TEXAS A&M UNIVERSITY

NMR NEWSLETTER

No. 408

September 1992

TABLE 1 DEUTERATED SOLVENTS

Cat. No.	Description	Formula	Min. Isotopic Purity (%D)	Density (g/cm³)	MP (°C)	BP (°C)	$-\chi_v \times 10^6$ @ (°C)
D-11	Acetic Acid-d_4	CD_3CO_2D	99.5%	1.1	17	118	0.551 (32)
D-120	Acetic Acid-d_4 + TFA						
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%				

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Cat. No.	Size	Instrument Model	Type
WCV-100 (S-100A)	11" X 26"	HA-100, HA-100D, HR-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 and FT	Gridded-Two Color
WCV-XLFT-100	11" X 26"	XL-100 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"	CFT-20, FT-80, FT-80A	Gridded-Two Color
WCV-11 (XL-200)	X 17"	All Model [illegible] 16 1/2" or 16	Blank

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FORTHCOMING NMR MEETINGS

MRI in the Applied Sciences, Duke University, Durham, North Carolina, **October 25-28, 1992**; Contact: Society of Magnetic Resonance in Medicine, 1918 University Ave., Suite 3C, Berkeley, CA 94704; (510) 841-1899; FAX: (510) 841-2340.

1993 Keystone Symposia on Molecular & Cellular Biology, Taos, New Mexico: **March 8-14, 1993,** *Frontiers of NMR in Molecular Biology - III*; Organizers: T. L. James, S. W. Fesik, and P. E. Wright; Information: Keystone Symposia, Drawer 1630, Silverthorne, CO 80498; Telephone: (303) 262-1230.

34th ENC (Experimental NMR Conference), St. Louis, Missouri, **March 14-18, 1993**; Contact: ENC, 815 Don Gaspar, Santa Fe, New Mexico 87501; Phone: (505) 989-4573; Fax: (505) 989-5073. See TAMU NMR Newsletter 408, 45.

High Resolution NMR Spectroscopy (a residential school), University of Sheffield, England, **April 1993**; Organizer: Dr. B. E. Mann (Sheffield); For information, contact Ms. L. Hart, The Royal Society of Chemistry, Burlington House, Piccadilly, London W1V 0BN, England; Tel.: 071-437-8656.

Additional listings of meetings, etc., are invited.

D. E. Woessner
9204 Middle Glen Drive
Dallas, Texas 75243

August 12, 1992
(received 8/15/92)

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

Re: **Ruminations from Middle Glen**

Dear Barry:

After checking out of Mobil on July 10, I have had a little time to reflect on my first four decades in NMR. As I look back, I am amazed by the continuing growth in both theoretical and experimental NMR together with the range of physical and chemical phenomena that NMR can illuminate. NMR is the most versatile form of spectroscopy. It is also the most complex form of spectroscopy and, unfortunately, is widely un-understood and is viewed as esoteric by the lay public, especially in modern management circles. In ancient days, however, Mobil, like several other oil companies, recognized that NMR had tremendous potential in the industry. NMR in Mobil began in Dallas shortly after the early NMR papers were published by academic researchers. The potentials of NMR in finding oil and analyzing petroleum were recognized very early, as evidenced by NMR well logging patents by some early academic NMR pioneers. In fact, I was employed by Mobil to carry out research aimed towards the use of NMR to find oil. It is ironic, that, after the recent heroic developments of of **NUMAR** and **Schlumberger** to do really useful pulse NMR in the oil well bore hole, Mobil decided to eliminate NMR (both expertise and equipment) in Dallas. Since this action closes an era, I may submit to the **TAMU NMR Newsletter** a brief history of "Forty Years of NMR at Mobil/Dallas."

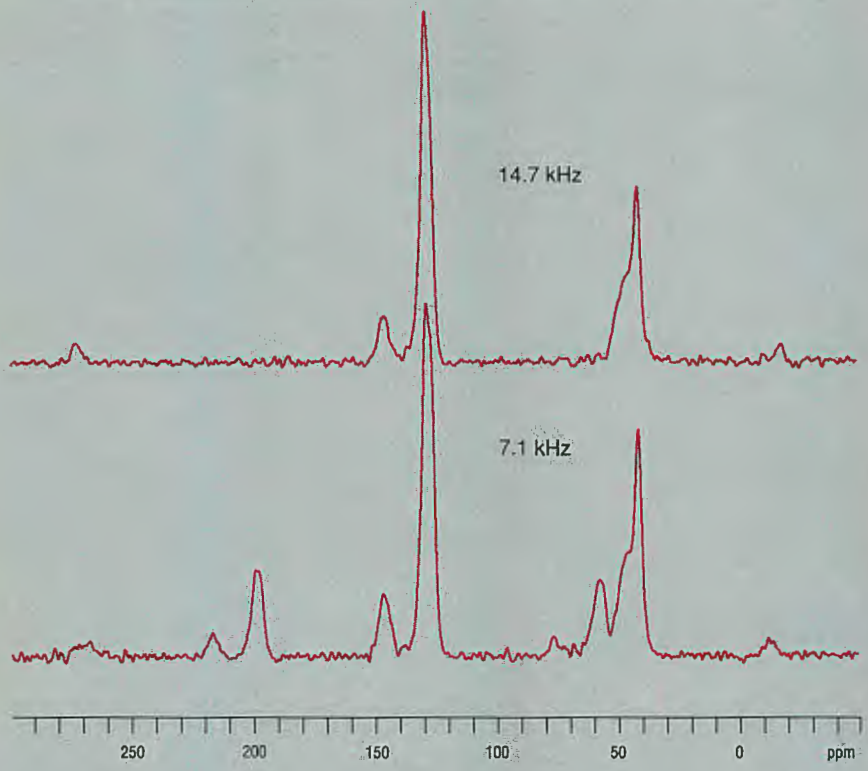
When I entered graduate school at Illinois in 1952, I had not even heard of NMR. However, when I was exposed to it, I became enchanted by it because NMR was new and also incorporated a number of areas that I had previous interests in: chemistry, physics, mathematics, and electronics. The field of NMR seemed ideal at that time, and for the same reasons it continues to be an ideal field of research. It has continued to grow. I was pleased to see in the August 1991 issue of the **Journal of Magnetic Resonance** an article entitled "Letting the Cat Out of the Bag" by Pierre Laszlo that described some of his and Paul Schleyer's experiences in NMR in the early 1960's. I was also greatly pleased to read Wallace Brey's invitation of other contributions to the Journal describing the "early days." One of the reasons that I was pleased was that I had already started writing "NMR Memoirs of D. E. Woessner" (only just over a page at that time).

It might be useful to insert the human element into NMR literature. One way to do this might be to publicize some of the historical aspects of developments in NMR, along the lines of the Citation Classics Commentaries that are published in **Current Contents**. This could be done effectively because NMR is slightly less than five decades old and almost all of the key players in the NMR game are still alive and can reveal personal and motivational details concerning NMR developments, items that rarely appear in articles in scientific journals. In fact, a knowledgeable science historian could interview NMR players and describe, in many volumes, the intellectual, inspirational, and serendipital elements in the progress of a major scientific endeavor.

Best regards,

Don

Varian introduces a 5mm variable temperature CP/MAS probe with a spinning advantage.



The above ^{13}C CP/MAS spectra of non-uniform cylindrical polystyrene beads (approximately 3mm length and 2mm diameter) were run on a UNITY®-400 MHz NMR spectrometer. These spectra were easily obtained over a range of spinning speeds, up to 15 kHz. The spectrum shows maximum signal intensity at the isotropic chemical shifts and minimum spinning sideband intensities.

Varian's 5mm VT MagicAngle•nmr™ probe for CP/MAS applications, offers an advantage in sample spinning often required to spin non-powder samples. Coupled with very high decoupling power to eliminate strong dipolar interactions for the ultimate in line narrowing, this probe is the choice for the most reliable and easy operation over the temperature range. This probe can be used with a wide or even narrow bore magnet!

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- Multinuclear operation
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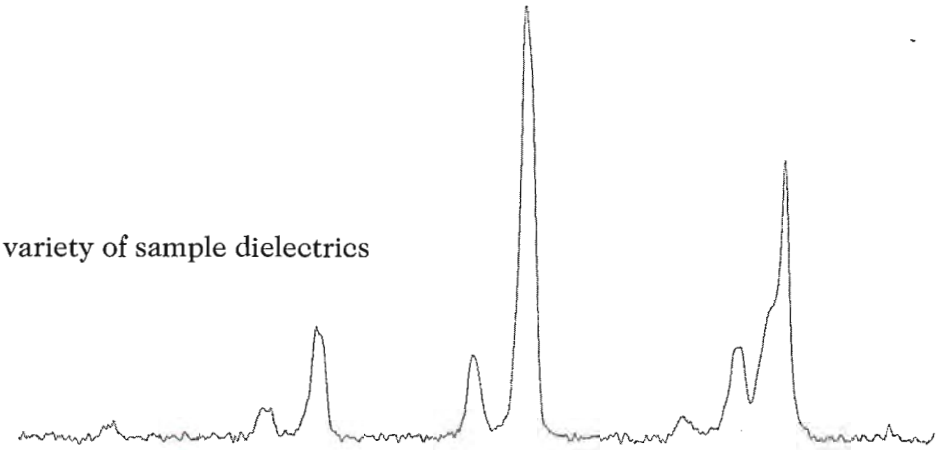

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- Computer-controlled temperature selection
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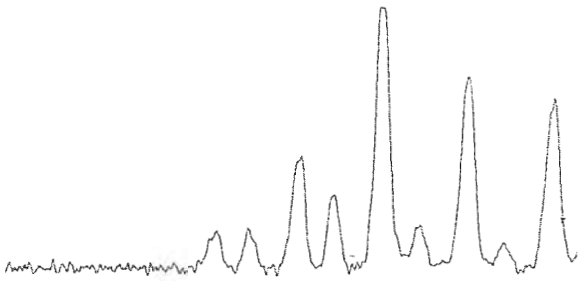
- Quick disassembly system and no-frost exterior
- Easy experimental set-up
- The ability to change sample temperature quickly



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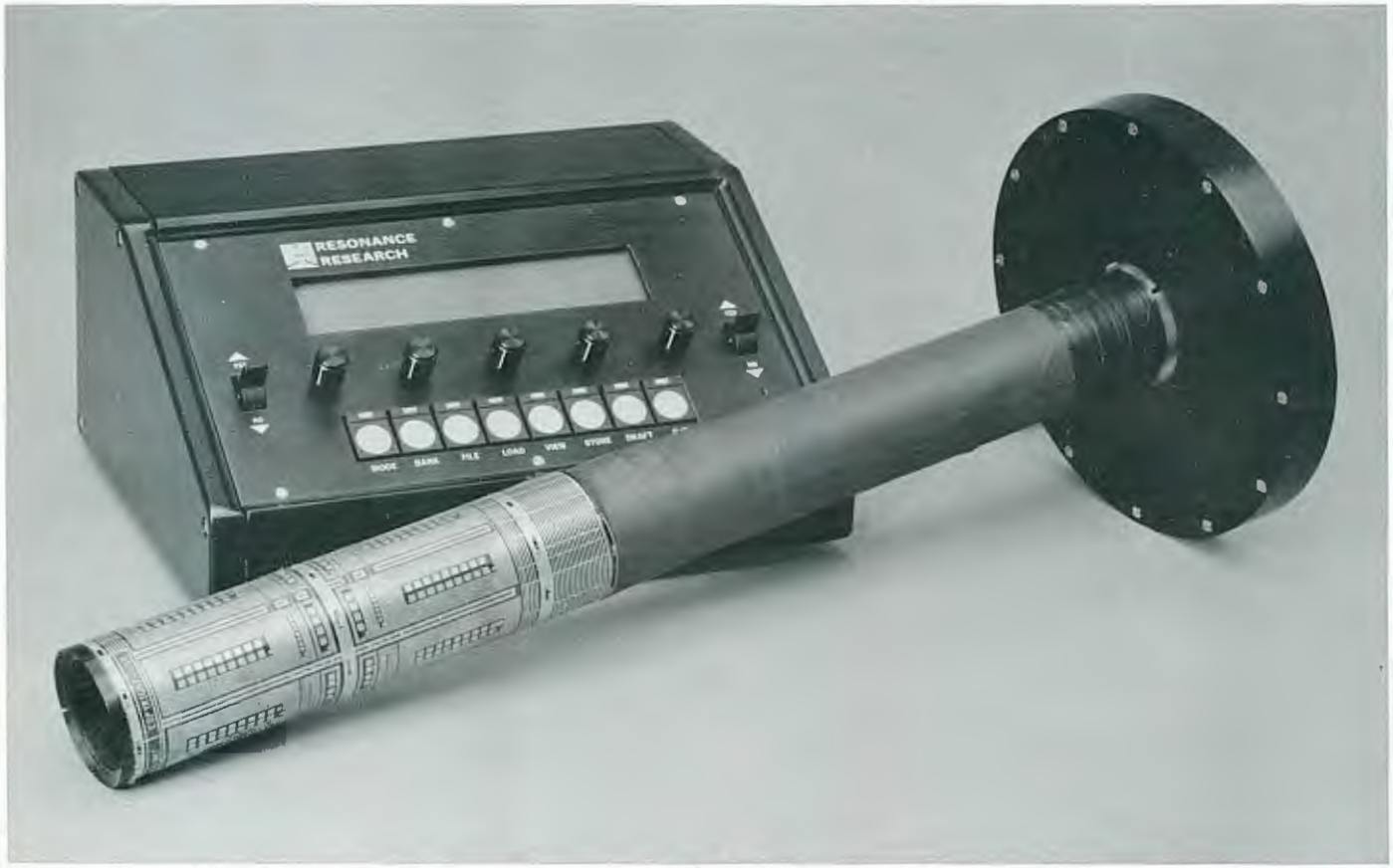
MHU-463 is capable of control up to 10th order axial and 6th order radial terms with substantially lower power dissipation and without loss of nominal bore dimensions.

The system's magnetic field correction coils require a computer controlled array of current sources supplied by Resonance Research which can be configured to operate under direct manual or external computer control.



RESONANCE RESEARCH

FEATURES

- Unmatched homogeneity volume to bore size ratio
- Industry standard shim tube dimensions
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- Field correction capability to 10^{th} order in axial and to 6^{th} order in radial components
- Low power deposition
- High dynamic range
- Capability for real-time z analog corrections (lock input)

SPECIFICATIONS

Number of channels:	48
Maximum current:	600 mA
Maximum voltage:	±30V
Cooling requirements:	Forced air flow provided
Cable:	6 meters (20 ft)

MECHANICAL (Typical Configurations)

Magnet system	**53/332**	**51/391**	**51/440**
Outside Diameter:	53.2mm	51.2mm	51.2mm
Inside Diameter (shim):		40.1mm	
Field Center to Top of Tube (minimum):		86.0mm	
Magnet Field Center:	332mm	391mm	440mm
Base Thickness:		25mm	
Base to Field Center:	370mm	430mm	480mm
Mounting Hole Pattern:			
Magnet	2 X M5 95.25 mm PCD;		
Probe	2 X M3 46.0 mm PCD		
	2 X M5 143.0 mm PCD		

(other lengths and patterns available on request)

ACTIVE PARAMETERS

Field strengths are specified for individual gradients within the family; in situations where several gradients within a given family must be run at high values, it is possible that the maximum fields may not be achievable.

