TEXAS A&M UNIVERSITY
NMR
NEWSLETTER

No. 390

March 1991

Continued on page 2

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-d4	CH_3COOD	99.5%	1.1	17	118	0.551 (32)
D-120	Acetic Acid-d4 + TMS		99.5%				
D-13	Acetone-d6	CD_3COCD_3	99.8%	0.87	-94	57	0.460 (20)
D-121	Aceonte-d6 + 1% TMS		99.8%				
D-129							
D-14							543 (20)
D-21							0.611
D-122							
D-130							
D-28	Choloroform-d	$CDCl_3$	99.8%	1.50	-64	62	0.740 (20)
D-31	Chloroform-d + 1% TMS		99.8%				

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WCV-60 (A-60C)	11" X 26"	A-60, A-60A, A-60D	Gridded-Two Color
WCV-XL (XL-200)	11" X 26"	XL-200 (CP and FT)	Gridded-Two Color
WCV-XLFT-100	11" X 26"	XL-400 (CP and FT)	Gridded-Two Color
WCV-XL-200	11" X 26"	XL-200, XL-300, XL-400	Gridded-Two Color
WCV-990-2	11" X 16 3/4"	EM-990	Gridded-Two Color
WCV-20 (CFT-20)	11" X 16 3/4"	CFT-20, FT-80, FT-80A	Gridded-Two Color
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FORTHCOMING NMR MEETINGS

In Vivo Magnetic Resonance Spectroscopy Tutorial and Participatory Workshop, St. Louis, Missouri, **April 4 - 7, 1991**; See Newsletter 385, 58.

32nd ENC (Experimental NMR Spectroscopy Conference), St. Louis, Missouri, **April 7 - 11, 1991**; Contact: ENC, 750 Audubon, East Lansing, MI 48823; (517) 332-3667.

1991 Keystone Symposia on Molecular & Cellular Biology, Keystone, Colorado: **April 8-14, 1991**, *Frontiers of NMR in Molecular Biology*; *Proteolysis in Regulation and Disease*; *Protein Folding, Structure and Function*; See Newsletter 384, 46.

Hahn Symposium, Berkeley, CA, **April 20, 1991**; See Newsletter 390, 58.

Fifth Washington University-ENI/Emerson Electric Co., Symposium on NMR, St. Louis, Missouri, **May 20, 1991**; See Newsletter 388, 52.

Second Annual Workshop on Magnetic Resonance Imaging of Materials, Mass. General Hospital NMR Center, Charlestown, MA, , **May 13 - 14, 1991**; See Newsletter 388, 43.

Contrast-Enhanced Magnetic Resonance, a workshop of the Society of Magnetic Resonance in Medicine, Napa, California, **May 23-25, 1991**; Contact: S.M.R.M., 1918 University Ave., Suite 3C, Berkeley, CA 94704; (415) 841-1899, FAX: (415) 841-2340.

11th Delaware NMR Symposium, Univ. of Delaware, Newark, DE, **June 4, 1991** See Newsletter 390, 35.

Tenth International Meeting on NMR Spectroscopy, St. Andrews, Scotland, **July 8-12, 1991**; Contact: Dr. John F. Gibson, Secretary (Scientific), The Royal Society of Chemistry, Burlington House, London W1V 0BN, England; See Newsletter 387, 69.

Gordon Research Conference on Magnetic Resonance, Brewster Academy, Wolfeboro, NH, **July 15-19, 1991**; Chairman: R. Griffin; Information from Dr. A. M. Cruickshank, Gordon Research Center, Univ. of Rhode Island, Kingston, RI 02881-0801; Tel.: (401) 783-4011 or -3372; FAX (401) 783-7644.

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.

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

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.

Table of Contents, cont'd.

* * * * *

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If the mailing label on your envelope of this issue is adorned with a large **red dot** or circle: this decoration means that you will not be mailed any more issues until a technical contribution has been received by me.

Page Length ~~Request~~ Instruction

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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. 392 (May) ----------19 April 1991
No. 393 (June)----------17 May 1991
No. 394 (July)---------- 21 June 1991
No. 395 (August)---------- 19 July 1991

* * * * *

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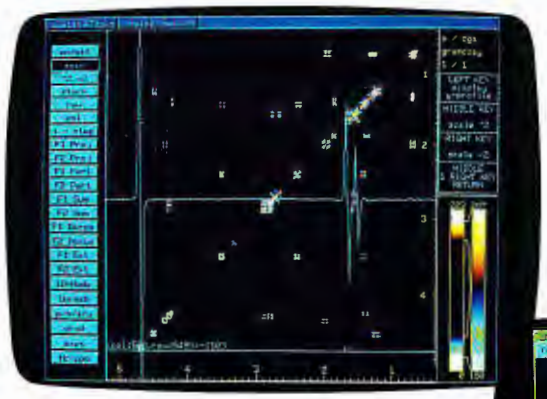

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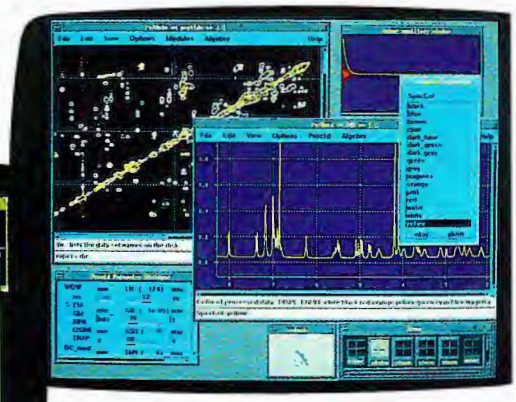
UXNMR on your SGI/IRIS.



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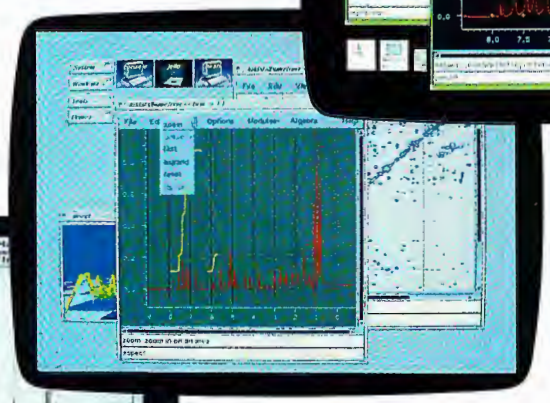

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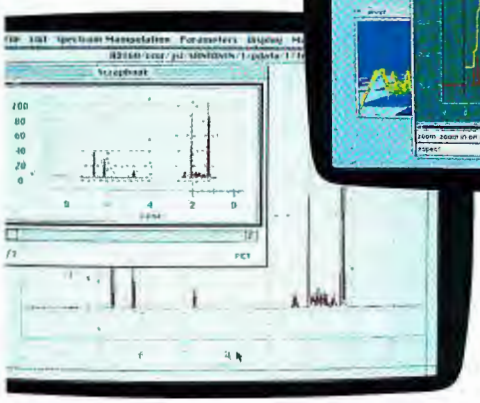

WINNMR on your Mac II or PC/PS system.

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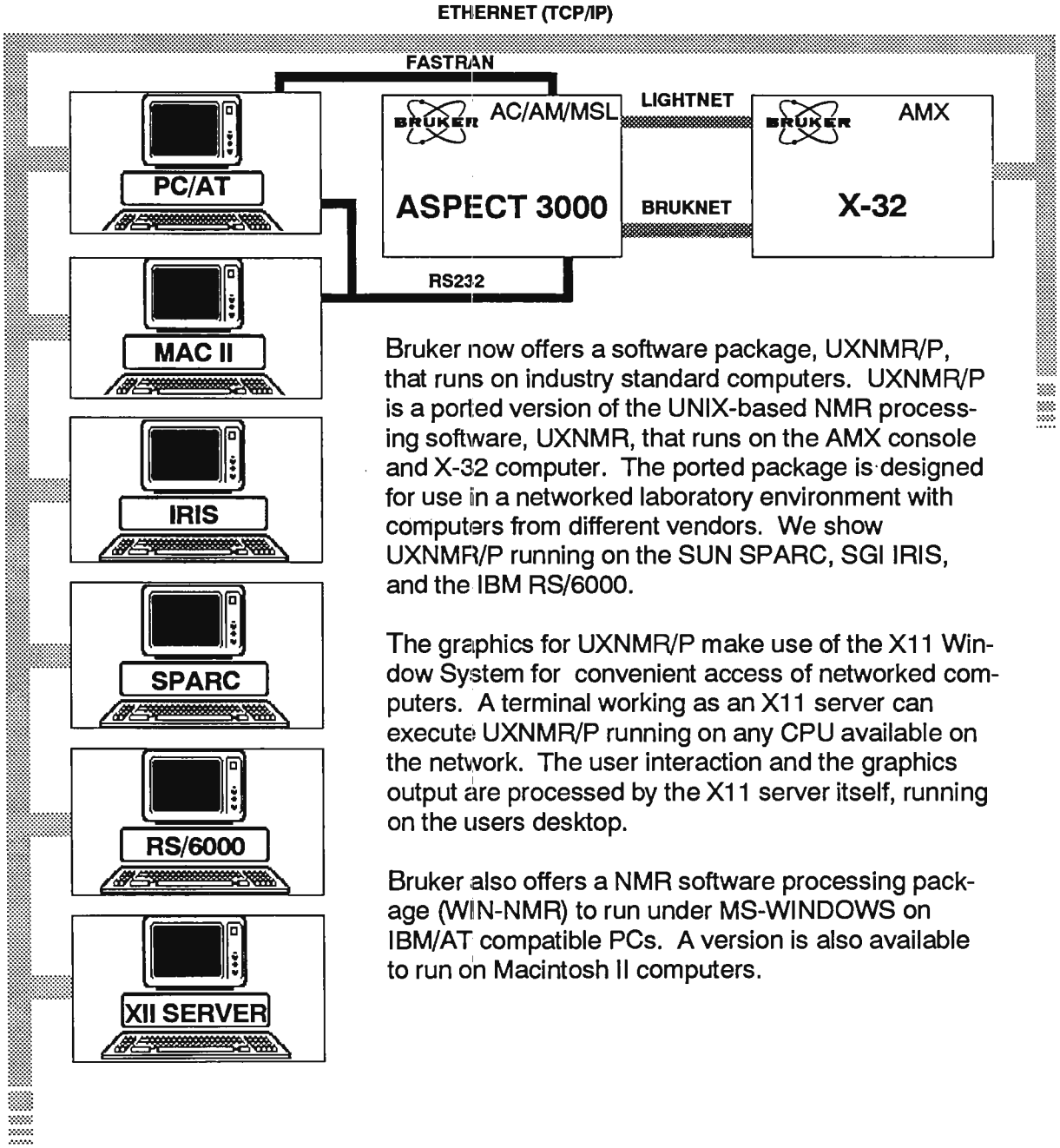

Bruker now offers a software package, UXNMR/P, that runs on industry standard computers. UXNMR/P is a ported version of the UNIX-based NMR processing software, UXNMR, that runs on the AMX console and X-32 computer. The ported package is designed for use in a networked laboratory environment with computers 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.

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discipline : Chemistry
vakgroep : Molecular Spectroscopy

Dr. B.L. Shapiro
TAMU NMR Newsletter
966 Elsinore Court
PALO ALTO, CA 94303
U.S.A.

Uw kenmerk	Uw brief van	Ons kenmerk	Inkiesnummer	Datum
		WV/DW/917	653109	8-1-1991
Onderwerp				(received 1/30/91)

Dear Dr. Shapiro,

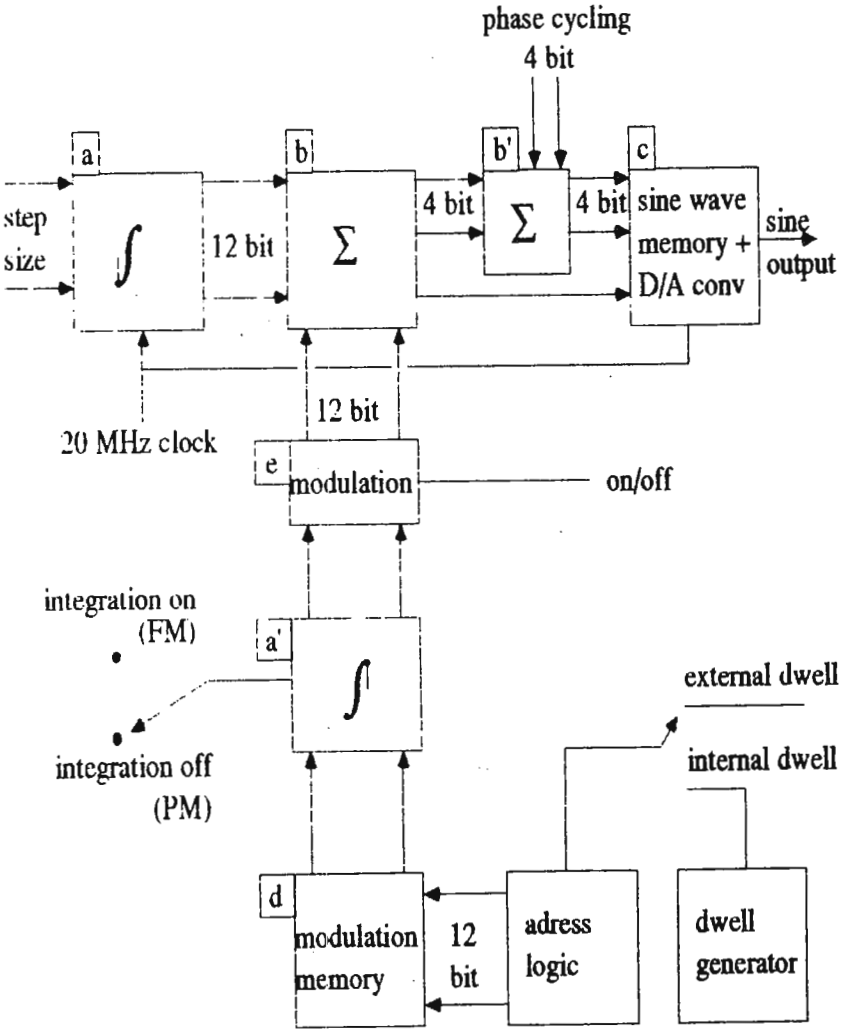
In modern NMR spectroscopy there is an increasing demand for precise computer control of the amplitude, frequency and phase of the rf source of the spectrometer. We have built a device, based on direct digital frequency synthesis, that allows fast frequency switching (and therefore also sweeping) without phase jumps and in addition has the capability to switch back to the original frequency, with a phase equal to the phase the original signal would have had if no frequency switching had occurred. The block diagram is shown in the figure. Block c is the heart of the instrument and consists of a memory that stores 4096 digital values of a sinewave. A sinewave with a precisely defined frequency can be generated by addressing the sinewave memory at predetermined intervals, provided by block a, at the clock rate. A frequency change can be obtained by a separate integrator block a' and the successive frequency step sizes which form the input of this integrator and which control the frequency (or phase modulation), are stored in a modulation memory (block d). Block b adds the carrier address stream from the block to the modulation address stream from block a'. For an experiment where the frequency sweep is terminated, the modulation address information is simply switched off by block e. Clearing this register means that the frequency and phase from then on is completely determined by the step size of block a. The modulation memory is cycled through by means of the address logic by internal or external advance. In this way only one modulating period (sine, triangle or arbitrary) has to be stored in the modulation memory. For phase cycling purposes block b' has been added.

The instrument has been implemented in two Bruker AM spectrometers. A more detailed report will be published elsewhere.

Please credit this contribution to the subscirption of Prof.dr. E. de Boer.

Best wishes for you and your Newsletter in this new year!

