THE
NMR
NEWSLETTER

No. 452

May 1996

Automated processing for reproducible quantitation

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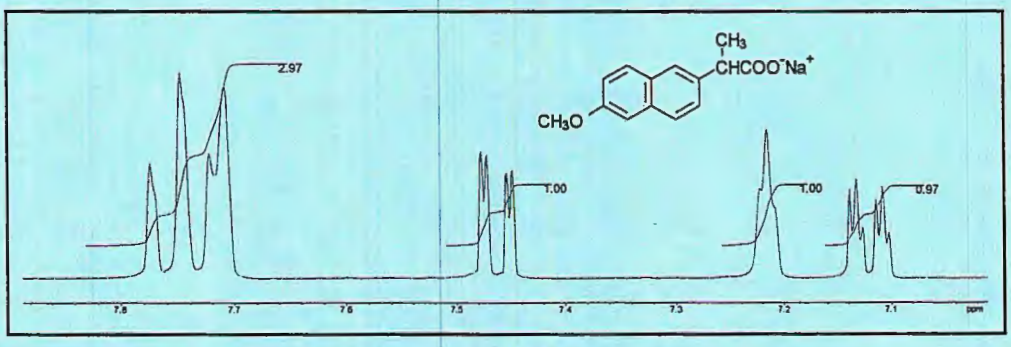

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FORTHCOMING NMR MEETINGS

NMR NEXUS '96, Canadian NMR Summer School, Winnipeg, Manitoba, Canada, **June 10-14, 1996**; For further information, phone 204-984-4543; Email: nmr.school@ibd.nrc.ca; http://www.ibd.nrc.ca/; Fax: 204-984-4722.

WWW Electronic Poster Session Announcement, **June 17-21**; see Newsletter 452, 52.

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 452, 59.

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.

4th International Conference on Magnetic Resonance Microscopy "Heidelberg Conference in Albuquerque", **Sept. 21-15, 1997**: Contact: E. Fukushima, The Lovelace Institutes, 2425 Ridgecrest Drive SE, Albuquerque, NM 87108-5127; (505) 262-7155; Fax: (505) 262-7043. See Newsletter 449, 37.

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Lensfield Road
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Ray Freeman *Telephone:* **(01223) 336450**

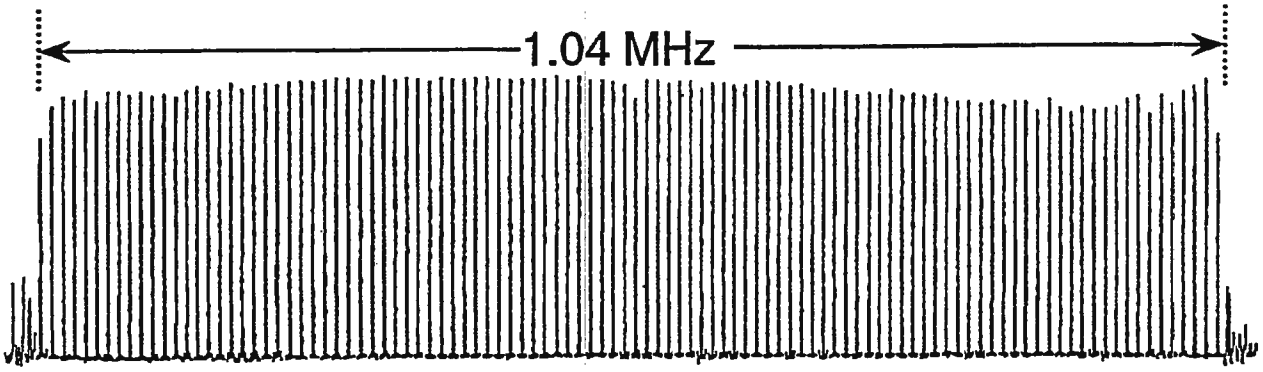
Professor Bernard Shapiro,
966 Helsingførs Court,
Palo Alto,
California, 94303
USA.