The specified values assume operation within the normal range of currents and therefore are conservative. Substantially higher individual gradients are achievable within the design if required.

Additional adjustable gradients are implicit in the design but are not speciifed.

Gradient	^{1}H Hz/cm^n
z0	10000
z1	10000
z2	1250
z3	5000
z4	875
z5	250
z6	125
z7	10

Gradient	^{1}H Hz/cm^n
x / y	3250
zx / zy	2500
c2 / s2	1500
z2x / 2y	1500
zc2 / zs2	1250
c3 / s3	400
z3x / z3y	1000
z2c2 / z2s2	1000
zc3 / zs3	600
z4x / z4y	750
z3c2 / z3s2	750
z2c3 / z2s3	500
z5x / z5y	250
z4c2 / z4s2	500
z3c3 / z3s3	250

For more information contact:
Resonance Research
43 Manning Rd.
Billerica, MA 01821
Tel (508) 671-0811
Fax (508) 663-0483

in the Western US
GMW
Box 2578
Redwood City, CA 94064
Tel (415) 368-4884
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RESONANCE RESEARCH

Department of Medicinal Chemistry
Central Research Division
Pfizer Inc
Eastern Point Road
Groton, CT 06340

Central Research
NMR Spectroscopy

August 6, 1992
(received 8/14/92)

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

Dear Dr. Shapiro,

Installation of our new AMX spectrometer involved a careful assessment of factors contributing to t_1 noise in multi-dimensional experiments. Temperature control is achieved with Bruker's BVT-2000 temperature regulator, a Haake bath for controlled temperature gas, and a gas-withdrawl liquid N_2 tank as the gas source. Given the room temperature stability (measured as +/-1°C at the console over 24 hours) and the suppression ratio of the temperature controller (50:1), I was surprised to note less than ideal temperature stability (Figure 1) (observed as a change in the frequency of 10mM TSP in D_2O, which has a temperature coefficient of 7.8Hz/°C at around 30°C at 600 MHz).

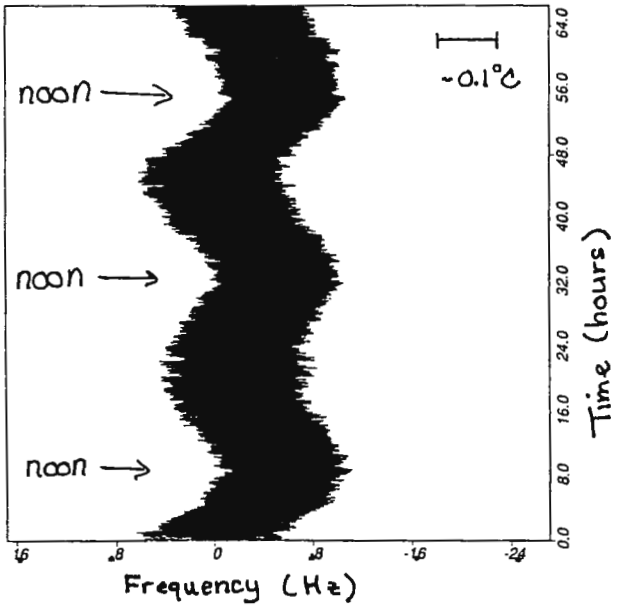
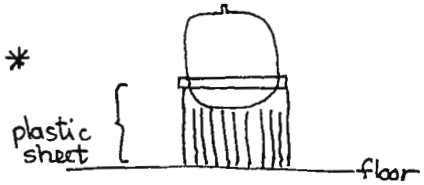


Figure 1.

Long-term temperature stability with standard set-up. Flow = 300 L/H, no skirt. Day/night variation is 0.091 °C (maximum) in 24 hours.

Stability improved dramatically with two changes in operations: an increase in gas flow (to 400 L/H) improved the short-term (minutes) stability approximately 2-fold (Figure 2), and adding a skirt to the base of the magnet improved the long-term stability approximately 5-fold (Figure 3). The idea of a magnet skirt was suggested by my colleague at Pfizer, Sandwich, Alistair Swanson, who wrapped the base in aluminum foil. I chose to use 4 mil clear landscaping plastic cut into 2-4cm strips from the floor to just below where the skirt is attached to allow for probe access. I wonder how much the spectrometer manufacturers will charge for this new 'temperature-stability enhancing modification' to the magnet??



Sincerely,

Walt

Walter Massefski, Jr.

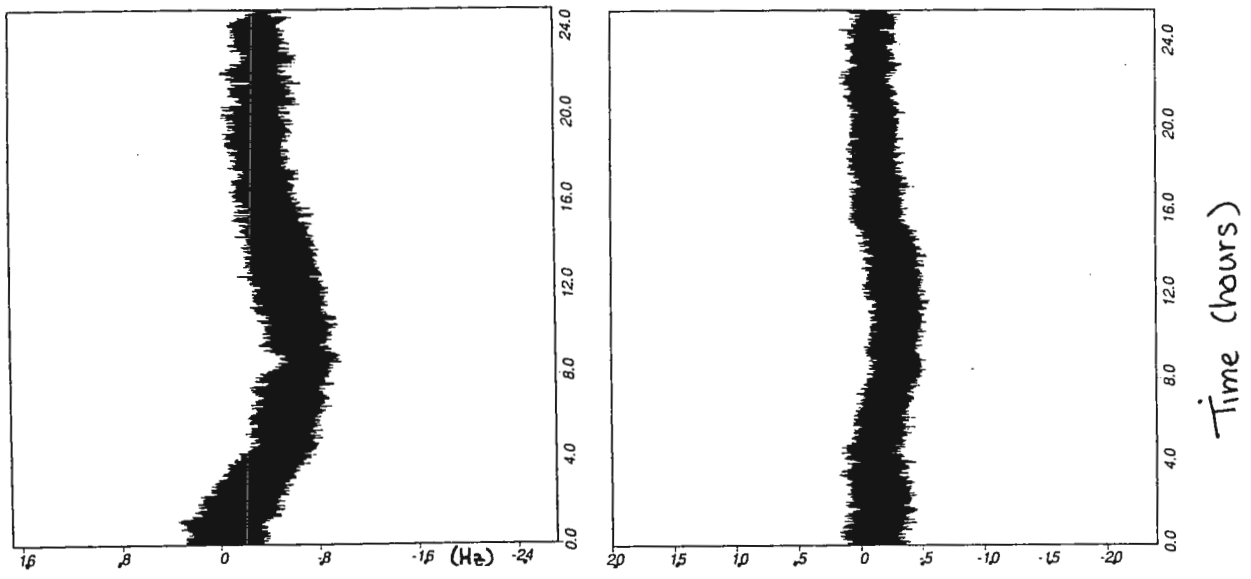
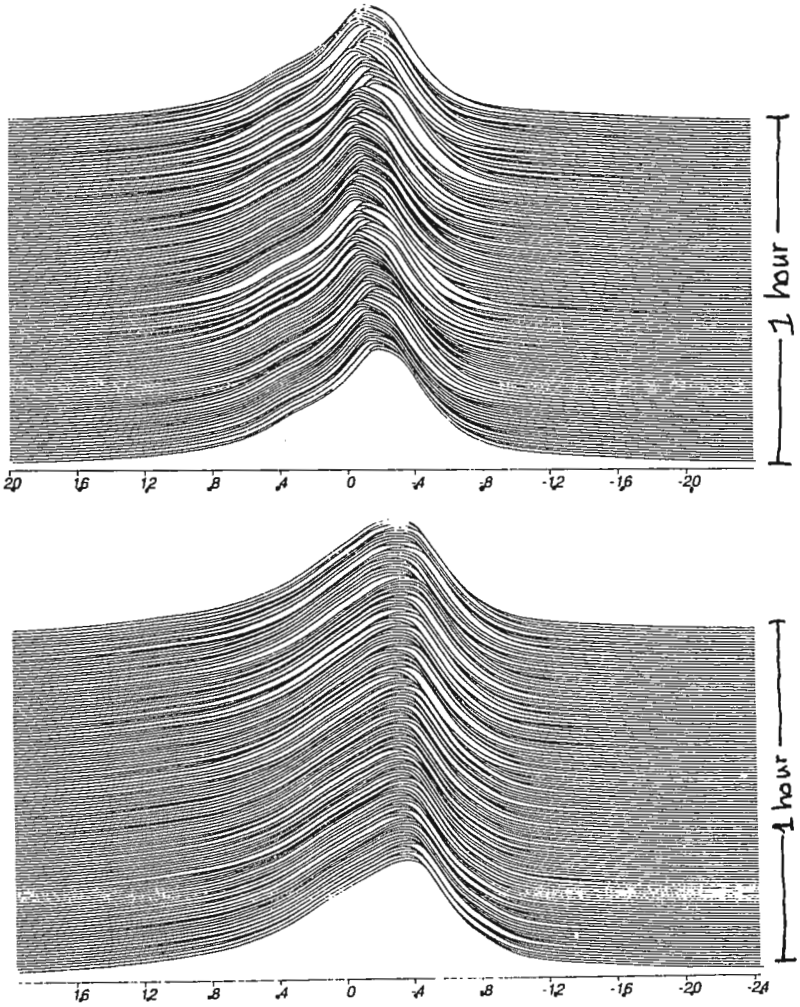


Figure 2. No skirt (left), with skirt (right). 24-hour stability is 0.091°C and 0.022°C, respectively. (Despite the numbers, the scales are the same.)

Figure 3.

Short-term stability with 300 L/H flow (top) and 400 L/H flow (bottom). Each trace is 30 seconds. Stability (maximum trace to trace variation) is 16 millidegrees and 7 millidegrees, respectively. 400 L/H flow allows a single 4400 cu.ft. N_2 tank to last for 6½ - 7½ days.



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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	

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

July 28, 1992
(received 8/3/92)

On the importance of spinning of quadrupolar nuclei

Dear Prof. Shapiro,

In our recent work about solid state structure investigations of alkaline salt/crown ether complexes via solid state NMR, especially of quadrupolar nuclei, we came upon an example that we think is worth being reported here.

Usually we measure our compounds first under static conditions as MAS averages also valuable structural information. Then in many cases we do MAS, too, to verify and sometimes improve our results. The temperature dependent ^{23}Na spectra of sodiumnitrate-15-crown-5 yielded in the whole temperature range of 200 K $\leq$ T $\leq$ 375 K a single resonance. Dipolar broadening ranged from a full width half maximum (FWHM) of 3.2 kHz (200 K) to 1.5 kHz (375 K). No sign of any quadrupolar interaction was encountered in the spectra.

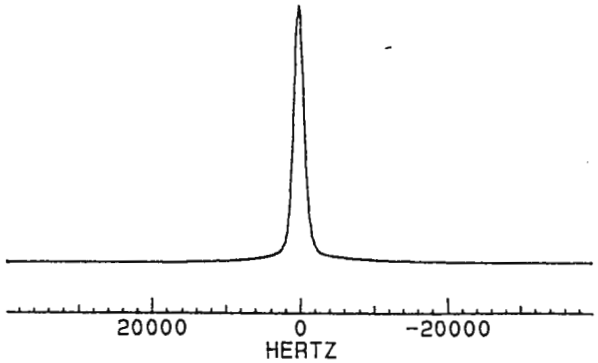
To our surprise the room temperature MAS spectrum showed spinning sidebands over the whole spectral range. Only with a spectral width of 500 kHz we could sufficiently cover the whole range of spinning sidebands of 360 kHz (this is not an unusual value, the quadrupole coupling constant of pure sodiumnitrate is 336 kHz). The static and MAS spectrum recorded at ambient temperature depicted in the figure demonstrate this effect. The pattern of the spinning sidebands reflects the line shape for a I. order quadrupolar perturbation. The splitting of the sidebands is due to spectral folding, as the depicted MAS spectrum was recorded with a spectral width (SW) of 100 kHz.

Relaxation rates of central transition and satellite transitions are known to be different. So probably this behaviour can be explained by different relaxation rates that are affected unequally by MAS.

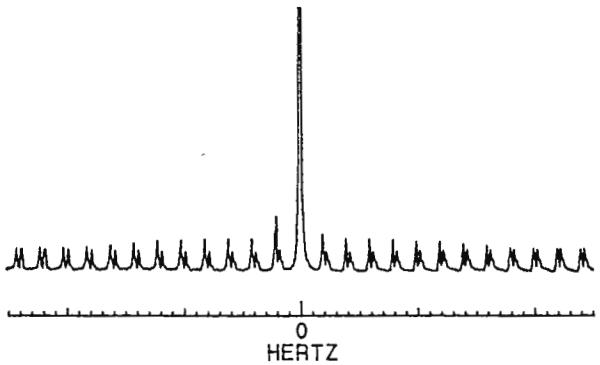
Sincerely yours,

P. Burkert T. Pietraß

$NaNO_3$-15-c.-5, static (SW 80 kHz)



$NaNO_3$-15-c.-5, MAS, 4 kHz (SW 100 kHz)



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Prof. Dr. R.R. Ernst

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Postadresse:
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ETH-Zentrum
8092 Zürich
Switzerland

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

Zürich, July 29, 1992
(received 8/3/92)

NMR OF TRAMWAYS

Dear Barry,

I am sure that during the past 30 years, we have inadvertedly publicized the Zürich tramway system even more frequently than the tourist office has. All our published 2D spectra, so far, carried the trade mark of the streetcars, in the form of excessive t_1–noise, provided they have been recorded between 5 am and midnight. The site of downtown ETH is known to be one of the worst and instable magnetic environments in the world, all due to our beloved tramways that still run on dc. Indeed, we could easily keep track of all streetcars starting and stopping anywhere in Zürich, just by recording NMR spectra.

At the beginning of 1992, we became the lucky owners of an AMX 600 Bruker spectrometer. As expected, stability was far from ideal and the old problems seemed to perpetuate. We were disappointed and even considered to discontinue working in high resolution NMR.

A few weeks ago, the situation has changed dramatically. Bruker replaced the traditional field–frequency system, based on analog circuits, by a brand new digital lock system. We expected some improvement but basically remained pessimistic. However, the results obtained surpassed even our fondest dreams. The stability of the magnetic field has indeed improved by at least a factor 30.

