TEXAS A&M UNIVERSITY

NMR NEWSLETTER

No. 359

August 1988

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TEXAS A&M NMR NEWSLETTER NO. 359, AUGUST 1988 ADVERTISER INDEX

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FORTHCOMING NMR MEETINGS

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

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

American Chemical Society, 196th National Meeting, September 25 - 30, 1988; Los Angeles, CA; Includes several symposia on NMR; Contact B. R. Hodsdon, 1155 16st Street NW, Washington, CD 20036, (202)872-4396.

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

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

Second Missouri Magnetic Resonance Symposium, October 25, 1988; St. Louis, Missouri; Contact Dr. D. W. Larsen (314) 553-5341 or Dr. F. D. Blum (314)341-4451, Bitnet: C2828@UMRVMB.

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. 361 (October)——23 September 1988
No. 362 (November)——21 October 1988
No. 363 (December)—18 November 1988
No. 364 (January) —— 16 December 1988

Lehigh University

Department of Chemistry
telephone (215) 758-3470

Seeley G. Mudd Building 6
Bethlehem, Pennsylvania 18015

June 15, 1988
(received 7/5/88)

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

Dear Professor Shapiro,

We are currently studying interactions between tRNA and some cationic, water-soluble porphyrins. Proton NMR spectra of yeast $tRNA^{Phe}$ were acquired on a Bruker 500 MHz spectrometer using a 1331 water suppression sequence. The resonance signals were distinguished according to the proton assignments of Heerschap, Redfield, and others (1).

Shown in Figure 1 is a series of stacked plots for tRNA with various amounts of tetra-4N-methyl pyridyl porphyrin (T4NMPyP) added. For each trace, only the imino spectral region (8.5-15.0ppm) is presented. Two signals are observed to shift upfield in response to the addition of porphyrin, specifically, those assigned to non-hydrogen bonding imino protons on base ψ55 (peaks "s" and "x" in the spectra). An additional proton resonance assigned to a methyl group on base T54 (1.0ppm; not shown) also shifts upfield. Resonance shifts and generalized linebroadening are noted upon addition of either T4NMPyP or its copper or zinc analogs to tRNA.

Based upon the NMR data and earlier CD and uv-vis work (2), it appears that the porphyrins interact with tRNA in a 1:1 ratio at the TψC loop, but the interaction is neither classical intercalation nor simply electrostatic binding. We contend that porphyrins occupy a unique binding site on tRNA as compared to the classical intercalator, ethidium bromide, which is known to reside in the mouth of the P10 loop stacked over base U8. Negligible nuclear Overhauser enhancements indicate long internuclear distances; porphyrins may cap the TψC loop or lodge in a fold of the tertiary structure.

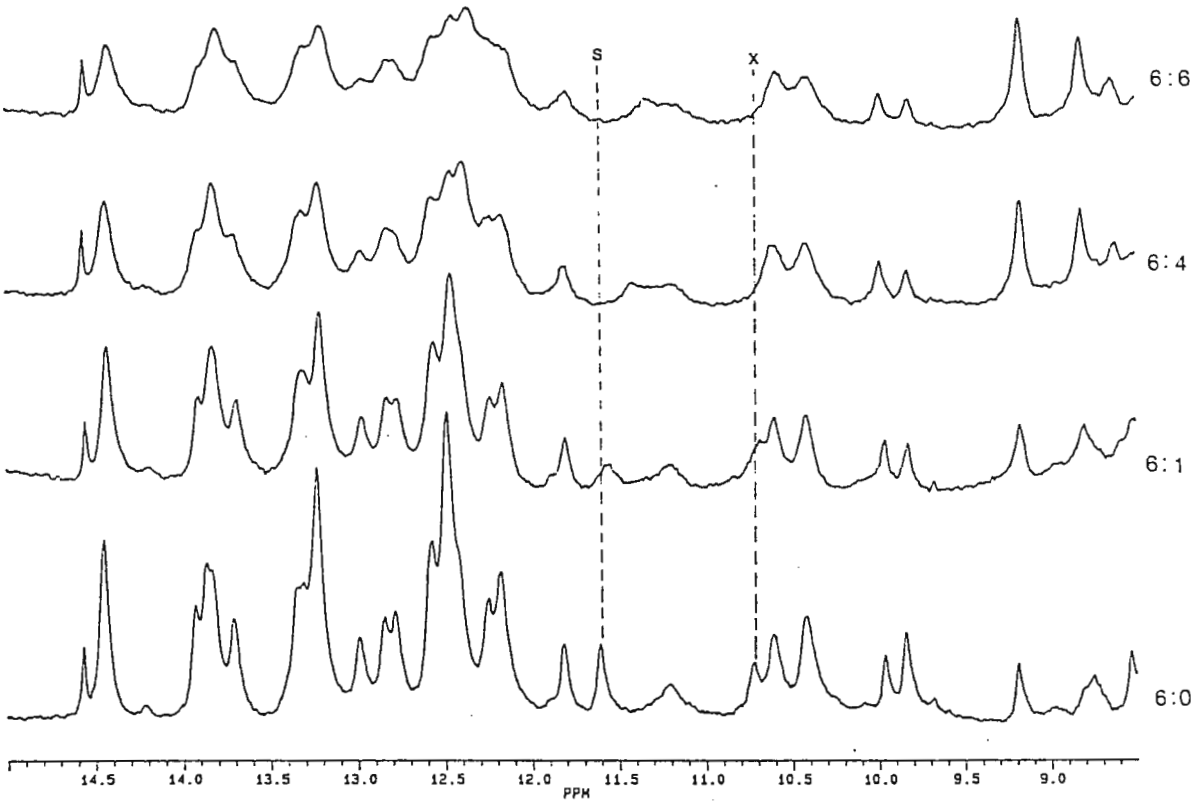
In addition, the manganese porphyrin does not exhibit the same effect on the tRNA ^{1}H spectrum, suggesting that axial thickness may play a role in determining the binding. To define the steric constraints of the interaction, ^{1}H and ^{19}F NMR will be used to observe bulkier, fluorinated porphyrins and tRNA in solution.

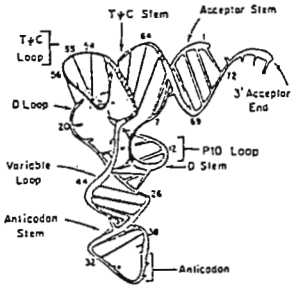
Please credit this entry to the joint Air Products/Lehigh University account maintained by Bill Anderson.

Best regards,

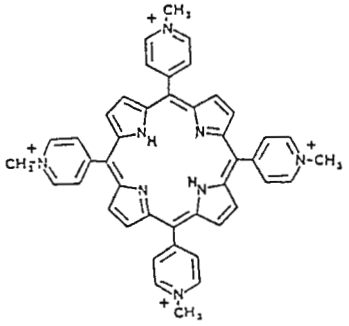
Bill Anderson Bill Birdsall Natalie Foster Jan Robert

Titration of tRNA with T4NMPyP
(Molar Ratio tRNA:T4NMPyP on right)





tRNA phenylalanine specific



tetra-4N-methyl pyridyl porphyrin

REFERENCES

(1) [a]Heerschap, A.; Haasnoot, C.A.G.; Hulbers, C.W. Nucl. Acids Res. 1982, 10, 6981-7000.
[b]Heerschap, A.; et. al. Nucl. Acids Res., 1983, 11, 4501-4520.
[c]Roy, S.; Redfield, A.G. Biochem., 1983, 22, 1386-1390.

(2) Foster, N.; Singhal, A.K.; Smith, M.W.; Marcos, N.G.; Schray, K.J. Biochim. Biophys. Acta, 1988, In Press.

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BERKELEY, CALIFORNIA 94720

NMR Facility

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

July 7, 1988
(received 7/9/88)

Dear Barry,

EASY SET-UP AND PHASING OF DEPT SPECTRA

The initial DEPT experiments have been incorporated into the manufacturers' libraries of pulse sequences without any modifications. Consequently, the 0-order phase correction usually differs from a spectrum obtained by a single pulse sequence (with or without decoupling) by 90°. For this reason, a DEPT with a 45° theta pulse is often acquired in order to get the proper phase parameters. In our facility we modified the sequences to have a receiver phase shift of 90° compared to the original ones (e.g 1133 instead of 0022). In addition, we added the .AU programs DEPT45.AU, DEP90.AU and DEPT135.AU where the variable proton pulse is set to the respective angles. The Bruker experiments were further modified to include pulse multipliers/divider in the .AU program. In the AS set-up routine, only the Decoupler 90° and the Observe 90° pulse needs to be specified.

The EX library of the Nicolet or GE NMC and GENT NMR programs can be modified for the same phase cycling. Pulse multipliers or dividers are unfortunately not supported.

The local preference is to acquire a decoupled spectrum first. If DEPT spectra are needed, the same phase correction can then be used. This is also quite helpful when running automated acquisition and processing. On our Bruker AM-400 we changed the automatic acquisition of DEPT 45-90-135 spectra to BB, DEPT -90 -135.

Sincerely,

Rudi

Rudi Nunlist

Some Biologically-interesting Compounds from

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CAT. NO.	COMPOUND[§]	QUANT.[†]	PRICE[‡] U.S.$
83-12205	L-Alanine-3-^{13}C	0.5g	200.
		1g	355.
83-12301	L-Aspartic-3-^{13}C Acid	0.05g	200.
		0.1g	375.
		0.25g	750.
83-12302	L-Aspartic Acid-4-^{13}C	0.1g	150.
		0.25g	300.
		0.5g	475.
83-82003	Benzoic Acid-α-^{13}C	1g	60.
		5g	250.
83-80513	Carbon-^{13}C Tetrachloride	0.5g	115.
		1g	170.
83-80515	Chloroform-^{13}C	0.5g	280.
		1g	500.
83-80517	Dichloromethane-^{13}C	0.25g	250.
		0.5g	430.
83-08000	N-(4-Ethoxy-1-^{13}C-phenyl)-acetamide (Phenacetin)	0.5g	170.
		1g	310.
83-62040	Ethyl Acetoacetate-1,3-$^{13}C_2$	0.5g	150.
		1g	225.
83-62044	Ethyl Acetoacetate-2,4-$^{13}C_2$	0.5g	185.
		1g	280.
83-30000	D-Glucose-1-^{13}C	0.25g	135.
		0.5g	250.
		1g	325.
		5g	900.
		10g	1400.
83-12201	Glycine-1-^{13}C	1g	105.
		5g	475.
		10g	850.
83-12204	Glycine-2-^{13}C	0.5g	130.
		1g	200.
		5g	900.
83-12206	Glycine-1,2-$^{13}C_2$	0.5g	160.
		1g	290.
83-42009	Hexadecanoic Acid-1-^{13}C (Palmitic Acid)	1g	90.
		5g	350.
		10g	500.
83-62035	3-Methylbutanoic Acid-1-^{13}C (Isovaleric Acid)	1g	175.
		5g	500.
83-42011	Octadecanoic Acid-1-^{13}C (Stearic Acid)	1g	105.
		5g	375.
83-42001	Octanoic Acid-1-^{13}C	1g	105.
		5g	375.
83-62019	Sodium L-Lactate-3-^{13}C	0.25g	135.
		0.5g	250.
		1g	475.
83-62023	Sodium Pyruvate-2-^{13}C	0.5g	215.
		1g	385.
83-62024	Sodium Pyruvate-3-^{13}C	0.25g	175.
		0.5g	300.
		1g	500.
83-62000	Urea-^{13}C	1g	185.
		5g	875.
		10g	1650.

[§]All ^{13}C-labelled compounds are a minimum 99 atom % ^{13}C unless otherwise stated.
[†]Please request prices for quantities that you do not see listed.
[‡]Prices are FOB Miamisburg, Ohio for delivery in North America; please request prices for delivery to the other continents.

3858 Benner Road • Miamisburg, Ohio 45342 U.S.A.
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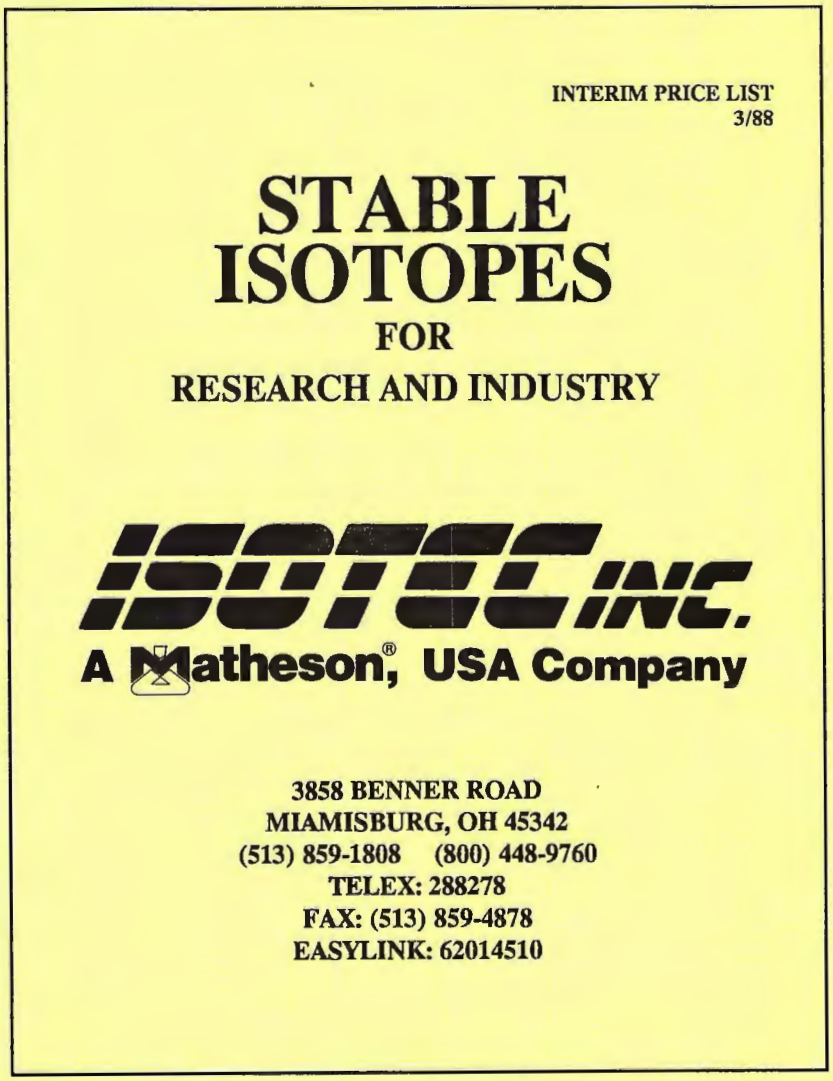
About Isotec Inc.

Isotec Inc. has been separating and enriching the noble gas stable isotopes, including ^{3}He, ^{21}Ne, ^{83}Kr, ^{129}Xe and ^{131}Xe, for approximately eight years. About five years ago, *Isotec Inc.* began the separation and enrichment of the stable isotopes of carbon, nitrogen and oxygen. Now we're making compounds (a few of which are listed on the other side of this page) from these raw materials (and deuterium).

Please let us know what you need—isotope, compound, quantity—we may already have it in stock; if not, we will be pleased to quote on your needs. Remember that there are significant cost advantages to producing one's own stable isotope enriched raw materials. Let us help you take advantage of our advantages.

We would also be interested in your requirements for enriched ^{17}O—desired enrichment, acceptable enrichment, preferred form and quantity. It's somewhere in our columns; we hope to be able to provide you with enriched ^{17}O within the year—your input now will help us do this more efficiently and economically. Please call or write Myra Gordon or Maureen Kalloo at *Isotec Inc.* with your wish list.

If you haven't received a copy of our Interim Price List, which looks something like the figure to the right, please ask us to send you one.



It lists hundreds more products (including all the noble gas stable isotopes, ^{15}N-and ^{18}O-labelled compounds, ^{12}C-, ^{14}N- and ^{16}O-labelled compounds and a large selection of metal isotopes, many of which have a nuclear spin) than are listed on the other side of this page—and more package sizes for many of those listed on the other side.

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Geadresseerde	Ons kenmerk	Datum	Blad No
Professor B.L. Shapiro	11332/WV/DvdW	1-7-1988 (received 7/13/88)	

Dear Professor Shapiro,

Solid state NMR imaging

For about a year we have been working on a ^1H NMR imaging technique for solids where use is made of line narrowing by magic angle spinning. When the NMR linewidth is sufficiently reduced, reasonably small field gradients can be employed with the promise of high resolution. Since the object under study is rotating several choices can be made for the field gradient (static, rotating synchronously, rotating asynchronously). Here we want to show some results from experiments where the field gradient rotates synchronously with the same frequency as the spinner.

Fig. 1 shows two images of polybutadiene in polybutadiene/polystyrene blends, blended in different ways. For polybutadiene MAS is sufficient to narrow the ^1H NMR line but this is of course not true for rigid solids. Fig. 2 shows the image of polystyrene powder forming a ring in the MAS rotor. Here in addition to MAS a MREV-8 pulse sequence is used to eliminate dipolar broadening.

Best regards,

Dave Cory Hanno de Boer Wiebren Veeman

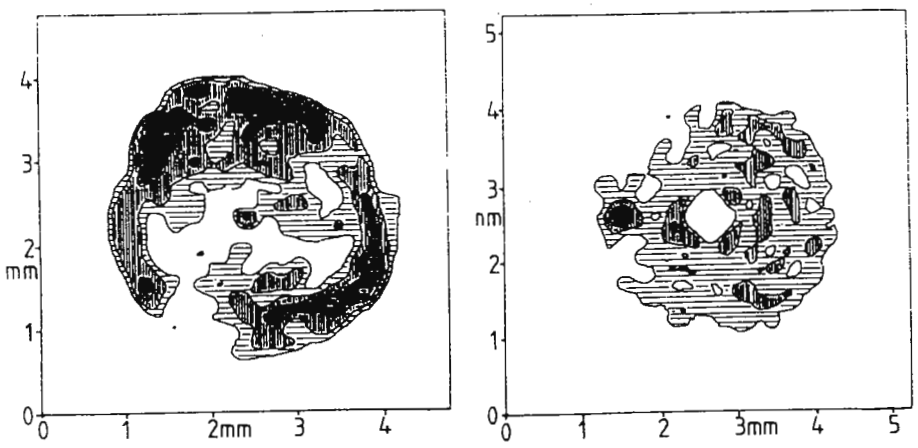

Fig. 1: ^1H NMR images of polybutadiene in polybutadiene/polystyrene blends obtained with MAS:

a) mechanically blended.
b) blended by coprecipitation from toluene.

