

Viscosity Effects on NOEs in Paramagnetic Proteins	La Mar, G. N., and Dugad, L. B.	2
Simulation of NMR Experiments by Density Matrix Operators, Part II	Chen, W., and Broido, M. S.	7
Positions Available	Boicelli, A.	8
Sorbitol-3-Phosphate in the Mammalian Lens; Positions Available	Szwergold, B. S., and Brown, T. R.	11
Bore Tube Conversion on an Old Oxford Horizontal Magnet	Fukushima, E., and Majors, P. D.	15
50cm Plots on QuietJet Printers	Taylor, R. B.	16
Position Available	Gerasimowicz, W. V.	16
Max T. Rogers MSU NMR Facility; Plotting Varian VXR Spectra on an Apple Laserwriter	Yesinowski, J. P., and Johnson, K. M.	21
Resorcinol-Formaldehyde and Other Polymers	Ward, R. L., and Happe, J. A.	22
Homonuclear Decoupled COSY Spectra	Bigler, P.	26
Deuteron Echoes in Occult HD Molecules	Volz, M. P., Fedders, P. A., Conradi, M. S., and Norberg, R. E.	28
NOE Difference Experiments: Small Molecules	Booth, H.	32
Determination of Lactate Using 2D DQCT; Position Available	Pillai, R. P., and Ng, T. C.	35
Installation of a 600MHz NMR Spectrometer in Zuerich	Baumann, R., and Wuethrich, K.	38
NMR Symposium at Washington University, May 24, 1989	Schaefer, J.	42
Proton Images of Solid Bone	Ackerman, J. L., and Garrido, L.	45
Position Available	Sibert, J.	46
Solvent Suppression in TOCSY	Williamson, M. P.	48
Position Available	Eliel, E. L.	51
500 to 400 to VAX to FTNMR	Fry, D. C., Greeley, D. N., and Pitcher, R. G.	52
³¹ P Metabolites in Unanesthetized Neonatal Rats	Smith, M. B., Williams, G. D., and Palmer, C.	55
Fifth International Symposium on Magnetic Resonance in Colloid and Interface Science	Dybowski, C. R.	57
<i>Life in the Rotating Frame. Part VI</i>	Goldberg, W.	58
TAMU NMR Newsletter Policies and Practical Considerations (slightly revised, February 1989)	Shapiro, B. L.	59
Product Operator Formalism on the PC	Dorman, D. E.	61
TAMU NMR Newsletter: <i>Incredibly Important Notice</i>	Shapiro, B. L.	62

A monthly collection of informal private letters from Laboratories of NMR. Information contained herein is solely for the use of the reader. Quotation is *not* permitted, except by direct arrangement with the author of the letter, and the material quoted *must* be referred to as a "Private Communication". Reference to the TAMU NMR Newsletter by name in the open literature is strictly forbidden.

These restrictions apply equally to both the actual Newsletter participant-recipients and to all others who are allowed open access to the Newsletter issues. Strict adherence to this policy is considered essential to the successful continuation of the Newsletter as an informal medium of exchange of NMR information.

TABLE 1 DEUTERATED SOLVENTS

Cat. No.	Description	Formula	Min. Purity (%)	Density (g/ml)	MP (°C)	BP (°C)	$-X_v \times 10^6$ @ (°C)
D-11	Acetone-d ₆	CD ₃ COCD ₃	99.6%	1.17	17	56	0.551 (32)
D-120	Acetone-d ₆	CD ₃ COCD ₃	99.6%	1.17	17	56	0.551 (32)
D-13	Acetone-d ₆	CD ₃ COCD ₃	99.6%	1.17	17	56	0.551 (32)
D-121	Acetone-d ₆ + 1% TMS	CD ₃ COCD ₃	99.8%	1.17	17	56	0.460 (20)
D-129	Acetone-d ₆ + 1% TMS	CD ₃ COCD ₃	99.8%	1.17	17	56	0.460 (20)
D-14	Chloroform-d	CDCl ₃	99.8%	1.50	-64	62	0.740 (20)
D-21	Chloroform-d	CDCl ₃	99.8%	1.50	-64	62	0.740 (20)
D-122	Chloroform-d	CDCl ₃	99.8%	1.50	-64	62	0.740 (20)
D-130	Chloroform-d	CDCl ₃	99.8%	1.50	-64	62	0.740 (20)
D-28	Chloroform-d	CDCl ₃	99.8%	1.50	-64	62	0.740 (20)
D-31	Chloroform-d + 1% TMS	CDCl ₃	99.8%	1.50	-64	62	0.740 (20)

Cost-conscious quality NMR solvents offered by Wilmad, such as CDCl₃, are frequently priced lower than more traditional sources. Included in this offering are the most common solvents, like Acetone-d₆, Benzene-d₆, D₂O, and DMSO-d₆, as well as some of the most unusual solvents for specialty applications, like 1,1,2,2-Tetrachloroethane-d₂, Octane-d₈, and Trifluoroacetic Acid-d.

VARIAN BOX/500 SHEETS

Cat. No.	Size	Instrument Model	Type
WCV-100 (S-100A)	11" X 26"	HA-100, HA-100D, HA-100D	Gridded-Two Color
WCV-60 (S-60C)	11" X 26"	HA-60, HA-60D, HA-60D	Gridded-Two Color
WCV-XL (S-XL)	11" X 26"	HA-60, HA-60D, HA-60D	Gridded-Two Color
WCV-XLFT (S-XLFT)	11" X 26"	HA-60, HA-60D, HA-60D	Gridded-Two Color
WCV-XL-200	11" X 26"	XL-200, XL-300, XL-400	Gridded-Two Color
WCV-20 (CFT-20)	11" X 16 3/4"	CFT-20, FT-80, FT-80A	Gridded-Two Color
WCV-11" (S-11")	11" X 17"	All Models	Blank

We provide the largest variety of paper and pens for NMR recorders or plotters available anywhere. Included in these listing are the newest spectrometers from Varian, Bruker, (and IBM), General Electric and JEOL, as well as the latest models, such as the Hewlett Packard 7475A, 7550A, and Thinkjet 2225A, Zeta 8 or 8A, and Western Graphtec 4730 plotters and printers.

Searching for the Unusual Requirement? WILMAD HAS YOUR ANSWER!

The most comprehensive offering of "widgets, gadgets and specials" for NMR spectroscopy, including:

Spatula for 5mm NMR Tubes
Three types of Valve NMR Tubes
 (including the new J. Young Valve Tube)
Solvent Jet NMR Tube Cleaners
pH Electrode for 5mm NMR Tubes

Taperlok® NMR Tubes
A multitude of Coaxial Inserts
Alumina NMR Tube for Si-29 Studies
Ultra-thin wall NMR Tubes
Throwaway "THRIFT" and "ECONOMY" NMR Tubes

Serving the Spectroscopic Aftermarket



WILMAD GLASS COMPANY

Route 40 and Oak Road • Buena, NJ 08310 U.S.A.

609-697-3000 • TWX 510-687-8911

FAX 609-697-0536

TEXAS A&M NMR NEWSLETTER

NO. 367, APRIL 1989

AUTHOR INDEX

Ackerman, J. L. 45	Dybowski, C. R. 57	Johnson, K. M. 21	Sibert, J. 46
Baumann, R. 38	Eliel, E. L. 51	La Mar, G. N. 2	Smith, M. B. 55
Bigler, P. 26	Fedders, P. A. 28	Majors, P. D. 15	Szwergold, B. S. 11
Boicelli, A. 8	Fry, D. C. 52	Ng, T. C. 35	Taylor, R. B. 16
Booth, H. 32	Fukushima, E. 15	Norberg, R. E. 28	Volz, M. P. 28
Broido, M. S. 7	Garrido, L. 45	Palmer, C. 55	Ward, R. L. 22
Brown, T. R. 11	Gerasimowicz, W. V. 16	Pillai, R. P. 35	Williams, G. D. 55
Chen, W. 7	Goldberg, W. 58	Pitcher, R. G. 52	Williamson, M. P. 48
Conradi, M. S. 28	Greeley, D. N. 52	Schaefer, J. 42	Wuethrich, K. 38
Dorman, D. E. 61	Happe, J. A. 22	Shapiro, B. L. 59, 62	Yesinowski, J. P. 21
Dugad, L. B. 2			

TEXAS A&M NMR NEWSLETTER

NO. 367, APRIL 1989

ADVERTISER INDEX

Bruker Instruments, Inc. 23	Nalorac Cryogenics Corporation 29
Doty Scientific, Inc. 13	New Methods Research, Inc. 9
Fremont Magnetic Resonance 39	Otsuka Electronics (U.S.A.) Inc. 47
GE NMR Instruments 3, inside back cover	Programmed Test Sources, Inc. 37
Isotec Inc. 33	Spectroscopy Imaging Systems Corporation 17
JEOL 43, outside back cover	Varian 19
MR Resources, Inc. 53	Wilma Glass Company, Inc. inside front cover
MSD Isotopes 49	

SPONSORS OF THE TAMU NMR NEWSLETTER

Abbott Laboratories	New Methods Research, Inc.
The British Petroleum Co., Ltd. (England)	NMR Technologies Inc.
Bruker Instruments, Inc.	Norell, Inc.
Cryomagnet Systems, Inc.	The Procter & Gamble Company, Miami Valley Labs
The Dow Chemical Company	Programmed Test Sources, Inc.
Eastman Kodak Company	Shell Development Company
E. I. du Pont de Nemours & Company	Siemens Medical Systems, Inc.
GE NMR Instruments	Spectroscopy Imaging Systems Corporation
JEOL (U.S.A.) Inc., Analytical Instruments Division	Spectral Data Services, Inc.
The Lilly Research Laboratories, Eli Lilly & Company	Unilever Research
Millipore Corporation, Waters Chromatography Division	Union Carbide Corporation
The Monsanto Company	Varian, Analytical Instrument Division

FORTHCOMING NMR MEETINGS

NMR Symposium at Washington University, May 24, 1989; See Newsletter 367, 42.

3rd Chianti Workshop on Magnetic Resonance Relaxation, May 28 - June 2, 1989; San Miniato (Pisa), Italy; Contact: L. Banci, Dipartimento di Chimica Bioinorganica, Università degli Studi di Firenze, Via Gino Capponi 7, 50121 Firenze, Italy. See Newsletter 362, 68.

9th International Meeting on NMR Spectroscopy, Sponsored by the Royal Society of Chemistry, July 10-14, 1989; University of Warwick, Coventry, England; Contact: Dr. John F. Gibson; (01) 437-8656; See Newsletter 364, 72.

NMR Spectroscopy In Vivo (Clinical Applications), July 10-12, 1989; Lyon France; Contact Prof. M. Amiel - see Newsletter 364, 73.

10th ISMAR Conference, July 16-21, 1989; Morzine (Haute-Savoie), France. (Note the newly announced location.); Contact: P. Servoz-Gavin, Departement de Recherche Fondamentale, Centre d'Etudes Nucleaires de Grenoble, B.P. 85X, 38041 Grenoble Cedex, France.

Fifth International Symposium on Magnetic Resonance in Colloid and Interface Science, August 7-11, 1989; See Newsletter 367, 57.

The Society of Magnetic Resonance in Medicine - Eighth Annual Scientific Meeting and Exhibition, August 12-19, 1989; Amsterdam, The Netherlands; Contact: The S.M.R.M. Business Office, 1918 University Ave., Suite 3C, Berkeley, CA 94704; (415)841-1899, FAX (415)841-2340.

Eastern Analytical Symposium, September 24 - 29, 1989; New York City; Contact: EAS, P. O. Box 633, Monchanin, DE 19710-0633; (302) 453-0785.

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. 369 (June) ----- 19 May 1989
No. 370 (July) ----- 16 June 1989
No. 371 (August) ----- 21 July 1989
No. 372 (September) ----- 18 August 1989

UNIVERSITY OF CALIFORNIA, DAVIS

BERKELEY • DAVIS • IRVINE • LOS ANGELES • RIVERSIDE • SAN DIEGO • SAN FRANCISCO



SANTA BARBARA • SANTA CRUZ

DEPARTMENT OF CHEMISTRY

DAVIS, CALIFORNIA 95616

Viscosity Effect on NOEs in Paramagnetic Proteins

March 3, 1989

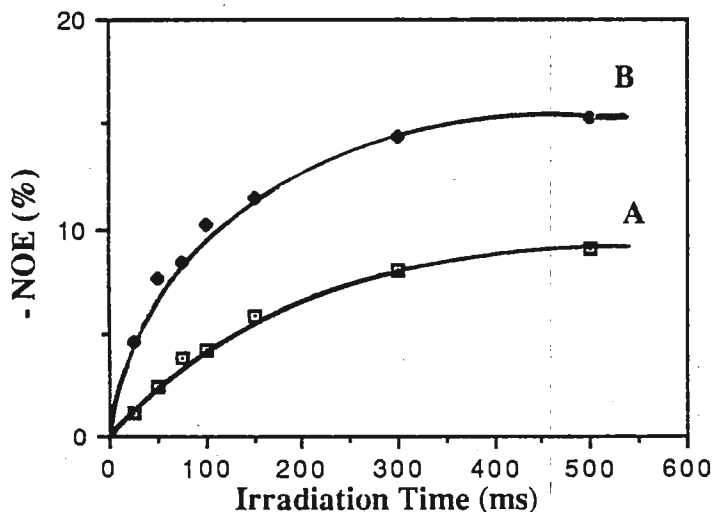
(received 3/11/89)

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

Dear Barry:

We have continued our study of the effect of solvent viscosity on the steady-state ($\eta = \sigma/\rho$) and truncated NOE ($\eta = -\sigma\tau$) of paramagnetic biopolymers. These studies, first reported in our last letter, were initiated to seek a basis for our qualitative observations that NOEs in paramagnetic proteins appear more useful in larger systems.

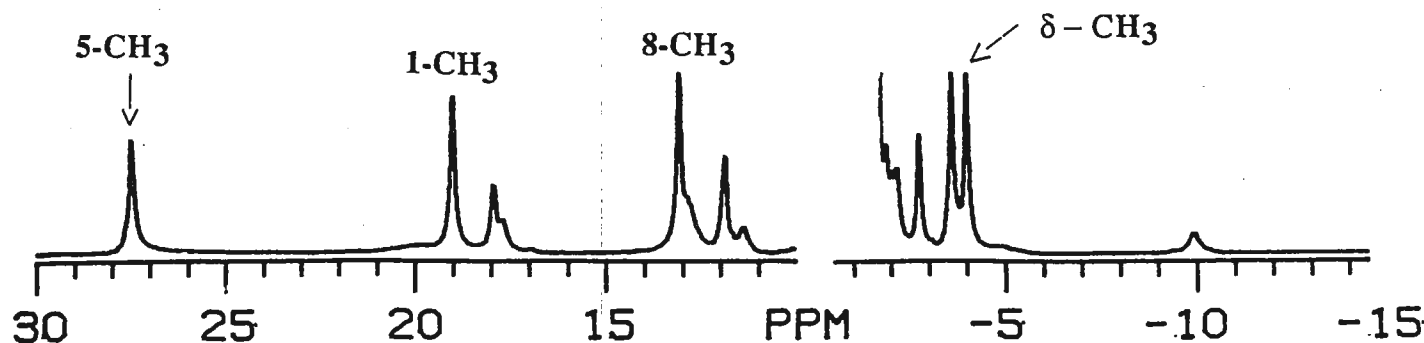
Our initial premise, that intrinsic relaxation, being dominated by a T_{1e} that is independent of molecular motions, appears to be confirmed by our observations for the low-spin ferric myoglobin complex. Hence, saturation of the 5-CH₃ peak leads to an NOE to the δ -CH₃ of Ile FG5/99. The time plots of the NOE shown here, in neat ²H₂O (A) and 30 % ethylene-glycol-d₈/70 % ²H₂O (B), show that the initial slope (σ) doubles in (B), corresponding to the doubling of viscosity. Moreover, the steady state NOE is also twice as large in (B), confirming that ρ is unaffected by viscosity. These results appear to rationalize our earlier observations of improved steady-state NOEs for isostructural functional groups in larger enzymes.

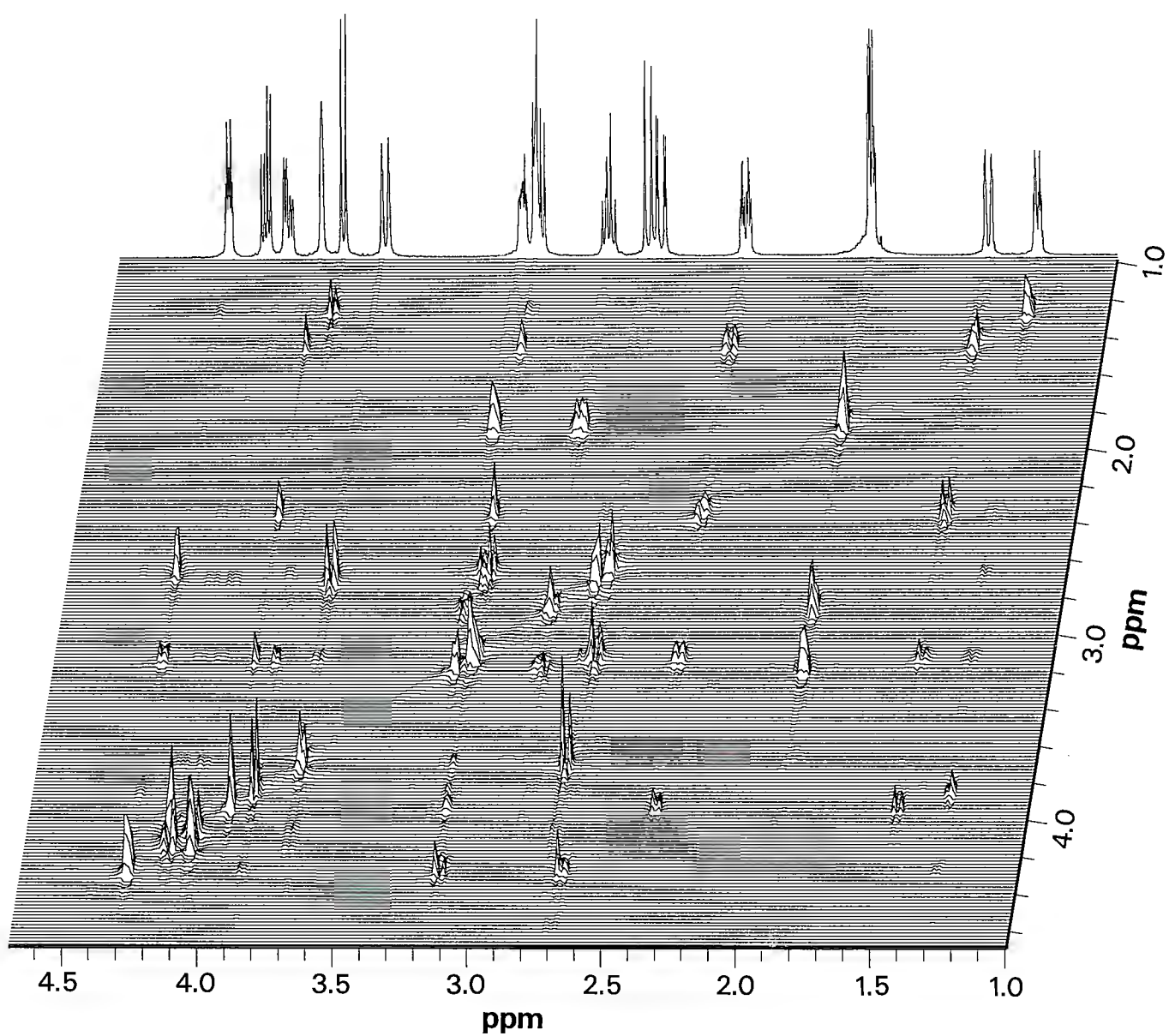


Best regards,

Gerd N. La Mar
 Professor of Chemistry

L.B. Dugad
 Research Associate



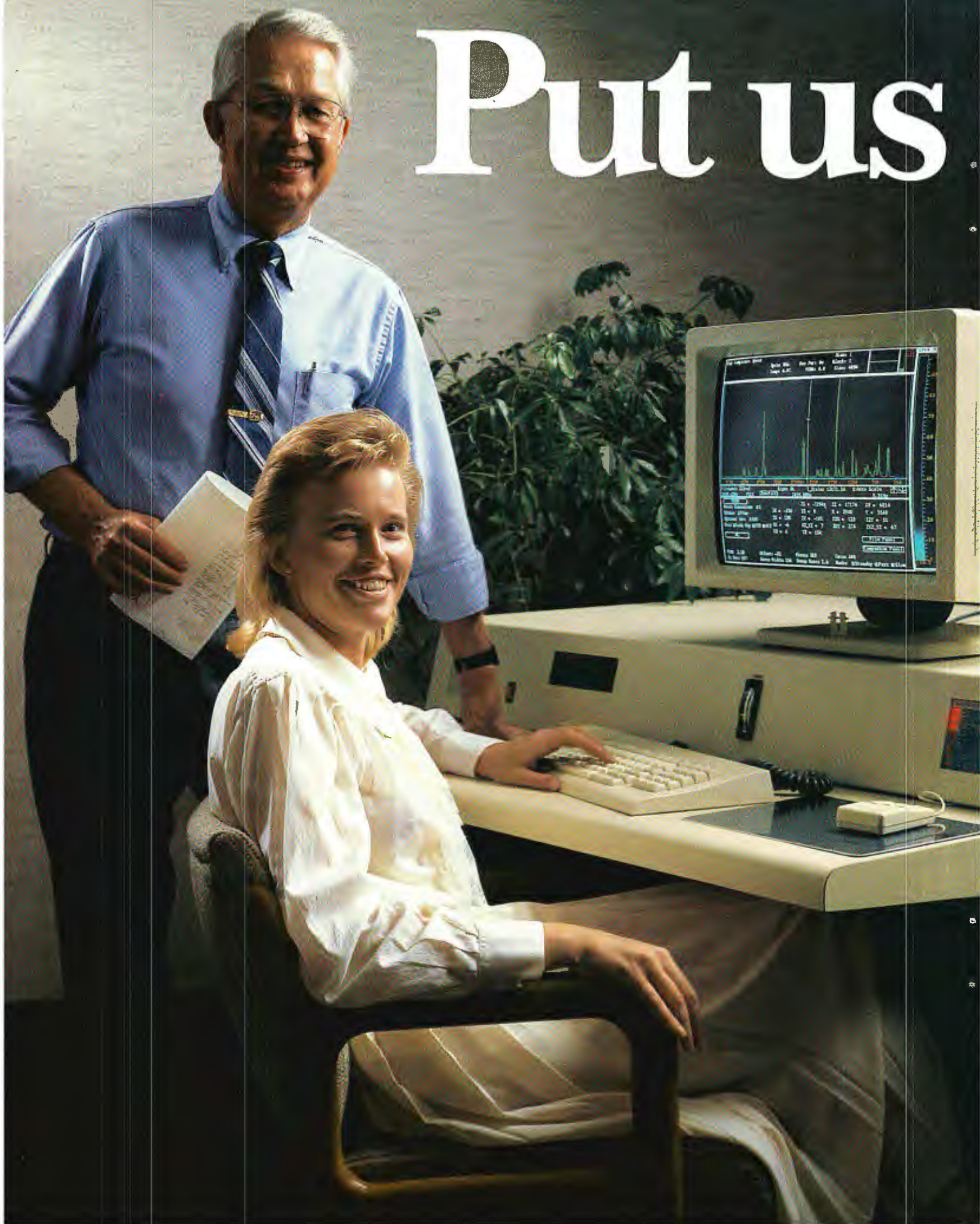


HOHAHA of Strychnine on an Omega 600



GE NMR Instruments

Put us

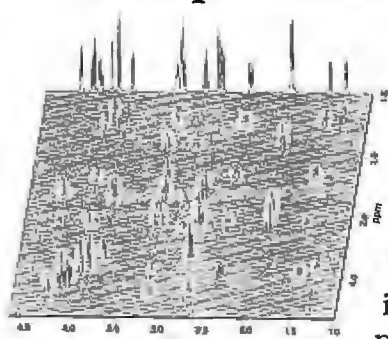


to the test.