The figures demonstrate the achieved stability improvement by recording ^{13}C sidebands in the proton resonance spectrum of 3% chloroform (with natural abundance ^{13}C) in acetone–d_6 with centerband suppression by a difference experiment. The two–scan pulse experiment taken at an interval of 60 sec used the sequence $(\frac{\pi}{2})_{1H}$–τ–$(\frac{\pi}{2})_{13C}(\pm\frac{\pi}{2})_{13C}$–$\tau$–$(\pm\text{acquisition})_{1H}$. The subtraction of the two FIDs should, in the ideal case, lead to complete suppression of the centerband, retaining only the ^{13}C–satellites. A series of 400 such experiments was performed between 6 pm and 7.30 am the following day. Every fourth trace is displayed in stacked plot mode by means of the program package UXNMR–P with a recent expansion supplied by Dr. Beat U. Meier and coworkers, Spectrospin AG. Figure 1 demonstrates the performance of the old analogue lock system. While at night, the centerband suppression is below 30%

FIG. 1

old analogue
lock system
(BSN-20)

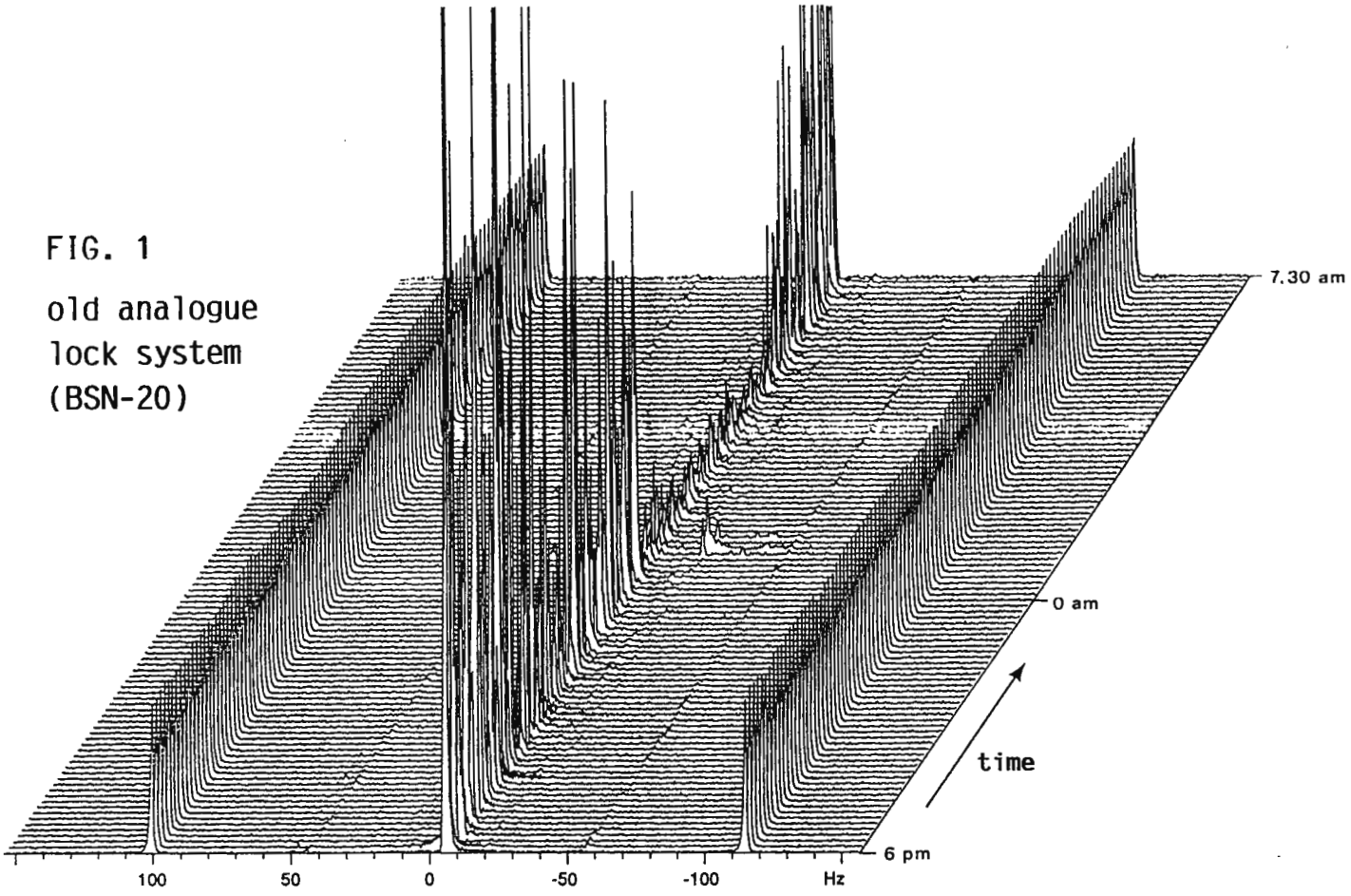
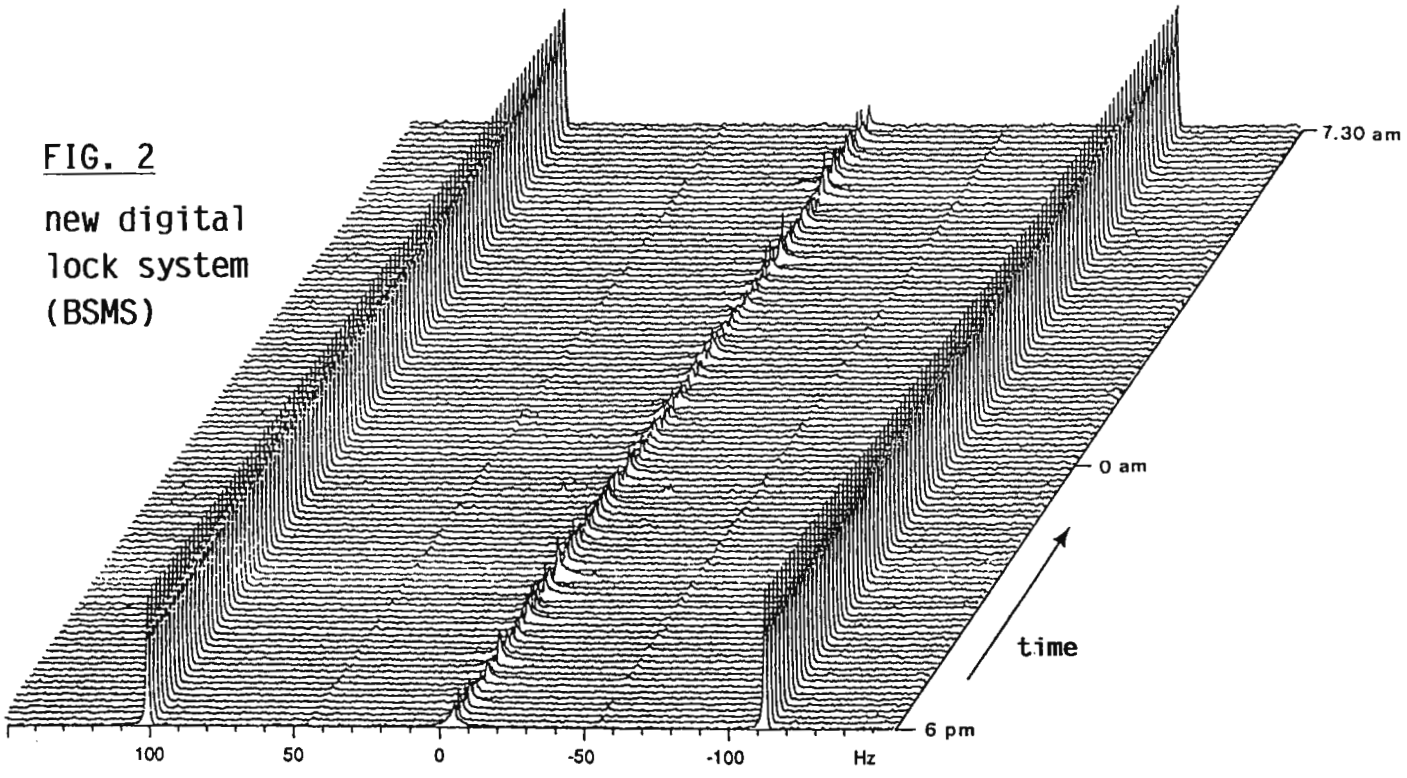


FIG. 2

new digital
lock system
(BSMS)



of the sidebands, at daytime the centerband can exceed the ^{13}C–satellites by up to a factor 6 due to magnetic field variations of ±5 μTesla. This is obviously fully unacceptable for HMQC experiments. On the other hand, figure 2 reveals the performance of the new digital lock to be by more than an order of magnitude better. The centerband, even at daytime, never exceeds 25% of the satellite intensity. Now we are back in business.

The Bruker AMX 600 spectrometer with its new digital lock is probably the most stable NMR system in the world. Of course, one can still run into problems when the lock signal is very weak and a compromise between the lock stability and its own noise contribution at high lock gain has to be made. But this happens now at signals one order of magnitude lower than before. Indeed, we are quite happy and grateful to the Bruker engineers on the occasion of this victory over the Zürich tramway system.

Best regards.

Sincerely yours,

Th. Schulte-Herbrüggen

Thomas Schulte–Herbrüggen

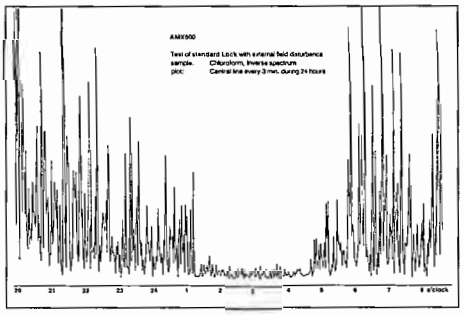
Richard

Richard R. Ernst

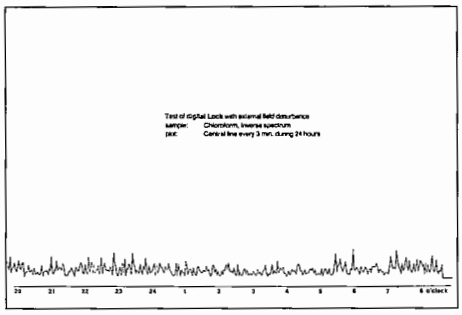
The Digital Lock

A Major Breakthrough in NMR Technology by BRUKER

The new BRUKER digital lock system is the first significant advance in lock technology in the last 20 years. The improvement in stability is comparable with the S/N improvement achieved with the introduction of FT spectroscopy. The unique performance is based on a completely new approach, starting with the Bloch equations and ending with the consequent application of state-of-the-art digital regulation theory.

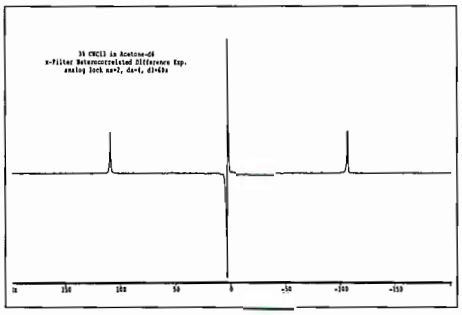


Interference source: Streetcars in Zürich generate magnetic disturbances which cause intolerable noise in a long-term stability test.

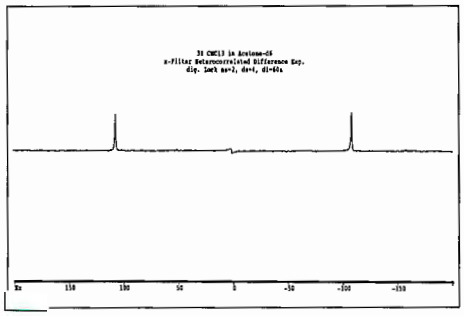


BRUKER's digital lock provides an elegant solution and guarantees silence even in the rush hour!

ANALOG → **DIGITAL**



The magnetic interference results in poor central peak suppression in an inverse H,C experiment with chloroform.



Suppression is significantly improved with the digital lock system.

The digital lock system is just a part of BRUKER's new smart magnet control system, the BSMS. It provides a solution for one of today's most difficult problems — magnetic field stability, not only in the presence of severe magnetic disturbances. The digital lock improves spectrometer stability in general and reduces the influence of the surroundings and lock signal parameters such as relaxation time, line shape and signal-to-noise.

BRUKER

For Ultimate NMR Performance

To match the new level of quality which the digital lock provides for our high-performance spectrometers, additional new features have been encorporated in the BSMS, using the most advanced electronic technology.

Ultra-High Stability Current Sources

The current sources for the BSMS have been completely redesigned. With 18-bit DAC resolution, shim currents can be set in 120 nAmp steps. Drift is nearly unmeasurable, providing the stability that is essential for 2D or 3D experiments lasting hours or even days.

User Interface with Alpha-Numerical Display

The new, extended Keyboard/Display Panel provides for a real dialog between the user and the BSMS. Various menus allow a wide variety of configuration changes and calibration. A special menu for maintenance and service helps to make the system completely transparent.

Intelligent Sample Control and Magnet Maintenance

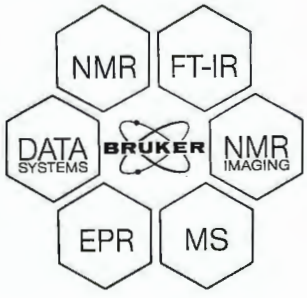
An intelligent regulation algorithm for the pneumatic sample spinning control guarantees the ultimate in spinning stability and accuracy. The He and N_2 cryogen levels are under permanent surveillance and can be displayed whenever desired.

Modular Service Concept

The well-designed modular structure of the BSMS allows very fast and efficient service of the unit. Every component, shim current source or power supply, is a plug-in subunit and can be exchanged in seconds.

BSMS Bruker Smart Magnet Control System

available as an accessory for the AMX and ARX series.



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

August 6, 1992
HvH 92-A-148
(received 8/10/92)

Chemical Shift Anisotropy of Anomeric Protons in Carbohydrates

Dear Dr. Shapiro:

While seeking to measure long-range ^{1}H-^{13}C *J*-couplings in various carbohydrate systems we recently utilized a ^{13}C-ω_1-half-filtered {^{1}H,^{1}H} ROESY experiment [1]. In the resulting spectra, we noticed consistently a significant difference in intensity between diagonal peaks that constitute ^{13}C-coupled ^{1}H multiplets, particularly for those of the anomeric protons of the glycosyl residues. This difference in intensity is due to differential relaxation of the ^{1}H magnetization components, originating from the interference of two relaxation mechanisms, namely ^{1}H chemical shift anisotropy (CSA) and ^{13}C-^{1}H dipolar interaction.

In order to quantify this effect we measured the relevant cross-relaxation rates on a mixture of α- and β-glucose that are selectively ^{13}C-labelled (>98%) at the anomeric carbon (C1). We used the pulse sequence proposed by C. Dalvit [2]. The anomeric regions of the spectra obtained at different mixing times (50, 100 and 220 ms) are shown in Fig. 1. The antiphase multiplets in the right-hand column are pure effects of cross-correlated cross-relaxation between CSA of the anomeric protons (H1) and dipolar interaction between H1 and $^{13}C1$. From the measured cross-relaxation rates we obtained chemical shift anisotropies $\Delta\sigma^{\alpha}$~–6 ppm and $\Delta\sigma^{\beta}$~–3 ppm, for the α- and β-anomeric proton, respectively. In our calculations we assumed the one-bond ^{1}H-^{13}C *J*-couplings to be positive and the ^{1}H chemical shift tensor to be axially symmetrical with the principal axis along the C1-H1 bond. A detailed account of our findings will appear elsewhere.