Fig. 2: ^1H NMR image of polystyrene powder in a MAS spinner (i.d. 3.5 mm), obtained with MREV-8 and MAS.

Montana State University
Bozeman, Montana 59717

Department of Chemistry
College of Letters and Science

Telephone (406) 994-4801

July 12, 1988
(received 7/16/88)

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

Dear Barry,

Title: Attempts to optimize the efficiency of a "hands on" NMR facility by integrating it into a computer network.

Most of the articles in your newsletter deal with important experimental advances in NMR spectroscopy. Certainly, all of your readers wish they could spend all of their time engaged in that kind of work. However, most of us also have a major obligation to provide NMR services to others and we expend a great deal of effort trying either to fulfill or evade this responsibility. It is the purpose of this letter to describe our efforts to structure an NMR lab in a university setting to provide the most efficient and comprehensive NMR services. I also hope to stimulate information exchange on this subject from others.

For nearly ten years our NMR facility consisted solely of a broadbanded Bruker WM-250. Graduate students and a faculty member scheduled NMR time on a weekly basis. The instrument was scheduled seven days a week, 24 hours a day. All instrument operation was done by the individual users. This included VT operation and probe changes. Virtually all experiments were simple 1-D accumulations in part because there was insufficient time available for 2-D accumulations and processing. In upgrading our facility we were motivated to meet the following goals:

1. We wanted to be able to use our departmental computer network for offline processing and archiving.

2. We needed to have enough instrument time so that the individual users could experiment with more advanced techniques and become skilled at them.

3. We desired to use the computer network to carry out training on NMR data processing and more efficient use of the spectrometers.

The first two goals were achieved by purchasing an AC 300 carbon/hydrogen instrument and an AM 500 instrument which is

broadbanded and has MAS and wideline capability. The older 250 MHz instrument was upgraded so that its data system is essentially identical to the systems on the new instruments. All instruments are linked by a fiber optics communications system and linked to our microVAX network by ethernet. Because of a strong need for solids and wideline capabilities a Chemagnetics CMC 200 was added. The Chemagnetics instrument is interfaced to a microVAX which is on the ethernet. A large 4 x 8550 VAX cluster is being used for DePaking and spectral simulation. All four NMR machines are located adjacent to each other in an open room plan for enhanced information transfer among users and ease of servicing.

This configuration offers these opportunities:

1. Each console can be kept optimally busy with data accumulations but users are often able to find an NMR console for data processing.

2. The computer network has terminals in each office and laboratory so it is possible to monitor experiments conveniently and to be aware of instrument availability in a convenient fashion.

3. The computer network provides for more elaborate data processing and archiving without any interference with experiments.

4. Through the network, users in other departments, in other units of the university system, and in the technical park adjacent to the campus can have access to instruments, experiments, and data.

We are still in the instrument setup and shakedown phase of this project. While all the hardware is more or less in place, important decisions on network software have not been made. We believe that our network will offer better instrument use patterns and will present improved opportunities for instruction in the use of NMR spectrometers in data processing and in the interpretation of NMR data. We are aware of some other organizations which are interested in analogous networking ventures. With this letter we hope to stimulate exchanges of information and experiences which will facilitate development in this area.

Yours very truly,

Edwin H. ~~Abbott~~
Professor and Head

Edward A. Dratz
Professor

ENIRICERCHE S.p.A.
20097 San Donato Milanese, Italy

15 July 1988
(received 7/21/88)

PROTON FT-PGSE EXPERIMENTS OF MICROEMULSIONS

Dear Dr. Shapiro:

In the application of Fourier transform pulsed-gradient spin-echo (FT-PGSE) NMR to isotropic solutions such as microemulsions the usual goal is to obtain the self-diffusion coefficients (D) of all the distinct molecular species present using the frequency resolved NMR signals. This note is an observation made while performing experiments on a system composed of hexadecane, a nonionic surfactant C12H25-(OCH2CH2)4-OH, H2O, and a small amount of $N(CH_3)_4^+$ Cl-. Three of the four components should have resolved signals since the proton signals of the oil and of the aliphatic part of the surfactant have the same chemical shifts.

Samples of different oil and H2O composition were studied by PGSE, on a JEOL FX-60 NMR spectrometer at Lund University, as a function of temperature as before (1). It became evident that the only visible signals of the surfactant were those arising from the ethylene oxide protons. The signals from the relatively immobile protons of the amphiphile, those of the hydrophobic portion, were absent. Selection of a gradient duration long enough to reveal only the signals of the slowest diffusing molecule illustrates the point (Figure 1). Elimination of the overlapping surfactant proton signal was fortuitous. The D-oil was obtained directly without the need for application of a biexponential decay function to a composite peak.

In the case of a microemulsion composed of C12H26/Na^+ $C_{12}H_{25}C_6H_4SO_3^-$/H_3O^+ the only observable proton signal of the amphiphile was that of the methyl group. The portion of the amphiphilic molecule which was relatively rigid was not detected due to efficient T2 relaxation.

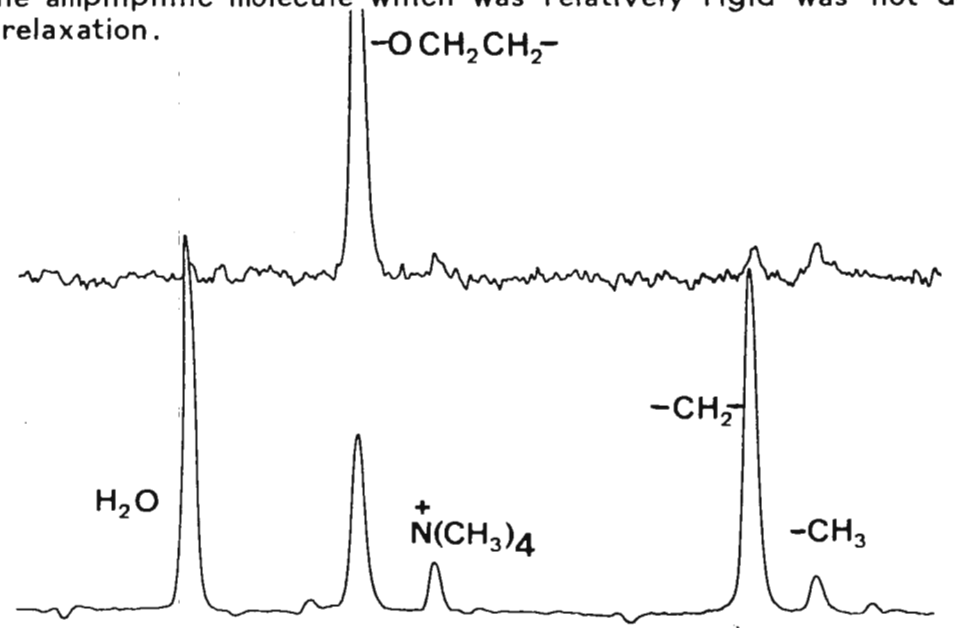

Figure 1. Proton PGSE NMR spectra of a microemulsion composed of C16H34/ C12H25-(OCH2CH2)4-OH/ H2O/ $N(CH_3)_4^+$ Cl- (by weight 32:5:8:1) at 32°C measured at 60MHz as before (1). Gradient strength was ca. 4G/cm with durations of 24ms (bottom trace) and 45ms (top trace). The interval between gradient pulses was 140ms.

Please credit this to Carlo Corno's account. Sincerely,

Wallace O. Parker, Jr.
Eniricerche S.p.A.

Mikael Jonstromer
University of Lund, Sweden

(1) U. Olsson, K. Shinoda, B. Lindman, J.Phys.Chem., 90, 4083 (1986).

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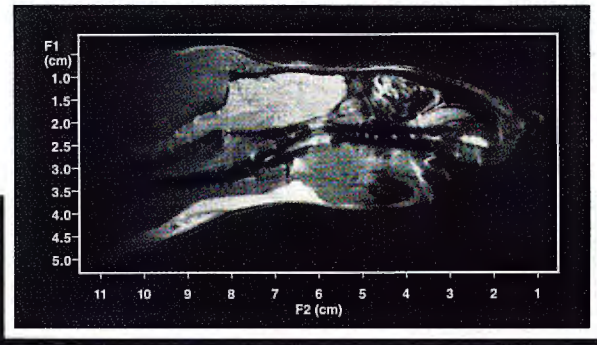

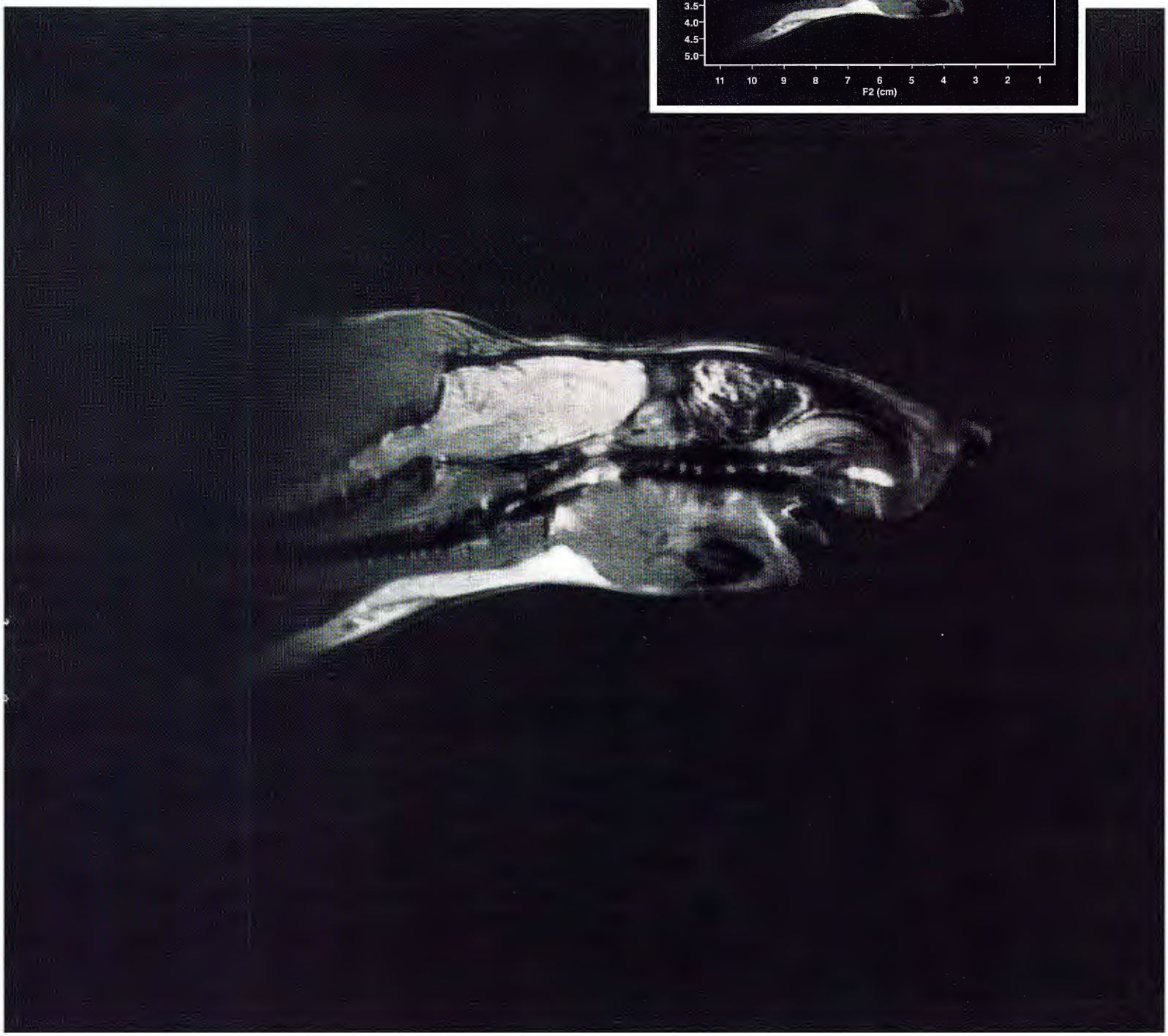

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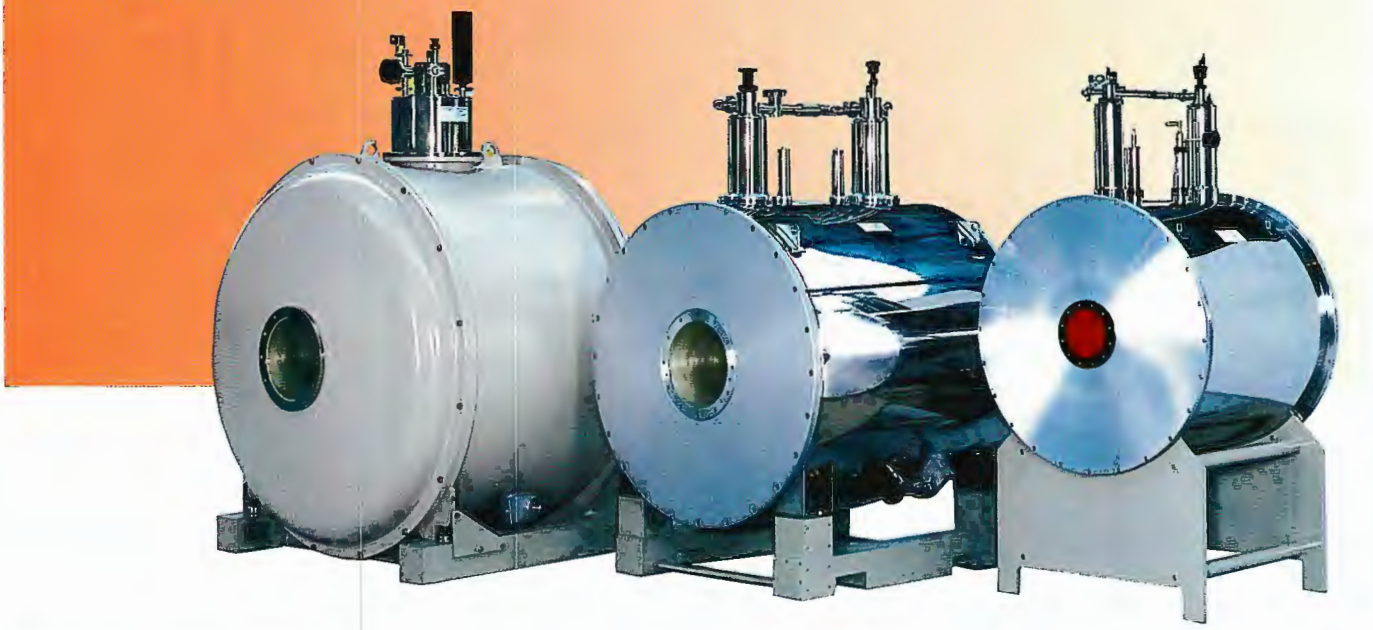
Magnet	Center Field	Magnet Bore	Clear Bore	Maximum Gradient	Plotted Homogeneity	HHLW Resolution	5 Gauss On-Axis	5 Gauss On-Radius
300/180	7.05T	183 mm	125 mm	4.0 G/cm	80 mm DSV ±6 ppm	35 mm DSV 0.1 ppm	5.60 m	4.45 m
200/330	4.7T	330 mm	254 mm	2.3 G/cm	140 mm DSV ±5 ppm	70 mm DSV 0.1 ppm	6.95 m	5.60 m
200/400	4.7T	400 mm	324 mm	1.8 G/cm	140 mm DSV ±4 ppm	80 mm DSV 0.1 ppm	8.50 m	6.75 m
85/310	2.0T	310 mm	225 mm	3.0 G/cm	100 mm DSV ±5 ppm	70 mm DSV 0.1 ppm	4.50 m	3.63 m

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



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

Spectroscopy Imaging Systems
1120 Auburn Road
Fremont, California 94538
(415) 659-2600

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

Duke University

NUCLEAR MAGNETIC RESONANCE SPECTROSCOPY CENTER

LEONARD D. SPICER, DIRECTOR
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(919) 684-6287

July 17, 1988
(received 7/25/88)

Professor Barry Shapiro
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto, CA 94403

Update on Changing Zeta 8 Pen Speed
"It Pays to Read the User's Manual #2"

Dear Professor Shapiro:

Last year, in TAMU 346-23, Jim Loo outlined a "trained monkey" procedure to slow down the Zeta 8 pen plotter speed to prevent "pen skipping" when recording narrow NMR lines. Jim's instructions read something like the following:

1. Press the CLEAR button.
2. Press the DOT (Fast) control and PAUSE buttons at the same time. This puts one in the Parameter Mode Select (PMS) mode.
3. Use the PEN SELECT button to select a pen number corresponding to the first digit of a function number. Press RETURN to code in this digit.
4. Move the PEN SELECT button to the pen number corresponding to the second digit of the FUNCTION number. Press RETURN again to code in this digit.
5. Press the DOT (Fast) Control and PAUSE buttons at the same time. The Zeta will then respond to the function coded.
6. The CLEAR button restores the Zeta to "normal" settings.

In Jim's letter, the function #38 codes for full normal Zeta speed, while the functions #37 through #31 decreases the plotter speed by 1/8th factors. Thus to code for 50% speed, the function #34 is used.

We tried Jim's function codes on the Zeta 8 and 836 plotters which were installed with our GN NMR systems and found that they did not work. However, the Zeta 8 which was installed with the XL system responded exactly as Jim described. A check of the Zeta manual which accompanied the XL revealed the PMS function codes for speed control to indeed be 31 to 38. The Zeta manuals which accompanied the GN systems instead list only PMS function code 14 to slow the pen speed, while #'s 31-34 now define other functions.

We found that programming in function code 14 gave about 50% slower plot speeds on the Zetas with the GN systems. Apparently Zeta has used different PROM (programmable read only memory) chips in its early and later version of

BOX 3711, DUKE UNIVERSITY MEDICAL CENTER
DURHAM, NC 27710

the Zeta plotter. The user's manual with your plotter should provide a quick reference as to which version of the plotter you have in your laboratory.