There's only one way to be certain you're getting the best NMR system—test it yourself. Challenge its capabilities with your samples. Compare its results against your requirements.

See for yourself how GE spectrometer and CSI imaging systems measure up.

In RF performance



HDHAHA of Strychnine on an Omega 600

For outstanding RF stability in phase-sensitive work, inverse transfers and INADEQUATE experiments:

■ Amplitude stability of .043% in 90° pulse test of single acquisition in doped

water, repeated 10 times. Amplitude stability of .17% with 1 microsec. pulse.

■ Average deviation in 13° test of 0.5% in amplitude, representing stability of 0.07° in phase.

■ High-performance 200 kHz ADC with up to 32 MByte of 64 bit on-board memory for direct acquisition of experiments into memory.

In gradient control

The GE Acustar™ and Microstar™ shielded gradient systems improve image quality and localization by

eliminating eddy current effects for submillisecond settling times and better signal-to-noise performance. They also expand applications into oil core analysis, chemical toxicity testing, and monitoring of microscopic processes and reactions.

In data processing

GE opens NMR data processing and system operation to users at every level of expertise.

Mouse-directed panel menus let beginners use the system immediately. And programming designed by GE specifically for NMR applications lets experienced users attempt the most complex experiments.



In customer service and support

At GE, we're with you before and after the sale, with convenient financing packages and prompt service—as well as equipment upgrades, software updates and advanced applications.

To arrange a demonstration or for more information, write us today at 255 Fourier Ave., Fremont, CA 94539.

Or call 800-543-5934.

You'll be pleased with the results.



GE NMR Instruments

Alpha HDR

The New Standard in Digitizer Performance

Dynamic range vs. spectral width; spectral width vs. digital resolution. Trade-offs have been required due to NMR system hardware limitations. With the Omega™ Data system's Alpha HDR digitizer, no trade-offs are necessary. As shown in Figure 2 with a 16-bit dynamic range, 200 KHz spectral width, 64-bit complex acquisition word size, and up to 32 MBytes (4 MWords complex) of on-board acquisition memory available, the spectrometer is no longer the limiting factor when designing the most demanding experiments. Other outstanding features of the Alpha HDR include variable dwell periods, phase shifts of each sampled data point as small as 0.05 degrees, and segmentation of the digitizer memory into as many as 64K blocks. These features further distinguish the GN-series spectrometer equipped with the Omega Data System as the leader in NMR technology.

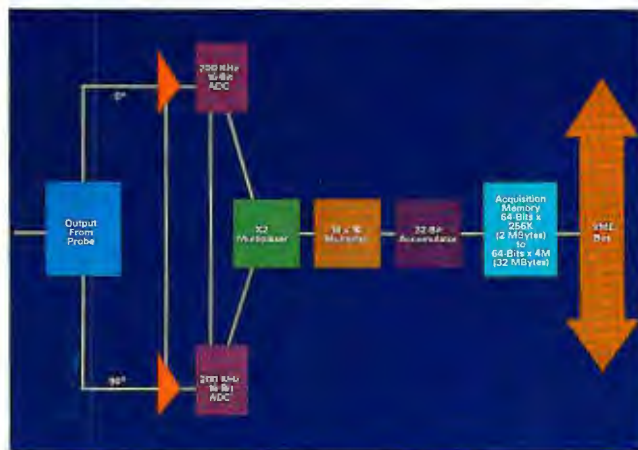


Fig. 1
The Alpha HDR digitizer.

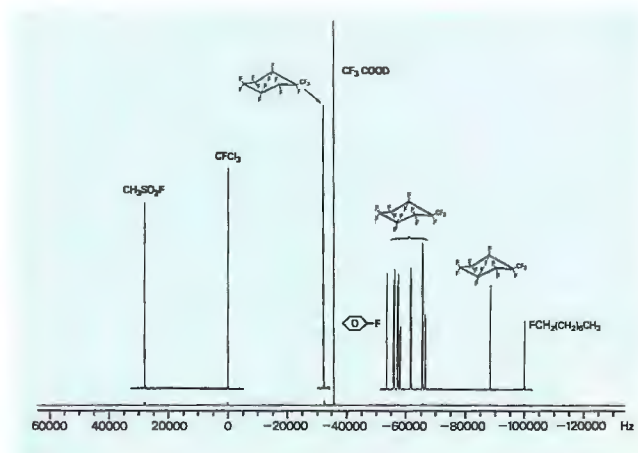


Fig. 2
200 KHz spectral width ¹⁹F spectrum acquired on a GN-500 Omega System. Note the extremely flat baseline obtained with the Alpha HDR.



GE NMR Instruments

255 FOURIER AVENUE, FREMONT, CA 94539
(415) 683-4408, TELEX 910 381 7025 GE NMR FRMT
PRAUNHEIMER LANDSTRASSE 50, D-6 FRANKFURT 90
WEST GERMANY 4969 760 7431, TELEX 041 2002 GEG

HUNTER COLLEGE

of The City University of New York

Department of Chemistry • 695 Park Avenue, New York, N.Y. 10021 • (212) 772-5330

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

(received 2/25/89)

Simulation of NMR Experiments by Embedded Density Matrix Operators **Part II***

Dear Barry,

As stated in Part I (TAMU, June, 1988), the set of basis functions of an N spin $1/2$ system can be defined such that the $2^N \times 2^N$ density matrix can be represented by character strings of N letters, with these letters corresponding to specific product operators. Because the symmetry of the nonzero elements in such matrices remains invariant under Jacobi diagonalization, the transformation between the starting basis set of simple product wavefunctions and the basis set of Eigenfunctions can be done through a very efficient book-keeping procedure on the character strings, rather than by time and memory-consuming matrix multiplications.

Using this method, we have (so far) been able to calculate all the energy levels and single quantum transition frequencies for a 14 spin system in which all spins are mutually coupled; this took 13 hours of CPU time on a μ -VAX II. The complete 1D spectrum for a 10 spin system with 45 coupling pairs was simulated within 2 hours of CPU time. With a less congested coupling network, larger systems could be simulated. Figure 1 shows the time needed for such calculations using both our program and the standard Jacobi method. (On our machine, the largest system which can be handled by the Jacobi method is for $N = 6$; for $N=7$, after 14 iterations (~1200 seconds of CPU time), the off-diagonal terms no longer decrease.)



Chen Wenqiao



Michelle Broido

*Two-dimensional simulations are "in progress" (the first simulation was completed as this was typed) and will appear in **Part III** or the ENC; the delayed submission of this (relative to the promise in **Part I**) tells us what we all know -- nothing goes as smoothly as one hopes.

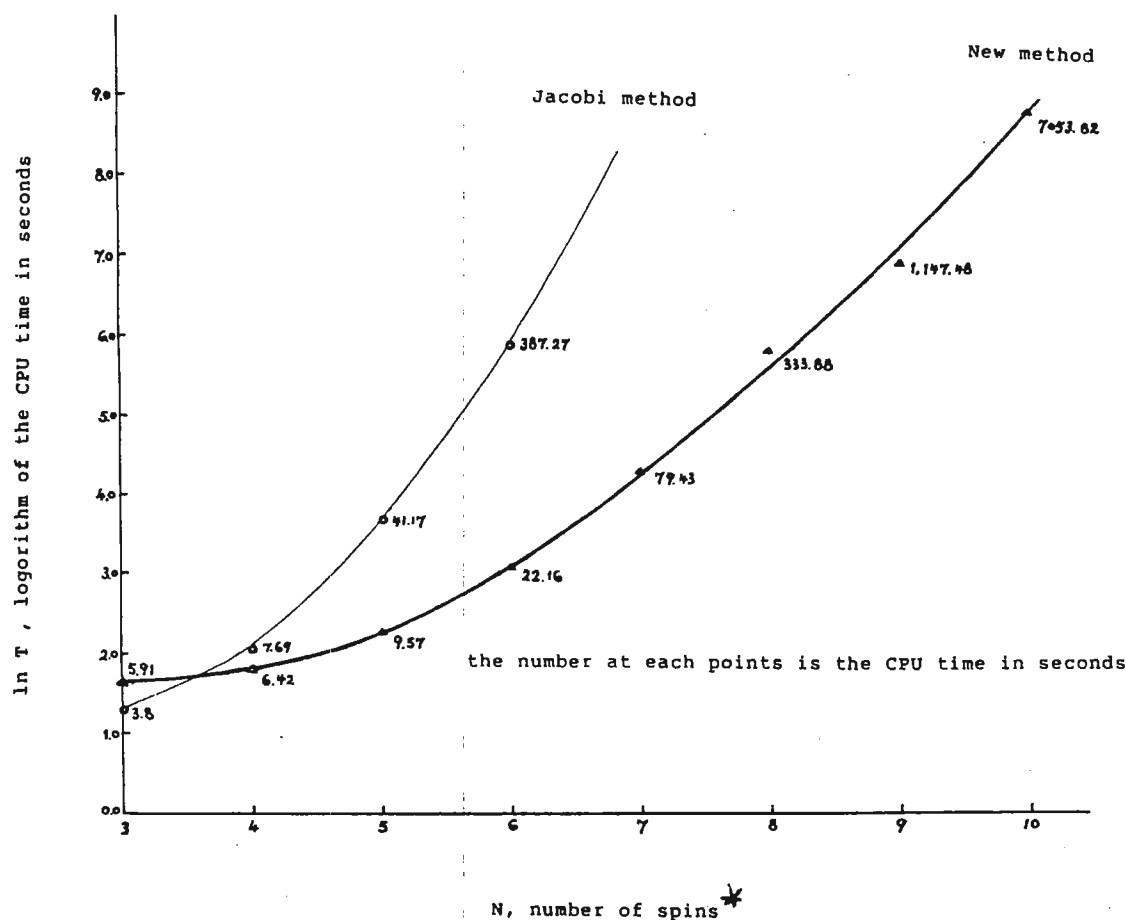


Fig. 1

* all mutually coupled.

The NMR Research Laboratory of the San Raffaele Hospital is looking for Post-docs with some experience in the "in vivo" NMR spectroscopy. The Laboratory is equipped with: multinuclear low resolution NMR (customized) spectrometer for relaxation studies, variable field, variable frequency; multinuclear high resolution Varian VXR 500S Spectrometer; and in vivo multinuclear SISCO 4.7T/330 Spectrometer. The Laboratory has the access to a Siemens Magnetom 1.5T for imaging and in vivo spectroscopy, to a Toshiba 0.5T for imaging and to a cyclotron/PET facility in the premise. Computational support is given by a vectorial main frame IBM 3090 and an IBM 4381. The main fields of interest of the NMR Research Laboratory are: Organ transplantation, diabetic pathologies, multiple sclerosis, sport medicine and physiology, and clinical chemistry. The San Raffaele hospital is a scientific institution for researches clinically oriented, is a 1300 bed hospital and is part of the University of Milano School. For more info on the facility and on the positions available please contact: Professor A. Boicelli, Director - Laboratori Ricerca NMR, San Raffaele Hospital, Via Oggettina, 60, 20132 MILANO (ITALY). Tel. 0039 - 2 - 2170.2706, Telefax 0039 - 2 - 2170.2482, Telex 326314 HSRAF I

SpecStation™ NETWORKING FROM NMRI

THE COMPUTER NETWORK THAT WILL GROW WITH YOU

DESIGN

- Turnkey networks designed utilizing your existing computers and spectrometers or adding new systems
- Currently running on Sun-3's and-4's, and VAXstation™ systems
- Currently porting software to Apollo, Ardent, Silicon Graphics, Stellar and Sun 386i
- SpecStation terminals starting at under \$2,500 (high-speed Ethernet multi-window SpecStation graphics)
- PC SpecStation network graphics servers will be supported later in 1989.
- Supports serial graphics terminals for other users on network
- Share access to common devices including PostScript™ and color printer/plotters
- Transfer data from *all* modern NMR and many FT-IR instruments

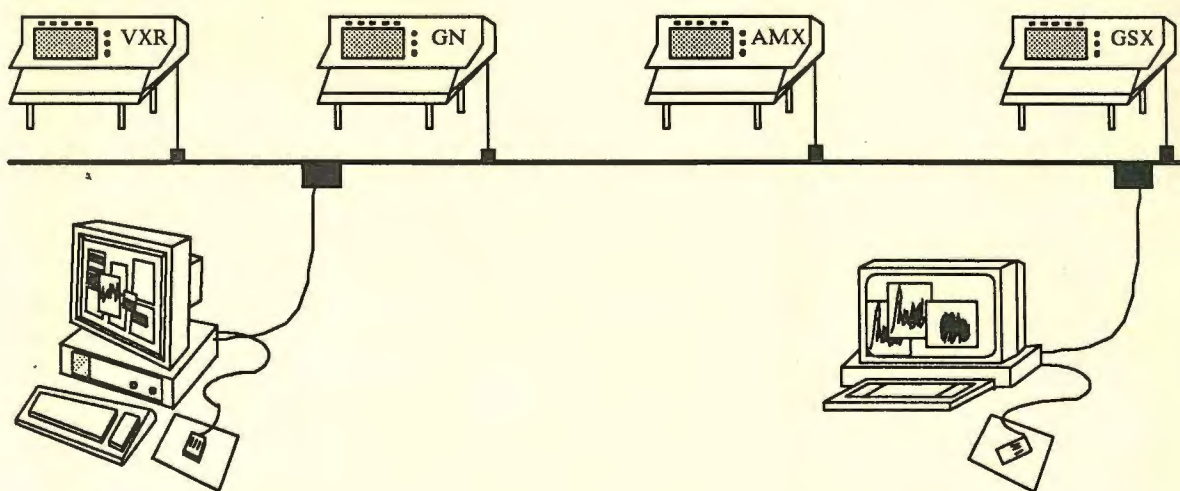
SOFTWARE

- 1D and 2D NMR, FT-IR, IMAGE and statistical analysis software available
- Process and analyze data at any SpecStation

CUSTOMER SUPPORT

- On-site installation and training available
- Continuing technical support and software upgrade service available
- 800 phone and modem customer support
- Chemistry Ph.D. Customer Support team committed to solving your problems
- 48 SpecStations installed in 1988

THE 100th SpecStation™ SYSTEM FROM NEW METHODS RESEARCH, INC. WILL BE DELIVERED THIS SPRING!



SpecStation™ COMPUTERS AND NETWORKS.

TURNKEY LABORATORY COMPUTER NETWORKING
AND SOFTWARE FROM NMRi;
OPTIMIZED FOR TODAY'S (AND TOMORROW'S) BEST
WORKSTATION COMPUTERS.

FOR BRUKER•CHEMAGNETICS•GE NMR•JEOL•VARIAN INSTRUMENTS

New Methods Research, Inc., 719 E. Genesee Street, Syracuse, New York 13210
(315) 424-0329 FAX (315) 424-0356

NMR2/Model, NMR2/IMAGE, NMR/MED, and SpecStation are trademarks of NMRi; VAXstation is a trademark of Digital Equipment; PostScript is a trademark of Adobe Systems. © NMRi 1989

Truman R. Brown, Ph.D.
Chairman, Nuclear Magnetic Resonance
and Medical Spectroscopy

7701 Burholme Avenue
Philadelphia, Pennsylvania 19111
215 728 3049
FAX 215 728 2822

March 8, 1989
(received 3/9/89)

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

Re: IDENTIFICATION OF SORBITOL-3-PHOSPHATE, IN THE MAMMALIAN LENS

Dear Dr. Shapiro:

About a year ago Dr. R.G. Gonzalez and co-workers published an intriguing observation about an increase in concentration of two novel phosphorylated metabolites in the lens of the diabetic rat (Gonzalez et al. Mag. Res. Med. 6:435-444, 1988). These compounds, while clearly phosphate monoesters resonate further downfield in the ^{31}P NMR spectrum (5.8 and 6.5 ppm), and have a lower pK_a (5.5 pH units) than any previously reported compounds of this class. In addition, in the ^1H coupled ^{31}P NMR spectrum they appear as doublets with an unusually high coupling constant of 10.5 Hz.

Because these metabolites were unidentified, and because in our laboratory we have made it a habit to identify phosphorylated metabolites in biological tissues we took up the "challenge." Unlike our previous identifications, however, where we were able to buy or synthesize the candidate compounds and confirm their identity by spiking, these metabolites did not correspond to any of the readily available compounds. Consequently we undertook a purification of the more abundant of the two metabolites (6.5 ppm) and after several false starts were finally able to isolate 1 mg of material from 75 g of calf lenses.

The identification of the material as sorbitol-3-phosphate was achieved by combination of NMR techniques such as 2D COSY (Fig 1) and ^{13}C spectroscopy (Fig 2), and confirmed by chemical synthesis of the compound from glucose. Presently we are in the process of identifying the second metabolite (5.8 ppm).

As suggested by Gonzalez, the presence of these metabolites in the lens and the increase in their concentration in the diabetic rats may indicate the presence of a new pathway of glucose metabolism in this tissue. Our identification of sorbitol-3-phosphate provides support for this hypothesis.

A more detailed description of our work is given in a forthcoming paper in J. Biol. Chem.

Please credit this contribution to the account of Dr. Truman Brown.

Sincerely

Benjamin S. Szwergold
Research Associate

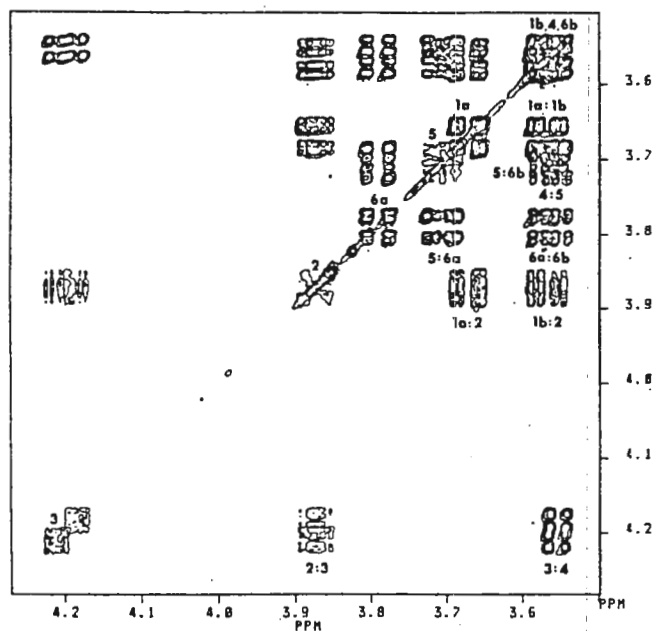


Fig.1 ^1H COSY spectrum of the 6.5 ppm unknown. Data was obtained as a 1024 x 1024 matrix with 64 scans per each time point; pulse width - 6 μs (60°), relaxation delay 3.75 s, SW2 = 700 Hz, SW1 = 750 Hz. Transformed in both dimensions using an unshifted sine-bell filter.

