THE NMR NEWSLETTER

No. 451

April 1996

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| | Frequency Range | Resolution | Switching Time[1] | Phase-Continuous Switching[2] | Rack-Mount Cabinet Dim.[3] | Remote-Control Interface | Price Example[4] |
|---|---|---|---|---|---|---|---|
| **PTS 040** | .1-40 MHz | optional .1 Hz to 100 KHz | 1-20μs | optional | 5¼"H×19"W | BCD (std) or GPIB (opt) | $5,330.00 (1 Hz resol., OCXO freq. std.) |
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1 Switching Time is dependent on digit (decade) switched; see detailed instrument specifications.

2 For applicable digits, see detailed instrument specifications.

3 Bench cabinets are 17" wide.

4 Prices are U.S. only and include Manual and Remote (BCD) Control; PTS 3200 Digital Front Panel.


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## FORTHCOMING NMR MEETINGS

Carbon Isotopes in Metabolic Research: Mass Spectrometry, NMR, PET, and Radiotracer Methods, Dallas, Texas, **April 26, 1996**; Contact: L. Greening, (214) 648-5886 or D. Christensen, (214) 648-8013 at the Univ. of Texas Southwestern Medical Center.

Society of Magnetic Resonance, Fourth Scientific Meeting and Exhibition, New York, NY, **April 27 - May 3, 1996**; Contact: SMR Office, 2118 Milvia St., Suite 201, Berkeley, CA 94704; (510) 841-1899; Fax: (541) 841-2340. E-mail: info@smr.org . Future meetings: 1997, April 12-18, Vancouver, BC, Canada; 1998, April 18-24, Sydney, Australia; 1999, Philadelphia, PA; 2000, Denver, CO.

NMR Symposium at the 38th Rocky Mountain Conference on Analytical Chemistry, Denver, Colorado, **July 22-25, 1996**; Contact: Dr. Joel R. Garbow, Monsanto Company, 700 Chesterfield Parkway North, St. Louis, MO 63198; (314) 537-6004; Fax: (314) 537-6806; e-mail: jrgarb@snc.monsanto.com; See Newsletter 445, 48.

42nd International Conference on Analytical Sciences and Spectroscopy, London, Ontario, Canada, **Aug. 10-13, 1996**; Chair: M. Stillman, Dept. of Chemistry, University of Western Ontario, London, ON, Canada N6A 5B7; (519) 661-3821; Fax: (519) 661-3022; E-mail: 42info@uwo.ca.

XVIIth International Conference on Magnetic Resonance in Biological Systems, Keystone, Colorado, **August 18 - 23, 1996**; Contact: ICMRBS, 1201 Don Diego Avenue, Santa Fe, NM 87501; (505) 989-4735; Fax: (505) 989-1073. See Newsletter 448, 36.

Missouri Magnetic Resonance Symposium (MMRS) and FACSS Meeting, Kansas City, MO, **Sept. 29 - Oct. 4, 1996**; Contact: (MMRS) Frank D. Blum, Dept. of Chemistry, Univ. of Missouri-Rolla, Rolla, MO 65409-0010; 573-341-4451 fblum@umr.edu. (FACSS) 198 Thomas Johnson Dr., S-2, Frederick, MD 21702-4317.

38th ENC (Experimental NMR Conference), Orlando, FL, **March 23 - 27, 1997**; Contact: ENC, 1201 Don Diego Avenue, Santa Fe, NM 87501; (505) 989-4573; Fax: (505) 989-1073.

Additional listings of meetings, etc., are invited.

**Department of Chemistry**
**Gordon House**
**29 Gordon Square**
**London WC1H 0PP**
**Tel: 0171-380-7471 (direct)**
**Fax: 0171-380-7464**

**Professor J. C. Lindon**

BIRKBECK COLLEGE
University of London

26 February 1996
(received 3/5/96)

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

Dear Barry,

**Protein-ligand interactions from diffusion coefficients**

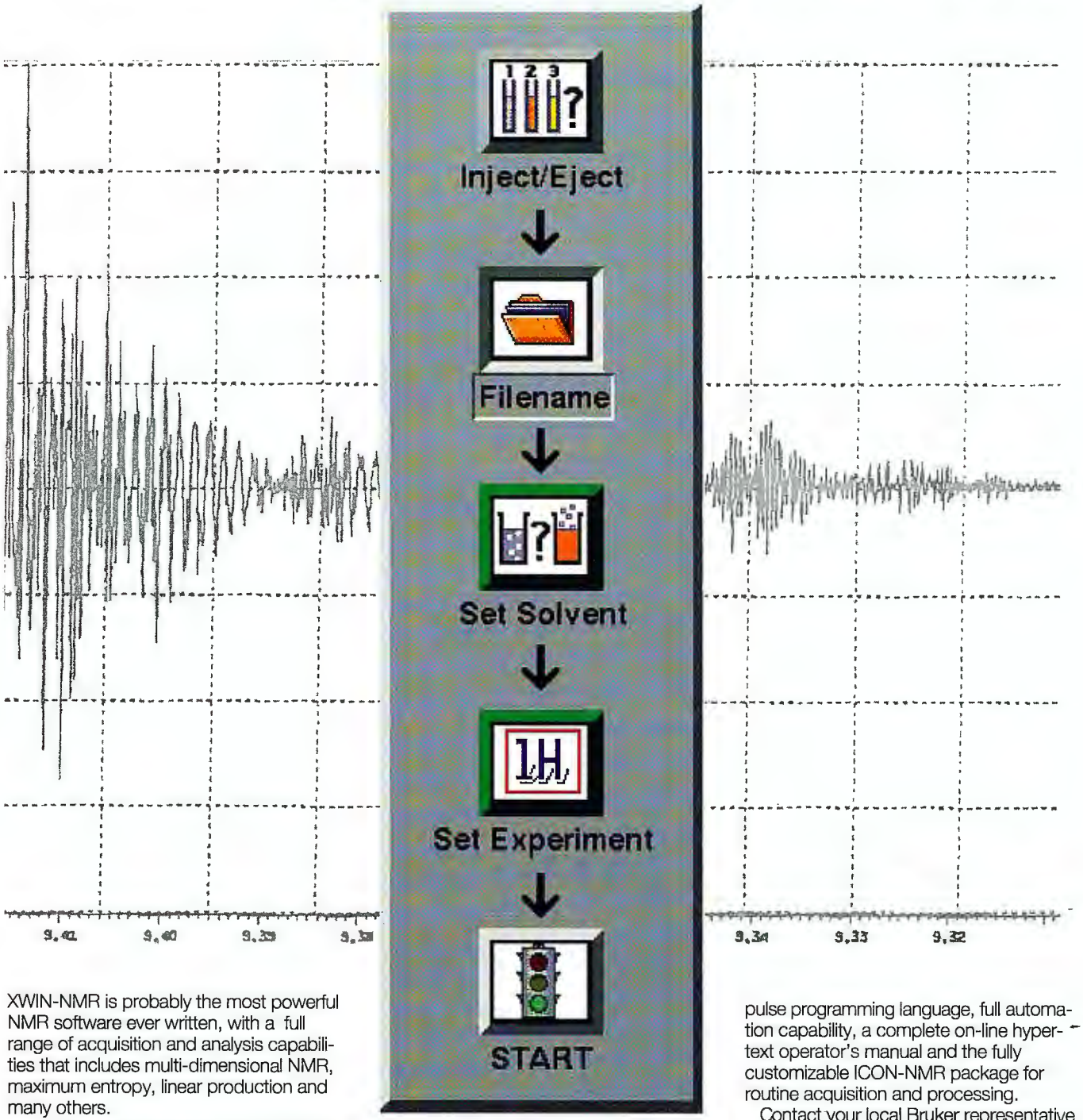
The measurement of molecular diffusion coefficients using NMR spectroscopy with gradients crops up in many areas of application from imaging of the brain after a stroke [Moseley *et al*, *Neurology*, **45**, A286 (1995)] to separating the resonances of endogenous metabolites in tissue extracts [Barjat *et al*, *J. Magn. Reson.*, **B108**, 170 (1995)]. We have been doing some experiments to measure diffusion coefficients both to provide spectral editing and to investigate intermolecular interactions. This has involved modifications to the now standard LED sequence [Gibbs and Johnson, *J. Magn. Reson.*, **93**, 395 (1991)].

We have incorporated bipolar gradients, shaped gradients and solvent suppression to enable us to measure diffusion coefficients of small molecule ligands which bind to macromolecules in the fast or intermediate exchange regime. For example the binding of 4-trifluoromethylbenzoic acid to human serum albumin has been studied using this method and fitting the observed diffusion coefficient for both the proton and fluorine resonances gives a dissociation constant of $2.2 \pm 0.3 \times 10^{-3}$ mol at 9 equivalent binding sites. The benefit of this approach is that extrapolation to the value for the diffusion constant for the fully bound ligand is not necessary since this is, to all intents and puposes, simply the value for the protein itself. The usual methods of monitoring changes in chemical shift or relaxation rate (either directly or by linewidth studies) all rely on either extrapolating, or fitting in the model, the values for the fully bound form. We have checked that by using the diffusion coefficient of albumin itself for the fully bound drug, it is posible to model the observed changes in chemical shifts and relaxation rates and obtain plausible values for bound shifts and relaxation rates. This work is currently being written up for publication.

Yours sincerely,

MAILI LIU JEREMY NICHOLSON JOHN C. LINDON

# It doesn't get any easier than this:

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March 6, 1996

(received 3/12/96)

Dr. B. L. Shapiro, Publisher
The NMR Newsletter
966 Elsinore Court
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**Exchange modulation of Spin Echoes**

Dear Barry,

Charles Pennington from Ohio State (private communication-papers to be submitted) as an outgrowth of his work on superconductivity has described the dynamics of spin correlations in spin echo experiments using his "super charged/superconducting approach" as well as the solution to the coupled Bloch equations for exchange between sites A and B:

$$\dot{M}_A = i\omega_0 M_A - \frac{1}{2\tau_1}(M_A - M_B)$$
$$\dot{M}_B = -i\omega_0 M_B - \frac{1}{2\tau_1}(M_B - M_A) \qquad (1)$$

where $\omega_0$ is the resonance frequency, $2\,\tau_1$ is the exchange time, and the magnetizations are expressed as $M_x + iM_y$.

We have generalized his approach by considering the "more complete" Bloch equations:

$$\dot{M}_A = -i(\omega_0 - \Delta)M_A - \frac{M_A}{T_2} - \frac{1}{\tau}(M_A - M_B)$$
$$\dot{M}_B = -i(\omega_0 + \Delta)M_B - \frac{M_B}{T_2} - \frac{1}{\tau}(M_B - M_A) \qquad (2)$$

where $2\Delta$ is the frequency difference of the two sites and $\tau$ is the exchange time.

Equations (2) are solved for the standard echo sequence, $\left(\frac{\pi}{2}\right)_x$, $(\pi)_y$ at time T, echo at time 2T (and beyond) to yield

$$\begin{pmatrix} M_A(2T+t) \\ M_B(2T+t) \end{pmatrix} = i\begin{pmatrix} a_{11}(T+t) & a_{12}(T+t) \\ a_{21}(T+t) & a_{22}(T+t) \end{pmatrix}\begin{pmatrix} a_{11}^*(T) & a_{12}^*(T) \\ a_{21}^*(T) & a_{22}^*(T) \end{pmatrix}\begin{pmatrix} M_A(0) \\ M_B(0) \end{pmatrix} \tag{3}$$

where $a_{11}(t) = \exp(-Gt)(\cos Rt + i\frac{\Delta}{R}\sin Rt)$, $a_{22}(t) = \exp(-Gt)(\cos Rt - i\frac{\Delta}{R}\sin Rt)$, and $a_{12}(t) = a_{21}(t) = \exp(-Gt)\sin Rt / R\tau$ with $G = T_2^{-1} + \tau^{-1} + i\omega_0$, and $R = \left(\Delta^2 - 1/\tau^2\right)^{1/2}$.

For $\tau^{-1} \to 0$ (no exchange) the solution reduces to

$$M_{Ay}(2T+t) + M_{By}(2T+t) = \left[M_A(0)\cos(\omega_0 + \Delta)t + M_B(0)\cos(\omega_0 - \Delta)t\right]\exp\left[-\left(T_2^{-1} + \tau^{-1}\right)(2T+t)\right] \tag{5}$$

as expected, whereas for finite values of $\tau$ but still in slow exchange, Eq. (3) contains terms modulated at frequency R and which allow an accurate determination of the exchange time from the modulation of the spin echo with time. Two examples, calculated for different exchange rates, of the time dependence of the $\cos\omega_0 t$ component of a spin echo from spin A where only $M_A$ is inverted are shown below. There is a clear difference in the modulation frequency. This approach has an advantage over linewidth measurements of exchange in that exchange rates between multiple sites are resolvable in principle.

Please credit this to the account of B. D. Nageswara Rao.

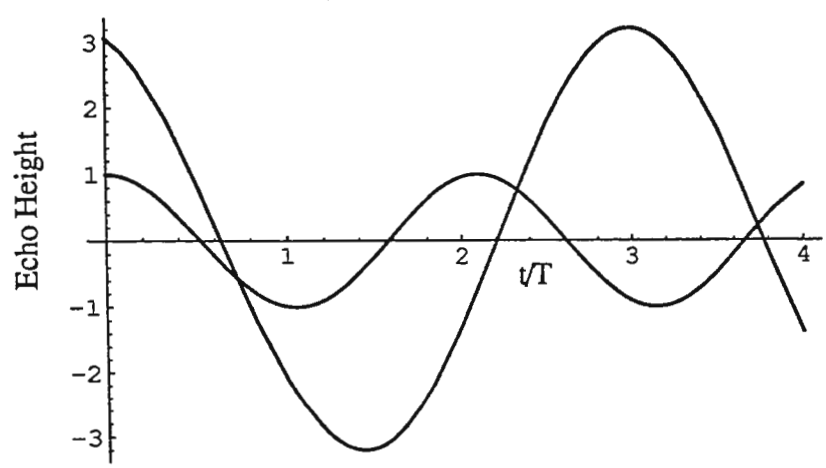
Best regards,

Lingyang Zhu

Jerry Kaplan

Marvin Kemple

Echo height of site A in terms of $M_A(0)$ versus time in units of T, the pulse separation, with damping removed. Parameters for the curve of smaller amplitude are $\tau^{-1} = 0$, and $\Delta T = RT = 3$. Parameters for the curve of larger amplitude are $T/\tau = \sqrt{5}$, $\Delta T = 3$, and $RT = 2$. Note that the smaller amplitude curve is oscillating at frequency $\Delta$.