Sincerely,

Leonard D. Spicer Donald E. Mika Anthony A. Ribeiro

EMORY UNIVERSITY
Department of Chemistry

1515 Pierce Drive
Atlanta, Georgia 30322
404/727-6585

July 18, 1988
(received 7/21/88)

Prof. Barry Shapiro
Editor, TAMU NMR Newsletter
966 Elsinore Ct.
Palo Alto, CA 94303

Dear Barry:

Status Indicator for Broad Band Decoupler on GN Spectrometers

We recently received a GN-500 spectrometer with a broad band decoupler unit needed to carry out proton detected heteronuclear experiments. Although the instrument is performing very well for these experiments, one shortcoming is that there is no indicator of the status of the BB decoupler. As it turns out, this deficiency can be easily rectified. Inspection of the circuit diagram for the BB decoupler control shows that there are outputs for driving LEDs to monitor RF on, modulation on, and 90 and 180 degree phase shifts. These lines make their way from the control module to connector J7 in the mother board for the "8-slot" RF unit. The circuit diagram shows pins 28 to 31 respectively have the LED drivers connected to them, but, as is indicated in the circuit, these are not connected to anything further down the line. What we have done is simply connect 4 leads of a multiconductor cable to these pins on J7 and a fifth to the 5V line, and run the cable out to a box in which we have installed 4 garden variety LEDs. The 5V is connected to the + side of the LED and the LED output lines to the other side. This provides a suitable indicator.

Sincerely

David Live

P.S. We have a working Bruker 18" magnet for an HFX 90 as well as a console in questionable condition that we will shortly have to dispose of. These will unfortunately have to be consigned to the junk pile unless we can find someone who wants to take them.

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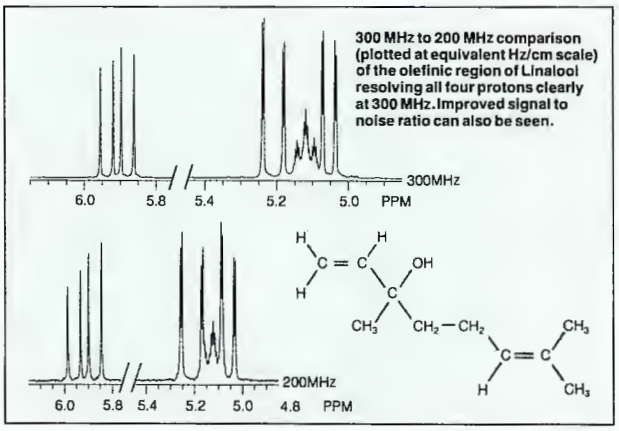

300 MHz to 200 MHz comparison (plotted at equivalent Hz/cm scale) of the olefinic region of Linalool resolving all four protons clearly at 300 MHz. Improved signal to noise ratio can also be seen.

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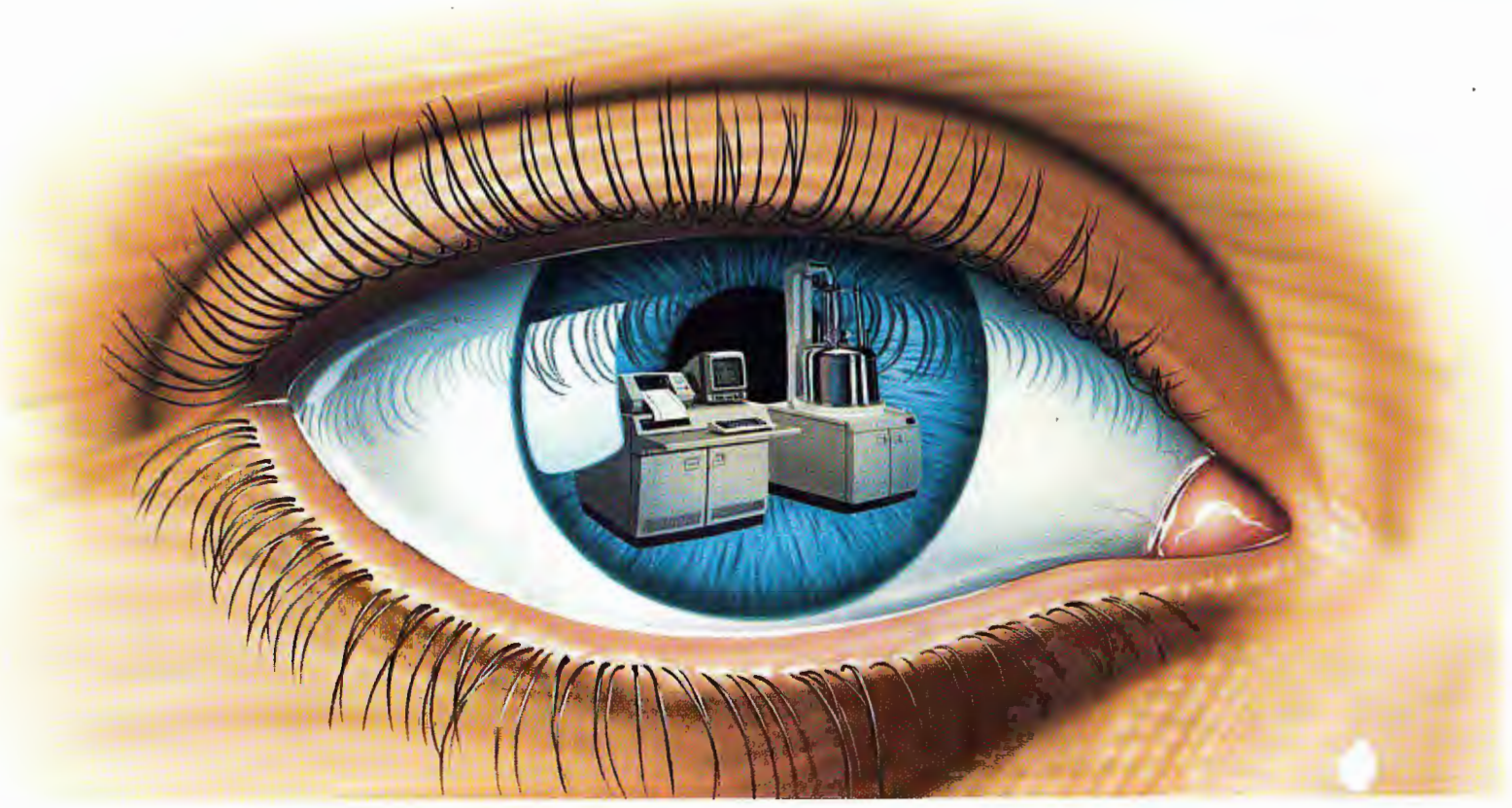

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Department of Medicinal Chemistry and Pharmacognosy

July 17, 1988
(received 7/20/88)

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

RUNNING 2D HOMONUCLEAR HARTMANN-HAHN (HOHAHA) EXPERIMENTS ON NT-SPECTROMETERS

In preparing this contribution, we thought you'd appreciate the continued usage of the format we introduced with our last contribution. Your quote from Shaw, "If you face is dirty wash it, don't cut your head off..." was well taken. In retrospect we did perhaps go overboard. Hence, we have resumed the use of paragraph breaks and indentations! We'll have to think of an alternative means of recovering the space that this wastes, not to mention that wasted by this paragraph!

More seriously, a number of methods are being developed for transferring magnetization beyond the vicinal homo- or heteronuclear coupling partner. One of the first such experiments to appear was the homonuclear relayed coherence transfer or RELAY experiment developed by Eich, Bodenhausen and Ernst.[1] While it is in principle possible to transfer magnetization across several bonds using single and double relay experiments, fixed delays must be optimized to accomplish this task. Bax and Drobny[2] have considered the optimization of the fixed delays in rather considerable detail; it may not always be possible to find a single value which provides all of the information required. Perhaps as a result of this drawback and other reasons, a group of experiments which rely on isotropic mixing are rapidly gaining in prominence.

There are a growing number of experiments which utilize isotropic mixing sequences to transfer coherence. These include: CAMELSPIN[3] or ROESY[4] for rotating frame NOE; TOCSY[5] and HOHAHA[6] for transfer of scalar coupling information. In the latter, Waugh[7] has shown that once Zeeman contributions are removed that the spin system will evolve under the influence of the scalar coupling constant, J. Thus, as the duration of the mixing interval increases, magnetization will propogate progressively further through a given network of coupled spins.

Quite obviously, the trick is to get these very useful and interesting experiments to run on "vintage" but dependable spectrometers such as the NT-300 in our lab. HOHAHA experiments cannot be run on an NT-series instrument using the conventional proton pulse power amplifier. The proton power amplifier cannot deliver pulses for the protracted periods necessary in these experiments. An alternative which we have found satisfactory is to use the proton decoupler amplifier. There are, however, several minor modifications which must be made to run the HOHAHA experiment. First, the pulse sequence, which is shown schematically in Figure 1, must be written so that decoupler pulses are delivered using P2/P1 commands in the pulse sequence. The 293-C pulse program can be obtained from us upon request. When the HOHAHA experiment is run, the instrument should be set for the low power transmitter using the TL command. We also routinely dial in 70dB of attenuation using the front console attenuators beneath the shim power supply although this is probably not absolutely necessary.

Next, on systems where MLEV proton decoupling is not done for heteronuclear experiments, it is necessary to connect the conditional jump lead on the back of the pulse programmer. Instructions for this connection are given in the NMC-1280 manual. Minor recableing is also necesary in the form of cross connecting the decoupler lead to the transmitter coupler input (J-904).

Third, it is necessary to disable the final decade of resolution control on the decoupler, limiting it to ±1 Hz resolution. This is done by lifting pins **12, 14, 16, and 18** on IC-27 in the pulse programmer (refer to Schematic No. 107398 for the location of IC-27). Next, it is necessary to set the decoupler frequency (F_2) and the receiver (SF) to exactly the same frequency. A one pulse spectrum should be obtained by writing a simple pulse program to generate the observe pulses from the decoupler. If the FID builds coherently and successive 1D spectra have exactly the same phase the instrument is basically ready to use. If there are phase anamolies from one spectrum to the next, shift the F_2 and SF frequencies by the same amount and try again. Usually a frequency within a few Hz will afford a usable matach. Finally, it should be recalled that the decoupler amplifier is a Class C amplifier and will still have some residual output when not actually running. This is

problemsome in that it puts a regular noise pattern in the data collected using the HOHAHA expeirment. A spectrum recorded using the pulse sequence shown in Figure 1 is presented in Figure 2 above the diagonal and clearly illustrates the regular, "grid-like" noise pattern. This is a rather irritating and expensive way of generating graph paper. Fortunately, however, the residual power output of the decoupler can be taken care of by using a pair of back-to-back diodes in line between the decoupler and J-904. We found it most convenient to do this using a small Pomona box with male and female BNC connectors to allow the diode box to be connected in line quite easily. Finally, we have found it beneficial to insert a Minicircuits BHP-150 high pass filter in line between the trans-coupler and the preamp. At this point, the instrument is set up for pulse calibration and data acquisition.

Prior to continuing, a note about pulse calibration is in order. Specifically, we have found that pulse widths should be calibrated and the probe tuned for each sample if the best data are to acquired using this technique.

Following the calibration of the 180° pulse, parameters can be entered into the pulse sequence shown in Figure 1. The spectra shown in Figure 2 were acquired using 10 mg of strychnine and were recorded in just under 3 hrs with 384 x 1K data points using the pulse sequence shown in Figure 1. Trim pulses consisting of 2.5 msec were used in all cases. The mixing times in Figure 2 were set to 119 msec. Note that the "grid-like" noise has been eliminated by the inclusion of the back-to-back diodes and the BHP-150 filter in the section of the spectrum shown in Figure 2 below the diagonal. The data were processed using sinusoidal multiplication prior to both Fourier transformations with zero filling to 512 points prior to the second to afford a final matrix of 512 x 512 points. The data was also magnitude calculated prior to plotting. For comparison purposes, the results shown in Figure 2 beneath the diagonal were compared to a phase sensitive HOHAHA spectrum recorded on a GN-500 graciously provided to us by Dr. B.K. John of GE NMR Instruments. The results presented in the two spectra were quite comparable despite the fact that the data presented in Figure 2 are magnitude calculated.

We have found the results obtained with the HOHAHA experiment to be very beneficial in the assignment of the proton NMR spectra of several alkaloids and for the determination of long range coupling pathways in polynuclear aromatic compounds.[8] Doubtless many other useful applications of the experiment will be found as additional groups gain access to the technique.

Gary E. Martin Larry D. Sims Luis R. Soltero

Milton D. Johnston, Jr.
Univ. of South Florida

Andrew S. Zekzter
Abbott Laboratories

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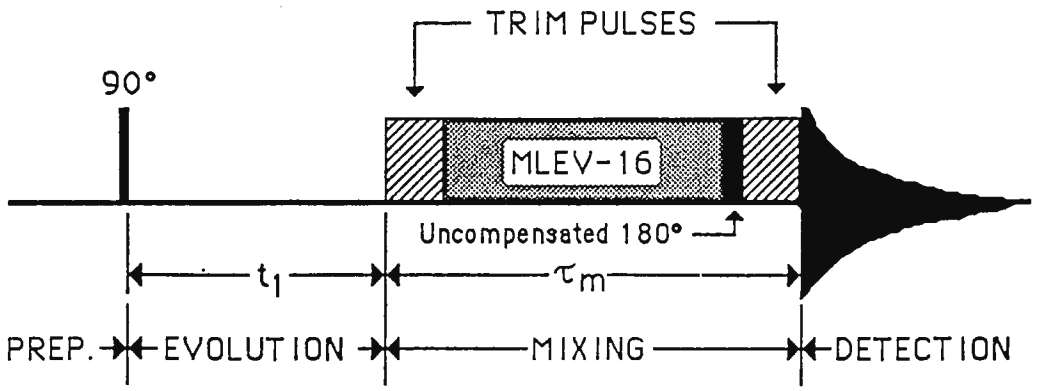

Figure 1. HOHAHA pulse sequence employing an MLEV-17 sequence flanked by trim pulses. The uncompensated 180° pulse refocuses errors due to phase shifter inaccuracies after an even number of cycles. The trim pulses were normally set to 2.5 msec and serve to defocus magnetization components not coaligned with the ±x axis.

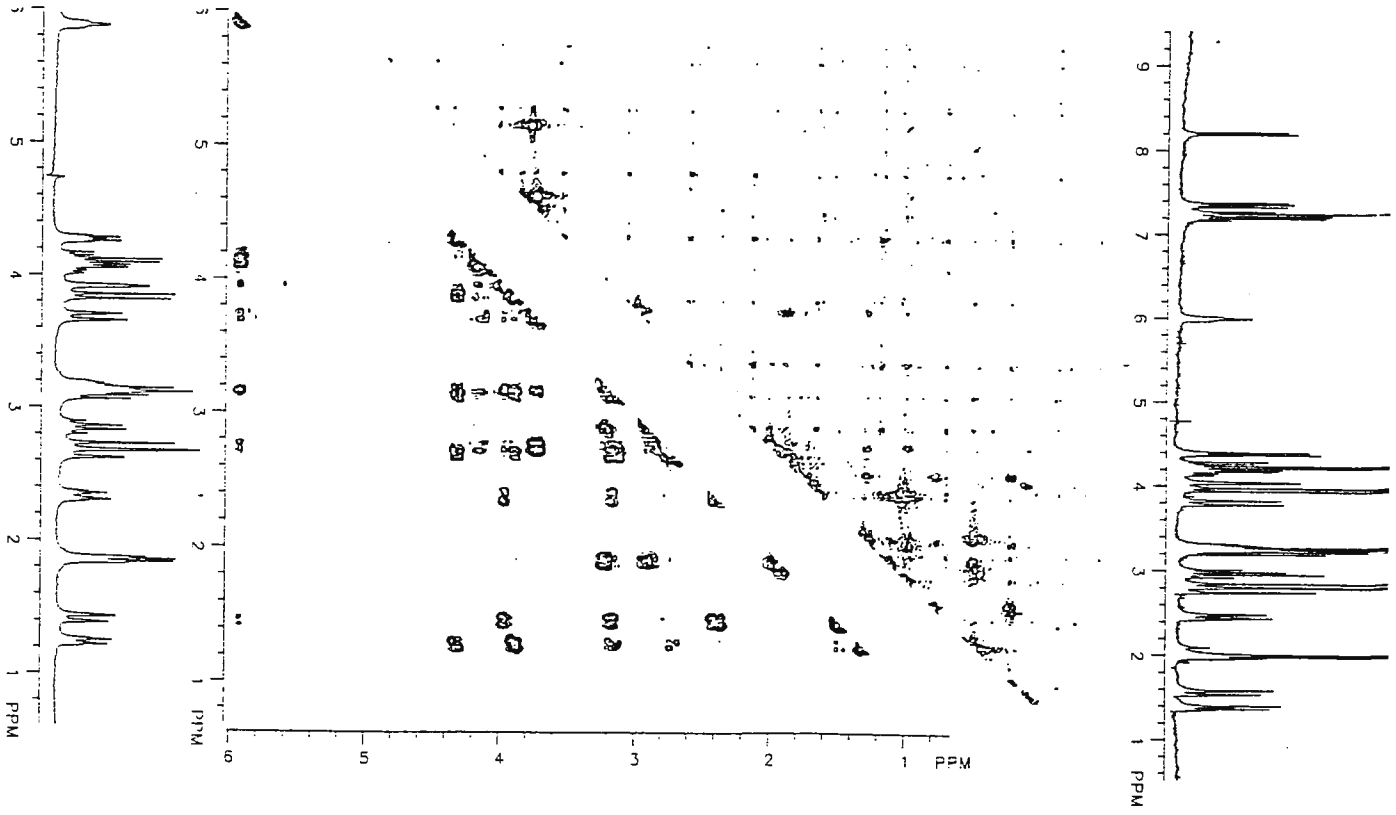

Figure 2. 300 MHz HOHAHA spectra of strychnine. The spectrum shown above the diagonal was recorded using the pulse sequence shown in Fig. 1 without the back-to-back diodes -- note the "grid-like" regular noise pattern in the spectrum. The spectrum shown below the diagonal was recorded under identical conditions except that the back-to-back diodes were in line between the decoupler and connector J904 (see text). Here it should be noted that the noise in the spectrum above the diagonal is completely eliminated providing a much more usable spectrum.

