear indication given me that payment is in the works) by October 1, if your Newsletter mailings are to continue without interruption.

I believe that aside from these fiscal necessities, the Newsletter continues in good health. The mailing list continues to grow in a slow, orderly fashion, as it has for lo these many years. The self-regulating nature of the mailing list apparently continues to be effective (The rapid responses of the great majority of those receiving the notorious 'Ultimatum' notices also suggest that the Newsletter continues to serve useful purposes.). No significant changes in the operating policies of the Newsletter are envisioned as we now approach the completion of the 30th year of unbroken (albeit occasionally 'bent') monthly publication.

B.L.S.
1 May 1988

TEXAS A&M UNIVERSITY
NMR NEWSLETTER

No. 356
May 1988

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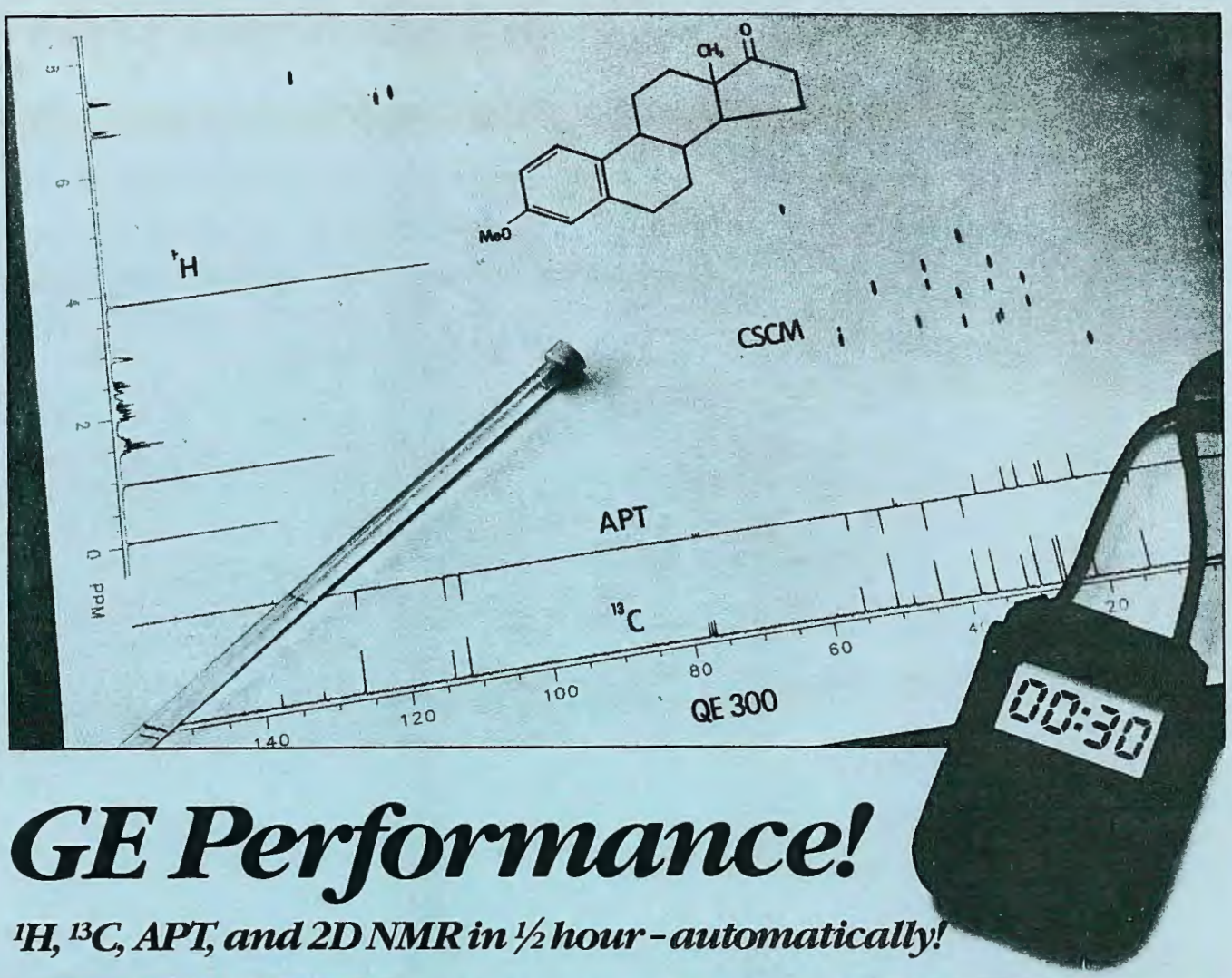
Editorial/Policy re 'Used' Material Appearing in the Newsletter.