3 April 96
(received 4/9/96)

Dear Barry,

"Mega-Wurst"

If anyone ever cares to build a 20 GHz spectrometer for protons, it would require a bandwidth of 1 MHz to decouple the entire range of carbon-13 shifts. Already WURST-400 decoupling can deliver such a bandwidth (although only at *normal* radiofrequencies). As evidence we show below the offset dependence of the proton signal from sodium formate (J = 200 Hz) at a decoupler level $\gamma B_2/2\pi$ = 14.3 kHz, measured on a Varian Unity-500 at Darmstadt. All the operating parameters are a little extreme. For example, the frequency has to be swept at a rate in excess of *1 GHz per second*, and the undulations in the amplitude of the decoupled line are probably attributable to the radiofrequency circuitry and coil, rather than shortcomings of the decoupling sequence.



In collaboration with Kurt Wüthrich and Gerhard Wider, we were able to show that by backing off from these extreme conditions we could keep the cycling sidebands down below the 0.2% level, even with synchronous decoupling.[1] This is significantly better spectral purity than that achieved by GARP or WALTZ-16 under equivalent conditions.

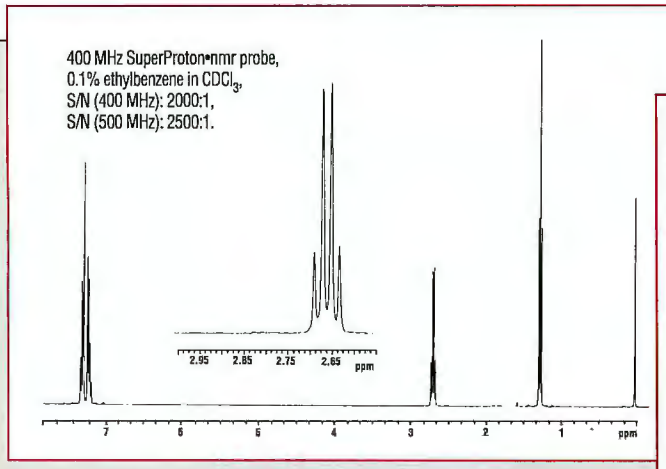
Best wishes,

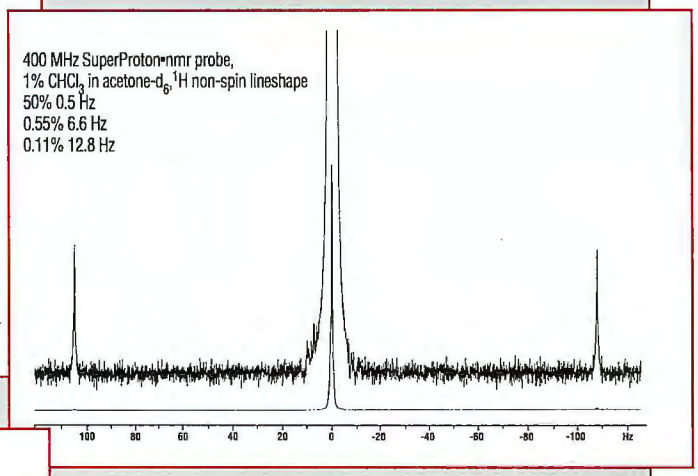
Ray Ēriks

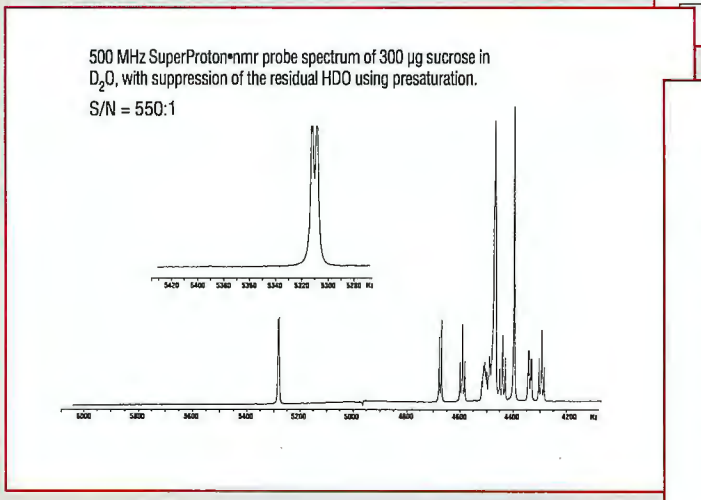
Ray Freeman, Ēriks Kupče

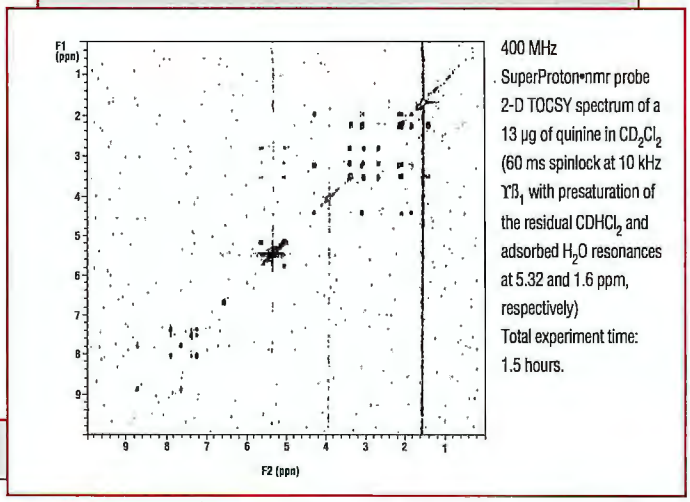
[1] Kupče, Freeman, Wider and Wüthrich, J. Magn. Reson. A. (in press).

Break Through the NMR Detection Limit Barrier









400 MHz SuperProton•nmr probe 2-D TOCSY spectrum of a 13 µg of quinine in CD_2Cl_2 (60 ms spinlock at 10 kHz γB_1 with presaturation of the residual $CDHCl_2$ and adsorbed H_2O resonances at 5.32 and 1.6 ppm, respectively)
Total experiment time: 1.5 hours.

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- Excellent spinlock performance
- Available for 400 and 500 MHz systems

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Extending the ^{1}H and ^{19}F NMR Detection Limits

Features	Benefits
Sensitivity advantage of a factor of 4 relative to a standard 5 mm ^{1}H or ^{19}F probe	Factor of 16 reduction in data-collection time; ability to obtain high sensitivity 1-D and 2-D spectra of samples in very low concentrations (e.g. 60 µg of MW 600 in 600 µl)
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Excellent spinlock performance	High sensitivity for homonuclear experiments utilizing high-power spinlock