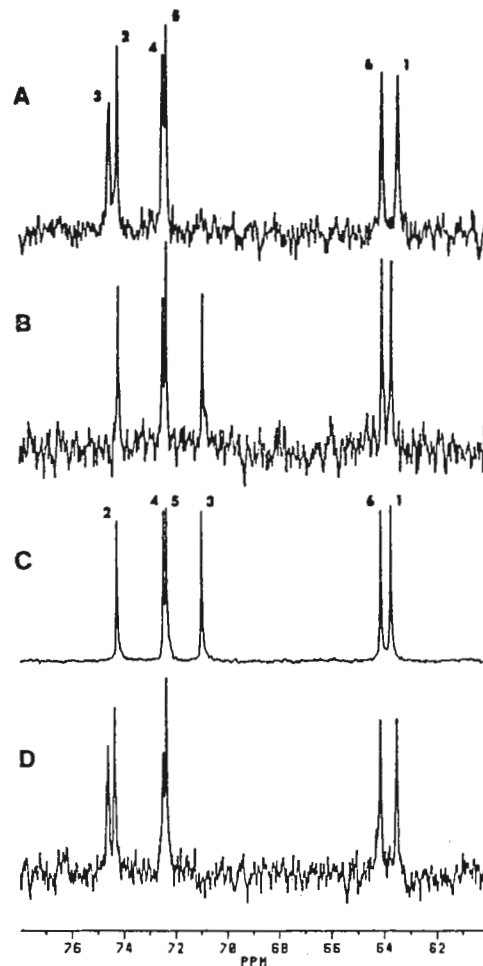


Fig. 2 A. Proton decoupled ^{13}C NMR spectrum of the 6.5 ppm unknown (pH 6.5). Obtained with 8 μs pulses, 1.4 s relaxation time, sweep width 5000 Hz, and 70,000 scans. Processed with a 5 Hz line broadening. B. Proton decoupled spectrum of the dephosphorylated material (parent compound of the 6.5 ppm unknown). Acquisition parameters same as above except for the number of scans; 40,000. C. Decoupled ^{13}C spectrum of sorbitol. Parameters same as above; 64 scans. D. ^1H decoupled ^{13}C spectrum of synthetic S3P. Number of scans = 3000, other parameters same as in A.

POSTDOCTORAL POSITIONS AVAILABLE

There are several postdoctoral positions available in the NMR Department at the Fox Chase Cancer Center. We have an ongoing substantial research effort on in vivo spectral localization, metabolic studies on tumors, tissue culture cells, lenses and NMR microscopy.

Interested individuals should inquire at the following address.

Dr. Truman R. Brown
Department of NMR and Medical Spectroscopy
Fox Chase Cancer Center
7701 Burholme Avenue
Philadelphia, PA 19111



Doty Scientific, Inc.

MAGIC ANGLE SPINNING

MAS PROBE FEATURES:

Available for all spectrometers.

Complete multinuclear range
using plug in capacitors.
Provides continuous coverage
from ^{31}P to ^{103}Rh .

High sensitivity and efficiency:
3 μs 90° pulses with 160 W
for ^1H at 300 MHz and 500 W
for ^{13}C at 75 MHz with 350 μl
sample volume.

Ultra high speed spinning option
5 mm spinning 14 kHz routine
17 kHz optimum.
7 mm spinning 9 kHz routine
11 kHz optimum.

Choice of 5 mm, 7 mm, and 19 mm
sample sizes.

Variety of rotors and spinner
assembly materials available.

Standard VT range:
-120 °C to 160 °C.

SPECIAL PROBE OPTIONS:

CRAMPS probes.

Fast Flipping Variable Angle
probes for DAS 30° to 90°.

Fiber optics spin detection.

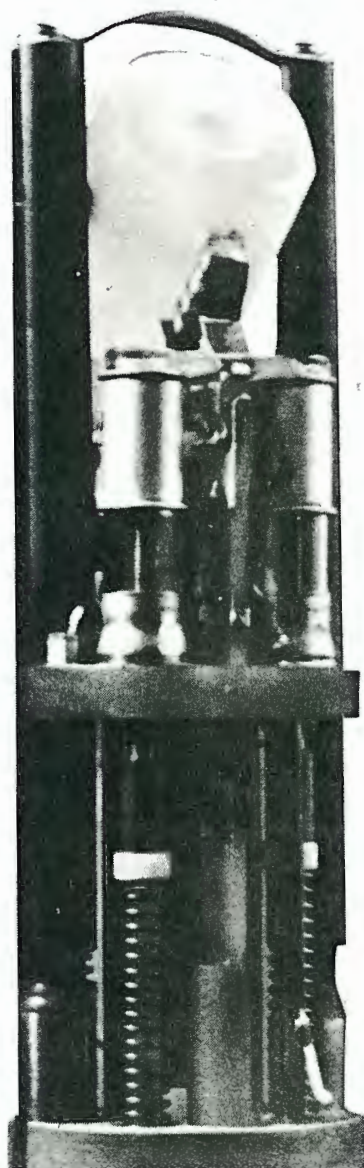
Triple tuned probes.

VARIABLE TEMPERATURE OPTIONS:

Extended temperature -180 °C to
250 °C on standard MAS probes.

High temperature MAS probes to
700 °C.

Low temperature MAS probes
to 6 K spinning, 3 K static.



DOTY SCIENTIFIC VT MAS PROBES

Note - Prices on this page do not include the necessary starter kits (page 5).

Domestic Prices
below 250MHz
\$ 13,000.

VT CP/MAS Double Tuned Probe for Supercon

Options:

Ultra high speed (5 mm - 14 kHz, 7 mm - 9 kHz) 3,500.

Multinuclear Observe Channel 2,500.

(Complete multinuclear capability
using plug in capacitor "wands", can
provide continuous coverage from ^{31}P
to ^{103}Rh in a single probe)

Capacitor wands (each) 200. to 600.

Adders for higher field probes:

250 MHz to 350 MHz 500.

360 MHz to 400 MHz 1,000.

500 MHz 1,500.

600 MHz 2,000.

Fast flipping variable angle (30° to 90°) 1,500.

Stepper Motor and controller for DAS 3,500.

(Variable angle not available on all probes)

Extra-narrow bore probes (37 mm) 800.

Extra-wide bore probes (over 73 mm) 1,000.

Triple tuned 2,000.

Top loading 1,500.

19 mm rotor diameter 2,500.

Fiber optics spin rate monitoring 1,000.

(Not on CRAMPS or variable angle probes)

Extended VT on standard narrow bore probes 2,000.

Extended VT on standard wide bore probe 1,500.

(Standard range is -120 °C to 160 °C,

Extended VT range is -180 °C to 250 °C)

VT CP/MAS Probe for Electromagnet with ext. ^2H lock 14,000.

Additional Features:

Multinuclear 1,500.

Triple Tuned 2,500.

CRAMPS Probe: Single tuned VT probe ^1H or ^{19}F
8 kHz 5 mm MAS, 1.5 μs 90° pulses 10,500.

High Temperature MAS Multinuclear Probe:

-100 °C to 1700 °C (7 mm only)

Single tuned probe 23,000.

Double tuned probe 26,000.

Low Temperature MAS Multinuclear Probe:

2 K to 400 K (5 mm only)

Double tuned probe 33,000.

LOVELACE MEDICAL FOUNDATION

2 March 1989

Bore tube conversion on an old Oxford horizontal magnet.

Dear Professor Shapiro,

We have a 1.9 T Oxford 30 cm diameter horizontal bore superconducting magnet (Model B26694) which has been in continuous use for 4.5 years. Recently, an Oxford engineer performed several operations which have significantly improved the magnet. I describe these operations and results so that they may benefit others with the same magnet.

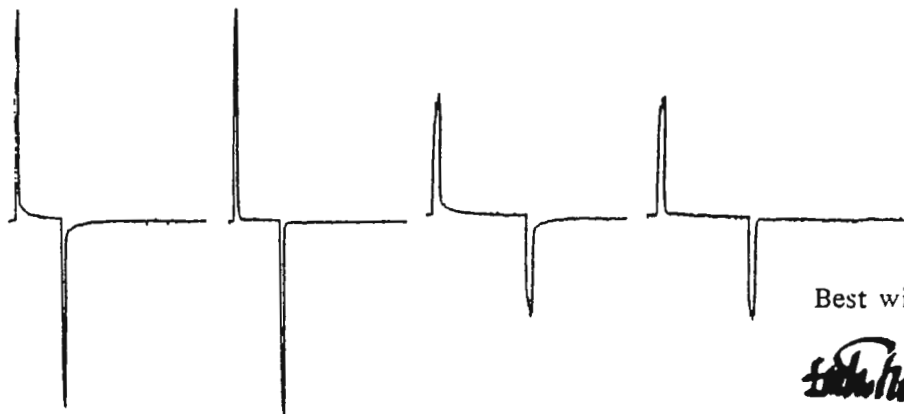
One of the problems we have had is the eddy current which alters the switched magnetic field gradients. We had tried several cures which offered partial solutions (smaller gradient coils, reshaping the driving pulse according to the difference between the actual gradient time dependence and the driving pulse, etc.) which led us to the conclusion that we should combine as many solutions as possible for the best results. Therefore, we ordered a conversion from the old aluminum bore-tube to a fiberglass one.

This conversion has made a significant difference in the performance (or shall we say, correctability) of the switched gradients, especially the rapidly changing components. The before/after figures below show the time-derivative of the field in the magnet for 5 and 10 ms z-gradient pulses from a Nalorac shim/gradient coil with an inside diameter of 25 cm and a switched gradient of 1.5 gauss/cm.

Another problem we had after 3.5 years was a small leak in the vacuum jacket which caused us to pump the vacuum jacket once a month. The engineer replaced all the o-rings on the magnet and installed a charcoal sorb in the vacuum space. We also discovered that there was no insulation in the vacuum space between a hatch (intended for a refrigerator) and the nitrogen can. This hatch exists only on old Oxford magnets and is a plate 14.4 cm in diameter bolted to a flange on the top center of the magnet. Sixteen sheets of aluminized mylar were cut to size and stuffed into the hole under this plate. The liquid nitrogen consumption has been reduced by about 1/3 and we now refill (from a 160 liter dewar) every ten days instead of the one week refill interval we used before. The liquid helium consumption has also diminished and we now go six weeks between ordering compared with the previous four. (We fill the dewar and then use up the remainder of the 100 liters to top-up one week later.)

An welcome bonus was that the Oxford engineer shimmed the superconducting shims better than we used to be able to shim the room temperature shims with a large (4.5 cm dia. x 9 cm long) sample. The improved shim (ca. 1/8 ppm on the 4.5 cm dia. cylindrical phantom of water which is 4 or 5 times better than before!) has allowed us to do many experiments more easily compared to the past.

We now have an extra bell-housing which was (is) attached to the old bore tube that we will sell (cheap) to anyone who needs it. Oxford does not stock this item so that you would normally have to pay Oxford to make you one for the bore tube conversion (unless you are willing to have your magnet down for several weeks while Oxford attaches the old housing to the new bore tube at Oxford) and then end up with your old, useless one (like we did).



Best wishes,

Eiichi Fukushima, Paul D. Majors

DOW CORNING U.S.A.

February 13, 1989
(received 2/22/89)

MIDLAND, MICHIGAN 48640
Telephone: (517) 496-4000

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

50 CM PLOTS ON QUIETJET PRINTERS

Dear Dr. Shapiro:

Thank you for your very kind pink reminder. I offer a tip which I hope will be useful to Varian owners with Sun data systems as well as preserve my subscription.

I was recently forced to rely on my HP QuietJet printer for output of spectra as well as text. I found that the Varian formats for graphic output did not work properly. The QuietJet_192 format only plotted the left half of the display and rotated plots were non-existent in the QuietJet format. A phone call to Varian yielded the information that the ThinkJet formats should work for the QuietJet and that the _96R and _192R formats were for rotated plots but that plots longer than one page were not available. After some experimentation, I found that the ThinkJet_192R format only plotted the left half of the display but that the ThinkJet_96R format works well. It can be simply formatted to produce 50 cm plots by editing the vmnr/devicenames file. Change wcmamax = 500.0 and wc2maxmax = 180.0 and make a new printcap (see Varian manual). To produce long plots, set wcmaw=wc=500 and wc2max=wc2=180. Plot dimensions are now determined as usual but with a 50 cm maximum length.

This plotter gives very acceptable quality plots and is quicker and cheaper than the HP-7550A. I am now using it regularly for routine output.

Best Regards,

Dick

Richard B. Taylor, PhD

POSTDOCTORAL POSITION

A postdoctoral position is currently available in our laboratory applying multinuclear solid- and solution-state NMR methods to the study of guest-host interactions between clay minerals (naturally occurring zeolites) and various substrates. The structure, dynamics, and thermodynamics of the adsorbed or intercalated species will be examined. The appointment is for one year and is renewable for a second year.

Our laboratory is equipped with Bruker MSL - 300 (solids/solutions), Jeol GX-400, and Jeol FX-60Q spectrometers. Excellent facilities and resources are available for molecular modelling, computations, and graphics.

Interested applicants should send a resume as soon as possible to:

Dr. Walter V. Gerasimowicz,
USDA, ARS
Eastern Regional Research Center
600 East Mermaid Lane
Philadelphia, PA 19118
(215) 233-6439

A Siemens and Varian joint venture. You benefit from single-source access to systems combining Siemens expertise in clinical MR imaging with Varian's leadership in high-resolution NMR.

Only *Spectroscopy Imaging Systems* gives you both the resources of a large corporation and the innovative responsiveness of a dedicated company. You also receive the combined expertise of site planners, system engineers, technical support personnel, and application scientists.

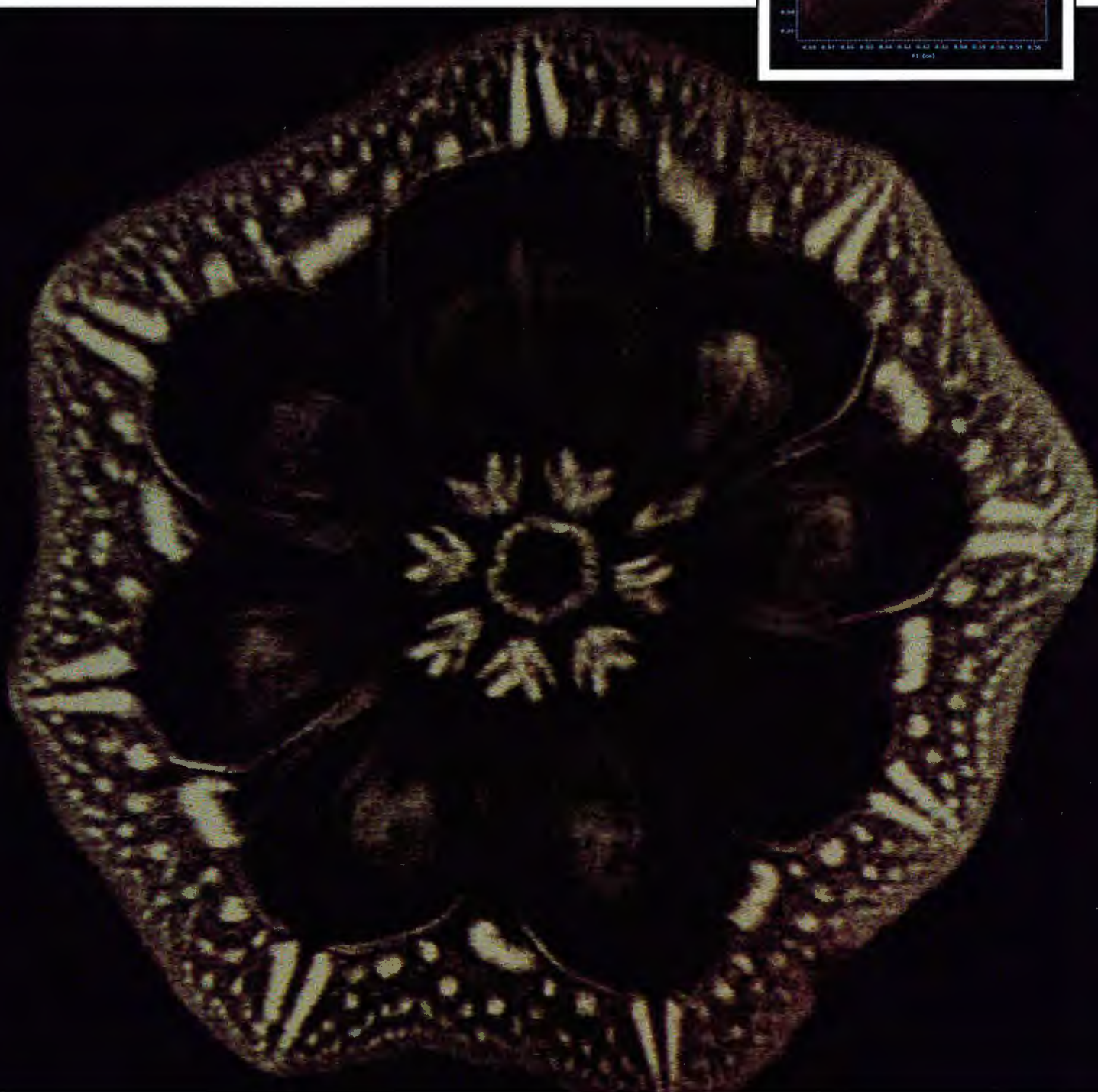
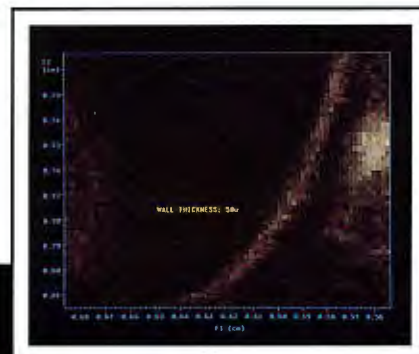
For systems, service and support in imaging spectroscopy, call 415-659-2600 today. Or write to 1120 Auburn Street, Fremont, California 94538.

Now... Explore Higher Performance NMR Imaging!

Spectroscopy Imaging Systems Corporation

A Joint Venture of Varian and Siemens

(415) 659-2600

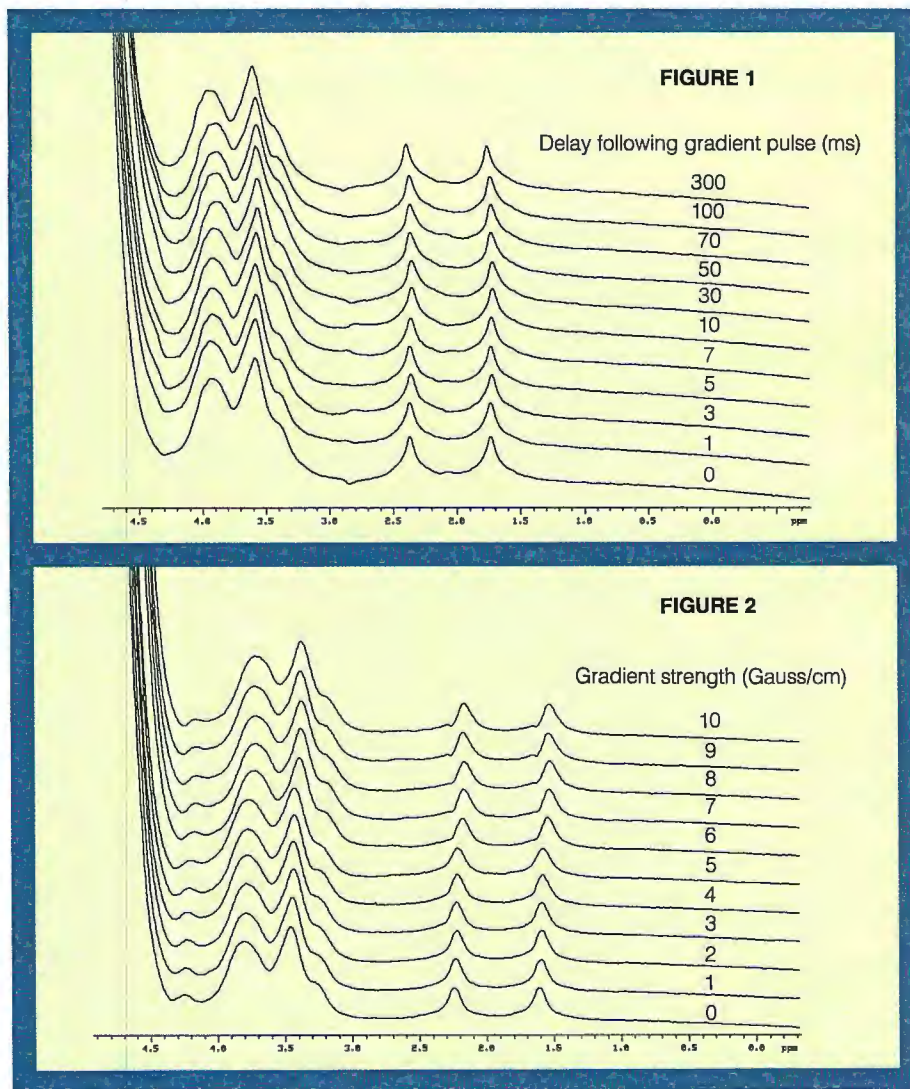


High Performance Auxiliary Gradient Coils

Dedicated RF coils, optimized for a specific application or a particular sample or both, have long been a tradition in NMR imaging as well as NMR spectroscopy. Similarly, dedicated gradient coils yield dramatic performance improvements in terms of rise and fall times, maximum gradient strength, duty cycle and eddy currents. Sophisticated eddy current compensation techniques reduce any residual spatial or temporal field variations due to eddy currents to a few hundredths of a percent, thus yielding distortion free spectra and images.

At Spectroscopy Imaging Systems we are able to obtain in-plane resolution of $20\mu \times 20\mu$ in an axial cut through an okra of 1.3 cm diameter. (See image shown on front side.) An enlargement of a portion of a seed capsule within the okra displays a wall thickness of 50μ (inset on front side). The gradient coil used to obtain this excellent performance has a diameter of 125 mm and was mounted in an Oxford Instruments 4.7 Tesla, 330 mm bore magnet without removing the standard set of gradient coils. The auxiliary gradient set is capable of generating greater than 10 Gauss/cm in all orientations using the same gradient power supply as the primary gradient coil.