Amoco Corporation

Amoco Research Center
Post Office Box 400
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312-420-5111

July 15, 1988
(received 7/22/88)

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

Dear Dr. Shapiro:

Remote Monitoring System for an Automated Varian VXR Spectrometer

We have recently completed the design and debugging of a remote monitoring system for our automated VXR 300 spectrometer. This spectrometer is equipped with the ASM 100 autosampler and is used for the routine operation of our proton and carbon NMR service at the Research Center. As such, it is required to operate 24 hours a day, 7 days a week without failure. However, the spectrometer occasionally does fail, due to the sensitivity of the VXR 4000 host computer to any static discharge in the vicinity of the instrument. This causes the computer to stop and requires a reboot to be performed. This is a real problem in the automation mode when the system is left unattended, and the spectrometer fails after the operators have gone home. The system we have designed allows us to monitor the status of the spectrometer from any remote location which has a computer and a modem. Also, we can remotely reboot the spectrometer using a software command and return the spectrometer to the original automation mode.

The system is designed around an IBM PC AT computer, which is the link between the remote computer and the VXR host computer. The PC is configured to emulate a Televideo 920 terminal using the software program "Crosstalk" and is connected to the VXR 4000 host computer Diagnostic/Modem port (J212) via RS-232 cable. The configuration of the VXR to PC interface is described in Figure 1. The off-site access to the system is accomplished through the use of the software package "Carbon Copy" from Meridian Technology. "Carbon Copy" is a communication program that allows one PC to call another PC and have control over the operating system of the computer that was called. "Carbon Copy" has the security feature that requires a password before entry is allowed to the system. The complete hardware configuration is shown in Figure 2. Once the connection is established, commands typed on the PC will be sent to the VXR. The Varian remote control software is now activated by typing the REMOTE(DON) command and the VXR display is transferred to the PC and emulation is now possible. In this way, we are now able to check the status of the VXR from any location that has an IBM compatible PC, Crosstalk and Carbon Copy software, and a modem.

This system currently has several limitations which include: graphics characters cannot be transferred to the remote display; routines that require the use of the spectrometer knobs are not functional; and the bottom 7 lines of display are not visible on the remote terminal due to the difference between the number of lines the remote terminal will support and the number available on the Varian monitor. The function keys on the remote keyboard, i.e., F1, etc., are not operable but can be emulated by using the forward slash key "/" and the number key followed by a return e.g. /1 'return' = F1.

VARIAN
INSTRUMENTS
AT WORK

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RESONANCE

MARCH 1988

NMR-32

VXR-600: PRELIMINARY RESULTS

Varian recently announced the availability of the newest member of our spectrometer line, the VXR-600. Using an Oxford Instruments 600 MHz magnet, the VXR Series S console with its ultra-stable rf, and the VXR-5000 data system, the VXR-600 is a state-of-the-art instrument.

Preliminary results with the first VXR-600 indicate that the magnet-related parameters (resolution, lineshape, ease of shimming, overnight stability) are all quite good. The spectra on these pages, while not illustrating "specs", show that the VXR-600 is capable today of producing quality results of the type for which the VXR-600 is being purchased: two-dimensional nmr studies on molecules of biological interest, and high-field solid-state nmr applications including wideline ^2H.

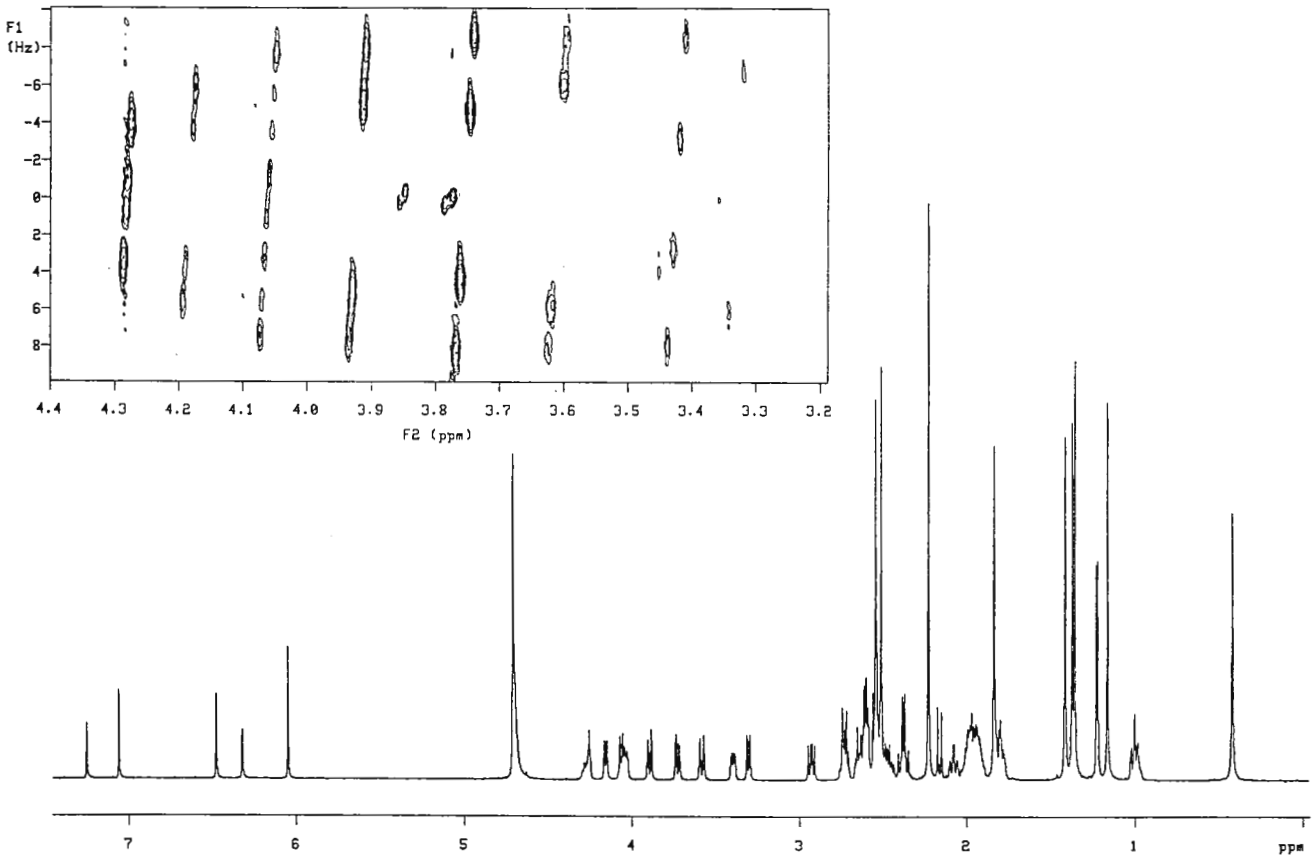


Figure 1: 1-dimensional spectrum (bottom) and an expansion from the homonuclear 2D-J spectrum (inset) of Vitamin B-12, 10 mmolar in D_2O. The 2D-J spectrum is unrotated.

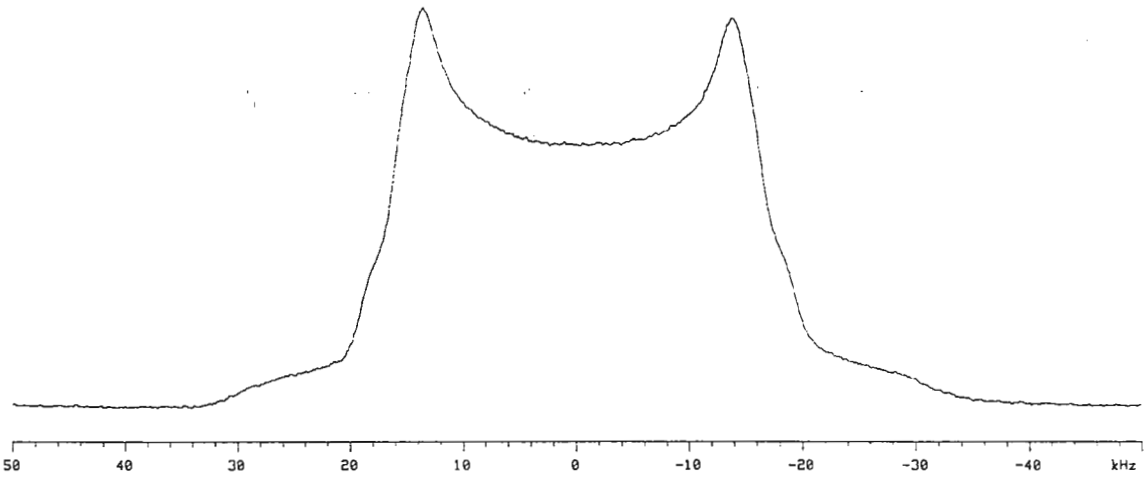


Figure 2: Wideline ^2H spectrum of methionine-d_3. The shoulders at ±18 kHz (approx.) are not seen at lower fields.

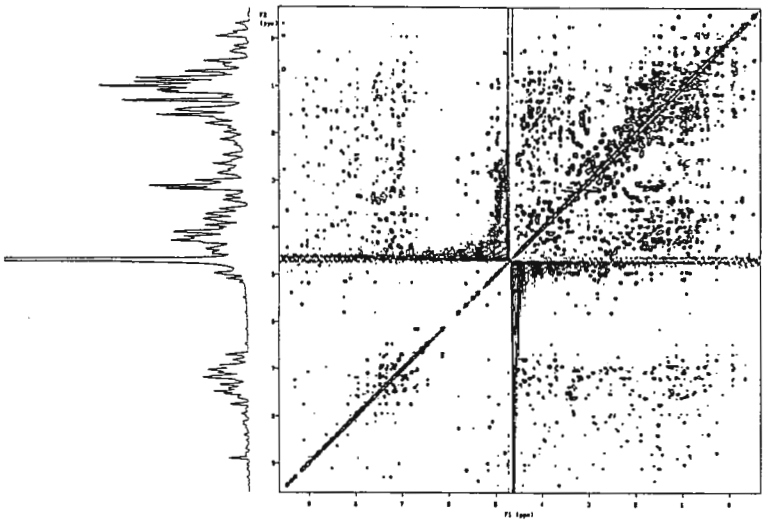
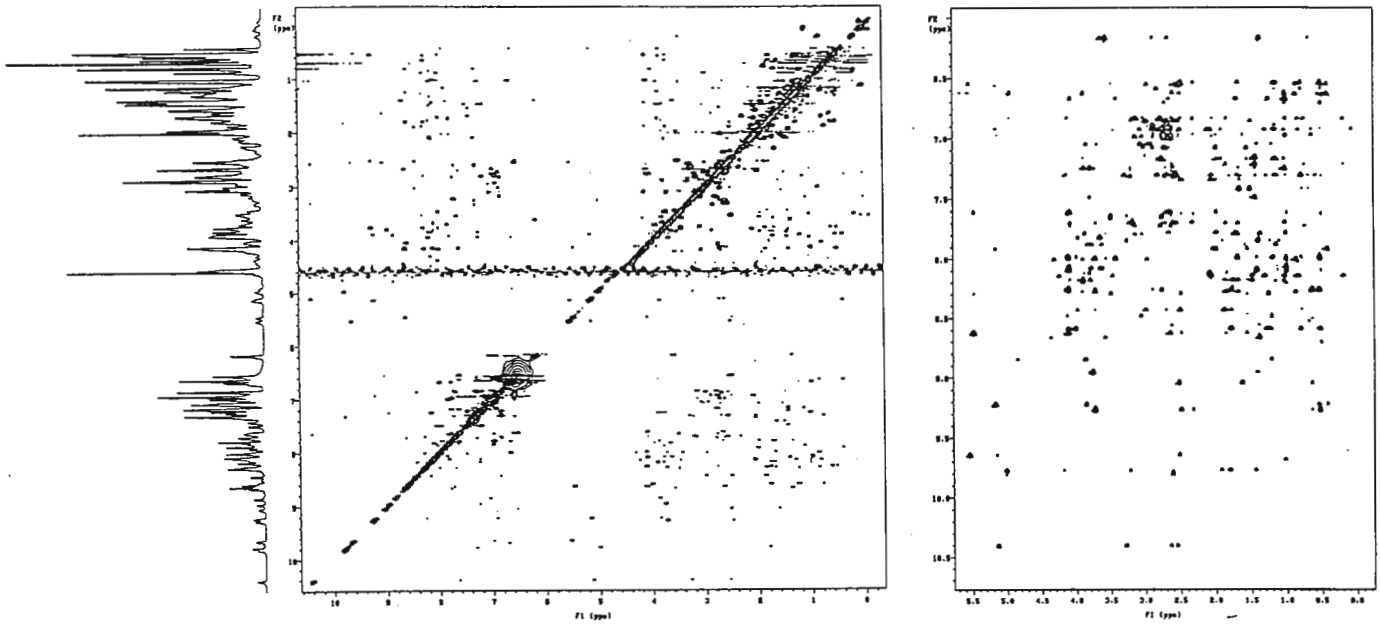


Figure 3 (left): Phase-sensitive 2-dimensional NOE (NOESY) spectrum of lysozyme, approximately 5 mmolar in D_2O, overnight accumulation. No presaturation or other water elimination was used whatsoever. The spectrum is unsymmetrized. The projection of the data on the F2 axis is also shown.

Figure 4 (below): Phase-sensitive 2-dimensional NOE (NOESY) spectrum of BPTI, approximately 4 mmolar in H_2O, overnight accumulation with water presaturation. The spectrum is unsymmetrized. (left) Full spectrum, along with the projection of the data on the F2 axis. (right) Expansion of the fingerprint region at a slightly higher vertical scale.



The most essential feature of this remote operation system allows us to reboot the VXR computer using a software command from the remote station. This required the slight modification of the CPU control board on the VXR 4000 computer to accept an additional input to the reset circuitry, as shown in Figure 3. One of the data lines from the PC parallel port is connected to this input and is activated by an assembly language program that momentarily sends the line low to reboot the VXR. The program will be submitted for publication in the VARIAN newsletter and can be obtained from one of us upon request.

R. W. Dunlap, F-9
312-420-5154

J. J. Klein, B-5
312-961-7966

RWD\TAMU88\jh

Figure 1: VXR to PC Configuration

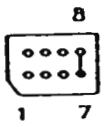
A) RS232 Cable for IBM AT (and PC) to the VXR Modem Port

IBM AT (PC)	RS232 Signal Line	VXR Modem Port
1 (8)	Carrier Detect DCD	8
2 (3)	Receive RCV	3
3 (2)	Transmit Data XMIT	2
4 (20)	Data Terminal Ready DTR	20
5 (7)	Signal Ground	7
6 (6)	Data Set Ready DSR	6
7 (4)	Request To Send RTS	4
8 (5)	Clear To Send CTS	5
9 (N/A)	Ring Indicator RI	22

B) Crosstalk Parameters for Communication with the VXR:

Speed — 9600 baud
Data Bits — 8
Stop Bits — 1
Parity — None
Duplex — Full
Emulate — TVI920
Mode — Call

C) VXR CPU Board Baud Rate Jumper
J21 (Port1) Set for 9600 Baud



359-24 Figure 2: Hardware Configuration

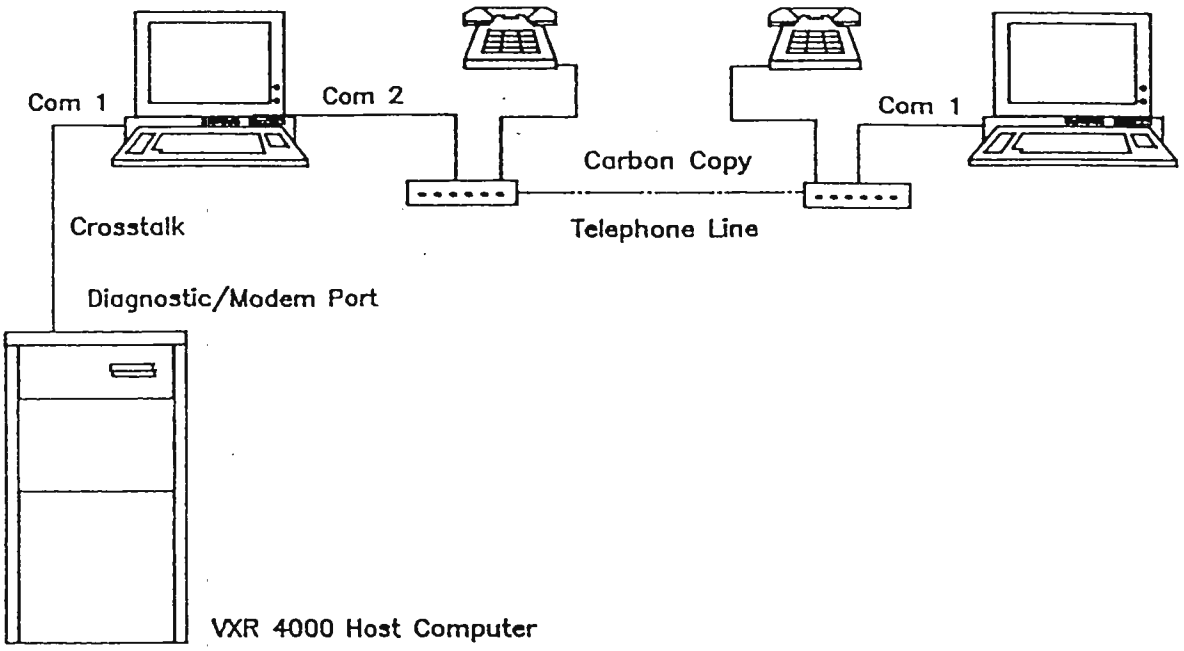
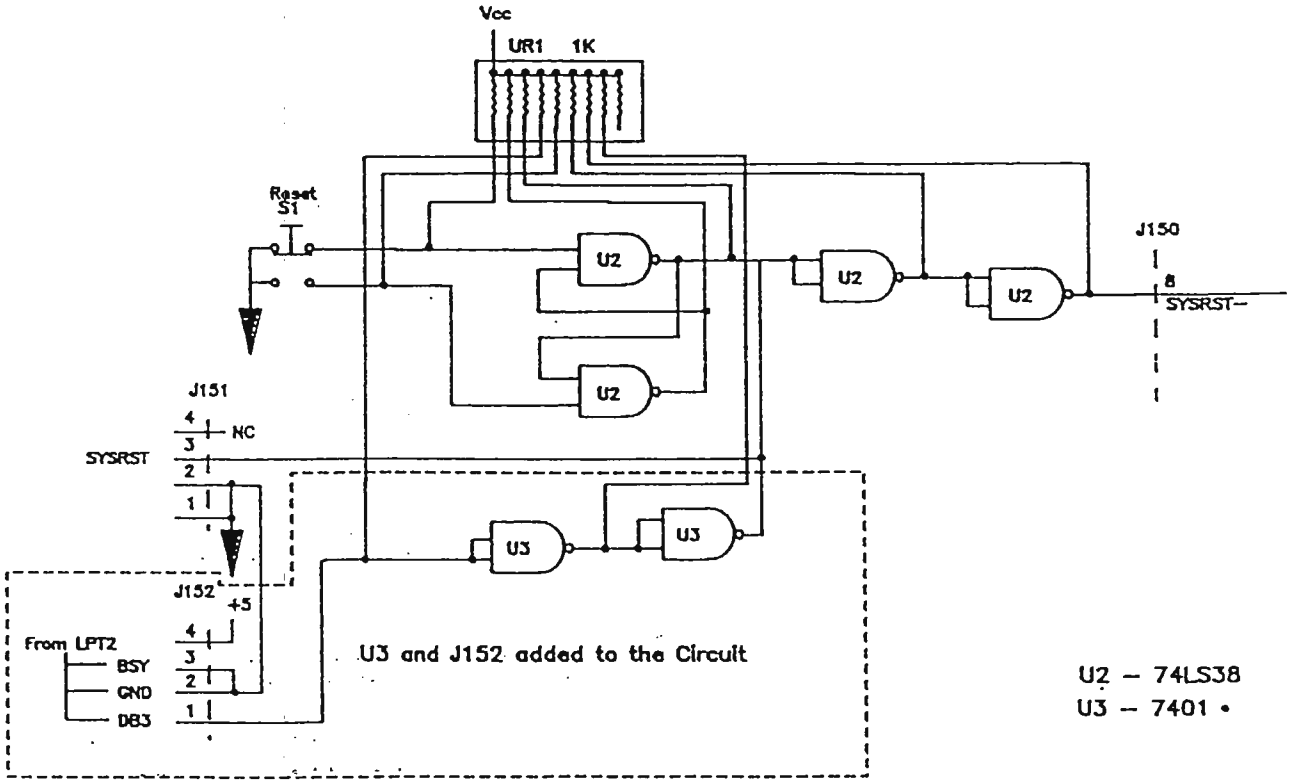