Jan van Os Gert-Jan Steeg Wiebren Veeman



C SEARCH

ORG.MAGN.R

$C_{26}H_{38}O_6$

$CDCL_3$

C - 1:	36
C - 2:	27
C - 3:	73
C - 4:	170
C - 5:	20
C - 6:	38
C - 7:	139
C - 8:	122
C - 9:	31
C -10:	31
C -11:	49
C -12:	36
C -13:	19
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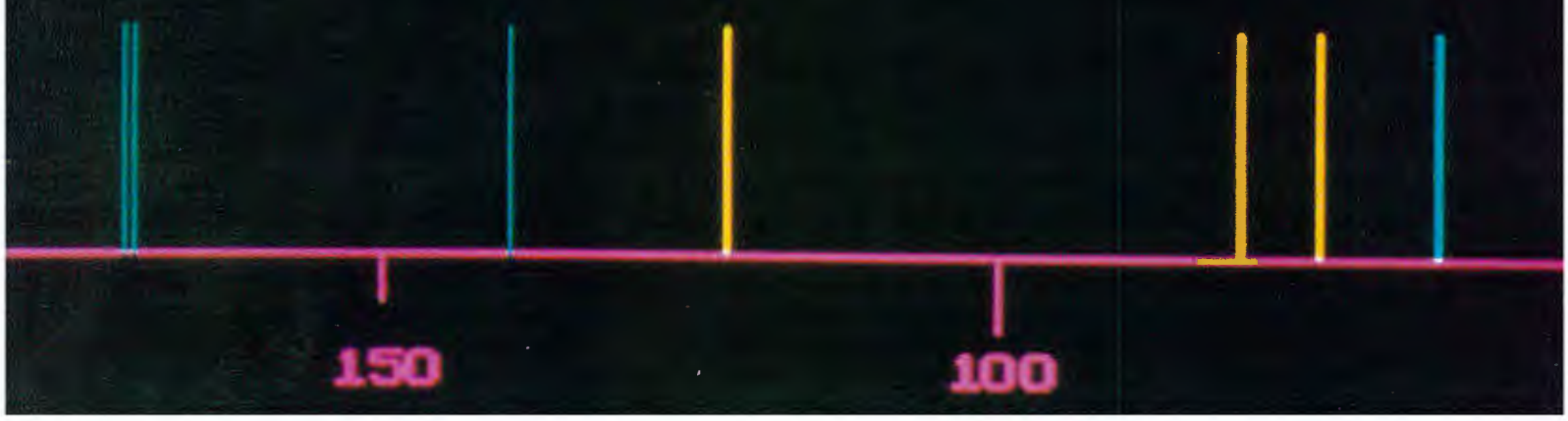

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Faculty of Science
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January 22, 1991
(received 2/7/91)

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

Dear Dr. Shapiro:

The importance of DNA-RNA interactions during transcription of the genetic code and the interest in using exogenous antisense DNA to prevent translation of mRNA has focussed recent attention on NMR studies of DNA-RNA hybrid structure. Such hybrids contain AT and AU base pairs the imino protons of which are not readily distinguished by standard NMR methods. Our interest in this area has led us to focus on INAPT experiments to correlate the imino protons of AT or AU base pairs with C5 carbons through three intervening bonds. The AT and AU base pairs can then be distinguished because the C5 carbon of thymidine is quaternary and that of uridine is not.

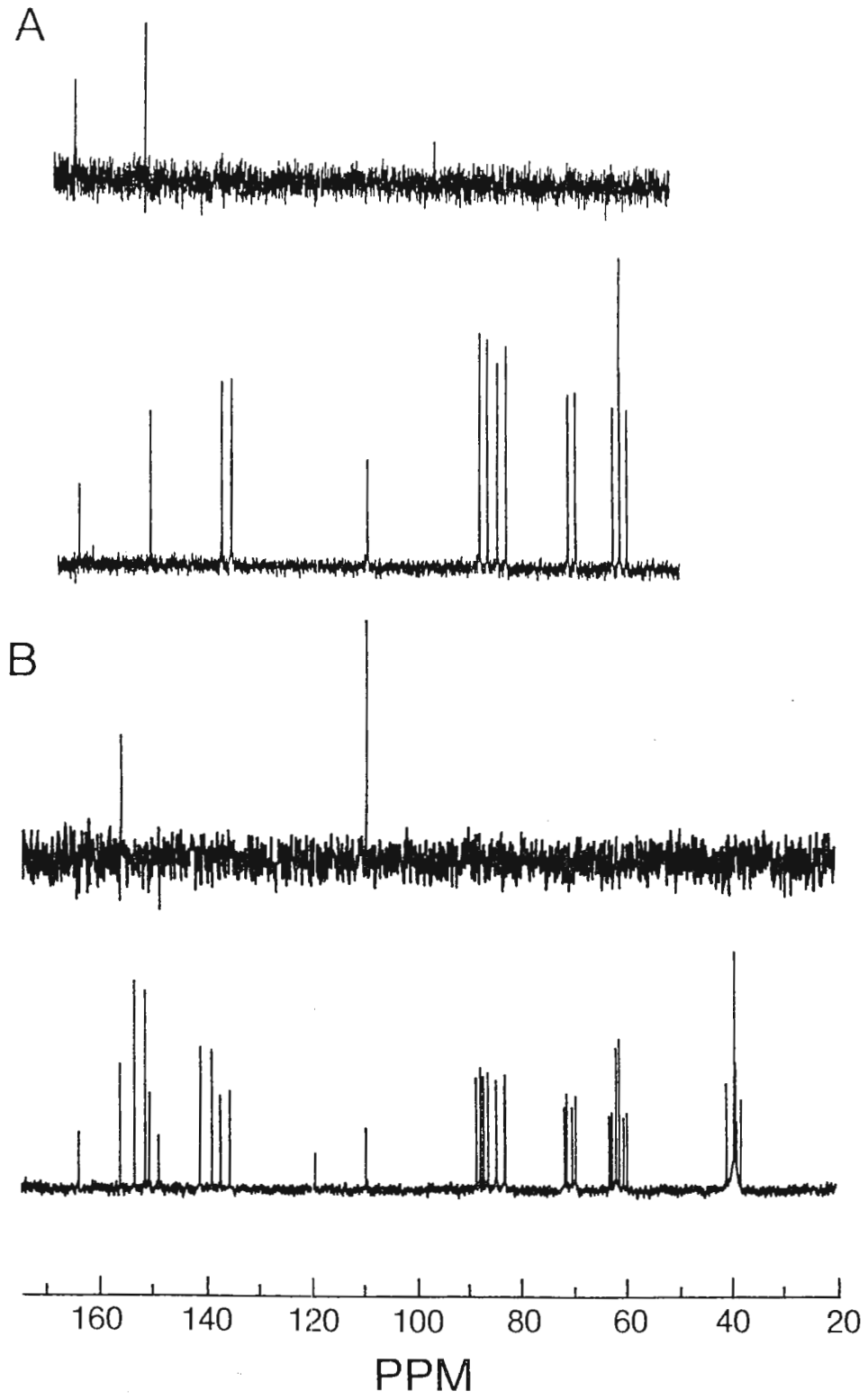
The INAPT experiment has been used infrequently in the study of nucleic acids and polarization transfer from the exchangeable amino and imino hydrogens of nucleoside bases has not been reported. The exchangeable hydrogens of nucleoside base pairs have two and three bond couplings to carbons of the nucleoside base when they are not exchanging rapidly. There is a change in these long range couplings upon base pair formation which suggests a reorganization of the bonding electrons. The INAPT spectrum of thymidine with selective application of ^{1}H pulses to the imino hydrogen is presented in Figure 1. The spectrum shows that polarization transfer *via* a two bond coupling between the imino hydrogen and C2 and C4 is a viable but inefficient process. No polarization transfer is observed *via* the three bond coupling to C5 in this system. Polarization transfer from other hydrogens of the nucleoside proceeds *via* three bond scalar couplings. The INAPT spectrum in the AT base pair is also presented in the figure and the results indicate that three bond scalar couplings including a carbon-nitrogen bond may be used to transfer polarization from proton to carbon.

Yours sincerely,

W. Gmeiner, Ph.D.

J.W. Lown
Professor

:aw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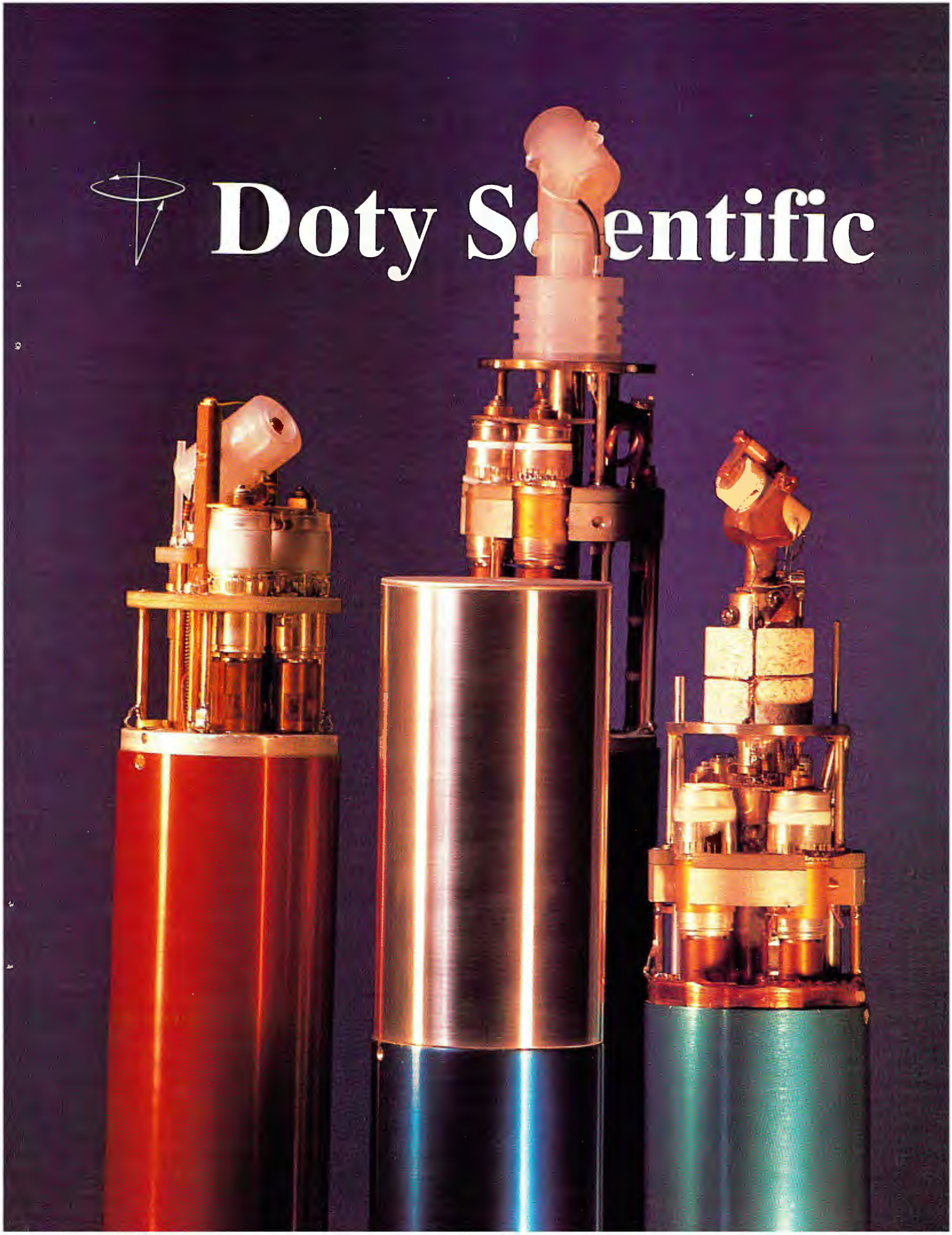

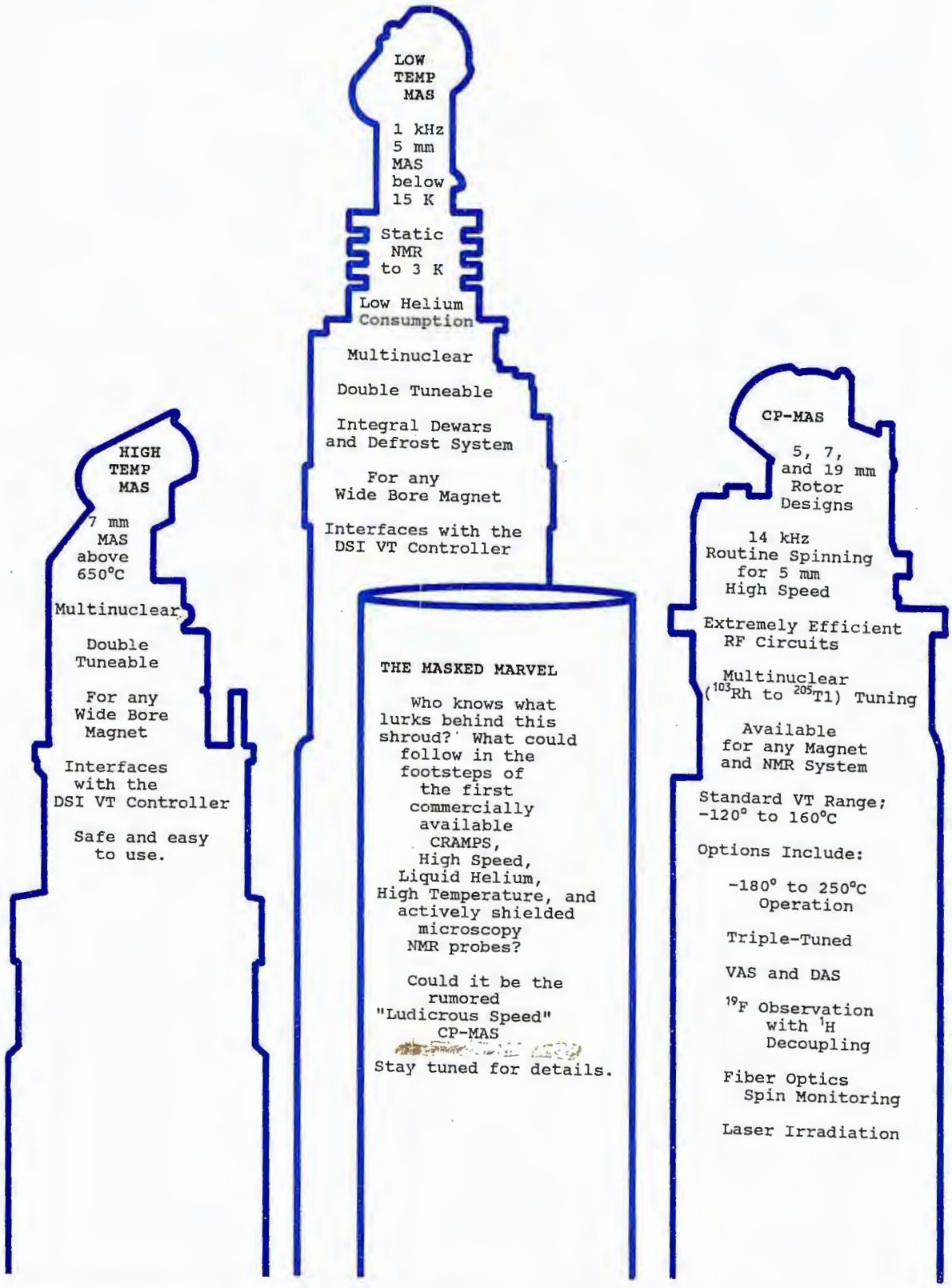

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Jan 15, 1991 (received 2/7/91)

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

Error Estimates of LPSVD Analysis of 31P NMR

Dear Dr. Shapiro,

Recently we have begun using a home-written version of a linear prediction and singular value decomposition (LPSVD) algorithm (1) to quantitate in-vivo 31P NMR spectra. LPSVD has the desirable qualities of being non-iterative, independent of phase, and requiring almost no user assistance. However, a drawback of the algorithm is that error estimates for each fitted spectral parameter are not readily determined. It has been suggested that the Cramer-Rao lower bound (2) can be used to estimate the error of LPSVD, but these estimates depend heavily on the correctness of the assumed model function. Moreover, the Cramer-Rao bounds reveal no information about systematic error which may be present in LPSVD.

