TEXAS A&M UNIVERSITY

NMR NEWSLETTER

No. 394

July 1991

TABLE 1 DEUTERATED SOLVENTS

Cat. No.	Description	Formula	Min. Isotopic Purity (%D)	Density (g/cc)	MP (°C)	BP (°C)	$-\chi_v \times 10^6$ @ (°C)
D-11	Acetic Acid-d_4	CD_3COOD	99.5%	1.12	17	118	0.551 (32)
D-120	Acetic Acid-d_4		99.5%				
D-13	Acetone-d_6	CD_3COCD_3	99.8%	0.84	-94	57	0.460 (20)
D-121	Aceonte-d_6 + 1% TMS		99.8%				
D-129							
D-14							543 (20)
D-21							0.611
D-122							
D-130							
D-28	Chloroform-d	$CDCl_3$	99.8%	1.50	-64	62	0.740 (20)
D-31	Chloroform-d + 1% TMS		99.8%				

Need Deuterated Solvents?

WILMAD HAS YOUR SOLUTION!

Cost-conscious quality NMR solvents offered by Wilmad, such as $CDCl_3$, are frequently priced lower than more traditional sources. Included in this offering are the most common solvents, like Acetone-d_6, Benzene-d_6, D_2O, and DMSO-d_6, as well as some of the most unusual solvents for specialty applications, like 1,1,2,2-Tetrachloroethane-d_2, Octane-d_8, and Trifluroacetic Acid-d.

VARIAN

BOX/500 SHEETS

Cat. No.	Sheet Size	Instrument Model	Type
WCV-100 (S-100A)	11" X 26"	HA-100, HA-100D	Gridded-Two Color
WCV-60 (A-60C)	11" X 26"	A-60, A-60A	Gridded-Two Color
WCV-XL (XL-100)	11" X 26"	XL-100 (CW)	Gridded-Two Color
WCV-XLFT-100	11" X 26"	XL-100 (CW and FT)	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" X 17"	CFT-20, FT-80, FT-80A All Models 16 1/2" or 16	Gridded-Two Color Blank

Use Plotter Paper and Pens?

WILMAD HAS YOUR CHART!

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. 394, JULY 1991 AUTHOR INDEX

TEXAS A&M NMR NEWSLETTER NO. 394, JULY 1991 ADVERTISER INDEX

SPONSORS OF THE TAMU NMR NEWSLETTER

Abbott Laboratories
Bruker Instruments, Inc.
Burroughs Wellcome Co.
Cryomagnet Systems, Inc.
The Dow Chemical Company
The Du Pont Merck Pharmaceutical Company
Eastman Kodak Company
E. I. du Pont de Nemours & Company
GE NMR Instruments
Hitachi Instruments, Inc.
JEOL (U.S.A.) Inc., Analytical Instruments Division
The Lilly Research Laboratories, Eli Lilly & Company
Merck Sharp & Dohme/Isotopes
Millipore Corporation, Waters Chromatography Division
The Monsanto Company
New Methods Research, Inc.
NMR Technologies Inc.
Norell, Inc.
The Procter & Gamble Company, Miami Valley Labs
Programmed Test Sources, Inc.
Shell Development Company
Tecmag
Unilever Research
Union Carbide Corporation
Varian, Analytical Instrument Division

*The Newsletter's fiscal viability depends very heavily on the funds provided by our **Advertisers** and **Sponsors**. Please do whatever you can to let them know that their support is noted and appreciated.*

FORTHCOMING NMR MEETINGS

33rd Rocky Mountain Conference on Analytical Chemistry, Denver, CO, **July 28 - August 2, 1991**; For information on the NMR Symposia, contact Dr. H. Eckert, Dept. of Chemistry, Univ. of California at Santa Barbara, Goleta, CA 93106, (805) 893-8163; For general information, contact: P. Sulik, Conference Chair, Rocky Mountain Instrumental Laboratories, 456 S. Link Lane, Fort Collins, CO 80524, (303) 530-1169.

Tenth Annual Scientific Meeting and Exhibition, Society of Magnetic Resonance in Medicine, San Francisco, **August 10-16, 1991**; Contact: S.M.R.M., 1918 University Ave., Suite 3C, Berkeley, CA 94704; (415) 841-1899, FAX: (415) 841-2340; See Newsletter 391, 55.

Two-Dimensional NMR Spectroscopy (ACS Short Course), New York, NY, **August 23 - 25, 1991**; See Newsletter 392, 33.

New Developments and Applications of Magnetic Resonance and Optical Spectroscopies (ACS Symposium), New York City, **August 25-30, 1991**; See Newsletter 393, 48.

International Conference on NMR Microscopy, Heidelberg, Germany, **September 16 - 19, 1991**; See Newsletter 385, 28.

1991 Joint Meeting FACSS/Pacific Conference, Anaheim, California, **October 6-11, 1991**; NMR/EPR Program Section Chairman: Prof. Cecil R. Dybowski, Chemistry Dept., Univ. of Delaware, Newark, DE 19716. Contact: FACSS, P.O. Box 278, Manhattan, KS 66502-0003.

Eighth Australian NMR Conference, Lorne, Victoria, Australia, **February 2-6, 1992**; See Newsletter 391, 38.

Eleventh Annual Scientific Meeting and Exhibition, Society of Magnetic Resonance in Medicine, Berlin, Germany, **August 8-14, 1992**; Contact: S.M.R.M., 1918 University Ave., Suite 3C, Berkeley, CA 94704; (415) 841-1899, FAX: (415) 841-2340.

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. 396 (September) -------- 16 August 1991
No. 397 (October) -------13 September 1991
No. 398 (November) -------11 October 1991
No. 399 (December)-------8 November 1991

OSU

Oklahoma State University

DEPARTMENT OF CHEMISTRY
COLLEGE OF ARTS AND SCIENCES

STILLWATER, OKLAHOMA 74078-0447
PHYSICAL SCIENCES 106
405-744-5920

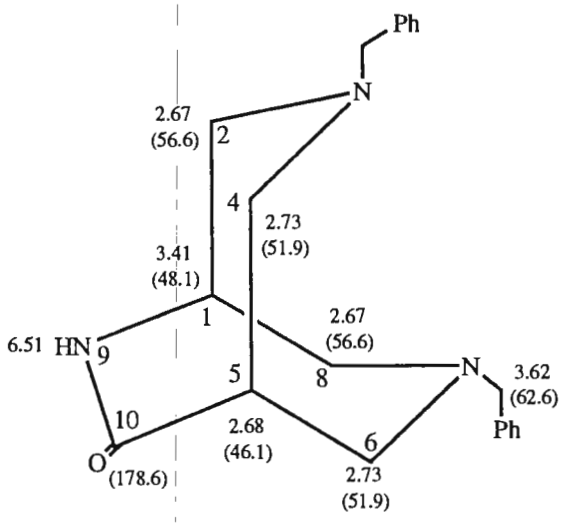
Dr. B. L. Shapiro
966 Elsinore Court
Palo Alto, CALIFORNIA 94303

June 17, 1991
(received 6/20/91)

TITLE: Proton and carbon analysis of 3,7-dibenzyl-3,7,9-triazabicyclo[3.3.2]decan-10-one

Dear Dr. Shapiro:

Our contribution this time concerns our studies on several novel heteroatom-substituted bicyclo[3.3.2]decane systems which are somewhat rare. We have obtained the following[1] and ^{13}C data on the example illustrated below. As one might expect, the nonequivalence of the protons alpha to nitrogen are observed as well as the bridgehead protons. Of course, there are sets of ^{13}C signals in the ring system which are nonequivalent. Although we assume a chair-chair conformation, we have not been able to eliminate a chair-boat from consideration in certain



^1H in δ values
(^{13}C in ppm)

[3,7-Dibenzyl-3,7,9-triazabicyclo[3.3.2]decan-10-one]

solvents. A HETCOR 2-D plot in $DCCl_3$ confirmed the assignments, but it was not possible even at 400 MHz to resolve individual signals of pseudo axial and pseudo equatorial protons for H(2,4) and H(6,8). These materials will serve as useful synthons for certain medicinal agents in our laboratory. We trust this will be acceptable for our contribution this time.

Sincerely yours,

Darrell

K. Darrell Berlin
Regents Professor

UNITY BRINGS NMR IMAGING DOWN TO SIZE

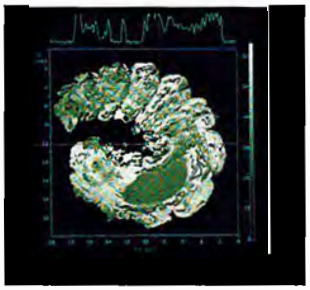
VARIAN COMBINES SPECTROSCOPY WITH MICROIMAGING

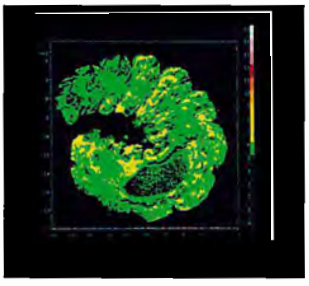
Built for the highest performance levels, the new UNITY™ NMR spectrometers combine high-field microimaging with high resolution spectroscopy. UNITY's state-of-the-art microimaging experiments for small samples address applications that range from biological and pharmaceutical to agricultural and material science.

Designed to bridge the operational gap between liquids, solids, and microimaging, this instrument can perform all three techniques and rapidly switch from one to the other with ease.

A wide variety of experiments can be accomplished at high performance levels due to the modular system architecture that's adaptable to a variety of configurations. Upgrading to additional capabilities can be accomplished at any time simply by adding the necessary system components.

Get the image of the future: invest in the most flexible technology of today to better progress toward the research of tomorrow. Invest in a UNITY NMR spectrometer. For additional information, please call the Varian office closest to you.





Varian is your full-line company for analytical instrumentation
NMR
UV-Visible-NIR
Atomic Absorption
Solid Phase Extraction
Liquid Chromatography
Gas Chromatography
GC/MS

Varian Associates
611 Hansen Way D-298
Palo Alto, CA 94303
U.S.A.
Tel: 1-800-356-4437

Varian AG
Steinhauserstrasse
CH-6300
Zug, Switzerland
Tel: (42) 44 88 44

Varian GmbH
Alsfelderstrasse 6
D-6100 Darmstadt
West Germany
Tel: (0 61 51) 70 30

Varian Instruments Ltd.
3rd Matsuda Bldg
2-2-6 Ohkubo-Shinjuku
Tokyo, 169, Japan
Tel: (3) 204-1211

NMR WITH A FUTURE

varian

Varian NMR Instruments

triple resonance Triple•nmr™ probe

For the most demanding and ambitious applications in Biomolecular NMR

Designed for high-field heteronuclear n-dimensional ***multiple resonance*** experiments
- e.g. Triple resonance experiments

Customer choice of two probes:
- ^{1}H observe $\{^{13}C/^{15}N\}$
- ^{13}C observe $\{^{1}H/^{15}N\}$

Extraordinarily ***high ^{1}H sensitivity***

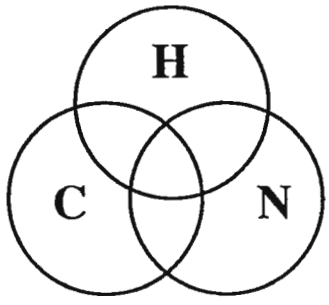
High performance for samples in buffered media

Short ^{13}C pulse widths for excellent broadband excitation

Robust under simultaneous high power pulses

Variable temperature 5 mm probe

Compatible with Varian's three channel UNITY® NMR Spectrometers



Order today!

Call your Varian sales representative. Australia (3) 543 8022. Austria (1) 69 55 450. Belgium (02) 721 4850. Brazil (11) 829 5444. Canada (416) 457 4130. Denmark (42) 846166. France (1) 69 86 38 38. Germany (6151) 70 30. Italy (2) 753 1651. Japan (3) 204 1211. Korea (2) 5611626. Mexico (5) 533 5965. Netherlands (3403) 50909. Norway (9) 86 74 70. Spain (01) 430 0414. Sweden (8) 82 00 30. Switzerland (42) 44 88 44. UK (932) 24 37 41. US 800-356-4437. Other International (408) 734-5370.

THE UNIVERSITY OF UTAH

May 17, 1991
(received 5/22/91)

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

Analysis of Spinning Sidebands for the Quadrupolar Nuclei with Half Integer Spin

Dear Barry,

NMR spectroscopy of quadrupolar nuclei in solids generally yields broad lines even when magic angle spinning is applied. Broadening of the central transition ($-1/2 \leftrightarrow +1/2$) of quadrupolar nuclei with half integer spins is caused by the anisotropy of the second order quadrupolar interaction. Recently we have developed a new approach for the simulation of lineshape for a quadrupolar nucleus under such a complicated variable angle sample spinning situation. It is known that an analytical expression for the central transition frequency can be obtained by average Hamiltonian theory (*1*). The perturbation in the modulation of the sample spinning can be written as a finite Fourier expansion in the angle $\omega_S t$:

$$\omega(\beta,\theta,\phi,t) = \sum_{m=-4}^{4} W_m \, e^{im\omega_S t}$$

with

$$W_m(\beta,\theta,\phi) = \left(\frac{e^2qQ}{2I(2I-1)\hbar}\right)^2 \frac{(I(I+1)-3/4)}{\omega_L} \sum_{l=0,2,4} \sum_{n=-l}^{l} A^l_n \, D^{(l)}_{n,-m}(\phi,\theta,0) \, d^{(l)}_{-m,0}(\beta),$$

where ω_S is sample spinning speed and the coefficient A are a function of asymmetry parameter η. β is the angle between spinning axis and external magnetic field. The Euler angles θ and ϕ specify the spinning axis in the principal frame of the electric field gradient tensor. The coefficient in the Fourier expansion W_0 gives the frequency of the central line, while the W_m with $m \neq 0$ describe the frequency modulation by the sample spinning which results in the sidebands. A method for computing the intensities of both the centerband and the sidebands for spin-1/2 nuclei has been reported by Sethi *et al* (*2*). The new method is readily extended to the case of quadrupolar nuclei and requires that only a set of banded homogeneous equations be solved to obtain the intensities for each spinning axis direction described by θ and ϕ:

$$\begin{bmatrix}
\ddots & \ddots & \ddots & \ddots & \ddots & 0 & 0 & 0 & 0 \\
\ddots & -3 & w_{-1} & w_{-2} & w_{-3} & w_{-4} & 0 & 0 & 0 \\
\ddots & w_{+1} & -2 & w_{-1} & w_{-2} & w_{-3} & w_{-4} & 0 & 0 \\
\ddots & w_{+2} & w_{+1} & -1 & w_{-1} & w_{-2} & w_{-3} & w_{-4} & 0 \\
\ddots & w_{+3} & w_{+2} & w_{+1} & 0 & w_{-1} & w_{-2} & w_{-3} & \ddots \\
0 & w_{+4} & w_{+3} & w_{+2} & w_{+1} & 1 & w_{-1} & w_{-2} & \ddots \\
0 & 0 & w_{+4} & w_{+3} & w_{+2} & w_{+1} & 2 & w_{-1} & \ddots \\
0 & 0 & 0 & w_{+4} & w_{+3} & w_{+2} & w_{+1} & 3 & \ddots \\
0 & 0 & 0 & 0 & \ddots & \ddots & \ddots & \ddots & \ddots
\end{bmatrix}
\begin{bmatrix} \vdots \\ C_{-1} \\ C_{-2} \\ C_{-1} \\ C_0 \\ C_{+1} \\ C_{+2} \\ C_{+3} \\ \vdots \end{bmatrix} = 0$$

where $w_m(\beta,\theta,\phi) = W_m(\beta,\theta,\phi)/\omega_S$. Since terms with fourth rank Wigner rotation matrices are included in the frequency expression, the width of the banded matrix is expanded to include -4 to $+4$, rather than the -2 to $+2$ range required earlier by second rank interactions. In the powder sample for the set of all crystallites for which the spinning axis lies in a particular direction defined by θ and ϕ the magnetization is given by