Figure 3: Modified Reset Circuit on CPU Control Board



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Doctor Muriel DELEPIERRE
Département de Chimie Organique
U 266 INSERM, UA 498 CNRS
UER des Sciences Pharmaceutiques et Biologiques
4 avenue de l'Observatoire
75006 PARIS - France.

UNIVERSIDAD DE BUENOS AIRES
FACULTAD DE CIENCIAS EXACTAS Y NATURALES

Buenos Aires, july 11,1988.
(received 7/25/88)

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

Dear Dr. Shapiro:

Since in our lab we are still using CW instruments (60 and 100 MHz) we quite naturally suffer from lack of sensitivity.Some time ago we found that CORRELATION SPECTROSCOPY (J.Dadok & R.F.Sprecher,J.Magn.Res. 13,243,(1974) and R.K.Gupta et al.,ibid 275,(1974) could be a faster cure than modifying our two spectrometers for F.T. Programs for this spectroscopy were already available from the Nicolet Users Society,(NUS-620).

The method works.It has its advantages.It can be used on almost any CW spectrometer.It allows a limited range to be swept giving very good resolution with nearly the same improvement in sensitivity (ca.80%) as F.T. and it does not suffer from solvent signal problems.But it requires a very good sweeper, with well defined initial and final frequencies and a very linear sweep.

A digital sweeper which meets these requirements was proposed some time ago by S.K.Kan (Rev.Sci.Instr. 44,1725, (1973)) but,according to its author,it suffered from some imperfections like a slight baseline ondulation and phase noise.

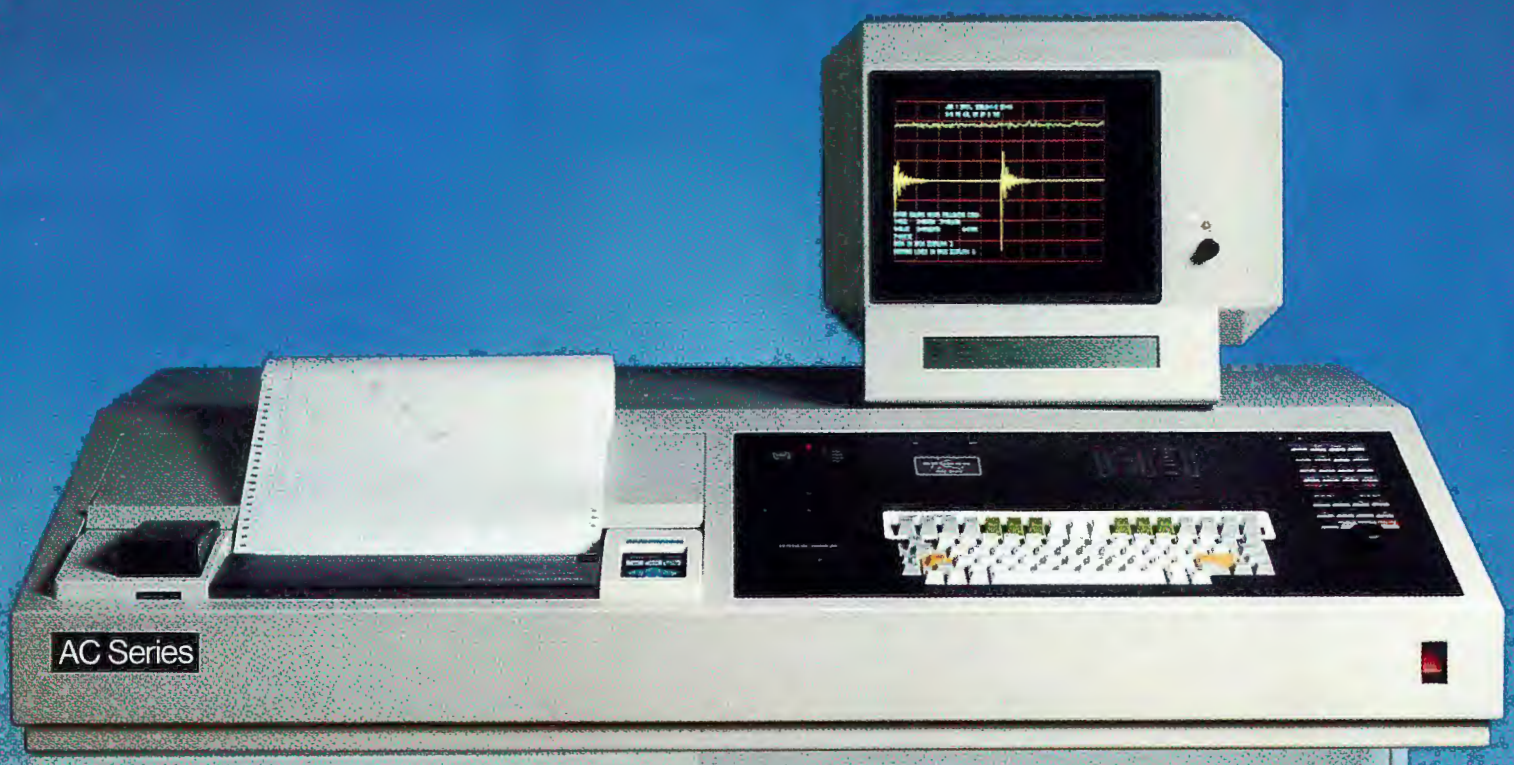
We have been able in our lab to solve these problems and a prototype,working in the audio range,has its initial and final frequencies defined to within .01 Hz and stable to within one ppm.It sweeps in .01 Hz increments and is absolutely linear. It can be swept either manually or by the computer for as many times as the resolution allows.

The only drawback we found was that,being a TTL device,it produces some R.F. interference which shows in some spectra as beats.But with generous decoupling and filtering we hope to be able to solve this problem in the new versions of the instrument we are building and to have it published somewhere.

Yours, sincerily.

V.J.Kowalewski.

Now, Bruker makes research routine.



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Spectrometer Performance — AC Series

Summary of Specifications for Basic System Configurations AC Series 100, 200, 250, 300 MHz

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^{1}H	^{1}H	0.1% EB	<10	35	85	120	175
$^{1}H/^{13}C$	^{1}H	0.1% EB	<15	20	45	70	90
	^{13}C	ASTM	<15	20	50	70	100
		10% EB		15	40	50	70
QNP	^{1}H	0.1% EB	<15	20	45	70	90
	^{19}F	0.05% TFT	<20	20	45	70	100
	^{31}P	48.5 mM TPP	<20	25	50	60	70
	^{13}C	ASTM	<15	20	50	70	100
		10% EB		15	40	50	70
VSP multinuc. ^{31}P–^{15}N	^{1}H	0.1% EB	<15	20	40	60	80
	^{31}P	48.5 mM TPP	<15	15	30	35	40
	^{13}C	ASTM	<15	20	50	70	100
		10% EB		15	40	50	70
	^{15}N	90% Form.	<25		5	8	10
			SSB %	**Lineshape (Hz)**			
All Probes (5 mm)	^{1}H	$CHCl_3$	<1	7/15	7/15	7/15	7/15
	^{13}C	C_6H_6	<0.5	3/7	3/7	3/7	3/7
				Resolution (Hz)			
All Probes (5 mm)	^{1}H	ODCB		0.2	0.2	0.2	0.2
	^{13}C	C_6H_6		0.2	0.2	0.2	0.2

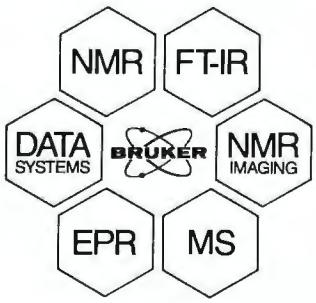
Magnet:	Helium refill interval (days)			
Standard	120	120	120	120
Long hold time	200	200	190	180
Ultra-long hold time	365	365	350	340

EB = ethylbenzene (for ^{13}C with ^{1}H-dec.); ASTM=60% C_6D_6 in dioxane;
TFT=trifluorotoluene; TPP=triphenylphosphate; Form.=formamide (^{1}H-dec. without NOE);
SSB=spinning sidebands;
Lineshape: ^{1}H=$CHCl_3$ linewidth at ht. of ^{13}C-satellites/at 20% this level
^{13}C=C_6H_6 linewidth at 0.55%/0.11% level (^{1}H-dec.).
Dimensions and weights are approximate; voltage+10/–5% max. variation, otherwise power stabilizer required; other line freq. or voltage upon request.

	Standard	Options
Transmitter Freq. range	$^{1}H/^{13}C \pm 100$ kHz	full multinuc.[2]
Offset steps	100 Hz	0.01 Hz[1,2]
Phase shift steps	90°	<0.5°[1,2]
^{1}H-decoupler Freq. range	150 kHz	
Offset steps	0.01 Hz	
Phase shift steps	90°	<0.5°[1,2]
Max. power	5 W	40W[2]
Attenuator range	60 dB	80 dB[2]
Quad. receiver Spectral width	100 Hz – 100 kHz (cont.)	125 kHz[2], 50 kHz
Filter width (100 Hz Steps)	100 Hz – 100 kHz	100 Hz – 100 kHz; 150 kHz[2]
ADC	12-bit	16-bit
Computer Memory	256 K / 24-bit	up to 1280 K / 24-bit
Max. FT size	128 K words	512 K words
Multi-pulse timing resol.	0.1 μsec	0.025 μsec[2]
FT time (software)	1 K/1.5 sec	
Co-processor		8 K/1.5 sec[2]
Array processor		64 K/1.2 sec
Graphic display: colors	8	8
Pixel matrix	256 x 512	256 x 512
Plotter	A3 flat bed 8 colors	drum[2], HP with auto-feed
Printer	dot-matrix	
Storage	160 MByte disk, floppy	tape
Communication	RS 232	KERMET/ETHERNET® Fiber optic LAN

1. Standard with the AC 300E 2. Standard for AC-P version

Nitrogen refill interval (days)	Minimal room height (cm)	Weight approx. (kg)
9	276	250
10	263	220
14	297	250
Console	w 126 x d 85 x h 91 cm	
Height with display	123 cm	
Weight approx.	300 kg	
Power consumption	220 V/50 Hz/3 kW	



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Département de chimie

July 18, 1988
(received 7/25/88)

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

Getting rid of paramagnetic ions

Dear Dr. Shapiro:

We are presently conducting ^{31}P-NMR studies on rabbit skeletal muscle aldolase (D-fructose-1,6-biphosphate D-glyceraldehyde-3-phosphate-lyase, EC 4.1.2.13). As it is a new field of research for us, we have encountered several niggling technical problems and one of these being "how to get rid of paramagnetic ions".

We have used the ion-exchange resin, Chelex-100^{TM}, to solve this problem. In order to get rid of the ions on the inner surface of the 10mm tube, we put a slurry of Chelex in deionized water in the tube and spin it. Monitoring by X-ray fluorescence analysis shows a clear drop in the amounts of paramagnetic ions in our tube. X-ray fluorescence constitutes the only practical way for us to determine whether or not there are paramagnetic enemies in our tubes or solutions.

However, our real problem has been the treatment of the sample. We could not pass it through a Chelex column since it would dilute our preparation and we only have a very limited volume solution that is only 0.5 mM in phosphate. Another alternative was to shake our aldolase solution with a small amount of Chelex for about 30 minutes as described in the BIO-RAD instructions and then separate the two components. However, it proves to be very difficult to decant the solution from the Chelex. It takes about two hours to get the solution clean of Chelex by centrifuging and decanting techniques. Also, by manipulating the sample, the problem of recontamination can occur and we can also lose part of our solution.

It appears that the simplest way is that based on the technique used by Saul and Don (1). We place a small amount of Chelex in a 1.5 ml conical propylene microcentrifuge tube (Eppendorf) containing our solution and then shake for 30 minutes. We then cut the cap off the Eppendorf so that it will fit in the larger centrifuge tube (see fig. 1), make a hole in its base with a 27-gauge needle and put it in another Eppendorf

of the same size also with the cap off. We place the whole thing in a larger centrifuge tube (see fig. 1), and after centrifuging for 5 minutes, we have our sample in the bottom Eppendorf and our Chelex which is dry of our sample in the other. Thus we do not lose any sample while saving a lot of precious time and having an extra purification of our aldolase solution since it must pass through the Chelex to get to the bottom Eppendorf. Of course, the hole must be small enough so that the Chelex particles can not pass through it.

I hope this contribution, simple as it is, will help someone working with biological material save paramagnetic ions purification time and also save many frustrations.

Please credit this to Serge Lacelle's account.

Sincerely,

Luc Tremblay

(1) Saul A. and Don M., (1984) Anal. Biochem., 138, 451-453.

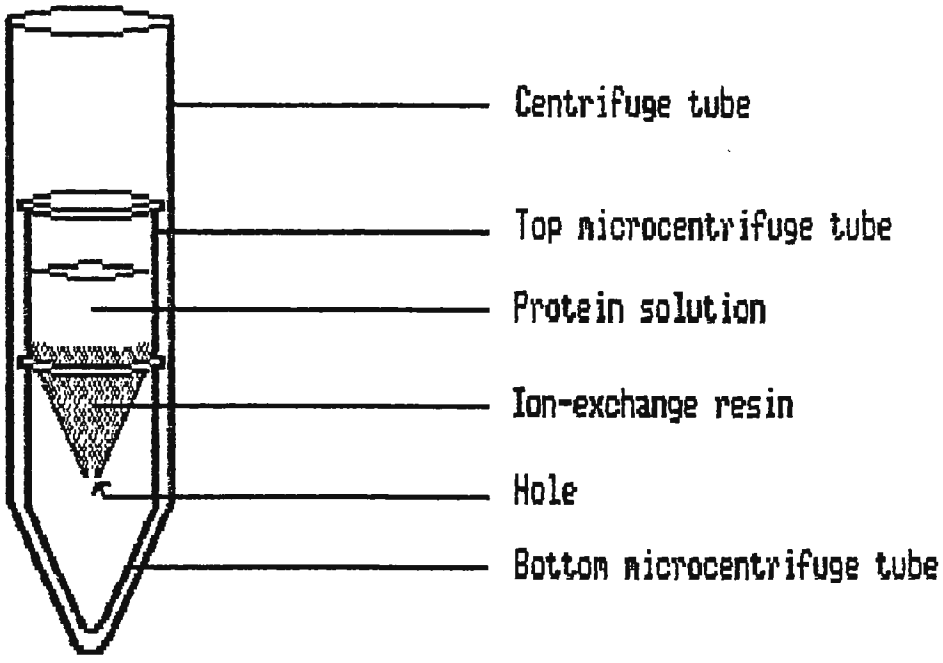

Figure 1: The technical setting

Fremont Magnetic Resonance
47647 Hoyt Street
Fremont CA 94539
(415)656-2398

FMR's charter is to provide the NMR researcher with technological assistance and customized hardware to allow existing equipment to be upgraded to the level required for today's NMR experiments. This includes:

- o Custom NMR probes and probe upgrades for high resolution NMR instruments.
- o Custom surface coils for CSI Instruments
- o On site analysis to determine the performance levels and limitations of current instrumentation. Often a more complete understanding of perceived instrument limitations can provide insight into their solution.
- o Instrument modifications to increase performance or provide new capabilities.
- o New instrument modules and upgrades for existing hardware to increase performance and provide new capabilities.

Surface coils

A simple approach to surface coil design can produce a circuit with excessive sensitivity to sample loading making it difficult or impossible to match some loads. This leads to inefficient data collection. FMR can produce surface coils with much greater tolerance to different samples, either with adjustable tune and match for the greatest sensitivity, or with no adjustable components for the greatest ease of use. These latter surface coils may also be ordered in a hermetic design that may be autoclaved.