We have therefore conducted Monte Carlo studies (3) of LPSVD's performance on real, high SNR data with varying levels of added noise. The advantage of conducting such a simulation is that true statistical measurements of the errors are given for the test data set at each SNR level. It then remains to be shown that analyses of other real spectra would produce similar error measurements. The results of the study can then allow us to make claims regarding the certainty of the quantitation of our data.

The first results of our studies have produced encouraging results. Figure 1 shows the original 31P spectra used in the study, which was obtained from a rat gastrocnemius muscle using a 1 cm diameter surface coil operated by a 2T GE 1280 system. The fitted areas of the Pi, PCR, and beta-ATP peaks were examined and their mean error and standard deviations are shown in figure 2. Also shown is the algorithm's success at detecting the presence of a peak (fig. 3).

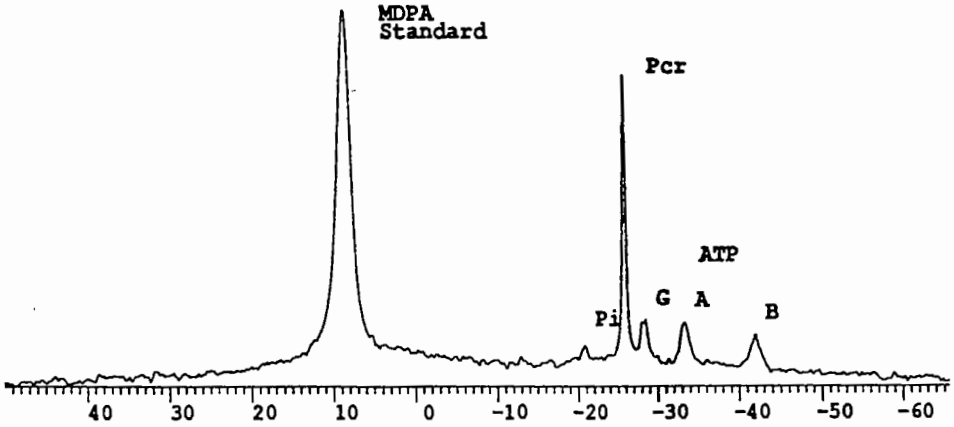

Figure 1. 31P spectra used in Monte Carlo simulations.

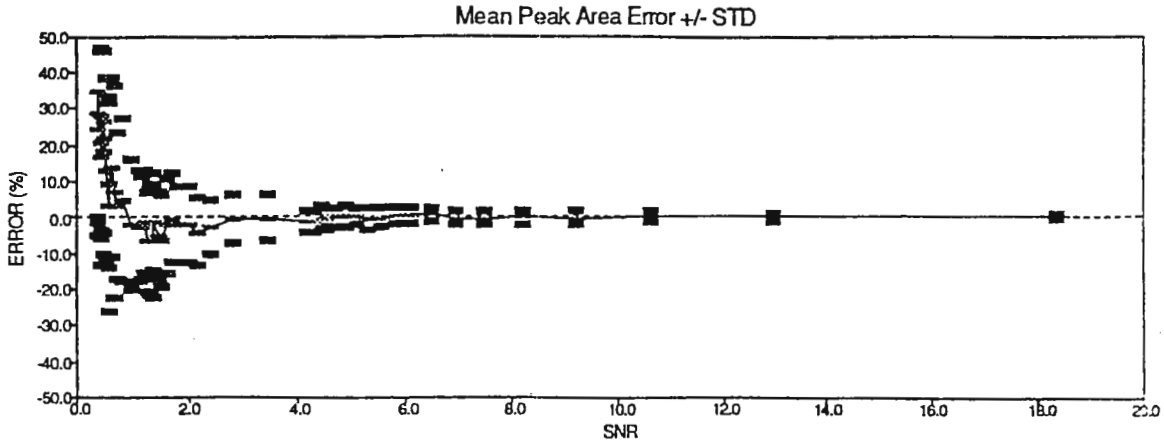

Figure 2. Percent error of peak areas +/- std (n = 100).

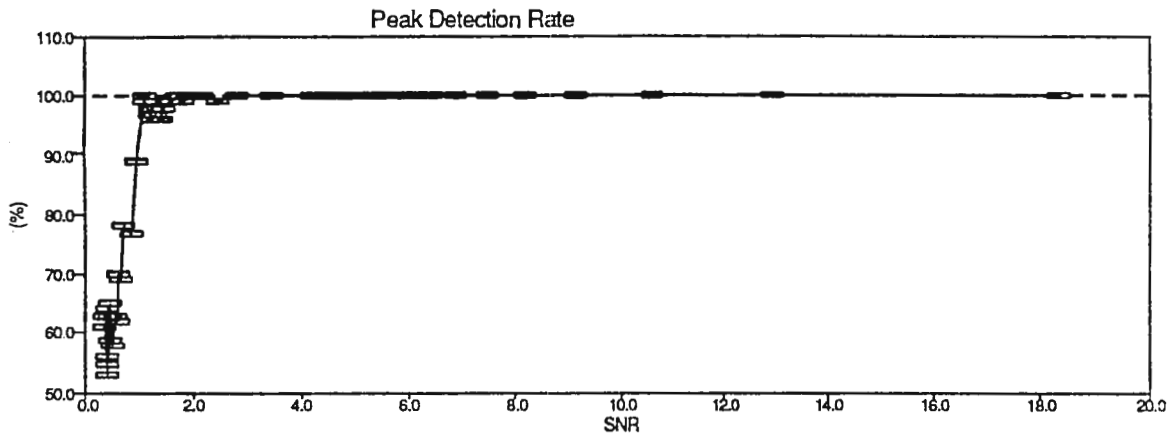

Figure 3. Percent successful peak detection (n = 100).

The initial results show that at signal-to-noise levels of greater than 2.0, LPSVD estimates the peak areas to with a standard deviation of less than 10 percent. Moreover, the systematic error is negligible (within 5%). However, at lower SNR levels, the systematic error and its variance quickly increase. The peak detecting ability of LPSVD corresponds well with the accuracy of its area estimation. With more extensive Monte Carlo simulations on real and synthetic data, we should be able to make reliable estimates of the accuracy of LPSVD in the analysis of NMR data.

(1) R. Kumaresan and Donald Tufts. IEEE Transactions ASP, 30, No. 6, 833 (1982)
(2) H. Barkhuijsen, R. DeBeer, and D. van Ormondt, J. Mag. Res., 67, 371 (1986)
(3) W.H. Press et al, Numerical Recipes, Cambridge University Press, 529 (1987)

Sincerely,

Mark Elliott
Research Assistant

Michael M. Weiner, MD
Professor of Medicine and Radiology

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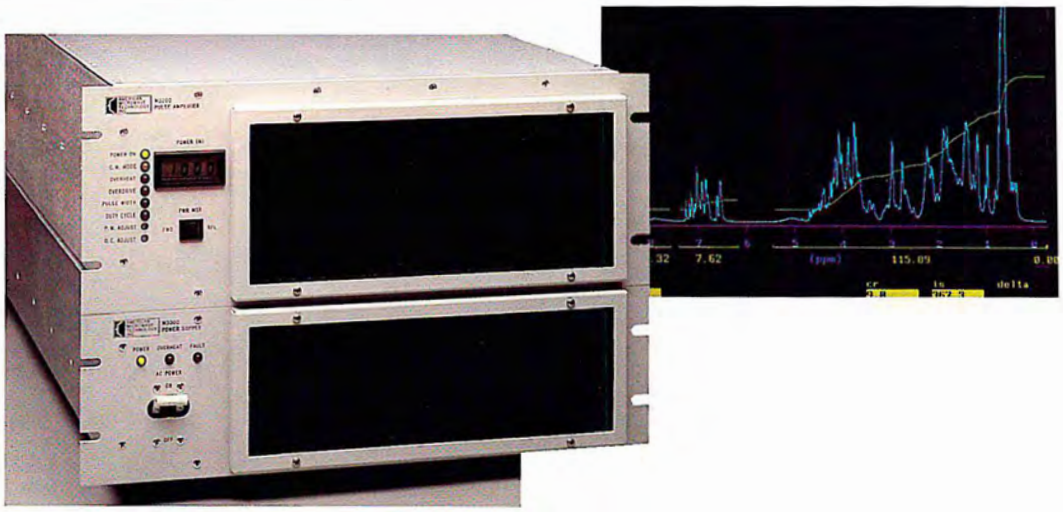

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Linearity (±1dB to 200Mhz)		0-800W	0-250W
(To 220MHz)		0-600W	0-200W
Gain (typ.)		65dB	60dB
Gain Flatness		±4dB	±3dB
Input/Output Impedance		50 ohms	
Input VSWR		< 2:1	
Pulse Width		20mS	
Duty Cycle		Up to 10%	
Amplitude Rise/Fall Time		200nS typ.	150nS typ.
Amplitude Droop		5% to 10mS typ; 7% max	
Phase Change/Power Output		10° to rated power typ.	
Phase Error Overpulse		4° to 10mS duration typ.	
Noise Figure		11dB typ.	8dB typ.
Output Noise (blanked)		< 20dB over thermal	
Blanking Delay		< 5uS on, 2uS off, TTL signal	

Protection

1. VSWR- will withstand infinite VSWR at rated power
2. Input overdrive- up to +10dBm
3. Over duty cycle/pulse width
4. Over temperature

Supplemental Characteristics:

Connectors (on rear panel)

1. Input- BNC (F)
2. Output- Type N (F)
3. Blanking- BNC (F)
4. Interface- 25pin D(F),EMI filtered

Indicators, Front Panel

1. Peak power meter
2. Over temperature
3. Over duty cycle
4. Over pulse width
5. CW Mode
6. Overdrive

System Monitors

1. Thermal
2. DC power supply fault
3. Over duty cycle
4. Over pulse width

Front Panel Controls

1. A.C. power
2. Pulse width
3. Duty cycle

Cooling	Internal forced air	
Operating Temperature	+10 to 40°C	
A.C. Line Voltage	120/240 VAC, ±10%, 50-60Hz (3200, 220/240V only)	
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Biomedical NMR Research Group
Dr. Hommo T. Edzes
c/o Department of Chemistry, Section Physical Chemistry

Date February 4, 1991 Telephone: +31 20 548 5366 Telefax: +31 20 461 479

Mailing address: De Boelelaan 1083, 1081 HV Amsterdam, The Netherlands.

vrije Universiteit *amsterdam*

Analysis and Identification of Phospholipids in Tissue Extracts.

(received 2/11/91)

Dear Dr. Shapiro,

Analysis of generic phospholipids in crude tissue lipids extracts by high-resolution ^{31}P NMR is attractive: sample handling is minimal and NMR intensities are directly proportional with lipid concentration. We have been able to resolve virtually all phospholipids in extracts of diverse origin in the solvent system chloroform-methanol-0.2 M K_4EDTA in water, which was recently introduced by Meneses and Glonek(1). Linewidths are about 1 Hz (at 162 MHz) but phospholipid resonances are crowded mainly in the range of 0.5 to -1.0 ppm. We found assignment of observed resonances to specific phospholipids to be unreliable if based on chemical shifts as these vary from sample to sample. In particular when extracts of different origin are compared, shifts often could not be matched with certainty to a specific phospholipid. Addition of reference standards does not offer full relief, because standards are not easily available for all phospholipids, and resonances of different phospholipid species may coincide.

We have developed a strategy to identify most of the observed ^{31}P resonances by 1D and 2D NMR, in combination with published data on phospholipid composition in different tissues. First, valuable information is gathered by studying the phospholipid ^{31}P NMR profile at a number of different compositions of the solvent system. All resonances are shifting, but direction and magnitude of the changes add to the information contained in the shifts themselves. A complete picture is obtained of all observable resonances, and one or two standard compositions of the solvent sytem are established which yield optimum separation of all resonances(2,3, to be published). The optimum compositions subsequently can be used in a comparative analysis of samples of the same origin, as we have found little variation so far between extracts from the same type of tissue. Second, assignment of the major peaks in an unfamiliar extract can be confirmed by a 2D ^{31}P-^{1}H NMR correlation experiment with isotropic proton mixing(3). For each phospholipid resonance, a characteristic proton 'fingerprint' is obtained which identifies the phospholipid.

The 2D NMR experiment used is shown in the Figure. As an example of its application, we will focus on the region of generic phosphatidylcholines. Three PC's do occur commonly: PC, PC plasmalogen (PC_{plas}), and 1-alkyl PC (PC_a). Meneses and Glonek observed two resonances in this region which they assigned to PC_{plas} (-0.78) and PC (-0.84 ppm). PC_{plas} is a major component in heart tissue, but the intensity of the peak at -0.78 ppm was much too low. We did observe, however, a separation of the 'PC' peak into two large resonances when the proportion of methanol and water in the solvent system was increased. The 2D spectrum clearly establishes that the two peaks are of different generic origin. The proton 'fingerprint' of one of the two resonances has been assigned previously to PC whereas the other one fits in with the lipid backbone resonance pattern of PE_{plas}(4). The component at -0.78 ppm is assigned to PC_a, in agreement with the assignment made by Bradamante et al(5). Its proton fingerprint fits the patterns observed for 1-alkyl glycerophospholipids observed in selected other extracts. We are now

completing a 'catalogue' of the phospholipid proton 2D 'fingerprints'.
This is a joint project with Dr. T. Teerlink, Department of Clinical Chemistry, and Prof. Dr. J. Valk, Department of Diagnostic Radiology, University Hospital, Vrije Universiteit.
Please credit this contribution to the subscription of Dr. J. Bulthuis.

References:

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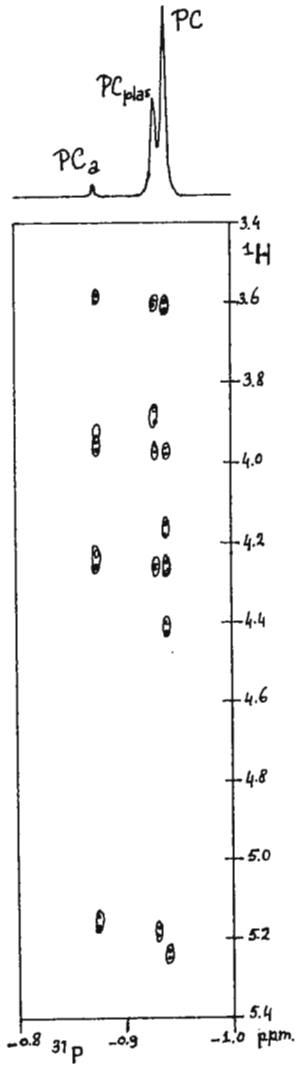
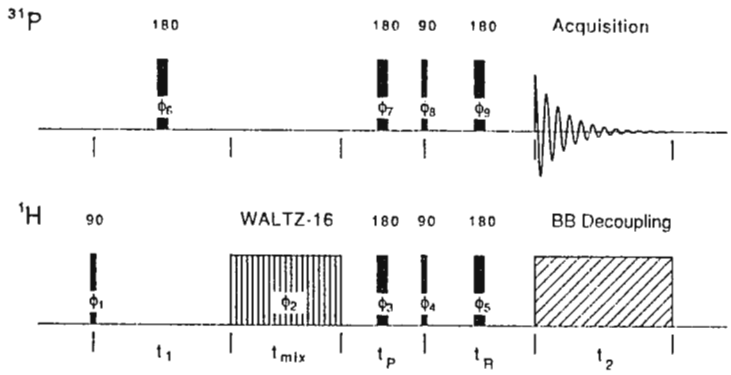
Yours sincerely,

Hommo T. Edzes



Below: 2D experiment: phase-sensitive fully refocussed INEPT with isotropic proton mixing
Right: PC region of 1D and 2D NMR spectra of rabbit heart tissue extract in chloroform-methanol-water (0.2 M K_4EDTA), proportions 100:90:45.