Sincerely,

Leszek Poppe

Herman van Halbeek

[1] G. Otting and K. Wüthrich; *Quart. Rev. Biophys.* **23**, 39-96 (1990).
[2] C. Dalvit; *J. Magn. Reson.* **97**, 645-650 (1992).

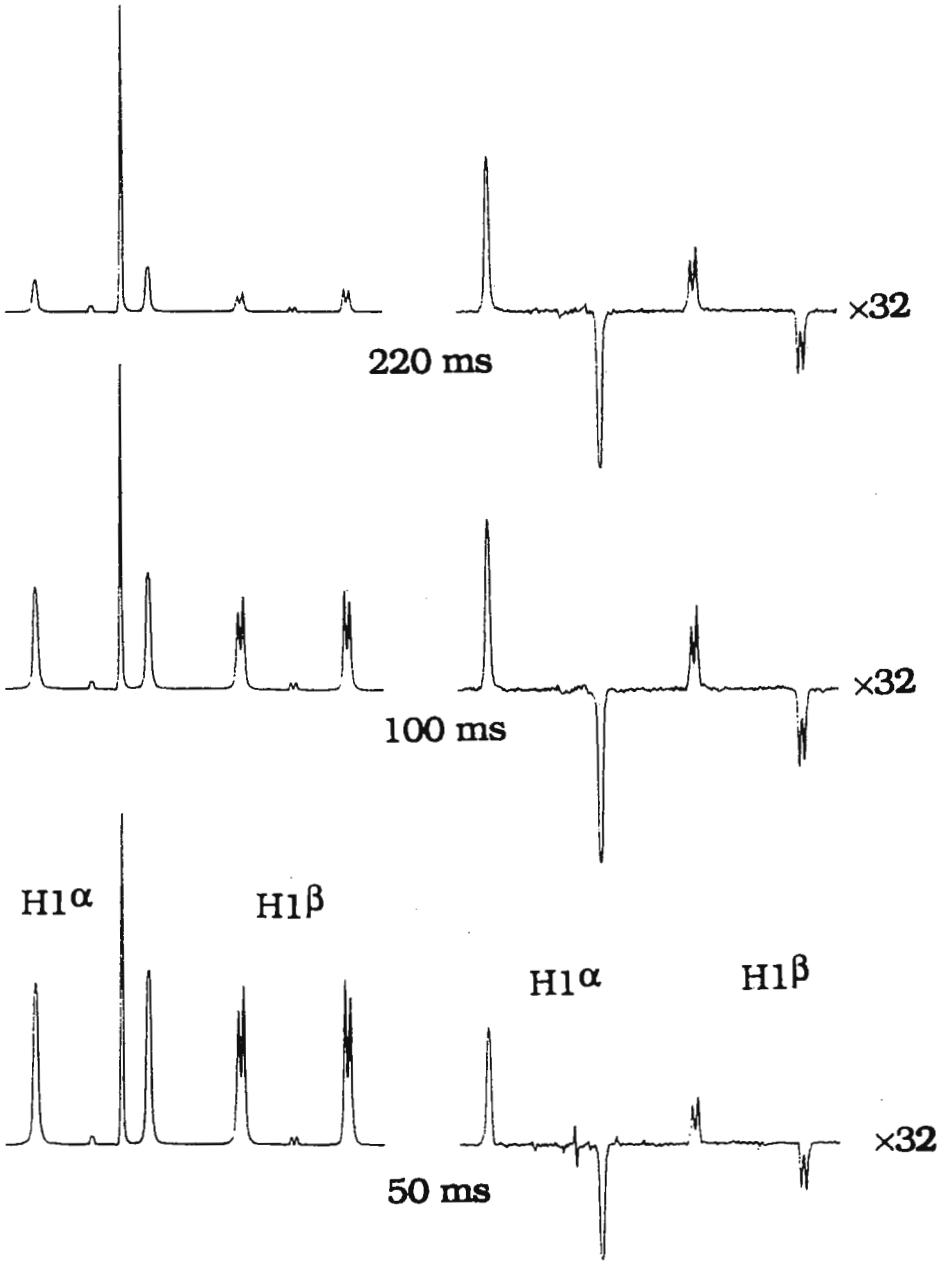

Fig. 1. **(Right)** Anomeric regions of the ^{1}H-NMR spectra of [^{13}C1-]glucose in D_2O recorded at 500 MHz (Bruker AM-500) with the pulse sequence published by Dalvit [2]. The mixing times were 50 ms (**a**), 100 ms (**b**) and 220 ms (**c**). **(Left)** Reference spectra showing the H1 signals for the α- and β-anomer of [^{13}C1-]glucose after spin-locking.

Structure Elucidation

CSEARCH

Offers identical presentation on Sun, Silicon Graphics, or VMS

Reads ^{13}C NMR peak tables directly

Confirms calculated with measured spectrum

Finds structurally related compounds by spectral matching

Calculates spectra for compounds not in the database

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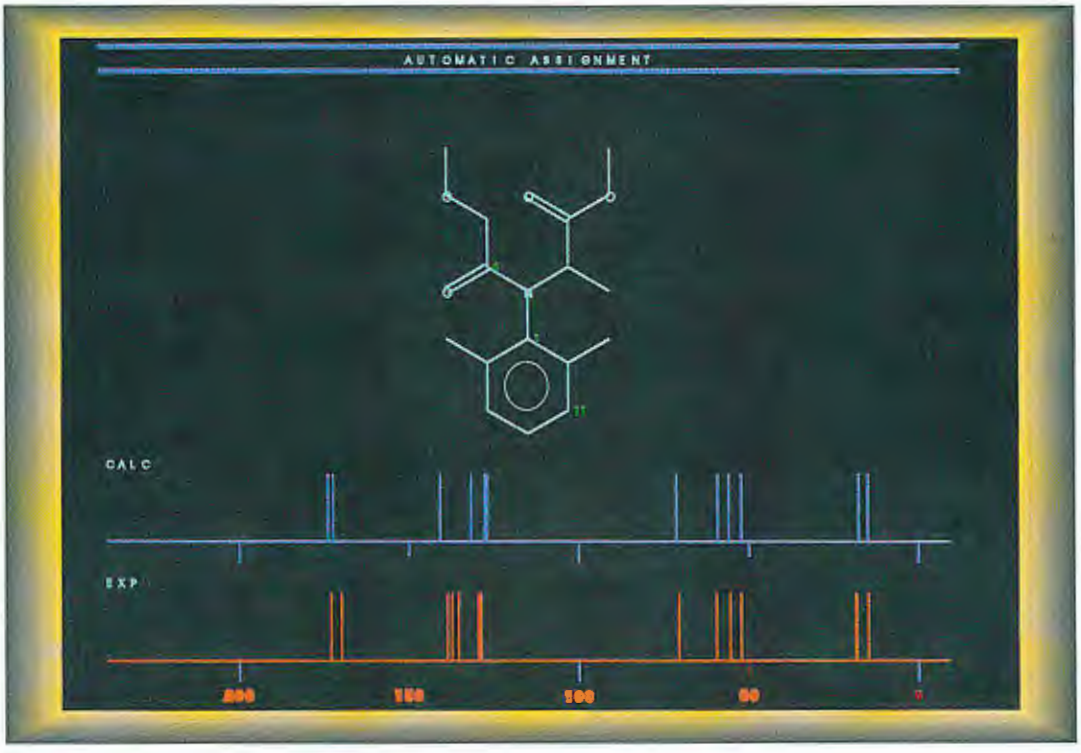
Comparison of the calculated spectrum of a proposed structure with its measured spectrum. The intelligence of CSEARCH is demonstrated by favorable comparison for a structure not contained within the database of 52,000 spectra.



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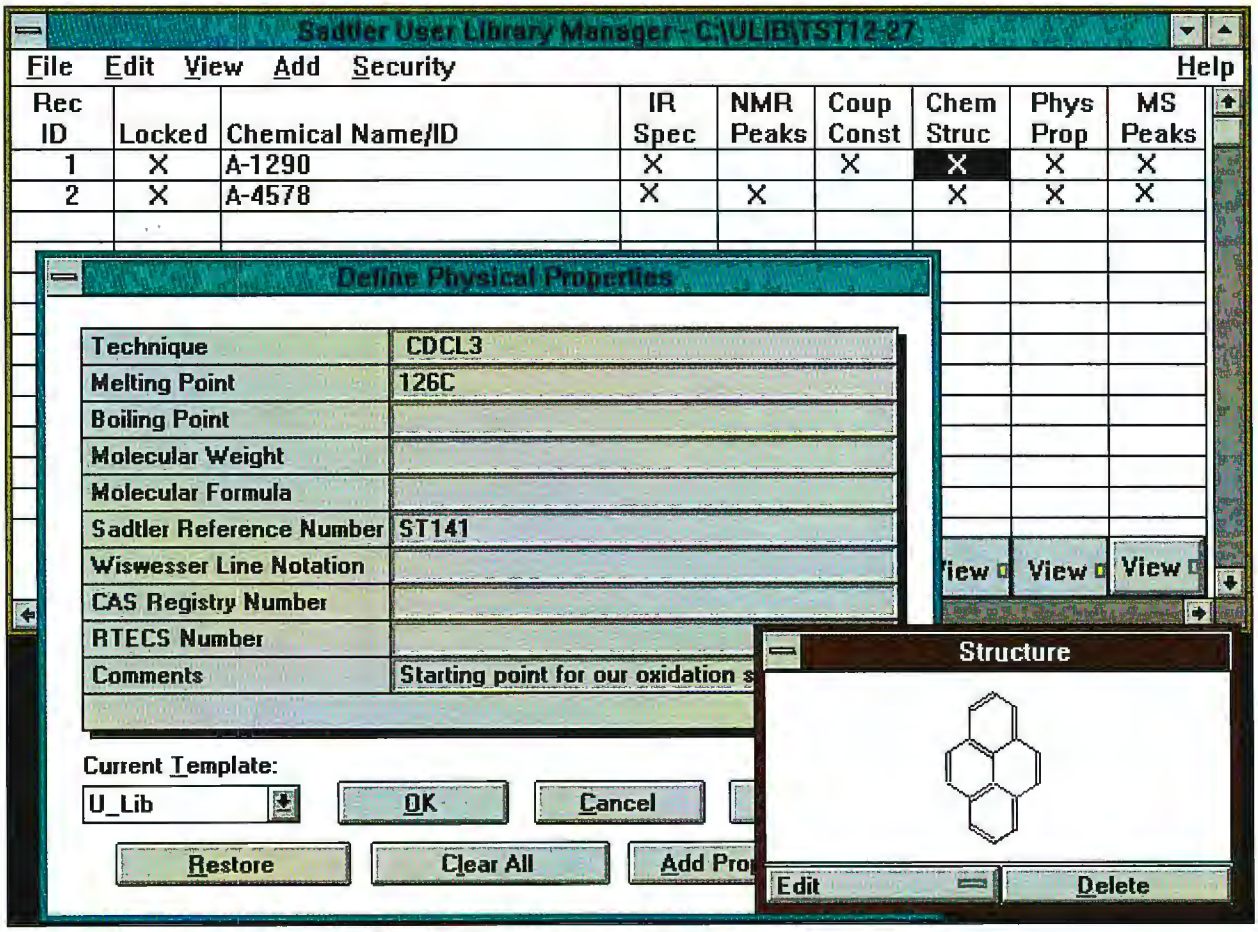

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August 6, 1992 (received 8/14/92)

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

DEUTERIUM-PROTON CROSS-POLARIZATION

Dear Barry:

Most practitioners of deuterium NMR (with few recent notable exceptions[1-4]) have concentrated on deciphering the subtleties of dynamically modified Pake patterns. We recently decided to take **the road less traveled by** and try a series of deuterium NMR experiments involving magic angle spinning, cross-polarization and preliminary "REDOR-like" experiments.

We have examined the deuterium spectrum of a sample of 90% randomly deuterated glycine using a variety of MAS experiments. As expected, one easily obtains deuterium rotational echoes (Fig. A). What we think is much more exciting is **the observation of cross-polarization between protons and deuterons.** The FID from a ^{1}H-^{2}H CP/MAS experiment, obtained using a Chemagnetics CMX 300 system and probe, is shown in the accompanying figure B. Spin temperature alternations was used; no signal was observed following a proton 180° pulse.

These experiments have obvious implications for the study of polymer blends, interfaces and micelles. Indeed, we already have preliminary evidence for intermolecular ^{1}H-^{2}H cross-polarization in a blend of a perdeuterated polymer with its protio counterpart. The experiment is straightforward; just deciding to try it **has made all the difference.**

Best regards,

Nick

NZ:lmw
Enc.

Nicholas Zumbulyadis
Corporate Research Laboratories

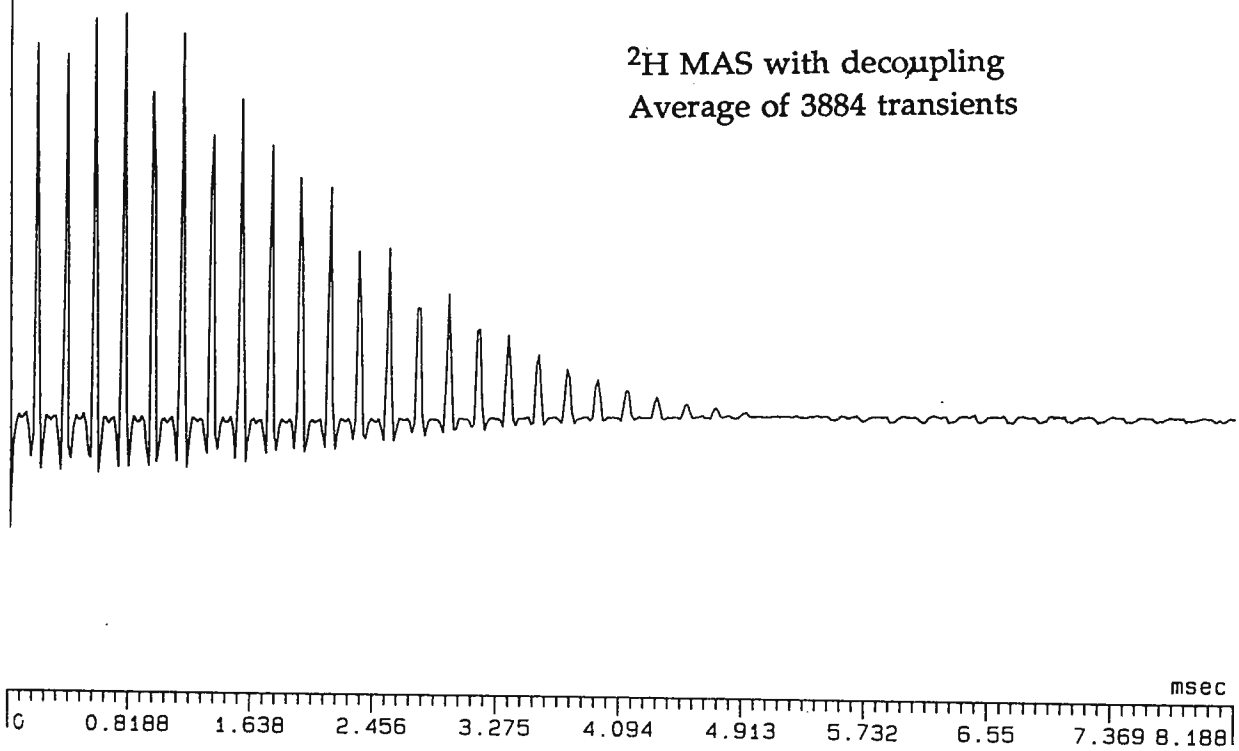
[1]C. Ye, B. Sun, and G. E. Maciel, J. Magn. Res. **70**, 241(1986).
[2]R. J. Schadt, R. Y. Dong, E. Gunther, and B. Blumich, J. Magn. Res. **96**, 393(1992).
[3]A. Schmidt, R. A. McKay, and J. Schaefer, J. Magn. Res. **96**, 644(1992).
[4]L. G. Butler, 33rd ENC.