Imaging coils

Custom imaging coils with the excellent rf linearity not possible with a simple Helmholtz configuration are available for single frequency operation from 20 to 500 MHz with sizes from 1 cm to 30 cm.

High Resolution Probe Upgrades

FMR offers a wide varety of new high resolution probes for most NMR systems and upgrades for existing probes to a higher performance level or to do different nuclei. This is usually done for much less than the cost of a new probe. This option is available for most NMR probes including the top loading NT Series probes. Performance specifications based on a console in good working order with a noise figure of less than 2 dB are shown in the summary below. Call for more information or different requirements.

5mm ^1H Probe

90° Pulse 10 us @ 50 Watts
Resolution 0.2 Hz (ODCB)
Lineshape 10/20 ($CDCl_3$)

Sensitivity (0.1% EB)	
200 MHz	80:1
300 MHz	160:1
360 MHz	220:1
400 MHz	300:1
500 MHz	400:1

Reverse Polarization Transfer Probe
^1H {^{31}P-^{15}N} 5mm Probe

		5mm	10mm
90° Pulse @ 50 Watts		15us	25us
Resolution (ODCB)		0.25	0.4
Lineshape ($CDCl_3$)		10/25	18/40
Gamma H_2 (10W)			
^{15}N		2KHz	1KHz
^{13}C		3KHz	2KHz
Sensitivity (0.1%EB)			
	200 MHz	60	100
	300 MHz	120	200
	360 MHz	170	280
	400 MHz	200	330
	500 MHz	300	500

Broadband Probes

High Band broadband probes cover the range from ^{31}P to ^{15}N for solutions up to 0.2 M salt. ***^{31}P performance is 15% less for narrow bore magnets.***

5mm	200	300	360	400	500
^{31}P	30	60	85	100	150
^{13}C	30	60	85	100	150
^{2}H NA=4	5	10	14	16	25
^{17}O NA=100	20	40	55	65	80
^{15}N	4	8	10	12	16
10mm					
^{31}P	130	260	340	375	450
^{13}C	150	300	360	400	500
^{2}H NA=4	15	30	40	50	60
^{17}O NA=100	110	220	270	300	400
^{15}N	15	30	35	40	50
12mm					
^{31}P	150	300	400	500	550
^{13}C	180	360	500	600	700
^{2}H NA=4	20	40	50	60	75
^{17}O NA=100	125	250	300	350	500
^{15}N	15	30	40	50	60
20mm					
31P	250	500	600	750	
^{13}C	300	600	700	800	
^{2}H NA=4	30	60	70	90	
^{17}O NA=100	180	350	450	550	
^{15}N	25	50	70	80	

fm
Fremont Magnetic Resonance
47647 Hoyt Street
Fremont CA 94539
(415)656-2398

Instrumentation Note 1

System Noise Figure

One important parameter which indicates how well a spectrometer is working is the noise figure of the preamp and total system. An inexpensive method of determining the system noise performance is the "hot/cold resistor" test. Done with care, this can provide valuable insight into spectrometer performance.

The first step is constructing the noise source. Take a piece of RG58 coax cable and carefully strip the cover back about 2 inches. Now slide the shield back as far as possible (about 1 inch), cut the exposed center conductor to a 1/2 inch in length and strip off the insulation on a 1/4 inch of the conductor. Solder a 1% 50 Ohm metal film resistor to the center lead. Cover with shrink tubing and shrink the cover tight leaving the unconnected end of the resistor exposed. Slide the shield down over the resistor and solder the shield and the unconnected end of the resistor together to make a shielded noise source. Take care to make all leads as short as possible. In most environments, it is very important to use a shielded noise source to help reduce outside RF noise. Interference renders this measurement useless.

Carbon resistors increase resistance as much as 20% when cooled in liquid nitrogen. For this reason, two sources need to be prepared if carbon resistors are to be used. One which measures 50 Ohm at 20°C and one which measures 50 Ohm in liquid nitrogen. Determine the values with an Ohm meter. The metal film resistors change only a few percent when cooled by liquid nitrogen and are therefore preferred.

Connect the shielded 50 Ohm noise source to the input of the preamp in place of the probe. If the entire system noise figure is to be determined, leave all devices for transmitter coupling in place. Set the spectrometer to operate on the frequency of interest with a large sweep width (+/- 10,000 Hz) and a gain level set to fill the digitizer more than half way when acquiring a single scan. Collect a scan and determine the RMS of the noise after fourier transform with no line-broadening. This can be done with computerized routines or by plotting the noise and drawing a line on top and bottom of the noise such that about 10% of the maximum noise excursions fall outside the lines. Any obvious spikes in the noise spectrum can be ignored, but their presence indicates interference in the NMR system.

Repeat the above process with the 50 Ohm liquid nitrogen noise source in place of the probe while the source is being cooled by liquid nitrogen. Process the data such that the same normalization constants are used and determine the RMS value of the noise as above.

These two RMS noise values can be used to calculate the instrument noise figure as indicated by the equation below:

$$NF(dB) = -1.279-10\log_{10}[1-(RMS_c^2/RMS_w^2)]$$

where RMS_c is the value of the RMS noise in liquid Nitrogen and RMS_w is the value at room temperature (20°C)

A table relating noise figure to the ratio of the cold RMS noise value to the warm RMS noise value (RMS_c/RMS_w) as calculated by the above equation is shown in the table below:

Ratio	NF(dB)	Ratio	NF(dB)
0.99	15.73	0.90	5.93
0.98	12.74	0.85	4.29
0.97	11.01	0.80	3.16
0.96	9.78	0.75	2.31
0.95	8.83	0.70	1.65
0.94	8.06	0.65	1.11
0.93	7.41	0.60	0.66
0.92	6.86	0.55	0.29
0.91	6.37		

This test is easy to do, but requires careful experimental technique. If a noise level difference cannot be observed between a hot and cold resistor, make sure the system can observe an NMR signal from a standard sample. If you are unable to obtain a reproducible system noise figure, problems in the system noise profile could be presenting limitations on spectrometer performance. A good procedure is to strip the spectrometer receiving system to the bare minimum. If possible take out any unnecessary lock filters (turn off the lock) and decoupler filters from the receiving system. Remove the transmitter coupler if possible. With this minimum system determine the system noise figure. Anything more than 3.0 dB needs to be improved. Now add back the removed components one by one. Repeat the test after the addition of each component to determine how they affect the system noise figure. Todays spectrometers typically give overall system noise figure of less than 2.0 dB. Call for further information or to discuss problems with this measurement on your instrument.

National Center of Neurology and Psychiatry

NATIONAL INSTITUTE OF NEUROSCIENCE
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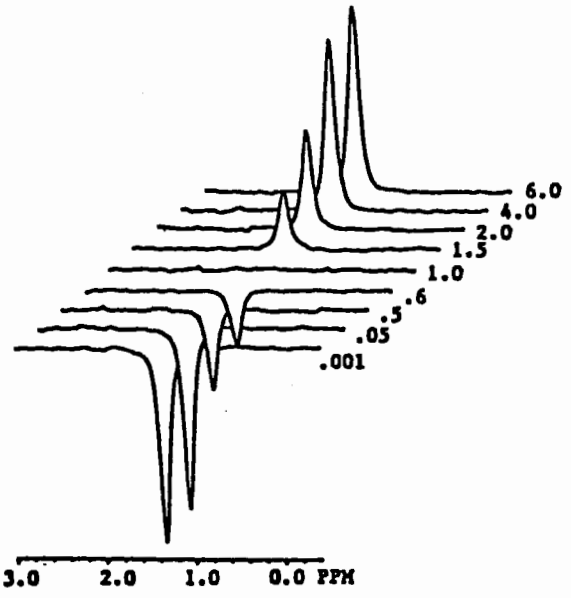
July 20, 1988
(received 7/25/88)

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

Approaching Perfection In An Imperfect System: Determination Of Relaxation Times In Vivo.

Dear Dr. Shapiro:

Spin-lattice relaxation times(T1) for metabolites are needed to determine true signal intensities and provide information about molecular motion of metabolites in the cells. Problems with obtaining T1 values by use of surface coils stem from the fact that it is difficult to fully invert signals in the inversion-recovery experiment due to the RF inhomogeneity. These problems were well discussed and the optimum methods for acquiring these values were proposed. Obtaining T1 values for specific metabolites in vivo is further complicated by spectral resolution and biological variables. This is particularly true of H-1 NMR signals. We have developed two variations of the inversion-recovery sequence with surface coils to overcome these problems and determine accurate T1 values for specific metabolites, for instance, lactate in the brain. The pulse sequence combines with the H-1 editing sequence and gives <u>full inversion</u> of the signals as shown in the figure of the edited lactate signal from the rabbit brain. The delay time in seconds is shown in each spectrum. The T1 value of lactate in the rabbit brain after death determined from these experiments is 1.45 ± 0.09 seconds(*).



Dr. B. L. Shapiro
page 2 of 2

We would like to announce that Philips Gyroscan S-15A imaging/spectroscopy system with a 2 tesla whole body magnet which is broad band and capable of obtaining high resolution NMR spectra from localized volumes within human body will be installed in July 1988 at the National Hospital. A collaborative research project may be possible to study the metabolic alterations in brain and muscles and to develop NMR techniques for clinical investigation and diagnosis. This project will be partly supported by the Agency of Science and Technology in Japan.

Sincerely,

Takashi Ogino Toshio Yano

(*) This experiment was performed in collaboration with Dr. E. J. Novotny and Prof. R. G. Shulman at the Magnetic Resonance Center, Yale University.

Position Available

Staff Research Associate position in Analytic Chemistry Instrumentation Facility. Duties include operating and maintaining both NMR spectrometers and optical spectrophotometers. Current equipment includes (GN-500, QE-300, NT-300, Joel-200), (Nicolet 5DX FT-IR, Cary UV-visible, Jasco CD). M.S. or Ph.D degree with background in FT/NMR and optical spectroscopy preferred. More information can be obtained from Dr. Robert Lee, Academic Coordinator, NMR Facility, UC Riverside, Riverside, CA 92521. Tel No:(714)787-3648.

Polyflon Company

Non-magnetic Capacitors for NMR Applications

An array of Polyflon non-magnetic capacitors, many of which are used in NMR/MRI applications.

Polyflon's rugged trimmer and variable capacitors meet rigid NMR/MRI specifications.

Polyflon's variable, trimmer and fixed capacitors meet critical non-magnetic, high voltage, high power, high Q NMR specifications.

A test report was recently submitted to Polyflon by a University deeply involved in NMR research. Polyflon trimmers were subjected to various tests in order to gather essential data (Q, DF, L, ESR, EPR) for use in the design of NMR RF equipment. Tests were conducted at 10, 100, 200, 400, 500, and 600 MHz. These trimmers were first exposed to a 4.7 Tesla magnetic field for detection of the presence of any magnetic particles in the trimmers. These tests conclusively showed there were no magnetic particles present.

Maximum dissipation displayed by the trimmers was 0.000125 with a minimum less than 0.0001, resulting in Q measurements from 8,000 to more than 10,000, at 600 MHz. (NOTE: Customers, who have tested Polyflon trimmers in their own laboratories, have measured Q's greater than 20,000 at 500 MHz.) The inductance of the trimmers was less than one nHy and maximum ESR at 160 milliohms with minimum EPR at 100 megohms.

Polyflon trimmers and variable capacitors provide linear tuning with no reversal in capacitance during capacitor adjustment. They are used in NMR pulsed applications with operating voltages up to 15 Kv peak and duty cycles ranging up to 10 %. Standard range of variable capacitor designs are available with a minimum capacitance of less than 1 pF to a maximum capacitance of 125 pF.

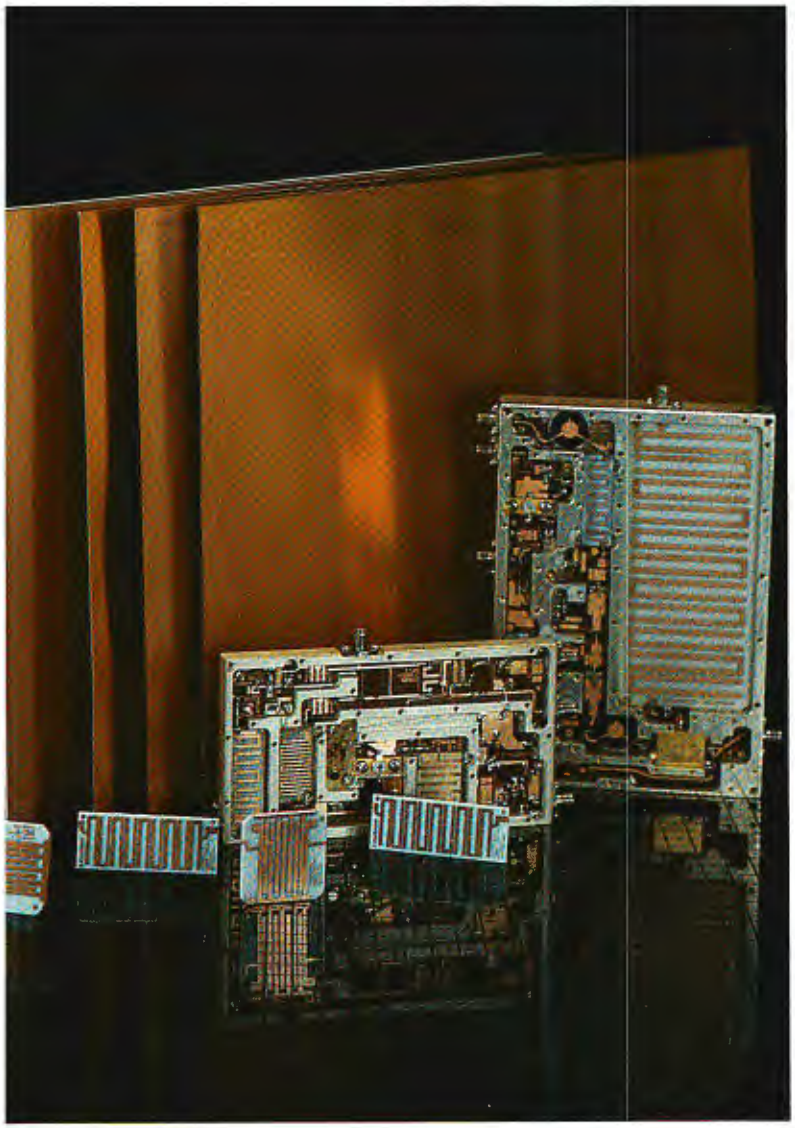
Fixed capacitors, used for 6 Kv peak pulsed applications, are available with capacitance values from 50 pF to 250 pF. For 10 Kv peak voltage applications, fixed capacitors are available with capacitance values ranging from 25 pF to 150 pF.

Polyflon also designs and manufactures variable, trimmer and fixed capacitors to customer specifications.

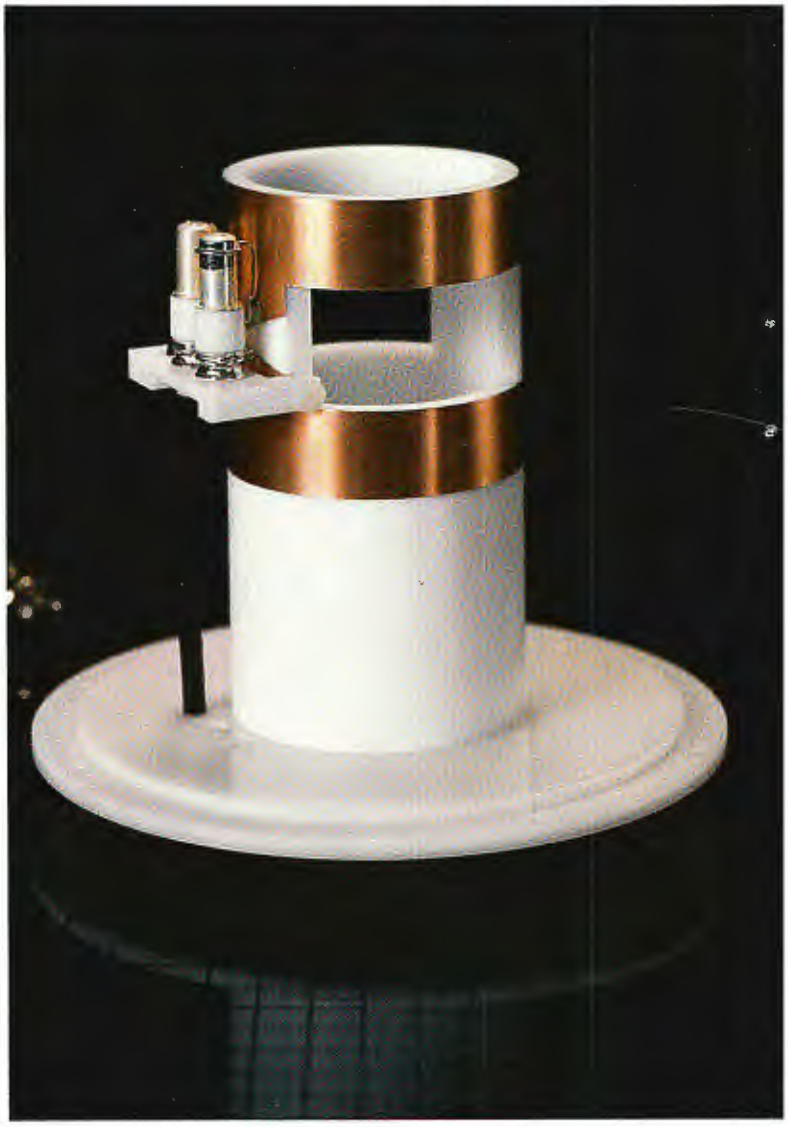
Polyflon Capacitor Advantages:

- **Non-magnetic**
- **Low dissipation factor (low loss)**
- **Extremely High Q**
- **Low ESR**
- **High EPR**
- **Linear tuning (no capacitance reversal)**
- **High voltage capability**
- **Rugged design**
- **Long operating life**

Material Technology for NMR Product Applications



Pure PTFE dielectric is used in all Polyflon CuFlon® microwave substrates, assuring low loss.



80 MHz copper plated PTFE saddle coil assembly.

Polyflon uses only pure PTFE dielectric in its CuFlon® microwave substrates which provides one of the best low loss dielectrics in the industry.