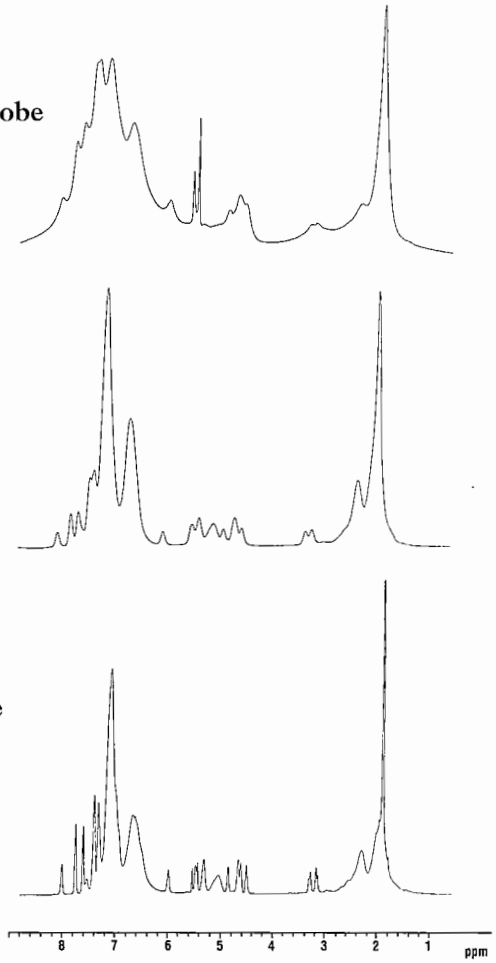


# Highest Resolution for Solid-Phase Synthesis

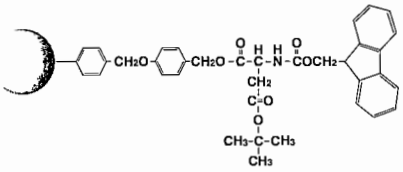
A. Vertical spinning probe

B. CP/MAS probe

C. $^{1}$H Nano•nmr probe




*$^{1}$H spectra of Fmoc-L-aspartic-ß(t-butyl) ester Wang resin in $CD_2Cl_2$ obtained utilizing a vertical spinning probe (A), a CP/MAS probe (B), and a $^{1}$H Nano•nmr probe (C). J. Magn. Reson. (1996), in press.*

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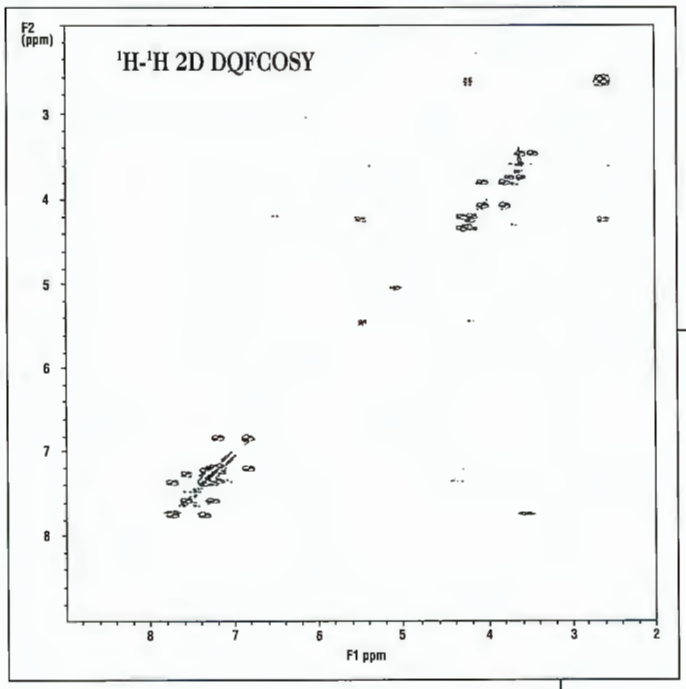
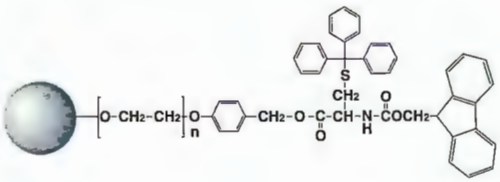
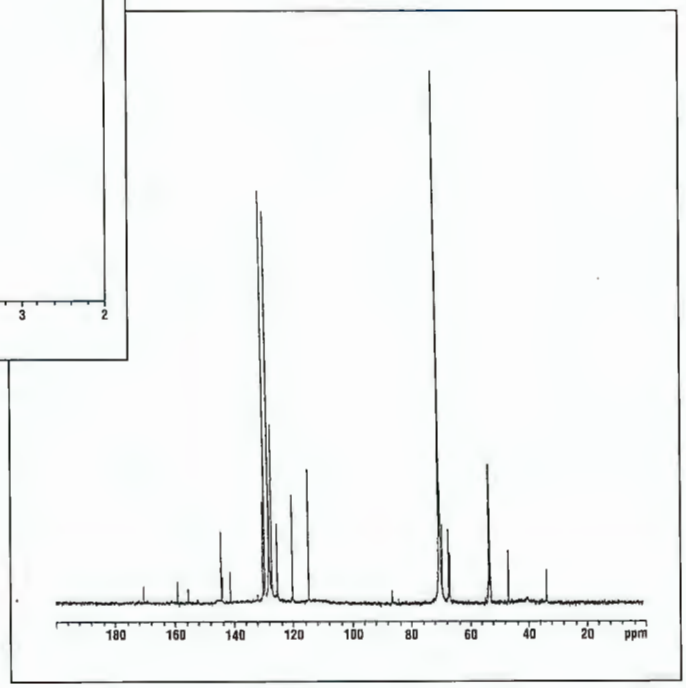
## High Resolution $^{1}H$ 2D Spectra from Solid-Phase Synthesis Gels




## High Resolution $^{13}C$ Spectra from Solid-Phase Synthesis Gels

Princeton University Department of Chemistry
Princeton, New Jersey 08544-1009

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Senior NMR Spectroscopist
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Prof. Bernard L. Shapiro
Editor/Publisher
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Palo Alto, CA 94303

February 29, 1996
(received 3/11/96)

**Re: $^{31}P,^{1}H$ - $COSY^{DD}$ of an RNA 24-mer**

Dear Barry,

Many thanks for your patience and understanding. As you can see one of us (I.P.) has relocated somewhat to the South recently. Since this move the Princeton area experiences exceptional weather conditions, such as weekly snowstorms, unusual cold, gusty winds, etc. Could this be transferred from Syracuse...?

In any case, we keep working together, as this letter shows, too. Our favorite molecule, a 24-mer RNA hairpin provides an opportunity to revive an old issue, namely fully dispersive phasing of COSY type peaks ($COSY^{DD}$). Such modified data processing has been shown to be advantageous (1,2) and offers enhanced sensitivity (virtually for free), simplified fine structure of cross peaks and easier analysis of connectivities (2) when compared to the conventional absorptive-absorptive, or *AA* approach.

The *DD* approach is useful for any spectra with partially overlapping antiphase peak patterns. We applied it to the $^{31}P,^{1}H$ - COSY spectrum (3) of the RNA hairpin sample (our appreciation to Prof. Heinz Rüterjans, whose courtesy allowed us to acquire the spectrum in his laboratory in Frankfurt, Germany, during the visit of I.P.). Correlation peaks in this spectrum consist of the usual patterns, which have antiphase structure along both dimensions. The attached Figure presents our spectrum with conventional *AA* phasing and that with *DD* phasing using identical contour levels (only positive levels for the *DD* spectrum). In the *DD* version the four-peak patters are simplified to a virtual singlet (with the exception of the 5', 5" correlation peaks, where the residual $^{2}J_{H,H}$ couplings are well visible and are of diagnostic value). One can appreciate the sensitivity increase by counting contour levels for any selected peak. Overlapping regions benefit most by the increased resolution. The assignments are shown with light gray over the spectra and match very well those published recently along with the results of preliminary structure refinement (4).

Sincerely, with our best regards,

Cai Songlin

Phil

István Pelczer
Princeton

SongLin Cai
Syracuse

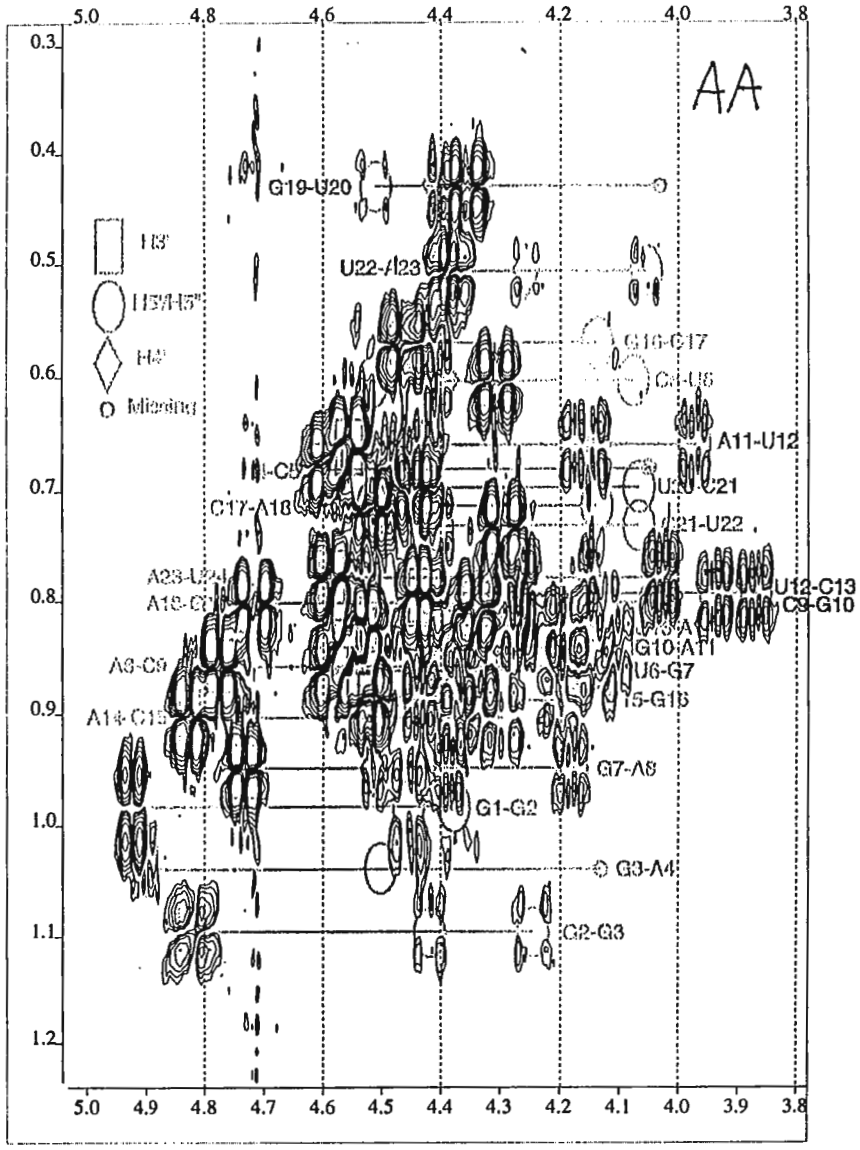
Philip N. Borer
Syracuse

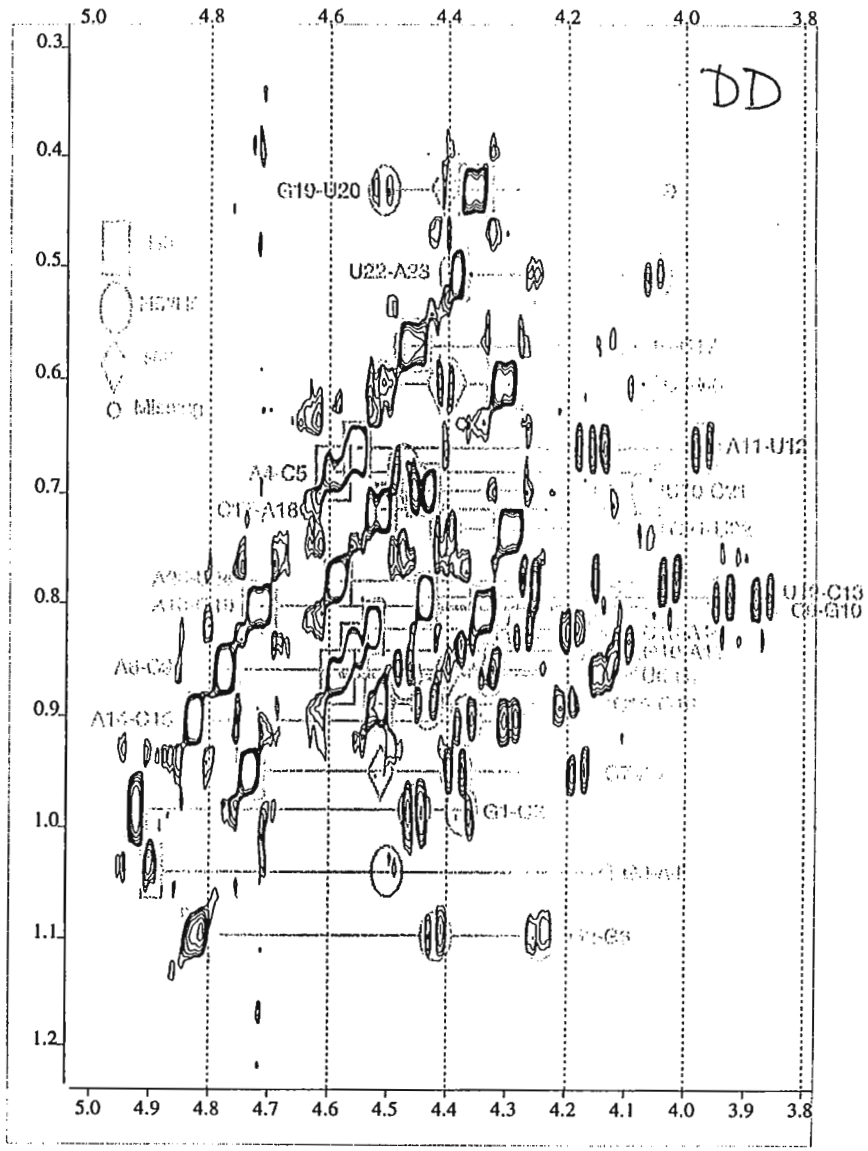
(1) Pelczer, I., Bishop, K. D., Levy, G. C., and Borer, P. N.; Modified Presentation of Double Quantum Correlation Spectra - Application to DNA Oligomers *J. Magn. Reson.*, 91(1991)604-606