The excellent control of eddy currents using the smaller gradient insert is demonstrated in Figures 1 and 2. Figure 1 shows a series of spectra of the high field region of a sample containing $[2-^{13}\text{C}]$ acetate and $[1-^{13}\text{C}]$ glucose in $\text{H}_2\text{O}/\text{D}_2\text{O}$. With the sample in the center of the gradient coil, a 1 second gradient pulse of 1 Gauss/cm was applied. The gradient pulse was followed by a variable delay, a 90°

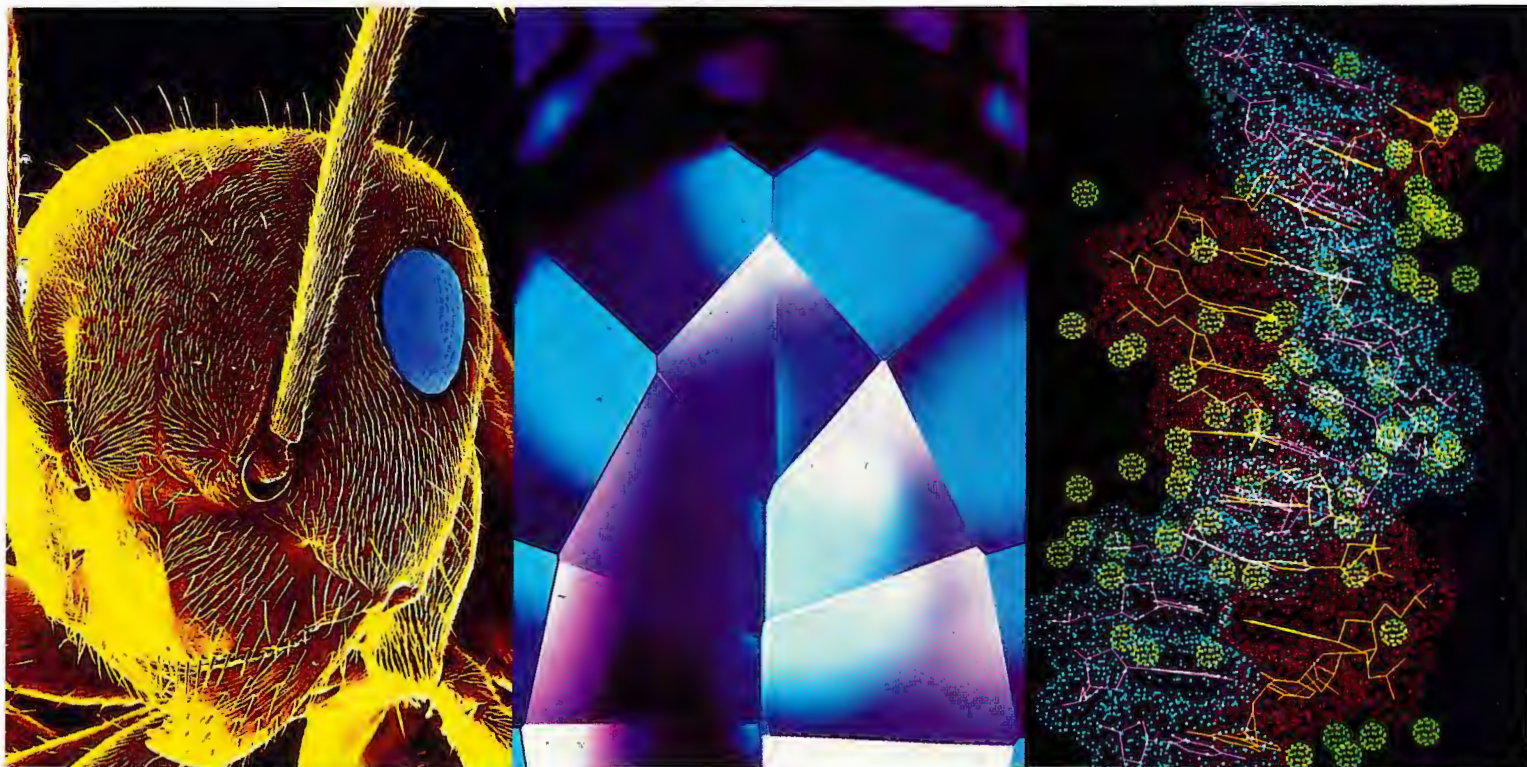


RF pulse and acquisition of the resultant free induction decay. All spectra are essentially undisturbed even at delay times below 10 ms.

Figure 2 shows a typical series of spectra of the same sample at an off-center location in the magnet. A gradient pulse of 5 ms duration with an amplitude range of 0 to 10 Gauss/cm was applied, and the

FID recorded at a fixed delay time of 3 ms. The sample was positioned at 22.3 mm off the center, corresponding to local gradient field of 95 kHz. All spectra are virtually undisturbed, even at 10 Gauss/cm.

VARIAN NMR FLEXIBILITY NOW RESOLVES PROBLEM DIVERSITY



MICROIMAGING

SOLIDS

LIQUIDS

UNITY lets you switch from one to the other with ease

Combine high performance capabilities with unparalleled flexibility using Varian's new UNITY™ NMR spectrometer. This unique spectrometer is a true, multi-capability instrument that performs high resolution microimaging as easily as it analyzes liquid and solid samples.

UNITY's revolutionary system architecture employs a modular design that addresses all NMR applications with a single instrument.

Analyze liquid samples using a variety of techniques over a wide range of nuclei. Perform CP/MAS, wideline and multipulse for solid samples. Examine microimaging samples with ease. Maximum flexibility has been built in to cover future experimental capabilities for every application.

Resolve problem diversity: invest in the most flexible technology of today to better address the research of tomorrow. Invest in a UNITY NMR spectrometer. For additional information, please call **800-231-8134**. In Canada, call **416-457-4130**.

Varian is your full-line
company for analytical
instrumentation

UV-Visible-NIR

LIMS

Atomic Absorption

NMR

Liquid Chromatography

Gas Chromatography

NMR WITH A FUTURE

varian 

MICHIGAN STATE UNIVERSITY

MAX T. ROGERS NMR FACILITY

CHEMISTRY BUILDING • EAST LANSING • MICHIGAN • 48824-1322

February 23, 1989
(received 2/28/89)

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

MAX T. ROGERS MSU NMR FACILITY:PROGRAM FOR PLOTTING VARIAN VXR SPECTRA ON APPLE LASERWRITER II

Dear Barry:

First of all, one of us (J.P.Y.) would like to note a change of address. I have moved from Caltech to join the Chemistry faculty at Michigan State University, and to head the Max T. Rogers NMR Facility (named in honor of the late Professor Rogers, a long-time pioneer in magnetic resonance at MSU). In addition to the current instrumentation, a Bruker WH-180 equipped for solids and a Bruker WM-250 with an Aspect 3000 computer, we are having installed before summer four Varian spectrometers: a Gemini 300, a VXR-300, a VXR-400 devoted to solid state studies, and a VXR-500. The three VXR spectrometers will have Sun 3/60 computers networked together and to our existing Sun 3/160 work station.

Since we already had an Apple Laserwriter II on the workstation, we wanted to be able to use it for plotting spectra. One of us (K.J.) has modified for use with the Varian software an existing public-domain program (*hpgl2ps*) that converts the language used to drive Hewlett-Packard plotters (HPGL) to the language used to drive the LaserWriter (PostScript). This program (or filter), which is written in C and is about 2000 lines long, is easily interposed between the output of the *Vnmr* data acquisition program and the spooler (*lpr*) used to drive the LaserWriter by a simple modification of the *Vnmr* shell script */vnmr/bin/vxrplot* and some other printing and plotting related tables (i.e. */etc/printcap/vnmr/devicenames*, etc.). A similar set of modifications to */vnmr/bin/vxrprint* can enable the LaserWriter to function as the system printer as well. VXR Series S users can obtain full information and the modified *hpgl2ps* by sending us either a 1/4" SUN-style cartridge tape to the above address or a mail message containing an INTERNET or BITNET address where the program and information can be electronically mailed to kermit@horus.cem.msu.edu (INTERNET) or kermit@msucem (BITNET).

Yours sincerely,



James P. Yesinowski

(517)-353-9412



Kermit M. Johnson

(517)-353-5115



Lawrence Livermore National Laboratory

February 22, 1989

(received 2/25/89)

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

Resorcinol-Formaldehyde and other polymers

Dear Barry:

We received a Bruker MSL-300 last April and have been learning its idiosyncrasies ever since. As with the best of plans, our new laboratory was not complete until the end of summer so the magnet was de-energized and moved cold to the next wing of the building. After re-energizing, the magnet was reshimmed and specs were met on three liquid state and one MAS probes within less than a week. This time the resolution was a factor of two better than that initially obtained! We have had little trouble with the system and are quite pleased with it. I should say that one comment that has been made is if you are going to jump back and forth between solids and liquids and many different nuclei you better know what you are doing -- in many ways a reasonable request.

We are interested in low density foam material and have found the MSL quite suitable for these studies. Resorcinol and formaldehyde react in the presence of a basic catalyst to form a polymer of low density and high transparency. Of particular interest are reasons for the variation in densities and therefore the condensation mechanism since "the major characteristics of gels (density, pore size distribution, homogeneity, mechanical properties) always depend on the initial stages of polymerization"¹.

¹Powxviel, J.C., Boilot, J.P., Beloeil, J.C., and Lallemand, J.Y., "NMR Study of the Sol/Gel Polymerization". J. Non-Crystalline Solids 89 (1987) 345-360.

As the condensation proceeds, initial products can be observed by high resolution C-13 NMR to form and then disappear. At this stage of the reaction the solution is not particularly viscous nor are there signs of gel formation. At this time no spectrum is observed by high resolution C-13 NMR techniques for solutions as high as 20 wt% in resorcinol. CPMAS studies of the gel, before it has undergone extensive washing and processing under supercritical conditions, reveal the presence of unreacted formaldehyde and broad featureless peaks with the polymeric spectra superimposed. The CPMAS spectra of the final low density crystalline-like product appear in fig.1 The densities of the final products are 58 (A) and 90 mg/cc (B) respectively. These spectra and the signal strength as a function of contact time do not suggest any parameters that can be related to the differences in density. One suggestion is that beads of polymeric material are formed and then linked to varying degrees by further reaction with formaldehyde. We are currently performing experiments

WIDE LINE
NMR
CRAMPS
VTMAS
IMAGING
2D-NOE

NMR Dream Machine.

How many things can a spectrometer do, and do them well?

At Bruker, we've always believed that the scope of an NMR investigation should be limited only by the spectroscopist's imagination, not by the instrument. That's why we designed the MSL.

The MSL is our flagship for NMR applications in solids, liquids and imaging which require unparalleled range and versatility—without compromising performance or simplicity of operation. Therefore, the design of the MSL is based on an open architecture which makes it highly adaptable to new experiments and modifications. In fact, we are often pleasantly surprised to find MSL users routinely performing experiments which even we never dreamed of. Perhaps this is why the MSL has been the

unquestioned market leader in its field for more than four years.

The choice is yours.

MSL spectrometers are available either with a 90 MHz electromagnet or with a 100, 200, 300, 400 or 500 MHz superconducting magnet. They offer vertical cryomagnet bore diameters from 89 to 150 mm. The unique MSL System Process Controller lets you generate virtually

every experiment you've ever dreamed of, and together with its battery of distributed microprocessors, controls every function and parameter of the system.

All of this makes the MSL the ideal research spectrometer for everything from catalysts to coal, polymers to proteins, rocks to rats.

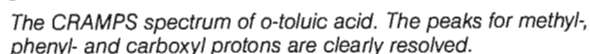
So, stop dreaming and call your nearest Bruker sales representative for more information or a demonstration. Once you've seen the MSL in action, it may even convince you that dreams can come true.



Bruker Instruments, Inc.
Manning Park, Billerica, MA 01821
In Europe: Bruker Analytische
Messtechnik GmbH, Silberstreifen
D-7512 Rheinstetten 4, W. Germany

Analytical Systems Worldwide

3



Australia: BRUKER (Australia) Pty. LTD., Earlwood, New South Wales, Tel. 02-5589747
Belgium: BRUKER SPECTROSPIN S.A./N.V., Brussels, Tel. (02) 648 53 99
Canada: BRUKER SPECTROSPIN LTD., Milton, Ontario, Tel. (416) 876-4641
England: BRUKER SPECTROSPIN LTD., Coventry, Tel. (0203) 855200
France: SADIS BRUKER SPECTROSPIN S.A., Wissembourg, Tel. (088) 94 98 77
India: BRUKER INDIA SCIENTIFIC Pvt. LTD., Andheri (West), Bombay, Tel. 22 62 72 32
Italy: BRUKER SPECTROSPIN SRL, Milano, Tel. (02) 23 50 09, 2 36 40 69
Japan: BRUKER JAPAN CO. LTD., Ibaraki, Tel. 0298-52-1234
Netherlands: BRUKER SPECTROSPIN NV, Wormer, Tel. (75) 28 52 51
Scandinavia: BRUKER SPECTROSPIN AB, Åkersberga, Sweden, Tel. (07 64) 6 80 60
Spain: BRUKER ESPANOLA S.A., Madrid, Tel. 341-259-20-71
Switzerland: SPECTROSPIN AG, Fällanden, Tel. 1-82 59 111
W. Germany: BRUKER ANALYTISCHE MESSTECHNIK GMBH, Rheinstetten, Tel. 0721-5161-0
 BRUKER ANALYTISCHE MESSTECHNIK GMBH, Karlsruhe, Tel. 0721-5967-0
 BRUKER-FRANZEN ANALYTIK GMBH, Bremen, Tel. 0421-8700-80
USA: BRUKER INSTRUMENTS, INC., Billerica, MA 01821, (508) 667-9580
 Regional Offices in Chicago/IL, Wilmington/DE, Houston/TX, San Jose/CA

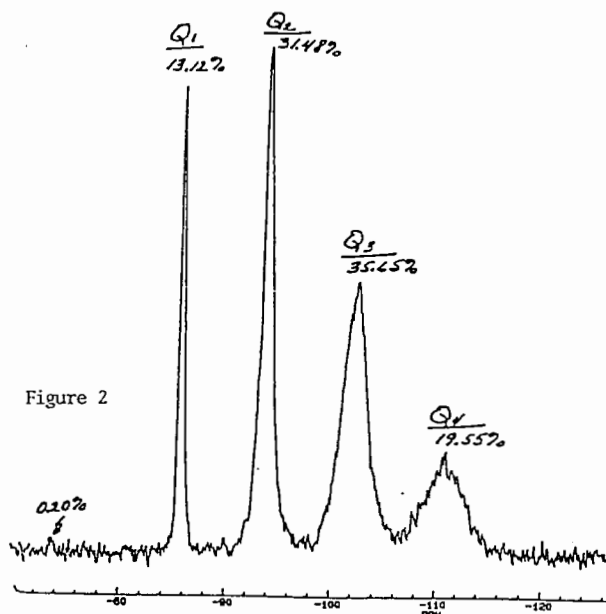
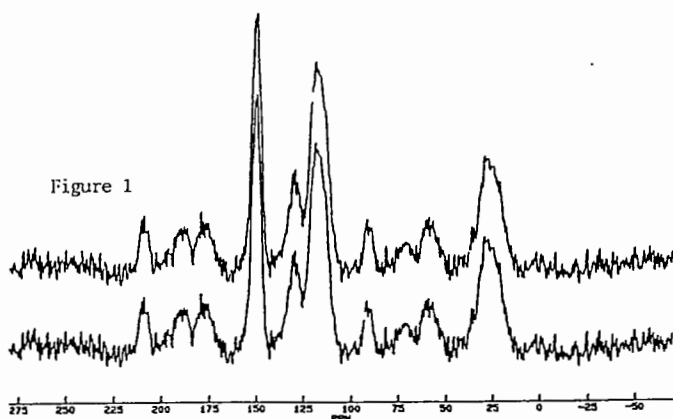
where the reaction mixture is spiked with C-13 labeled formaldehyde at various times during the reaction period. We hope that this might lead to information relevant to the difference in densities.

We are also interested in the production of silicon aerogels by the hydrolysis of tetramethoxysiloxane (TMOS). We have studied the hydrolysis of TMOS by high resolution Si-29 liquid state NMR and determined the distribution of Silicon between $(RO)_nSi(OSi)_m$ species where $n+m=4$. Values of m are 0,1,2,3, or 4 and designated Q_m . In the liquid state each environment is further distinguished by the presence of multiple peaks. After a period of time, gelation usually occurs and no further high resolution data can be obtained. A number of samples that had gelled as long ago as 15 months were examined by Si-29 MAS techniques with proton decoupling. We anticipate that spectra such as fig. 2 will define variations in species distribution as ageing progresses.

Sincerely,

Ray Ward
Raymond L. Ward

Jim Happe
James A. Happe



Back Issues, Anyone?

A limited supply of back issues of the Newsletter is available, for a limited time. Before very long, I will need to make space by throwing out some of the older issues, so now is the time to see if you need some replacements, birthday gifts, Beethoven's birthday/Christmas/Chanukah presents, etc. Very reasonable prices, especially if I don't have to issue invoices, deal with government purchasing offices, etc. Quantity discounts available. Please enquire.

B.L.S.



CH-3012 Bern, Freiestrasse 3
Telefon 031 654311

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

7.3.1989
(received 3/11/89)

HOMONUCLEAR DECOUPLED COSY SPECTRA

Dear Professor Shapiro

Highly resolved H/H COSY spectra have meanwhile proven to be an invaluable tool not only to establish coupling networks of complex molecules, but furthermore to evaluate chemical shifts and coupling constants in a straightforward manner. One of the problems is certainly the (sometimes too) high information content of the highly resolved cross peaks and the partial decrease in sensitivity due to unresolved small couplings in COSY spectra if acquired in the phased mode. In this context we tried to simplify the structure of cross peaks by selectively decoupling protons in both dimensions. Whereas this procedure is straightforward in the second time domain (t_2), with the irradiation of only one proton, decoupling in the first time domain (t_1) may lead to additional problems especially in the case, where not only one, but a few protons should be decoupled simultaneously.

As an example we acquired the COSY spectrum of eugenol with a modified COSY sequence (Fig. 1) -including double quantum filtering and the possibility to record phased spectra- on our BRUKER AM 400 spectrometer. A few selective 180 degree pulses P_2 (5 ms, $S_1=10L$), spaced by Δ (2 ms) to change between various offset frequencies $f_1 \rightarrow f_N$, are applied in the middle of the evolution period. Normal (pulsed) homodecoupling at f_N is accomplished during the detection period. Fig. 2 shows an expansion of the 2D contour plot in the region of the 9 and 9' protons of eugenol. Whereas for Fig. 2a the methylene protons at C(7) are decoupled in both dimensions, giving a simplified but symmetric cross peak, an additional 180 degree pulse is applied at $t_1/2$ for Fig. 2b to selectively decouple both the methylene protons at C(7) and the methine proton at C(8) in the first dimension. This leads to a further data reduction in the first frequency domain (only the coupling interaction $J_{H(9),H(9')}$ is left). To compensate for the delay introduced in the middle of the evolution period the acquired data has to be shifted in t_1 by $(P_2 + \Delta) \times N / 2 \times IN$ (with the t_1 increment IN) data points for proper phasing.