Eastman Kodak Company
Rochester, NY 14650-2110
Tel. (716)722-1409 - FAX (716)477-6498

90% Randomly Deuterated Glycine

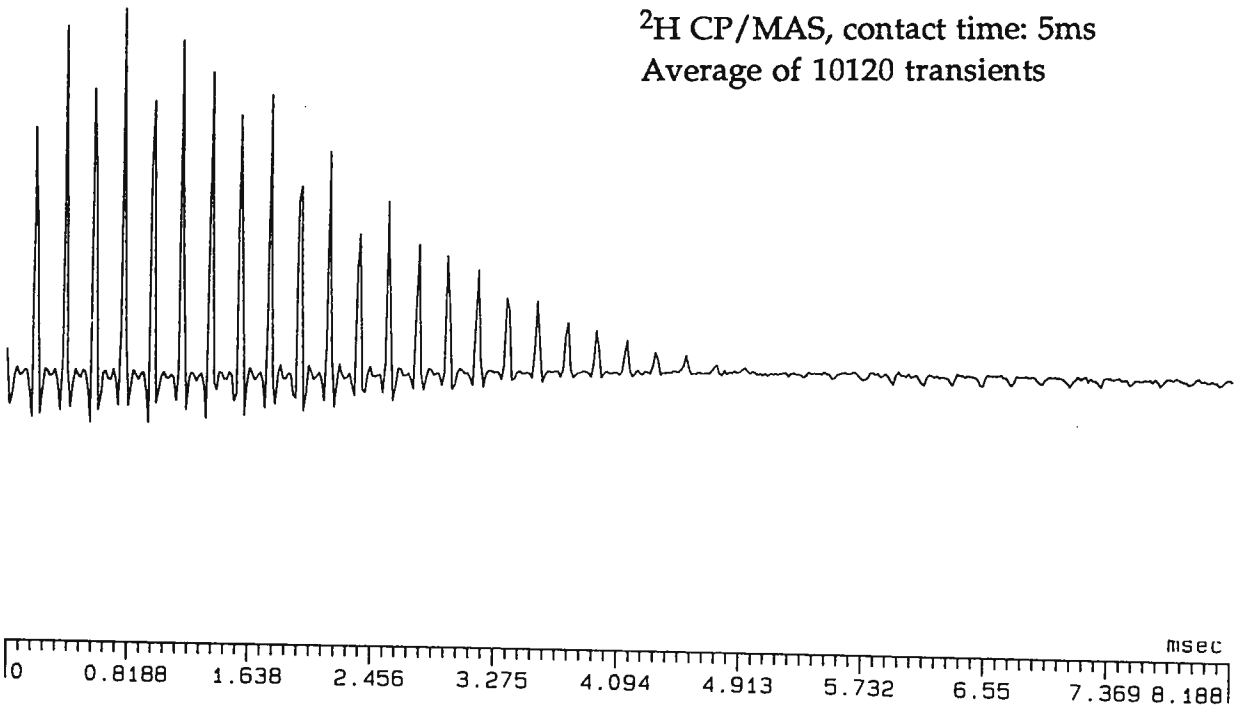
A

^{2}H MAS with decoupling
Average of 3884 transients



B

^{2}H CP/MAS, contact time: 5ms
Average of 10120 transients



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Chemagnetics utilizes the latest technology to put multi-channel flexibility in the hands of the spectroscopist. Dual 16 bit, 1 MHz digitizers offer maximum dynamic range over the widest bandwidths. And each RF channel in the CMX is equipped with its own pulse programmer, so experiments are never limited by pulse programmer capacity.

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▪ DOR	▪ TOCSY
▪ Wideline	▪ COSY
▪ CRAMPS	▪ Proton/Fluorine

Two Channels

Imaging	Solids	Liquids	Liquids+Imaging
	▪ Rotational Resonance ▪ CP/MAS ▪ HETCOR	▪ HCCH-TOCSY ▪ Reverse Detection ▪ HETCOR ▪ X-Decoupled	▪ ^{1}H, ^{19}F, Gradient Field Spectroscopy

Three Channels

Solids	Liquids	Liquids+Imaging
▪ REDOR	▪ Triple Resonance ▪ ^{1}H, ^{13}C, ^{15}N, HMQC-TOCSY	▪ H/X Gradient Field Spectroscopy

Four Channels

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These spectra of AlPO-11 below illustrate these effects. The top spectrum shows single resonance DOR at 625 Hz spinning. The bottom trace shows significant improvement at 980 Hz; note especially the appearance of the octahedral peak at 30 ppm. The middle trace shows 625 Hz DOR with proton decoupling; the peaks are narrower than at 980 Hz, especially the octahedral signal.

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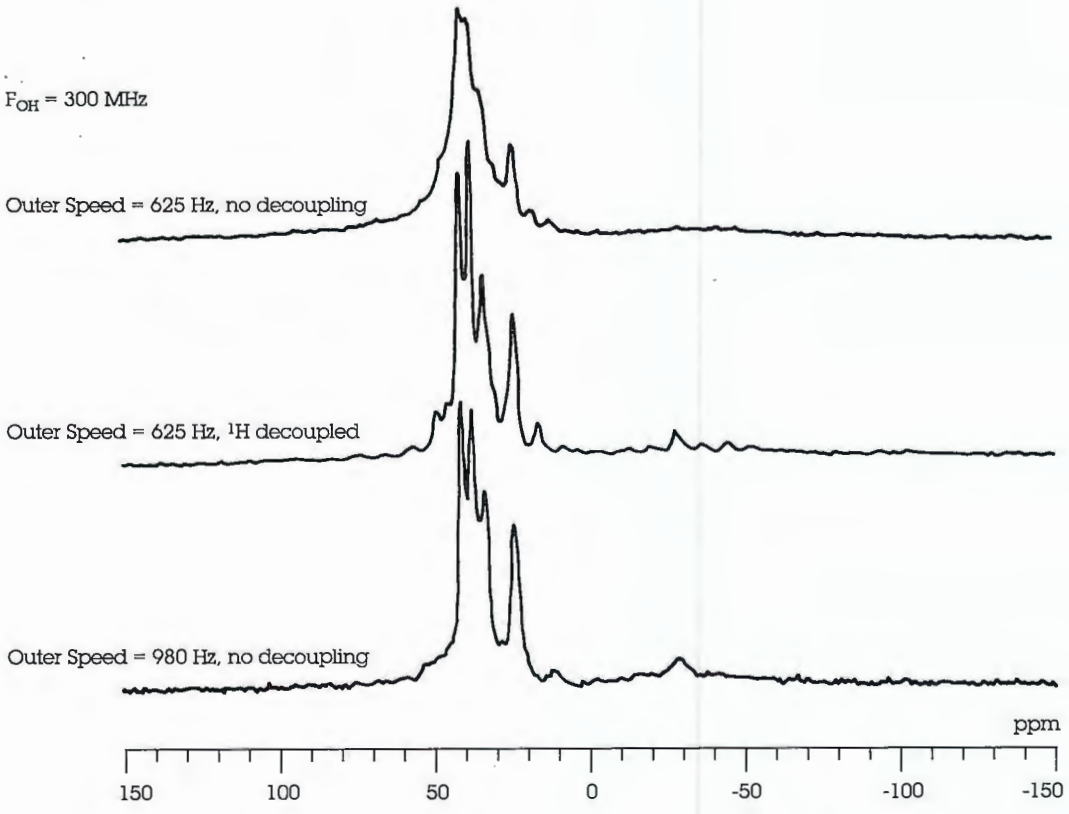

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August 4, 1992 (received 8/7/92)

Dr. Bernard L. Shapiro
TAMU NMR Newsletter
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e-mail: mweiner@ccb.ucsf.edu

Dear Dr. Shapiro:

Reliability and reproducibility of <u>in-vivo</u> NMR spectral data processing

As you may or may not know, the Nuclear Magnetic Resonance Spectroscopy Group at the DVA Medical Center in San Francisco, CA is one of the major research sites for clinical ^{1}H and ^{31}P NMR spectroscopic imaging (MRSI). This phase-encoding method provides beautiful images of metabolite distributions, usually of the brain, which are reconstructed from a huge amount of spectra per examination. For a typical brain examination in three spatial dimensions 12 x 12 x 12 = 1728 ^{31}P spectra are acquired, which after 4D-FT processing yield a three-dimensional data set of 32 x 32 x 16 = 16384 spectra, approx. 3000 of which contain information from the brain and can potentially be used for data analysis. Obviously, automated spectral processing is desired. Efforts by this group to develop a robust and automatic spectral processing routine for in-vivo ^{31}P brain data, including automated analysis of the "broad component", have been unsuccessful so far.

Manual processing of selected spectra by different or even the same operators may lead to systematic errors in data analysis due to, for instance, operator dependent (say different) setting of the baseline or phase of the typical low-signal-to-noise-in vivo-spectrum with its overlapping resonances. Therefore, operators need to be thoroughly trained so that operations are performed in a reproducible and consistent way. This letter describes intra- and inter-operator reliablity and reproducibility studies of spectral processing. Comparisons of intraclass correlation coefficients θ were used. These coefficients are calculated from metabolite mean square values, and they provide an indication of the contribution of noise and true measure to an experimental value. For example, θ = 0.96 indicates that 96 % of the measured values are true measures and 4 % are due to noise.

For ^{31}P MRSI, 12 spectra from various clinically relevant brain regions were selected from each of 8 normal controls (total = 96 spectra). All of them were processed by two trained operators familiar with NMR1 (commercially available software) processing of ^{31}P spectra, including manual phasing, manual baseline selection through the middle of the noise, and automated curve-fitting. The relative contribution of a metabolite resonance to the sum of all resonances (% metabolites) was calculated for all seven resonances (PME, Pi, PDE, PCr, γ, α, and β ATP). One of these operators fitted the same data on another occasion a month later to provide data for assessment of intra-operator reliability. Intraclass correlation coefficients were calculated across operators (*inter-operator reliability*) or across repeat measurements for one operator (*intra-operator reliability*) for each resonance. The coefficients for *inter-operator* processing, θ = 0.73 - 0.93, were moderate to strong, except for PME, for which θ = 0.45. PME resonates at the low-field end of the spectrum and is usually overlapping with Pi and PDE on the shoulder of the "broad component" (made up of immobile and less mobile phospholipids). Its area is therefore more susceptible to changes in phasing and baseline selection. This is also reflected in the lowest *intra-operator* reliability for the PME peak area (θ = 0.75). The corresponding coefficient for all other ^{31}P metabolite resonances were between 0.86 and 0.99. As expected, in ^{31}P MRSI spectral processing, the *intra-operator* reliabilities were greater than the *inter-operator* reliabilities.

For ^{1}H MRSI, *inter-operator* reliability in spectral data processing was tested by NMR1 curve-fitting 9 ^{1}H MRSI spectra each from 11 subjects (total = 99 spectra) by two trained operators. Again, % metabolites were calculated for all three major resonances (choline, creatine, and N-acetyl-containing metabolites). *Intra-operator* reliability was tested by NMR1 curve-fitting 4 spectra each from 11 different subjects 3 times over (total = 132 spectra). The intraclass correlation coefficients for *inter-operator* reliabilities and *inter-operator* reproducibilities were calculated: they were equally excellent (between 0.96 and 0.99) for all ^{1}H metabolite resonances and thus indicated very strong intraclass correlations. This is not a big surprise, considering that magnitude data were used and the ^{1}H MR resonances in these kind of MRSI spectra are not overlapping.

All of these numbers suggest the following:

1.) A reliable assessment of % metabolites can be made by two different operators, provided they are thoroughly trained.
2.) An even better assessment, especially of ^{31}P metabolite contributions, can be achieved if only one operator processes all spectra in a given set of control and disease data.
3.) Better use caution with inferences from PME peak areas!
4.) Evaluations of reliability of spectral processing are strongly encouraged in each laboratory, and a training data set for ^{1}H and ^{31}P spectral processing may be used to "check out" new group members in spectral data processing.

The results obtained for ^{31}P MRSI data emphasize the need for improved processing techniques to increase reproducibility of spectral processing. The development of completely automated operator-independent curve-fitting software would greatly improve data processing.

This whole processing exercise, so labor intensive it may have been, helped us understand much better the power and limitation of our MRSI technique. We are curious if any of you, dear reader, have done similar analyses or are working on automating in-vivo spectral data processing.

Sincerely, your curious investigators

Dieter J. Meyerhoff   Cynthia Husted   Michael W. Weiner

MWMWMWMWMWMWMWMWMWMWMWMWMWMWMWMWMWMWMWMWMWMWMWMWMWMWMWMWMWMWMWMWMWMWMWMWMWMWMWMWMW

LOVELACE MEDICAL FOUNDATION

We are seeking a postdoctoral fellow with interest in NMR studies of flowing systems to start in the fall of 1992. Knowledge of NMR physics, hardware, or software is essential. Experience in applying echo-planar imaging would be helpful. [See Physics Reports 198, 197 (1990) for background.] Appointments are for one year, renewable by mutual agreement. Lovelace Medical Foundation is a small, private, research laboratory in a city of 600K at an altitude of 1600 meters above sea level. Please send inquiries or informative biographies including addresses and telephone numbers of your references to Eiichi Fukushima, Lovelace Medical Foundation, 2425 Ridgecrest Dr., SE, Albuquerque, NM 87108, USA. FAX number 505/262-7043.