Excellent lineshape performance	High spectral resolution and sensitivity; excellent solvent suppression
Deuterium lock	Standard deuterium field-lock and shim

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April 15, 1996
(received 4/16/96)

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

Detection of non-Protonated Carbons in Labeled RNA Oligomers

Dear Dr. Shapiro:

Recently, we faced a rather difficult task of assigning the imino protons on a 40-mer RNA aptamer binding ATP[1]. One of the approaches we used to tackle this problem involved chemical synthesis, in collaboration with Roger Jones at Rutgers University, of molecules incorporating G residues selectively with different labeling patterns. In one case the base was uniformly ^{15}N labeled, and in the other both uniformly ^{15}N labeled and ^{13}C labeled at C2. To discriminate these two, the HNCa experiment developed for proteins[2] was adapted. As the exchange rate of imino and amino protons in nucleic acids does not allow the use of presaturation, we added the WATERGATE suppression routine at the end of the pulse sequence. Otherwise, the experiment is simpler to apply to RNA than to proteins as we do not have to deal with the selective excitation of aliphatic and carbonyl carbons. Thus, we could use hard ^{13}C pulses throughout the experiment. The experiment can be applied equally well as a general approach in uniformly ^{13}C, ^{15}N labeled RNA to detect ^{13}C sites without proximal non-exchangeable protons. Determination of the shifts of such carbons may be useful in characterizing base hydrogen bonding.[3]

Figure 1 shows a 1D spectrum of imino proton region of the 40 mer, and correlations of guanine and uracil imino protons to the carbon atoms neighboring the imino nitrogen. GC6 appear much weaker in the spectra than CG2 as a result of the splitting of GC6 peaks by the coupling to C5 and C4. The experiment can be used to correlate the amino protons as well (data not shown), only the evolution interval for ^{15}N-^{1}H INEPT transfer must be cut in half because of the faster evolution due to the presence of two protons in the amino group.

Sincerely yours,

Radovan Fiala

[1] Sassanfar, M. and Sostak, J. W., Nature 1993, 364, 550-553.
[2] Grzesiek, S. and Bax, A., J. Magn. Reson. 1992, 96, 432-440.
[3] Borer, N. P. Et al. Nucl. Acids. Res. 1988, 16, 2323-2332.

Please credit to David Live's account.