(2) Pelczer, I., and Carter, B. G.; "Data processing in Multidimensional NMR" Chapter for the book: *Protein NMR Protocols*, in the Series: *Methods in Molecular Biology* (Ed.: D. G. Reid), Humana Press, NJ (in press)

(3) Sklenár, V., Miyashiro, H., Zon, G., Miles, H. T., and Bax, A.; Assignment of the $^{31}P$ and $^{1}H$ resonances in oligonucleotides by two-dimensional NMR spectroscopy *FEBS Lett.*, 208(1986)94-98

(4) Borer, P. N., Lin, Y., Wang, S., Roggenbuck, M. W., Gott, J., Uhlenbeck, O. C., and Pelczer, I.; Proton NMR and Structural Features of a 24-nucleotide RNA Hairpin *Biochemistry* 34(1995)6488-6503


AA
H3'
H5'/H5''
H4'
Missing
G19-U20
U22-A23
G16-C17
A11-U12
C17-A18
U12-C13
C9-G10
A3-C9
A14-C15
G7-A8
G1-G2
G3-A4
G2-G3


DD
G19-U20
U22-A23
A11-U12
A4-C5
C17-A18
U12-C13
C9-G10
A3-C9
A14-C15

# *Chemagnetics Technology Solid-State NMR Probes for Narrow-Bore Magnets*

*March, 1996*

Otsuka Electronics USA Inc.

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- Broadband Double Resonance. The decoupler channel tunes $^{1}H$-$^{19}F$, and the observe channel tunes over the range $^{31}P$-$^{15}N$.
- Variable-temperature equipped.
- Efficient. Signal-to-noise is high thanks to a large sample volume.

**Stable Spinning is achieved with a modification of the highly successful, patented Pencil™ rotor technology.**

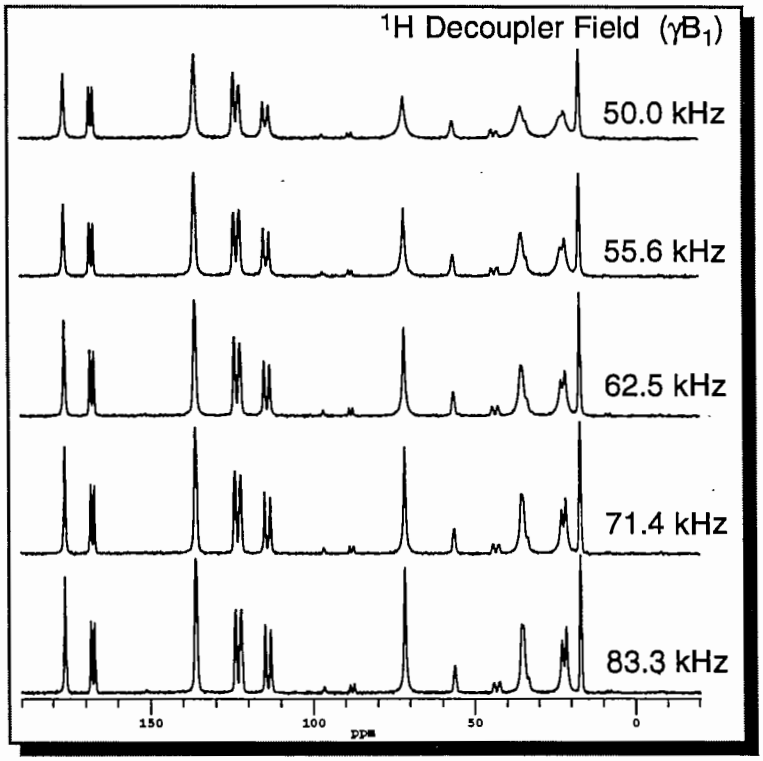
- Probes will be available with 3.2, 4.0, 5.0, and 6.0 mm spinning modules.
- Spinning speed specifications are comparable to Chemagnetics wide-bore probes.

**Sustainable Decoupling Powers are sizzling.**

- Don't give up power for a narrow-bore magnet. The new Chemagnetics 40 mm probe delivers decoupling RF fields comparable to wide-bore probes.

**Accessories will Allow you to Adapt the Chemagnetics Narrow-Bore probe to any Spectrometer.**

- All existing Chemagnetics MAS controllers interface to the narrow-bore probe.
- A narrow-bore VT stack is controlled by the Chemagnetics Variable Temperature controller.
- All of this hardware can operate independently of your spectrometer configuration.




**Demonstrated High Power $^{1}H$ Decoupling**

At left are five $^{13}C$ CP/MAS spectra of 4-butoxybenzoic acid, taken with different decoupling levels, using a CMX-400 Infinity. Note the resolution enhancement achieved only at high decoupler fields.

All spectra were acquired using the 40 mm diameter Chemagnetics double resonance MAS probe, fitted with a 5 mm spinning module. Spinning speed = 8.0 kHz; decoupling time = 25.6 ms; contact time = 2 ms. The probe fitted with 5 mm spinning module is capable of 83 kHz decoupling level. The power available from a $^{1}H$ amplifier will determine achievable proton decoupling level.

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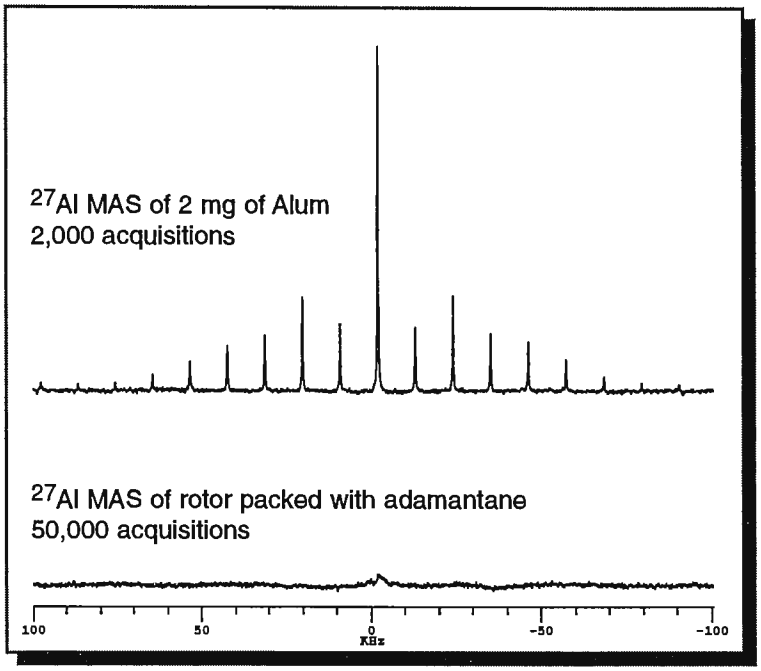
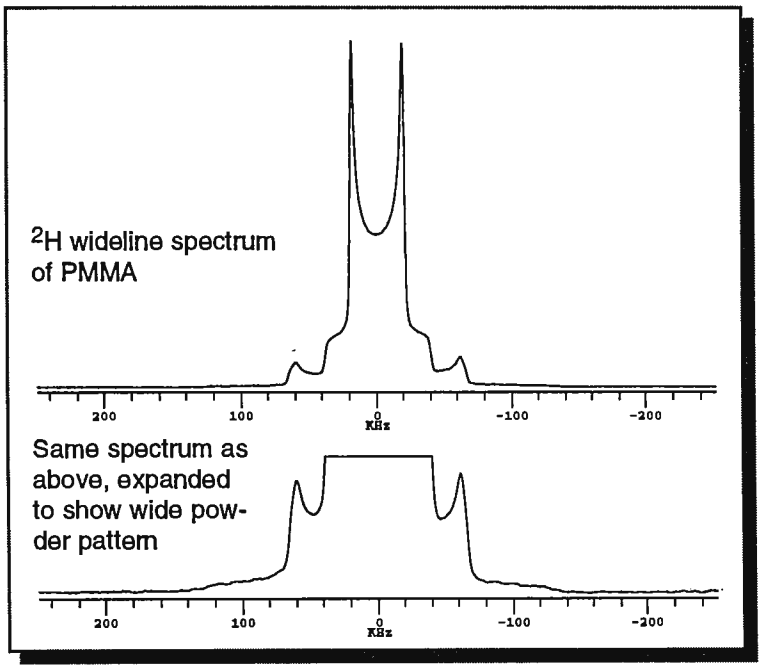
# *Chemagnetics Narrow-Bore Probe Technology*

### Low $^{27}$Al Background in 40 mm Probes

Our probe engineers take great care to limit background signals in all our probes. Our zirconia supply for rotor material is guaranteed to be very low in aluminum, and we strive to eliminate aluminum in other probe components.

The spectra on the right were acquired on a CMX-400 Infinity fitted with a 40 mm probe, and 5 mm spinning module. Pulse width (solid $\pi/2$) = 2.0 µs; recycle delay = 0.1 s; sweep width = 200 kHz; spinning speed = 11 kHz.




### Large Bandwidth

Although one should consider a separate, dedicated probe for extensive wideline work, the spectra on the left demonstrate that the 40 mm MAS probe has sufficient bandwidth for demanding MAS work and for some wideline work. The $^{2}$H spectrum of PMMA was acquired on a CMX-400 Infinity using a 40 mm, double-resonance, Chemagnetics MAS probe fitted with a 5 mm spinning module. Pulse width = 2 µs; spectral width = 500 kHz; recycle delay = 30 s.

Probe Specifications: 40 mm diameter; 3.2, 4.0, 5.0, or 6.0 mm Vespel spinning module
Spinning Speed: (5mm module) 1 - 12 kHz
Decoupler Field (with Chemagnetics high power amplifiers, 5 mm module): 83 kHz
Operating Frequency Range: Decoupler Channel - $^{1}$H-$^{19}$F, Observe Channel $^{31}$P-$^{15}$N

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UNIVERSITY OF MISSOURI-ROLLA
*Missouri's Technological University*

Frank D. Blum
Department of Chemistry
142 Schrenk Hall
Rolla, Missouri 65409-0010
(573)-341-4451 (or 4420)
fblum@umr.edu

March 9, 1996
(received 3/14/96)

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

Dear Barry:

# - ANNOUNCING -

**MMRS-6**
Missouri
Magnetic
Resonance
Symposium

***Industrial Applications of NMR***
***Nuclear Magnetic Resonance Diffusion Measurements***
***Peptide/Protein Characterization with NMR Spectroscopy***
***Poster Session***

This year, the 6th Missouri Magnetic Resonance Symposium (MMRS-6) will be held jointly with the 23rd Federation of Analytical Chemistry and Spectroscopy Societies (FACSS XXIII) Meeting from Sept. 29 - Oct. 4, 1996 in Kansas City, MO, USA at the H. Roe Bartle Convention Center. The MMRS sessions are tentatively scheduled from Tuesday (AM) through Wednesday (PM). The preliminary schedule is attached.

Contributed papers from MMRS participants are now being solicited for this symposium in all areas of magnetic resonance. **Full time students giving posters in the MMRS poster session** (probably Wed. PM) **are eligible to apply for a stipend ($35) to cover registration costs.** To apply, a copy of the abstract of the poster presentation should be sent to: Frank D. Blum, Department of Chemistry, University of Missouri-Rolla, Rolla, MO 65409-0010, 573-134-4415, fblum@umr.edu. There are a limited number, so apply soon.

Registration for the meeting will be handled through FACSS. For registration and abstract materials, contact: FACSS National Office, 201-B Broadway St., Frederick, MD, 21701-6501, USA (301-846-4797, FAX 301-694-6860).

I would be pleased for you to share this information with your readers.

Sincerely,

Frank

Frank D. Blum
Curators' Professor of Chemistry
and Senior Investigator
Materials Research Center

P.S. As I write, the Missouri Miners basketball team is about to play in the finals of the first round NCAA Division II regional tournament. If we win, we go to the sweet 16 in Louisville for the national championship. This is noteworthy because at *Missouri's Technological University*, it is not possible to major (*i.e.* graduate) in anything that does not require significant intellectual ability and commitment. In contrast to most athletic programs these days, we really have *student- athletes*. I am very proud of them. I'm off to the game, **GO MINERS!**

# MMRS-6 and FACSS-23
# September 29 - October 24, 1996

## Industrial Applications of NMR
Patrick Smith, Dow Chemical, Organizer, Presiding

**H. N. Cheng**, Hercules, NMR Studies of Polymer Reactions.
**Betsy McCord**, DuPont, NMR Determination of Microstructure in Novel Ethylene and Alfa-Olefin Polymers.
**Dennis Hasha**, Dow, Characterization of Metallocene Catalysts.
**Warren T. Ford**, Oklahoma State, Solid State $^{13}C$ NMR Analysis of Hypercrosslinked Polystyrene.
**Pat Smith**, Dow Chemical, Characterization of Crosslinking in Ion-exchange Resins.
**Fred Schilling**, ATT, Structural Characterization of Inorganic Glasses by Solid-State NMR.
**Joel Garbow**, Monsanto, Polymers of Substituted N-Phenylnorbornene-5,6-dicarboxamide: Characterization of Structure and Dynamics.
**Mike Hewitt**, Kodak, Remote Access to NMR Spectrometers.
**Claire Conboy**, Dow, Water Suppression Techniques.
**Henry Yue**, Dow Corning, An Industrial Application of NMR Microimaging for Porosity Deretmination in Ceramic Matricies.
**Frank Blum**, U. Missouri-Rolla, Dynamics of Adsorbed Poly(vinyl acetate).