Since nothing is added to the PTFE, the dielectric constant of 2.1 remains consistent over an extremely wide range of frequencies.

The unique, proprietary electroplating technology for PTFE, developed by Polyflon, permits design and fabrication of superior RF components.

An intimate bond is produced between the metal surfaces and the PTFE, with no other element or media between them. The dissipation factor and Q is then that of the PTFE substrate itself.

Very low loss components are produced with this technology that can be used with excellent results at very high microwave frequencies.

Polyflon's CuFlon substrate materials, found in many NMR/MRI applications, are used in various coil designs such as surface, solenoid and saddle configurations. CuFlon is also used as a substrate in pulsed RF amplifiers, wide-band RF transformers, and chip capacitors for tuning and matching elements.

Polyflon's expertise in the PTFE plating technology can and does provide customers with rugged and reliable products for critical NMR/MRI applications.

Call or write for information.

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

July 5, 1988
(received 7/25/88)

Models for simulation of NOE cross-peak volumes

Dear Barry:

Information on coordinates of macromolecule protons, as well as on relaxation (T_1) and correlation (τ_c) times make it possible to compute the relaxation matrix and therefore cross-peak volumes of NOESY spectra [1-3]. Iterative fitting of the theoretical cross-peak volumes to the experimental NOESY spectrum can be used when elucidating the spatial structure of the molecule. However, this procedure takes much computer time for large molecules (e.g., proteins), to say nothing of the difficulties intrinsic to optimization problems. Another approach implies the hierarchic algorithm of spatial structure reconstruction according to NMR spectroscopy data. For proteins, the local structure of amino acid residues (see, e.g. [4,5]) is the first to be determined (approximate values of angles φ, ψ and χ), then the secondary structure to be revealed (α-helices, β-structures, β-turns, etc.) and, finally, the elements of the secondary structure are to be arranged into the three-dimentional protein structure. Evidently, the local structure can be analysed by the cross-peaks volumes only if influence of protons adjacent to the given fragment is negligible, or they have to be taken into account.

To elucidate different contributions of the environment into the volumes of cross-peaks and to study the possibility of application of simplified mathematical treatments we examined models I-IV (Fig. 1). Model I takes into account full set of relaxation rates and every adjacent proton. Models II, III and IV correspond to increasing order of simplification. Although model II provides the most accurate values of cross-peak volumes as compared to other simplified models, its practical application is substantially restrained since it requires data on the selective spin-lattice relaxation times. Results obtained by means of model III, utilizing coordinates and nonselective spin-lattice relaxation times of the dipeptide fragment protons, only slightly depend on complete and accurate information on nonselective proton relaxation times. This model seems most applicable.

Data obtained by means of the most complete model I (Fig. 2), show that assertion "the less mobility, the greater cross-peak volume" is true only for short τ_m, and hence one has to use extreme care when exploiting difference in cross-peak volumes for mobility estimation and for calibration of proton-proton separation by using standard distances.

The results in full details will be presented for publication in "Bioorgan. Khem." (USSR).

With best wishes,

Sincerely yours Vladimir A. Arseniev A. Sobol

V. F. Bystrov A. S. Arseniev A. G. Sobol

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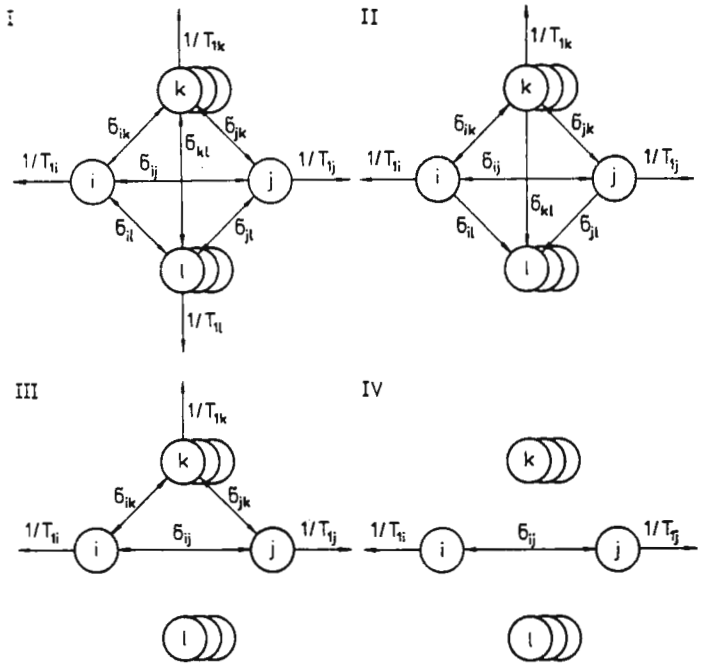

Fig. 1

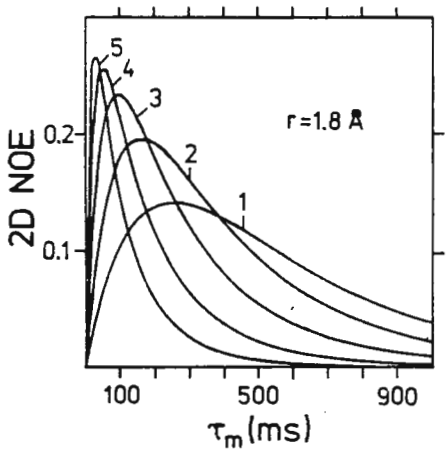

Fig. 2

Fig. 1 Schemes for magnetization transfer routes in models I-IV. Indexes i and j denote the protons with cross-peaks under discussion, k - other protons of dipeptide fragment, l - other protons of the molecule, σ - rate of cross-relaxation, T_1 - nonselective spin-lattice relaxation time.

Fig. 2 Dependence of the NOE cross-peak volumes on mixing time (τ_m) for the geminal protons at different correlation times: curves 1-5 correspond to the correlation times 1, 2, 4, 8 and 15 ns.

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DEPARTMENT OF PHARMACEUTICAL CHEMISTRY

SAN FRANCISCO, CALIFORNIA 94143

(received 7/20/88)

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

Title: Computer Assisted Sequential Assignment of Proteins

Dear Barry,

The most difficult and time consuming aspect of protein structure determination by nmr is the sequential assignment of chemical shifts. The logic of the sequential assignment process is quite simple. However, the difficulty is that the quantity of data involved is large, and much of it is potentially ambiguous. Protein nmr spectroscopists could save a great deal of time by using computer programs designed to apply the sequential assignment logic directly to nmr data in the form of peak coordinates and intensities. We have therefore written a set of computer programs for assisting in the sequential assignment of proton chemical shifts in proteins.

The programs work in several steps. First, the chemical shifts of the N, C-alpha, and C-beta protons for each spin system are determined by identifying scalar coupling in COSY and HOHAHA spectra. Next, NOESY cross peaks that give sequential information according to the sequential assignment logic are identified. Finally, the spin systems are assembled into long lists which have a high probability of being sequential in the primary sequence.

As input to the programs, the user must supply a list of chemical shift coordinates for peaks in the fingerprint region of the COSY spectrum, peaks in the alpha-beta region of the COSY spectrum, and peaks above the N proton region in the HOHAHA and NOESY spectra. The programs also require arbitrary names for the fingerprint peaks for identification of the spin systems, and integrated intensities of the NOESY peaks for ranking spin systems according to their probability of being sequential. Several output files are created. One consists of a list of spin system names followed by chemical shifts traced out to the C-beta protons. Also, for each spin system, a list of all other spin systems which may precede or follow the given spin system in the primary sequence are listed along with the NOESY cross peak intensities and coordinates that suggest the sequential relations. Long lists of spin systems having a high probability of being sequential are output as well.

The spectroscopist must trace the spin systems beyond the beta protons by hand to determine spin system type, and to establish correspondence of the computer generated lists of sequential spin systems with the primary sequence according to this information. The lists of potential next and previous spin systems may be used to extend the assigned spin systems by using spin system type information. For a test data set acquired for a mutant of bovine pancreatic trypsin inhibitor, 48% of the spin systems with finger print peaks were automatically placed in sequential lists of length 3 or longer, and lists of possible next and previous spin systems were created for most of the other spin systems. By combining knowledge of the spin system types (determined manually) with the program output, it was possible to assign unambiguously 86% of the spin systems. The total time required to achieve this result was approximately two weeks. Most of this time was spent on peak identification and integration, and on tracing the spin systems beyond the C-beta protons. The computer time required for the process is negligible. The programs are written in Fortran, and should be ready for general release by October.

Best Wishes,

Charles Eads

Charles D. Eads

Irwin D. Kuntz

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July 18, 1988
(received 7/25/88)

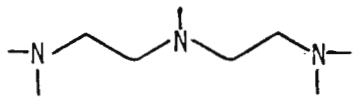
Dr. B. L. Shapiro
966 Elsinore Court
Palo Alto, CA 94303

Chiral Lithium

Dear Barry:

Responding to the dreaded pink note. -

In recent work we have found neopentyllithium-^{6}Li, **1**, to be a compound whose structure and state of aggregation is largely determined by the ligands to which it can be complexed. We have identified from the ^{13}C, ^{6}Li coupling pattern of the $CH_2(\alpha)$ of **1** tetramers and dimers in diethyl ether-toluene, dimers alone in diethyl ether, monomers and dimers in THF and **exclusively** monomers in THF or diethyl ether containing an equivalent of N,N,N',N',N"-pentamethyldiethylenetriamine, PMDTA. Carbon-13 NMR of the latter solution at 160K, see Figure, indicates that



PMDTA

with the exception of two methylenes all carbons of complexed PMDTA are magnetically non-equivalent. This means that the complex must be locked into one unsymmetrical rotamer. In such a structure lithium and the CH_3N nitrogen are chiral centers, see Figure. Further, inversion at the $(CH_3)_2N$ nitrogens and rotation about the carbon lithium "bond" are both slow relative to the NMR time scale, as is also carbon lithium bond exchange. In fact the latter process is still slow by 240K as evidenced by triplet splitting of the neopentyl $^{13}CH_2$ resonance. Above 170K the two $(CH_3)_2N$ ^{13}C doublets individually average to single lines at their centers, due to inversion at nitrogen, the result of sequential reversible nitrogen lithium bond dissociation, see dashed lines in Figure. By 250K all the methyl resonances have averaged. We ascribe this phenomenon to rotation within the complex. Taking into account both processes in the NMR line-shape analysis of the N-methyl carbon resonance yields for nitrogen inversion(i) $\Delta H_i^{\ddagger}$ = 11.4 kcal and $\Delta S_i^{\ddagger}$ = 14.6 eu whereas for rotation (r) $\Delta H_r^{\ddagger}$ = 6 kcal and $\Delta S_r^{\ddagger}$ = -20 eu. We envisage rotation to proceed through a loose transition state involving extra solvation.

In the THF solutions containing neopentyllithium we have observed a third process, the exchange of PMDTA between its lithium complex and its free state in solution.

Ordinarily lithium ligand exchange is very fast relative to the NMR time scale. In the present case the trifunctionality of the amine slows down all lithium nitrogen bond exchange processes. Needless to say these and related experiments are still under way.

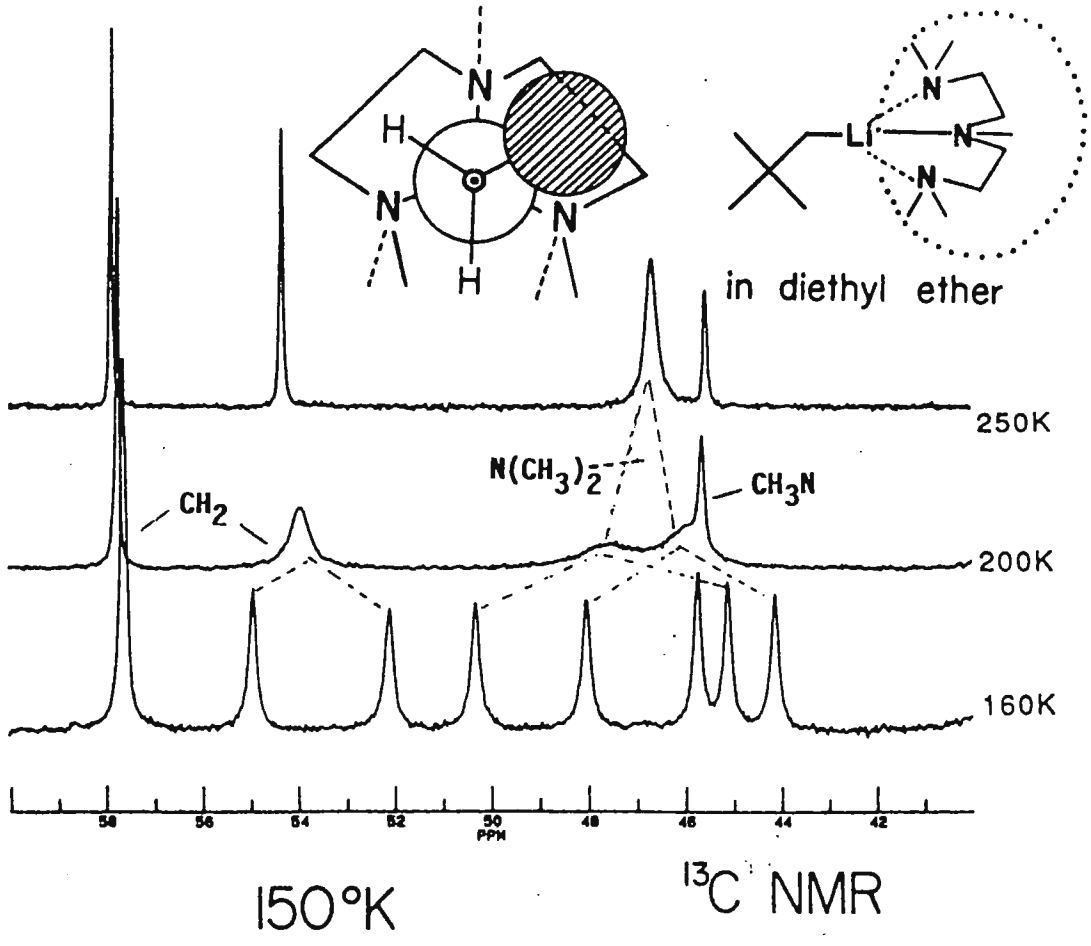
With our best regards.

Sincerely,

Gideon Fraenkel
Professor of Chemistry

William R. Winchester
Research Associate

ls



TAMU NMR Newsletter

Policies and Practical Considerations

(Revised July 1988)

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

1. **Policy**:

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

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

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

2. **Public Quotation and Referencing:**

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

3. **Participation is the prime requisite for receiving the TAMU NMR Newsletter:**
In order to receive the Newsletter, you must make at least occasional technical contributions to its contents.

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

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

4. **Finances**:

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

The **Subscription** fee is currently $120.00 per year, with a 50% academic or personal discount being available. Subscriptions are available only for the twelve monthly issues which begin with the October issue and run through that of the following September. However, a subscription can be initiated at any time, and the issues back to the previous October will be provided as long as copies remain available.

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

Continued

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

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

5. **Practical Considerations**:

a) All technical contributions to the TAMU NMR Newsletter will always be included in the next issue if received before the published deadline dates.

b) Please provide short titles of all topics of your contributions, so as to ensure accuracy in the table of contents.

c) Contributions should be on the *minimum* (NOTE!!) number of 8.5 x 11" (21 x 27.5 cm) pages, printed on one side only. There must be margins of at least 0.5 - 0.75" (1.3 - 2.0 cm) on all sides. Please observe these limits. Black ink for typing, drawings, etc., is essential. All drawings, figures, etc., should be mounted in place on the 8.5 x 11" pages. We are not equipped to handle pieces of paper larger than 8.5 x 11" (21 x 27.5 cm).

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

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

ii) **PLEASE** avoid excessive margins. *Instruct your secretaries to avoid normal correspondence esthetics or practices, however time-honored or 'standard'!* This page has margins on both sides of 0.6" (*ca.* 1.55 cm), which is very adequate. Margins of the same size at the top and bottom are sufficient also, but don't worry if there is more space at the end of your document, for I can often use such spaces for notices, etc. You are reminded that regardless of the standard paper length you use, *all material* - letterhead, text, figures, addresses printed at the page bottom, *everything - must not exceed 10" (ca. 25.3 cm) from top to bottom.*

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

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

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

6. **Suggestions**: They are always welcome.

B. L. Shapiro

Address for all correspondence:

Dr. Bernard L. Shapiro
Editor/Publisher
TAMU NMR Newsletter
966 Elsinore Court
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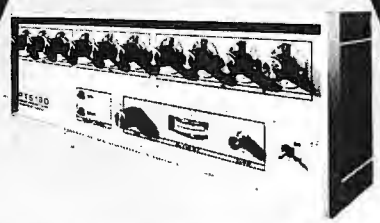

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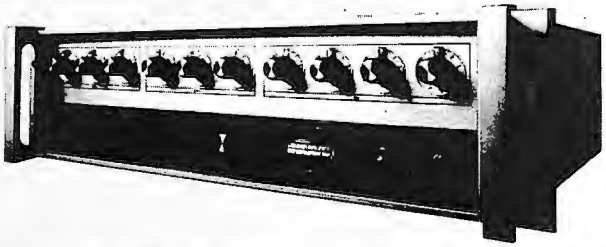

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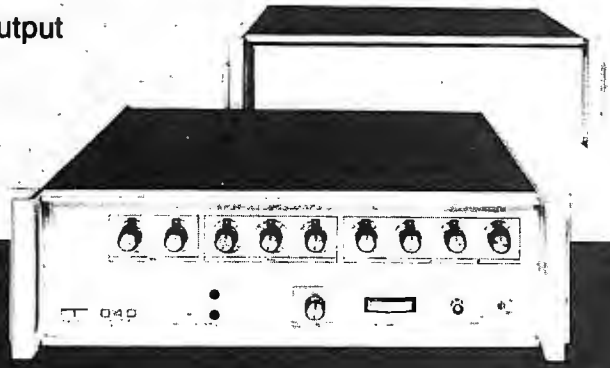

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July 6, 1988
(received 7/19/88)

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

Dear Barry:

Methods in Quantitative NMR Analysis

As part of a central analytical service facility, we are frequently asked to give quantitative information on a variety of proprietary, as well as competitive, products. In view of the extreme importance (and blind faith) that our clients give the results of our integrations, we felt that it might be worthwhile at this time to remind our fellow readers of the precautions one needs to take when undertaking quantitative analysis by NMR.