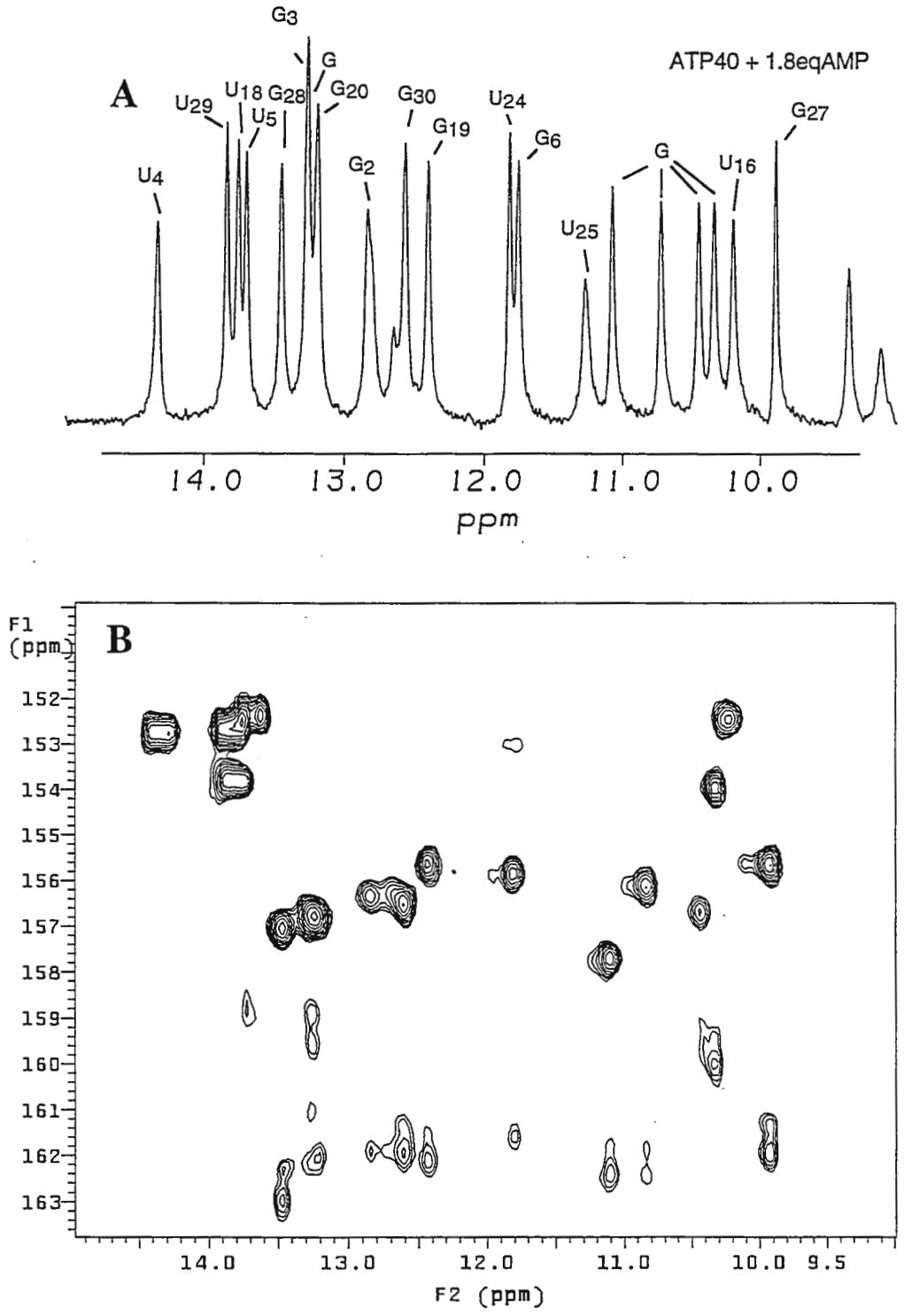

Figure 1. (A) imino region of proton 1D spectra. (B) 2D ^{1}H-^{13}C correlation of imino protons to carbons adjacent to the imino nitrogens. Varian Unity Plus 500, 5°C, 16.8 ppm and 1k (real) points in ^{1}H dimension, centered at 11 ppm, 31.8ppm and 64 (complex) points in ^{13}C dimension, centered at 155 ppm,128 scans per increment, total acquisition time approx. 7 hours.