## Nuclear Magnetic Resonance Diffusion Measurements
Cynthia K. Larive, U. Kansas, Organizer, Presiding

**Charles S. Johnson, Jr.**, U. North Carolina, Multidimensional Diffusion Ordered NMR: Promise and Limitations.
**Cynthia K. Larive**, U. Kansas, Examination of Peptide Aggregation using Diffusion Measurements.
**Brian Antalek**, Kodak, Diffusion and Scaling Behavior of Polymer-Surfactant and Polymer-Protein Aggregates.
**Peter Stilbs**; Royal Institute of Technology, Sweden, Polymer-surfactant Interaction as Studied by FT-PGSE NMR Self-Diffusion Methods.
**Lucio Frydman**, U. Illinois-Chicago, Pulse-Gradient, Spin-Echo Studies of Anisotropic Diffusion in Liquid-Crystalline Phases.

## Peptide/Protein Characterization with NMR Spectroscopy
Cynthia K. Larive, U. Kansas, Organizer, Presiding

**Dallas Rabenstein**, U. California-Riverside, NMR Studies of the Binding of Peptides by Heparin.
**W. Robert Carper**, Wichita State U., Characterization of the Active Site of Gluconolactonase using $^{113}Cd$ and other Divalent Metals as Catalytic Probes.
**Steven Van Doren**, U. Missouri-Columbia, NMR Structural Studies of a Metalloproteinase and its Inhibitor.
**John Likos**, Monsanto, Multi-Dimensional NMR Studies of Oncomodulin, a B-Parvalbumin.
**Melissa Starovasnik**, Genetech, The E-domain of Staphylococcal Protein A: Solution Structure and Fv-binding Surface.
**Jim Satterlee**, Washington State U., Solution Structures and Dynamics of Heme Protein"

## MMRS Poster Session
Frank Blum, Organizer, Presiding

To be determined.

**For more information contact**: Frank D. Blum, Department of Chemistry, University of Missouri-Rolla, Rolla, MO 65409-0010, 573-134-4415, fblum@umr.edu.

# Set your site on Oxford Instruments

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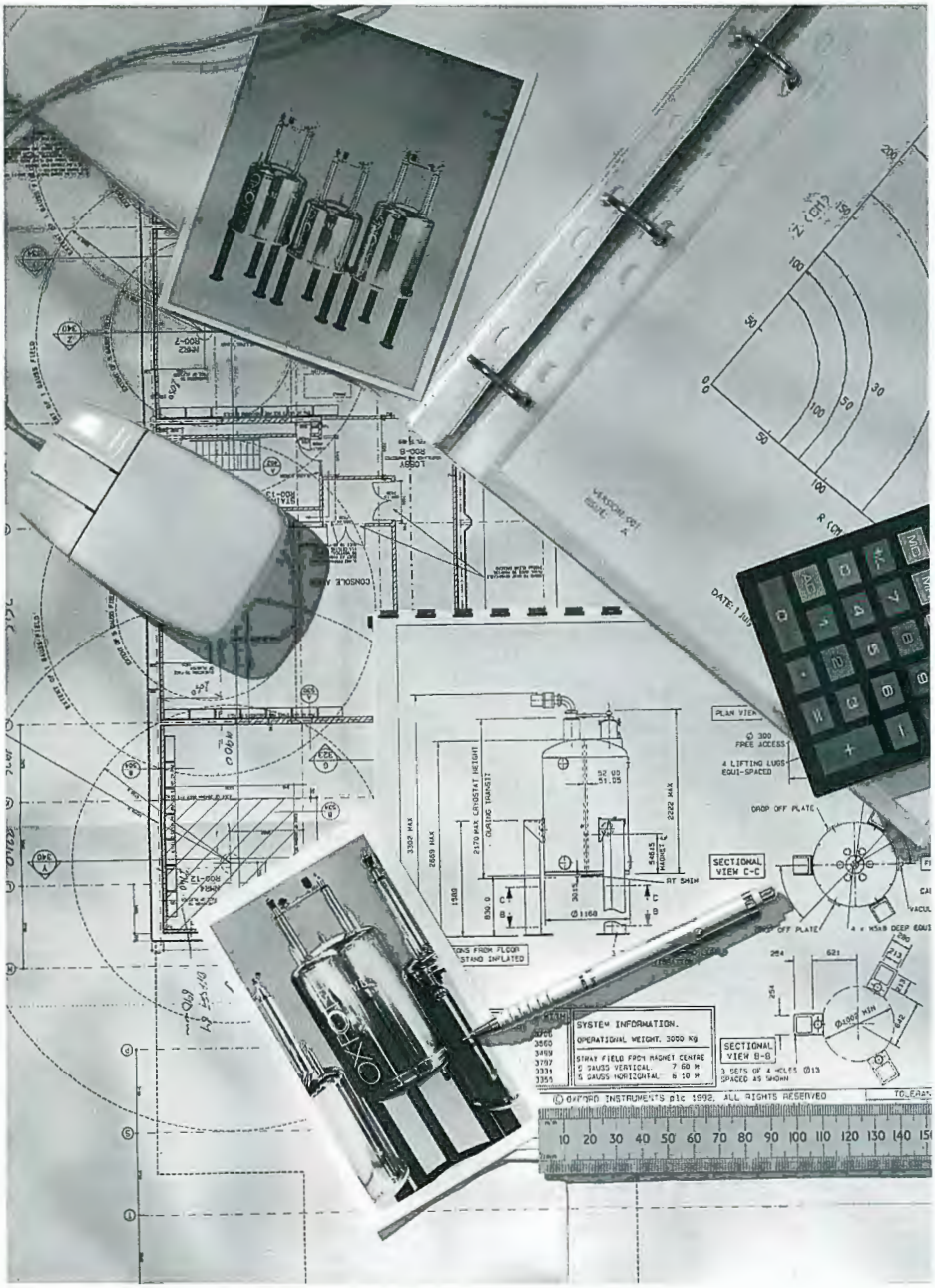


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# *The Oxford Instruments Heritage*

Oxford Instruments are the pioneers of NMR magnet systems and associated cryogenic technology. After more than 30 years, we are still leading the way maintaining our worldwide reputation for transforming scientific ideas into usable, practical technology:

- Oxford Instruments were the first company to introduce NMR quality super-conducting magnets at 400, 500, 600 and 750 MHz.
- We designed and built the world's first compact superconducting storage ring for X-ray lithography.
- 20 Tesla magnets are routinely produced for physics research.

Making this happen are the people of Oxford Instruments, their expertise and dedication makes them our greatest asset and a unique resource for our customers.

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# *Standard specifications*

| Magnetic field Strength ($^1$H-MHz) | Room Temperature Bore Diameter (mm) | Field Stability ($^1$H-Hz/Hour) | Maximum Helium Refill Interval (Days) | Minimum Operational Ceiling Height (m) |
|---|---|---|---|---|
| 750 | 51 | 15 | 60 | 3.8 |
| 600 | 51 | 10 | 120 | 3.4 |
| 500 | 51 | 10 | 150 | 3.2 |
| 400 | 54 | 8 | 365 | 2.8 |
| 360 | 54 | 8 | 365 | 2.8 |
| 300 | 54 | 3 | 365 | 2.8 |
| 270 | 54 | 2.7 | 365 | 2.8 |
| 200 | 54 | 2 | 365 | 2.8 |
| 100 | 54 | 1 | 365 | 2.8 |
| 500 | 89 | 15 | 120 | 3.4 |
| 400 | 89 | 10 | 180 | 2.8 |
| 360 | 89 | 10 | 365 | 2.8 |
| 300 | 89 | 3 | 365 | 2.8 |
| 270 | 89 | 2.7 | 365 | 2.8 |
| 200 | 89 | 2 | 365 | 2.8 |
| 100 | 110 | 1 | 119 | 2.8 |

**We would be delighted to discuss your custom specification requirements for any specialist systems. For more information please contact your local Oxford Instruments sales and service organisation.**


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$B_0$ gradients: A spectroscopist's best friend?

Wissembourg, March 7th ,1996

(received 3/20/96)

Dear Barry,

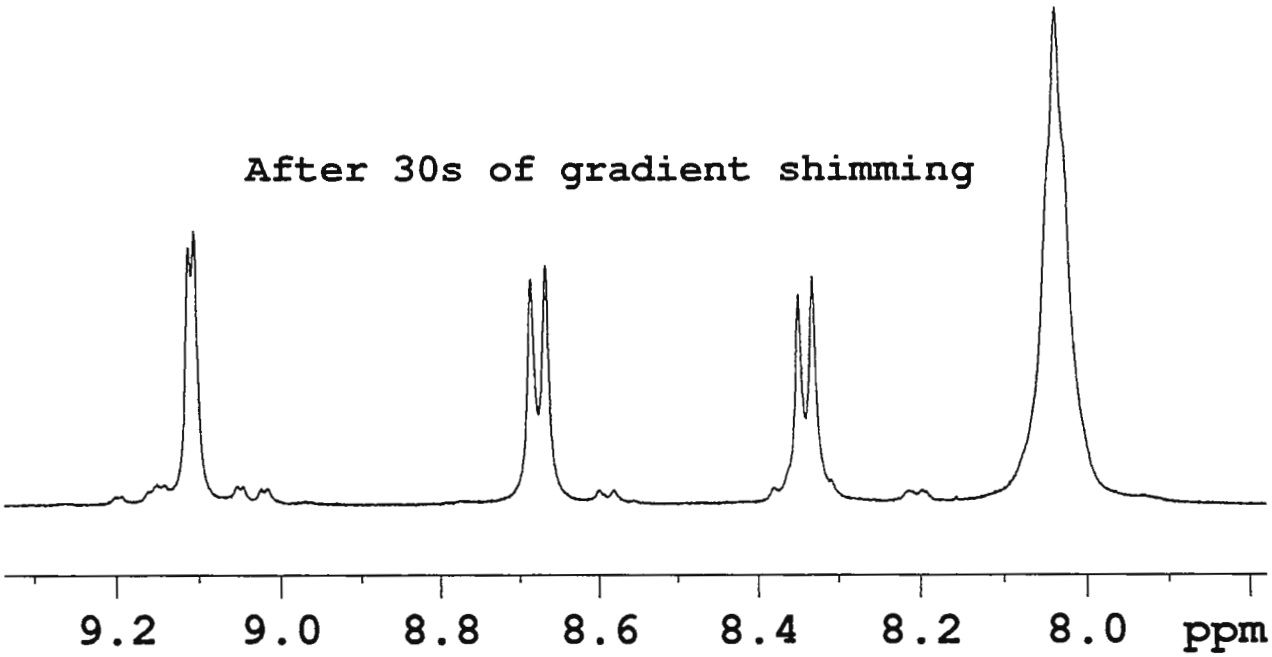
$B_0$ pulsed field gradients have become part of the standard techniques of the high resolution NMR spectroscopist. They are used in almost all experiments and people have now become accustomed to inverse experiments showing no ridges.
Recently, $B_0$ pulsed field gradients have shown that they can do even more for the quality of life of the human being behind the spectrometer. They can do in a matter of minutes what used to take hours depending on the weather or the mental condition of the user. They can shim!
For the lucky ones who work on proteins in $H_2O$, three axes gradients can use the very strong water signal to map exaclty the $B_0$ inhomogeneity inside the RF coil. The result of the investigation is then fed to a computer that will compute the optimum solution to the problem. In the mean time, people can stare at the process on the screen or go and have one of their favorite drinks.
For people who work on samples in deuterated solvents where the residual solvent peak is not as strong, one axis gradient can shim the sample along the most troublesome direction (the Z axis) and provide in less than a minute optimum shims (no time for coffee this time).
As an illustration we show below two spectra recorded on our favorite molecule (50 mM Gramicidine). The first one recorded immediately after introduction inside the magnet, the second after 30s of gradient shimming.
We might wonder what novelty gradients will bring to us next....