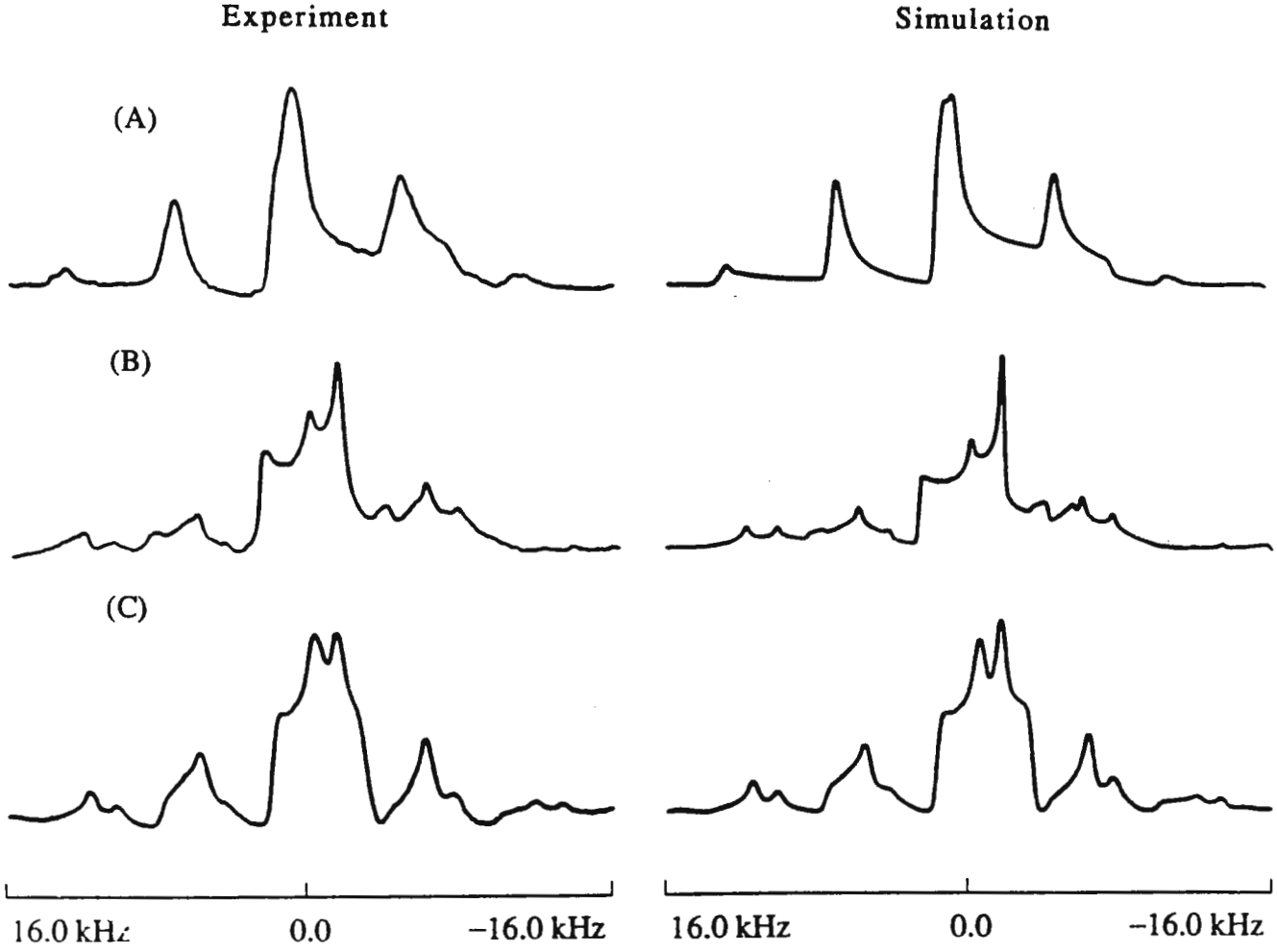
$$M_+(\beta,\theta,\phi,t) = \sum_N \left| C_N(\beta,\theta,\phi) \right|^2 \exp\{i[W_0(\beta,\theta,\phi) - N\omega_S]t\}.$$

Department of Chemistry
Henry Eyring Building
Salt Lake City, Utah 84112

This magnetization creates a series of peaks at frequencies $W_0(\beta,\theta,\phi) - N\omega_S$, each with intensity $|C_N(\beta,\theta,\phi)|^2$. Integrating θ and ϕ over the sphere produces the powder lineshapes including the spinning sidebands. The banded matrix method used in the simulations is computationally much more efficient than the standard method using Bessel functions. The complete details and analysis of this technique have been submitted for publication.

Shown in the following Figure are the experimental (left) and theoretical (right) ^{23}Na NMR spectra of Na_2SO_4 operating at 52.921 MHz. The angles of the spinning axis β are 45°(A), 54.7°(B) and 65°(C), respectively. The spinning speed is 6 kHz. Previously determined values of quadrupolar coupling constant $e^2qQ/\hbar$ = 2.6 MHz and asymmetry η = 0.6 were used in the simulation. Good agreement between the experiment and theory is obtained. Na_2SO_4 lineshapes are quite sensitive to β.

(*1*). A. Samoson, E. Kundla, and E. Lippmaa, *J. Magn. Reson.* **49**, 350 (1982).
(2). N. K. Sethi, D. W. Alderman, and D. M. Grant, *Mol. Phys.* **71**, 217 (1990).



Best wishes

Zhiwen Zheng D W Alderman Dave

Zhiwen Zheng D. W. Alderman David M. Grant

Not even a coffee break.

Seven days a week, twenty-four hours a day. For over three years, a QE automated NMR analytical spectroscopy system ran experiments at Searle Labs in Chicago with only a few hours of downtime.

This sterling work record helped Searle gather high quality data from both fully automated macro and manual routine experiments. Today, two QE automated systems:

- ► Accommodate the experiments of over 30 chemists
- ► Operate 24 hours a day, seven days a week
- ► Perform both manual and fully automatic macro experiments during the day
- ► Perform fully automatic macro programs at night and on weekends
- ► Adapt to a demanding user schedule of 15 and 30 minute time slots

Work smarter with the new QE Plus

With more speed and efficiency, the new QE Plus does the job even better—to meet your needs for automated performance day after day, year after year.

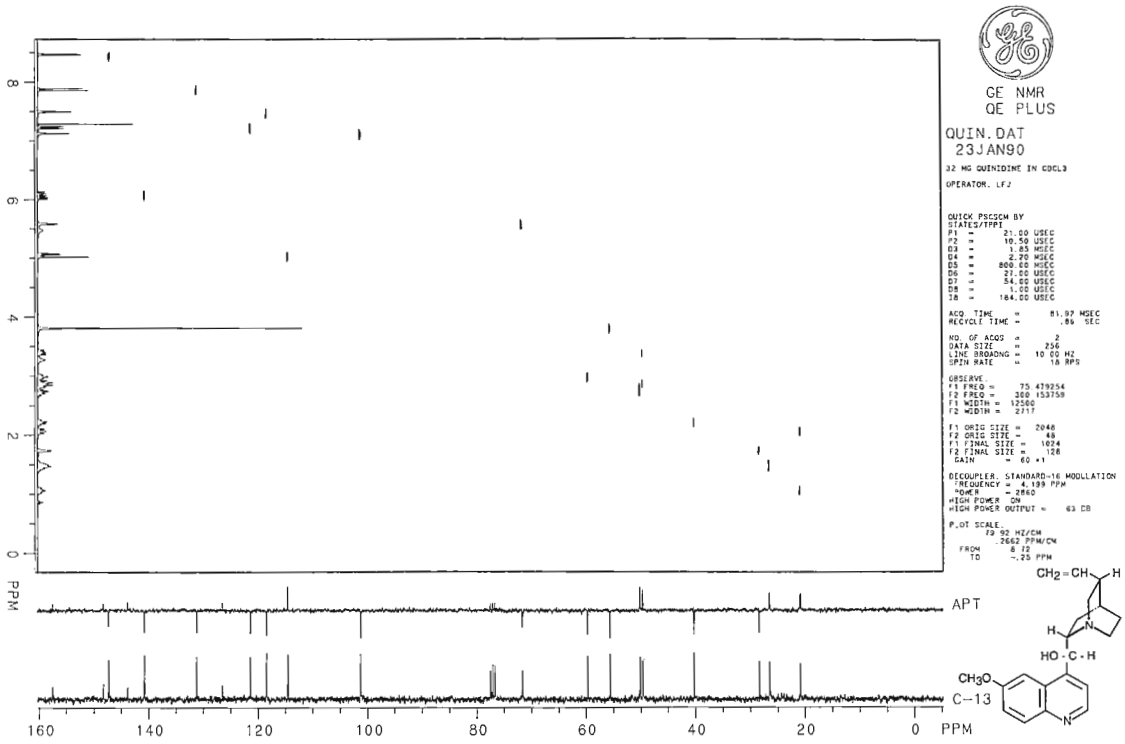
For more information on the new QE Plus, write 255 Fourier Ave., Fremont, CA 94539 for a full color QE Plus brochure. Or call 800-543-5934.

And get to work.

GE NMR Instruments

Put us to the test.

The new QE Plus—for increased throughput and greater automated capabilities.



An automated run using MACRO mode operation on a sample of 32 mg of quinidine in 0.5 ml chloroform-d (0.20M). Data were obtained using the 5 mm broadband probe. ^{1}H, ^{13}C, APT and phase sensitive 2D data were collected, processed and plotted—including the 2D contour—in only 8.2 min.

University of Missouri-Rolla

Joseph R. Duke
Department of Chemistry
142 Schrenk Hall
Rolla, Missouri 65401-0249
(314)-341-6453 (or 4420)
jd@blumiris.chem.umr.edu

May 30, 1991 (received 6/3/91)
Dr. B. L. Shapiro
968 Elsinore Court
Palo Alto, CA 94303

Dear Barry,

LOCAL ORDERING POTENTIALS IN ^{2}H POWDER PATTERN SIMULATIONS

SHBS forms lamellar liquid crystals with water. In these liquid crystals, the mobility of the phenyl ring is increased with respect to the dry surfactant. We have done lineshape simulations using two different models for phenyl group reorientation, free rotational diffusion and anisotropic viscosity. These are based on the method of Freed *et al.* (1-2) as adopted in our lab for ^{2}H NMR (3). For free rotational diffusion the motion is simulated by the inclusion of a fast reorientation about the molecular symmetry axis (the 1'-4' axis) (10 MHz), and a slower motion perpendicular to this axis (1 kHz). For anisotropic viscosity the motion is simulated by reorientation about a space fixed axis, the director axis. There are two rotational diffusion rates, one about the director axis (10 MHz) and one perpendicular to it (1 MHz). In this model, a restoring potential is used to mimic the intermolecular interactions which produce long range order. Spectra from the anisotropic viscosity model (bottom of Figure) seem to fit the experimental spectra best suggesting the head-group motion is restricted with respect to reorientation away from the liquid crystalline director axis, but allowed to rotate freely about this axis. The local ordering, therefore, dominates to large extent the spectral features. Thus, these experiments provide evidence of the local environment in terms of structure as well as dynamics. We have submitted a paper on this topic.

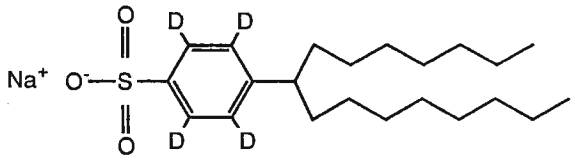
Incidentally, I am planning to graduate soon with a Ph.D. in the area of NMR spectroscopy. I would be pleased to know of any job openings in this area.

1. Freed, J. H. Spin Labeling: Theory and Applications, Vol. 1; Academic Press, Inc., New York, 1976.
2. Schneider, D. J.; Freed, J. H. Spin Labeling: Theory and Applications, Vol. 8; Academic Press, Inc., New York, 1989.
3. Jagannathan, S.; Polnaszek, C.F.; Blum, F.D. *J. Chem. Inf. Comput. Sci.* **1987**, *27*, 167.

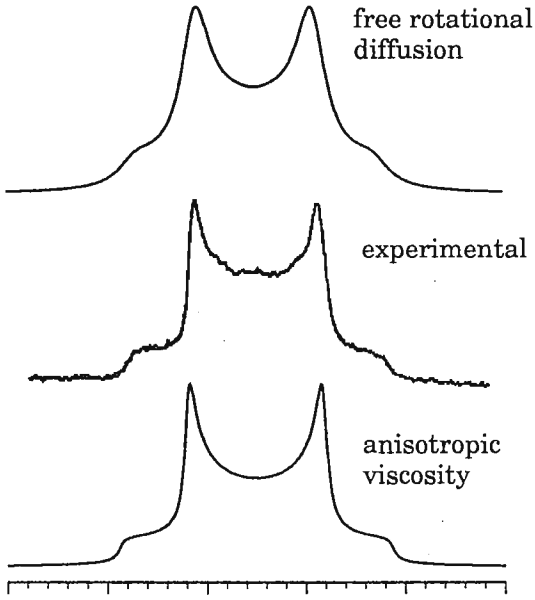
Sincerely,

Joe R. Duke

*Please credit this contribution to F. D. Blum.



Sodium 4-(1'-heptylnonyl)benzenesulfonate (SHBS)



^2H spectra for SHBS- d_4 in a liquid crystal. The top and bottom spectra are simulations.

POSITION AVAILABLE

UNIVERSITY OF QUEENSLAND, BRISBANE, AUSTRALIA

The University of Queensland Centre for Magnetic Resonance wishes to recruit a scientist to participate in a multidisciplinary in vivo magnetic resonance imaging and spectroscopy project. We are seeking a Ph.D. level person who should have experience with magnetic resonance imaging hardware design and construction, pulse programming and image processing software. Expertise with in vivo methodology is not a prerequisite. A major portion of the appointment, which is for three years in the first instance, will be spent in the laboratories of a multinational pharmaceutical company based in England, applying skills learnt at the University of Queensland, in a collaboration with an established biological magnetic resonance group within the company. The salary for this position is in the range $ Aust 40-44K while working in Australia, and at least £20K while in the UK. All removal and airfare expenses will be met. Interested persons should contact **Professor D M Doddrell, Centre for Magnetic Resonance, University of Queensland, Brisbane 4072, AUSTRALIA; Telephone 61-7-3654100, Fax 61-7-3653833.**

UXNMR* and WINNMR.*

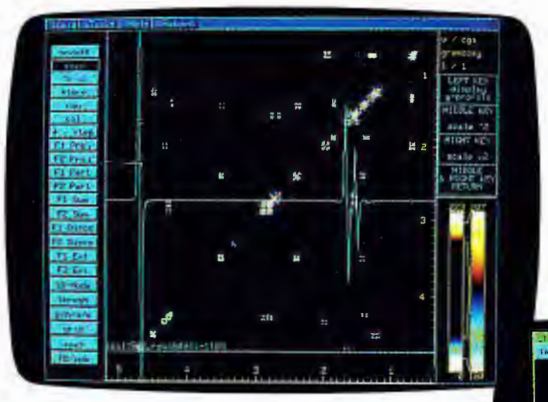
Bruker gives you exceptional freedom in the choice of industry-standard computers by delivering NMR software for multi-vendor networks. The UNIX*-based software (UXNMR) is available for SGI IRIS* workstations, IBM RS/6000* systems, and SUN* SPARCstations.* The new graphics use X-Windows* and the Motif interface toolkit. Via Ethernet* (TCP/IP), it can be run remotely from any X-server. Our software for personal computers (WINNMR) is based on MS-Windows* for 80X86 computers, and on Mac*-OS for Macintosh* IIs. WINNMR allows NMR processing with convenient access to all your favorite PC and Mac software packages. To see how you can enhance your NMR networking, simply ask for more information about our NMR software.

Standard software.

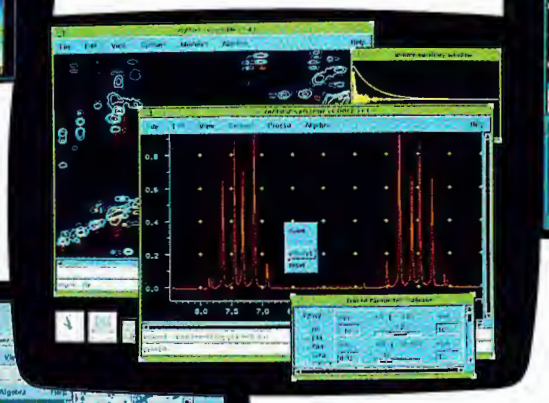
Exceptional freedom.

From Bruker.