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Every rotor is individually tested above its rated speed.

Low Frictional Heating: 2°C max, from 1 kHz to 7.5 kHz

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High RF Homogeneity: ±5% for 0.77 ml sample volume

Hartmann-Hahn match and high rf field strength are not sufficient for CP at high speeds. The spinning frequency offset imposes the requirement of high B_1 homogeneity. High rf homogeneity can double SNR.

- Multinuclear channel tunable from ^{31}P to ^{39}K in most WB's.
- Zirconia construction. Standard VT range: -140 to +180°C.
- Upper temperature limit may be extended to 280°C.
- Triple-tuned option with dual, BB channels: ^{1}H-X-Y.
- Also available for Varian narrow bore (44 mm RT bore).

10 mm Rotor Specifications

Rotor Wall (mm)	material	Sample Length (mm)	Volume (ml)	RF Homogeneity (±%)	Maximum Safe Speed* (kHz)
0.7	zirconia	17.6	1.02	15	6.7
1.0	glass ampule	18	0.35	15	3
0.7	zirconia	13.2	0.77	5	7.4
1.2	Si_3N_4	13.2	0.60	4	10.5

* For 1.2 g/cm^3 sample density

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4.7	^{15}N	20	25	1,400	4
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Bethesda, Maryland 20892

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

July 20, 1992
(received 7/30/92)

Dear Barry:

$^1J_{C\alpha H\alpha}$ and protein conformation

We generally assume that $^1J_{C\alpha H\alpha}$ in proteins has a uniform value of ~140 Hz. However, as von Philipsborn and co-workers (1) have demonstrated using conformationally constrained cyclic peptides, the magnitude of this coupling varies with the local geometry around the C_α carbon. From this work it was implied that particularly the backbone angle ϕ, between C_α and its adjacent nitrogen, dominates the $^1J_{C\alpha H\alpha}$ dependence. This would occur by interaction of the lone pair nitrogen electrons with the C^α-H^α bonding orbitals. A smaller effect was ascribed to ψ, the angle between C^α and its adjacent carbonyl.

In practice it is quite difficult to separate the effects of ϕ and ψ, because the degree of variation in ϕ is not very large, and ϕ and ψ cannot be varied independently. A large set of data for different ϕ/ψ combinations is therefore necessary before one can reliably separate the effects of ϕ and ψ, particularly because, as we found, parameters other than ϕ and ψ also have a significant effect on the $^1J_{C\alpha H\alpha}$ value.

Proteins present ideal collections of conformationally tightly constrained amino acids. Particularly if the protein structure is known at high resolution, either from X-ray crystallography, or potentially from a very detailed NMR study, all dihedral angles are known with good precision. Provided complete assignments for the protein are also available, it is relatively straightforward to measure the large and well resolved $^1J_{C\alpha H\alpha}$ couplings. For proteins that have been enriched with ^{13}C, sensitivity also does not present any problems in such measurements. However, for larger proteins, the line width of H^α when it is coupled to $^{13}C^\alpha$ is more than double relative to being attached to ^{12}C and frequently exceeds 20 Hz, making it difficult to measure the J splittings very precisely. In practice we found it impossible to measure these splittings with a precision better than ~1 Hz. Therefore, instead of trying to measure the value from the splitting, we designed some 2D experiments where the intensity of the C^α-H^α (or CO-H^α) resonance is modulated by $^1J_{C\alpha H\alpha}$. This results in highly reproducible values for $^1J_{C\alpha H\alpha}$, which agree very well with the direct measurements. For non-overlapping correlations in the 2D spectrum we are confident that our error is smaller than ~0.5 Hz.

For 203 residues in the proteins staphylococcal nuclease (assigned and provided to us by Dennis Torchia & co-workers) calmodulin (labeled, purified and assigned by Mitsu Ikura) and BPTI (provided by the NIH self-service store, and assigned by Gerhard Wagner and friends) we were able to measure the $^1J_{C\alpha H\alpha}$ values, plus we had reliable backbone angles from previous

crystallographic work. Figure 1 shows how these couplings deviate from their random coil values, depending on ϕ and ψ. The most obvious feature is that residues in an α-helical conformation have large values of ~147 Hz, whereas β-sheet residues are close to their random coil value. Non-glycine residues in the "forbidden" ϕ,ψ region near ψ=45°/ϕ=55° have the smallest values for $^1J_{C\alpha H\alpha}$ (~133 Hz). The surface is described quite well by the following empirical equation:

$$^1J_{C\alpha H\alpha} = 140.3 + 1.4\sin(\psi+138^o) - 4.1\cos2(\psi+138^o) + 1.7\cos2(\phi+30^o)$$

As mentioned above, there remains a significant rms deviation of 2.0 Hz which cannot be explained by ϕ and ψ, and which may be caused by a variety of factors, including hydrogen bonding of the amide and carbonyl, and the effects of solvent and electric fields.

Kindest regards,

On Vacation

Geerten Vuister Frank Delaglio Ad Bax

(1) Gil, V.M.S., von Philipsborn, W. (1989) *Magn. Reson. Chem.* **27**, 409-430.

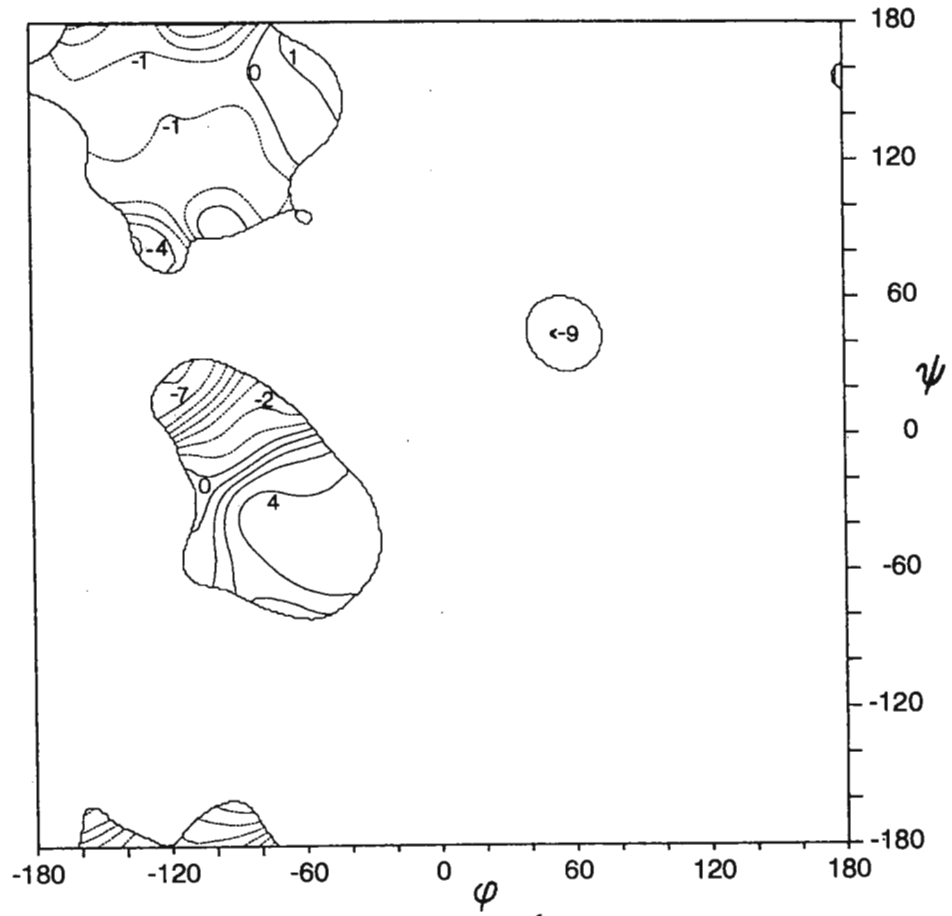

Figure 1. Contour plot showing the deviation of $^1J_{C\alpha H\alpha}$ from its random coil value, as measured for 203 residues in the proteins staphylococcal nuclease, calmodulin, and BPTI.

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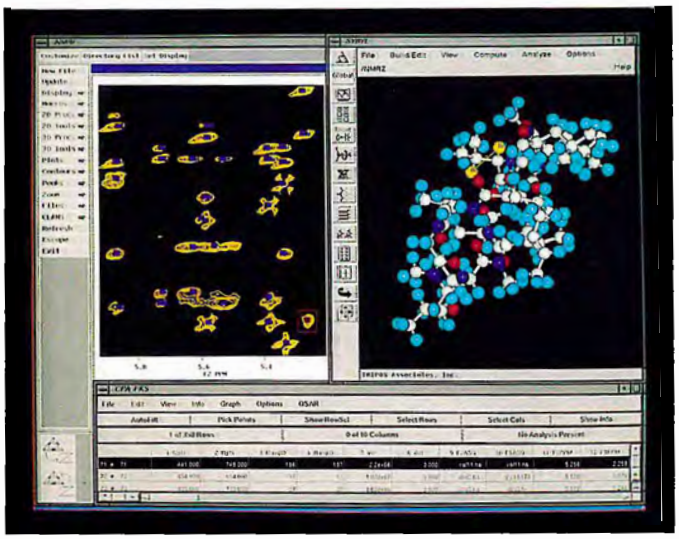

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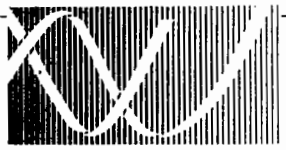

C A B M

CENTER FOR ADVANCED BIOTECHNOLOGY AND MEDICINE
675 Hoes Lane • Piscataway, NJ 08854-5635 • 201/463-4665

(received 8/17/92)
August 6, 1992

Progress with Heteronuclear PFG NMR

Dear Dr. Shapiro,

This past June we took delivery of a Varian Pulsed Field Gradient (PFG) Triple Resonance Probe and gradient amplifiers. PFG NMR is an exciting area of technology development with especially useful applications in protein and nucleic acid NMR[1-12]. Using PFGs it is possible to select in a single scan only those nuclei which develop homonuclear or heteronuclear multiple quantum coherences (MQCs). Since the H_2O solvent protons cannot usually develop MQC, they are invisible in these experiments. In this way it is possible to selectively observe protein protons with complete water suppression and without attenuation of important resonances[4-12]. PFGs select coherence transfer pathways in a single scan, eliminating the need for phase cycling, reducing the number of scans needed to obtain each FID of a multidimensional NMR data set, and increasing the dynamic range available for digitizing the data of interest.

Comparisons of 2D ^{15}N-HSQC spectra recorded with either phase cycling or using PFGs are shown in Figures 1 and 2. These two data sets were recorded using identical total measuring times (ca. 3 hrs.) and maximum values of the ^{15}N evolution time t1, processed with identical window functions, and displayed in absolute value mode. We were especially pleased to see that, for a 5 kDa protein, the ^{15}N-^{1}H correlation peaks of the PFG-version exhibit signal-to-noise ratios equal to or greater than those obtained in the phase-cycled version of the experiment. This is not too surprising since in this sample even weak solvent preirradiation results in saturation of some H^{α} resonances and attenuation of the H^{N} signals by spin diffusion effects. In addition, much higher values of the gain setting are possible in the PFG experiment since complete solvent suppression is obtained in a single scan. A more detailed comparison of preirradiation and PFG methods for solvent suppresion in ^{15}N-HSQC will be presented elsewhere. We anticipate that PFGs will provide a great benefit to 3D and 4D NMR experiments of protein samples in H_2O solvent.

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6. Hurd, R.E. **1990** *J. Magn. Reson.* 87, 422.
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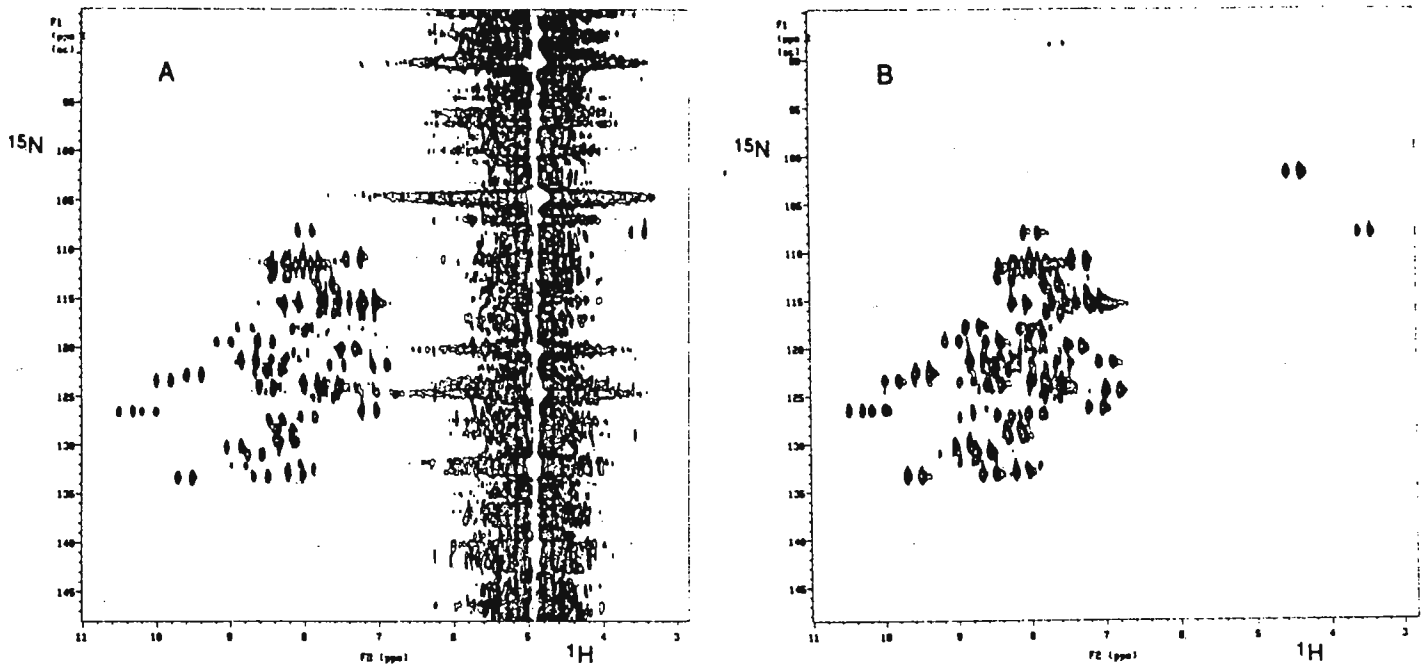

Fig. 1. Comparison of Phase-Cycled and PFG 2D [^{15}N]HSQC Experiments. (A) Phase-cycled [^{15}N]HSQC recorded with weak preirradiation to suppress the solvent H_2O signal. The residual H_2O solvent peak in this spectrum is especially strong because the probe has not been optimally shimmed for solvent suppression by preirradiation and because the spectra are presented in absolute value mode. (B) PFG-[^{15}N]HSQC experiment recorded using no solvent-suppressing preirradiation or trim pulses. The spectra were recorded without ^{15}N decoupling during the detection period. This provided clear evidence that the peaks under the H_2O resonance were indeed amide protons with $^1J(^1H\text{-}^{15}N)$ coupling constants of 90 Hz. The PFG experiments provide excellent suppression of the H_2O signal without many of the artifacts associated with solvent preirradiation. The sample is 1 mM of 100% uniformly ^{15}N-enriched [V30, A51]-pancreatic trypsin inhibitor dissolved in H_2O at pH 6.5. In this analog of BPTI, the Cys^{30}-Cys^{51} disulfide bond has been removed by mutagenesis.