Sincerely,

M. PIOTTO

C. BREVARD

Christian



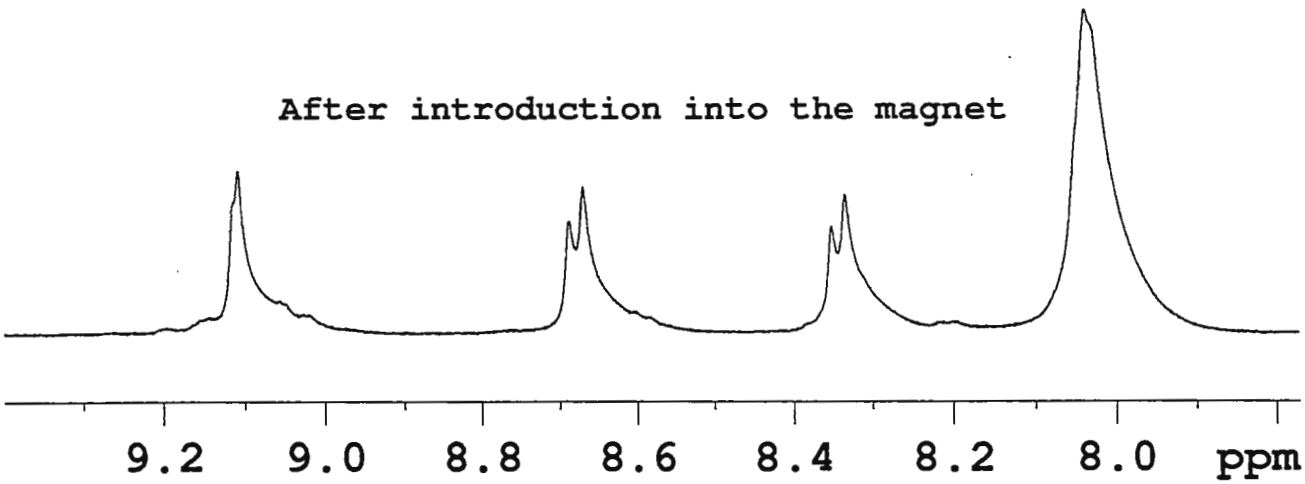




Figure1: DRX 500 1D spectrum of 50 mM Gramicidine

NEW

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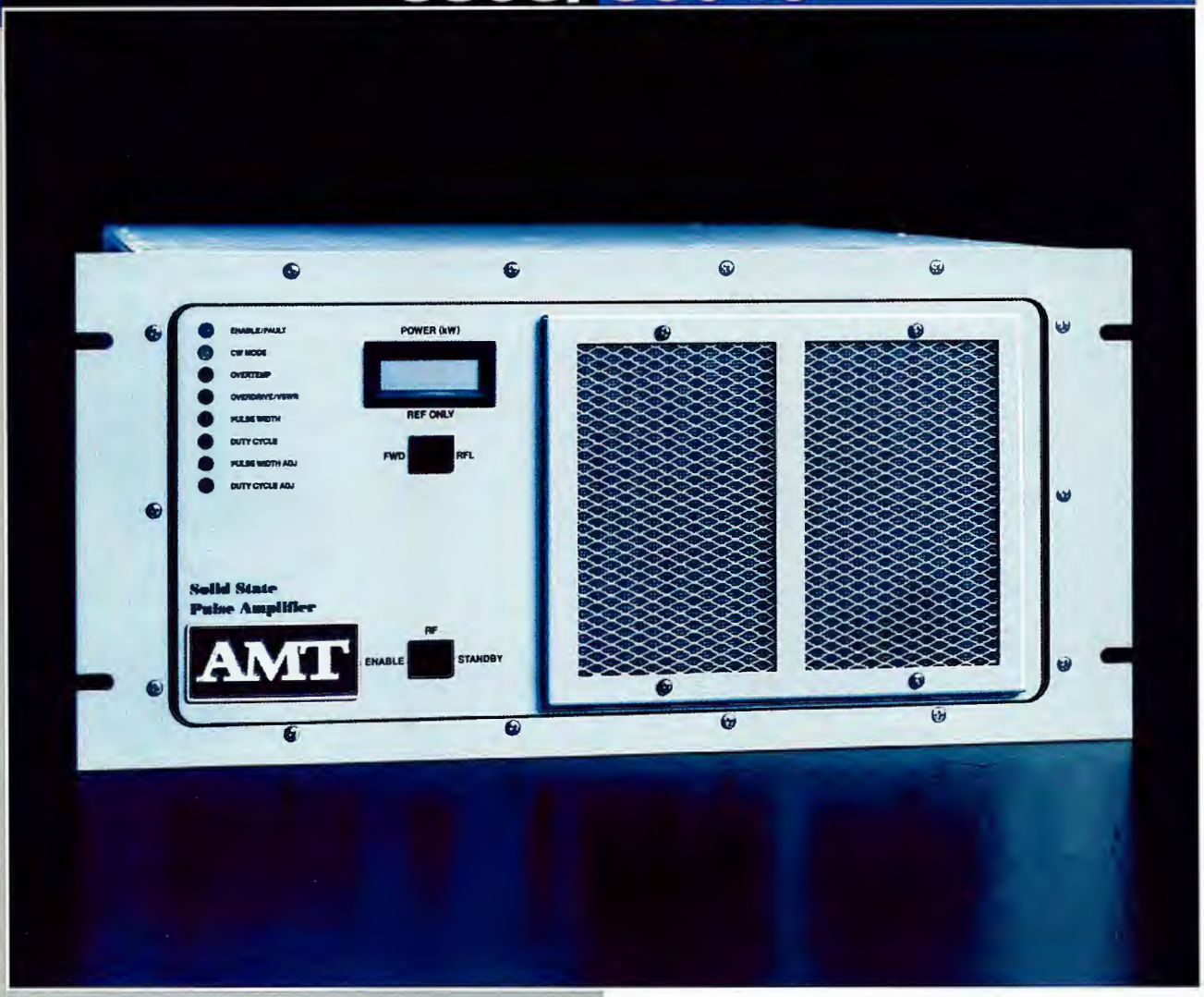


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|---|---|---|
| Frequency range | 30-310 MHz | 30-310 MHz |
| Pulse power into 50 ohms | 700 W | 400 W |
| CW power into 50 ohms | 70 W max. | 40 W max. |
| Linearity (±1.5 dB from linear) | 0-500 W | 0-300 W |
| Amplitude rise/fall time | 250 ns typ. | 250 ns typ. |
| Amplitude droop | 5% to 10 ms pulse width | 5% to 10 ms pulse width |
| Phase change/output power | 10° typ. to rated power | 10° typ. to rated power |
| Phase error over pulse | 4° to 10 ms duration, typ. | 4° to 10 ms duration, typ. |
| Harmonic levels | -25 dBc | -25 dBc |
| Gain (0 dBm input) | 65 dB ± 5 dB | 62 dB ± 5 dB |
| Input/output impedance | 50 ohms | 50 ohms |
| Input VSWR | <2:1 | <2:1 |
| Pulse width | 10 ms, 100 ms w/long pulse | 10 ms, 100 ms w/long pulse |
| Duty cycle | Up to 10% | Up to 10% |
| Noise figure | 8 dB typ. | 8 dB typ. |
| Output noise level (blanked) | <20 dB over thermal | <20 dB over thermal |
| Blanking delay | <1 μs "ON", 1 μs "OFF", TTL signal | <1 μs "ON", 1 μs "OFF", TTL signal |
| Protection | • VSWR: infinite VSWR at rated power<br>• Over duty cycle/pulse width<br>• Over temperature<br>• Output overdrive | • VSWR: infinite VSWR at rated power<br>• Over duty cycle/pulse width<br>• Over temperature<br>• Output overdrive |
| Cooling | Internal forced air | Internal forced air |
| Operating temperature | Ambient: +10° to +40°C | Ambient: +10° to +40°C |
| Line voltage | 208/230 VAC, ±10%, 1 Ø, 47-63 Hz | 208/230 VAC, ±10%, 1 Ø, 47-63 Hz |
| AC power requirements | 1200 VA | 850 VA |
| Package | 19 inch rack mount | 19 inch rack mount |
| Size (HWD) | 8.75 x 19.0 x 24.0 inches<br>222 mm x 483 mm x 610 mm | 8.75 x 19.0 x 24.0 inches<br>222 mm x 483 mm x 610 mm |
| Panel height | 10 inches, 254 mm | 10 inches, 254 mm |
| Unit weight | 90 lbs., 41 kg | 75 lbs., 34 kg |
| Shipping Weight | 135 lbs., 62 kg | 120 lbs.,45 kg |



## SUPPLEMENTAL CHARACTERISTICS:

**Connectors, rear panel**

- RF input: BNC (F)
- RF output: Type N (F)
- Noise blanking: BNC (F)
- Interface: 25 pin D (F), EMI filtered

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- Digital peak power meter
- CW mode
- Over pulse width/duty cycle
- Power On/Standby
- Over temperature

**System monitors**

- Forward/Reflected RF power
- Over pulse width/duty cycle
- DC power supply fault
- Thermal fault

**Front panel controls**

- Pulse width
- Duty cycle
- Forward/Reflected power
- RF enable/standby

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# NIJMEGEN SON RESEARCH INSTITUTE

FOR MOLECULAR STRUCTURE, DESIGN AND SYNTHESIS

Son National
HF-NMR Facility
Toernooiveld 1
6525 ED Nijmegen

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

(received 3/22/96)
Nijmegen, 03/15/96

## THERMAL STABILITY OF SILICON-BASED HYBRID MATERIALS

Dear Dr. Shapiro,

In cooperation with the Philips Research Laboratories we have been studying combined organic and inorganic materials. Much of this work involves $^{29}$Si NMR to study the inorganic network formation. Unfortunately, we found that we can not use Cross-Polarization because part of the $Q_4$ sites remain invisible in the CP experiment due to the large proton-Si($Q_4$) distances in very condensed materials. Therefore we have to resort to Single Pulse Excitation (SPE) experiments, these become very time-consuming, however, because of the large spin lattice relaxation times. With the upgrade of our CXP 300 spectrometer to a DMX 300 we purchased Brukers 10mm MAS probehead, which proves to be very convenient for these studies. It readily spins 5 kHz which is adequate for the systems under study, and the experiment time was reduced by a factor of 4 compared to a 7mm probehead, as one would expect on the basis of the increased volume. Using this probehead we got some striking results studying the thermal stability of the hybrid materials.

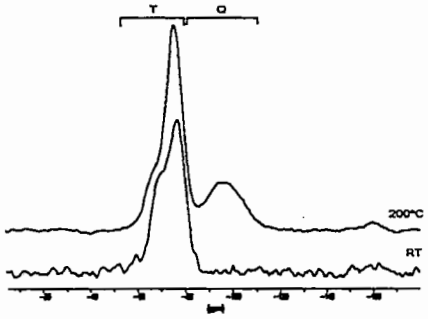



Figure 1: $^{29}$Si MAS NMR spectra of a 0.80 PhTES / 0.20 Al(OBu$^s$)$_2$EAA sample before and after a heat treatment at 200°C

Addition of Al(OBu$^s$)$_2$EAA to PhTES (phenyl triethoxysilane) or Glymo (3-glycidoxypropyl trimethoxysilane) containing TEOS-based materials leads to a decreased thermal stability of the trifunctional siloxanes. $^{29}$Si MAS NMR shows that breaking of the Si-C bond and formation of an extra Si-O bond occurs after heating to 200°C for these materials (Figure 1). Before heat treatment resonances of $T_2$ and $T_3$ sites can be observed (-60 - -80 ppm). Heat treatment leads to the formation of a broad resonance centered at -100 ppm, the region

National HF-NMR Facility
FAX xx31-24-3652112

Dr. S.S. Wijmenga, supervisor biomolecular NMR, tel. xx31-24-3653884/3652885, e-mail sybren@nmr.kun.nl
Dr. A.P.M. Kentgens, supervisor solid-state NMR, tel. xx31-24-3652078/3652369, e-mail arno@solidmr.kun.nl

NSR Center

SON National HF-NMR Facility
Nijmegen

normally ascribed to Q-atoms. MeTES containing materials are stable up to (at least) 200°C. $^{27}$Al MAS NMR spectra show the presence of tetrahedral species which are thought to be incorporated in the network, and octahedrally coordinated (non-network) aluminum which serve as charge-balancing cations. Preparation of samples using $Na[AlOR_4]$ instead of $Al(OBu^s)_2EAA$ in order to replace some of the octahedrally coordinated aluminum by sodium (figure 2) shows that the undesired decomposition reaction is caused by octahedral (non-network) aluminum. Up to 4-5 Si-C bonds are broken per octahedral aluminum, indicating a catalytic effect.

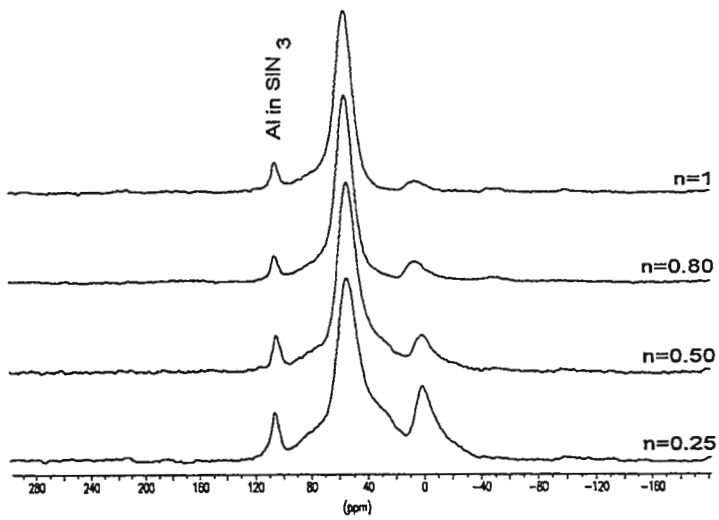



Figure 2: $^{27}$Al MAS NMR of 0.42 PhTES / 0.38 TEOS / 0.20 $Al(OBu^s)_3$ samples after heating to 200°C ( $n$= $NaOEt/Al(OBu^s)_3$ ratio). (MAS speed 13.0 kHz). The signal at 105 ppm is an impurity in the rotor material