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

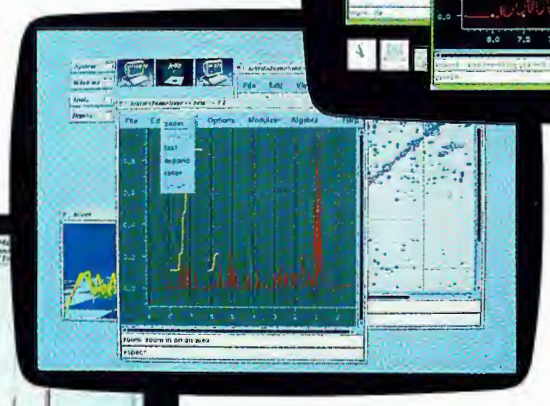


UXNMR on your ASPECT X-32.



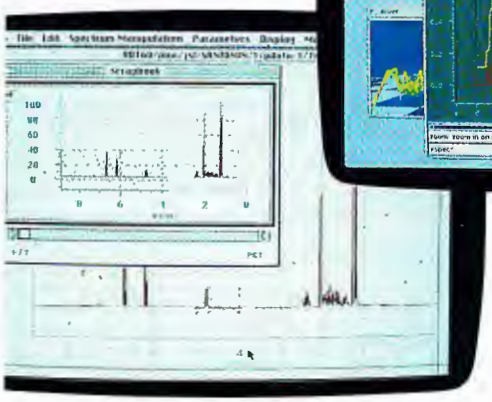
UXNMR on your SGI/IRIS.

UXNMR on your IBM RS/6000.



WINNMR on your Mac II or PC/PS system.

UXNMR on your SUN SPARCstation.

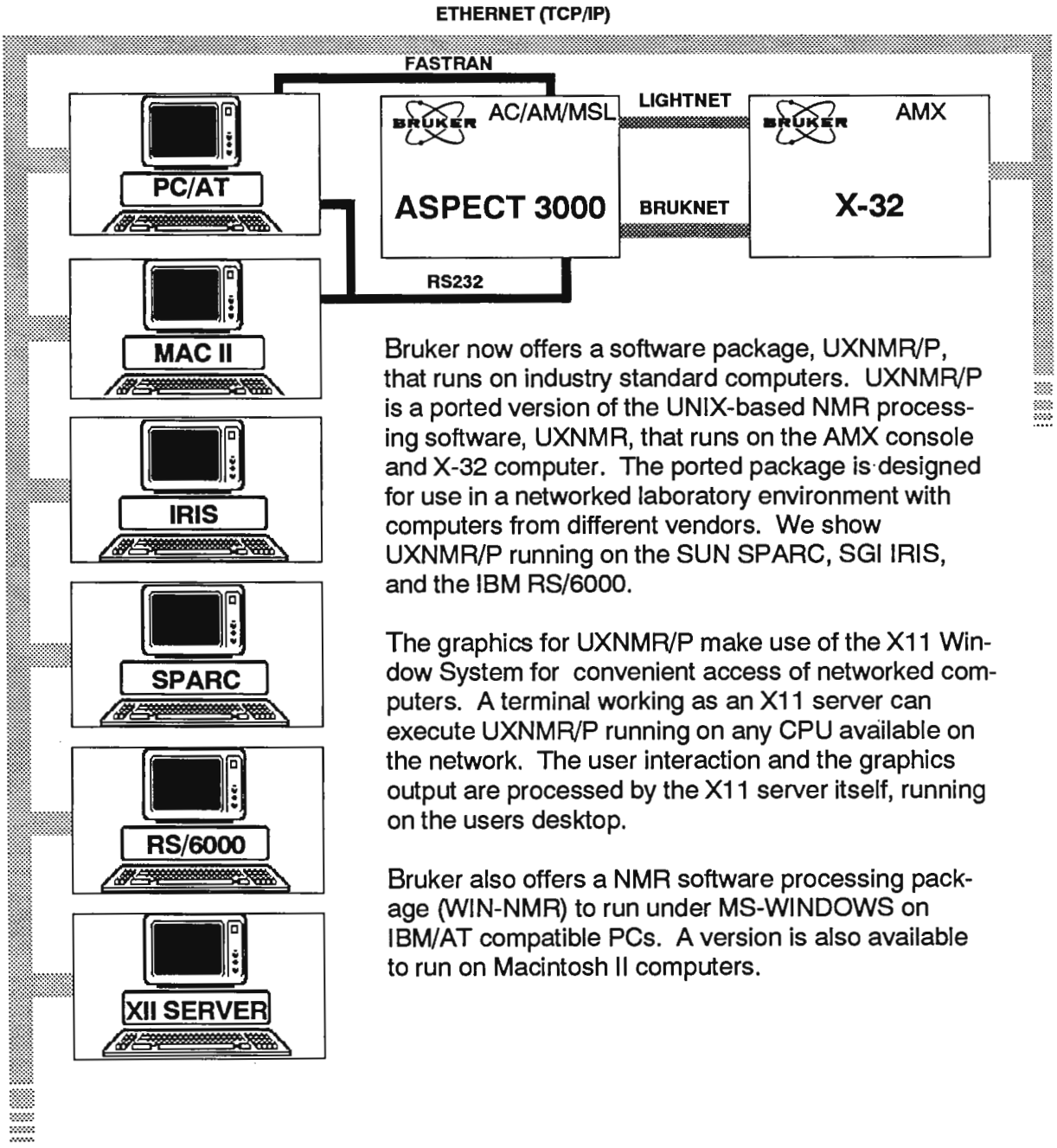


* UXNMR and WINNMR are trademarks of Bruker Analytische Messtechnik GmbH. UNIX is a registered trademark of AT&T. IRIS is a registered trademark of Silicon Graphics Inc. IBM and RISC system/6000 are registered trademarks of International Business Machines Corp. SUN is a registered trademark of Sun Microsystems, Inc. SPARCstation is a trademark of Sun Microsystems, Inc. The X-Window System is a trademark of Mass. Inst. of Tech. ETHERNET is a trademark of XEROX Corp. MS-Windows is a trademark of Microsoft Corp. Mac and Macintosh are registered trademarks of Apple Computer, Inc.

BRUKER

Comprehensive Support for Innovative Systems.

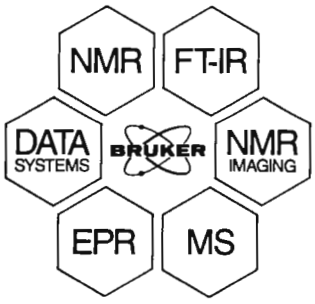
BRUKER SOFTWARE AND NMR NETWORKING



Bruker now offers a software package, UXNMR/P, that runs on industry standard computers. UXNMR/P is a ported version of the UNIX-based NMR processing software, UXNMR, that runs on the AMX console and X-32 computer. The ported package is designed for use in a networked laboratory environment with computers from different vendors. We show UXNMR/P running on the SUN SPARC, SGI IRIS, and the IBM RS/6000.

The graphics for UXNMR/P make use of the X11 Window System for convenient access of networked computers. A terminal working as an X11 server can execute UXNMR/P running on any CPU available on the network. The user interaction and the graphics output are processed by the X11 server itself, running on the users desktop.

Bruker also offers a NMR software processing package (WIN-NMR) to run under MS-WINDOWS on IBM/AT compatible PCs. A version is also available to run on Macintosh II computers.



Australia: BRUKER (Australia) Pty. LTD., Alexandria, New South Wales, Tel. 02-5506422
Belgium: BRUKER SPECTROSPIN S.A./N.V., Brussels, Tel. (02) 648 53 99
Canada: BRUKER SPECTROSPIN (Canada) LTD., Milton, Ontario, Tel. (416) 876-4641
England: BRUKER SPECTROSPIN LTD., Coventry, Tel. (0203) 855200
France: SADIS BRUKER SPECTROSPIN SA, Wissembourg, Tel. (088) 73 6800
India: BRUKER INDIA SCIENTIFIC Pvt. LTD., Andheri (West), Bombay, Tel. 22 62 72 32
Italy: BRUKER SPECTROSPIN SRL, Milano, Tel. (02) 23 50 09
Japan: BRUKER JAPAN CO. LTD., Ibaraki-ken, Tel. 0298-52-1234
Netherlands: BRUKER SPECTROSPIN NV, Wormer, Tel. (75) 28 52 51
Scandinavia: BRUKER SPECTROSPIN AB, Täby, Sweden, Tel. (08) 758-03-35
Spain: BRUKER ESPANOLA S.A., Madrid, Tel. 341-259-20-71
Switzerland: SPECTROSPIN AG, Fällanden, Tel. 1-82 59 111
Germany: BRUKER ANALYTISCHE MESSTECHNIK GMBH, Rheinstetten, Tel. 0721-5161-0
BRUKER ANALYTISCHE MESSTECHNIK GMBH, Karlstruhe, Tel. 0721-5967-0
BRUKER-FRANZEN ANALYTIK GMBH, Bremen, Tel. 0421-2205-0
USA: BRUKER INSTRUMENTS, INC., Billerica, MA 01821, 508-667-9580
Regional Offices in Chicago, IL, (708) 971-4300 / Wilmington, DE, (302) 478-8110
Houston, TX, (713) 292-2447 / San Jose, CA, (408) 434-1190

10/90

HMRI

HUNTINGTON MEDICAL RESEARCH INSTITUTES

WRITER'S TELEPHONE NUMBER:
(818) 397-8532

June 3, 1991 (received 6/10/91)

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

Dear Dr. Shapiro:

Observation of ^{15}N-labeled metabolites in isolated rat brain by ^{1}H-^{15}N HMQC.

Selective observation of ^{1}H coupled to ^{15}N by heteronuclear multiple quantum coherence (HMQC) spectroscopy permits indirect detection of ^{15}N-labeled compounds with a substantially higher sensitivity than direct ^{15}N NMR (1). Using this technique in Bruker AM-500 spectrometer, we have recently observed the amide protons of biologically ^{15}N-enriched [γ-^{15}N]glutamine in isolated rat brain in 2 min of acquisition time (Fig.A) (2). Comparison with the conventional ^{1}H spectrum of the same brain (Fig.B) shows that proton signals from all other cerebral metabolites not spin-coupled to ^{15}N were suppressed. Sensitivity enhancement over direct ^{15}N detection was 50-fold. In an isolated liver of a similarly ^{15}N-enriched rat, [^{15}N]urea protons were observed in 16 min (Fig.C). The HMQC method is likely to be effective for in vivo study of cerebral and hepatic nitrogen metabolism.

References.
1. A. Bax, R.H. Griffey, B.L. Hawkins, *J Magn Reson.* **55**, 301 (1983).
2. K. Kanamori, B.D. Ross, and F. Parivar, *J. Magn. Reson.* **93**, 319 (1991).

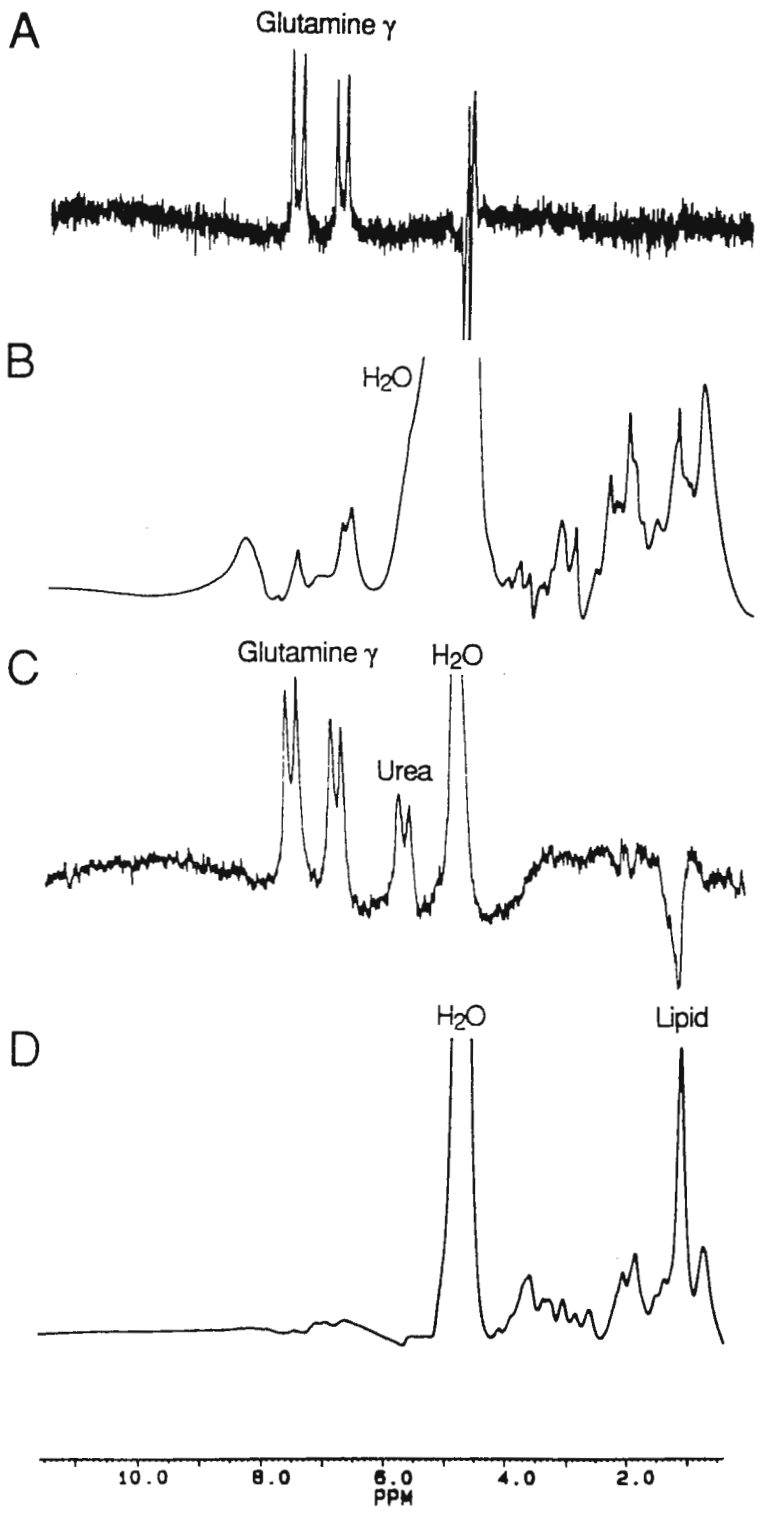
Yours sincerely,

Keiko Kanamori, Brian D. Ross, Farhad Parivar

Please credit this contribution to the subscription of John D. Roberts, Division of Chemistry and Chemical Engineering, California Institute of Technology, Pasadena, CA 91125

MAGNETIC RESONANCE SPECTROSCOPY LABORATORY
660 S. Fair Oaks Avenue, Pasadena, CA 91105

HMRI



MAGNETIC RESONANCE SPECTROSCOPY LABORATORY
660 S. Fair Oaks Avenue, Pasadena, CA 91105

Look to...

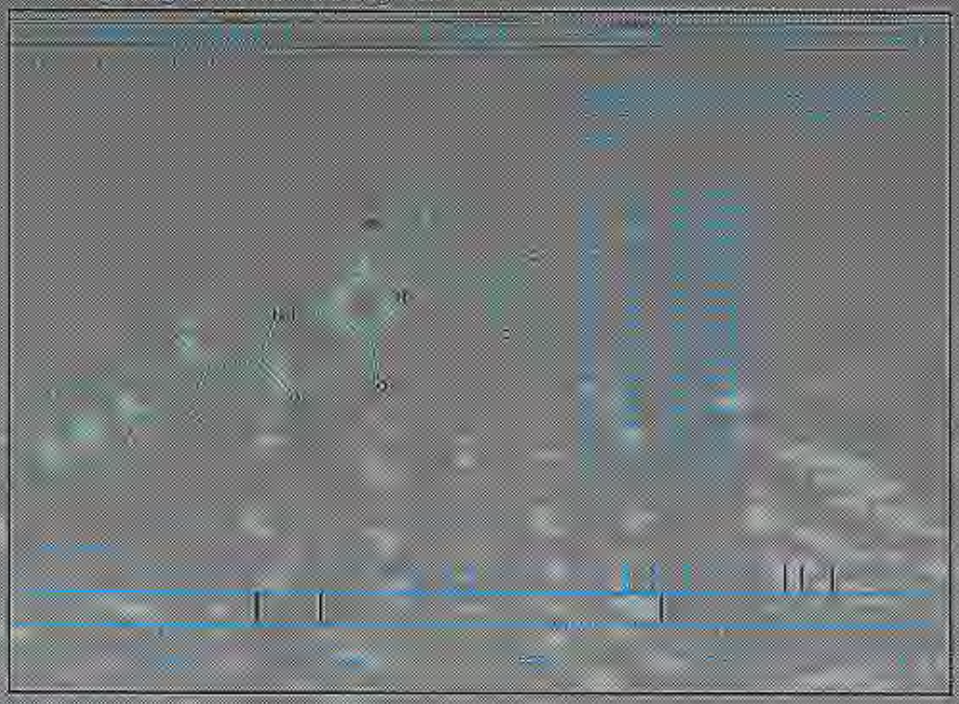
SADTLER

For:

- In House Data Management Systems
- Integrated Libraries and Spectra
- Structure Identification Using Integrated Techniques
- ASE Automated Data
- Spectral Processing Systems

CALL SADTLER TODAY and Keep Pace with the '90's

The assigned Carbon 13 below is from CSEARCH. Color is used in the APT spectrum to highlight the assignments



BIO-RAD

Sadtler Division

Main Office, 3316 Spring Garden Street, Philadelphia, Pennsylvania 19104
Telephone: (215) 382-7800. Telefax: (215) 662-0585. TWX: 710-670-1186.