Phase cycling:																	
	ϕ_1 (+TPPI)	+Y	-Y	+Y	-Y	+Y	-Y	+Y	-Y	+Y	-Y	+Y	-Y	+Y	-Y	+Y	-Y
	$\phi_{2,4}$	+X	+X	+X	+X	+X	+X	+X	+X	+X	+X	+X	+X	+X	+X	+X	+X
	$\phi_{3,5}$	+X	+X	-X	-X	+X	+X	-X	-X	+X	+X	-X	-X	+X	+X	-X	-X
	$\phi_{6,7}$	+X	+X	-X	-X	+X	+X	-X	-X	+X	+X	-X	-X	+X	+X	-X	-X
	ϕ_8	+X	+X	+X	+X	-Y	-Y	-Y	-Y	-X	-X	-X	-X	+Y	+Y	+Y	+Y
	ϕ_9	+X	+X	-X	-X	-Y	-Y	+Y	+Y	+X	+X	-X	-X	-Y	-Y	+Y	+Y
	Receiver	+X	-X	+X	-X	-Y	+Y	-Y	+Y	-X	+X	-X	+X	+Y	-Y	+Y	-Y

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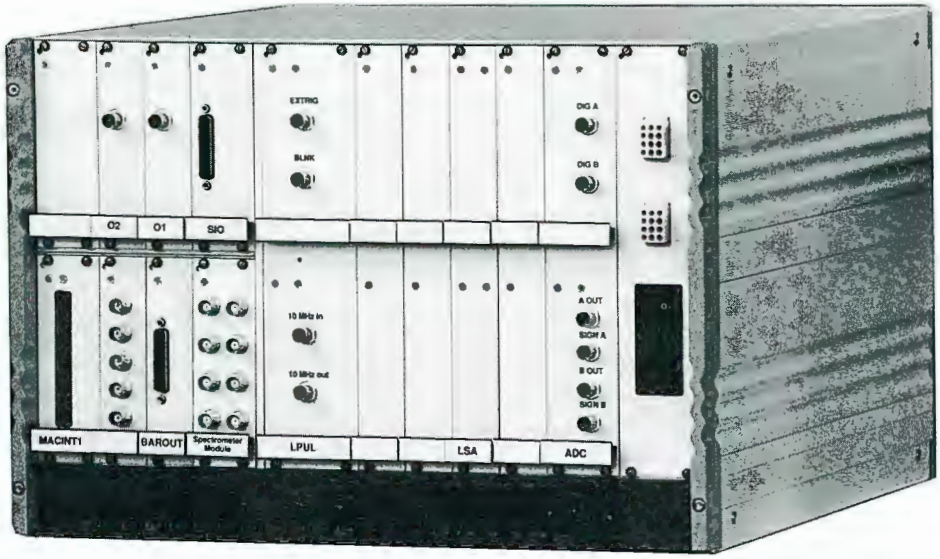

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Zürich, January 18, 1991 / hu
(received 1/30/91)

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

Palo Alto, CA. 94303
USA

Proton exchange from internal hydration water in proteins

Dear Barry

Looking at protein hydration in aqueous solution using NOESY (1) one finds that all NOE cross peaks between polypeptide protons of the protein and the hydration water protons are at the ω_1 chemical shift of the bulk water signal. This result holds also for internal water molecules, which are not in direct contact with the bulk water (1,2). Since the proton chemical shifts of these interior waters are not known, it has so far not been possible to distinguish between the following two possible situations: (i) the water chemical shifts in the protein interior and the bulk water are indeed identical; (ii) the apparent chemical shift degeneracy with the bulk water is caused by chemical exchange which is rapid enough to conceal any separate ^{1}H resonance of the interior water molecules. This distinction can be made by comparing the chemical shifts of the NOESY cross peaks with the interior water protons in samples with and without an extrinsic shift reagent (Co^{2+} shifts the resonance of the bulk water by about 0.01 ppm/mM (3)).

In NOESY experiments with BPTI at 4°C, pH 3.5, after addition of Co^{2+}, all water-polypeptide cross peaks were still at the chemical shift of the bulk water signal. This shows that situation (ii) prevails. We can deduce an upper limit of 20 ms for the lifetime of the interior water molecules with respect to chemical exchange with the bulk water, where it is assumed that a separate signal exists for the interior water within ± 0.4 ppm of the bulk water signal, so that its direct detection is prevented by the water suppression technique used (1). The corresponding exchange rate of $\geq 50\ s^{-1}$ would then cause sufficient line broadening to obscure also the presence of the NOE cross peaks between the interior water and the polypeptide. At the same time the chemical exchange provides an efficient relay of magnetization between the interior water and the bulk water, so that the polypeptide—water cross peaks are observed at the frequency of the bulk water signal.

Gottfried Otting Edvards Liepinsh Kurt Wüthrich

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THE UNIVERSITY OF UTAH

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FAX: 581-7087
davis@adenosine.pharm.utah.edu

January 29, 1991
(received 2/5/91)

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

vnmr2felix: Program for Sun-based Varian Spectrometers.

Dear Barry:

We have recently written a program that takes Varian "fid" files and converts them into a form that is readable by FELIX (Hare Research, Inc.). Previously available Fortran programs were either unacceptably slow, or just didn't work. After conversations with Dennis, we decided that the best course of action was to write a C program for this task. We are running this program, vnmr2felix, on SparcStations, a Sun 386i, and a SGI 4D/20. Conversion times for a 2D data set consisting of 800, 4096 point FIDs are $\approx$ 50 seconds. Simple command line switches specify input and output as well as the printing of various conversion statistics.

The source code, and a README file describing the program and how to install it can be obtained by E-mail request to davis@adenosine.pharm.utah.edu. Some SGI users may be *sans* C compiler, if this is the case let us know that you need to be mailed a uuencoded executable for your Iris. Vnmr2felix will also be bundled as part of Felix. If you don't have E-mail access and are convinced that you can't live without this delightful program, Kevin Banks at Hare Research can probably help you out.

Please accept this contribution to initiate a subscription.

Sincerely yours,

Darrell R. Davis

John L. Daschbach

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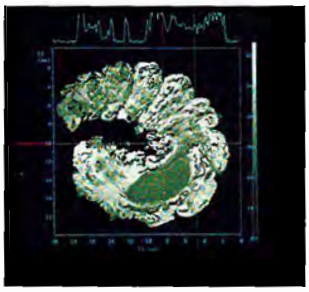

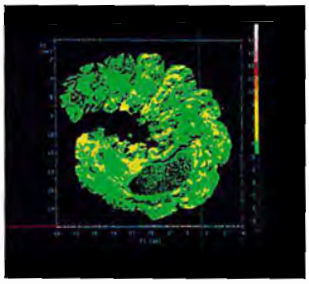

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Dr. B.L. Shapiro, Editor
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto, CA 94303

(received 2/7/91)

Dear Dr. Shapiro:

NMR, STROKE AND TEMPERATURE

NMR imaging and spectroscopy methods are proving to be of considerable value in studies of experimental stroke. Under general anesthetics animals (including humans) lose the ability to regulate body temperature, and in most laboratory conditions this means that body temperature falls. A further decrease in brain temperature takes place when the cerebral blood supply is disrupted in creating the experimental injury. Interestingly it is only recently that the effects of small temperature changes on the severity of ischemic injury have been studied in detail.

We have used NMR imaging to follow the development of brain injury caused by 10 minutes of forebrain ischemia in rats whose brain temperatures were kept at 33, 35, 38 or 40°C during ischemia and for 1 hour following reperfusion (control of body temperature is not sufficient). In normothermic (38°C) animals repeated imaging examinations showed enhanced intensity corresponding to edema appearing over about 4 days in the hippocampus, striatum, and in some cases the neocortex. At lower temperatures the images showed no signs of tissue injury. None of the hypothermic (40°C) animals survived beyond a few hours unless body and head temperature was rapidly cooled to normal while the brain was being reperfused. Even with this cooling the animals did not survive beyond about 30 hours, and imaging studies at 24 hours showed marked uniform enhancement, suggesting extensive edema and hence tissue injury throughout the brain. Histological examination after 4 days (1 day in the 40°C rats) confirmed the NMR imaging results, with neuronal damage evident in the regions showing enhanced image intensity.

We are now using ^{31}P NMR to compare cerebral metabolite and pH changes during and following ischemia at different temperatures. For this we use an animal holder with a circulating water bath around the head to control brain temperature.

These pronounced effects of quite small changes in temperature emphasize the need for extreme care in studies of cerebral ischemia. The effects may be important when using drugs which can affect tissue perfusion, and hence brain temperature. There are also a number of evident clinical implications regarding patients with fever or undergoing neurosurgical procedures.

Please credit this to Ted Schaefer's account.

Sincerely,

Jim Peeling

James Peeling

Garnette Sutherland

Garnette Sutherland

The University of Leeds LEEDS LS2 9JT

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Telex 556473 UNILDS G Fax (0532) 336565

From J. D. Kennedy
School of Chemistry

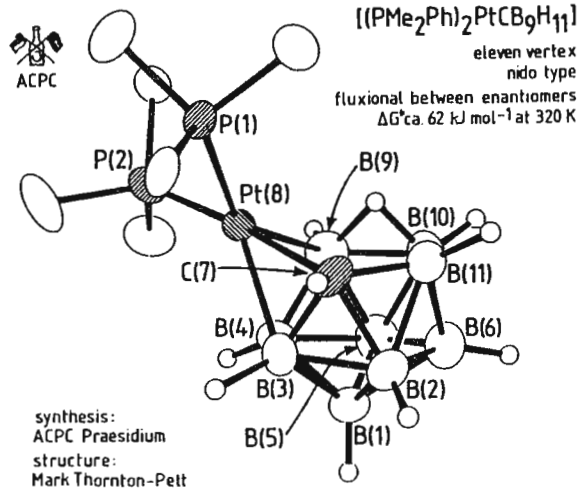
(received 1/30/91)

Dear Dr Shapiro

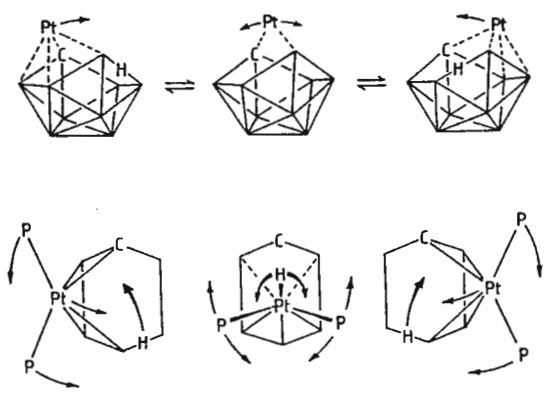
Fluxionality of a Platinaheteroborane by Multieliment NMR

We use NMR as a crucial tool in the examination of the structure, behaviour and bonding of the metallaboranes and metallaheteroboranes that we make. Many of these polyhedral compounds exhibit fluxionality, which is of course an "NMR phenomenon". A recent interesting example, requiring a variety of NMR experiments, is $[(PMe_2Ph)_2PtCB_9H_{11}]$, an ACPC* compound we are examining in collaboration with Bob Štíbr's group in Řež u Prahy, Czechoslovakia.

At 275 K the NMR is consistent with its asymmetric solid-state structure. The nine separate ^{11}B resonances are assigned by $[^{11}B$-$^{11}B]$-COSY45 experiments together with the incidence of coupling to 195Pt for the positions adjacent to platinum. $^1H(FT)$-$\{^{11}B(CW,selective)\}$ work thence assigns the ^1H spectrum; we have found this proves to be much more convenient and rapid than $[^1H$-$^{11}B]$ 2D correlation experiments (although the information obtained is not quite as complete, we can live with that; the large difference in instrument time is one of the principal governing factors). The ^1H and ^{11}B assignments are then confirmed by $[^1H$-$^1H]$-COSY work, done with simultaneous $\{^{11}B(CW$, broadband noise$)\}$ decoupling, although some anomalous (in terms of a static structure) cross-peaks arising from exchange correlation were also observed. Two ^{31}P resonance positions are observed, and two pairs of P-methyl ^1H resonance positions.



At higher temperatures, coalescence in each of the ^{31}P, ^{11}B, and ^1H spectra is observed, with $\Delta G^{\ddagger}$ *ca.* 62 kJ mol^{-1} at 325 ±10 K. In the ^{11}B spectrum a 2:2:2:1:1 pattern is observed, with the assignments showing a movement of the platinum atom between two *tetrahapto* $\{CB_3\}$ sites across the formal mirror plane of the $\{CB_9H_{10}\}$ unit, *i.e.* the fluxionality is an effective interchange between enantiomers (Scheme). The coalescence in the ^{31}P spectrum shows that this is associated with an exchange of the phosphorus sites, but the nature of the coalescence of the pairs of P-methyl ^1H resonances, of which each coalescing pair contains one resonance from each PMe_2Ph ligand [as determined by ^1H-$\{^{31}P(CW$, selective$)\}$ work], shows that there is not a general scrambling, but that the shift of the platinum across the open face of the $\{CB_9H_{10}\}$ subcluster is linked with a specific half-twist of the $\{PMe_2Ph\}_2$ ligand sphere, probably as illustrated in the Scheme. We were surprised that the fluxionality was therefore quite different to that of the isoelectronic and quasi-isostructural congener $[(PMe_2Ph)_2PtB_{10}H_{12}]$, which we examined some time ago (*J. Chem. Soc., Chem. Commun.*, 1980, 305), and which shows a contrarotation of the $\{PMe_2Ph\}_2$ and $\{B_{10}H_{12}\}$ ligand spheres *within* the constraints of a *tetrahapto* borane-to-metal bonding, with no evidence for platinum site-migration.



We intend to submit the work sometime as part of a quest for a publication in *J. Chem. Soc., Dalton Transactions*.

Yours in harmonic resonance

John Kennedy

John Kennedy
(also on behalf of some other ACPC* members Tom Jelínek, Xav Fontaine, Bob Štíbr, and Mark Thornton-Pett).