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

Sincerely yours,

Mart Peeters

Ingrid Snijkers-Hendrickx

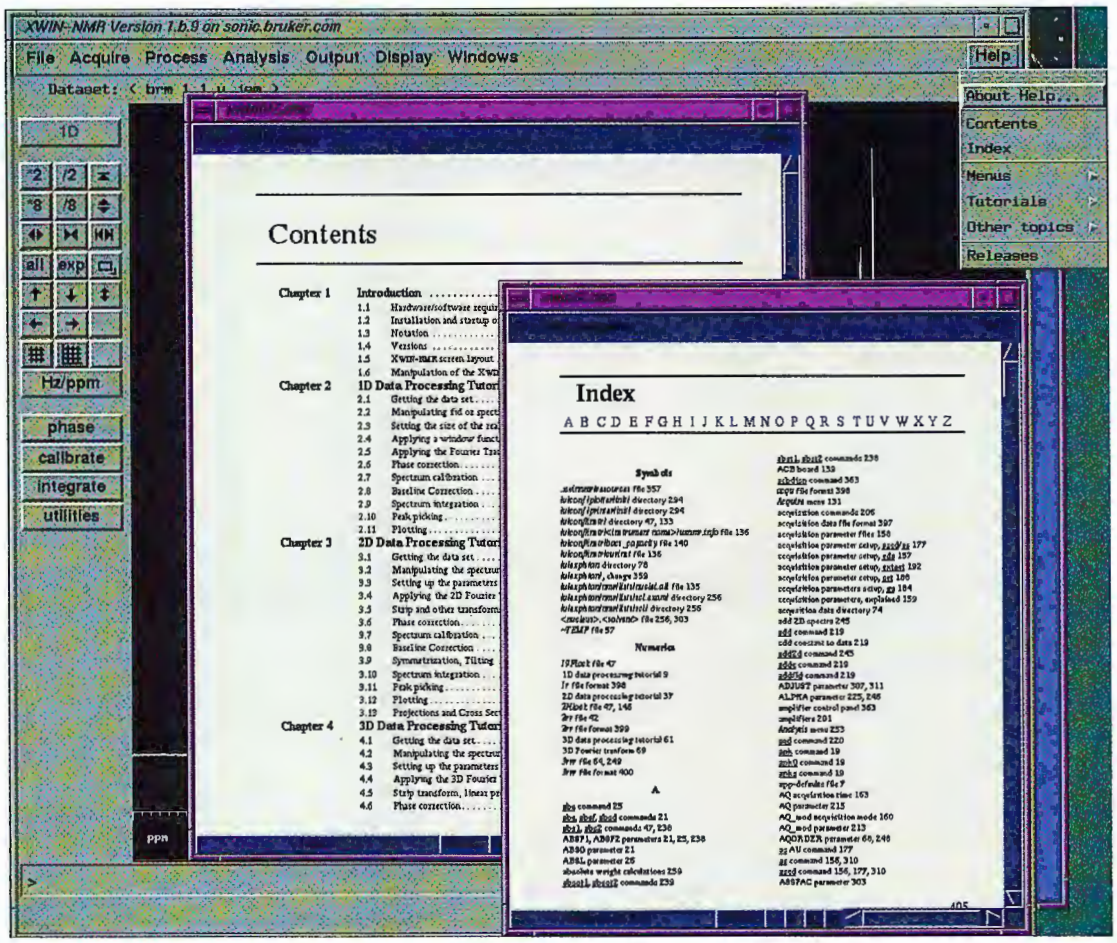
Arno Kentgens

The NMR Revolution advances...

BRUKER

# XWIN-NMR™ Software:

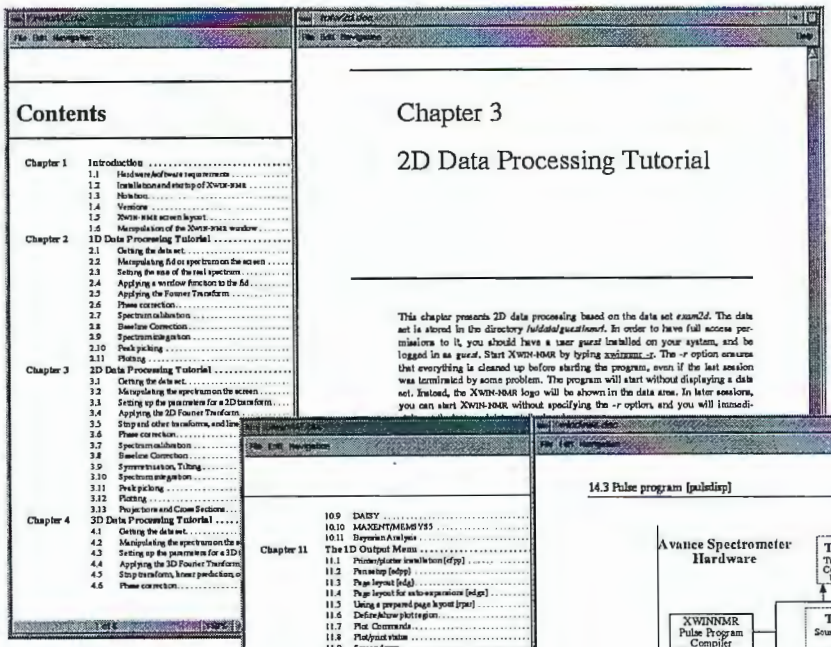
## *On-line help is just a click away!*




Bruker's new NMR software package, *XWIN-NMR™*, comes complete with an **on-line** manual. *XWIN-NMR™* uses the Frame-Viewer utility to display the manual. Additionally, the table of contents and a keyword index for the manual are organized as hypertext for fast display of a desired item. Now "help" is just a click away!

# ...The NMR Revolution advances

Here are some pages from the on-line manual, showing the Table of Contents that was used to find the page.



Contents

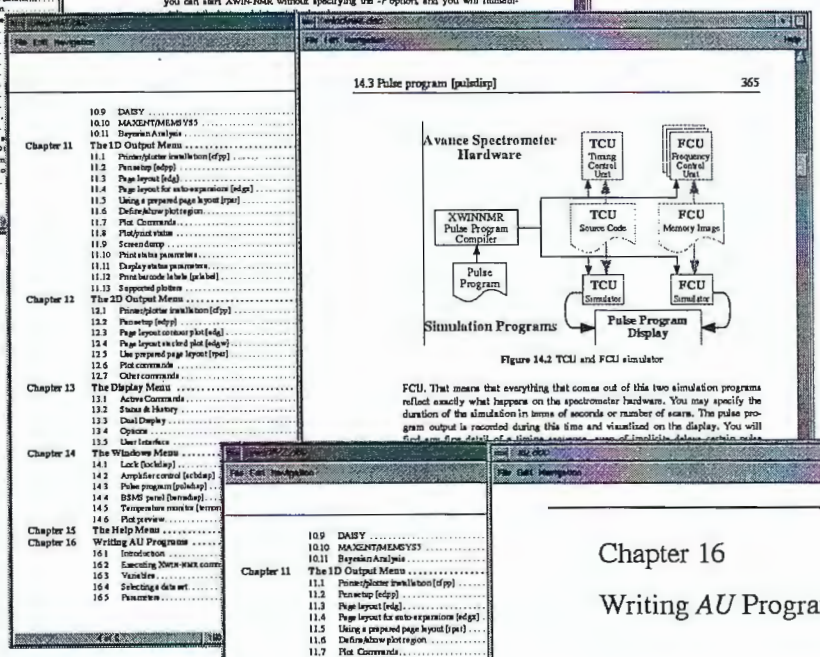
Chapter 1 Introduction
Chapter 2 1D Data Processing Tutorial
Chapter 3 2D Data Processing Tutorial
Chapter 4 3D Data Processing Tutorial

Chapter 3

2D Data Processing Tutorial

This chapter presents 2D data processing based on the data set *exam2d*. The data set is stored in the directory */u/data/guest/nmr/*. In order to have full access permissions to it, you should have a user *guest* installed on your system, and be logged in as *guest*. Start XWIN-NMR by typing xwinnmr -r. The *-r* option ensures that everything is cleaned up before starting the program, even if the last session was terminated by some problem. The program will start without displaying a data set. Instead, the XWIN-NMR logo will be shown in the data area. In later sessions, you can start XWIN-NMR without specifying the *-r* option, and you will immedi-

**Top:** Shows the tutorial help available with *XWIN-NMR*™ for 2D processing.



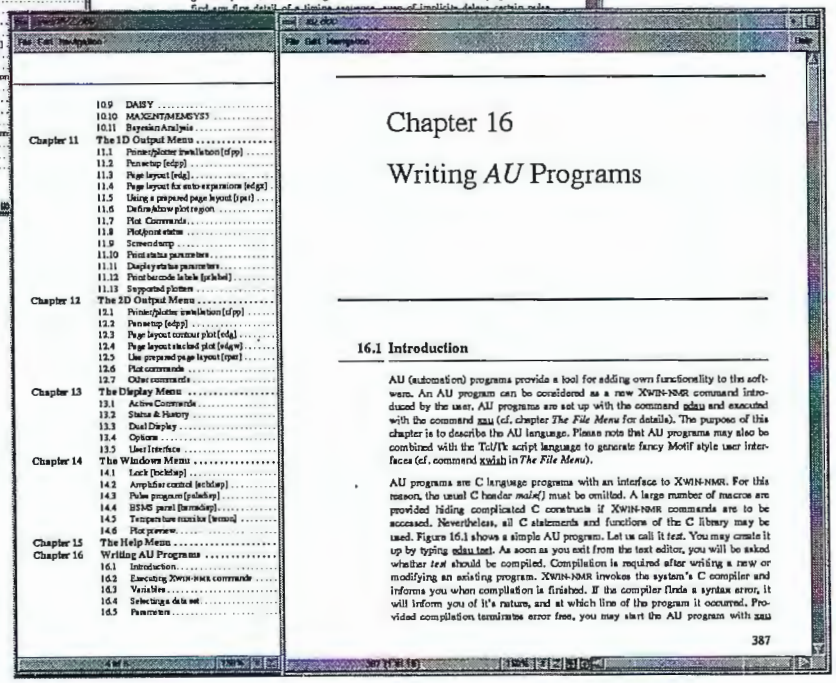
14.3 Pulse program [pulsdisp] 365



Figure 14.2 TCU and FCU simulator

FCU. That means that everything that comes out of this two simulation programs reflect exactly what happens on the spectrometer hardware. You may specify the duration of the simulation in terms of seconds or number of scans. The pulse program output is recorded during this time and visualized on the display. You will

**Middle:** Explains functionality of the pulse program display software.



Chapter 16

Writing *AU* Programs

16.1 Introduction

AU (automation) programs provide a tool for adding own functionality to the software. An AU program can be considered as a new XWIN-NMR command introduced by the user. AU programs are set up with the command edau and executed with the command xau (cf. chapter *The File Menu* for details). The purpose of this chapter is to describe the AU language. Please note that AU programs may also be combined with the Tcl/Tk script language to generate fancy Motif style user interfaces (cf. command xwish in *The File Menu*).

AU programs are C language programs with an interface to XWIN-NMR. For this reason, the usual C header *main()* must be omitted. A large number of macros are provided hiding complicated C constructs if XWIN-NMR commands are to be accessed. Nevertheless, all C statements and functions of the C library may be used. Figure 16.1 shows a simple AU program. Let us call it *test*. You may create it up by typing edau test. As soon as you exit from the text editor, you will be asked whether *test* should be compiled. Compilation is required after writing a new or modifying an existing program. XWIN-NMR invokes the system's C compiler and informs you when compilation is finished. If the compiler finds a syntax error, it will inform you of it's nature, and at which line of the program it occurred. Provided compilation terminates error free, you may start the AU program with xau

387

**Bottom:** Explains the fundamentals of writing and using AU programs.

For more information on *XWIN-NMR*™ software and our other new products, contact your local sales representative.

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**Applications and Support Group**

BRUKER

Bruker Spectrospin Limited

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

Bruker Spectrospin Ltd
Banner Lane
Coentry CV4 9GH
UK

March 4 1996
(received 3/9/96)

Dear Prof. Shapiro,

**Adiabatic is good for you!**

It has been quite some time since I contributed to the Newsletter and I am aware I have been spared your wrath in the form of multicoloured ultimatums and dotted envelopes solely due to the "efforts" of my colleagues at Bruker. To show that I am still alive and kicking the spectrometer, here is a simple but useful experiment.

The benefits of adiabatic pulses have been recognised for years in MRI and localised spectroscopy applications but the analytical spectroscopist has kept his loyalty to the good old "hard" pulse. Not always rightfully so! For instance, in inversion recovery $T_1$ experiments a full passage hyperbolic secant pulse should always be preferred to a square pulse for the magnetisation inversion.

In the figure the effect is illustrated of using a hyperbolic secant half passage as a $90^o$ pulse. Because of the independence of RF field inhomogeneity the adiabatic pulse excites a slightly larger effective volume than an accurately determined "hard" $90^o$ pulse. This results in the small positive difference between the hard pulse and adiabatic pulse spectrum (approximately 3%). When the probehead is not optimally tuned (a common situation in open access NMR) then the hard pulse is no longer $90^o$ and the difference becomes larger: about 20% in our example.

So the conclusions are: Don't think shaped pulses must be selective, and: Adiabatic is good for you!

Sincerely yours,

Joost A.B. Lohman
Senior Applications Scientist

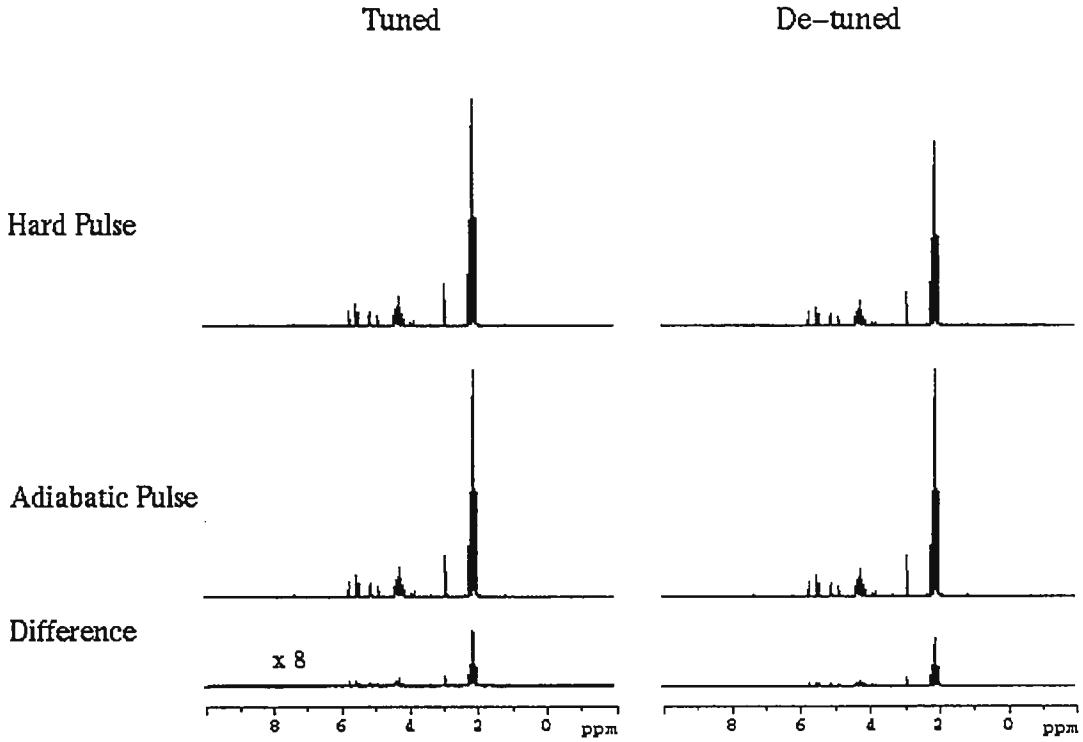


Figure 1: 500 MHz NMR spectra of Sucrose-octo-acetate in Acetone-$d_6$. The hard pulse was 6.1 μs at approximately 25 Watt; in the case of a tuned probe this corresponded to a $90^o$ pulse. The adiabatic pulse was a half passage hyperbolic secant pulse of 500 μs at approximately 12 Watt. Reducing the pulse power to about 4 Watt resulted in only a minor reduction in signal strength. In order to excite the whole spectrum with uniform phase, the pulse was applied at -1 ppm.