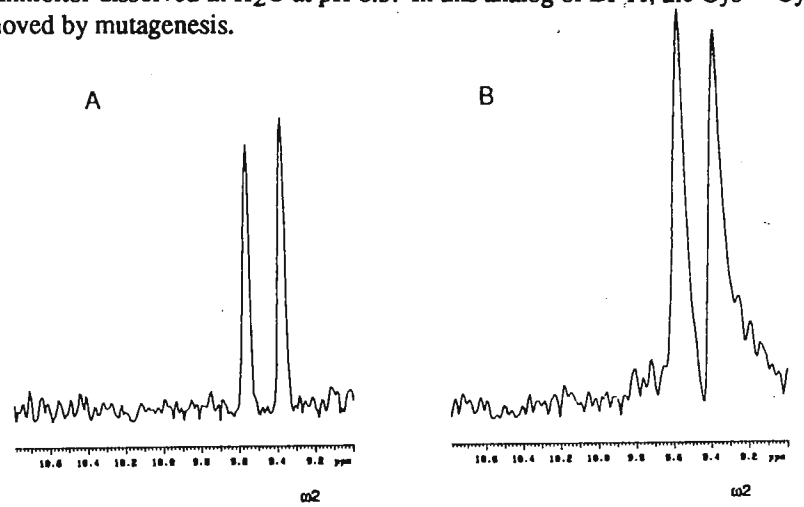

Fig. 2. Comparison of Cross Sections Through Phase-Cycled and PFG 2D [^{15}N]HSQC Experiments. Cross sections along ω2 through the ^{15}N-^{1}H correlation peak of residue Tyr-35 of (A) the phase-cycled and (B) PFG-[^{15}N]HSQC spectra shown in Figure 1.

Yu Chin Li
CABM NMR Lab
Rutgers Univ.

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Timothy Saarinen
Product Manager
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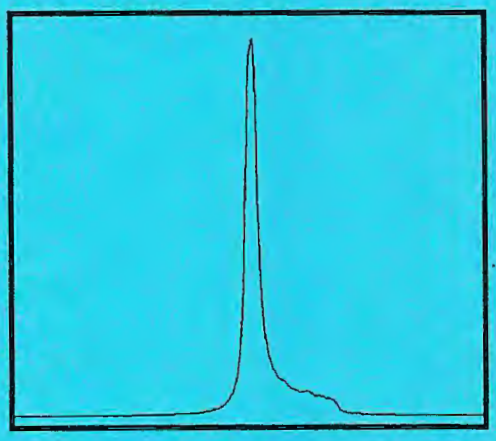

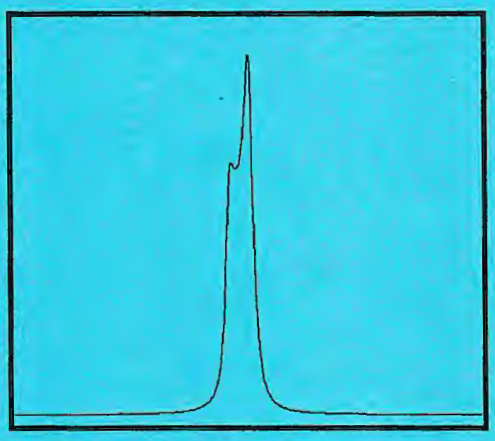

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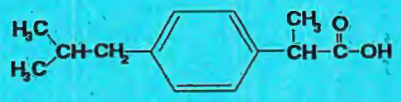

DEPARTMENT OF HEALTH & HUMAN SERVICES

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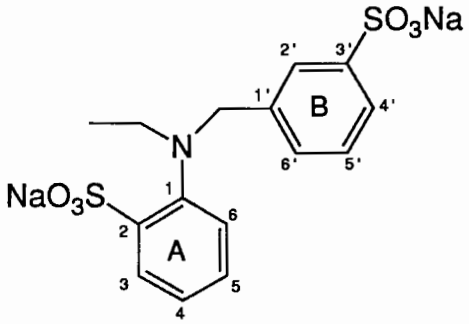
August 1, 1992
(received 8/7/92)

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

NMR Exchange Spectra of Tertiary Amines

Dear Barry,

While investigating the following tertiary amine



we observed that both sets of methylene protons are enantiotopic in basic solution but diastereotopic in strong acid solution where the amine exists primarily as the ammonium salt. In addition, broadening of certain signals, in both the ^{1}H and ^{13}C spectra, which are not involved in any type of conformational, *e.g.* inversion, processes was seen at moderately acidic pH. Here exchange, which is apparently slow on the NMR time scale, occurs between the free amine and its ammonium salt. In the proton NMR spectrum, signals due to the ethyl-methylene protons ($\Delta\nu_{acid\text{-}base}$=212, 272 Hz) nearly disappear, and those of the benzylic-methylenes ($\Delta\nu$=80, 180 Hz), H-4 ($\Delta\nu$=230 Hz), and H-6 ($\Delta\nu$=324 Hz) are considerably broadened. In the C-13 NMR spectrum, the resonances due to C-1 disappear while those of carbons 1' and 4 and the ethyl-methylene carbons are substantially broadened.

With regard to the first, proton interchange process, Martin Saunders examined a somewhat analogous aliphatic amine, $MeN(CH_2Ph)_2$, nearly 30 years ago and obtained similar results.[1] Leyden expanded this work and described a more exact kinetic treatment for the amine-salt exchange process.[2] Saunders made a critical point which bears repeating for people who are interested in, or confronted with, inversion processes of amines, *viz.*, inversion at nitrogen can occur only with the free amine and not with the ammonium salt. The nature of any prochiral proton resonances, such as those of methylene protons, thus depends on both the rate of nitrogen inversion (which is relatively rapid on the NMR time scale) and the proportion of amine present as the free base (which, depending on solution pH, may be quite small). The fact that Saunders' methylene protons remained diastereotopic, even when NH-exchange (he used HCl, not DCl) became sufficiently rapid to eliminate NH-coupling with these protons, suggested that the ammonium salt was being deprotonated and reprotonated without undergoing inversion. We are also studying the *p*-sulfophenyl (A-ring) analog to determine the pH value at which its methylene protons change from diastereotopic to enantiotopic.

Sincerely,

Gene

Gene Mazzola

[1]M. Saunders and F. Yamada, *J. Am. Chem. Soc.*, **85**, 1882 (1963).
[2]W.R. Morgan and D.E. Leyden, *ibid*, **92**, 4527 (1970).

TU Delft

1992

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

Your reference and date	Our reference	Office telephone	Date
			July 7, 1992. (received 7/31/92)

Subject

Sub-division

Editing techniques for the detection of glutamate and glutamine in vivo.

The quantitative detection of glutamate (Glu) and glutamine (Gln) by in vivo ^{1}H-NMR is severely hampered by strong overlap of the α, β and γ signals of Glu and Gln. The α peaks of Glu and Gln overlap completely and are very close to the water resonance. The β peaks also overlap considerably and are spread out over a wide range, reducing the peak height. Therefore the γ peaks were chosen for a selective detection by means of double quantum and spin echo editing techniques. Using the chemical shifts and coupling constants of Glu and Gln the optimal DQC preparation and evolution time and phase settings, and the optimal spin echo time with regard to selectivity and signal to noise ratio were determined. Simulations were used because the effect of the pulse sequences on these strongly coupled spin systems can not be calculated analytically. The simulated results were compared with experimental 7 T spectra of 20 mM solutions of pH=7.0 with linewidths comparable to in vivo.

The best DQC results were obtained with gradient DQC selection, and the two 90^0 preparation pulses 180^0 out of phase. Gln is optimally detected at 7 T with a preparation (τ) and evolution time (t_1) of 128 and 10 ms. Glu is then suppressed a factor 10 with respect to Gln, but the signal loss of Gln is a factor 5. Glu is optimally detected in a two shot sequence (t_1=11.7 and 8.0 ms resp., the signals are added) with τ=20 ms. Gln is then suppressed a factor 7.2 with respect to Glu, but the signal loss of Glu is a factor 12, which is intolerable.

In a spin echo sequence both Gln and Glu were optimally detected with two shot sequences. For Glu echo times of 126 and 92 ms were chosen, the acquired signals were subtracted. The gamma Gln signal intensity is then a factor 8 suppressed with respect to Glu, see Fig. a. Gln is optimally detected by subtracting the signals obtained with echo times of 60 and 126 ms, the gamma Glu peak is then almost completely suppressed, see Fig.b. In the spin echo editing sequences the signal loss for both Glu and Gln is about 40% compared to a normal spin echo sequence with an echo time of 136 ms.

Concluding, selective detection of Glu and Gln is possible via double quantum filtering and spin echo editing techniques, at the cost of considerable signal loss. For lower field strengths (1.5 T) editing is the only possibility to separate Glu and Gln in vivo, but at higher field strength dedicated data fitting techniques are to be preferred.

J.E. van Dijk

W.M.M.J. Bovée

The figures appear on page 42.

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UCD NMR FACILITY DAVIS, CALIFORNIA 95616

August 6, 1992
(received 8/7/92)

Dr. Barry Shapiro
966 Elsinore Ct.
Palo Alto, CA 94303

Title: Re-formatting Omega Hardcopy

Dear Dr. Shapiro:

We have several GE-NMR spectrometers and workstations that run the Omega 6.0 software. The laserwriter hardcopy produced by the default settings in the Omega software produces thin lines and small font sizes (see Figure A). This hardcopy is often marginal for usage in publications. Following inquires to GE, Jerry Dallas of their Applications Group suggested the following procedure.

1) Write a script "save_ps" to capture the hardcopy PostScript file temporarily created by the Hard Copy panel when plotting:

```
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while ( 1 )
        set FL = ( 'ls  *.ps' )
        if ( $#FL ) then
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        endif
sleep 1
end
#
```

We place this script in /home/base/genmr/plot; and it must be executable.

2) Use a UNIX shell to execute the script; let it run continuously so that it will capture the PostScript file as soon as it is created by the Hard Copy panel.

3) Use the Hard Copy panel to make a plot in the usual fashion as shown in Figure A. Executing the plot command in Hard Copy will create the temporary PostScript file that will be captured by the save_ps script and saved to the file called ps_save.

4) Now that the ps_save PostScript file has been saved, it can be edited by vi or (more conveniently) the SUN text editor. We commonly make the following changes:

a. Replace 0 setlinewidth with 2 setlinewidth throughout the file.
b. Under the section "/draw tics" change /Times-Roman findfont 12, to findfont 16.
c. Under "/showlabel", change /Times-Roman findfont 9, to findfont 16.

This will make the plot, axis, and axis tic marks thicker and darker, and puts axis units and labels in a larger font, as shown in Figure B.

5) Use the UNIX command "lpr ps_save" to print the modified PostScript hard copy file (Figure B).

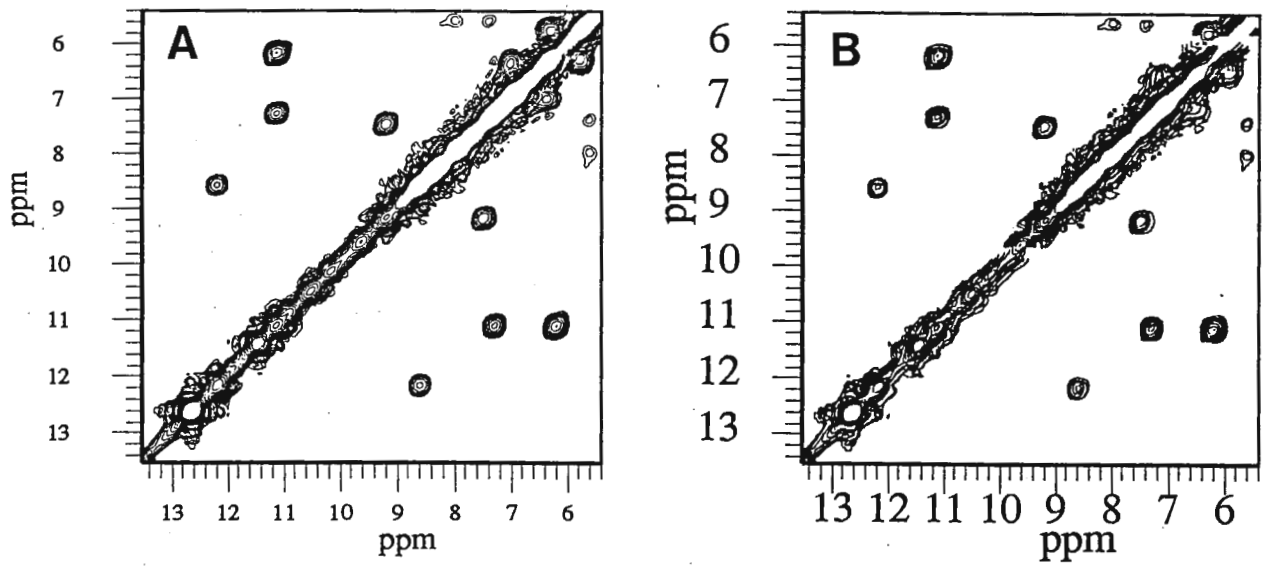
We have found that with practice the above procedure goes easily and rapidly; we now employ it for making all final figures for publication.