As of late, we have been comparing the reproducibility of our work among our staff, as well as with other labs. We have little trouble obtaining reproducible results when we have high signal-to-noise and good separation of peaks. Errors typically range between 1-3%. However, when working in a crowded region of a spectrum, or with mediocre signal to noise, or overlapping peaks, errors skyrocket up to 5-65%. We find ourselves, therefore, taking a path that others have taken before (1,2,3) to ensure the best possible data. We have found their suggestions both timely, and well worth repeating. A brief summary of the main causes of error, as well as the steps that can be taken to minimize these errors follows.

1) Saturation: Proper choice of instrumental parameters must always take into account the relaxation times of the species involved.

2) Noise: One must scan sufficiently long such that adequate signal-to-noise is obtained.

3) Baseline characterization: Poor baseline characterization around and under the peaks frequently occurs and must be compensated for by software means.

4) Digitization error: The spectral data point density should be high enough to resolve the narrowest feature in the transformed spectrum. Zero filling yields additional peak definition and is quite helpful.

5) Lack of consistency: This manifests itself principally in two ways--improper phasing of peaks (an operator error that can be corrected), and integration errors due to improper choice of integration limits. In the case of overlapping peaks, curve fitting yields the best results. And even in the case of nonoverlapping peaks, it can help greatly. An easily overlooked fact is that a Lorentzian line has a broad tail, and one must integrate over a large frequency range to achieve reasonable accuracy. To reduce truncation error to 5%, one must take the integration limits to 6.3 times the half width! Otherwise, underestimation of the peak area occurs. Consistency in calling the limits helps dramatically, especially when concentration is being determined from a calibration curve of an internal standard. Here too, curve fitting can help, as it takes into account the broad tail of the peak shape. However, this does not take into account non-ideal peaks.

Although all this may sound elementary, these are important considerations that are all too easily (and frequently) forgotten.

Sincerely,

Janice K.Gard John C.Burquin Pierre A.Berger

1) Becker, E.D., Ferretti, J.A., Gambhir, P.N.; Anal. Chem. 1979, 51, 1413-1420.
2) Weiss, G.H., Ferretti, J.A.; J. Mag. Reson. 1983, 55, 397-407.
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359-52

DEPARTMENT OF THE NAVY
NAVAL RESEARCH LABORATORY
WASHINGTON, D.C. 20375—5000

IN REPLY REFER TO:
18 July 1988
(received 7/25/88)

Dear Barry:

DISTINGUISHING DIFFERENTIAL DEVELOPMENT OF MQ COHERENCE IN LIQUID CRYSTALS

We show here an example of multiple-quantum (MQ) evolution in a liquid crystalline (LQ) system in which coherences from different parts of the same molecule do not intermix.

A 2:1 molar mixture of 4'-cyanophenyl-4-n-heptylbenzoate and 4'-cyanophenyl-4-n-butylbenzoate exhibits nematic liquid crystal behavior in the temperature range from 25° to 50° C. The proton NMR spectra of the nematic phase (Fig. 1a) corresponds to a central narrow peak and a doublet suggesting that two distinct regimes of dipolar interaction are present. The central peak arises from the protons associated with the alkyl chains of the parent molecules. The doublet probably originates from isolated proton spin-pair dipole-dipole interactions on the phenyl ring system. A solid-echo ($90° - \tau - 90°$) pulse sequence separates these unique regions within the molecule; in Fig. 1B, the 90° pulses are 168 μsec apart, and the signal from the (more or less isolated) proton spin pairs on the phenyl moieties can be distinguished from the weakly-coupled alkyl chain protons.

We have combined this dipolar echo selection with multiple-quantum NMR.[1-3] The spins are first prepared by means of a variable delay solid-echo sequence, and the subsequent development of MQ coherence is then monitored. In the standard MQ spectrum in Fig. 2A (no solid-echo delay), multiple-quantum transitions are apparent out to n=8 under these conditions. Do the intensities of the coherences arise from separate regions of the molecule? In order to carry out accurate spin-counting calculations, one must account for the development of spin coherence in each distinct population. In the case of Fig. 2B, where a selective (dipolar-echo) pulse sequence has been applied, we see the dominance of the 2-quantum intensity, presumably from the isolated proton spin pairs. Differential development of proton spin coherence can then be distinguished (in this case) for *different* segments of the *same* molecule. Furthermore, the selected starting conditions determine the population (or combination of spin ensembles) whose coherence dynamics are being assessed. Over a wide range of multiple-quantum preparation periods τ, the proton pairs do not interact appreciably with the remaining alkyl protons of the molecule. The resultant spin-counting indicates two effective system sizes *i. e.* one distribution corresponds to spin clusters of approximately 13 protons consistent with the sum average of those from the alkyl chains of the parent species; the other distribution corresponds to the isolated proton spin-pairs of the phenyl head groups. A full accounting of these studies has been submitted for publication.

With best regards,

Walt Al

W. V. Gerasimowicz* A. N. Garroway J. B. Miller
Code 6122, Chemistry Division

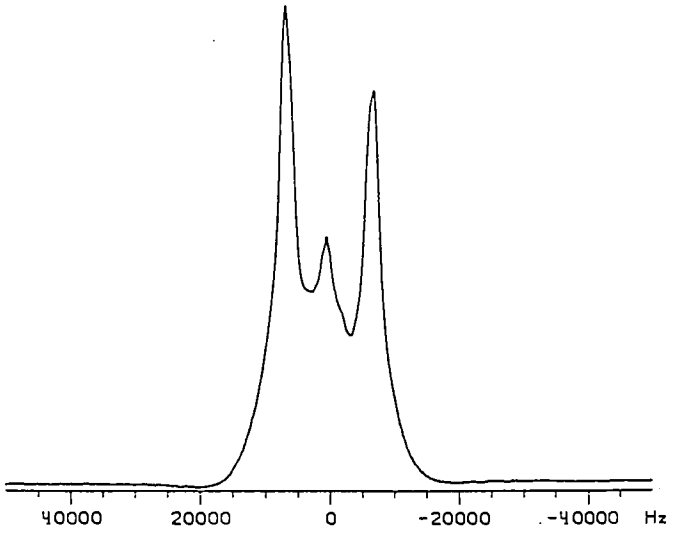
References
1. D. P. Weitekamp, Adv. in Magn. Reson., 11, 1983, 111 - 274.
2. J. Baum, M. Munowitz, A. N. Garroway, and A. Pines, J. Chem. Phys., 83, 1985, 2015 - 2025.
3. D. N. Shykind, J. Baum, S. - B. Liu, A. Pines, and A. N. Garroway, J. Magn. Reson., 76, 1988, 149 - 154.

*Permanent Address: U.S.D.A., Eastern Regional Research Center, 600 East Mermaid Lane, Philadelphia, PA 19118

Z-SPEC

FIGURE 1

A. ^{1}H NMR OF LQ MIXTURE



B. DIPOLAR ECHO SELECTION

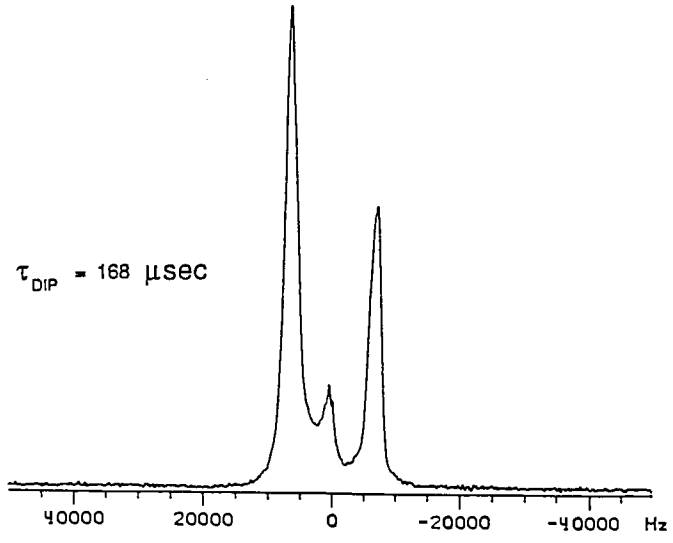
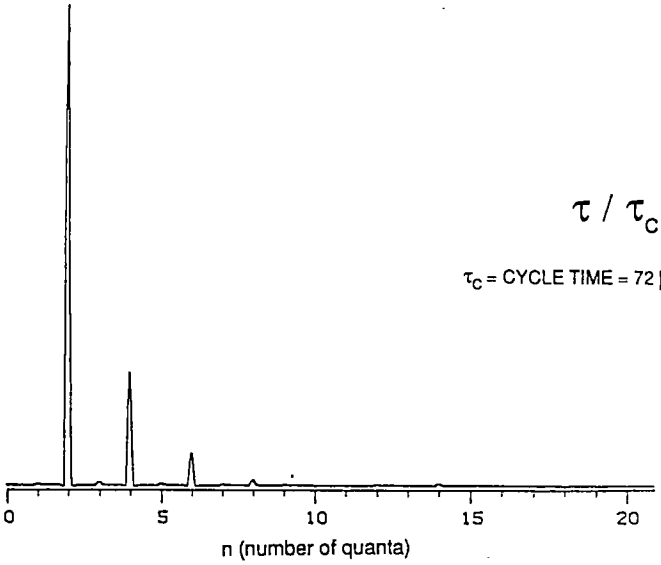
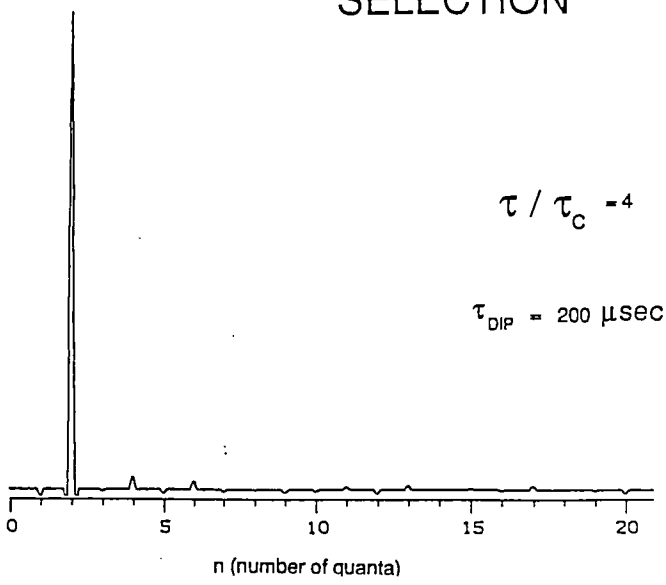


FIGURE 2

A. MQ WITHOUT DIPOLAR ECHO SELECTION



B. MQ WITH DIPOLAR ECHO SELECTION



TEXAS A&M UNIVERSITY
NMR NEWSLETTER

No. 359
August 1988

Table of Contents, cont'd.

* * * * *

Newsletter Subscription and Sponsorship Renewals

Invoices for the 1988-89 Newsletter year have been mailed out over a month ago to all **Subscribers**, and all of the Newsletter **Sponsor** organizations and **Advertisers** have also been contacted about renewing their annual support of this self-supporting publication. If you have not received an invoice or a letter by the time you read this notice, please let me know without delay.

It will be greatly appreciated if you will see that these invoices are processed promptly, in order that the costs of operating the Newsletter can be 'contained'. Fiscal considerations require that **Subscription payment must be received** (or clear indication given me that payment is in the works) **by October 1, 1988** if your Newsletter mailings are to continue without interruption. Many payments have already been received, and I thank those responsible for their promptness.

Several of our **Sponsors** have also responded as of this date, and I hope those yet to do so will be able to indicate their intention to renew their vital support of the Newsletter before long. The Newsletter's financial circumstances are always marginal, and this is more true than ever this year. Significantly increased income is needed, and we hope that our loyal **Sponsors** and **Advertisers** will be able to share with the **Subscribers** the task of meeting the ever increasing costs of the Newsletter.

B.L.S.
10 August 1988

Coming Next Month (in addition to much serious science):

- *Problem Solving Flowsheet;*
- *'Life in the Rotating Frame'. Part I.*

CSI 2T Applications

Shielded Gradients and NMR Microscopy

In spin warp imaging, there is a trade-off between minimum TE and maximum resolution. Even if rise and fall times were zero and phase encoding occured during the entire echo delay, a ±2 Gauss/cm gradient range and a TE of 2 msec would provide best case resolution of 0.32 mm. This translates to a 7 cm field of view in a 256 × 256 matrix. To improve resolution by a factor of 10, TE may be increased by a factor of 10 (which is not acceptable in a sample with short T2 values) or gradient strength may be increased by a factor of 10. The long echo times required for T2 weighted images create an undesired loss of signal in many non-T2 weighted image experiments. These effects, however, are tolerable at 2 Gauss/cm for resolution at the 100-200 micron level.

Clearly, added signal that would be available with a shorter TE would be useful. The current practical limits of high signal-to-noise NMR micro imaging are greatly reduced by high strength shielded gradients. A 50 micron resolution image of an Agapanthus bud is shown in Figure 1. Unlike very high field (>7 Tesla) micro NMR imaging, magnetic susceptibility effects at 2T do not compromise the 50 micron digital resolution obtained during these gradient strengths.

In a second example, (Figs. 2 and 3), 25 micron resolution is achieved in a small phantom by using a moderate access (5 cm) rf coil. The phantom consists of seven small capillary pipets in a 5 mm NMR tube. Data was collected as a 32 × 256 × 256 DEFT data set.

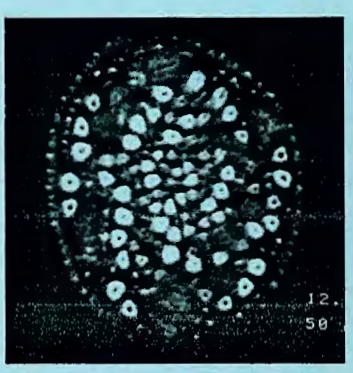


***Fig. 1**—Agapanthus bud Matrix 256 × 256, TR 200 Slice 2 mm, TE 30 FOV 12.8 mm, NEX 4, 45° Tip Angle DEFT Sequence*

***Fig. 2**—16 contiguous 1 mm slices FOV 6.4 mm, NEX 4. TR 150 msec, Field Strength 2T, TE 14 msec*

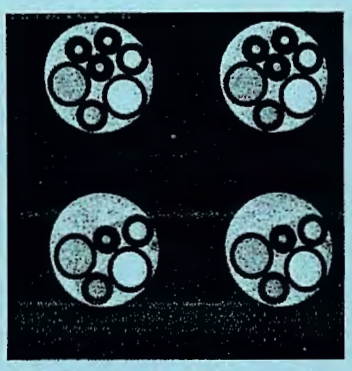


***Fig. 3**—Expanded view of four of the 16 slices shown in Fig. 2.*

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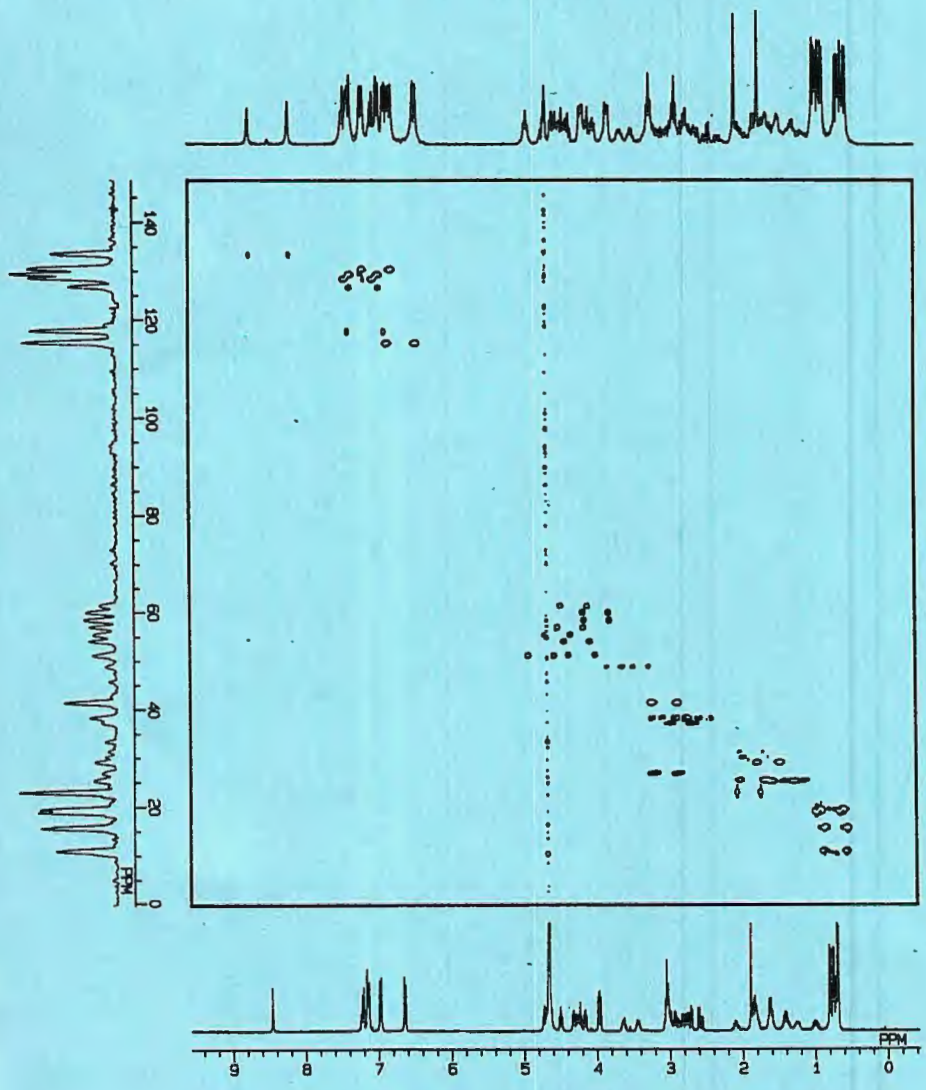
JEOL GSX-FT NMR Systems

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.)

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