Assistant Professor, The University of Akron Department of Chemistry.

The Department of Chemistry at The University of Akron is seeking an outstanding applicant whose research interests involve the use of SOLIDS STATE NMR SPECTROSCOPY. The applicant should have a Ph.D. in Chemistry and Postdoctoral experience. While the exact field of Chemistry is open, preference will be given to applicants with excellent qualifications, and whose use of solid state NMR complements the research activities at the U of A. The Department maintains a state-of-the-art NMR facility with 5 spectrometers from 200 to 600 MHz (two of these are dedicated to solid state experiments), and an extensive computer network with a suite of NMR related software. Details about the Department and the NMR facilities can be obtained on the WWW (http://atlas.chemistry.uakron.edu:8080/ and http://atlas.chemistry.uakron.edu:8080/cdept.docs/mspechome.html). Please send resume, two page research description, and three letters of reference to: NMR Search Committee, Department of Chemistry, The University of Akron, Akron, Ohio 44325-3601. The University of Akron is an equal employment opportunity organization.

The University of Akron

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Buchtel College of Arts and Sciences
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216-972-7372 216-972-5256 Fax

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

February 29, 1996
(received 3/4/96)

Subject: Heteronuclear Spin Locking Experiments

Dear Barry,

I have seen numerous inquiries and comments regarding the efficiency of heteronuclear spin locking (HSL) experiments, both in this publication and in several of the computer E-mail discussion groups over the past year. I thought that some comments about our experiences with these experiments might be helpful.

Most of the discussions I have seen center around the question of why one probe gives such good sensitivity and other probes perform this experiment so poorly. We have attempted to perform these experiments with $^{13}C$ detection (spin locking $^{1}H$ and $^{13}C$) on a 300 MHz spectrometer using three different probes; a 10 mm broadband probe; a 5mm $^{1}H$-$^{19}F/^{31}P$-$^{15}N$ broadband switchable probe optimized for $^{13}C$ detection (separate coils for the $^{1}H$ and $^{13}C$ frequencies); and a 5mm indirect detection probe (built for us by Nalorac, with a single coil double tuned for the $^{1}H$ and $^{13}C$ frequencies) optimized for $^{1}H$ detection. Our best results were always obtained on the last of the three probes described above, despite the fact that it has the poorest S:N specifications for $^{13}C$ detection ($^{13}C$ S:N = 50:1 for ASTM sample compared to 120:1 for the 5 mm switchable probe).

All of the above probes provided HSL spectra from samples of low MW molecules in very concentrated solutions (ca. 1M). However, we were only able to obtain HSL data from real-world samples (10-50 mM) using the solutions probe which has a coil double tuned for the $^{1}H$ and $^{13}C$ frequencies. We attribute this to the fact that with the double-tuned coil, the matching condition is satisfied for most of the sample volume in the active region of the coil (just as with a solids CPMAS probe). However, with a dual coil arrangement, the matching condition is precisely met for only a small fraction of the sample volume. More experimental data are provided in *J. Magn. Resonance*, **100**, 139-145 (1992).

I hope this puts The University of Akron back in good standing as far as our contributions to the newsletter.

Regards,

Peter

Peter L. Rinaldi
Professor of Chemistry
Director of the Molecular Spectroscopy Laboratory

# The NMR Newsletter - Book Reviews

Book Review Editor: **William B. Smith**, Texas Christian University, Fort Worth, TX 76129

## " NMR and Its Applications to Living Systems "

by

**David G. Gadian**

Oxford University Press, 198 Madison Avenue, New York, NY 10016; 1995
ISBN 0-19-855281-5 (Hbk), 0-19-855803-1 (Pbk); 278 pages + index. $80.00

This volume is addressed to those persons in medical and biological research who need background in NMR either to assess the work of others or to get started in the field for their own purposes. The chapter titles run as follows: 1. Introduction (includes a review of what is the basis of NMR and some of the application to living systems.); 2. The information available from NMR (a survey of applicable nuclei, measurement of pH, metabolites, perfusion and diffusion); 3. MRS and tissue biochemistry; 4. Physiological magnetic resonance imaging; 5. The theoretical basis of NMR; 6. The NMR parameters; 7. Instrument design and operation; and 8. Pulse sequences; all followed by five pages of index. The ordering of the text at first sight seems a little strange. One might have thought all the technical details would come first and the applications later, but the author knows his audience. To hold their attention through the heavy-weight NMR discussions, he intersperses a variety of applications.

This would make a good introductory text for medical and biochemically related professions.

## " High Resolution NMR in the Solid State; Fundamentals of CP/MAS "

by

**E. O. Stejskal** and **J. R. Memory**

Oxford University Press, 198 Madison Avenue, New York, NY 10016; 1994
ISBN 0-19-507380-0, 1994, 189 pages, $39.95

This volume bears a 1994 copyright, but came to my desk only in the fall of 1995. Since a detailed review appeared in the December 1995 issue of *Magnetic Resonance in Chemistry*, I will not go into great detail here. The chapters are as follows: I. Introduction to NMR principles; II. High Resolution Methods in the Solid State; III. Spin-spin Interactions; IV. Magic Angle Sample Spinning; V. Spectrometer and Probe Design. A series of appendices addresses the subjects of vectors, matrices, complex numbers, spinning side bands, Hamiltonian theory, transmission lines and phase-sensitive detection. This volume is much more grounded in theory that the volume reviewed above. As a person who has never had occasion to run a solids NMR, I found this volume a fascinating introduction to the subject and can highly recommend it to those wishing to make the jump from solution NMR. The index of 2+ pages seems adequate.

WBS

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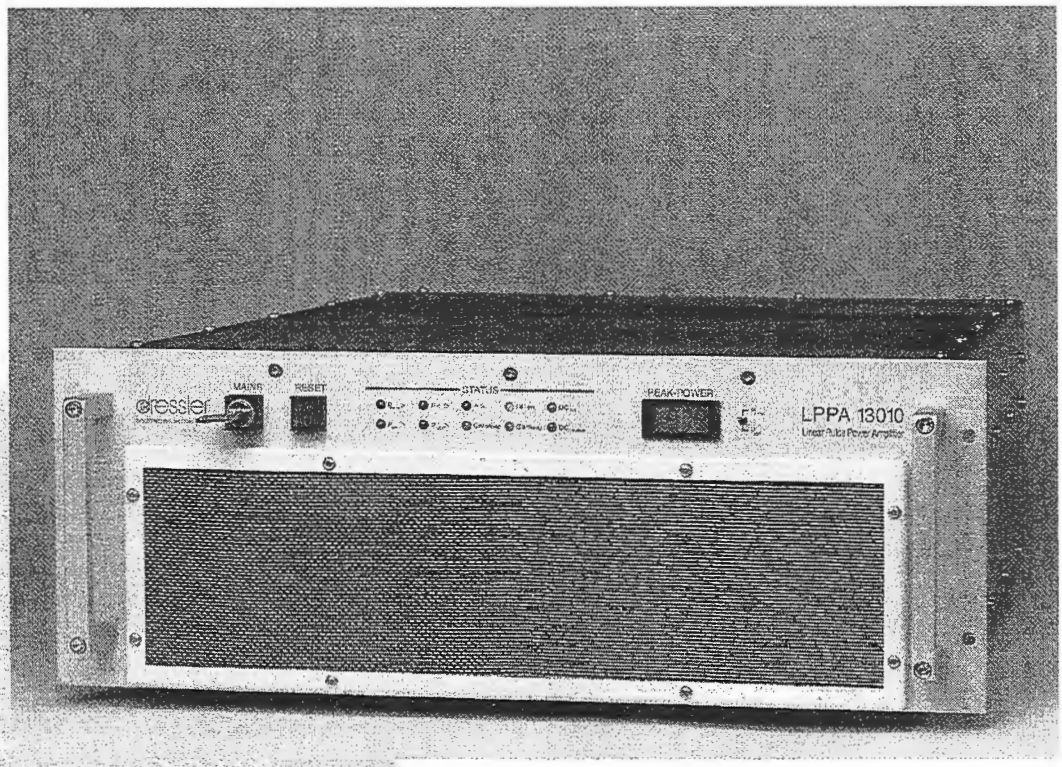


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| RF pulse power | 1000 W<br>700 W | 2000 W<br>1400 W | 1000 W<br>– | 400 W<br>– | 8000 W<br>– | into 50 Ohms<br>(105 - 130 MHz) |
| CW output power | 100 W | 200 W | 150 W | 100 W | 1000 W | into 50 Ohms |
| Impedance | 50 Ohms | 50 Ohms | 50 Ohms | 50 Ohms | 50 Ohms | N type |
| Gain | 60 ±4 dB | 63 ±4 dB | 60 ±2 dB | 56 ±2 dB | 69 ±2 dB | at Pmax. |
| Linearity | ±0.5 dB | ±0.7 dB | ±0.4 dB | ±0.8 dB | ±0.8 dB | 1 – 80% $P_{RF}$ |
| Harmonics 3xf | -14 ... -25 dBc | -14 ... -25 dBc | -18 ... -25 dBc | -15 ... -30 dBc | -18 ... -25 dBc | at Pmax. |
| Harmonics 2xf | -25 dBc or better for all models | | | | | |
| Pulse width | 20 ms | 10 ms | 20 ms | 20 ms | 10 ms | at Pmax. |
| RF pulse energy | 10 Ws | 10 Ws | 10 Ws | 10 Ws | 40 Ws | max. |
| Pulse droop | 0.1 – 0.3 dB | 0.2 – 0.4 dB | 0.2 dB | 0.1 – 0.3 dB | 0.3 – 0.4 dB | at Pmax. |
| Pulse rise time | 500 ns | 500 ns | 300 ns | 200 ns | 300 ns | |
| Pulse fall time | 60 ns | 60 ns | 30 ns | 30 ns | 30 ns | |
| Noise blanked | -160 dBm/Hz | Noise floor for all models | | | | |
| Noise unblanked | -120 dBm/Hz | Noise floor for all models | | | | |
| Displays | • 3 1/2 digit LCD meter for forward power or reflected power<br>• RESET push button and indicator<br>• RF on • Blanking • P forw. • DC RF • CW Mode<br>• PTT • P DC • P reflected • DC control • Thermal overload | | | | | |
| Interface | Description see text above<br>Input levels 5 – 24 V DC. Outputs 5 V TTL<br>Analog Inputs and Outputs 0 – 10 V DC | | | | | |
| Cooling | Forced air cooling. Air inlet at the front panel. 0 – 40 degrees C | | | | | |
| Weight | 18 kp | 22 kp | 18 kp | 18 kp | 170 kp | |
| Dimensions | 19" 4 Units = 178 mm all models | | | | 19" 40 Units = 178 cm | |
| Depth | 550 mm all models | | | | 550 mm | |
| Power supply | 180 – 240 V AC 50/60 Hz | | | | 380/400 V 3 phase | |
| Power Factor | 0.95 - 0.98 PFC corrected for the latest IEC-555 regulations | | | | | |

Specifications to change without notice March 1995

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Baltimore, MD 21224

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

March 11, 1996
(received 3/16/96)

## Efficiency of Liquid Helium Transfer Lines

Dear Dr. Shapiro,

I would like to relate a cautionary tale to the NMR community regarding the construction of liquid helium transfer lines. We own a 400 MHz superwidebore magnet, which was originally supplied with a 1/2" transfer line and a double-walled but undewared screw-on extender tube for the supply tank leg. After several liquid helium refills with this transfer line, we noticed that at most 60-70 liters of liquid helium could be added to the magnet given a full 100 liter supply dewar. This low efficiency was unaffected by varying the fill pressure between 0.5 to 1.5 psig, and a helium leak check showed that the line was properly evacuated and leak tight to helium. While we were concerned about the poor cryogenic efficiency of this transfer line, we gradually learned to live with more frequent helium refills.

During a fill several months ago, the original transfer line developed an internal leak and failed spectacularly, necessitating the use of a 3/8" transfer line borrowed from another magnet, plus the requisite 3/8"-to-1/2" adapter fittings. This smaller transfer line was long enough to be used with no extender tubes. To our pleasant surprise, the 3/8", fully-dewared transfer line filled our magnet with a loss of only 5 liters of liquid helium! Yet we were still not sure why the original line was so inefficient before it failed.

Eventually, we purchased a replacement 1/2" transfer line, which came supplied with an extender consisting of a length of thin-walled stainless steel tubing with no insulation of any kind. To our great dismay, the new transfer line lost all but 18 liters in a 100 liter fill! We received numerous explanations for this poor transfer efficiency, including excessively high or low fill pressure, ice blockages in the magnet, and poor vacuum in the transfer line. Indeed, the manufacturer claimed that a fill pressure of 3-4 psig was needed for this line, despite its relatively large diameter. Nevertheless, we found that the efficiency was not substantially affected by fill pressure and that neither the transfer line vacuum nor ice in the magnet stacks was at fault. Finally, we attached the double-walled extender from the original transfer line to the new line and again got about 60% efficiency, proving that the new transfer line itself was no less efficient than the original line.