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Dr. B.L. Shapiro
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National Institute on Alcohol
Abuse and Alcoholism
Rockville, MD 20857

Laboratory of Membrane
Biochemistry & Biophysics
12501 Washington Ave.
(301) 443-0552

Inserts for 4mm MAS Rotors

April 8, 1996

(received 4/18/96)

Dear Dr. Shapiro,

Our laboratory investigates biophysical properties of lipid membranes using various solid-state NMR methods. To observe protons, we utilize high-resolution two-dimensional experiments in combination with magic angle spinning on multilamellar vesicles. Hydrated membranes are neither solid nor liquid but fall into the in-between category of liquid-crystalline and have a consistency similar to a very greasy wax. When we first attempted to spin samples with such a consistency we found that if rotors were filled, the grease-like nature of the lipid forced the cap off the rotor during spinning. To solve this, the sample was reduced to a very small amount, approximately 2-3 mg. Now, spinning spread the lipid over the inner surface of the rotor which resulted in broad or split peaks due to a field inhomogeneity which was difficult to remove by shimming. This led us to design an insert for a 4 mm rotor that maintains a small volume of sample exactly at the center of the coil in a Bruker MAS probe. The material used for the insert is Kel-F and the internal sample equals approximately 8 µL. The improvement in the linewidth at half-height is routinely a factor of three to four. More importantly, the peak width at the baseline improved even more. Spectra of a typical lipid sample recorded without and with the insert in a rotor are shown below as well as a schematic of the insert design.

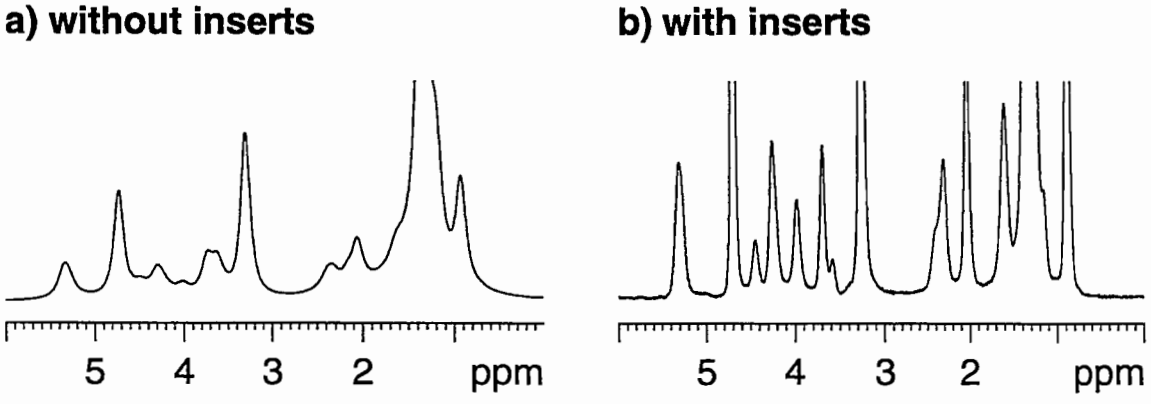


Figure 1. 1H–MAS NMR spectra of a phospholipid dispersed in 50 weight % D2O using a 4 mm rotor. Spinning speed = 5 kHz.