Yours sincerely

Peter Bigler

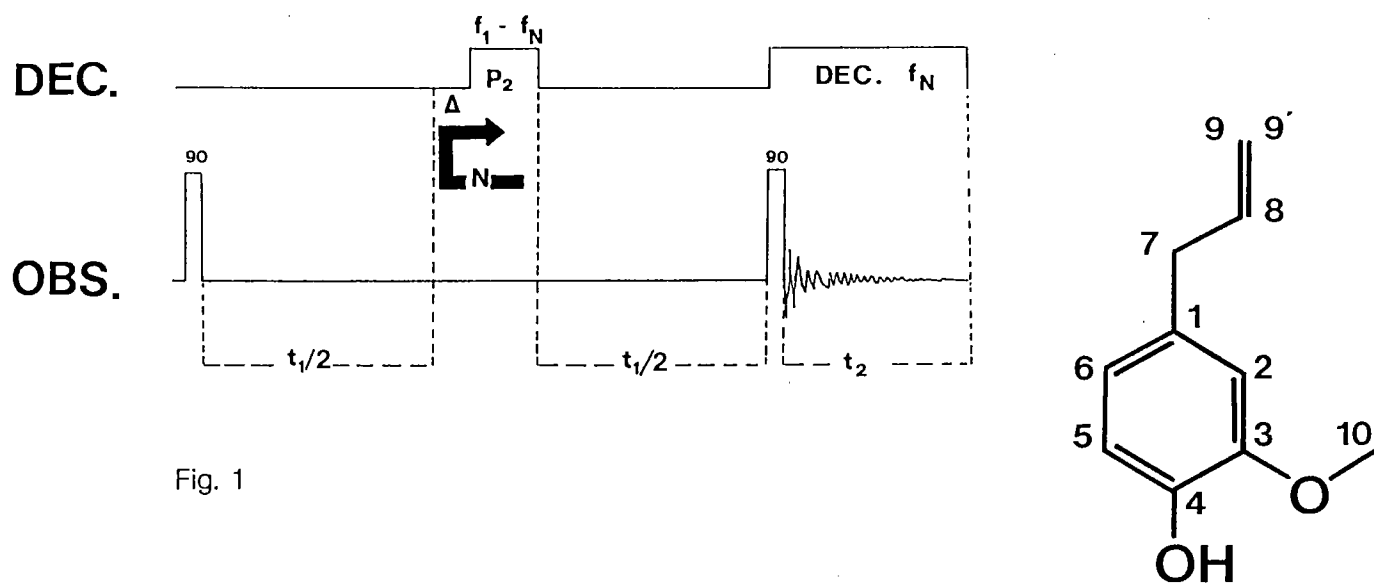


Fig. 1

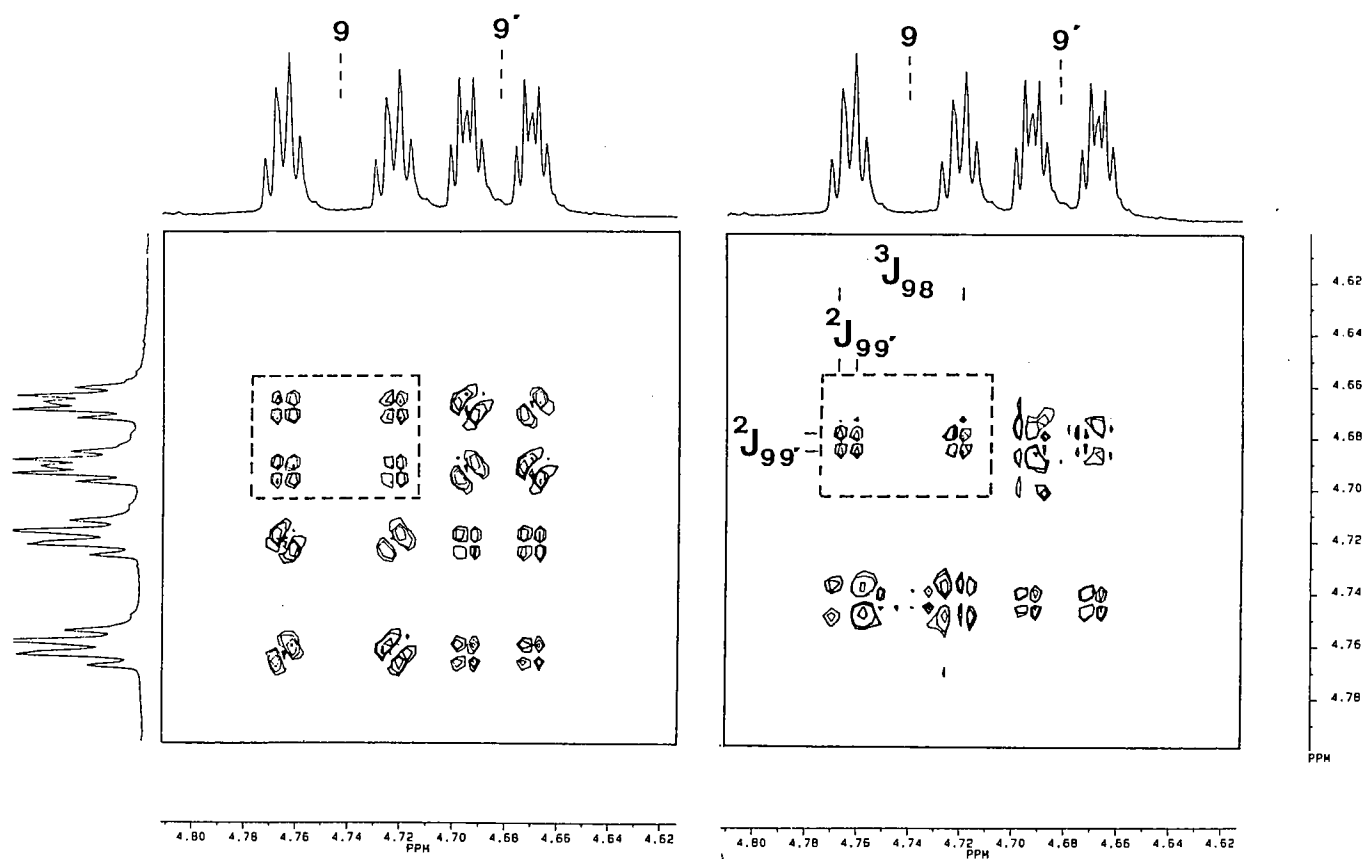


Fig. 2 a

Fig. 2 b



Department of Physics

March 6, 1989
(received 3/13/89)

Dr. B. L. Shapiro
Tamu Newsletter
966 Elsinore Ct.
Palo Alto, CA 94303

Dear Dr. Shapiro:

We have observed spin echoes at unusual times in deuteron NMR of HD molecules present in "hydrogenated amorphous silicon". Echoes following the second pulse of $90_x - \tau - 90_y$ pulse sequence are shown in Fig. 1 for $\tau = 204 \mu\text{sec}$. The FID and two pulses are not shown. Besides the usual echo at time τ following the second pulse, there are two auxiliary echoes at $k \equiv t/\tau = 0.56$ and 1.78.

The deuteron of HD has a quadrupole interaction of strength \mathbf{a} and a dipole coupling of strength \mathbf{b} to the proton. The spin one has two transition frequencies, $\omega_+ = \omega_0 + \mathbf{a} - \mathbf{b}$ and $\omega_- = \omega_0 - \mathbf{a} - \mathbf{b}$. A spin coherence initially at ω_+ accumulates phase $\omega_+ \tau$. The second pulse will switch part of the coherence to ω_- . At time t after the second pulse, the phase is $\phi = (\pm \omega_+ \tau) + \omega_- t$. The \pm sign arises because the second pulse phase inverts part of the coherence. Echoes occur at times t where ϕ is independent of \mathbf{a} and \mathbf{b} . Because the molecules experience a distribution of crystal field magnitudes and orientations in the amorphous silicon, there is a distribution of \mathbf{a} and \mathbf{b} values. But because \mathbf{a} and \mathbf{b} are both described by uniaxially symmetric 2nd rank tensors with the same symmetry axis, they have the same angular dependence. Thus, the ratio b/a is fixed, though \mathbf{b} and \mathbf{a} vary.

The above argument predicts allowed $|\Delta m| = 1$ echoes at $k = \frac{1-b/a}{1+b/a}$ and $\frac{1+b/a}{1-b/a}$. The ratio of b/a may be taken from molecular beam measurements, 0.264. This yields predicted echo locations of 0.58 and 1.72, in reasonable agreement with the observations. The auxiliary echoes are an unambiguous signature of HD molecules. Butterworth¹ and Mehring and Kanert² previously considered the combined effects of quadrupole and dipole couplings. But the identical angular dependences of the couplings in HD lead to new echoes, rather than just damping of the Solomon³ echoes.

Sincerely,

M. P. Volz P. A. Fedders Mark S. Conradi R. E. Norberg
M. P. Volz, P. A. Fedders, M. S. Conradi, and R. E. Norberg

1. J. Butterworth, *Proc. Phys. Soc.* **86**, 297 (1965).
2. M. Mehring and O. Kanert, *Z. Naturforsch.* **24a**, p. 332 and p. 768 (1969).
3. I. Solomon, *Phys. Rev.* **110**, 61 (1958).

Z•Spec Probes

OFFER

Indirect Detection Probes with...

- * High ^1H and X channel efficiency
- * Exceptional Lineshape and Resolution
- * Full X Channel Tuning (^{31}P - ^{109}Ag)
- * Outstanding water suppression capability

Z•Spec Probes are available with Nalorac Cryogenics Quest 4000 series NMR Spectrometers and as after-market Probe products for all major NMR spectrometer systems. For more information about Z•Spec Probe products contact Toby Zens, manager of Special Products Group.



NALORAC CRYOGENICS CORPORATION
837 Arnold Drive Suite 600, Martinez, CA 94553
Tel: (415) 229-3501 FAX (415) 229-1651

Z•Spec Probes

OFFER

Double Tuned Surface Coils with...

- * Multiple Plug in Coil Sizes
- * $^1\text{H}/\text{X}$ Detection or Decoupling $\text{X}(^{31}\text{P}-^{13}\text{C})$
- * Remote Tuning
- * High ^1H and X channel efficiency

Z•Spec Probes are available with Nalorac Cryogenics Quest 100 series NMR Spectrometers and as after-market Probe products for all major NMR spectrometer systems. For more information about Z•Spec Probe products contact Toby Zens, Manager of Special Products Group.



NALORAC CRYOGENICS CORPORATION
837 Arnold Drive Suite 600, Martinez, CA 94553
Tel: (415) 229-3501 FAX (415) 229-1651

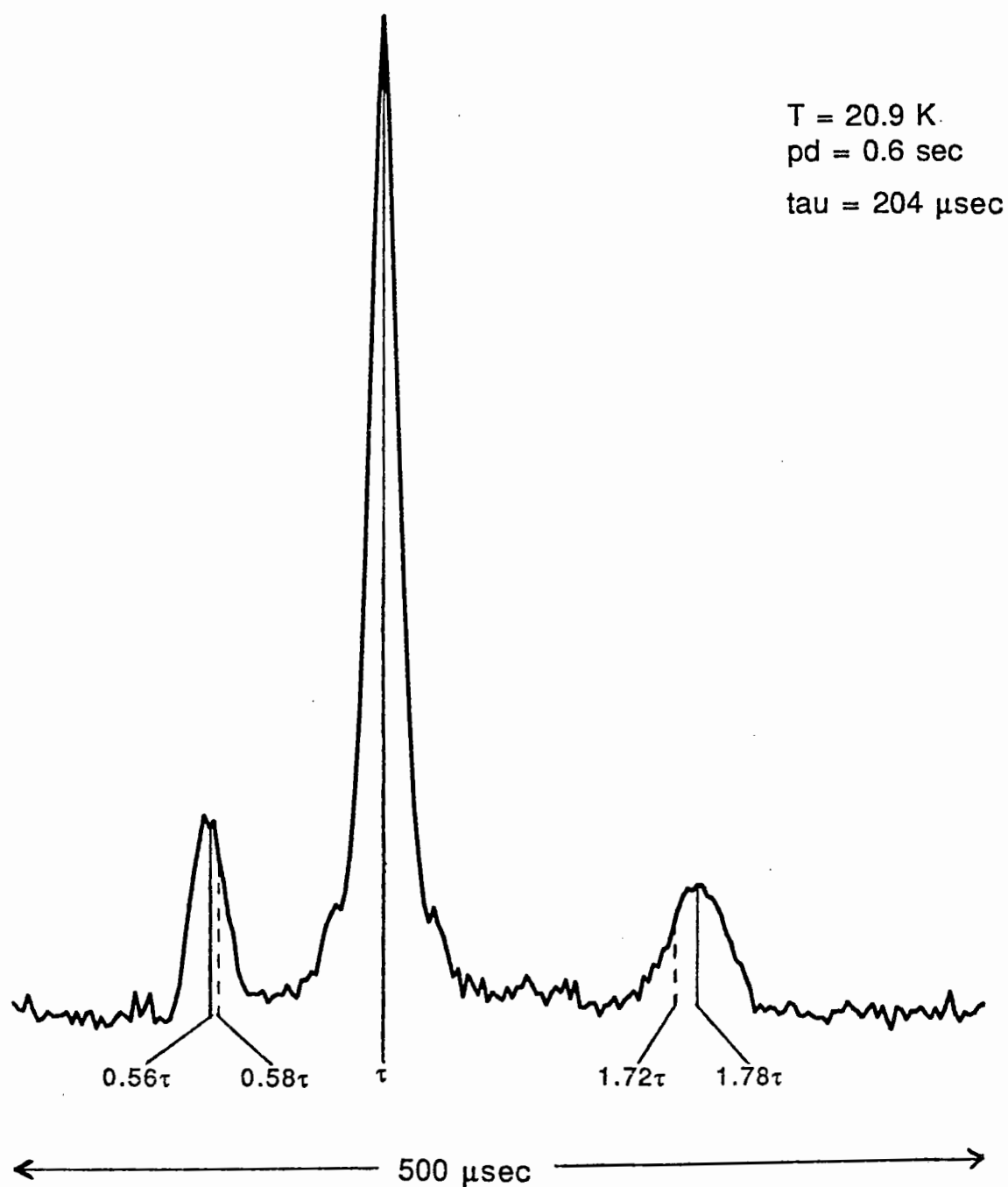


Fig. 1: Allowed Solomon-Butterworth echoes at 20.9 K. The echo positions expected from molecular beam measurements of a and b are indicated as dashed lines for comparison with the present data.



University of Nottingham

Department of Chemistry

UNIVERSITY PARK NOTTINGHAM NG7 2RD
TEL. NOTTINGHAM 484848

HB/vhf

7th March 1989

Dr Bernard L Shapiro
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto
California 94303
USA

Dear Dr Shapiro

N.O.e difference experiments: small molecules

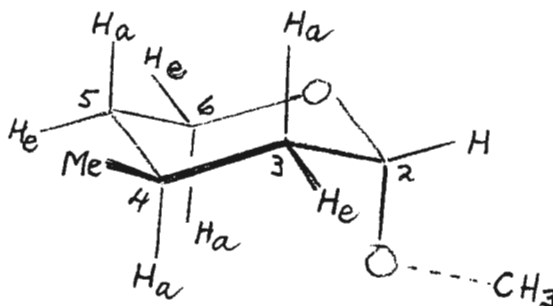
Our n.O.e. difference experiments on small molecules have given some satisfaction but, more often, frustration. Do the published successes in this area represent just a small fraction of all experiments carried out? Only about 1 in 3 of our attempts has given a clean result, i.e. few (or no) artefacts. The real frustration arises when we wish to confirm a crucial result only to find ourselves hampered by many artefacts.

We have carried out seven experiments on a solution in CD_2Cl_2 of trans-2-methoxy-4-methyltetrahydropyran, thought to be largely as (1), in which the methoxy singlet was irradiated. The proton T₁ values are in the range 3 to 6 s. for (1), and we initially tried an irradiation time of 10 s. In a clean result, we measured enhancements of 6% (2-H), 4% (6-H), 4% (4-H), 3% (5-H), 2% (3-H) and 1% each for 3-H_a and 5-H_a. A small negative enhancement showed up for 6-H_e. An irradiation time of 5 s. was more discriminating, and in a clean result we obtained enhancements of 6% for 2-H, 7% for 6-H_a, < 1% for 4-H and no enhancements for the remaining protons. This was in line with expectation, but what will the result be next time??

Yours sincerely

Harold Booth

Dr H Booth



INTRODUCING..... NMR SOLVENTS

from



Introductory Sale

HIGH QUALITY!!! LOW PRICES!!!

They're all checked by ^1H NMR at 400 MHz and they're not only **highly enriched**, they're very **CLEAN** and very **DRY**.

CAT. NO.	PRODUCT	ENRICHMENT MIN. ATOM % D	QUANTITY*	PRICE [†] U. S. \$
82-00732	Acetone-d ₆	99.9	10 x 1 g	14.
			10 g	14.
			5 x 10 g	69.
			10 x 10 g	135.
82-05012	Acetonitrile-d ₃	99.8	10 x 1 g	22.
			10 g	22.
			5 x 10 g	100.
82-84077	Benzene-d ₆	99.6	10 x 1 g	18.
			10 g	18.
			5 x 10 g	75.
			10 x 10 g	145.
82-80556	Chloroform-d	99.8	100 g	18.
			5 x 100 g	80.
			10 x 100 g	142.
			1 kg	135.

CAT. NO.	PRODUCT	ENRICHMENT MIN. ATOM % D	QUANTITY*	PRICE [†] U. S. \$
82-70001	Deuterium Oxide	99.9	100 g	45.
			5 x 100 g	215.
			10 x 100 g	390.
			1 kg	375.
82-70901	Deuterium Oxide	99.8	min. 10 kg	3500.
			25 kg	8500.
			50 kg	16250.
82-70002	Deuterium Oxide "100%"	99.96	10 g	15.
			5 x 10 g	50.
84-70001	Deuterium-depleted Water	< 5 x 10 ⁻⁵	25 g	25.
			4 x 25 g	90.
			10 x 25 g	200.
			20 x 25 g	300.
			30 x 25 g	400.
			1 kg	500.
82-00807	Dimethyl-d ₆ Sulfoxide	99.9	10 x 1 g	12.
			10 g	12.
			5 x 10 g	56.
			10 x 10 g	110.
82-00809	Dimethyl-d ₆ Sulfoxide (multi-dose septum vials)	99.9	10 g	12.
			5 x 10 g	56.
			10 x 10 g	110.
82-00061	Methanol-d ₄	99.8	10 x 1 g	43.
			10 g	43.
			5 x 10 g	171.
			10 x 10 g	325.

***PACKAGING:** Organic solvents are sealed in glass ampoules to maintain the high quality of the solvents. Dimethyl-d₆ sulfoxide is also available in multi-dose septum vials. The waters are packaged in glass screw-cap bottles, except for the "100%" deuterium oxide, which is packaged in multi-dose septum vials.

[†]Prices are FOB Miamisburg, Ohio for delivery in North America; please request prices for delivery to the other continents. Minimum order, \$50.

**Please request prices for quantities larger than are listed.
Please allow us to bid on your annual NMR solvent requirements.**

The other NMR solvents will soon be available; we will keep you informed. In the meantime, please ask us for any compounds in which you are interested. We have many more compounds than are listed in the Interim Price List 3/88 and we are now in a position to carry out many custom syntheses. **Please call us with your requests.**

ISOTECH INC.
A Matheson[®] USA Company

3858 Benner Road • Miamisburg, Ohio 45342

(513) 859-1808 • (800) 448-9760 • Telex: 288278 • Fax: (513) 859-4878



THE CLEVELAND CLINIC FOUNDATION

One Clinic Center 9500 Euclid Avenue Cleveland, Ohio 44195

A National Referral Center An International Health Resource

March 10, 1989
(received 3/16/89)

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

Determination of Lactate Using 2-D Double Quantum Coherence Transfer

Dear Barry:

We have been studying the metabolism of solid tumors in vivo using multinuclear NMR spectroscopy. Recently a 2-dimensional ^1H double quantum coherence transfer method to monitor lactate levels in vivo without contamination from overlapping lipid signals has been reported by the group from GE (1). Although this method uses pulsed field gradients to select the double quantum coherence and works best with self shielded gradients, we have implemented it on our 4.7 Tesla GE imaging/spectroscopy system without shielded gradients. Figure 1a shows the slice through 1.3 ppm (the ^1H chemical shift of the lactate CH_3 in the single quantum domain) in a 128 x 2K ^1H double quantum coherence transfer experiment performed on a RIF-1 tumor implanted subcutaneously in a C3H/HeN mouse. Although lipid and lactate resonances overlap at 1.3 ppm in the single quantum domain, the lactate DQ signal at 4.2 ppm (denoted by *) is well resolved from the lipid DQ signals at 6.2 and 7.4 ppm. We find significant changes in the level of lactate following treatment of the tumor with X-radiation. Figure 1b shows the decline in lactate 24 hrs following a single 60 Gy radiation treatment. We are following the dose and time dependence of this change as well as monitoring the changes in tumor blood flow. We hope to report our findings before the next dreaded pink slip.

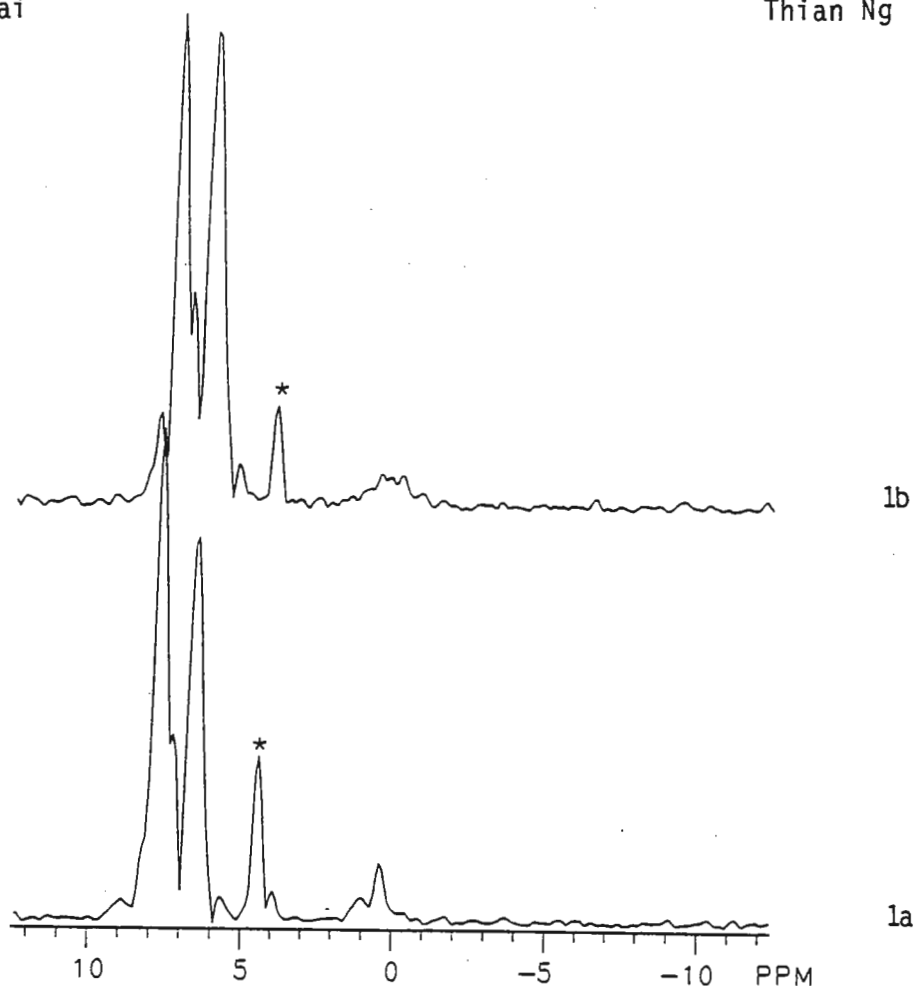
Reference:

(1) Sotak, CH; Freeman, DM and Hurd RH, J. Magn Reson 78:355-361(1988).