CSEARCH

for NMR Assignment

Save days while applying your structure assignments against a database of structure and chemical shift data. CSEARCH will enable your laboratory to operate more efficiently by employing a computer to do what computers do best: routine assignments. Free up both yourself and your spectrometer in order to create time and energy for the more demanding problems. CSEARCH and our extensive databases provide you with the tools for success in today's hectic development cycle.

- Read spectrometer generated peak lists directly
- Search spectra for similar or identical chemical shift patterns
- Request all functional groups for a chemical shift
- Request chemical shift range for a functional group
- Locate spectra by substructure searching
- Retrieve data based upon author, source or properties
- Estimate spectra from a structure proposal

BIO-RAD
Sadtler Division
Main Office, 3316 Spring Garden Street, Philadelphia, Pennsylvania 19104
Telephone (215) 382-7800 Telefax: (215) 662-0585 TWX: 710 670-1186

NATIONAL TAIWAN UNIVERSITY DEPARTMENT OF CHEMISTRY
ROOSEVELT ROAD SECTION 4
TAIPEI, TAIWAN, REPUBLIC OF CHINA
PHONE: 3635357 • 3636359

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

(received 6/7/91)
31 May 1991

Hypophosphite Two–Spin Order Relaxation and Biological Applications

Dear Barry,

We have previously used ^{31}P spin–transfer, ^{31}P longitudinal relaxation measurements and ^{31}P pulsed field gradient NMR measurements to study the molecular dynamics and transport of the hypophoshite ion ($H_2PO_2^-$; HP) in human erythrocytes [1–3]. Later experiments revealed differential longitudinal relaxation in solutions of low viscosity (i.e., the rate of molecular reorientation is within the extreme motional narrowing limit). This differential relaxation is caused by chemical shift anisotropy – dipole–dipole cross–relaxation. The presence of this cross–relaxation gives rise to new methods with which to study biological systems. Selective study of cross–relaxation, which is extremely sensitive to reorientational correlation–time, has been shown to be a useful tool for elucidating relaxation mechanisms and molecular dynamics [4–9].

The first step in this series of experiments was to properly characterize the ^{31}P relaxation mechanism, including terms for cross–relaxation, in D_2O solution [10]. The relative orientation of the chemical shift shielding tensor with respect to the molecular axes was determined from two–spin order relaxation measurements (e.g., [7–9]). The chemical shift anisotropy–dipole–dipole cross–interaction gives rise to the two–spin order signal. Next, we studied HP ^{31}P two–spin order relaxation in H_2O, glycerol solutions and in suspensions of human erythrocytes with different cell volumes [11]. Using two–spin order relaxation to study the molecular dynamics of the hypophosphite ion has the great advantage of allowing the separation of the intra– and intermolecular (i.e., random field) contributions to the total relaxation rate. The results revealed that random field effects can have significant effects on the relaxation of species in biological systems, and must be accounted for if accurate estimates of the reorientational correlation time are to be obtained.

Differential line broadening [4–6], which occurs when the rate of molecular reorientation is outside the extreme narrowing limit, is observed for the proton–coupled ^{31}P spectrum of HP (a triplet) in a solution of albumin (see Fig. 1). We are currently investigating the use of two–spin order relaxation and differential line broadening measurements to study binding processes.

Yours sincerely,

Lian-Pin Hwang W-S. Price Ching-Lung Tsai

Lian–Pin Hwang William S.Price Ching–Lung Tsai Baw–Ching Perng

1. Price, W. S., P. W. Kuchel, and B. A. Cornell. 1989. *Biophys. Chem.* 33:205–215.
2. Price, W. S., and P. W. Kuchel. 1990. *J. Magn. Reson.* 90:100–110.
3. Price, W. S., and P. W. Kuchel, 1990. *NMR in Biomedicine.* 3:59–63.
4. Hwang, L. P., P. L. Wang, and T. C. Wong, 1988. *J. Phys. Chem.* 92:4753–4758.
5. Wang, P.–L., and Hwang, L.–P. 1988. *J. Phys. Chem.* 92:623–625.
6. Wong, T. C., P. L. Wang, D. M. Duh, and L. P. Hwang. 1989. *J. Phys. Chem.* 93:1295–1302.
7. Chang, W. T., P. L. Wang, and L. P. Hwang. 1989. *J. Chin. Chem. Soc.* 36:417–421.
8. Chang, W. T., P. L. Wang, and L. P. Hwang. 1990. *J. Phys. Chem.* 94:1343–1346.
9. Chang, C. F., and L. P. Hwang, 1991. *J. Chin. Chem. Soc.* 38:1–4.
10. Tsai, C.–L., W. S. Price, Y.–C. Chang, B.–C. Perng, and L.–P. Hwang. 1991. *J. Phys. Chem.* In press.
11. Price, W. S., B.–C. Perng, C.–L. Tsai and L.–P. Hwang. 1991. Submitted to *Biophys. J.*

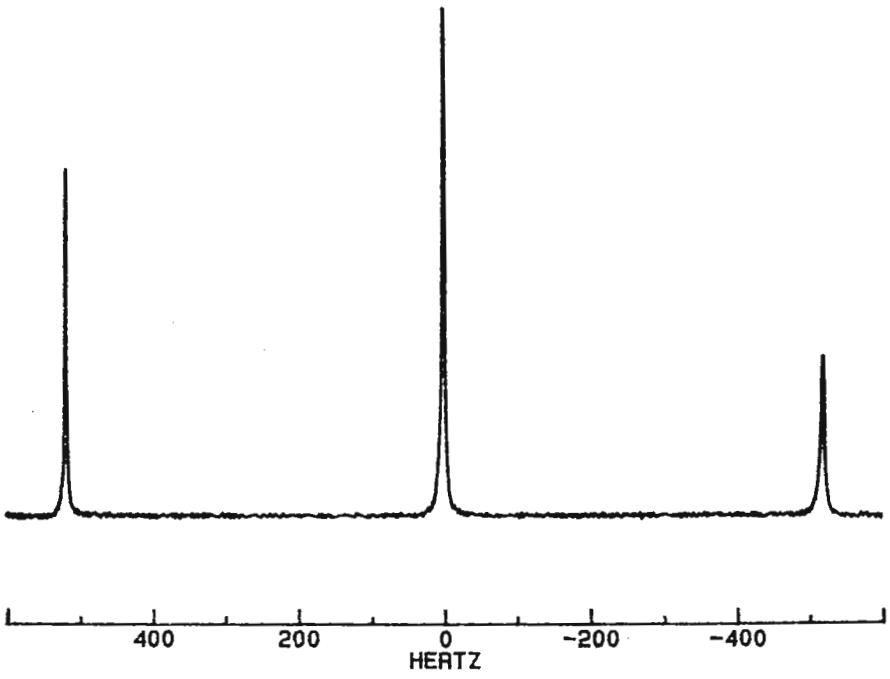


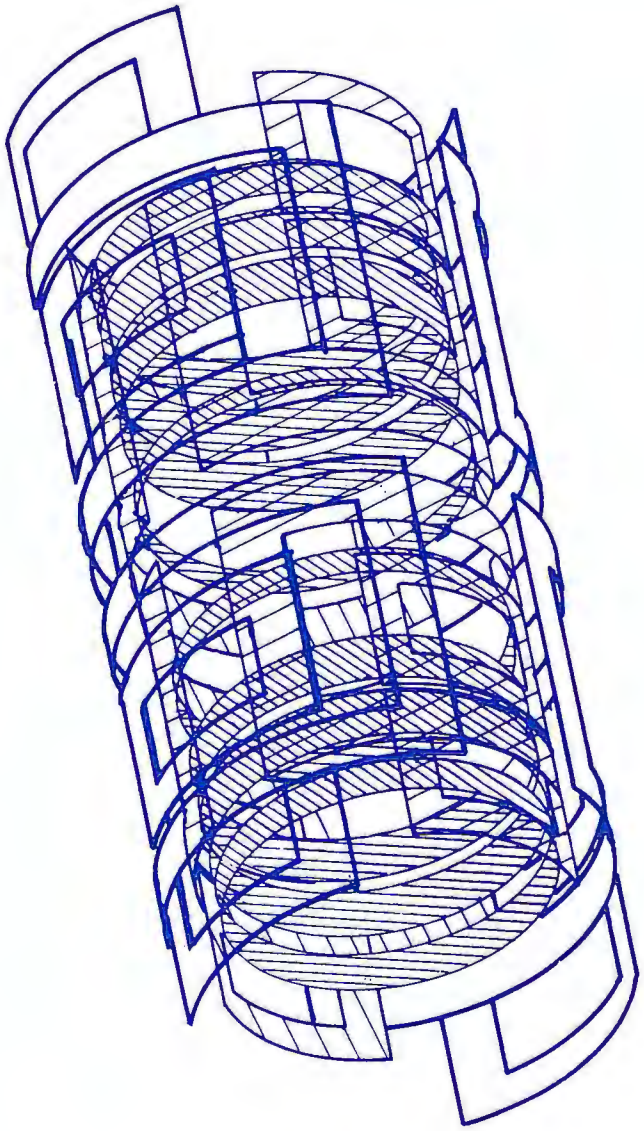
Figure 1. Differential linebroadening of the proton–coupled ^{31}P spectrum of HP (80 mM; pH 7.4) in bovine serum albumin (0.1 g/ml) at 121.4 MHz and 280 K. The effects of differential linebroadening are clearly shown by the difference in intensity of the satellite peaks. The line widths (from left to right) are 2.0, 3.3 and 5.5 Hz, respectively.

DOTY SCIENTIFIC:

EXPANDING THE BOUNDARIES OF NMR PROBE TECHNOLOGY

HIGH SPEED MAGIC ANGLE SPINNING

- 14 kHz specified spinning (air drive) in high-performance, multinuclear, 5mm VT MAS probes.
- Speeds over 17 kHz (air drive) under optimum conditions
- DAS - FAST FLIPPING VARIABLE ANGLE PROBES



LOW TEMPERATURE

- MAS at 1 kHz below 8 K.
- High resolution below 90 K.

HIGH TEMPERATURE

- 7mm, 4 kHz MAS above 650° C.
- High resolution to 380° C.

NMR MICROSCOPY

- X-Y-Z gradient coil systems for all supercon magnets.
- Pulse z-gradient fields up to 500 G/cm (4mm).
- Fully shielded design minimizes eddy current and pre-emphasis problems.
- Gradient linearity ± 2% over full sample volume.
- High sensitivity double-resonance rf systems.

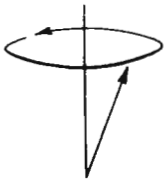
EXPANDING THE BOUNDARIES OF NMR

Doty Scientific began building Magic Angle Spinning probes over nine years ago. But we didn't stop there. You, the NMR scientists, have pushed the boundaries of NMR in every direction. We in turn have pushed the boundaries of NMR probes. You need reliable, state of the art probes to do your experiments. We design and build them.

In the last nine years we have been privileged to provide our customers with the first commercially available probes for a number of new NMR experiments. We're proud of our record.

THE DOTY SCIENTIFIC TIME LINE

- 1982 First 7 mm 5 kHz spinning
- 1982 First triple tuned MAS
- 1983 First single crystal probe
- 1984 First 5 mm 9 kHz spinning
- 1984 First multinuclear solids
- 1985 First 19 mm MAS
- 1987 First CRAMPS probe
- 1987 First actively shielded gradient probe
- 1987 First 5 mm 17 kHz spinning
- 1987 First 1000 G/cm pulse field gradient
- 1987 First 7 mm 9 kHz spinning
- 1988 First 6 Kelvin MAS
- 1988 First 650°C MAS
- 1990 First DAS probe
- 1991 First Supersonic 14 mm



Doty Scientific, Inc.

700 Clemson Road
Columbia, S.C. 29223
USA

Office: (803) 788-6497 Sales: (803) 699-3806
Fax: (803) 736-5495 Service: (803) 699-3807

UNIVERSITY OF WISCONSIN MADISON

DEPARTMENT OF CHEMISTRY

1101 UNIVERSITY AVENUE
MADISON, WISCONSIN 53706
FAX # 608-262-0381

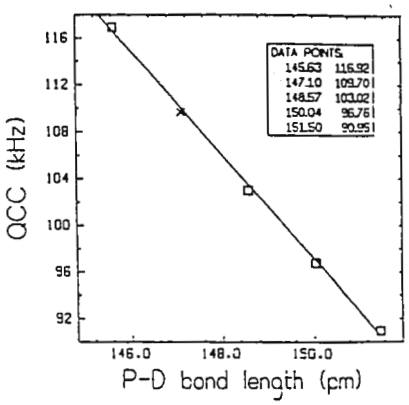
5 June 1991
(received 6/8/91)

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

RE: Deuterium Quadrupole Coupling Constants (Theoretical and Experimental).

Greetings, Barry!

We have recently been carrying out deuterium NMR relaxation time measurements and *ab initio* calculations of deuterium quadrupole coupling constants, χ_D, in small, garden variety molecules and ions, such as the phosphite ion, $PHO_3^=$. At a pH of 9 or higher there is no exchange (at least not over the period of five years that we have been monitoring a sealed sample) of the deuterium with the protons in the H_2O solvent. By measuring the ^2H longitudinal relaxation time as a function of temperature, one can obtain accurate experimental values for χ_D; the value we obtain is 94.7 ± 0.5 kHz. This result is in excellent agreement with Gaussian 86 *ab initio* calculations, *if* the proper bond length is used for the P-D bond. Because of vibrational motion the average P-D bond length is about three percent longer than the r_e value obtained from an *ab initio* calculation. The values for χ_D depend on the internuclear distance. For the phosphite anion they change as shown in the figure below. Using an r_{PD} value of 150.5 pm obtained from NMR measurements, the *ab initio* value obtained for χ_D in $PDO_3^=$ is 95.0 kHz, in excellent agreement with the experimental value. We would be most interested in hearing from other readers who have similar results on other molecules. Are we again a mailee in good standing? ✓
(ugh!)



Golf Ho!

Tom.

Thomas C. Farrar
Professor of Chemistry

TCF/mm

* Teach! ATS

394-22

Mobil Res. & Dev. Corporation
Dallas Research Laboratory
13777 Midway Road
Dallas, Texas 75244

June 14, 1991
(received 6/21/91)

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

Re: NMR Imaging of Pore Structures and Flow

Dear Barry:

Here at Mobil we have been getting very interesting NMR images of liquids in porous rocks from 1.0 to 100 mm diameter using our Bruker system with micro-, mini-, and macro-imaging probes together with a 15 cm bore vertical magnet and a 40 cm bore horizontal magnet. I wish to briefly mention some of our work performed with the mini-imaging probe.