* ACPC is IUPAC nomenclature for the Anglo-Czech Polyhedral Collaboration alias the Anglo-Czech Pivo Club

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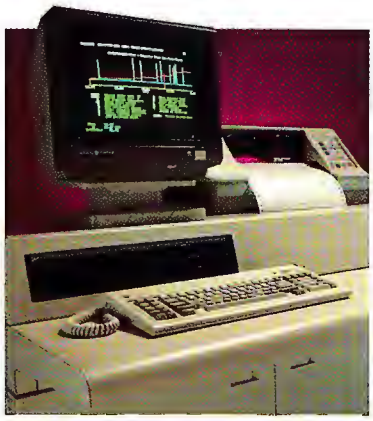

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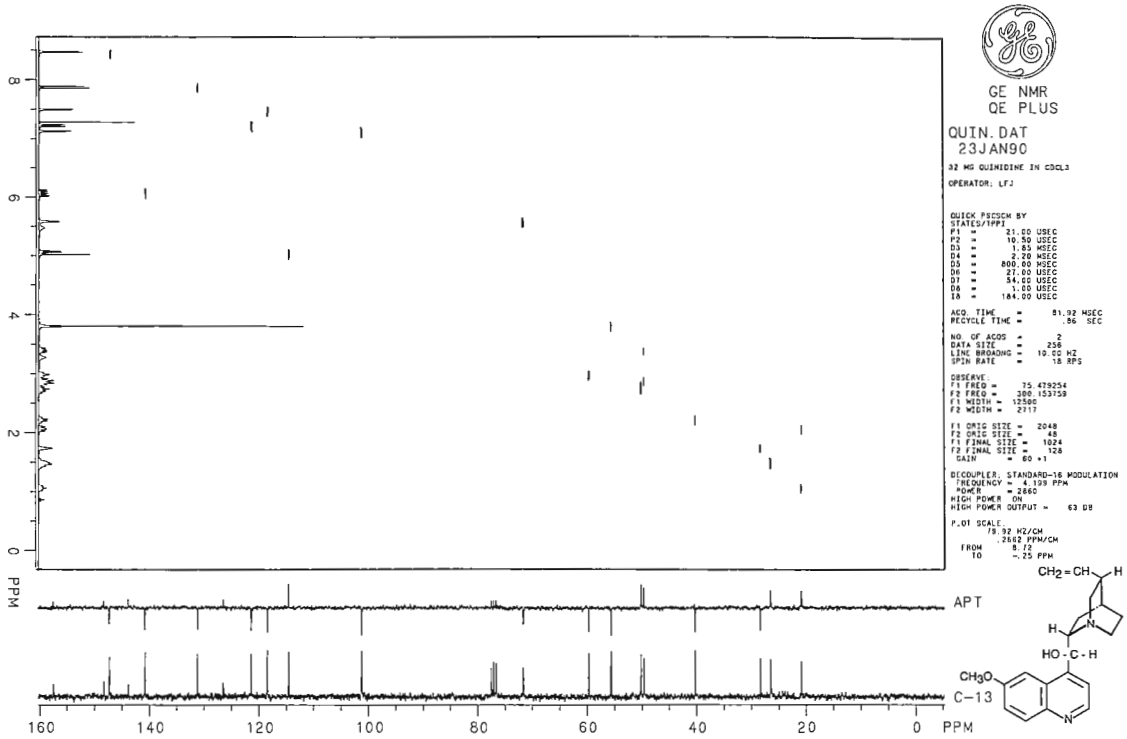

An automated run using MACRO mode operation on a sample of 32 mg of quinidine in 0.5 ml chloroform-d (0.20M). Data were obtained using the 5 mm broadband probe. ^{1}H, ^{13}C, APT and phase sensitive 2D data were collected, processed and plotted—including the 2D contour—in only 8.2 min.

CHEMICAL CENTER
UNIVERSITY OF LUND
PHYSICAL CHEMISTRY 2

December 14, 1990
(received 1/19/91)

Dear Dr. Shapiro,

We, like so many others, have recently discovered the great joys of $^{1}H-^{15}N$ heteronuclear experiments for the study of proteins. In our lab, expression of ~100% ^{15}N-labelled protein (calbindin D_{9k}) have so far worked very well, thanks to our wonderful technician Eva Thulin, who supplies us with decigram quantities of protein from every 5-liter batch.

We have applied the $^{1}H-^{15}N$ HSQC method to amide proton exchange experiments, carried out on our GE Ω-500. This makes nice, well resolved 2D spectra of Ca^{2+}-saturated calbindin D_{9k}, for which only 2 peaks are overlapped (in the amide-$^{1}H/^{15}N$ region). Naturally, it is of interest to follow the exchange-out behaviour of the amide protons over a large number of time-points. For all but the very slowly (and very quickly) exchanging protons, this requires fast acquisition of spectra. To this end, we progressively cut the total acquisition time shorter, and were pleased to find an 1-hour (16 scans, 128 × 2048) spectrum having satisfactory sensitivity. Then, prompted by our good friends at Scripps Clinic, we tried acquiring a spectrum with "minimal" phase cycling, as reported by Marion et al. [1], (2 scans, 128 × 2048), resulting in an acquisition time of merely 8 minutes. Such a spectrum is shown together with a 1-hour spectrum below.

This is the result of a straightforward reduction in phase cycling, and the pulse sequence is otherwise identical to the one we use for any "normal" HSQC experiment. We are sure that the experiment time could be reduced further, and our 8-minutes spectrum is certainly not a record (acknowledging where the idea originated from).

We feel that the results are highly promising for our future ventures into higher dimensions using these heteronuclear techniques on our Ω-500 spectrometer.

Sincerely,

Mikael Akke

Johan Kördel

1. Marion, D., Ikura, M., Tschudin, R., & Bax, A. (1989) *J. Magn. Reson.* **85**, 393-399.

Please credit this contribution to the account of Sture Forsén.

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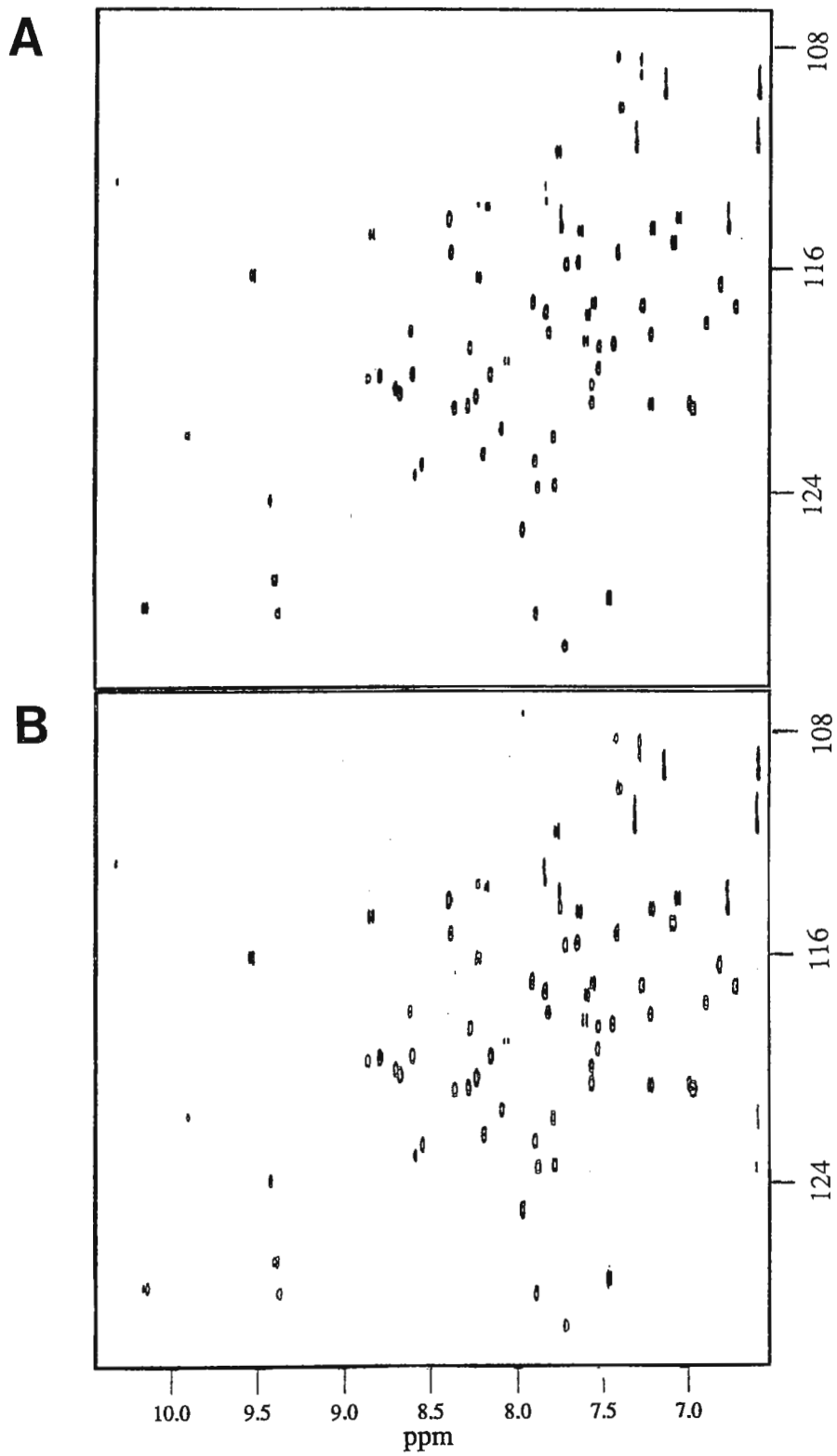

Expansions of 1 hour (A) and 8 min (B) ^{1}H-^{15}N HQSC spectra recorded on a 5mM solution of ~100 % uniformly ^{15}N labelled calbindin D_{9k}. The carrier is placed on the water frequency and the water is suppressed by presaturation during 1.2 seconds prior to the first pulse. The spectral widths are 6400 Hz in ω_2 (to avoid annoying folding of the water artefact) and 1120 Hz in ω_1. Both spectra were processed with a 30° shifted squared sine bell in t_2, zero filled to 4096 points, and a 60° shifted squared sine bell in t_1, zero filled to 256 points.

WESTERN RESEARCH INSTITUTE
P.O. Box 3395, University Station
Laramie, Wyoming 82071-3395
(307) 721-2011
January 29, 1991
received 2/1/91)

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

NMR STUDY OF PETROLEUM SOURCE ROCKS

Dear Barry:

One project that we have been involved with is the use of solid-state ^{13}C and ^{1}H NMR techniques to characterize petroleum source rocks. This work is being done in collaboration with Prof. Ron Surdam and his group in the University of Wyoming, Department of Geology and Geophysics. Presently we are characterizing samples from the Frontier and Mowry Formations in the Powder River and Big Horn Basins of Wyoming and Montana. These Cretacious rocks are source rocks for a number of oil fields in these basins.

One problem in using NMR for studying petroleum source rocks is that source rocks are typically low in organic carbon. The samples we are studying range from ~0.5 - 4 wt% organic carbon. Consequently, overnight signal averaging is usually called for. We are hoping to eventually acquire a large volume sample spinner (Zhang and Maciel, J. Magn. Reson. 85, 156, 1989), but in the meantime are using the Chemagnetics 9.5 mm zirconia rotor system.

We have noticed a number of things that improve the spectra, and are presently investigating these (Figure 1). Spectrum (a) is a CP/MAS spectrum of a Frontier shale sample having 2% organic carbon. Spectrum (b) is a CP/MAS spectrum of the sample after first washing the sample with HCl. Spectrum (c) is the sample after being subjected to hydrous pyrolysis at 280 °C. This sample had 1.9% organic carbon. Washing with HCl to remove paramagnetic impurities, and heating in the presence of water both appear to improve spectral quality. Washing the 280 °C hydrous pyrolysis residue with HCl does not appear to improve the spectral quality (spectrum d). We have noticed this behavior on other source rocks and are currently investigating the reasons for this behavior.

Thanks for your patience while we were trying to satisfy our TAMU NMR newsletter obligation.

Best regards,

Fran Miknis Dan Netzel

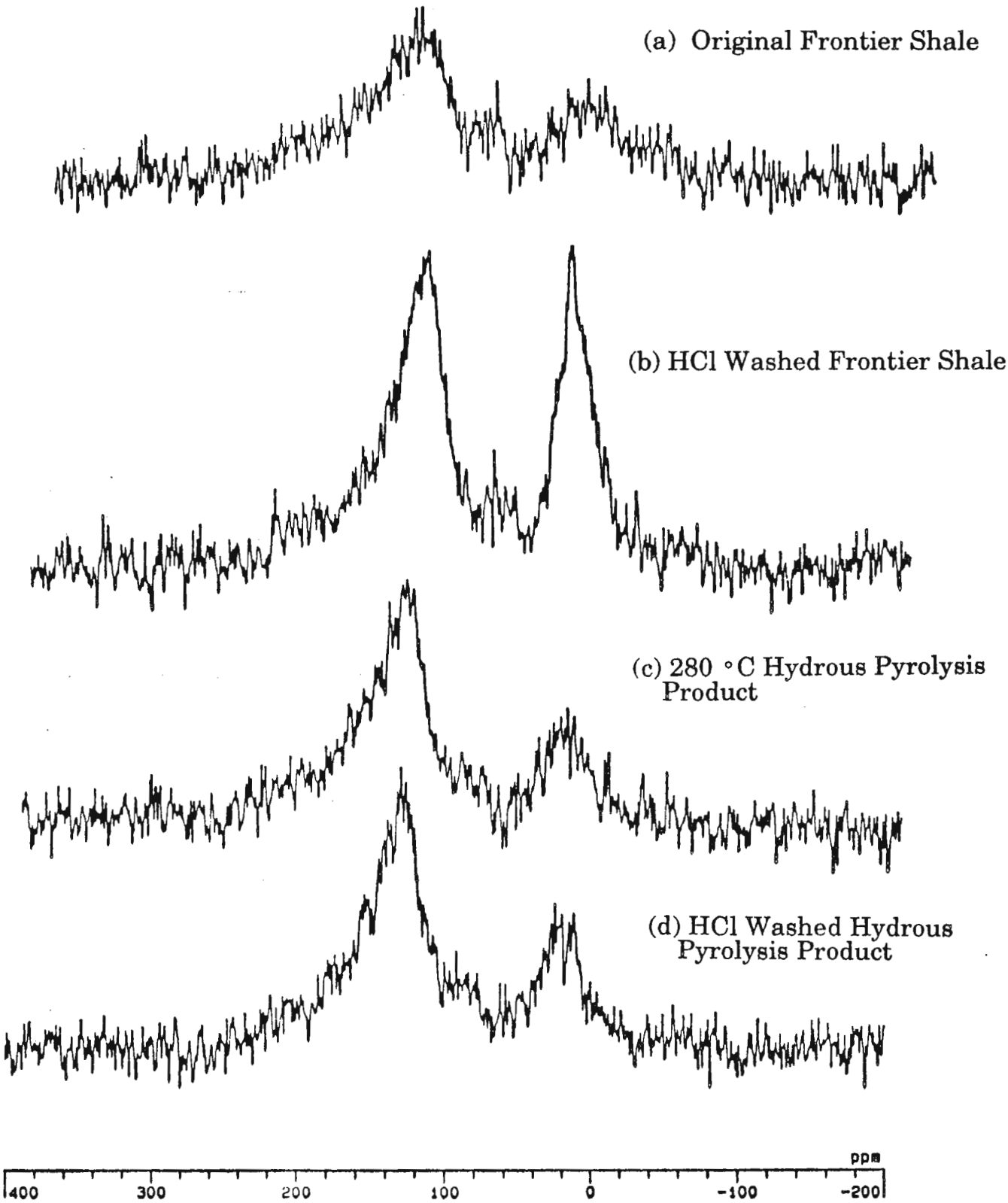

Figure 1. CP/MAS ^{1C} NMR Spectra of Source Rocks

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CHEMAGNETICS

UNIVERSITY OF DELAWARE
Department of Chemistry and Biochemistry

February 6, 1991 (received 2/9/91)

Dr. Barry Shapiro
TAMU NMR Newsletter
999 Elsinore Court
Palo Alto, California 94303

Quantitative Cross Polarization, Old Dogs Learning Old Tricks

Dear Barry,

Recently we have wanted to quantify carbon in solids. Of course, it is well known that the interplay between polarization transfer and spin relaxation makes relative-amplitude measurements in a single spectrum unreliable for quantification. The way around this impediment is to determine the full time dependence of each magnetization component, a parameterization of which gives the required amplitudes for relative quantification. We did experiments on several materials, including hexamethylbenzene (HMB), with our Chemagnetics m100S that show one must be exceedingly careful to obtain reliable data. As others who do these experiments have discovered, the Hartmann-Hahn match is critical. At optimal match, the largest HMB aromatic signal is achieved in our spectrometer for a contact time of ~8msec. With a mismatch of 1.3dB, the optimal time was in the range of 25-30msec, reflecting a substantial change of the polarization-transfer rate constant with mismatch.

This behavior was certainly expected. However, a subtler effect crept into our experiments. The probe tuning depends on repetition rate and contact time. For long contact times ($t_c > 20$ msec), the tuning for minimal reflected power is substantially different from that at shorter contact times. To obtain consistent results on a sample, we found it essential to readjust the probe tuning at each contact time to produce the same reflected power. Only under those conditions could we obtain reproducible quantitative results on known samples.