Sincerely,

Raj
Rajasekharan P. Pillai

Thian
Thian C. Ng



Position Available

The MR Research Center of The Cleveland Clinic Foundation is seeking a scientist with doctoral degree and less than 5-years research experience. The MR Center currently has a GE 4.7T/40-CM animal system and GE Signa 1.5T/whole body system. Both systems equipped with multinuclei localized spectroscopy and imaging. It is also well-equipped with a tissue culture lab, a biochemistry lab and an animal preparation room. Many projects are designed to investigate various tumor system in situ and in vitro (includes perfused cell studies) using NMR and other tumor biological assays. Salary depends on experience; background in tumor biology helpful. Interested applicants should forward a curriculum vitae to: Dr. Thian Ng, Director of MR Research Center, Division of Radiology, The Cleveland Clinic Foundation, 9500 Euclid Avenue, Cleveland, Ohio 44106, (216 444-8209).

HIGH-PERFORMANCE DIRECT SYNTHESIZERS

Accurate, stable, quiet frequencies on command, fast. For NMR, imaging, SATCOM, surveillance, ATE. Sources adapting to your needs with options. High demonstrated reliability. Thousands in use.

PTS 040

Range: 0.1-40MHz
Resolution: 0.1Hz-100KHz (opt.)
Switching: 5-20 μ s
Output: +3 to +13dBm: 50 ohm
Spurious Outputs: -75dB

Phase Noise: -75dBc, (0-15KHz)
Freq. St'd: Oven, TCXO, Ext.
Interface: BCD par. or GPIB
Size: 19"W, 5 1/4"H, 18"D
Price: \$4,800.00*

Other Options:
Progr. Attenuator, 0-90dB
(or 0-99dB with GPIB)
nx10MHz output 20-140MHz
or any 10MHz line (20-140)

PTS 120

Range: 90-120MHz
Resolution: 0.1Hz-100KHz (opt.)
Switching: 5-20 μ s
Output: +3 to +10dBm: 50 ohm
Spurious Outputs: -75dBc

Phase Noise: -75dBc, (0.5Hz-15KHz)
Freq. St'd: Oven, TCXO, Ext.
Interface: BCD par. or GPIB
Size: 19"W, 5 1/4"H, 18"D
Price: \$4,800.00*

Other Options:
Progr. Attenuator, 0-90dB
(or 0-99dB with GPIB)
nx10MHz output 20-140MHz
or any 10 MHz line (20-140)

PTS 160

Range: 0.1-160MHz
Resolution: 0.1Hz-100KHz (opt.)
Switching: 5-20 μ s
Output: +3 to +13dBm: 50 ohm
Spurious Outputs: -75dB

Phase Noise: -63dBc, (0-15KHz)
Freq. St'd: Oven, TCXO, Ext.
Interface: BCD par. or GPIB
Size: 19"W, 5 1/4"H, 18"D
Price: \$5,850.00*

Other Options:
Progr. Attenuator, 0-90dB
(or 0-99dB with GPIB)
nx10MHz output 20-140MHz
or any 10 MHz line (20-140)

PTS 250

Range: 1-250MHz
Resolution: 0.1Hz-100KHz (opt.)
Switching: 5-20 μ s
Output: +3 to +13dBm: 50 ohm
Spurious Outputs: -70dB

Phase Noise: -63dBc, (0-15KHz)
Freq. St'd: Oven, TCXO, Ext.
Interface: BCD par. or GPIB
Size: 19"W, 5 1/4"H, 18"D
Price: \$6,700.00

Other Options:
Progr. Attenuator, 0-90dB
(or 0-99dB with GPIB)
nx10MHz output 20-140MHz
or any 10 MHz line (20-140)

PTS 500

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

Phase Noise: -63dBc, (0-15KHz)
Freq. St'd: Oven, TCXO, Ext.
Interface: BCD par. or GPIB
Size: 19"W, 5 1/4"H, 18"D
Price: \$7,850.00

Other Options:
Progr. Attenuator, 0-90dB
(or 0-99dB with GPIB)
nx10MHz output 20-140MHz
or any 10 MHz line (20-140)

PTS 300 NEW

Range: 0.1-300 MHz
Resolution: 1 Hz
Switching:
20 μ s (10MHz, 100MHz steps)
5 μ s (1 MHz steps)
1 + 2 μ s (transient + delay) for all steps
1 Hz-100 KHz; (phase-continuous)

Output: +3 to +13dBm; 50 ohm
Spurious Outputs: Type 1
-70/65dB (typ/spec) Type 2
-60/55dB
Phase Noise: -68dBc, (0.5Hz-15KHz) -63dBc
Freq. St'd: Oven, TCXO, Ext.
Interface: BCD par.

Size: 19 x 3 1/2 x 17"
(relay rack or bench cabinet)
Price: Type 1 Type 2
\$5,800.00* \$5,300.00*

*Prices are US only, manual & remote, (BCD), 1 Hz res. with oven std.



NEW!
PHASE
ROTATION
AVAILABLE
ON ALL OF
OUR MODELS

Choice of TLU/DDS resolution with phase—continuous switching.

PROGRAMMED TEST SOURCES, INC.

P.O. Box 517, 9 Beaver Brook Rd., Littleton, MA 01460 508-486-3008 FAX Number: 508-486-4495

Institut für Molekularbiologie und Biophysik

Prof. Dr. K. Wüthrich

HPM-Gebäude

Durchwahl-Nr.: 01 / 377 24 73

Telefonzentrale: 01 / 377 44 11

Telex: 823 474 ehpz ch

Postadresse:

*Institut für Molekularbiologie
und Biophysik
ETH-Hönggerberg
CH-8093 Zürich*

Telefax 01/ 371 48 73

Prof. B.L. Shapiro
TAMU NMR Newsletter
966 Elsinore Court

Palo Alto, CA 94303
USA

1085

Zürich, March 9, 1989/hu

Installation of a 600 MHz NMR spectrometer in Zürich

Dear Barry,

Our 600 MHz spectrometer was initially installed in the basement of our laboratory building. We soon realized that the new magnet was very sensitive to environmental influences. In particular, it picked up perturbations when magnetic objects (e.g., chairs with metal wheels) were moved around in the laboratory above the magnet, and it was also sensitive to low frequency mechanical vibrations present in the building. Our search for a location devoid of these perturbing effects identified a room that seemed ideally situated as it is outside of the laboratory tract and is part of a one-story underground construction. Since this room was not sufficiently high to accommodate the 600 MHz magnet, we constructed a hole in the floor, into which the magnet was placed (Fig. 1). For this the ground plate of the building was cut, and the hole was actually dug into solid ground.

Although this construction was forced upon us by the small size of the available room, we soon realized that there are various advantages to it. For example, with this set-up sample changing and refilling of the liquid gases is much more convenient than in a conventional, standard laboratory. Furthermore, the field tests were significantly improved relative to the results obtained previously in the factory and in our laboratory, and the magnet is easier to shim. We tentatively attribute this to the fact that the walls and the bottom of the hole consist of 90 cm of heavily reinforced concrete (Fig. 1), so that the magnet is symmetrically placed in an "iron basket", as compared to the typical asymmetric environment of a magnet placed in the corner of a room.

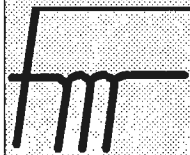
Sincerely yours,



R. Baumann



Prof. K. Wüthrich



Broadband Decoupler

- Broadband RF Circuits (5-500MHz).
- Accepts any Synthesizer Input (0dBm).
- Fast BiLevel RF Power Switching (< 1 usec).
- MLev & Waltz Modulation Schemes.
- 0.5 Watt Output to external RF Power Amplifier.
- Excellent On/Off isolation.
- 90°/180° Phase Shifts (Sub 90° available)

FMR's Broadband Decoupler is of modular construction, and can be purchased as a complete package or by the module. The modules are useful for updating existing instruments. FMR provides the modules and instructions for the do-it-yourselfer or will update your transmitter or decoupler in its facilities. The complete package is available as a stand alone transmitter when used with a synthesizer and power amplifier.

- Digital 90° and 180° Phase Shifter Module
- Mixer / Gate / Attenuator Module (Fast BiLevel Operation).
- 0-65 dB Attenuator Modules (**BiLevel requires two**)
- MLev / Waltz Modulator Module
- Driver RF Amplifier Module
- High Power Gate Module (+10dBm).
- Filter for one nucleus (specify frequency).
- Chassis and Power Supplies.

The external synthesizer and power amplifier necessary for the use of the Broadband Decoupler Package may already be available in the laboratory. If not, FMR offers:

- PTS 300 Synthesizer with 0.225° phase shifts.
 - 10-220 MHz Linear RF Amplifier.
- 100 Watt CW (150 Watt Peak) with NMR blanking.

FMR offers ¹H Probes with broadband decoupling coils for most NMR instruments for use with the above Broadband Decoupler. Call for details.

Repair Services

FMR Offers a mail in repair service for NMR instrument RF modules and probes at reasonable rates. Fast turn-around times are often possible. Call for further information.

Noise Figure Meter

Have you ever struggled with sensitivity tests on your NMR instrument? It is often very difficult to determine where the problem area lies:

- Shimming (resolution and/or lineshape).
- The probe.
- The NMR console's noise figure.

To help determine the problem area, FMR has developed an inexpensive noise figure meter kit with procedures to enable the NMR user to quickly determine the system noise figure.

The system noise figure is an important factor determining the NMR instrument's overall signal to noise performance level. The NMR instrument is designed to have the preamplifier gain and noise figure determine the total system noise figure. As a rule of thumb, a 1 dB increase in the noise figure decreases the signal to noise by 10-15%. For adequate signal to noise the overall system noise figure needs to be in the 1.0 to 2.5 dB range. If the system noise figure is outside that range, further tests are needed. If the signal to noise is not at the level it is supposed to be, there can be at least three problems:

- Not enough signal from the probe.
- Poor system noise figure.
- Noise from other sources

To be most useful, noise figure tests should be done routinely as part of preventative maintenance procedures on the NMR instrument. This gives a history of performance levels and makes it easy to see when something has gone wrong. They can however be useful without this history, since most working NMR systems have typical noise figures in the range (1.0-2.5 dB). If the NMR system is performing outside this range, other noise figure tests can help determine which module of the system is at fault.

FMR's Noise Figure Meter Kit consists of a meter capable of displaying the noise level in the audio channel going to the digitizer and a 50 ohm noise source for the preamp. The meter is calibrated with the noise source at room temperature. Then the noise source is cooled in liquid nitrogen. The system noise figure is then read directly from the meter. The entire procedure takes only a few minutes and can be repeated as a diagnostic procedure.

Ordering Information:

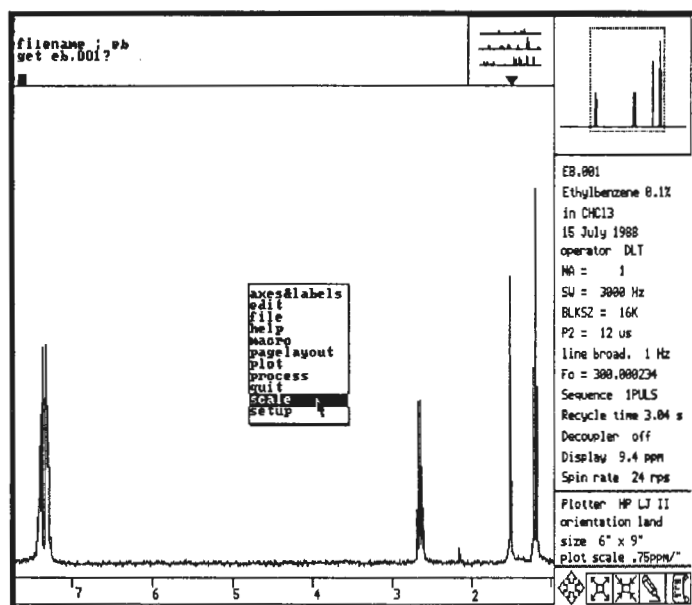
NMK-10007-2B Noise Meter Kit

\$500.00



PC based Plotting

Plotting takes a lot of time, even if your system is capable of multi-tasking and plotting in the background. And when its plotted all you have is a hard copy of the spectrum. With FMR's PC based plotting software package you can:



Screen Image

- Plot off-line in the convenience of your office or home.
- Plot spectra using PC based laser printers, dot-matrix printers or HPGL plotters.
- Annotate the spectra with labels, notes, figures, arrows and etc..
- Incorporate the annotated spectra as figures in articles, memos and letters using popular desktop publishing software packages.
- Perform minor spectral editing to a data file for publication quality results.
- Archive the data using inexpensive PC based disks and tape drives.
- Have a convenient digital library of your spectra in your office for immediate reference and comparison.

PLOT is a software tool requiring an IBM PC or compatible, and supports common graphics devices, mice, plotters and printers. The transformed data file is transferred to the PC by RS-232, Kermit, Ethernet, or by emulating the spectrometer's plotter. PLOT is WYSIWYG, which means you can preview your plot on the computer screen as it will appear on paper. Recently accessed spectra are kept in a stack similar to an HP calculator for easy manipulating and viewing.

PLOT will work with CGA, EGA, VGA, Hercules, AT&T, or Tandy graphics systems. It will employ a 8087 coprocessor if it finds one. It can output your data to an HP-compatible laser printer, an HPGL plotter, A Zeta plotter, an Epson-compatible dot matrix printer, to a file in any of the above formats, or to a file for input into Aldus PageMaker or Xerox Ventura Publisher.

Online HELP is available for each command, icon, and screen area. The user may view the help text on topic "xxx" by typing "help xxx", or get help on screen items by pressing the F1 key and then placing the cursor over the questionable item.

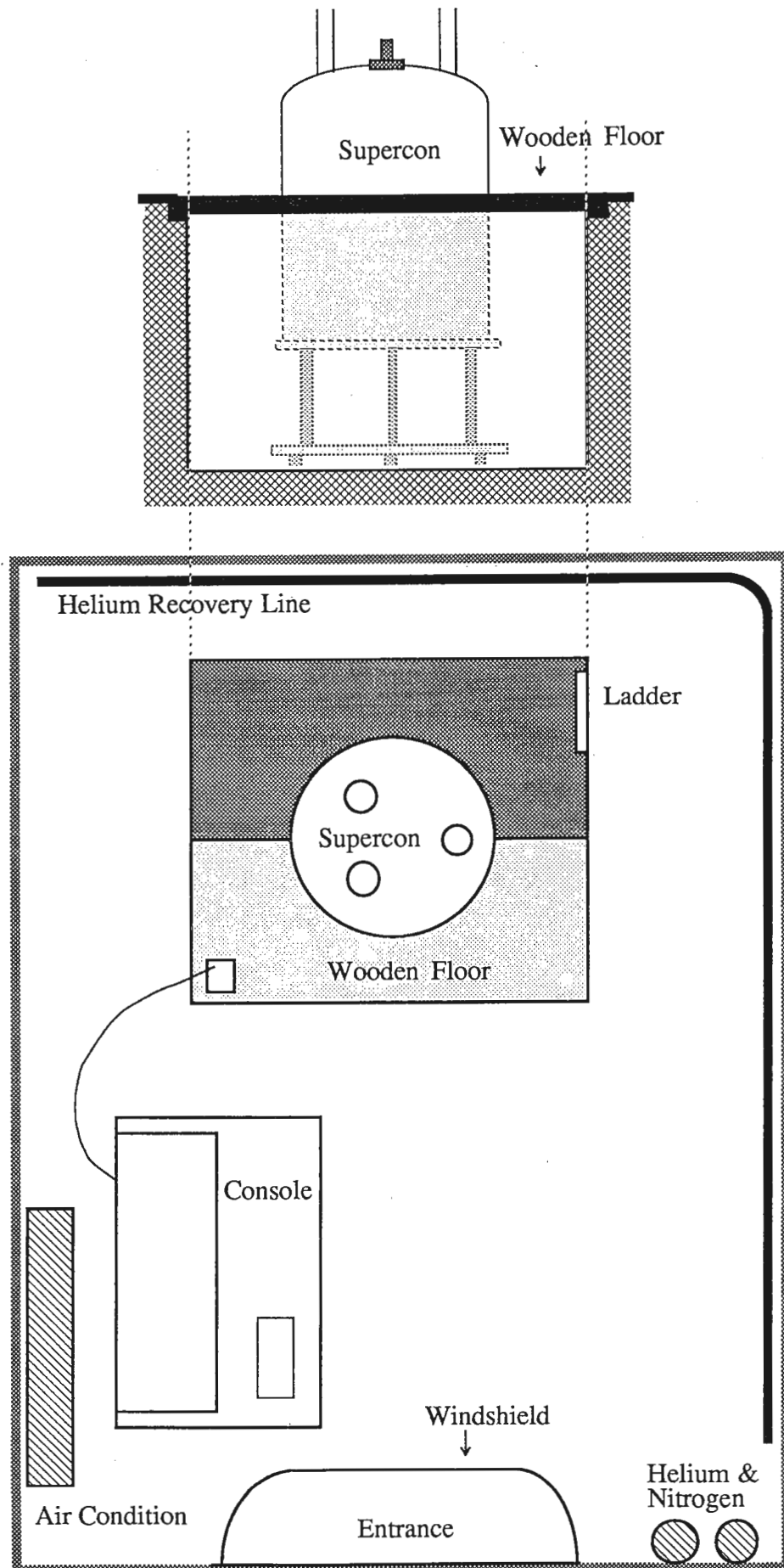


Fig. 1



Department of Chemistry

March 15, 1989

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

Dear Barry:

The Department of Chemistry of Washington University (St. Louis) is hosting a one-day NMR symposium on Wednesday, May 24, 1989. Emerson Electric Company is the sponsor of the symposium. There is no registration fee. Details about the symposium can be obtained from the Symposium Secretary, Mrs. Carol Brown (314 889 6350). The schedule of lectures is

R. Kaptein	Professor, University of Utrecht "Structures of Proteins and Protein-DNA Complexes from 2D and 3D NMR."
<i>(Refreshment break)</i>	
M. Mehring	Professor, University of Stuttgart "Pulsed Magnetic Resonance on Delocalized Electron Spins."
<i>(Lunch)</i>	
R. Tycko	Member, Technical Staff AT&T Bell Laboratories "Studies of Molecular Structure and Dynamics in Two Spectral or Spatial Dimensions."
<i>(Refreshment break)</i>	
A. N. Garroway	Head, Polymer Diagnostics Section Naval Research Laboratory "NMR Imaging for Solids."
I. Pykett	President, Advanced NMR Systems, Inc. "Ultra-High Speed MR Imaging Techniques and Their Clinical Applications."

Best wishes,

Sincerely,

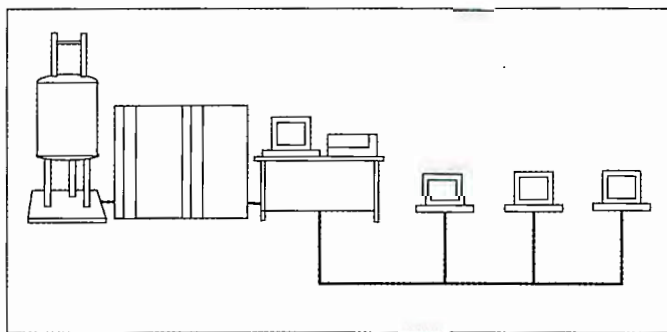
Jacob Schaefer
Charles Allen Thomas
Professor of Chemistry



Here Is Your Brand New \$10,000, 500 MHz FT-NMR Spectrometer.

Our GSX Series FT-NMR spectrometer was built for the laboratory which has a large family of NMR users, but the budget for only one NMR spectrometer.

When you invest in one GSX, you can get up to three others just by adding data stations, each of which costs approximately \$10,000.



The GSX FT-NMR spectrometer can be shared by up to four users simultaneously. While one does an experiment, others can be analyzing data, preparing manuscripts, or generating slides for the next scientific meeting.

The GSX is an advanced technology NMR instrument equipped with a multi-user, multi-tasking DEC 'microPDP-11' based' data system.

It offers the highest available sensitivity and resolution with automatic shimming, and such advanced technology features as automatic probe matching and menu-driven control.

Best of all, for the large laboratory, a single GSX can be configured to accommodate multiple users simultaneously. And that means more productivity and less aggravation for everyone.

While one user is running an experiment, a second can be analyzing data from a previous run, a third working on a manuscript, and a fourth preparing slides for the next scientific meeting.