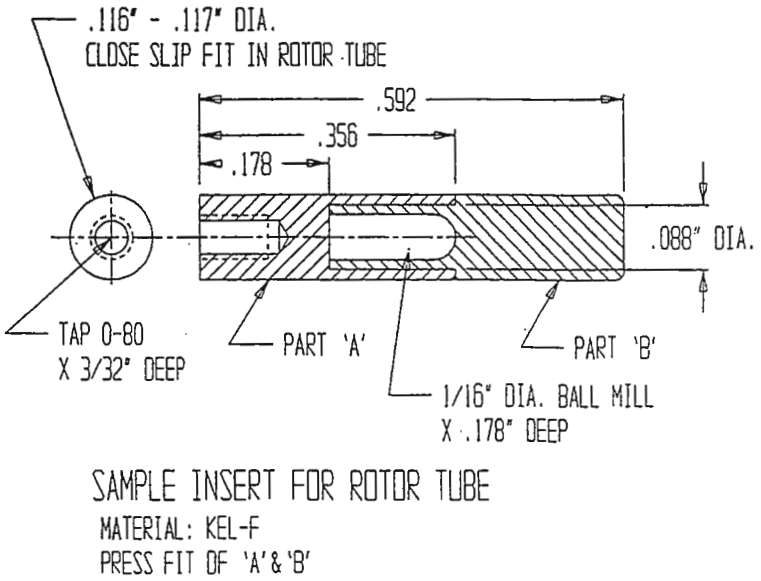


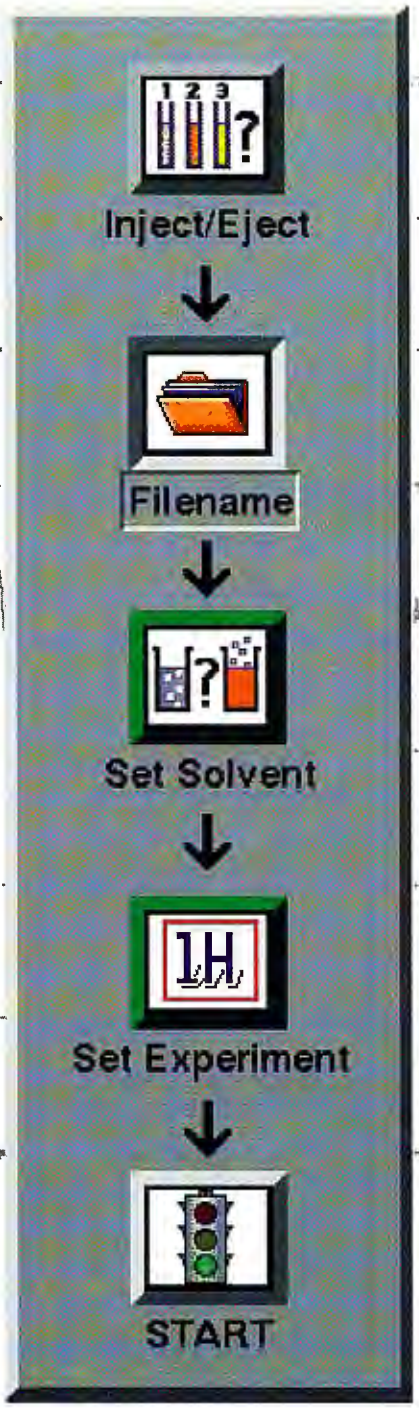
Figure 2. Schematic of the insert design for a 4 mm MAS rotor.

Our primary goal in this endeavor was to overcome limitations imposed by an uncooperative sample consistency and to improve resolution. But in cases where one had a limited sample availability, use of inserts would also reduce the quantity needed. Another advantage of using this insert is that the sample is located in a region with a very homogeneous B_1-field.

Sincerely yours,

Laura L. Holte Jim Sullivan Paul Fitze Klaus Gawrisch

It doesn't get any easier than this:



XWIN-NMR is probably the most powerful NMR software ever written, with a full range of acquisition and analysis capabilities that includes multi-dimensional NMR, maximum entropy, linear production and many others.

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

(608) 264-5407
E-Mail jralph@facstaff.wisc.edu
April 10, 1996
(received 4/15/96)

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

Dear Dr. Shapiro:

The Advantage of Fully Digital NMR and Pulsed Field Gradients

TAMU readers scarcely need be reminded that the digital revolution in NMR is here. As a newcommer to these advances, which I regrettably don't have on my own machine, I was surprised at just how far the technology had come and how dramatic the effect was on spectra.

A sample that we have been toiling with is an enriched lignin where we have yet to make serious assignments of most of the peaks but have been very successful in finding evidence for cell wall cross-linking structures. Ferulates in grass cell walls can cross-link arabinoxylan polysaccharides to lignin and we had suspected that this would be achieved via a radical process. Indeed, HMBC spectra have been able to unambiguously confirm this and, more importantly, to determine that ferulates act as nucleation sites for the lignification process. I recently had a chance to access the 750 MHz Bruker at the Madison NMR facility and am ecstaticwith the results, even though we used only one third of the amount of material and ran for only half the time on the 750!

Long range ^{13}C—^{1}H correlation (HMBC) spectra of a uniformly ^{13}C-enriched ryegrass lignin were run on two NMR machines, our own AMX-360 and the National NMR Facility's DMX-750. On the 360, 300 mg of sample was dissolved in 0.24 ml of acetone-d_6 and run for 60 h to give the top spectrum in Fig. 1. On the 750 (which currently only has a 5 mm probe, 122 mg in 0.4 ml acetone-d_6 was run for 24 h to give the bottom spectrum.