NMR imaging can be used to map the pore space distribution in liquid-saturated porous media. When the resolution is sufficient, the actual pore structure can be imaged. Three-dimensional imaging (both spin-warp and back-projection) is especially powerful for obtaining the structure of a whole system of pores. We found that imaging carbonate rocks, with their very complex pore systems, gives very interesting results. By use of connectivity algorithms, we can select networks of connected pores and estimate liquid flow paths. Alternatively, we can set up the imaging protocol to greatly attenuate the imaging signal from the stationary liquid when liquid is flowing through the sample. The relatively unattenuated signal indicates the major flow paths. Because of dead-end pore space, the latter method is the more accurate. In some cases, we found that only a fraction of the connected pore space contains the liquid flow path. We have used NMR imaging to visualize the various types of pore structures and flow paths in limestones with two-dimensional slices and three-dimensional rendering.

Sincerely,

Don

D. E. Woessner

DEW/Clone

Instant Upgrade of RF Amplifier Performance in Your NMR/MRI System

Install an AMT 3000 Series solid-state pulse power amplifier—6–500MHz at up to 1000 W—into your system. Instant upgrade!

Here's just one example: AMT's RF power envelope detection system guarantees full protection. That means you can operate at low-level CW with full-power peaks on demand.

Pre-saturation water suppression? Cross polarization in solids? No problem—*now!*

Additional Key Features:

- **Broadband Frequency Ranges—6–220MHz, 200–500MHz**
- **Key Power for Liquids & Solids—50, 150, 300, 1000 Watts**
- **Excellent Linearity—(±1.0dB)**
- **Low Pulse Droop—typically less than 5%**
- **Fast Low Noise Blanking—within 20dB of KTB in 1 μs**

For full information call your NMR/MRI system manufacturer or contact Lowell Beezley at AMT:
PH (714) 993-0802
FAX (714) 993-1619

Models Available:		
3205	6–220MHz	300W
3200	6–220MHz	1000W
3137	200–500MHz	50W
3135	200–500MHz	150W
3134	200–500MHz	300W

AMERICAN MICROWAVE TECHNOLOGY INC.
an MMD company

3080 Enterprise Street, Suite A, Brea, CA 92621 PH (714) 993-0802 FAX (714) 993-1619

Model 3200 Series
6 - 220 MHz, pulsed, solid-state, RF power amplifier systems

Electrical specifications:	**Models:**	**3200**	**3205A**
Frequency range		6 - 220 MHz	6 - 220 MHz
Pulse power (min.) into 50 ohms		1000 W	300 W
CW power (max.) into 50 ohms		100 W	30 W
Linearity (±1dB to 200Mhz)		0-800 W	0-250 W
(to 220MHz)		0-600 W	0-200 W
Gain (typ.)		65 dB	60 dB
Gain flatness		±4 dB	±3 dB
Input/Output impedance		50 ohms	
Input VSWR		< 2:1	
Pulse width		20 ms	
Duty cycle		Up to 10%	
Amplitude rise/fall time		200 ns typ.	150 ns typ.
Amplitude droop		5% to 10 ms typ; 7% max	
Phase change/power output		10° to rated power, typ.	
Phase error overpulse		4° to 10 ms duration, typ.	
Noise figure		11 dB typ.	8 dB typ.
Output noise (blanked)		< 20 dB over thermal	
Blanking delay		< 2 μs on/off, TTL signal	
Protection		1. VSWR: infinite VSWR at rated power 2. Input overdrive: up to +10 dBm 3. Over duty cycle/pulse width 4. Over temperature	

Supplemental characteristics:

Connectors, rear panel	1. RF input: BNC (F) 2. RF output: Type N (F) 3. Noise blanking: BNC (F) 4. Interface: 25 pin D(F), EMI filtered
Indicators, front panel	1. Peak power meter 2. Over temperature 3. Over duty cycle/pulse width 5. CW Mode 6. Overdrive
System monitors	1. Thermal fault 2. DC power supply fault 3. Over duty cycle/pulse width 4. Forward/Reflected RF power
Front panel controls	1. AC power 2. Pulse width 3. Duty cycle
Cooling	Internal forced air
Operating temperature	+10 to 40°C
AC line voltage	120/240 VAC, ±10%, 50-60Hz (3200, 220/240V only)
AC power requirements	2000 watts / 700 watts
Package	Rack mount
Size (HWD, inches)	12.25x19x24 / 5.25x19x24
Net weight	165 lbs / 42 lbs

3080 Enterprise Street, Suite A • Brea, CA 92621 • (714) 993-0802 • Fax (714) 993-1619

DEPARTMENT OF HEALTH & HUMAN SERVICES

Public Health Service

Food and Drug Administration
Bethesda MD 20892

May 22, 1991
(received 5/28/91)

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

^{1}H-Detected C-C TOCSY Experiments: *Tempting the Probe Gods*

Dear Barry;

Over the last year, we have been employing isotope enrichment techniques and the associated NMR techniques to our ongoing studies in polysaccharide structure determinations (Byrd, Freese, & Vann, 32nd ENC, St. Louis, MO). One of the problems which is particularly troublesome in these systems, namely small $^{3}J_{HH}$ coupling constants by which H-H correlations may be generated, is efficiently overcome by making ^{13}C an abundant spin and using the techniques developed by Fesik & Zuiderweg and by Bax, Kay, Clore, Gronenborn and coworkers.

We have generated several isotopically enriched polysaccharide systems; however, for clarity, we would like to show here the efficient principle of generating H-H correlations via C-C and H-C coupling interactions with glucose. Figure 1 shows calculations, done in collaboration with Mark Rance of Scripps Institute, for the TOCSY transfer functions amongst ^{13}C spins in a uniformly labeled glucose sample. The pertinent $^{1}J_{CC}$ couplings were measured in a conventional 2D J spectrum, and the plot shows transfer from C1 to sequential C2, C3, C4, C5, and C6 as a function of idealized isotropic mixing time. The dashed vertical lines correspond to mixing times of 6 msec, 12 msec, and 24 msec. It is clear that at 6 msec, one would expect transfer to both the 2 and 3 positions, while at 12 msec and 24 msec there should be transfers to C5 and C6 respectively. Experimental verification of this is provided in Figure 2, which shows f2 traces through the α-anomeric proton for the corresponding mixing times of Figure 1.

This experiment was performed using a 2D sequence in order to demonstrate the mixing. The actual sequence consists of a ^{1}H-^{13}C coherence transfer (INEPT) step with frequency encoding of the ^{1}H shift in t_1 followed by ^{13}C-^{13}C TOCSY mixing via a DIPSI-2 sequence and finally a coherence transfer back to ^{1}H via an INEPT process for acquisition (Bax, *et al.*, JMR **88** 425-431, 1990). The real benefit, and application in our lab, comes from utilizing the experiment as a 3D sequence where the resultant spectrum contains ^{1}H-^{1}H "TOCSY" planes separated by the ^{13}C chemical shift of the site where the first coherence transfer occurred.

This data was collected on a GN300 spectrometer using a home-built X-decoupler channel, ENI 550L amplifier (γB_2 was 5 kHz), and a Cryomagnet inverse probe. Our 3D studies using these sequences have been performed on a JEOL GSX-500 with basically standard inverse probe and components except for the use of the ENI 550L amplifier for the ^{13}C channel (γB_2 was 7.5 kHz). These experiments really do *tempt the probe gods*, since most manufacturers never intended to see spin lock RF pulses of up to 30 msec on the X-channels of liquid probes....but just be careful!

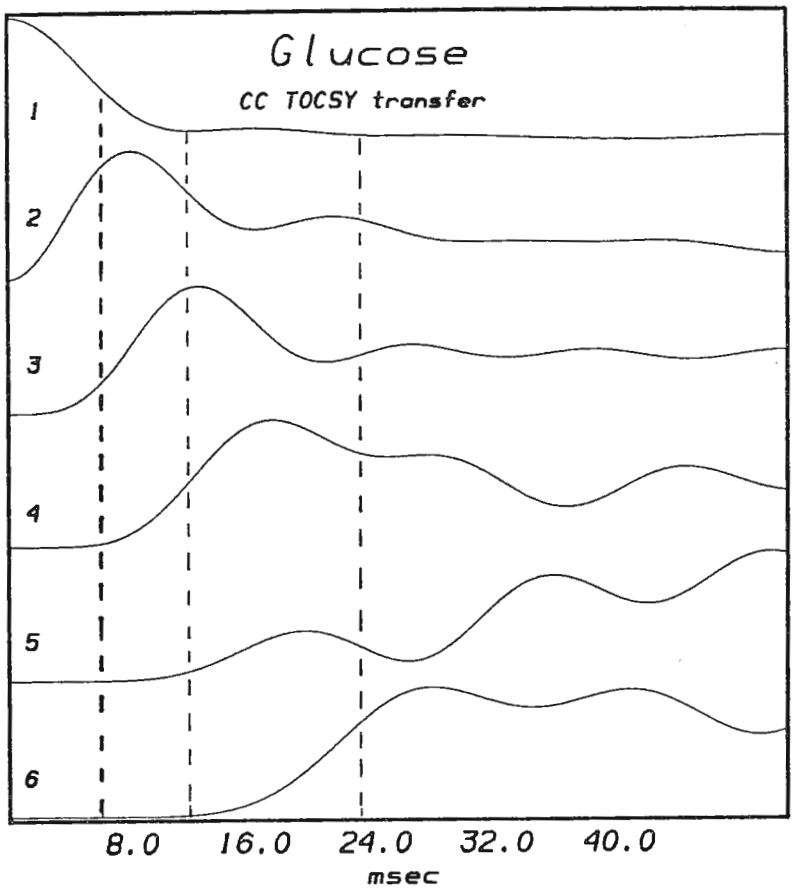


Figure 1: TOCSY transfer functions for Glucose. The calculation ignores relaxation and utilizes measured $^1J_{CC}$ values.

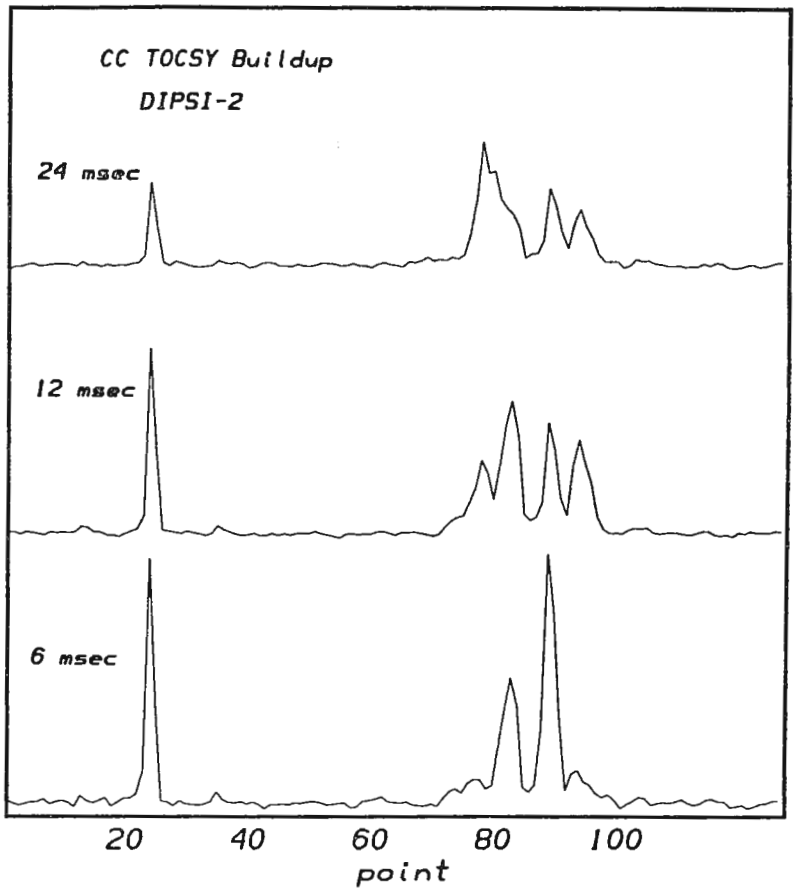
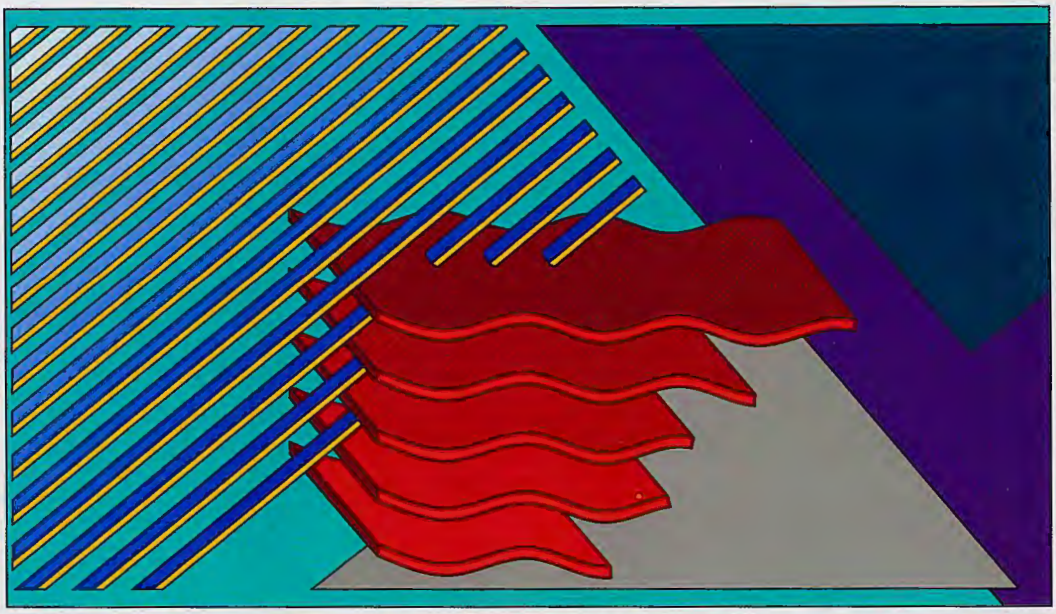


Figure 2. Traces through a 2D HCCH TOCSY experiment. Note the agreement with intensities both of the origin site and the sites of transfer.

Best regards;

R. Andrew Byrd
Biophysics Lab/FDA
8800 Rockville Pike
Bethesda, MD 20892

What's the difference between the new CMX spectrometer and the competition?



We took the complicated and made it simple.

Computer System
- Choice of SUN or Silicon Graphics computer
- X-Windows™ software **on** the spectrometer
- High-speed communications for "real time" display

Advanced Console Design
- Full *channel modularity*
- Full channel expandability
- Easy customer access

RF Design
- High IF design provides clean RF
- Ultra fast amplitude, phase, and frequency control
- Direct digital synthesis

Digital Design
- High-speed 100 MHz pulse programmer on **each** channel
- Complete pulse program flexibility
- Multiple expansion capability

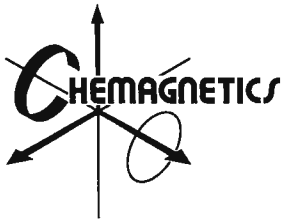
Solids Probes System
- PENCIL™ spinning system for high stability MAS
- Problem free spinning from – 150°C to + 250°C
- COAX design affords superior tuning and RF capability for multiple resonance experiments
- High-stability, microprocessor-controlled, speed controller

Ergonomics
- Superior comfort, functionality and increased work space for optimum productivity

Chemagnetics

2555 Midpoint Drive
Fort Collins, Colorado 80525
(303) 484-0428
1 (800) 4 OTSUKA

Dedicated to Design Excellence



Dear NMR Colleagues:

I'd like to invite you to an NMR workshop that you just shouldn't miss--especially if you're attending the Rocky Mountain Conference. Each year following the conference in Denver, Chemagnetics invites NMR scholars to give seminars on advanced solids topics and organizes a 2-day opportunity for NMR scholars to visit and get up-to-date on the latest in NMR. This year's workshop will be held August 2 & 3 at the new Chemagnetics facility in Ft. Collins, Colorado.

We are privileged to have luminaries from both academia and industry. Below is a list of seminars that will be presented on Friday. Saturday will feature hands-on demonstrations of advanced solids NMR techniques. Registrants may attend one or both days.