We are quite sure this effect has been noticed by many people. Indeed, one of us (CRD) recalls the need for "pre-equilibration" in multiple-pulse experiments 15 years ago to counter changes in probe tuning. However, it seems pertinent to remind ourselves and our friends that gremlins are waiting to spoil any experiment. For those who wish to do quantitative measurements, HMB is a good standard because it is ubiquitous in spectroscopy labs; since one must investigate contact times longer than 20msec, attention to probe tuning is critical. We prefer it over adamantane, which has a rather long polarization-transfer time constant, which means the probe-detuning problem will be exacerbated. Another convenient standard material is high-molecular-weight linear polystyrene, for which we get relative quantification of the sidechain and backbone carbons.

As you can see by the signatories, we have a new member of the Blue Hen NMR team. Dr. Martha Bruch has joined us in placed of Roger Crecely, who chose to "retire" last year. Her interest in NMR of biological systems and her expertise in 2-D (and higher dimensional) NMR gives us strength ia variety of areas.

By the way, for those interested in the 11th Delaware NMR Symposium, plans are under way. It will be June 4. Those on our mailing list will receive information by mail in the Spring. Those wishing to be on the list should contact either of us.

Yours truly,

Martha

Martha D. Bruch

Cecil

Cecil Dybowski

Colorado State University

Department of Chemistry
Fort Collins, Colorado 80523

Tel. (303) 491-6480
Fax (303) 491-1801

February 4, 1991
(received 2/9/91)

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

Title: Novel New Solid-State Probe Developments in the C.S.U. NMR Center

Dear Barry:

As you may already know, N.S.F. funding of our NMR facility at C.S.U. has been terminated. We are grateful to N.S.F. for twelve years of support and are naturally disappointed in the termination of support at a time when we feel we have so much to offer to users of our facility. In any case, we believe that, even without N.S.F. funding, we will be able to continue many of the activities that have characterized our facility during the past several years, although circumstances force us to be somewhat more selective in what we do. Readers who have any questions regarding our Center can call me (303-491-6480) or the Center's Manager, Dr. Bruce Hawkins (303-491-6455).

Readers may be interested in some of the recent solid-sample probe developments in the Center. Bruce Hawkins and John Stringer have successfully bench-tested a home-built DOR probe. Steve Dec has developed probes that may be of special interest to a variety of research groups, especially those studying catalysis. He has designed and made a MAS rotor, one end of which includes a rubber septum, that is capable of spinning ($\nu_{rot} \leq 5$ kHz) samples at temperatures up to 210 °C and under gas pressures up to 10 atm, with the flexibility of altering the pressurized gas in a given sample between NMR experiments. He has also designed and built a magic-angle hopper system that is capable of functioning at temperatures up to 210 °C and under static or dynamic gas pressures up to 10 atm; in this system the gas phase, which can be a flowing system if desired, can be changed *during* a given NMR experiment. We hope to have some spectra obtained on all of these new probes to show by the time of the ENC.

Sincerely,

Gary

Gary E. Maciel
Professor

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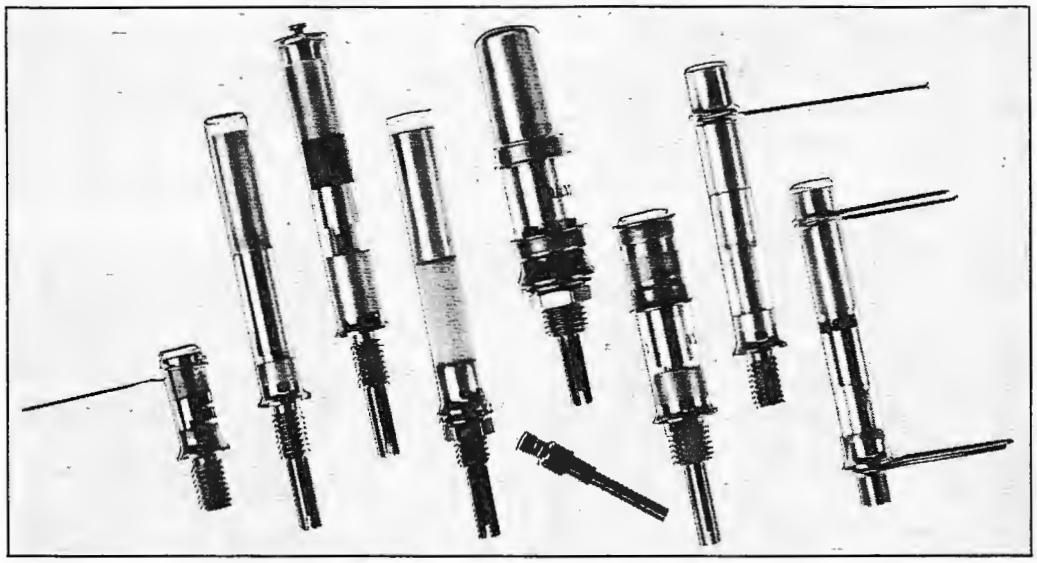

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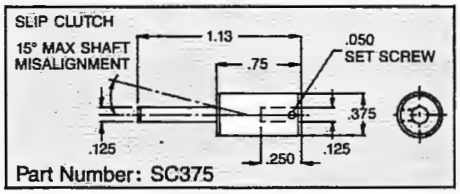

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Dear Barry,

(received 2/7/91)

NMR OF CULTURED CELLS

P.G. Scherer, J.P. Galons and R.J. Gillies; Depts. of Biochemistry, Physiology and Radiology; University of Arizona Health Sciences Center
Tucson, AZ. 85724 (602) 621-8521

For the past two years, we have been developing techniques for the growth and maintenance of mammalian cells at densities appropriate for high-field NMR. Preliminary reports of these activities have appeared elsewhere. We have used these systems to optimize growth and monoclonal antibody production of cultured hybridoma cells (Gillies et al. Magn. Reson. Med. (in press, March, 1991).

The system that we have developed is made of two components: a hollow-fiber bioreactor (HFBR) and a maintenance circuit (Fig 1). The HFBR is 25 mm in diameter and contains 1350 cellulose acetate/cellulose nitrate hollow fibers of around 0.3 mm diameter. The fibers occupy 45-50% of the available volume. These reactors are made by Microgon (Laguna Hills, CA), but only in batches of 100 or so. We have purchased 100 of these and are willing to part with them (at cost) provided they are used in an appropriate circuit. These HFBR units fit into custom 25 mm Helmholz-style probes. The spectrometer used is a wide-bore Bruker AMX-400.

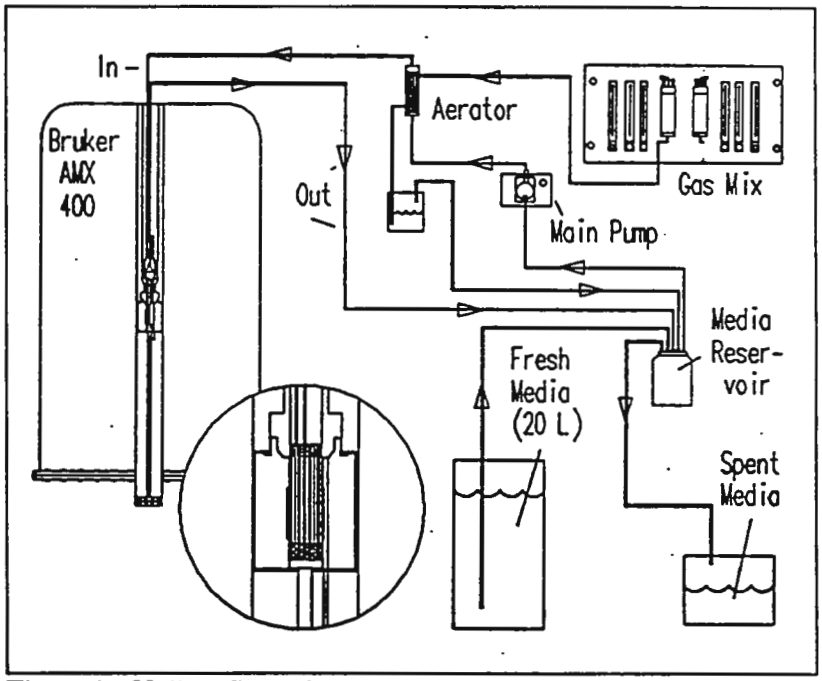

Figure 1. Hollow fiber bioreactor perfusion circuit.

The maintenance circuit contains a MAIN LOOP, which circulates media from the main vessel through a gas permeable hollow fiber oxygenator (Kinetek, St. Louis MO) through the reactor and back to the vessel. The composition of the growth medium in the main vessel is maintained at near steady state through the use of a "bleed-and-feed" circuit. Temperature is maintained by housing the whole circuit (except for the lines going to the reactor) in a homemade incubator. Temperature is sporadically monitored at various points in the circuit using homemade thermocouples and commercially available control units. We are currently working on a publication which will give these methods in detail.

These circuits have been used to grow high density cultures of hybridoma, Chinese Hamster Ovary, Ehrlich Ascites tumor and transformed 3T3 fibroblast cells. Under proper

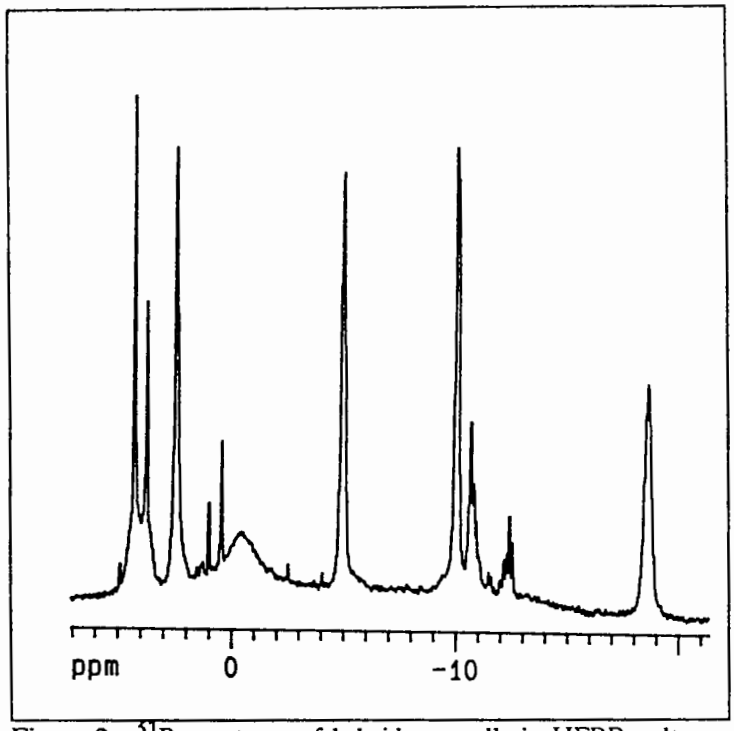

Figure 2. ^{31}P spectrum of hybridoma cells in HFBR culture.

conditions, growth can be rapid and cells can reach near-tissue densities on the shell side of the fibers. The sensitivity of spectra resulting from these cultures is striking. Reasonably good ^{31}P spectra can be generated in as little as 30 seconds. Spectra from longer accumulations reveal a number of hitherto unobserved peaks (Figure 2). The most striking aspect of these ^{31}P spectra is the high resolution which can be obtained. Even at very high cell densities, the (proton-decoupled) phosphorous linewidths are around 10 Hz. These sharp lines not only significantly improve the S/N, they also indicates that the "endogenous" linewidths of cells are somewhat less than previously thought. The resulting spectra have a quality rivaling that of some extracts.

We have also used these systems to investigate the metabolism of ^{13}C glucose in tumor cells. A significant problem with these studies is the fact that, because of sample size and coil configuration, the pulsewidths are quite long (ca. 60 usecs for a 90° pulse). This limits the efficiency of decoupling to a couple thousand Hz protons. Another major problem with these ^{13}C studies is the natural abundance peaks coming from the fibers. These carbons give both broad and sharp resonances. Therefore spectra from sequential acquisitions have to be subtracted to reveal those peaks derived from the ingested label.

Even with these problems, significant ^{13}C spectra can be obtained (Figure 3). These spectra reveal the presence of a number of glycolytic intermediates, as well as resonances in glutamate, alanine, lactate, and ribose. Since spectra can be obtained under non-saturating conditions, a number of peaks in the carbonyl region are also observed.

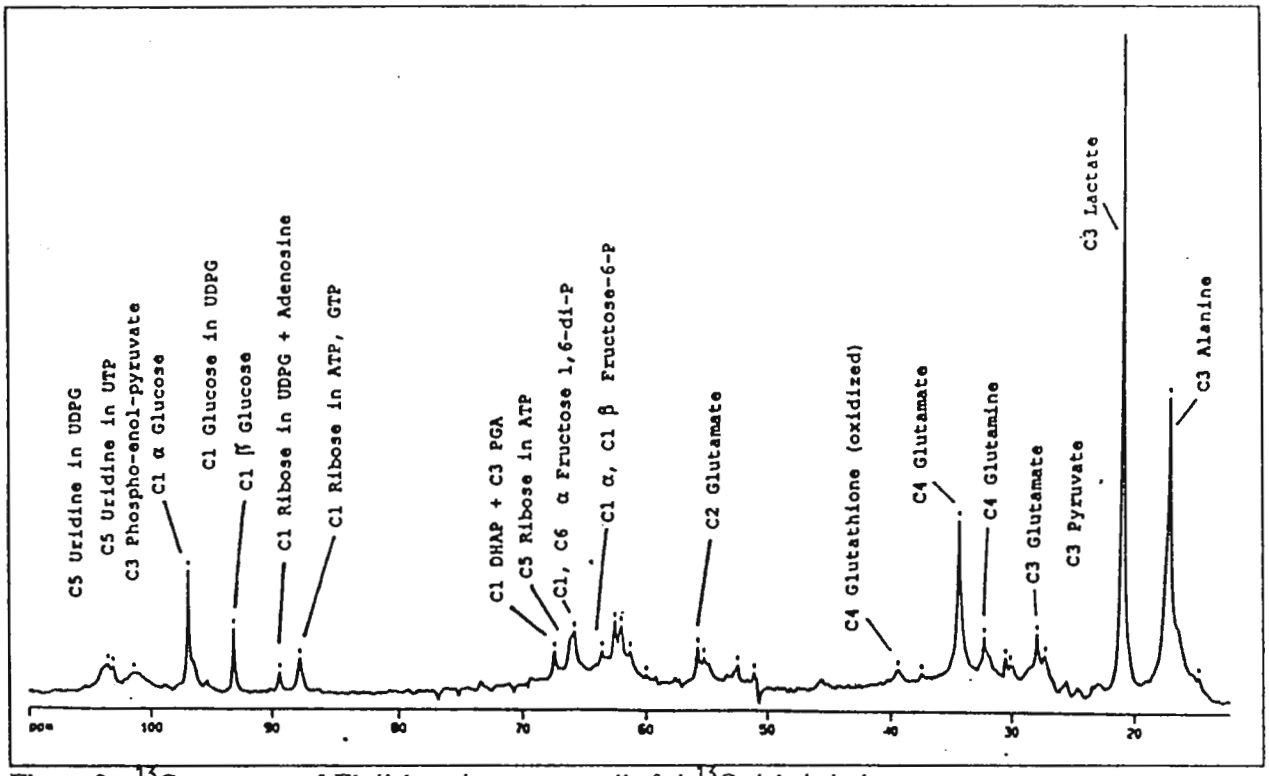

Figure 3. ^{13}C spectrum of Ehrlich ascites tumor cells fed $^{13}C_1$-labeled glucose.