The differences in the spectra, Fig. 1, are spectacular. Ignoring the obvious dispersion gain from the higher field instrument, the freedom from T_1-noise artifacts in the digital/gradient system (bottom spectrum) is striking. Peaks close to the methoxyl region are no longer ambiguous. In addition, the flat baseplanes and improved sensitivity allow us to look closer down to the noise level to reveal many more potentially valuable correlations. The only drawback is that we now have a great many more assignments to make in this spectrum!!

These plots and those from the accompanying letter on NMR-invisible methoxyls were, by the way, produced differently from my normal method. Until recently, I saved hpgl files to disk, converted them to Mac PICT format, and took them into Claris Draw for embellishing. The files often became huge and unwieldy. What works far better now is to save the postscript files directly from the Silicon Graphics (in UXNMR or XWinNMR), transfer those to the Mac, convert them to Adobe's pdf format with Adobe's Distiller, and pull these straight in to Adobe Illustrator. The files are about an order of magnitude smaller, print very quickly, and retain the full color information from the NMR — too bad TAMU isn't in color; the figures look great!

Best Wishes

John Ralph

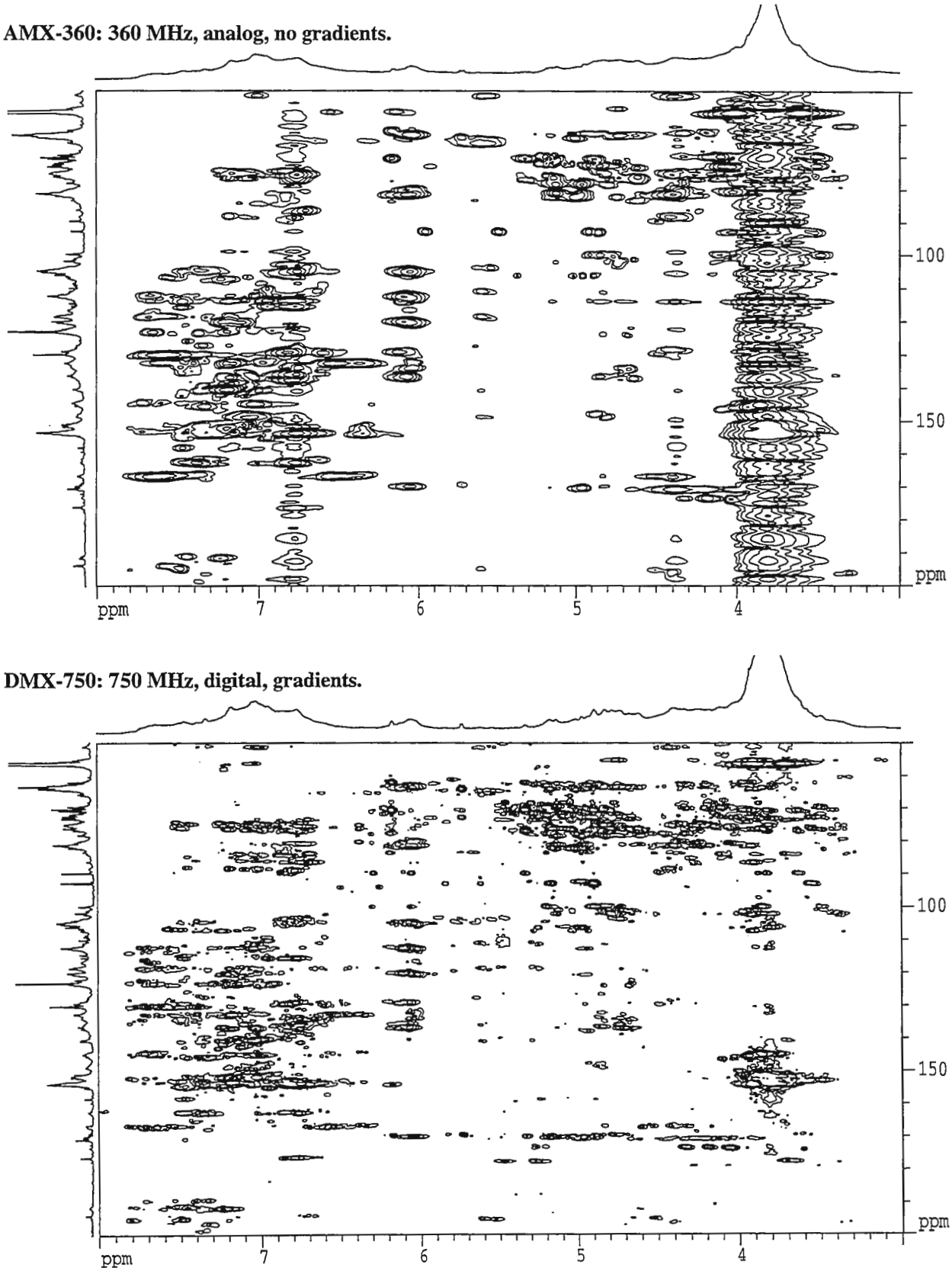

Figure 1. Comparison (unfair) of an HMBC spectrum of a ryegrass lignin with and without gradients and digital acquisition. Top spectrum: AMX-360, 300 mg, 60 h on our non-digital machine without gradients. Bottom spectrum: DMX-750, 122 mg, 24 h. Note particularly the absence of T_1-noise artifacts obfuscating the methoxyl region in the top spectrum (ca 4 ppm on the proton scale), the richness of detectable peaks due to the ability to approach the flat base-plane more closely (bottom spectrum), and the enhanced dispersion resulting from the MHz difference.

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April 10, 1996

(received 4/15/96)

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

Dear Dr. Shapiro:

NMR of Synthetic Lignins with NMR-Invisible Methoxyl Groups