Those presenting Friday and their topics are:

- **Dr. Gary Drobney,** *University of Washington* — "Solids NMR Study of DNA Structures."
- **Dr. Bob Griffin,** *MIT* — "Distance Measurements with MAS."
- **Dr. Mark Conradi,** *Washington University* — "High Temperature NMR, Technique & Application to the Anomaly in Metal Hydrides."
- **Dr. Bernie Gerstein,** *Iowa State University* — "Technology and Gimmicks for Low Temperature CPMAS Without Tears."
- **Dr. Naresh Sethi ,** *AMOCO Corporation* — "New Methods to Simulate NMR Powder Spectra."
- **Dr. Jim Haw,** *Texas A&M* — "Isotropic Reconstruction: The Second Chapter."
- **Dr. Yue Wu ,** *U. of California, Berkeley* — "DOR, Technique and Applications."
- **Dr. Dan Weitekamp ,** *California Institute of Technology,* — "MRI of Solids with RF Field Gradients and Multiple-Pulse Line Narrowing."
- **Dr. Chuck Bronniman ,** *Chemagnetics* — "Solid-State Imaging: Review and Prospects."

If you'd like to get up-to-date on the latest in solids NMR, I urge you to register today. Registrations are limited to 60 people on the first day and 15 for day two. There is no fee to attend the seminar, but registrants are responsible for their own transportation from Denver to Ft. Collins (approx. $13), for the hotel, and meals. To help defray your costs, rooms at Chemagnetics' discount rate of $33.50 single/$37.50 double have been booked at the nearby Ramada Inn. Please call today to register at 1-800-4-OTSUKA and ask for Sheila or Patti.

I look forward to seeing you.

Robert Wind

Dr. Robert Wind
Director of Research & Development

2555 Midpoint Drive Fort Collins, Colorado 80525 (303) 484-0428 FAX (303) 484-0487 1-800-4-OTSUKA

A member of the Otsuka family of companies

UNIVERSITA' DEGLI STUDI DI MILANO

Dipartimento di Scienze Molecolari Agroalimentari

DISMA Via Celoria, 2 I-20133 Milano Tel. 02-2663662/2365029/2362721

May 31, 1991
(received 6/21/91)

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

IN VIVO DETERMINATION OF INTRACELLULAR pH AND OTHER PHYSIOLOGICAL PARAMETERS BY ^{31}P NMR SPECTROSCOPY, IN THE STUDY OF PLANT RESPONSE TO ANOXIA.
POSITION WANTED FOR A RUSSIAN COLLEAGUE.

Dear Barry,

as probably I have been already dropped from your mailing list, I apologize for the trouble that my delay induces. It is a pleasure for me to send you a note from my young colleague, Dr. Enzio Ragg, as a contribution of our laboratory. At the same time, I would ask you a favor for a Russian colleague, who wants to find a position in a western country, to work in NMR field. He has 25 years experience in NMR application to inorganic, analytical, organometallic chemistry and asked me not to mention at present his name. If somebody of the NMR Newsletter readers is interested, would he please contact me.

Thank you very much, with kindest regards

Rosanna Mondelli

In vivo ^{31}P NMR spectroscopy was used to study plant response to O_2 deficit, comparing the highly tolerant rice seedlings with the intolerant wheat seedlings. NMR spectroscopy indicated a strong acidification of wheat cytoplasm (pH = -0.8 pH units) and a milder one in rice cytoplasm (pH= -0.4 pH units) in agreement with the higher tolerance to anoxia of rice. The previously reported (MENEGUS F. et al., Plant Physiol. 90,29,1989) cell sap alkalinization during anoxia in rice, as compared to acidification in wheat, was reflected in the alkalinization of vacuolar content in rice but not in wheat. The requirement of cytoplasm acidification, as predicted by the widely accepted model of plant resistance to anoxia (ROBERTS JKM et al., Proc. Natl. Acad. Sci. USA 81, 3379,1984) is therefore satisfied also in rice. Details of the above results are reported elsewhere.

Here we focus on some other events, concomitant with the changes of intracellular pH, that have received less attention. Oxygen deficit reduces ATP level in rice tissues to 50% of that found in air (Fig. 1). In wheat tissues ATP signals practically disappear under anoxia. The same is true for UDPG level which is hardly affected in rice, whereas it is strongly depressed in wheat. This last finding seems to be connected to the ability of rice coleoptile, but not of wheat, to elongate during anoxia.

Concomitant with the decrease of ATP and other phosphorylated compounds, there is an increase in cytoplasmic content of the inorganic phosphate (Pc). Cytoplasmic acidification and cytoplasmic phosphate build-up are simultaneous events.

UNIVERSITA' DI MILANO
Dipartimento di Scienze Molecolari
Agroalimentari

The increase of cytoplasmic phosphate content may be relevant in determing plant resistance to anoxia, because it negatively affects the phosphorylation potential: $P = [ATP]/[ADP][Pc]$.

During anoxia, therefore, P will be lower in wheat than in rice, not only because ATP level is lower, but also because the inorganic phosphate level is higher. Changes of ADP level induced by anoxia in the two species (^{31}P NMR spectra of $HClO_4$ tissue extracts) are not much different, so their influence on P is less important.

The qualitative changes of the phosphorylation potential, outlined here from the in vivo NMR measurements, help to explain the higher tolerance of rice with respect to wheat to anoxia.

Very sincerely yours

Faustino Menegus
Dr. F. Menegus

Ezio Ragg
Dr. E. Ragg

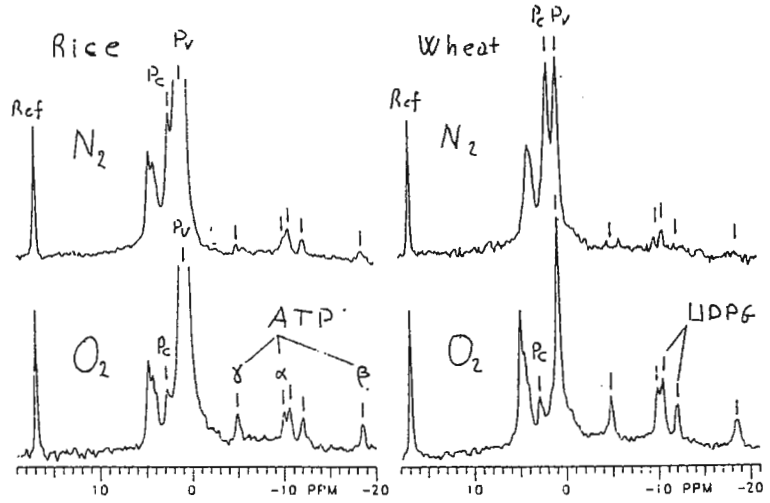


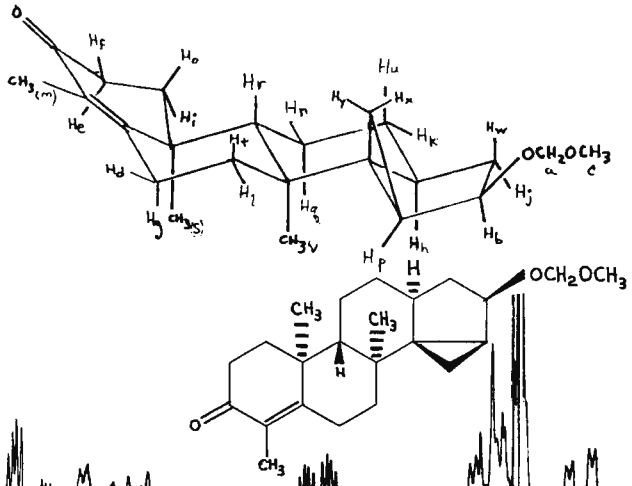
Fig. 1 - In vivo ^{31}P NMR spectra (121.4 MHz) of aerobic (O_2) and anoxic (N_2) rice and wheat tissues. Pc and Pv refer to the signals of inorganic phosphate pools of cytoplasm and vacuole respectively.

Why compromise in quality and even pay higher prices?
... for NMR tubes specify Norell -- accept no substitutes
We have the lowest prices in the industry!

BRUKER

```
JAB132.001
DATE 30-11-88

SF      500.135
SY       83.0
O1     7900.000
SI      32768
TD      32768
SW     5000.000
HZ/PT      .305

PW         5.0
RD         0.0
AQ       3.277
RG          32
NS          72
TE         297

FW        6300
O2     7925.000
DP      10H PO

LB         0.0
GB         0.0
CX       36.00
CY         0.0
F1       2.700P
F2         .401P
HZ/CM    31.950
PPM/CM     .064
SR     5426.67
```

I have evaluated Norell's XR-55 Precision NMR Sample Tubes and found them to be:
☒ Excellent ☐ Very Good ☐ Satisfactory ☐ Unsatisfactory
Norell's XR-55 NMR Sample Tubes were compared with the following competitive tube or tubes: WILMAD 528-PP
(give catg. no. and name of manufacturer)
Type of NMR equipment used in the above evaluation: BRUKER AM-500
Date Nov 30, 1988 Name Scott VIRGIL Title GRADUATE STUDENT
Address 12 OXFORD STREET # 302
CAMBRIDGE, MA 02138
Telephone: (617) 495-4034
We expect your findings from the supplied batch of Norell's tubes to be unbiased and we reserve the right to share your findings with others inquiring about our NMR sample tubes. Norell, Inc., 314 Arbor Ave., Landisville, NJ 08326 USA.



Comparison and evaluation carried out at Harvard University by Scott Virgil on a Bruker AM-500 NMR unit, on November 30, 1988.

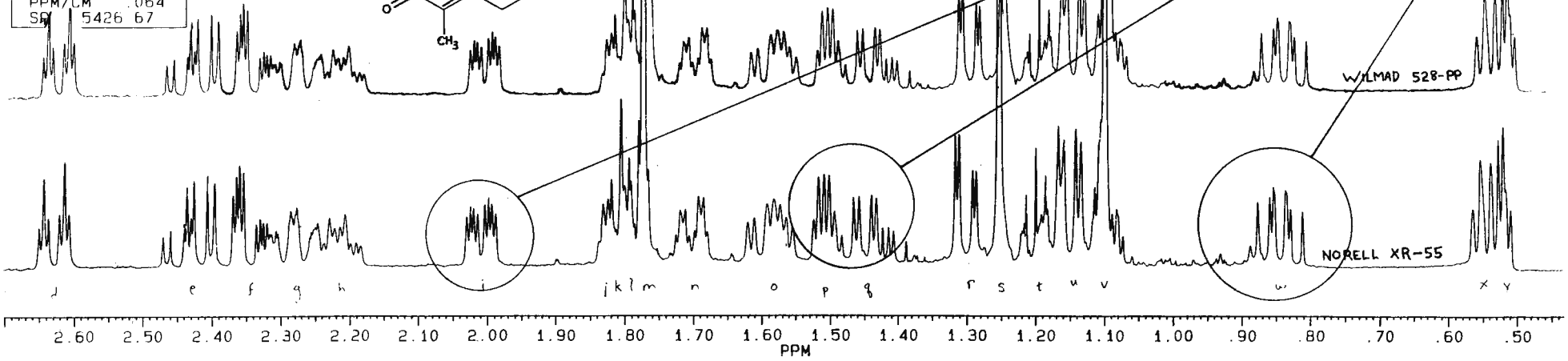
Look at the quality of resolution - as evidenced by the DEPTH of VALLEYS between very close peaks. You can also see that the XR-55 tube yields sharper & clearer signals. This is primarily due to its SUPERIOR CONCENTRICITY. Not only that you get BETTER RESOLUTION with our NMR tubes - you pay a substantially LOWER PRICE!!!

Please Note:

... our prices did not change since 1980!

... our QUALITY OUTSTRIPPED the Competition!

Norell's XR-55 Tube



NORELL, INC., 22 Marlin Lane, Mays Landing, NJ 08330 USA Tel: (609)625-2223; (800)222-0036; Fax: (609)625-0526

PRECISION NMR SAMPLE TUBES

CENTRE FOR NUCLEAR MAGNETIC RESONANCE

Department of Chemistry
Tel: 523187
DR. OLIVER W. HOWARTH

University of Warwick
Coventry CV4 7AL

Department of Physics
Tel: 523523 Ext. 2403
DR. RAY DUPREE

28th May 1991
(received 6/3/91)

SLOW PROTONATION IN ISOPOLYTUNGSTATES

Dear Barry,

We have been having a lot of fun recently trying to sort out the aqueous chemistry of tungsten (VI) using ^{183}W and ^{17}O NMR. It is proving possible to recognise the relationships between many anions which have up till now been either unknown, or only known through one-off methods of preparation. The figures below show two of the better known isopolytungstate anions. Both of them normally hold two protons in their central cavity, in order to achieve a favourable distribution of charge. We have shown that metatungstate can be made with just one internal proton. It pulls in a second one within about one day, in aqueous solution. The H/D exchange rate in the diprotonated species is similar.

The cavity in paratungstate B is less obviously closed off to the solvent, and sure enough one cannot now observe any separate resonance from the internal protons. However, we were initially surprised to observe significant line-broadening in the tungsten resonances. The broadening increases from nil to a maximum as the anion protonates. This was a surprise to us, because the tungsten-oxygen framework shows every sign of being stable on this timescale. We therefore tried moving from H_2O to D_2O solvent, and sure enough, all the linewidths more than doubled. It is not easy to get accurate linewidths in this system because the protonated paratungstate B anion undergoes further chemical changes, over a day or so, which alter the pH. (It goes to a new 11-tungsten species, but that's another story). Nevertheless, we are convinced that the widths shown in the Table below are reasonably reliable. As the broadening only occurs in the (tri)protonated anion, we reckon that it arises from protonation isomerism between a species with two internal and one external protons and one with three internal protons. Our oxygen data support this.

Although the cavity in paratungstate B is plenty large enough for protons, it is too small for any water molecule. Evidently protons do not move all that rapidly on an oxide-type surface without assistance from the solvent.

Table. Linewidth variations in acidified paratungstate B

Peak label	^2H_2O, 283K	^1H_2O, 283K	^1H_2O, 283K	^1H_2O, 268K	$^1H/^2H$
	pH=2.8[a]	pH=3.1	pH=4.2[b]	pH=3.1	pH ≈ 3
I	7.6[c]	3.4	3.0	6.0	2.8[d]
II	15.2	7.2	3.9	16.2	2.3
III	16.2	6.8	3.2	11.5	2.6
IV	5.2	3.0	2.3	4.6	2.8

[a] Almost fully protonated (i.e. H_3) [b] Just over half-protonated (i.e. $H_{2.6}$)

[c] Width at half height in Hz, after removal of broadening due to window function

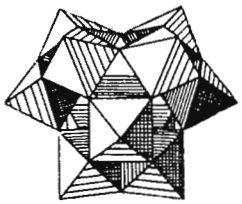
[d] Natural linewidth = 1Hz, as observed in the unacidified anion (i.e. H_2)

With best wishes

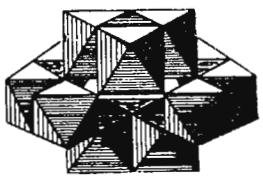
Oliver Jeremy.

Oliver Howarth and Jeremy Hastings

metatungstate, $[H_2W_{12}O_{40}]^{6-}$



paratungstate B, $[H_2W_{12}O_{42}]^{10-}$



394-34

MASSACHUSETTS GENERAL HOSPITAL ◆ HARVARD MEDICAL SCHOOL

MGH-NMR CENTER
NMR RESEARCH LABORATORIES
MGH IMAGING CENTER
MR EDUCATION CENTER

Bldg. 149, 13th Street
Charlestown, MA 02129
Phone: (617) 726-
Fax: (617) 726-5819

Phone: 617-726-3083
Internet: jerry@nmr-r.mgh.harvard.edu

June 18, 1991
(received 6/20/91)

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

Modifying Standard Bruker CPMAS Probes for ^{19}F

Dear Barry:

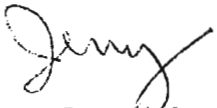
We have two standard Bruker 7 mm CPMAS probes for our MSL400. In both of these probes, the high frequency channel is tuned for protons. We recently had occasion to measure some ^{19}F MAS spectra, and were dismayed to find that the proton channel of neither probe could be tuned to the fluorine frequency, only 6 percent away. Bruker quickly confirmed that, indeed, they themselves had tried it and failed, and that our only recourse was to purchase a probe built for ^{19}F.

Owing to my inherently cheap nature and to our desire to get the experiment done that same day, we decided to take a closer look at the Bruker tuning circuitry.