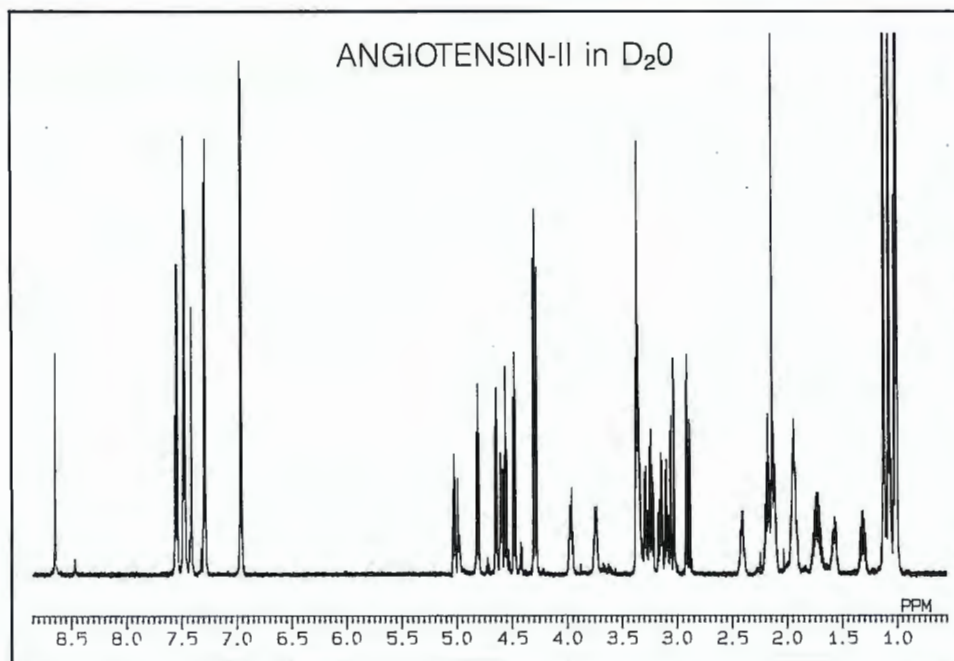
No one even needs to be in the NMR lab. Experiments can be queued up on the auto-sampler and run from remote locations — another lab, another office, even another building.

Come see our multi-user GSX FT-NMR spectrometer in operation.

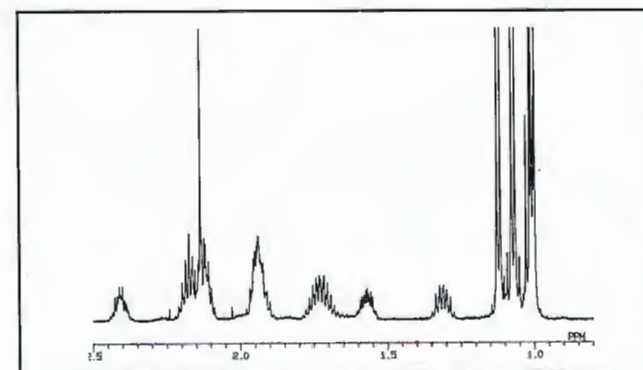
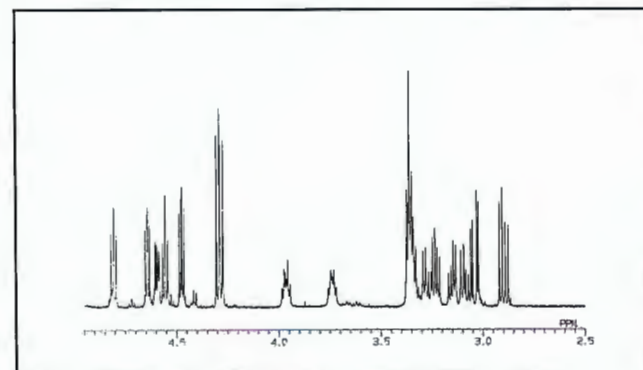
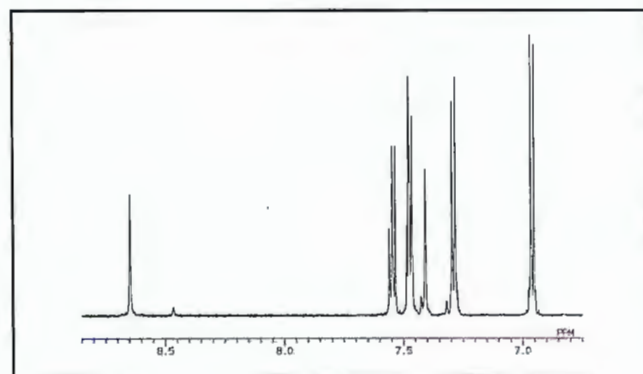
For a demonstration or literature, call (508)535-5900. Or write JEOL U.S.A. Inc., 11 Dearborn Road, Peabody, MA 01960.



GSX-600



JEOL in its continuing commitment to the scientific community announces an operational 600 MHz NMR spectrometer at its Akashima applications laboratory. The Oxford Instruments 14.1 Tesla superconducting solenoid with JEOL's well proven matrix shim system, benchmark Rf technology, and excellence in probe design combine to provide the highest dispersion in 600 MHz performance. If your research and applications in macro molecules or low gamma nuclei requires 600 MHz performance, you owe it to yourself to call JEOL.



The above data is of Angiotensin-II in D₂O and was processed under normal one dimensional conditions. If you are presently evaluating 600 MHz NMR spectrometers, we would be pleased to arrange for you to see the GSX-600.

Please Contact:

JEOL

Serving Advanced Technology

11 Dearborn Road, Peabody, MA 01960
(508) 535-5900



Please reply to

Massachusetts General Hospital
Boston, Massachusetts 02114



NMR LABORATORY
(617) 726-3081

March 13, 1989 (received 3/15/89)

Professor Bernard Shapiro
966 Elsinore Court
Palo Alto, CA 94303

Proton Images of Solid Bone

Dear Barry:

One of the (normally useful) key features of conventional proton MR imaging is that the solid component of bone provides no signal, and thus the bone itself is invisible. However, the examination of the solid phase of mineralized tissue by NMR could prove informative in the study of disorders associated with calcium or phosphorus deficient diets, dental caries, osteoporosis or Paget's disease, for example. NMR analysis could also contribute to the understanding of normal and abnormal cellular and molecular level processes such as growth, ectopic growth, resorption and repair. A number of groups (e.g., Griffin, Glimcher, et. al., Yesinowski, et. al., and others) have laid the foundations of solid state NMR spectroscopy of these materials.

We have been interested in developing imaging techniques for mineralized tissue, using the spectroscopic characteristics reported by these workers for the development of image contrast between the various inorganic phases. Both proton and phosphorus resonances in the calcium phosphates which make up bone mineral are useful. The very short T_2 's encountered in these solids require echo times TE under one msec, necessitating special apparatus and pulse sequences. For example, we can produce a proton image of a transverse section of a 2 cm diameter lamb shank bone (a primarily dense cortical material) with a backprojection technique employing a spin echo with TE = 600 μ sec. The gradient is on during the complete 90- τ -180-acquire sequence, which eliminates the need for rapid gradient switching.

The image in Figure 1 is the result of a 2 hr acquisition. In order to study just the solid phase, tissue was removed by exhaustive treatment with NaOCl, sonication, extraction with solvents of decreasing polarity, and vacuum drying. The proton signal contains contributions from the crystal lattice hydroxyl protons of hydroxyapatite (the major mineral component), strongly adsorbed water, and residual organic phase. High speed proton MAS spectroscopy is useful for quantifying the amounts of each of these components. It also suggests that much of the residual organic material, which totals under one mole percent of all protons, is likely to be collagen, as expected from the known chemistry of bone tissue. ^{31}P imaging, of course, does not require the removal of tissue, and may be practical *in vivo*. We will be reporting on these experiments shortly.

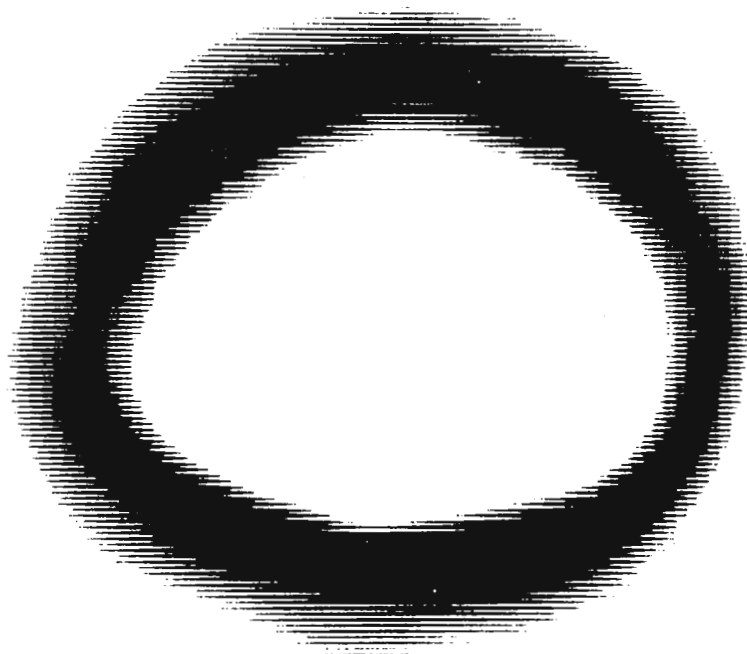


Figure 1. Proton image of 2 cm diameter lamb shank bone, 2.0 T field strength, spin echo, 600 μ sec TE, 130 msec TR, no slice selection, 200 projections, 256 averages.

Jerome L. Ackerman

Leoncio Garrido

CALIFORNIA INSTITUTE OF TECHNOLOGY

Manager of NMR Facilities, California Institute of Technology. The California Institute of Technology seeks an individual, preferably at the Ph.D. level, to oversee an NMR facility consisting of a range of NMR spectrometers. The ideal candidate will have a broad-based knowledge of NMR hardware and maintenance, the design and implementation of modern pulse sequences and related techniques, as well as versatility with associated computer software. Please send resume and salary history to Dr. John Sibert, Division of Chemistry and Chemical Engineering, 164-30, California Institute of Technology, Pasadena, CA 91125. Applications from minority and women candidates are encouraged; the California Institute of Technology is an equal opportunity employer.

When the Challenge is Research,

the Choice is
the Fully Capable,
Cost-Effective,
Accessible
VIVOSPEC™ Magnetic
Resonance Imaging
Spectrometer.

Designed for MR Spectroscopy and Imaging by MR Researchers, the First Truly Accessible System that Really Puts Users First.

With the VIVOSPEC SPECTROMETER, you get a full range of spectroscopy and imaging features and functions, including—

- MULTINUCLEAR SPECTROSCOPY AND IMAGING CAPABILITY
- TRANSFORMED SPECTRA IN REAL TIME FOR SHIMMING
- HETERO- AND HOMONUCLEAR DECOUPLER OPTIONS
- OPTIONAL SELF SHIELDED GRADIENTS
- ... and much more.

Consider the Research Challenges Already Being Addressed:

- METABOLIC STUDIES
- MUSCULOSKELETAL STUDIES
- NEUROLOGICAL STUDIES
- ISCHEMIA STUDIES
- PHARMACEUTICAL STUDIES
- ANESTHESIA STUDIES
- IN-VITRO STUDIES

New

Discover the Savings

Low loss cryostat for reduced cryogen consumption

Modularized design for reliability and ease of service

Full range of magnet sizes and strengths allows you to purchase only as much magnet as you need

Optional magnet shielding reduces facility requirements

And All at the Lowest Prices Available

CALL NOW FOR MORE DETAILS ON THE VERSATILE, INNOVATIVE VIVOSPEC Magnetic Resonance Imaging Spectrometer... a product of Otsuka Electronics.





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

14 Feb 1989
 (received 2/22/89)

*Roche Products Limited · PO Box 8 · Welwyn Garden City · Hertfordshire · AL7 3AY · Telephone (0707) 328128
 Telefax (0707) 338297 · Telex 262098 ROCHEW*

Dear Barry,

Solvent Suppression in TOCSY

We have been experimenting with various ways of suppressing the solvent signal when running TOCSY (alias HOHAHA) spectra in water. As commented by Esposito *et al.* (J. Magn. Reson. 1988, 80, 523) the difficulty is that all RF sources and the receiver must be phase locked. There is a practical difficulty here, in that, although the same power setting can be used both for the hard pulses and for the spin locking, a lower power setting is normally required for solvent suppression. The solution of Esposito *et al.* is to use a DANTE sequence to suppress the solvent signal, so that the same RF source is used throughout. In our hands, this implementation proved tricky to set up and not particularly successful.

Our Bruker AM-400 is modified so that O1 and O2 can be phase locked, thus opening up the possibility of using the transmitter for the pulses and spin locking (at the low power transmitter output, TLO) and the decoupler for solvent saturation. Clearly, this necessitates having the carrier positioned at the water frequency, as does the DANTE method. Solvent saturation is carried out using a long pulse from the decoupler, which should be phase cycled in parallel with the first hard pulse. There are two interesting points to make here. Firstly, although the decoupler and transmitter are phase locked, the phase generated by the decoupler is not the same as that generated by the transmitter; the relative phase shift varies with the power setting of the decoupler, but at the level used here it was 184°. Secondly, it proved impossible to change the phase of the decoupler pulse by using a phase command in the microprogram; all values of decoupler phase generated the same spectrum (184° out from that obtained using the transmitter). The trick is to insert a dummy hard pulse before the decoupler pulse, and phase cycle this as desired, corresponding to the microprogram

1 ZE

2 P1 PH1 ;P1 = 0, PH1 = (360) 184

3 P2:D PH2 ;P2 = 1 s presaturation, PH2 = B0 here but anything will do

P3 PH3 D0 ;start of TOCSY sequence, P3 = 52 µs in TLO

The figures compare the spectra produced with and without the dummy hard pulse, demonstrating a small but significant reduction in F₂ ridges from the solvent using the dummy pulse.

MERCK DEUTERATED SOLVENTS FOR NMR

MSD

MSD ISOTOPES

DIVISION OF MERCK FROSST CANADA INC., Montreal, Canada

Price List 49A

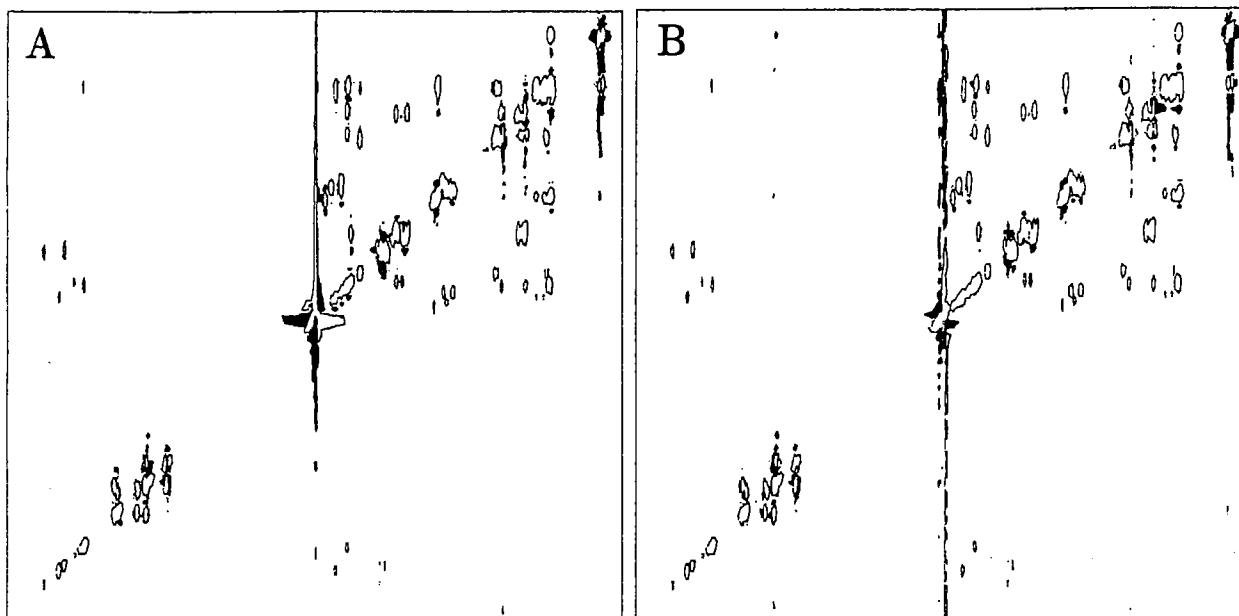




Yours sincerely

Mike

Michael P Williamson



TOCSY spectra of LHRH in H_2O/D_2O , mixing time 32 ms. Solvent saturated using the decoupler, other pulses from the transmitter in TLO mode ($90^\circ = 52 \mu s$). The pulse sequence is that of Rance (J. Magn. Reson. 1987, 74, 557), with 90° pulses acting as a z-filter each side of the spin locking (WALTZ). Negative levels are blacked in. (A) Without, and (B) with a dummy pulse.

Postdoctoral Position, University of North Carolina at Chapel Hill with Ernest L. Eliel

A postdoctoral position with Professor Ernest L. Eliel, University of North Carolina, Chapel Hill in the general area of stereochemistry will be available July 1, 1989.

Problems range from conformational analysis and nuclear magnetic resonance studies (including applications of rapid-injection nmr, a stop-flow kinetic technique) to enantioselective synthesis. Ideally, candidates should combine skill in synthesis with good background in nmr and in the use of computers, e.g. in molecular mechanics. Ph.D. or equivalent is required.

The appointment is for one year, at a stipend of \$17,500 and may be extended if mutually agreeable and subject to the availability of funds.

Applications, with a resume including a statement of previous experience and names of three references should be sent to

Ernest L. Eliel
Department of Chemistry
CB#3290 Venable & Kenan Laboratories
University of North Carolina
Chapel Hill, NC 27599-3290

The deadline for applications is April 15, but completed applications will be considered beginning March 1, 1989.


Hoffmann-La Roche

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

Direct Dial

March 9, 1989

500 to 400 to VAX to FTNMR

=====

Dear Barry:

We collect most of our data on large biomolecules on a VXR-500S spectrometer, which is controlled by a Sun 3/160 computer running UNIX. We prefer to do our processing with FTNMR on a VAX-8800, which is running VMS. We would like to transfer our data directly from the 500 to the VAX. Presently, such a transfer is not possible without the purchase of software for the VAX; such software is expensive for an 8800.

We are able to transfer data readily over Ethernet from our XL-400 to the VAX, using Varian's LimNet software. We have recently acquired a version of LimNet for the VXR-500S. After some fiddling, we have discovered how to move our data files from the 500 to the 400 to the VAX, and to format them for FTNMR. Our current protocol is as follows:

1. Using LimNet, we write the fid portion of a data set (ex.- data.fid/fid) from the Sun to the XL-400. We have found it necessary to reduce the burst size to low values (1 or 2) to avoid time-outs.
2. Using LimNet, we read the file from the XL-400 onto the VAX.
3. At this point, the file has a record length of 133 bytes. There is a program in the Sun version of FTNMR called "VARIAN1D", which will reformat a data file from a VXR-500 so that it can be read in and processed; this program requires a file with a record length of 512. Our colleague, Dr. Charles Cook, initially wrote a program for us called "PACKFID", which reorganizes the data file into 512 byte records. The output may then be run through VARIAN1D. Note that the portion of VARIAN1D which swaps high and low data bytes must be retained - it was commented out in our version.
4. We currently find it more convenient to read the entire data file into memory on the VAX, where it is then accessed by a program which combines PACKFID and VARIAN1D. In order to do this, one's page file quota must be larger than the largest file one wishes to convert (ex. - a 2-D data set of 512 fids x 2048 points is about 16,000 blocks; the page file quota must be greater than 21,000 pages). Speak to your system manager for details.
5. After numerous transfers of large 2-D data sets, and checking by re-transferring and using the VMS "differences" command, we have not encountered any corruption of data. A typical transfer time is as follows (512 fids, 2048 points each) -
 500 to 400: 40 minutes
 400 to VAX: 5 minutes
 fidpack/varian1d: 30 minutes; or 5 minutes if performed in memory

We hope this information is useful. We thank Dr. Art Pardi and Dr. Dennis Hare for helpful comments.

Sincerely yours,

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

HIGH CAPACITY WINCHESTER DISK DRIVE SYSTEM for BRUKER ASPECT COMPUTERS

NEW



Fully compatible with your Bruker or IBM Instruments Aspect 2000A or 3000 computer system. **167 MegaBytes!** Highly reliable NEC Winchester Disk Drive. These are brand new, custom-manufactured to MR's exacting specs. Easily user installable — required cables and installation instructions included. Standard 19" rack mount or stand-alone case. Priced 30% less than the price quoted by a well-known manufacturer! Industry standard 90 day warranty on parts and labor.

Model MDD-160F

167 Mbyte NEC Winchester Disk Drive (8" form factor) with built-in Mitsubishi 8" Floppy Drive with 151k words formatted capacity. **ASPECT STANDARD.** Brushed aluminum finish. 120/220 VAC available; specify when ordering.

\$8749.00

Model MDD-160

As above, but without floppy drive

\$7995.00



All prices FOB Gardner, Massachusetts.
Quantity discounts available; call factory.

MR Resources, Inc.
P.O. BOX 880
158 R MAIN STREET
GARDNER, MASSACHUSETTS 01440
(508) 632-7000
(800) 443-5486

CONTACT OUR SALES DEPARTMENT FOR FURTHER INFORMATION.

ATTENTION BRUKER & IBM INSTRUMENTS NMR USERS

MR Resources has purchased IBM Instruments' NMR Inventory. This stock is original BRUKER equipment and is fully compatible with all Bruker and IBM Instruments NMR spectrometers.

Special Sale! Limited Inventory!

We have for immediate sale a number of Aspect Computer Boards, Disk Drives, Probes, RF Modules, and other NMR Components and Accessories. Call for a complete list.

ASPECT BOARD REPLACEMENT and REPAIR SERVICE

We have available for service exchange every* board for the Aspect 3000 and 2000A NMR Computer. Send us your repairable board and we will ship you an exact working replacement.

Every board in stock for immediate delivery!

Guaranteed performance. 90 day warranty.

BOARD

EXCHANGE PRICE

• Digitizers	\$750.00
• Array Processor	750.00
• Processor Controller	750.00
• Disk Interface (2000A)	600.00
• Sweep Control	500.00
• 10 Real Time Clock Pulser	500.00
• Graphics Display Processor Boards	250.00
• All other 2000A and 3000 boards	500.00
• Aspect 2000 boards	CALL
• Aspect Power Supplies	CALL

MODULE REPAIR SERVICE

We also provide complete and quick repair services in our shop for the following Bruker and IBM Instruments NMR system modules:

- Graphic Display Processors and Monitors
- Keyboards
- Printers
- Disk Drives

MR Resources offers the best service and fastest response time in the industry. All work fully guaranteed.