POSITIONS AVAILABLE

The Biological Magnetic Resonance Facility at the University of Arizona has a number of post-doctoral positions available to investigate protein structure and cellular metabolism. Cellular metabolic studies are described above and are under the direction of Dr. Robert J. Gillies. High resolution structural studies of biologically active peptides and proteins (Biochemistry (1990) **29**, 2278) is undertaken under the direction of Dr. Neil E. MacKenzie, Dept. Pharm. Sci. (602) 626-4898.

Fremont Magnetic Resonance
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GOSSIP version 1.11 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.

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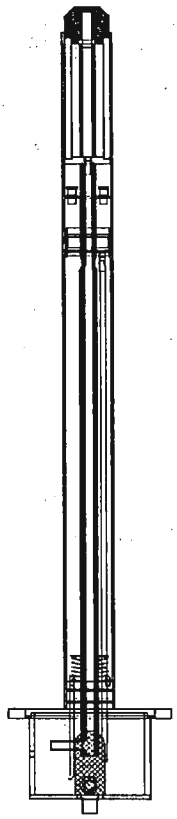

One port tuned for ^{1}H observation.

One port simultaneously tuned for both ^{31}P and ^{13}C observation.

Allows the QE console to observe ^{1}H, ^{31}P and ^{13}C spectra without tuning or switching the probe in any way.

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	^{1}H	^{31}P	^{13}C
Sensitivity	100:1	45:1	40:1
90° Pulse	10 usec	25 usec	30 usec
Resolution	0.3 Hz		
Lineshape	10/25 Hz		

Sensitivity samples:

^{1}H	0.1% Ethyl Benzene in $CDCl_3$
^{31}P	1% Trimethyl Phosphite in C_6D_6
^{13}C	ASTM (60% C_6D_6, 40% Dioxane)

Assumptions:
QE now meeting installation specifications.
2.0 dB Noise Figure.
Transmitter power at the probe:

^{1}H	100 Watts
^{31}P	150 Watts

Price List

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Heater / Sensor Assembly	$ 1,000.00
Installation	$ 2,000.00 plus travel expenses

THE ROYAL INSTITUTE OF TECHNOLOGY

Department of Physical Chemistry

Professor Peter Stilbs

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

February 1, 1991
(received 2/6/91)

Re: The Chinese Connection

Dear Barry,

Thank you for your kind "ultimatum". Everyone familiar with the JEOL FX series would agree that the weak point of these spectrometers is the obsolete data peripheral units, especially the first-generation 8" Floppy drive from PerSci. This has a mean time before (expensive) failure of a few 100 hours.

We now have great hope to keep our JEOL FX spectrometers alive for some more time after responding to an article in JEOL News 25A, No. 1; 1989 by Haolin Deng of the Instrumentation Analysis and Research Center of Zhongshan University, Guangzhou, People's Republic of China.

This article decribes a modification made at that location, in essence "replacing the PerSci floppy by an IBM PC". The connection is made through two boards, one replacing the floppy interface board in the TEXAS 980B computer, and one new board installed in the IBM-type PC. These two small (entirely TTL-based boards) are connected through a 3m ribbon cable. In addition, the JEOL software (floppy disk version FAFT72), together with a DOS *.COM-file is read into the PC from one MS-DOS compatible 5.25" floppy. The complete interface system, priced at 950 US dollars, arrived by EMS-DHL-type parcel post in a few days after order - and worked immediately!

Several advantages are apparent: 1) The modern PC disks are cheap and immensly more reliable than either the PerSci floppy or the DIABLO MHD, 2) There is "unlimited" storage capacity compared to before 3) Data are already "transferred" to the PC (data files reside as normal DOS binary files on the PC disk) - further transfer to a mainframe or workstation for further processing is easy (Kermit or whatever). In contrast, transfer from a TEXAS 980B computer requires quite some programming effort and is terribly awkward with today's standard 4) Data and pulse

Address: Prof. Peter Stilbs
The Royal Institute of Technology
Dept. of Physical Chemistry
S-100 44 STOCKHOLM, Sweden

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Electronic mail:
stilbs@sekth.bitnet
peter@physchem.kth.se

programming backup is easy 5) Since also JEOL "MENU's" now reside as normal DOS files, one can have a whole library of those on a non-active area on the PC, and copy them to the active area, when needed. In our FT-PGSE self-diffusion measurements, in particular, we really need a lot more than the normally 79 available, to cope with different conditions of measurement - without tedious reprogramming.

The unit does, however, not appear to work correctly, when coexisting with a DIABLO MHD unit on the 980B bus, although the latter is not actually active. This is so because in the case of an installed MHD, the FG/BG unit is connected through the I/O expander box to the 980B unit, so the power to the rack has to be on. The symptoms appear to be timing jitter in the FG/BG unit, causing irreproducible pulse and field gradient timing. Rebooting from the DIABLO MHD and running the applications from there removes these effects. We have not yet traced down these problems, but the DIABLO and I/O expander will not be needed anyway and will be disconnected shortly. Also, there is some IRQ conflict with an Ethernet board in the PC - one can not run DECnet PC-VAX/VMS applications or data transfer through DEC PCSA with the Chinese unit active - but we have hope to solve that shortly.

Finally it should be pointed out that the unit cannot at present be connected to an FX60 - the supplied software only works with quadrature detection spectrometers, and that Mr. Deng appears to have left Zhongshan University - correspondence should be adressed to Mr. Zhong Shizhou, in case any reader would like to purchase a unit.

Yours Sincerely

Peter Stilbs

***In Vivo* NMR Postdoctoral Position Available**

A postdoctoral position is available for *in vivo* MR spectroscopy at the MRS Unit , VA Medical Center and the University of California at San Francisco. This position is to continue the development of Spectroscopic Imaging techniques for animal studies of cerebral ischemia. The primary equipment to be used is a 7 Tesla imaging spectrometer system (Nalorac Cryogenics). Additional equipment available in this laboratory includes a 2.0 T whole body MRI/S system (Philips Gyroscan), a 2 T 30 cm GE CSI and a SLM fluorometer. Collaborative research will be carried out with Dr. Andrew Maudsley, though independent work is strongly encouraged. This position is part of an active MRS research group with a wide variety of projects in human and animal MRS.

Applicants should be familiar with computer programming, rf coil development and MR imaging. A background in MR/MRS and physics, chemistry or engineering is most desirable. Applicants should send a resume and names of references to:

Michael Weiner, M.D.
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NMR Technologies Inc.

Dr. David E. Axelson, Ph. D.
President

Head Office & NMR Spectroscopy Division
Advanced Technology Centre,
9650 - 20 Avenue, Unit 132
Edmonton, Alberta, Canada T6N 1G1
Tel: (403) 462-2798 Fax: (403) 428-5376

January 30, 1991
(received 2/6/91)

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

Magnetic Resonance Imaging of Sludges

Dear Barry,

As a complementary tool to current studies of clay-organic complexes, emulsion characterization, and the fate of tailings in heavy oil recovery operations, we have had occasion to attempt to obtain ^1H images of clay/water, clay/decalin and heavy oil tailings.

Figure 1 shows two slices taken from the middle and bottom of two vials containing kaolinite (4%, <0.2 μm)/deionized water and kaolinite in 3M NaCl. In the pictures shown here the darker areas represent longer T_2^*'s. Figure 2 gives another view of the same samples, while Figure 3 gives a schematic representation of the T_2^*'s as a function of the location within the emulsion. Instrumental limitations precluded imaging of the sludge / tailings samples we had due to the extremely short relaxation times apparently induced (in part) by high concentrations of paramagnetic impurities.

We also present these very preliminary data to ascertain whether there are other interested parties currently involved in similar or related studies.

MRI data were obtained courtesy of Drs. Peter Allen and Dan Doran (University of Alberta, Dept of Applied Science in Medicine, Edmonton) on a Bruker BNT 2.3/400 system. (multi-echo, single slice pulse sequence, 180° pulse width = 250 μs, 8 echoes, 64 phase steps, 256 x 256 element matrix, 3mm slice thickness, 10 cm field of view, TR = 3s, TE = 26 ms, gradient ~ 0.5G/cm, total time ca 30 minutes).

Sincerely,

Dave

D. E. Axelson A. Sharma R. Mikula D. Doran P. Allen
NMR Technologies Inc
Energy, Mines & Resources
CANMET/Coal Research Labs
Devon, Alberta
University of Alberta
Dept of Applied Sci in Medicine
Edmonton, Alberta

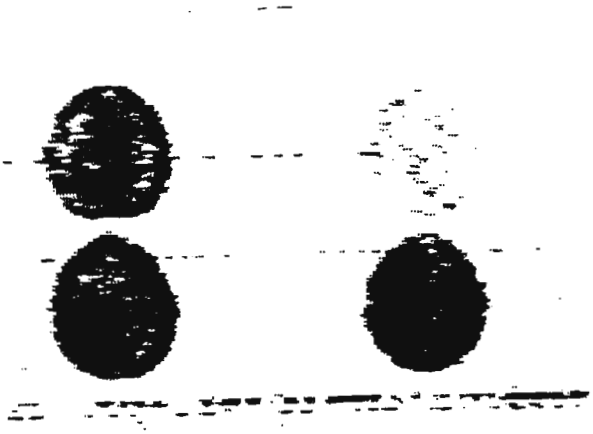

Figure 1
Slices from the middle(left side) and bottom(right side) of two vials containing kaolinite/deionized water(bottom image of each pair) and kaolinite/3M NaCl (top image).

Figure 2
Different view of same samples. Dark area at top of images is the (predominantly) water layer.

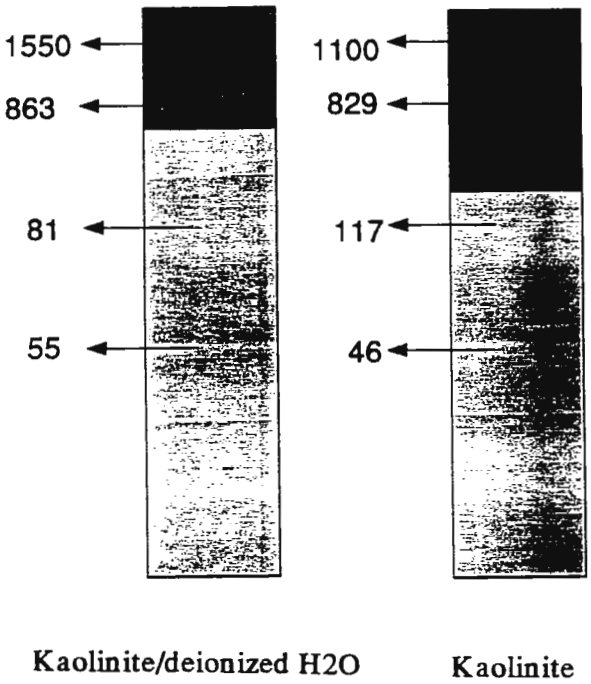

Figure 3
Schematic representation of change in apparent spin-spin relaxation times with position in vials. All values in ms.

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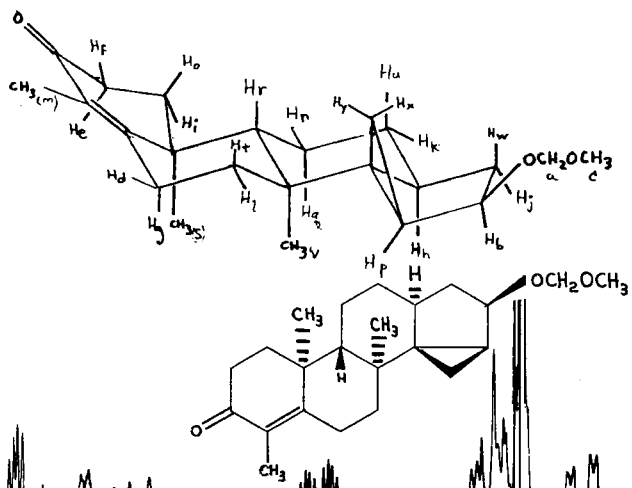

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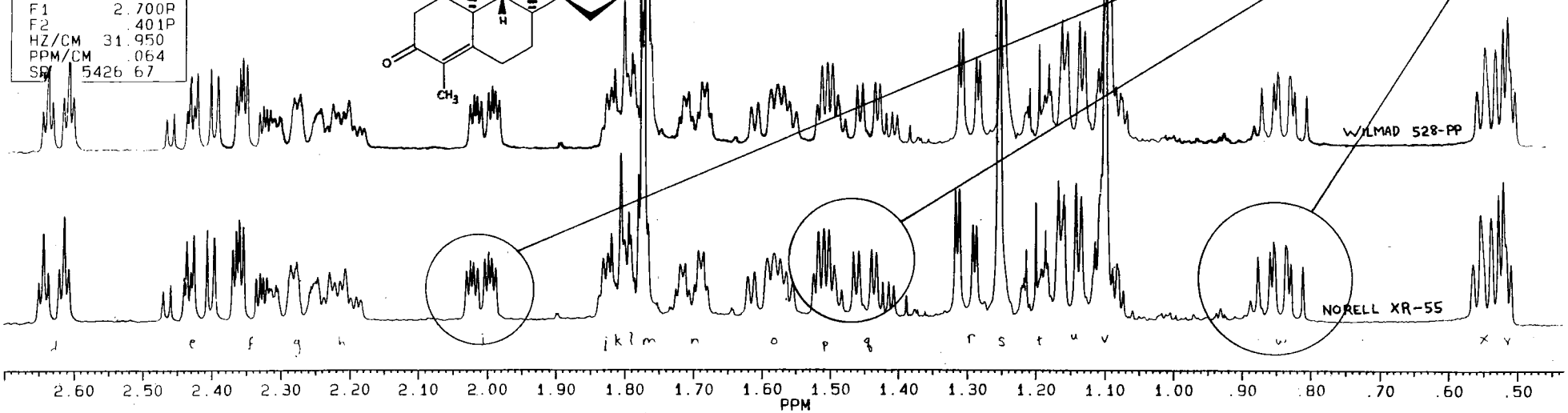

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(received 2/14/91)

To: Professor B. L. Shapiro
Editor TAMU Newsletter
966 Elsinore Court
Palo Alto CA 94303
U.S.A.

Dear Barry,

We are currently working on the solution structure of some biomolecules that are larger than those we have studied before. 3D Spectroscopy has become an important tool for our investigations. We have adopted the 3D NOE-NOE experiment (proposed by Ernst, Griesinger, Sørensen) that was first used in structural studies by the Kaptein group. We use this experiment for the determination of cross-relaxation rates of slow tumbling molecules. The idea behind our approach is, that spin-diffusion processes between 3 spin (say from **a** to **b** to **c**) can be observed and quantified using spin-diffusion peaks and back-transfer peaks in a 3D NOE-NOE spectrum. Cross-relaxation rates can be obtained from a spectrum $\tau_m^1=\tau_m^2$ (corrected for spin-diffusion) using the following formula:

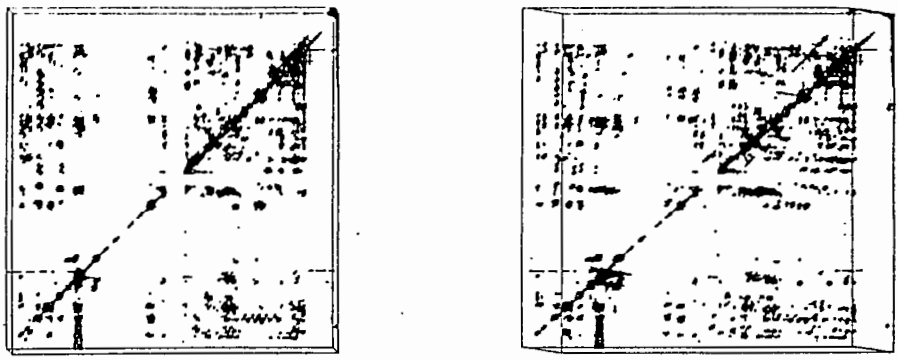
$$\sigma_{ij}\tau_m a_{ii}(0) = a_{iij} - 0.5\,\Sigma\, a_{jni} + 0.5\,\Sigma\, a_{ijn} + 1.5\,\Sigma\, a_{jin} + 2\, a_{iji}$$

We have taken a 3D NOE-NOE spectrum of a nonadecapeptide on our AMX 600 in 64 hours, with 256*192*512 data points. Processing was done on our Silicon Graphics Power station with the FELIX software of Dr. Dennis Hare (Fig.1 shows a stereo view). The evaluation of the large amount of cross-peaks is in progress.