The high frequency channel of these probes employs a resonant line or coupling loop, the length of which is adjusted by the ^{1}H tune knob at the probe base. Although this adjustment reaches its low frequency stop well before the vicinity of the ^{19}F frequency, it turns out to be a simple matter to loosen the clamp which supports the moving part of the line and allows it to slide with respect to a stationary member. By pushing the clamp away from the probe head by about 0.5 cm and tightening it again (it's got to remain loose enough to allow sliding) it becomes possible to reach the ^{19}F resonance. The matching capacitor has the range to match perfectly at the new frequency. The limit stop on the tuning rod at the probe base may be backed up to a new position suitable for the full ^{1}H to ^{19}F range.

Placing marks on the resonant line to indicate the clamp positions for each frequency allows the nucleus switching operation to be completed in under five minutes. The more ambitious among your readers might consider machining a new clamp with a longer slot so that the retuning is possible without removing the probe cover.

Best regards,



Jerry Ackerman

Fremont Magnetic Resonance
3315 Seldon Court
Fremont, CA 94539
415/623-0722
FAX 415/623-0851

File Transfers via Ethernet

GOSSIP provides transparent file transfers between a Nicolet 1280 and any Ethernet system supporting the tcp/ip protocol. You can configure your 1280 to upload every file as it is aquired, or you can manually select the files to upload in background.

At 19.2 KBaud, file transfers occur at about 21 K words/min. So a typical 512 x 4K 2D file can be transferred in about 100 minutes. Since the transfer occurs in parallel with the data aquisition, if the experiment takes more than 100 minutes, **data transfer takes practically no additional time**, just the time required to transfer the last block. Multi-block files larger than the available disk space on the host spectrometer can also be created.

GOSSIP version 1.12 offers automatic configuration and file conversion (conversion requires purchase of the appropriate data translation software from FMR). Configuration is achieved via a downloaded text file, therefore no user input at the PC is required. A keyboard and screen at the PC might be desired for greater flexibility.

Template macros for the Nicolet 1280 are provided that demonstrate : 1) data aquisition, then simultaneous data processing and file transfer, and 2) 2D data aquisition with concurrent file transfer.

Features

- No-hassle installation
- Automatic, customizable operation
- 20K data words/minute transfers
- Background transfers for 1D data
- Concurrrent background transfers for 2D or kinetics data
- 2D files can be larger than the spectrometer disk space

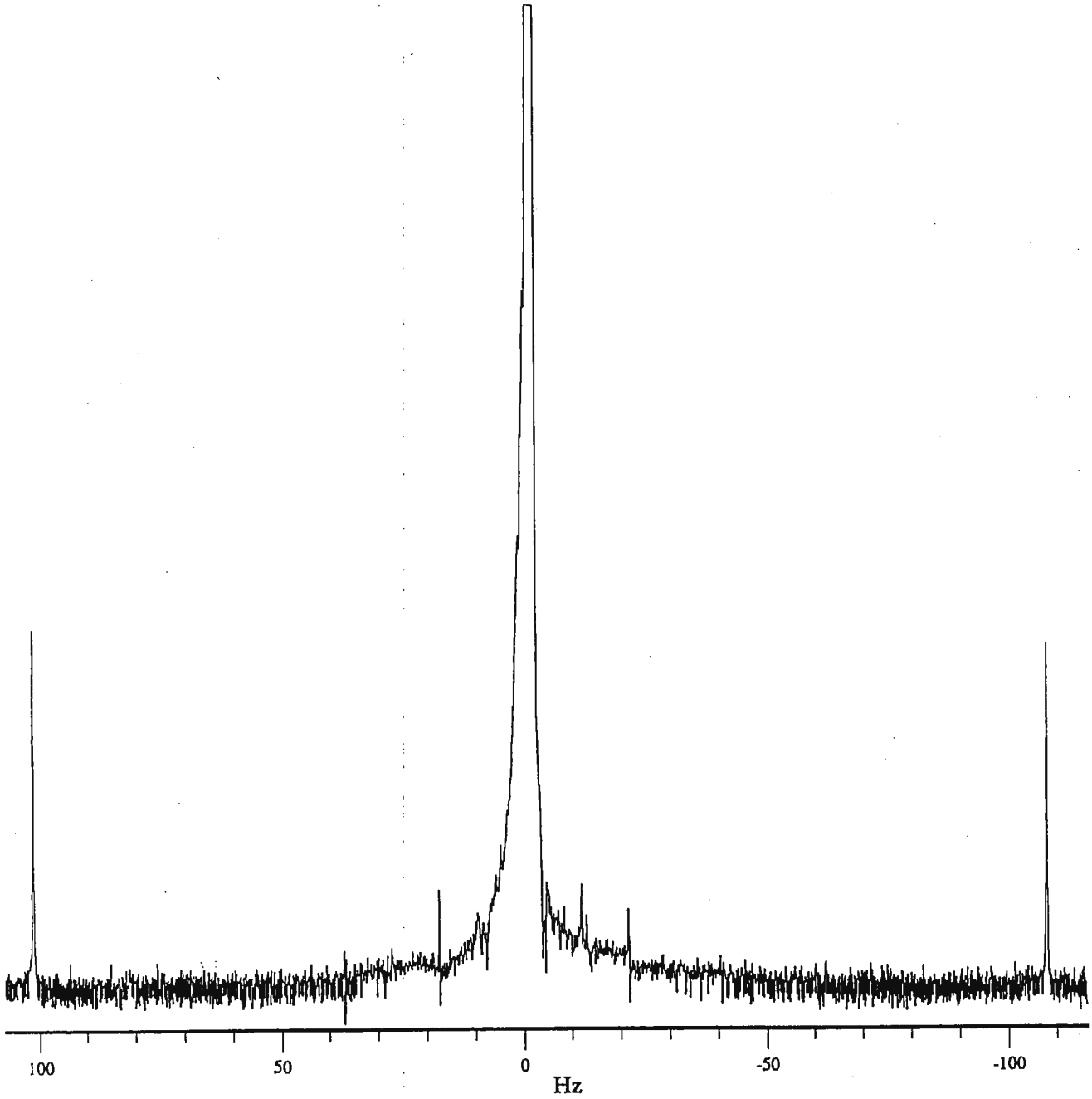
FMR offers flexible packaging of GOSSIP to meet your requirements. You may purchase a turnkey system, or aquire the parts independently and buy only the software from FMR. Since there need be no user interaction at the PC, you may choose a 'black box' PC, without keyboard or moniter.

GOSSIP is very reasonably priced, and is available from stock. It can be a very cost-effective way to add your older spectrometers to the lab network. Why not order a copy and see for yourself?

Fremont Magnetic Resonance
3315 Seldon Court
Fremont, CA 94539
415/623-0722
FAX 415/623-0851

Probes and Probe Upgrades

FMR offers probes for most systems. Upgrading an old, unused probe to a modern technology reverse detect or 1H can bring new usefulness to older systems.



500 MHz $CHCl_3$

The University of Texas Medical Branch at Galveston

School of Medicine
Graduate School of Biomedical Sciences
School of Allied Health Sciences
School of Nursing

Marine Biomedical Institute
Institute for the Medical Humanities
UTMB Hospitals

Marine Biomedical Institute

(received 6/24/91)

Barry Shapiro, Ph.D.
TAMU NMR NEWSLETTER
966 Elsimore Ct.
Palo Alto, CA 94303

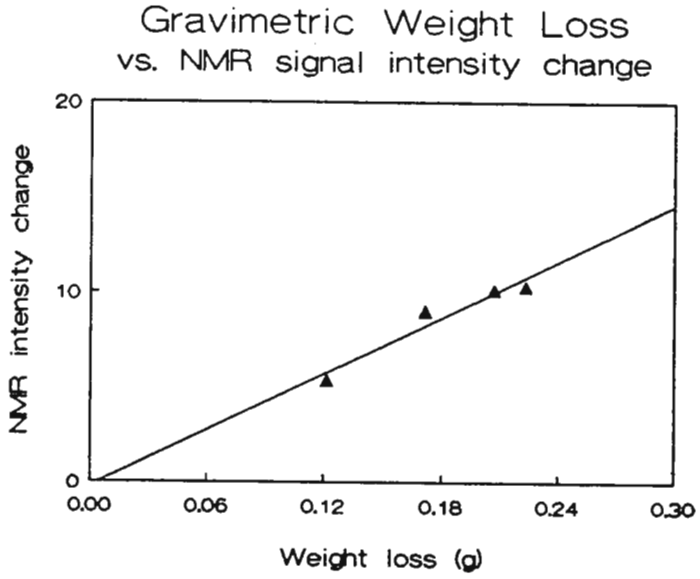
Measurement of Water Content in Bone Graft Samples

Hey Barry,

The tissue bank at Shrine Burns Institute here on our campus asked us to assess moisture content in bone samples using NMR spectroscopy. According to their technical manual[1], NMR is a reliable way to quantify the percentage water content. In order for their samples to be useful they must contain less than 5% water by weight. Their air drying procedure is carried out at room temperature according to standard procedures. To perform quality control of moisture content we have used the following procedure.

In order to calibrate the NMR method, we compared proton NMR signal intensity change to gravimetric weight loss in sections of fibulas weighing between 1 and 3 grams. Our gravimetric water determination was done by heating the sample in a vacuum dessicator containing DRIERITE ($CaSO_4$) at 65 degrees C until a constant weight was achieved (about 3 days). Other labs have successfully used higher temperatures for drying bone samples[2]. The NMR measurement was made by 12 signal averages using a 90 degree pulse and a 10 second interpulse delay. Samples were run on a horizontal bore system operating at 200 MHz. The broad-line background signal originating from protons in the plexiglass coil body was subtracted out of each spectrum. Bone water T_1 relaxation times,measured by inversion recovery, were around 1.5 seconds. Line widths ranged from 500 to 1800 Hz, and were found to be inversly related to water content. As shown in the graph, weight loss in bone samples varied linearly with change in NMR signal intensity. The constant relating NMR intensity change and water content was 0.02094 +/- 0.00157 (mean +/- SD). This corresponds to a 7.5% uncertainty in the NMR measurement vs. gravimetric. On the initial samples we tested, the average water content was 6.83% +/- 0.566, indicating that their standard drying procedure did not produce a useful sample for bone grafting.The simple expedient of using pre-dried air in the drying procedure instead of ambient air (typically 90% humidity) is expected to achieve optimal water content.

Please credit this to L.L. Smith's account.



References

1. American Association of Tissue Banks Technical Manual, Section 2, B2.155.

2. Joe Harris, AO Labs & Research, Inc., Indianapolis, IN, Personal Communication.

Yours in the double bond,

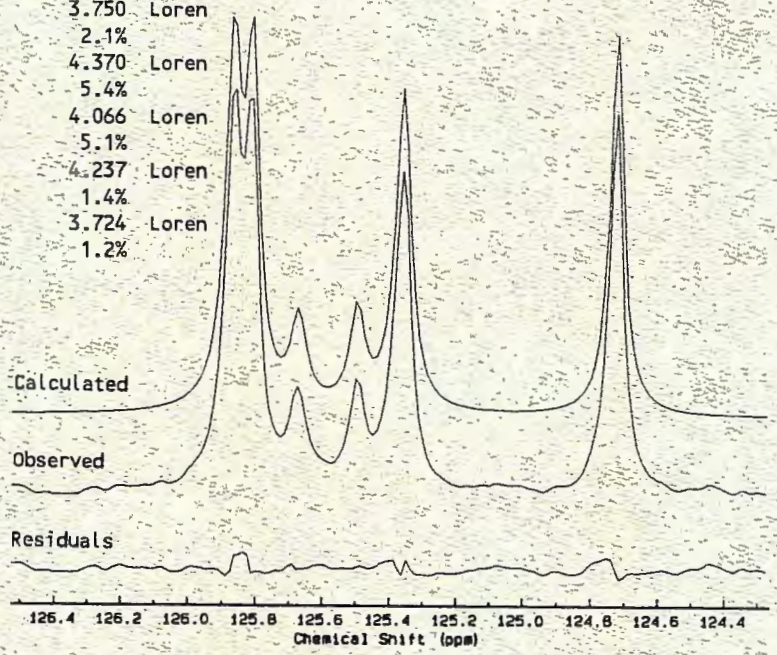
Mike Quast Ed Ezell Jose Gonzales Stacy Klinke

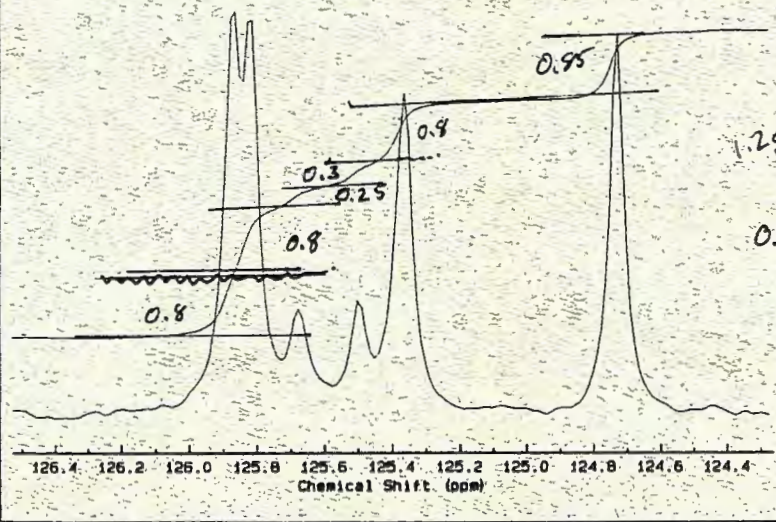
How does your present NMR software let you measure integrals?

Fitted Peaks from file: tmsa100.001
Chi Square = 183.800034 180 Points

Peak	Hz	ppm	Intensity	Area	Width	Shape
1	9499.27	125.878	0.1429	0.8915	3.972	Loren
;+/-	4.9E-0002	6.5E-0004	1.0%	2.9%	1.9%	
2	9494.95	125.821	0.1370	0.8070	3.750	Loren
;+/-	4.8E-0002	6.3E-0004	1.1%	3.2%	2.1%	
3	9484.09	125.677	3.485E-0002	0.2392	4.370	Loren
;+/-	1.5E-0001	2.0E-0003	3.5%	8.9%	5.4%	
4	9470.73	125.500	3.833E-0002	0.2448	4.066	Loren
;+/-	1.3E-0001	1.8E-0003	3.3%	8.4%	5.1%	
5	9460.49	125.364	0.1297	0.8634	4.237	Loren
;+/-	4.0E-0002	5.3E-0004	9.4E-0001%	2.4%	1.4%	
6	9411.88	124.720	0.1544	0.9033	3.724	Loren
;+/-	3.1E-0002	4.2E-0004	8.4E-0001%	2.0%	1.2%	

Like this?





Or like this?

1.2g = 10cm

0.95/1.2 = 0.71 } some?

0.8/1.2 = 0.67

cut & weigh!

0.3/1.2 = 0.25 } ?

0.25/1.2 = 0.21

Quantitative NMR with **NMR-286**

NMR is inherently quantitative but our software usually leaves us guessing at the peak areas. NMR-286 uses an interactive Marquardt driven peak fitting routine to help you get the most out of your spectra. NMR-286 is a ***must*** for researchers doing serious quantification of their spectra. Look at these features:

- Parameters can be frozen at will to incorporate prior knowledge of the spectrum.
- The output contains the best-fit parameters as well as their standard deviations and the full variance-covariance matrix.
- You can compare the calculated and observed spectra and see the residuals in Dual Display.
- Clusters of up to 30 overlapping peaks can be pulled apart, even if the component peaks appear only as shoulders.

NMR-286 is not just a line fitting program.

It is a *powerful* and *complete* PC based NMR processing package for one dimensional NMR. Here are some of its features:

- Lightning Fast FTs — the fastest that you will find on a PC. Sizes up to 32k supported.
- Window Functions. Select from eight different window functions.
- Plotting. Plotting is extremely flexible. Send the HPGL output to a plotter, a file or a laser printer.
- Automated Processing. Almost anything that you can do from the keyboard, you can write a program to do. A powerful editor is a part of NMR-286.
- Manual. A comprehensive reference manual comes with NMR-286. It explains not only the commands used but also the ideas behind them.
- Public File Formats. NMR-286 uses a file formats which are well described in the manual. You can import and export spectra as ASCII text files.
- **And much much more!**

Computer Requirements

A Personal Computer which conforms entirely to the IBM standard. CPU: 80286/287 or better. OS: MS-DOS 3.0 or greater. Memory: 640k required. Graphics: EGA, VGA, Hercules or AT&T400.