*Sorry, BC 130/140 Series fast digitizers, MSL, CXP, and imaging process controller boards not available for swap. These boards can, however, be repaired in our shop. Prices subject to change without notice.



MR Resources, Inc.
P.O. BOX 880
158 R MAIN STREET
GARDNER, MASSACHUSETTS 01440
(508) 632-7000
(800) 443-5486

CONTACT OUR SALES DEPARTMENT FOR FURTHER INFORMATION.

PENNSTATE



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

Department of Radiology

P.O. Box 850
Hershey, Pennsylvania 17033
(717) 531-8044

March 16, 1989
(received 3/20/89)

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

Dear Barry:

Cerebral Monitoring of ^{31}P Metabolites in Unanesthetized Neonatal Rats

We have begun an investigation of hypoxic-ischemic injury in seven day old rat pups. Since the neonatal rat has very little muscle strength and is content to remain for long periods in a dark, warm and restricted space, it is an ideal model for the study of unanesthetized cerebral metabolism by NMR. The hypoxic insult was provided by the administration of 8% oxygen to the neonates, who had their right common carotid artery ligated four hours earlier. This procedure will produce well documented cellular damage and changes in tissue metabolism in the anterior region of the right hemisphere (1). Specific localization of phosphorus metabolites have been performed using a 7 x 10 mm elliptical single-turn surface coil limited to the temporal region of the brain (Figure 1). A resulting eight minute spectrum is also shown in Figure 1.

We used a Bruker AM-400 WB spectrometer equipped with an animal holder which was constructed from a spare probe body. A plexiglass chamber, constructed from a split 70 mm diameter cylinder, was used for easy access and positioning of the animal and surface coil. In order to perform the experiments unanesthetized, each rat pup was wrapped and surrounded with gauze pads and thin surgical tape was used to precisely tape the head to the surface coil with minimal discomfort for the length of the experiment. The open end of the chamber was sealed with plastic wrap forming a small environmental gas chamber. Two small holes were cut in the plastic wrap as an exit port for the gas and to allow for adjustment of the Jackson Brothers tuning and matching capacitors. Mixtures of gas containing either 8% or 21% oxygen were maintained as monitored with a flow meter. The chamber temperature was regulated at 36-37°C with a waterbed and thermocouple arrangement. The waterbed consisted of a spiral geometry of 5 mm Tygon tubing which was glued to the plexiglass body. Water was heated with an externally located Haake bath.

Figure 2 shows a typical time course of metabolites during 21% oxygen (control), 8% oxygen and a subsequent 21% oxygen recovery period. Each spectrum required 7.5 or 15 minutes of 2 second repetitions of 8 μsec 70° rf pulses. In this example, PCr/P_i is found to vary from 1.7 to 0.24 during the three hours of hypoxia. Adenosine triphosphate, ATP (monitored most accurately from the -16 ppm β -phosphorus resonance), decreases

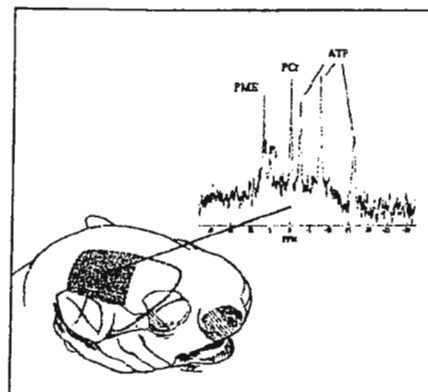


FIGURE 1

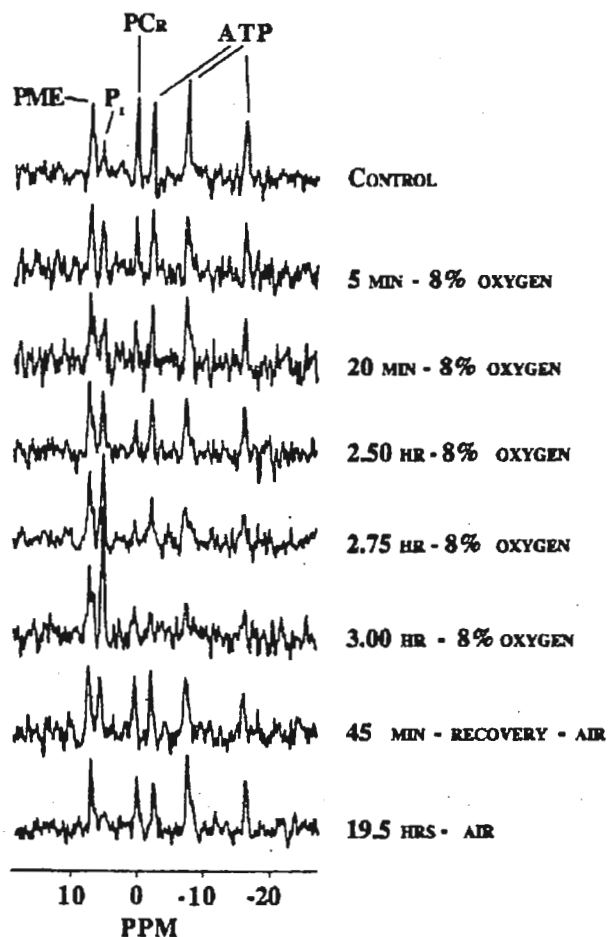


FIGURE 2

to less than one-half of its control value during the last hour, when the stroke occurs. Brain pH (measured from the P_i -PCr frequency) dropped by several tenths immediately after hypoxia and then partially recovered towards the normal value of 7.20. We believe this model to eliminate in a simple manner, the problems of depression of cerebral metabolism by anesthetic agents while providing stability and high signal-to-noise to test a number of questions concerning hypoxia/ischemia or neonatal asphyxia.

(1) Palmer C, Brucklacher RM, Vannucci RC. Ped. Res. 23(4):Part 2, 555A, 1988.

Sincerely,

Michael B. Smith, Ph.D.
Associate Professor of
Radiology and Physiology
Bitnet: MBS@PSUHMED

Gerald D. Williams, Ph.D.
Research Associate of
Radiology and NMR Lab Mgr.
Bitnet: JERRY@PSUHMED

Charles Palmer, M.D.
Assistant Professor of
Pediatrics

FIFTH INTERNATIONAL SYMPOSIUM

on Magnetic Resonance in Colloid and Interface Science

August 7-11, 1989

BITNET: ABL00354 @ UDACSVM
FAX: (302) 451-6335

March 21, 1989

NEWARK 1989

Dr. Barry Shapiro
Editor, Texas A and M NMR Newsletter
966 Elsinore Court
Palo Alto, CA 94303

Dear Barry:

I would like to make readers of the Newsletter aware of the Fifth International Symposium on Magnetic Resonance in Colloid and Interface Science, to be held at the University of Delaware, August 7 - 11, 1989. Like previous symposia at San Francisco, Menton, Torun and Muenster, this one is loaded with papers on magnetic resonance. Plenary lecturers include T. M. Duncan, H. Pfeifer, G. Martini, P. Lenahan, R. Griffin, P. Ellis, F. Blum and A. Legrand. In addition, contributions, both posters and talks, are invited.

Readers wishing more information on the symposium should contact me at the BITNET or FAX address above, or write to me at: Department of Chemistry and Biochemistry, University of Delaware, Newark, Delaware 19716. The preregistration deadline is June 1, 1989, which is also the deadline for submission of abstracts of talks and posters. I hope to see many of our colleagues and friends at this event, as it returns to the United States.

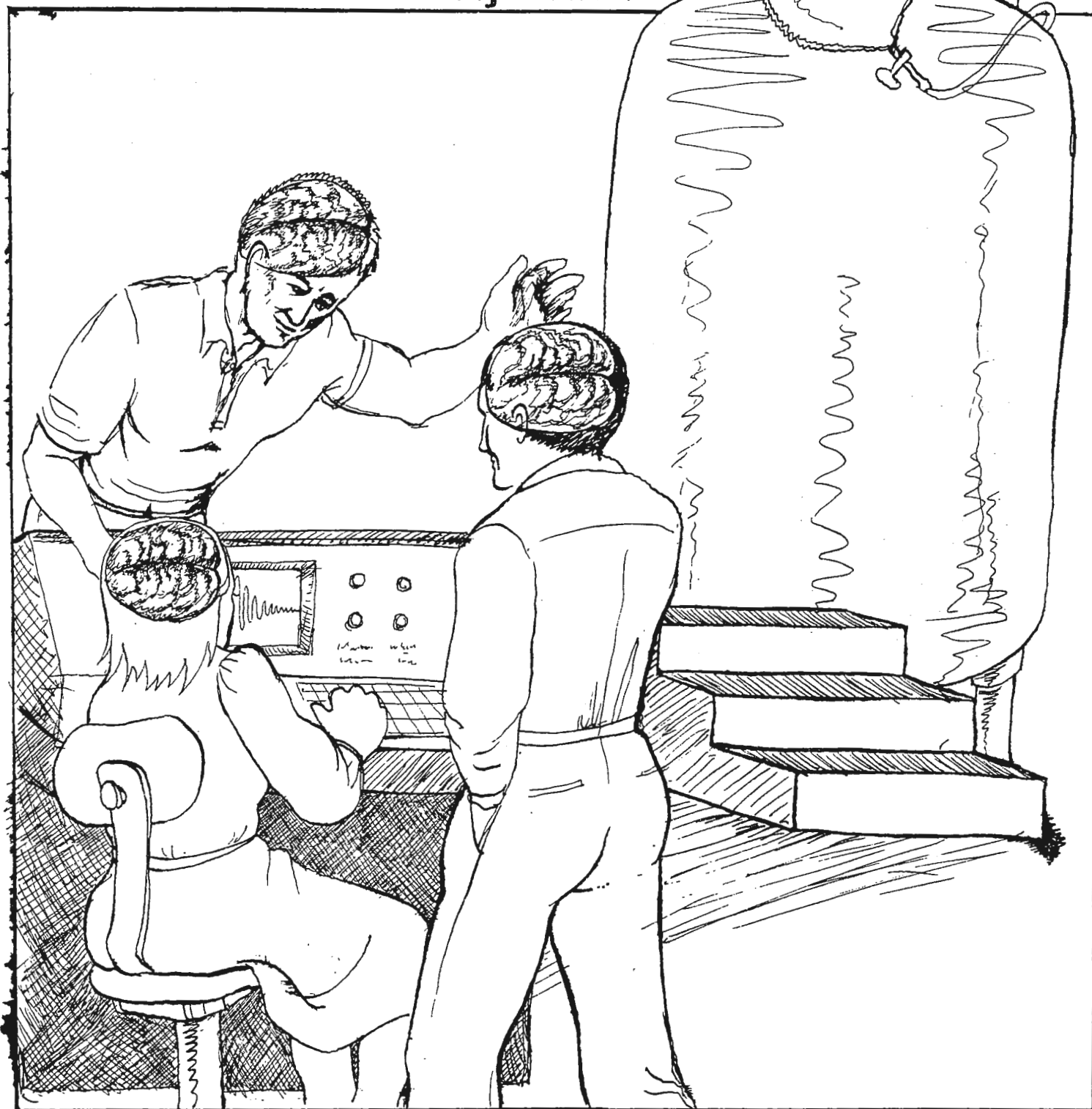
Yours truly,

Cecil

Cecil R. Dybowski
Professor of Chemistry
Symposium Chairman

CRD/djt

LIFE IN THE ROTATING FRAME



WENDY GOLDBERG '88

High Field NMR: 90° Pulse

TAMU NMR Newsletter

Policies and Practical Considerations

(Revised February 1989)

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 in its thirty-first 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 US\$120.00 per year, with a 50% academic or personal discount. Subscriptions are available only for the twelve monthly issues which begin with the October issue and run through that of the following September. However, a subscription can be initiated at any time, and the issues back to the previous October will be provided as long as copies remain available.

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

Continued

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

Another major, indeed most essential, source of funds for the Newsletter is **Advertising**. We earnestly encourage present and potential participants of the Newsletter to seek advertising from their companies. 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. Contributions may not exceed three pages without prior approval. Each page must have 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).

Foreign subscribers 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.

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.

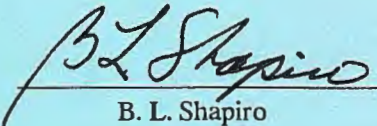
ii) **PLEASE avoid excessive margins.** *Instruct your secretaries to avoid normal correspondence esthetics or practices, however time-honored or 'standard'!* This page has margins on both sides of 0.6" (ca. 1.55 cm), which is very adequate. Margins of the same size at the top and bottom are sufficient 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.

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
Palo Alto, California 94303
U.S.A.

Telephone: (415) 493-5971.

16 February 1989

**Lilly Research Laboratories**

A Division of Eli Lilly and Company

Lilly Corporate Center
Indianapolis, Indiana 46285
(317) 276-2000February 21, 1989
(received 2/25/89)Mr. Barry Shapiro
Editor/Publisher
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto, California 94303

Dear Barry:

PRODUCT OPERATOR FORMALISM ON THE PC

Like many organic chemists with, uh, limited backgrounds in the theory of NMR and physics in general, I welcomed the "invention" of product operator formalism¹. Here, perhaps, was a method that I could use to understand just what it was that was going on inside that NMR tube. I had tried density matrices and they worked fine, but their complexity inhibited me from pushing them past the two-spin stage. I had a brief go at something called superoperators, but I didn't even get up to one spin with them. However, the use of product operator formalism (hereafter "POF") seemed to require only algebra and a little trigonometry, and I had managed to pass those courses in college.

I was a little less enthusiastic after I had used up several tablets in POF calculations. Even after switching to blackboards and a tree-like representation², I found that the method was rather tedious and very unforgiving of human error. Jon Paschal and I spent several weeks in the conference rooms, filling the blackboards with calculations that people seemed to delight in erasing before we could transfer them to more appropriate mass storage devices. One day Jon asked in exasperation why we didn't use a computer to do all the work. In the course of explaining why computer algebra is so difficult³, it occurred to me that perhaps I could use PROLOG, an

¹O. W. Sorensen et al., "Progress in NMR Spectroscopy," 16, 163 (1983).

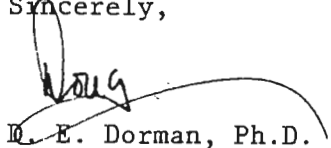
²F. J. M. van de Ven and C. W. Hilbers, J. Magn. Reson., 54, 512 (1983).

³R. Pavelle in T. H. Pierce and B. A. Hohne (eds.), "Artificial Intelligence Applications in Chemistry," ACS, 1986, chapter 8.

artificial intelligence programming language that I was attempting to learn, to do at least part of the job. I took a lap-top computer to the next conference I attended and spent the evenings trying out my ideas. I came up with a program, POFFIE, which did most of the work, but it required the user to work within the PROLOG environment, and Jon didn't seem to think that was acceptable. So I added a primitive user interface; and as a result, we now have the program, POFFINT, which can do a lot of the hard work involved in the use of product operator formalism.

Ironically, since that time I have been so busy trying to keep some of our older spectrometers going that I have not had much time to use POFFINT. Hence, I have not really explored its potential, if any, and I have virtually no documentation. We hope to get back to the program this year. In the meantime, if there is anyone out there who has a PC and wants to try it out, please let me know. I think that it should be reasonably portable, since it contains no graphics, but so far we have only tried it on an IBM XT, a Toshiba 3100 lap-top, and some AT and 386 compatibles.

Sincerely,


 D. E. Dorman, Ph.D.
 Research Scientist

TAMU NMR Newsletter: *Incredibly Important Notice.*

When a Newsletter subscriber/participant lets his (rarely hers) subscription lapse by ignoring the pink 'Reminder' and 'Ultimatum' notices, the name is removed from the mailing list according to our time-honored recipe (*q.v.*). More often than not, this is followed by remorse and a feeling of deprivation by the erstwhile communicant. This in turn usually results in a late technical contribution being sent in, with the request for reinstatement on the roles of the righteous. While I am willing to accomodate the tardy (with whom I readily identify), doing so makes a non-trivial amount of extra work. Extra work makes me grumpy, for it cuts into the time for other more valued activities. Thus deprived I tend to be uncharitable.

Therefore, to aid in keeping subscriptions in good standing, the following practice is now in effect: For the issue of the month after an 'Ultimatum' notice is sent out, the mailing label will be adorned with a large red dot or circle. This decoration means that no further issues will be sent until a technical contribution has been received. Please respond without delay to the receipt of a red dotted issue and save me the trouble of removing and re-adding your name from the mailing list. Better, of course, is to respond to the 'Reminder' or 'Ultimatum' notice. Thanks for your cooperation.

The requirement of an occasional technical contribution must, of course, continue, and any active NMR person/group/facility/department, etc., can surely generate one letter of interest per 9 or 10 months. Someone with a temporary difficulty can always discuss the problem with me. The possibility exists that someone with an occasional productivity or clearance problem can keep his/her subscription alive by providing a Book Review in lieu of the more usual type of technical contribution; please check with our book review editor, Professor Wm. B. Smith (Chemistry Department, Texas Christian University, Fort Worth, TX 76129) or me before doing this.

B. L. S.
 22 March 1989

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 occurred 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 $32 \times 256 \times 256$ DEFT data set.

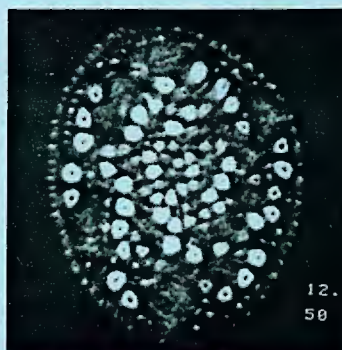


Fig. 1—Agapanthus bud
Matrix 256×256 , TR 200
Slice 2 mm, TE 30
FOV 12.8 mm, NEX 4,
45° Tip Angle DEFT
Sequence



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.



GE NMR Instruments

255 FOURIER AVENUE, FREMONT, CA 94539
(415) 683-4408, TELEX 910 381 7025 GE NMR FRMT
PRAUNHEIMER LANDSTRASSE 50, D-6 FRANKFURT 90
WEST GERMANY 4969 760 7431, TELEX 041 2002 GEG

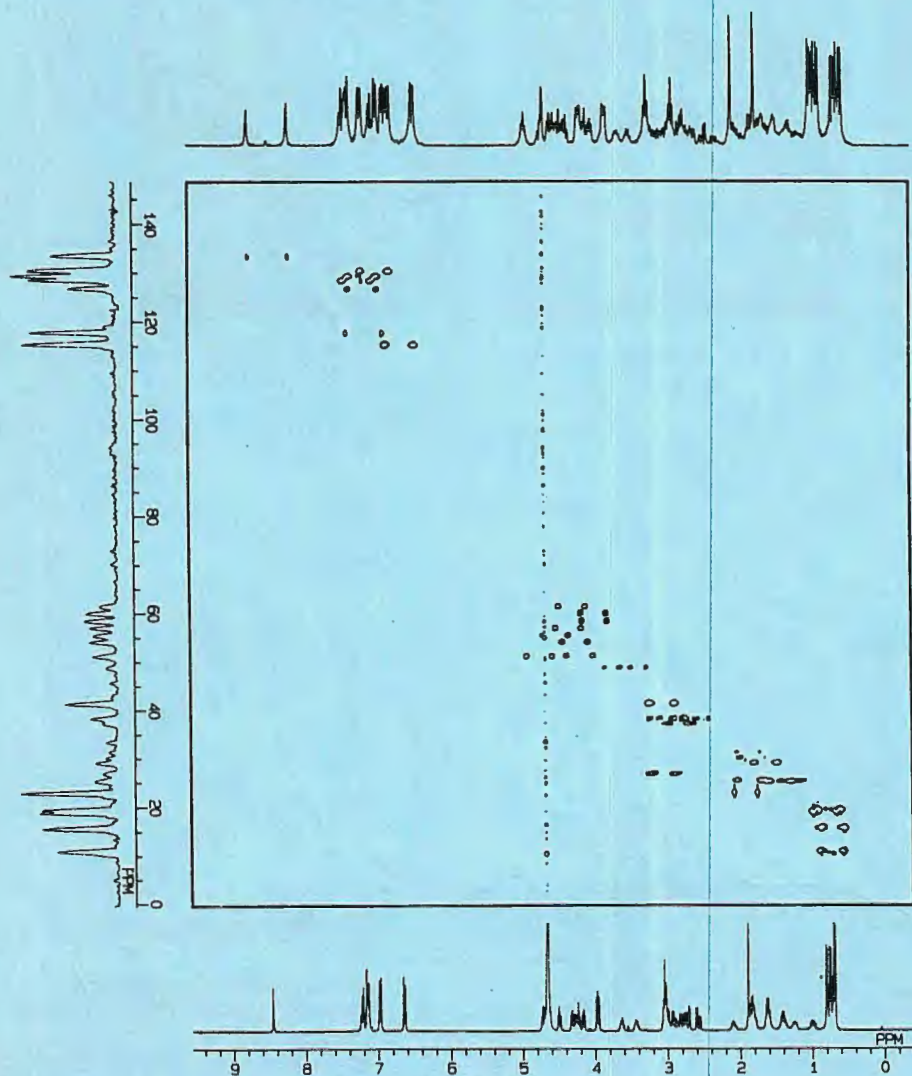
JEOL GSX-FT NMR Systems

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 (^1H [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.D2O



For further information call:

JEOL

Serving Advanced Technology

11 Dearborn Road, Peabody, MA 01960
(617) 535-5900