Sincerely,

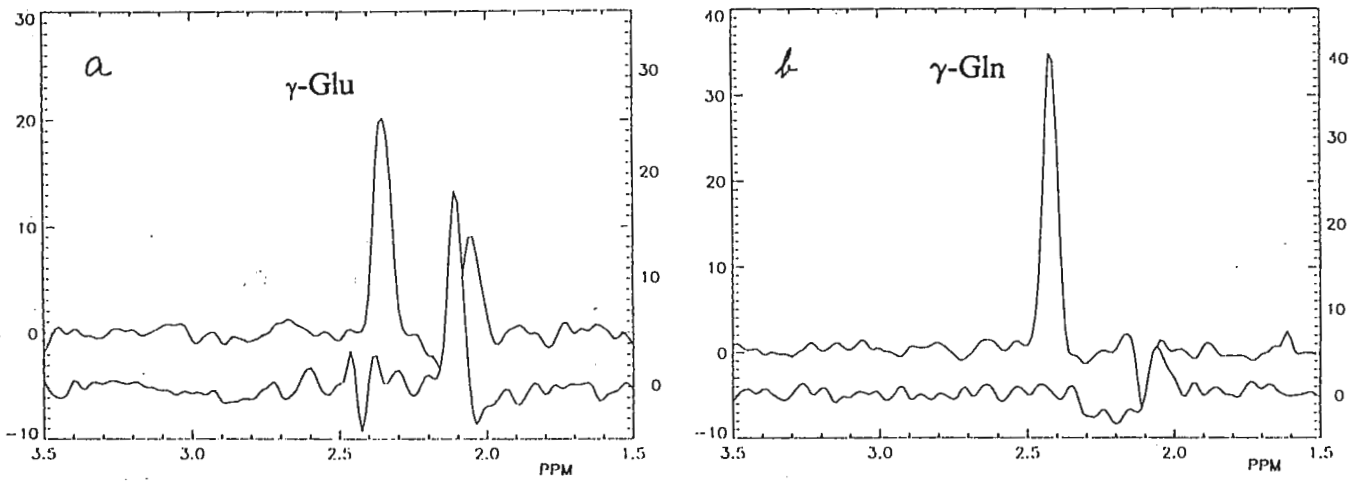
Jeff de Ropp

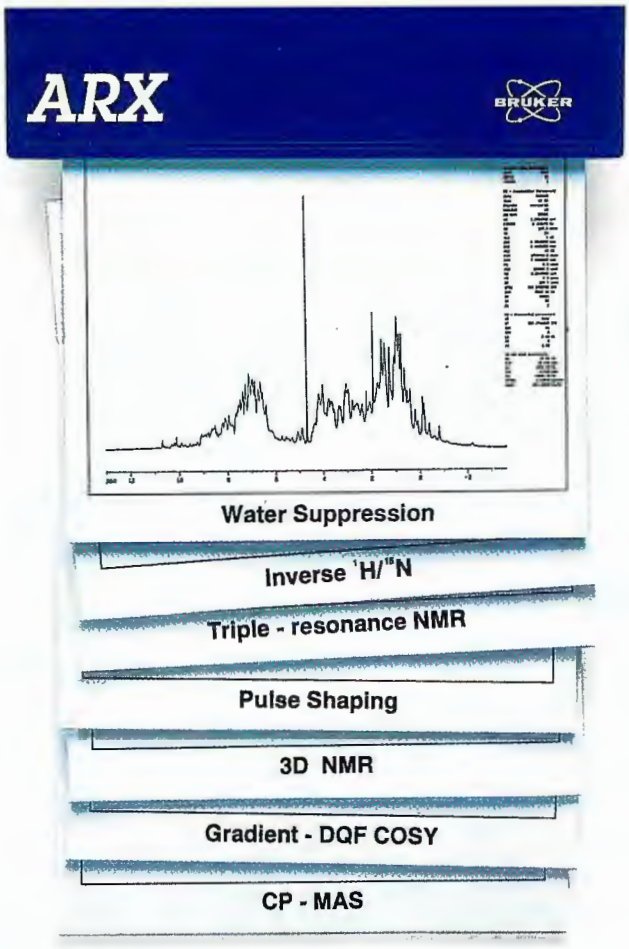
Steve Unger

Gerd La Mar
Director



- continued from page 38.





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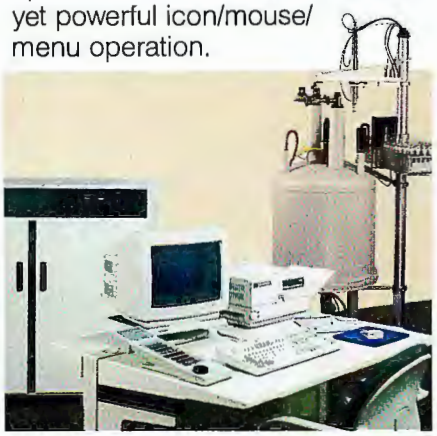

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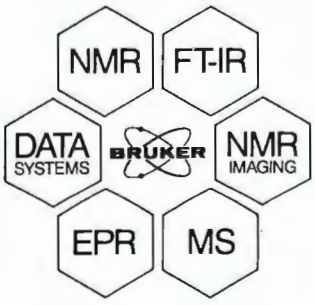
Performance Data

Summary of Specifications for common probeheads					
ARX		300	400	500	All
Probehead	Sample				
Resolution Test					
All 5 mm ^{1}H 5+10 mm ^{13}C	ODCB/C_6H_6	0.2	0.2	0.2	
Lineshape Test					**SSB %**
All 5 mm ^{1}H	10% $CHCl_3$	6/12	7/15	7/15	<1
All 5 (10) mm ^{13}C	80% C_6H_6	3/7 (3/7)	3/7 (3/8)	4/8 (4/8)	<0.5 (<1)
Sensitivity Test					
5 mm ^{1}H Selective	0.1% EB	175	250	450	<10
5 mm ^{1}H Inverse Detection	0.1% EB	135	190	350	<15
5 mm ^{1}H Dual, QNP, VSP	0.1% EB	100	140	200	<15
5/10 mm ^{13}C QNP, Dual	ASTM	100/320	160/450	180/600	<15/<20
5/10 mm ^{13}C QNP, Dual	10% EB	70/200	100/300	150/400	
5/10 mm ^{13}C VSP multinuc.	ASTM	100/320	160/450	180/600	<15/<20
5/10 mm ^{13}C VSP multinuc.	10% EB	70/200	100/260	150/320	
5/10 mm ^{15}N VSP multinuc.	90% Form.	10/35	15/55	20/70	<25/<30

EB = ethylbenzene (for ^{13}C with ^{1}H-dec.);
ASTM = 60% C_6D_6 in dioxane
Form. = formamide (^{1}H-dec. without NOE)
lineshape: ^{1}H = $CHCl_3$ linewidth at ht. of ^{13}C-satellites/at 2^{6} this level
^{13}C = C_6H_6 linewidth at 0.55% / 0.11 level (^{1}H-dec.)
SSB = Spinning sidebands measured with 8 transients
QNP: 5 or 10 mm ^{1}H, ^{31}P, ^{13}C, ^{15}N
VSP: 5 mm ^{15}N – ^{13}P; 10 mm ^{109}Ag – ^{31}P.

Console	Dimensions and weights are approximate; voltage +10/ – 5% max. variation; other line freq. and voltage upon request
dimensions electronics cabinet	w 130 x d 0.75 x h 1.20 m
weight	450 kg
power (dissipation)	220 V/50 Hz/16 A (ca. 4 kW)



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ENC 34th Experimental Nuclear Magnetic Resonance Conference
March 14–18, 1993, Adam's Mark Hotel St. Louis, Missouri

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

Dear Barry:

The 34th Experimental NMR Conference will be held at the Adam's Mark Hotel in St. Louis, Missouri from March 14-18, 1992. There will be sessions covering high resolution and multi–dimensional NMR in liquids, materials imaging, biological imaging, hardware, NMR in solids, calculations and data processing, and that old standby, miscellaneous. Two poster sessions will be held, and we anticipate having adequate space for the posters at the Adam's Mark. **The deadline for submitting poster abstracts will be December 30, 1992** (ENC is early this year!), so start planning now.

We have set dates and sites for future ENC's as well, and you should mark the following dates on your busy calendar:

March 14-18, 1993	Adam's Mark Hotel, St. Louis, MO
April 10-14, 1994	Asilomar Conference Center, Pacific Grove, CA
March 26-30, 1995	Boston Marriott Copley Place, Boston, MA
1996	to be determined
March 23-27, 1997	Clarion Plaza Hotel, Orlando, FL

A conference announcement will be mailed to those on our mailing list (*ie*, registered conferees from the past three ENC's) in October. Anyone wishing to be added to the mailing list should contact the Conference Office, address and phone at the bottom of this letterhead. **Please note that the office address and phone have changed from last year.**

I look forward to an exciting conference in St. Louis, and hope that you and your readers will be able to participate.

Sincerely,

Mary

Dr. Mary W. Baum, Chair, 34th ENC
fax: 908-594-1530
email: baum@msdrl.com

UNIVERSITY OF ST. ANDREWS

From: Dr. F. G. Riddell

DEPARTMENT OF CHEMISTRY,
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FGR/WC

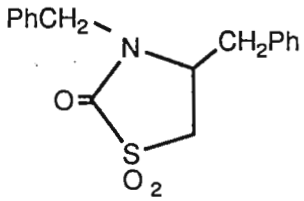
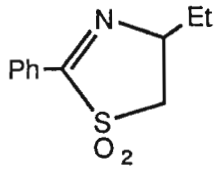
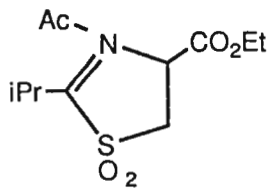
3rd August 1992
(received 8/10/92)

Professor B. L. Sharpiro
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto
CALIFORNIA 94303
USA

Dear Barry,

Some new ^{33}S resonances

Alan Aitken and Shaun Mesher in our department have been using the power of our MSL 500 at 34.8 MH_z to look for previously unobserved ^{33}S resonances. Using 4-5 ml of neat liquid or concentrated solution in our solenoid probe has enabled us to see quite a few new sulphur functional groups that were previously unknown. Here are some of them:

Functional Group	Compound	δ p p m (ref $Na_2SO_4^{2-}$)	$W^1/_2$ Hz
thiazolidin-2-one *S,S*–dioxide		– 6.5 (AM 300)	130
2-thiazoline *S,S*–dioxide		+ 36.6	220
thiazolidine - *S,S*–dioxide		+ 18.7	460
Sulphonyl Chloride	Et SO_2 Cl	+ 23	1980

UNIVERSITY OF ST. ANDREWS

From: Dr. F. G. Riddell

DEPARTMENT OF CHEMISTRY,
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SCOTLAND

Tel: (0334) 76161 Ext. 8364
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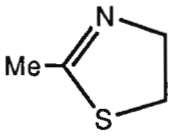
thiazole		– 217	4200
sulphinyl chloride	Et SO Cl	+ 217	6200
thiophenol	Ph SH	– 332	7050
arylthioether	PhSMe	– 390	19800

As expected the sulphones give sharper lines than the other functional groups.

The MSL 500 in this configuration is probably the best instrument yet for observing these types of resonances, especially the broader lines.

Best wishes,

Frank R Alan Aitken

F. G. Riddell **R. A. Aitken**

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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.

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Attention overseas subscribers: If you must use paper which is longer that 11", *please* take care that all material (including signatures, addresses - everything!) ends no more than 10" from the top of each of your pages. It is costly to make reductions, and henceforth I reserve the right to chop the excess length off any page, no matter what the result. Beware of the dreaded guillotine! Your cooperation in this matter will be greatly appreciated.

All Newsletter Correspondence

Should Be Addressed To:

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

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DEADLINE DATES

No. 410 (November) -------16 October 1992
No. 411 (December)-----20 November 1992
No. 412 (January 1993) - 18 December 1992
No. 413 (February) --------- 22 January 1993

* * * * *

Is your Oct. 1992 - Sept. 1993 subscription invoice paid?

BRUKER

PRESS RELEASE:

BRUKER INSTRUMENTS, INC. ACQUIRES
GE NMR INSTRUMENTS BUSINESS

Billerica, MA, August 27, 1992: Bruker Instruments, Inc. has acquired the analytical NMR (Nuclear Magnetic Resonance) and CSI (Chemical Shift Imaging) Instruments business of the General Electric Company. The acquisition, effective August 24, 1992, was announced by Dr. Frank Laukien, Executive Vice President of Bruker Instruments, who indicated that this is a strategic step in Bruker's long-term planning in the United States.

Many of the key technical personnel from GE NMR Instruments have already been hired by Bruker, and Bruker regards this major addition of talented and experienced NMR scientists and engineers as an excellent and unique opportunity to strengthen and expand its U.S. and West Coast NMR team. These additional scientists and engineers will combine with Bruker's existing West Coast operation at the 52,000 sq.ft. Fourier Avenue facility in Fremont, California, previously used by GE NMR Instruments.

As part of this acquisition, Bruker will provide on-going service, applications, software and hardware support worldwide to the installed GE NMR and CSI customer base, from its Fremont, CA, support center, as well as from Bruker service and support facilities in Massachusetts, Delaware, Illinois, Texas, Japan, the U.K., Germany, and Switzerland.

Bruker will also provide upgrades and enhancements to the existing GE NMR instrumentation, and offer some of the acquired technology to its own NMR customers. "GE NMRI has been the world leader in CSI Accustar™ gradients and coil technology, and has pioneered the applications of gradient enhanced spectroscopy to high-resolution NMR; Bruker is enthusiastic about offering these technologies and products on its enhanced Biospec™ CSI and AMX high-resolution NMR product lines.", explained Dr. Laukien.

For additional information call Dr. Malcolm Bramwell, Vice President and manager of Bruker operations in the Western United States, at phone number (408) 434-1190.

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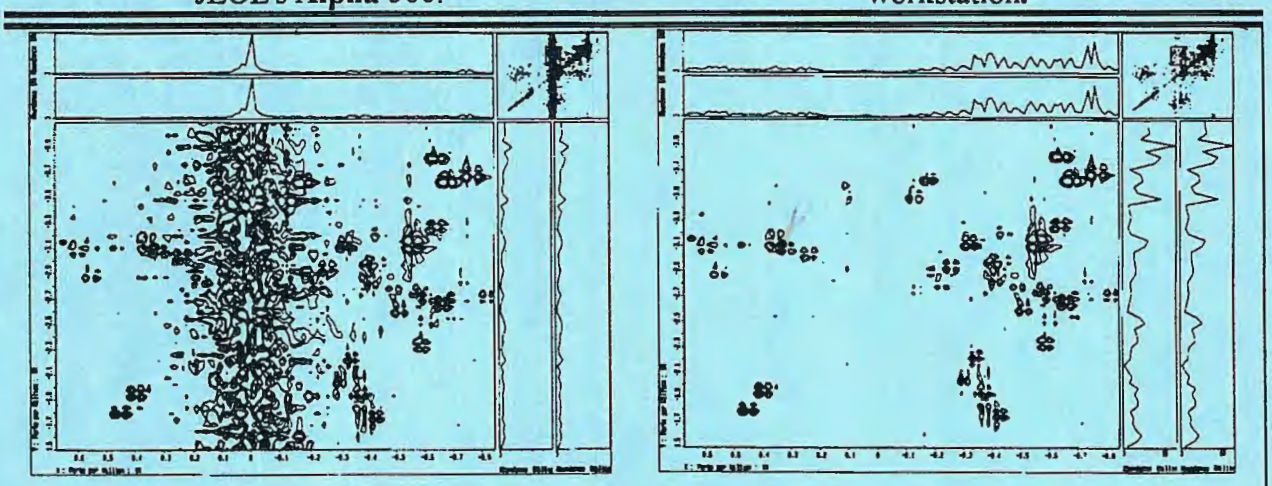
The same data, processed with a high pass filter using **DELTA**, JEOL's SGI based NMR workstation.



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