Please credit this contribution to Prof. F. Köhler , TU München

With best regards

Horst Kessler Stephan Seip Juris Saulitis



3D NOE-NOE spetrum of Ro 09-0198

390-52

UNIVERSITY OF VIRGINIA
DEPARTMENT OF CHEMISTRY
McCORMICK ROAD
CHARLOTTESVILLE, VIRGINIA 22903

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

February 14, 1991
(received 2/16/91)

Temperature increase caused by MAS

Dear Prof. Shapiro:

Sample heating due to rf fields is well known and is often a concern during solid NMR experiments where high rf fields are used. Another source of sample heating during magic angle spinning (MAS) experiments that has begun to receive more attention is the friction caused by spinning (1,2). We have examined the effect of MAS on the temperatures of a 90% adamantane, 10% KBr sample (AdKBr) and a 50% palmitoyloleoylphosphatidylcholine, 50% water sample (POPC).

In order to measure sample temperature, we used 50% CD_3OH, 49.9% D_2O, 0.1% HCl as a chemical shift thermometer (3). The relationship between temperature and the distance between the CD_3 and OD resonances was obtained by recording spectra with a standard solutions probe. Temperatures were measured by placing a thermocouple directly into the sample after spectrum acquisition. The relationship between temperature and shift difference was highly linear from 20 to 70°C, the slope was -0.65 Hz/deg.

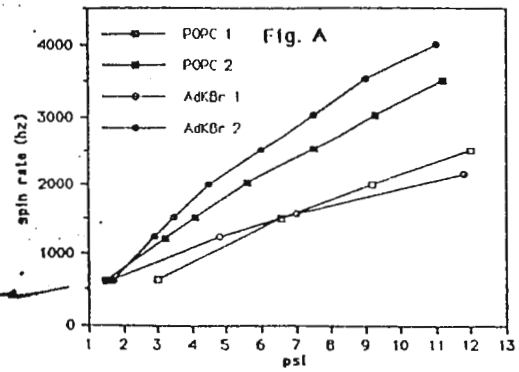
A Doty 5 mm VT CPMAS probe was used for the MAS experiments. A small container of the chemical shift thermometer solution was placed next to the sample inside the MAS rotor. Spectra of CD_3OD were recorded following a single pulse, the short pulse and very low duty cycle precluded rf heating. The spin and bearing gas were derived from a single nitrogen source and the spin/bearing ratio was controlled by adjustment of a valve on the spinner assembly. Two spin/bearing ratios were used during the experiments. Figure A shows that, as expected, a higher gas pressure was required to reach a given spin rate when the spin/bearing ratio was decreased. The temperature of the inlet gas was 21°C. Figure B shows that spinning the samples at 3.5-4.0 kHz can cause a temperature increase of about 20°C. One might wonder about the influence of the CD_3OD container inside the sample rotor. The above was repeated except that CD_3OD was added directly to the POPC sample; no CD_3OD container was present. The same dependance of temperature on spin rate was observed.

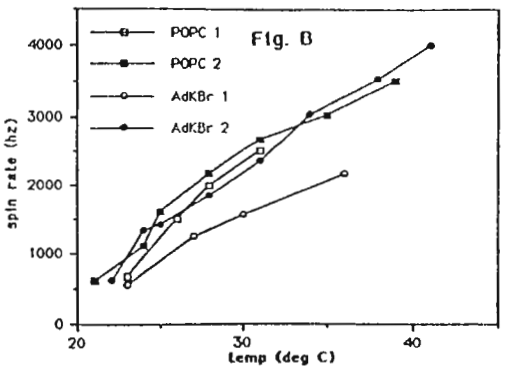
It is of interest to compare our results to other recent work. Spinning at 4 kHz caused a sample temperature increase of 7 (1) and 2.5^{o}C (2). In these studies (1,2) increases in spin rate above 4 kHz resulted in further heating. MAS heating is dependent on probe design (1), rotor material, and probably drive gas (2). We have observed that hydrated phospholipid membrane samples can be difficult to spin and, compared to samples that are readily spun, require a higher bearing and spin gas flow to reach a given spin rate. Inspection of difficult-to-spin samples after one or more hours of spinning at 2 kHz indicated that they experienced heating in excess of that described above. We now use a centrifugation technique for loading membrane samples into rotors; this technique usually yields samples that are readily spun.

The effect of MAS on sample temperature shown above and by others (1,2) indicates that if one wishes to know the sample temperature during an MAS experiment, in many cases one must place the temperature sensor very close to or preferably inside of the sample rotor.

1. Bjorholm, T. and Jakobsen, H.J., J. Mag. Res., *84*, 204, (1989).
2. Aguilar-Parrilla, F., Wehrle, B., Braunling, H., and Limbach, H.-H.; J. Mag. Res., *87*, 592, (1990).
3. Martin, M.L., Delpuech, J.-J., and Martin, G.J., *Practical NMR Spectroscopy*, Heydon and Son (1980), p336.

open symbols - lower spin/bearing ratio, closed symbols - higher spin/bearing ratio, gas inlet temp. 21°C, see letter for sample descriptions





Sincerely,

Jeff Ellena

Jeff Ellena

Laurie Kelsh

Laurie Kelsh

David H. Cafiso

Dave Cafiso

United States Department of Agriculture
Agricultural Research Service
Midwest Area
U.S. Dairy Forage Research Center
1925 Linden Drive West
University of Wisconsin
Madison, WI 53706

(608) 264-5407
E-Mail RALPHJ@VMS.MACC.WISC.EDU
January 18, 1991
(received 1/25/91)

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

Dear Dr Shapiro,

File Compression

The prospect of getting one of your dreaded Ultimatum's leads me to rush off this quick and rather cheap contribution. I shall try and get a real one off to you before the next deadline.

Archival and transfer of large data-sets in NMR is becoming more of a concern. While disks are certainly getting bigger and faster, files are also getting larger and the old adage of always being able to fill the available space holds true. Data compression can certainly help with storage and file transfer to other machines. We have recently become aware of a file compression utility for Bruker AMX spectrometers/data called 'compress', available for download via ftp on ucbvax.berkeley.edu (128.32.133.1).

There are many archival and compression programs around on a variety of machines but, as usual, these are usually much more conveniently implemented on microcomputers. On the MacIntosh™ there are a range of such Utilities, the most common being Stuffit (now commercialized as Stuffit-Deluxe), and DiskDoubler. DiskDoubler works flawlessly and seamlessly within your working environment on a Mac, and there is now even a Nubus card retailing for $129 for very speedy compression/decompression providing a rather cheap way of effectively doubling your disk size. As we back up to optical disks on the Mac (connected to NMR and Mass spectrometers over ethernet), compression on that platform is particularly convenient. We have found the following compressions using DiskDoubler:

1. A 64K FID (Bruker, ASPECT 3000, 24 bit word) file compressed by 34% (Method B).
2. A 16K FID (Bruker, ASPECT 2000, 24 bit word) file compressed by 31% (Method B).
3. A folder full of Bruker AMX files (FIDs and spectra, with associated parameter files and folders) compressed to 54% (Method B).

The AMX data structure, typical of analytical instrument files on UNIX machines, is quite an extensive directory full of parameter and data files, often themselves directories. These seemed to compress very highly, making compression of such data directories particularly useful. Needless to say, compression with these programs is exactly reversible and recreates the original file/directory for returning to the instrument if required.

Sincerely

John Ralph

John Ralph

P.S. I still have funding for a further PostDoc and for PhD students who are interested in plant chemistry with a high NMR component. Those interested in doing some good synthetic chemistry, 'playing' with NMR, 'playing' with MacIntosh computers, and think they are tough enough for the Wisconsin winters, should contact me directly.

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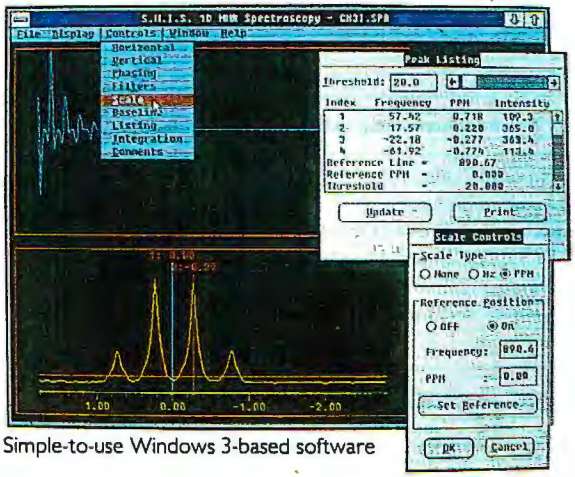

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Jerome L. Ackerman, Director of NMR Spectroscopy
Massachusetts General Hospital
NMR Center
149 13th Street
Charlestown, MA 02129

Phone: 617-726-3083
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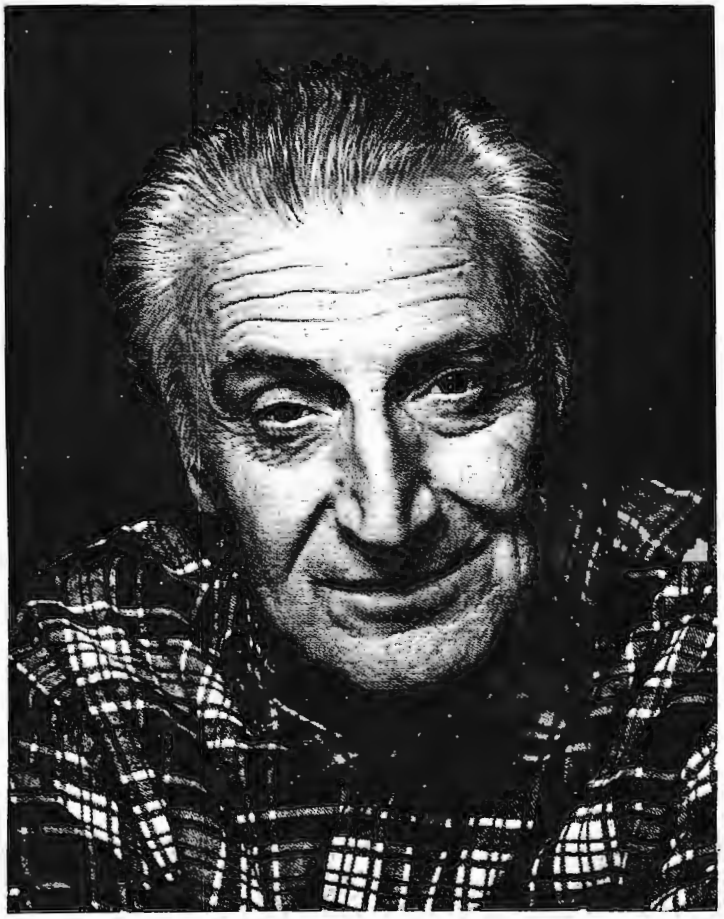
April 20, 1991



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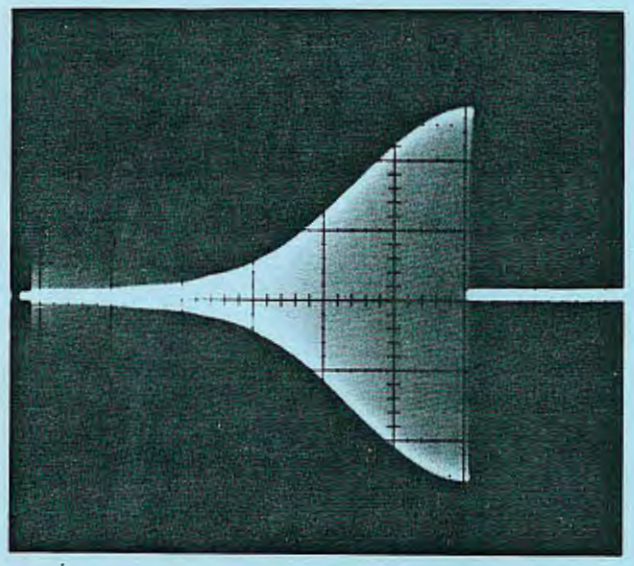
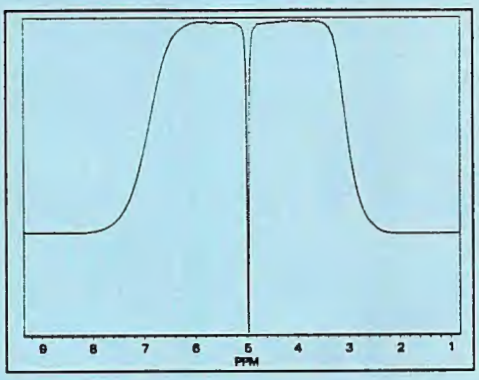
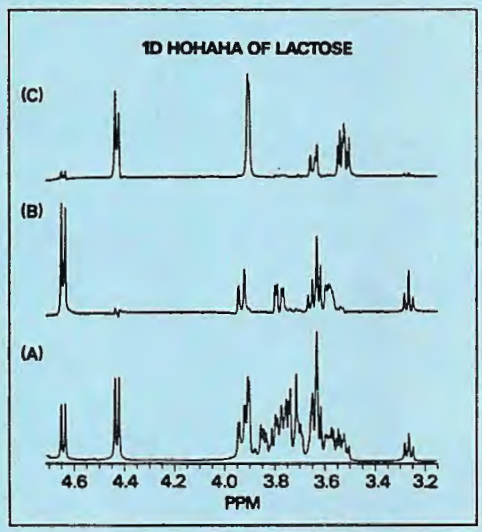


An oscilloscope trace of a half-Gaussian pulse. The pulse is defined by 250 points and the duration is 10 ms.



The result of applying a 180° half-Gaussian pulse to a sample of doped water. The water resonance has been broadened by introducing a large Z1 current in the room temperature shims. The half-Gaussian pulse width is 200 ms and the width of the "burned hole" is 12 Hz.



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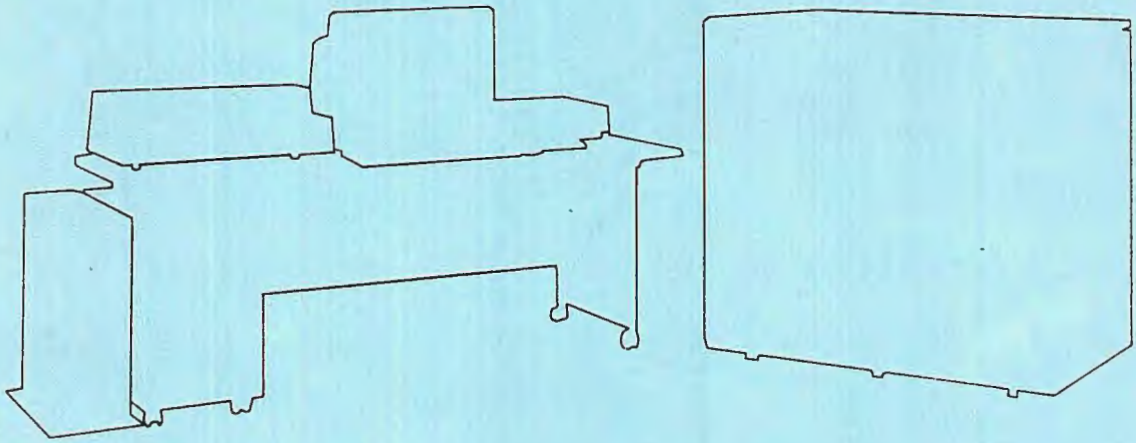

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