A subject of concern to several present and potential TAMU NMR Newsletter participants, as well as to myself, is whether the Newsletter ought to contain material which has recently appeared in the formal literature or is scheduled definitely to appear very shortly (*i.e.*, within a few weeks) after it would appear in the Newsletter.

I feel that a TAMU NMR Newsletter contribution should not duplicate, summarize, or abstract material which has been published, or which will appear in the formal literature within a small number of weeks of the Newsletter account.

On the other hand, let it be firmly emphasized that if the appearance of an article in a journal is several months away, a brief account (as an abstract with or without a 'Preprint Available' notice, a separate informal account, a selection of material from the manuscript, or what have you) sent in to the Newsletter fulfills one of its very important functions. We trust that a participant himself/herself will, in each case, apply the criterion of whether or not such a technical contribution will communicate some subject matter to the Newsletter recipients before they could read it elsewhere.

B.L.S.
1 May 1988



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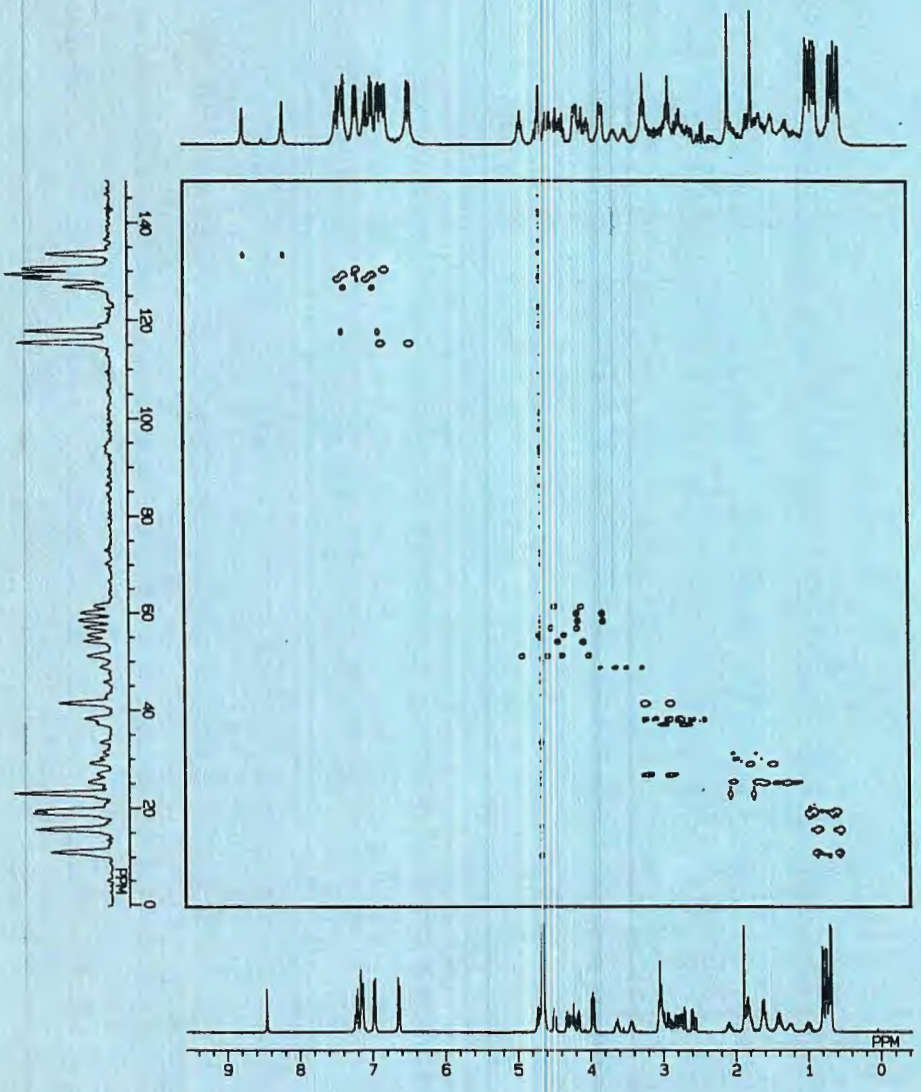
Subject: Rf STABILITY

All of the automation, elegant experiments, and high speed computer processing will do nothing for an NMR experiment if the spectrometer is not stable. The Rf section of the spectrometer must be reproducible and clean of spurious signals over periods of days for some experiments.

One of the most demanding experiments for spectrometer stability is the reverse detection (H [C]) experiment without C decoupling. Between the relatively sharp lines and the low magnitude of the satellites, this experiment graphically demonstrates the stability of the spectrometer. As the data below shows, a standard JEOL GSX Spectrometer has the Rf stability to do these experiments.

ANGIOTENSIN-II, 3mg.in.D20



*Sample courtesy of Dr. Jeffrey Hoch (Rowland Institute For Science, Inc.)

For further information call:

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