From all of this experimentation, we conclude that using undewared transfer line extensions dramatically decreases liquid helium transfer efficiency and that

single-walled extenders lead to particularly large transfer losses. It should be noted that in each fill, a tall-form supply dewar was used so that the extender was at least 16" below the neck of the dewar and thus completely surrounded by cold helium gas at all times. Nevertheless, we suspect that these extenders are not cooled sufficiently to prevent excessive boil-off unless they are submerged in liquid helium during the entire fill. Indeed, by monitoring the liquid level in the magnet during each fill, we could see that the liquid accumulation rate decreased steadily as the liquid helium in the supply dewar drained down, exposing more and more of the uninsulated extender tube to helium gas. Finally, once all but the last 6" of the extender was exposed to gas, no more liquid helium could be transferred to the magnet at all. Consequently, we strongly caution against the use of uninsulated extender tubes on liquid helium transfer lines unless ceiling height or other physical restrictions make their use unavoidable.

Sincerely,

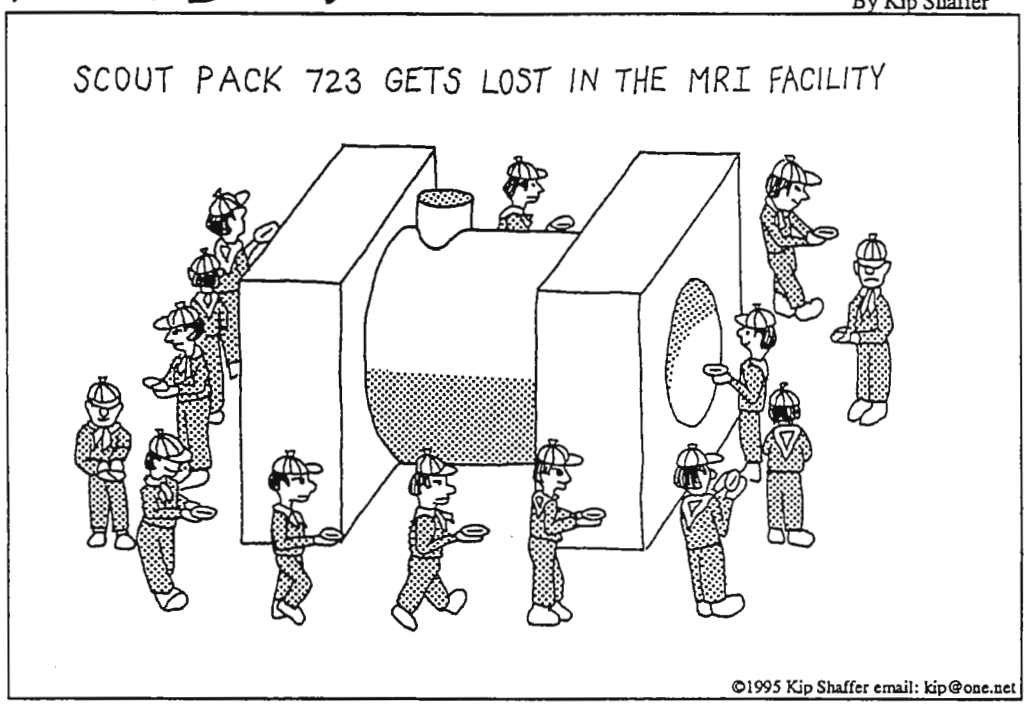
Ken Fishbein
Facility Manager, NMR Unit
NIH/NIA/GRC

Richard G. S. Spencer
Chief, NMR Unit
NIH/NIA/GRC

Phone: (410) 558-8512
FAX: (410) 558-8173
Email: fishbein@vax.grc.nia.nih.gov

**Field of Dreams**

By Kip Shaffer



Lilly

**Lilly Research Laboratories**
A Division of Eli Lilly and Company

Lilly Corporate Center
Indianapolis, Indiana 46285
(317) 276-2000

6 March 1996
(received 3/11/96)

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

Dear Barry:

**News from Lilly**

Thank you for all the nicely colored letters. It seems that my colleagues here have gone very shy about telling the outside world what they are up to. As a result you will have to hear the news from me.

Steve Maple sent one of our compounds to the Varian demonstration laboratory last year and got some very nice results from LC-NMR. The compound he sent is one that undergoes a series of interconversions analogous to anomerism in sugars, and therefore might not have been predicted to be separable by chromatography. As it turned out, however, the choice of this compound was a very shrewd one. The rates of interconversion are rather slow, and as a result it *is* possible to separate the various components. Only when one does stop-flow experiments is the interconversion detectable. In fact, this news was of particular interest to some of our chemists, who had been trying unsuccessfully to study some of the interconversion rates of this equilibrium in other ways. Steve has used these results as part of a justification of a new spectrometer. Said spectrometer is presently being installed, and I am sure that Steve will soon have all sorts of results that he will want to send to you.

Susan Reutzel continues to do amazing things with solid state NMR. She and her post-doc, Paula McGee, got the HETCOR experiment going last year. That has always been one of my favorite experiments in solution and I am delighted to be able to look at the same experiment in the solid state. They also got $^{15}N$ SSNMR going. Since virtually all drugs include nitrogen atoms in their structures, often at important sites, this promises to be a very important tool in the future. In fact, Paula promised me a contribution for TAMU on this subject last fall, but the rush of moving to a new position seems to have crashed that idea. But I am sure that Susan is working on a couple of contributions to send you, even as you read this.

In fact, all my colleagues seem to be busily doing science and having fun. And what does that leave for me to do?

Well, for the last few months I have been busy preparing an inventory of all the NMR instruments Lilly owns. The list isn't completed yet, but just in case you are interested we have something over two dozen instruments, ranging from 200 to 500 MHz in field. The oldest was purchased around 1980 (nobody seems to remember for sure), and the newest, as I mentioned above, is being installed now. Examination of the data showed us that we have what Steve Maple calls an "aging fleet." In recognition of this I am now trying to work out a five-year plan, complete with succession and update planning. This may not sound like fun, and in fact it isn't, but it is the type of thing old-timers like me do after they forget how to run spectrometers.

Doug Dorman

Pharmacia & Upjohn

March 12, 1996
(received 3/18/96)

Calibration of Z-gradients with Shigemi Microtubes

Dear Dr. Shapiro,

We have been using Shigemi NMR microtubes -- both 3 mm and 5 mm OD varieties -- successfully for some time now with very good results. In spite of the advertising which touts the magnetic susceptibility match for aqueous samples, we use them with literally all solvents. The very viscous solvents are sometimes difficult to manipulate, especially in a glovebox environment, but it can be done, and the resulting time-savings in instrument time and quality of spectra are worth the effort.

We recently found yet another use for the Shigemi NMR cells; on those occasions when gradient strengths need to be calibrated, the z-gradient has always been uniquely messy, and consequently, not too accurate. Bruker provides, under duress, a machined, solid plug of some rigid polymer which fits nicely into a 5 mm OD standard NMR tube. This plug has two holes drilled through it which are approximately 8 mm apart; the holes are a smaller diameter and perpendicular to the long axis of the plug. When filled with water (much easier said than done), these two holes provide a known geometry for a z-gradient profile (see below). However, the holes are difficult to fill with water, the exact distance between the holes is hard to measure accurately, and the resulting spectrum is messy due to much interfacial water distributed about the plug.

By using Shigemi microtubes, the above problems are solved. They are as easily filled as normal NMR tubes, the tolerances are such that the height of the vertical plug of water can easily be measured to 0.1 mm accuracy, and the interfacial water is a small fraction of the measured total. Thus the resulting z-axis profile is much more definitive (see below), and the calibration is easily made. I have never been able to obtain a Bruker-like plug for our 3 mm OD NMR tubes, but the Shigemi microtubes work almost as well in this instance also. The only difference is that the fraction of interfacial water is somewhat larger due to the reduced volumes, but the measurement and sample preparation are really just as straightforward as in the 5 mm OD case.

Below are examples of z-gradient calibrations using the Shigemi microtube and the Bruker plug in a 5 mm OD probe. The formula from NMR Imaging 101 which gives the gradient strength is also included.

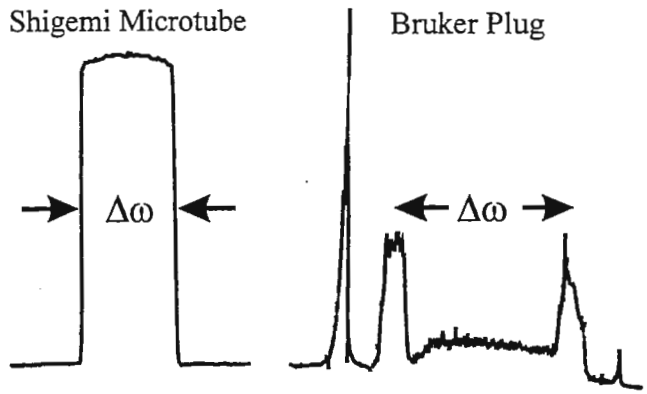
$$Gz = \frac{\Delta\omega(kHz)}{[4.258][d(cm)]}$$

Sincerely,

Paul Fagerness

Greg Walker

Steve Mizsak

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966 Elsinore Court
Palo Alto, CA 94303.

(415) 493-5971* - *Please call only between 8:00 am and 10:00 pm, Pacific Coast time.*

**Deadline Dates**

| | |
|---|---|
| No. 452 (May) | 26 April 1996 |
| No. 453 (June) | 24 May 1996 |
| No. 454 (July) | 28 June 1996 |
| No. 455 (August) | 26 July 1996 |
| No. 456 (Sept.) | 23 Aug. 1996 |

*Fax: (415) 493-1348, at any hour. Do not use fax for technical contributions to the Newsletter, for the received fax quality is very inadequate.

⇒⇒⇒ E-mail: shapiro@nmrnewsletter.com [Do not use the previous Compuserve number.]

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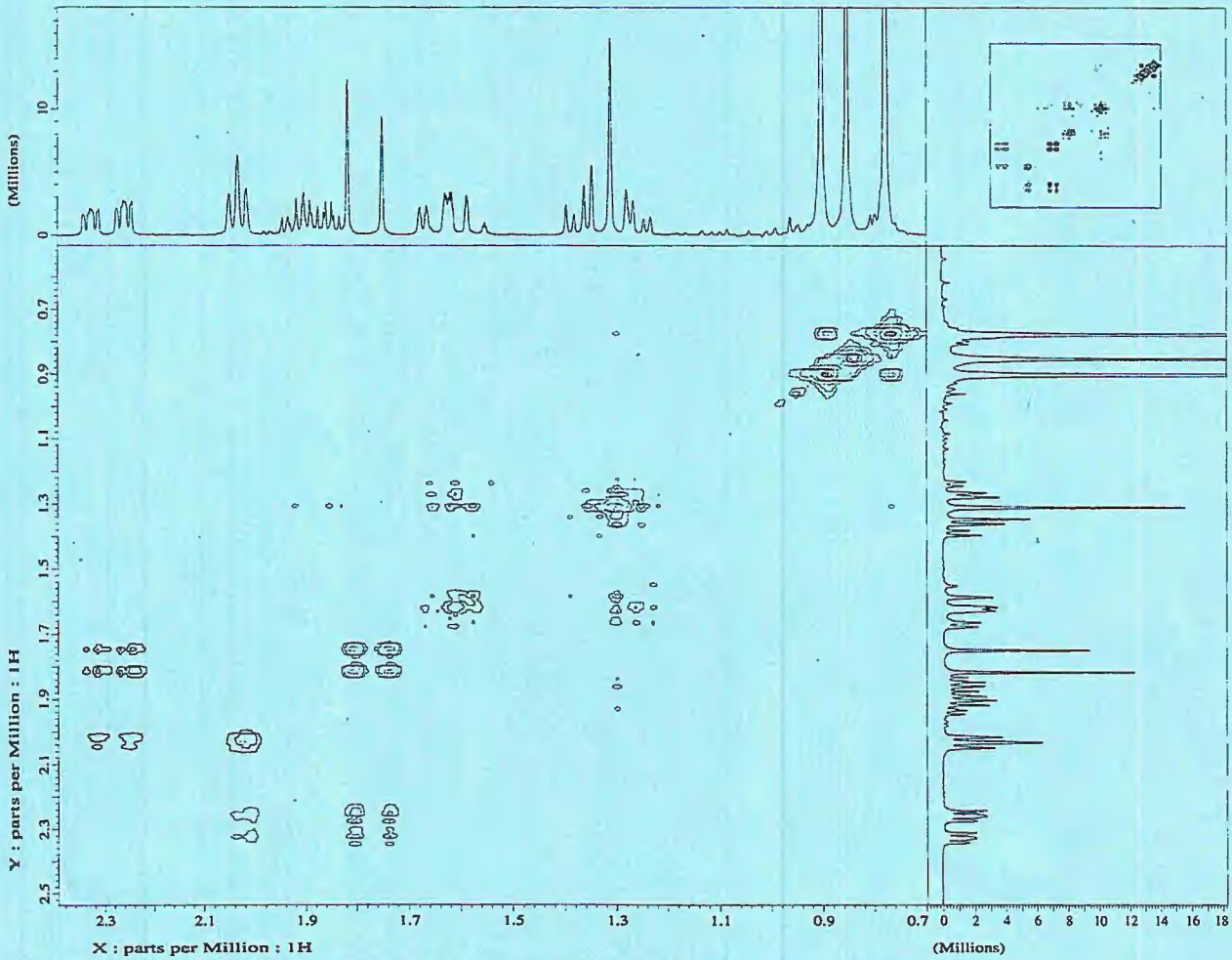


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Now you can use the ECLIPSE NMR Advantage to your advantage.

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