Spectrometers

NMR-286 is in itself independent of the spectrometer. It requires a translation program to convert the data files to NMR-286 format. Currently such programs exist for Bruker DISNMR, DISCXP, DISMSL, TOMIKON and UXNMR files as well as for Lybrics format files. Have another type of machine? Call!

Upgrade to 3.2

NMR-286 V3.1 users can receive an upgrade to Release 2 for a shipping and handling fee of $50.00. Contact SoftPulse for more information.

Software Support

Each purchaser will receive a year of free software support by facsimile.

NMR-286 Prices

Copies	Price per copy*
1	1500.00
2-5	1250.00
>5	1100.00

*All prices are in Canadian dollars. Canadian orders add 7% GST, Ontario orders add 8% PST. Academic institutions are eligible for a 33% discount on software. Purchase orders accepted. Shipping per order: Canada – $15.00, elsewhere $30.00. Foreign orders may be paid in the equivalent of U.S. dollars.

NMR-286 will help you use your valuable magnet time for acquiring data. You can process, examine, measure and plot your spectra when and where you want — in the lab, in your office or at home. Try it, you'll like it. You can even try it ***Risk Free***! Buy NMR-286 and if within 30 days you decide that NMR-286 is not for you, your purchase price will be refunded. Questions? Call, fax or write.

SoftPulse Software

NMR Processing Software for the Personal Computer

Box 504, Guelph Ontario N1H 6K9 Canada *Telephone (519)837-0276 Facsimile (519)821-8955*

Acorn NMR
46560 Fremont Blvd., #418
Fremont CA 94538-6482
Telephone: (415) 683-8595
FAX: (415) 683-6784

June 22, 1991

Dear Barry,

We have just established Acorn NMR as a spectroscopy service. As part of our preparations, we acquired a 10 year old NT360-WB. Shipping an old instrument across the country and getting it running is always fun. One nice feature of the NT console is the extensive operator feedback provided by all the lights and switches. These make debugging consoles much easier. The instrument is now up and running and we are in business. In the process of setting up we discovered a few modifications which have helped to make this older instrument more versatile. In the belief that they are of general utility, we would like to share some of them with the TAMU Newsletter readers.

To get rid of the old "clunky" TTY-43 keyboard, we substituted a 386 PC running the terminal emulator program within Windows 3.0. This configuration adds several new features to spectrometer operation. One very convenient feature is the ability to define a single mouse-activated ''button'' to execute a series of frequently used commands. This supplements the NT software limitation of having only one "link" list. For example, this gives us single button activation of various auto-shim sequences, another button for the ever useful ZGBCEMFTPS command string, and several others which are always available with a mouse click. The terminal emulation program also provides the ability to capture text output by the 1280, such as parameter lists, peak lists and pulse programs, into files on the PC.

Another useful feature of this configuration is that Windows lets you have multiple programs active simultaneously. It also permits rapid toggling between them which eliminates delays for each program to be started. Using this feature allows several programs to be simultaneously running in separate windows. A common configuration for us is 1) the terminal emulator for controlling the NT console, 2) GOSSIP for serial data transfers to and from the PC and 1280 at Baud rates up to 38,400, and 3) Felix/PC for data processing. All programs are available for immediate use.

Once data has been transferred to the NMR's PC, it is immediately accessible from one of several PCs at anyone's desk in the facility via a Local Area Network. Plots are output from Felix to our HP Series II laser printer, equipped with a Pacific Data Products ''Plotter in a Cartridge'' for HPGL emulation, obviating the need to purchase a dedicated plotter. Plots can also be output to a file in HPGL format, which can be imported into a variety of desktop publishing programs for customizing or incorporating into reports. For example, the sucrose plot included in this communication was imported into PageMaker. Using this capability in conjunction with the "captured" peak lists and other information from the 1280 allows us to prepare complete reports for our customers in both magnetic and hard copy form. They can then reprocess it later at their facilities. We can even E-mail the data and report for faster turn-around.

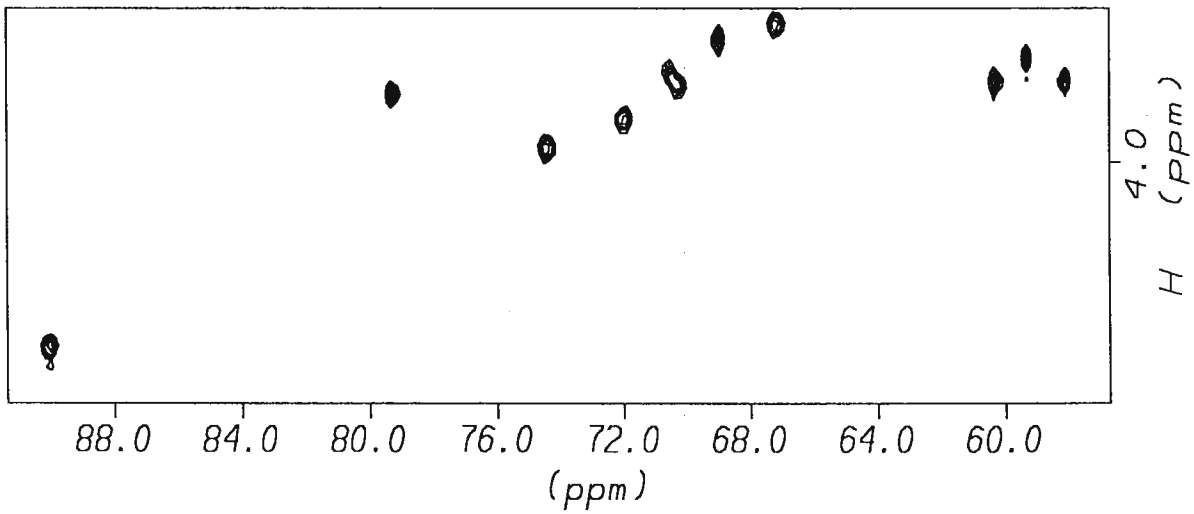
As we began working with 2D data, the advantages of Felix became more apparent. The 10 year old NT software is limited in its capability for user-interactive viewing and manipulation of 2D data. 2D FTs are also faster on our PCs: the sucrose spectrum shown (1K x 64) took 45 seconds for complete processing on a 486. Readers may also benefit from our experience with Felix 2D pro-

cessing, as it took a bit of trial-and-error to determine the correct combination of commands. An essential point is understanding how the data is actually stored in the computer. Complex NMR data is stored by different computers and software in different ways. Nicolet software stores complex data in two parts. The first half of the file contains the real half of the complex data set and the second half of the file contains the imaginary half. On the other hand, Felix stores the data intermingled, as alternating real and imaginary points. Listed below is a macro for transforming a magnitude (not phase sensitive) data set. The ''trick'' is in keeping the matrix type as real, even though we keep the imaginary part of the data until the last step of processing. The real and imaginary data are written to the matrix after the first FT as separate real and imaginary halves, with odd numbered rows being real and even numbered rows being the corresponding imaginary half. (This necessitates ''lying'' to Felix about the size of the data set and its real/complex nature, which can make the process rather confusing.) When a column is loaded for the 2nd dimension FT, it consists of alternating real and imaginary points, as Felix expects, and a complex FT is performed. (Thanks to Dennis Hare for explaining this.)

Gina Miner

Woody Conover

The plot below is the result of a 1K x 64 C-H correlation experiment on sucrose processed in Felix/PC with the mag2d.mac macro described.



Macro for 2D data processing in Felix/PC.

"mag2d.mac"

```
cl                                          ;close file & reset pointer
get 'Enter data file name:' name            ;time-domain data file
get 'No. of t1 slices?' nrow                ;number of spectra acquired
get 'Size of each?' ncol                    ;size (in COMPLEX points)
get '1st dimension Line broadening:' linbr1
get '2nd dimension Line broadening:' linbr2
get 'Enter name for matrix file:' mname     ;name for matrix file
eva nrowx2 (2*&nrow)
eva ncolx2 (&ncol*2)
bld &mname 2 &ncol &nrowx2 0                ;build matrix (real)
mat &mname write                            ;open matrix, write-enabled
def rrow 1                                  ;counter for matrix writing
for row 1 &nrow                             ;loop for 1st dimension FT
re &name
dat 1                                       ;data type = complex
em &linbr1
ft
sep                                         ;separate real/imag parts to
                                            ; left/right sides
dat 0                                       ;data type = real
si &ncol                                    ;size
sto 0 &rrow                                 ;write real half to matrix
eva rrow (&rrow+1)                          ;increment counter
si &ncolx2                                  ;double size
shl &ncol                                   ;get rid of 1st half
si &ncol                                    ;reset size
sto 0 &rrow                                 ;write imag. half to matrix
eva rrow (&rrow+1)                          ;increment counter
ty row=&row $                               ;type row number (no scroll)
next                                        ;end 1st loop

for col 1 &ncol                             ;loop for 2nd dimension FT
loa &col 0                                  ;load column from matrix
dat 1                                       ;data type = complex
si &nrow                                    ;size
zf &nrowx2                                  ;zero-fill 2x
em &linbr2
ft
ms                                          ;magnitude spectrum
rev                                         ;reverse spectrum
red                                         ;reduce to real
sto &col 0                                  ;write to matrix
ty col=&col $                               ;type column number
next                                        ;end 2nd loop
end                                         ;end of macro
```

Dalhousie University

Department of Chemistry
Halifax, Nova Scotia
Canada B3H 4J3
Tel: (902) 494-3305
Fax: (902) 494-1310

May 22, 1991
(received 5/30/91)

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

Dear Barry:

^{31}P nmr study of solid mercury phosphonates

We have recently examined the ^{31}P nmr spectra of several mercury phosphonates, $(EtO)_2P(O)HgX$, in the solid state. These compounds are characterized by very large $^1J(^{31}P,^{199}Hg)$ values ranging from 12.6 kHz (X=I) to 13.8 kHz (X=Cl) and values of $\delta_{11} - \delta_{33}$ which range from 135 ppm (X=CI) to 170 ppm (X=SCN). For some of the derivatives it was possible to analyze the MAS and static ^{31}P nmr powder line shapes to obtain estimates of the anisotropy in the $^{31}P,^{199}Hg$ spin-spin coupling constant. Values of $\Delta J(^{31}P,^{199}Hg)$ were 2700 Hz ($X=CH_3COO$), 1600 Hz (X=SCN) and 1500 Hz (X=I). The error in these values is estimated to be $\pm$ 250 Hz. Interestingly, the value of $\Delta J/J$ in the mercury phosphonates (0.1 to 0.2) is significantly less than the corresponding values in a series of mercury phosphines (0.4 to 0.7).

The most important conclusion from this work is that mechanisms other than the Fermi-contact mechanism make substantial contributions to $^1J(^{31}P,^{199}Hg)$, particularly in the mercury phosphines.[1] Further details will appear in the ACS publication, Inorganic Chemistry.

Best regards,

Yours sincerely,

Rod Wasylishen Bill Power Michael Lumsden

RW:jw

[1]G.H. Penner, W.P. Power, and R.E. Wasylishen, Can. J. Chem., 66, 1821 (1988).

P.S. We are preparing to write a review which will tentatively be titled, "Phosphorus NMR Studies of Solids." I would appreciate copies of any recent reprints and preprints that readers of the Newsletter would like us to include in the review. Thanks, Rod

Gradient Enhanced Spectroscopy SWAT

GE introduces the use of Switched Acquisition Time (SWAT) gradients to achieve pure phase 2D spectra with quadrature detection in both the acquisition (ω_2) and evolution (ω_1) dimensions without any phase cycling and without an additional set of t_1 data.

One example of a pure phase gradient enhanced COSY spectrum of a solution of 2,3-dibromopropionic acid in benzene-d6 is shown in Fig. 1. SWAT gradients and a single acquisition per block were used. Data was collected on an Omega 300WB with Microstar actively-shielded gradients. A 5mm inverse probe was built for use within the gradient coils.

Digital resolution of 1.2 Hz in ω_1 and 2.4 Hz in ω_2 was achieved by collection of a 512 x 512 matrix with t_1 evolution time of 840ms and a t_2 acquisition time of 420ms. A single acquisition per t_1 evolution data block and an average recycle time of 1.84s resulted in a 15 minute total collection period.

Since the SWAT gradient method encodes the necessary information in a single t_2 acquisition time, it avoids the collection of additional data blocks required by traditional pure phase methods. This time efficiency is especially important for collection of large multidimensional data sets.

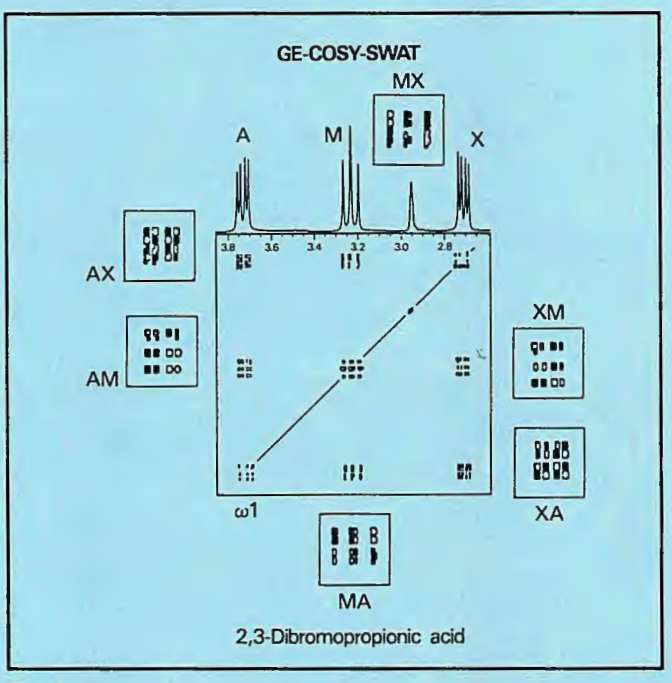


Fig. 1

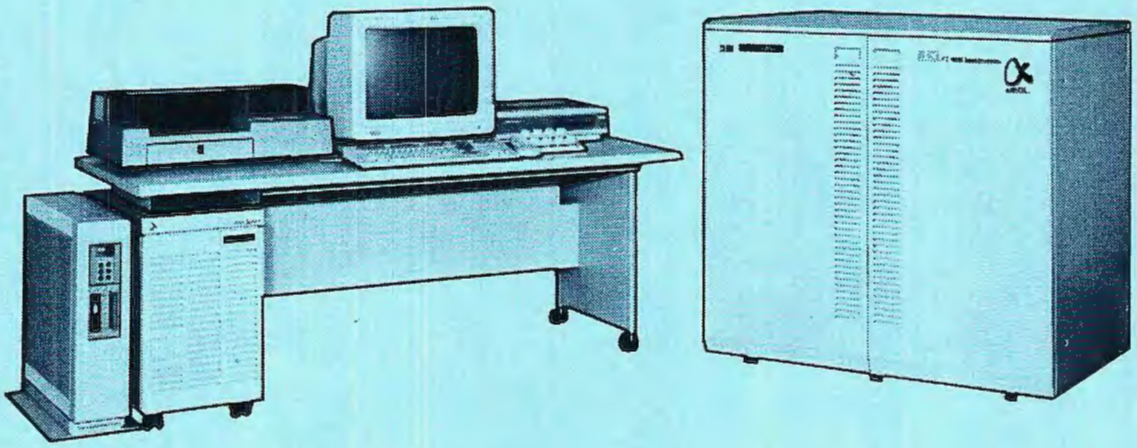
Contour plot of a 300 MHz pure-phase COSY spectrum of a solution of 2,3-dibromopropionic acid in benzene-d6 acquired with only a single acquisition per t_1 evolution time increment using the GE-COSY-SWAT method. Cross peaks are shown in expanded insets with positive peaks as darkened contours and negative peaks as open contours. A one dimensional spectrum is plotted across the top of the 2D spectrum.

GE NMR Instruments

8564 Printed in U.S.A.

IT'S NOW STANDARD: α

- High-speed phase coherent frequency switching
- Fine phase shifting (0.1°)
- High-speed attenuator control
- Programmable pulse shaping
- Linear power amplifiers
- Array processor
- User-friendly operating system



Come see the NMR with the **standard** features that are accessories from everyone else!

For more information please contact:

JEOL

11 Dearborn Road, Peabody, MA 01960 (508) 535-5900 (Phone) (508) 535-7741 (FAX)