NMR spectroscopy is an invaluable aid to determining details of the structures of plant cell wall components including lignin. And synthetic lignins (DHPs), made by peroxidase/H_2O_2-catalyzed polymerization of the lignin monomer coniferyl alcohol, are enormously valuable to elucidate reaction pathways and provide the NMR data required to interpret spectra from real plant materials. One problem with lignin and DHP spectra is systematic artifacts that arise from various machine imperfections, vibrations of many types, and our impatience for the spectra which dictates that we run acquisitions before the poor nuclei have 'relaxed' properly. The most troubling problem is T_1-noise, a particular problem around the intense methoxyl peaks (in both proton and carbon dimensions of 2D spectra), where artifacts obliterate or obfuscate the data in that region.

An elegant solution to the methoxyl region T_1-noise artifact problem is possible for synthetic lignins; for real lignins, this is not possible but, as noted in an accompanying letter, there is also an instrumental solution. The idea for synthetic lignins is to make the methoxyl group NMR-invisible, but normal in every other regard. That can be done by replacing the normal methoxyl carbon (which has *ca* 1% natural abundance of the NMR-active ^{13}C) by the ^{12}C isotope (^{13}C-depleted) which is not NMR-active. Replacing the protons with deuterons also removes the methoxyl from the proton spectrum.

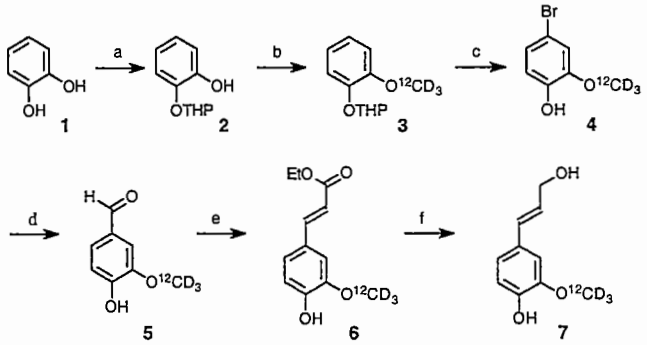


Figure 1. Synthetic scheme to produce coniferyl alcohol with an NMR-invisible methoxyl group.

As shown in Fig. 2, a variety of 2D NMR spectra of a DHP produced from the specialized coniferyl alcohol **7** are spectacularly superior to an equivalent DHP made from normal coniferyl alcohol. The use of short relaxation delays (1 s) heightens the problems from the methoxyls. The spectra also illustrate the value of spectral editing in 2D NMR experiments that has not yet attracted much attention from lignin chemists. The DEPT-HMQC experiment can aid in assignment by selectively inverting (for example) -CH_2 resonances (Fig. 2C).

The freedom from artifacts in these spectra (right side, Fig. 2) will allow more detailed assignments to be made and aid in the characterization of real plant lignins.

Best Wishes

John Ralph

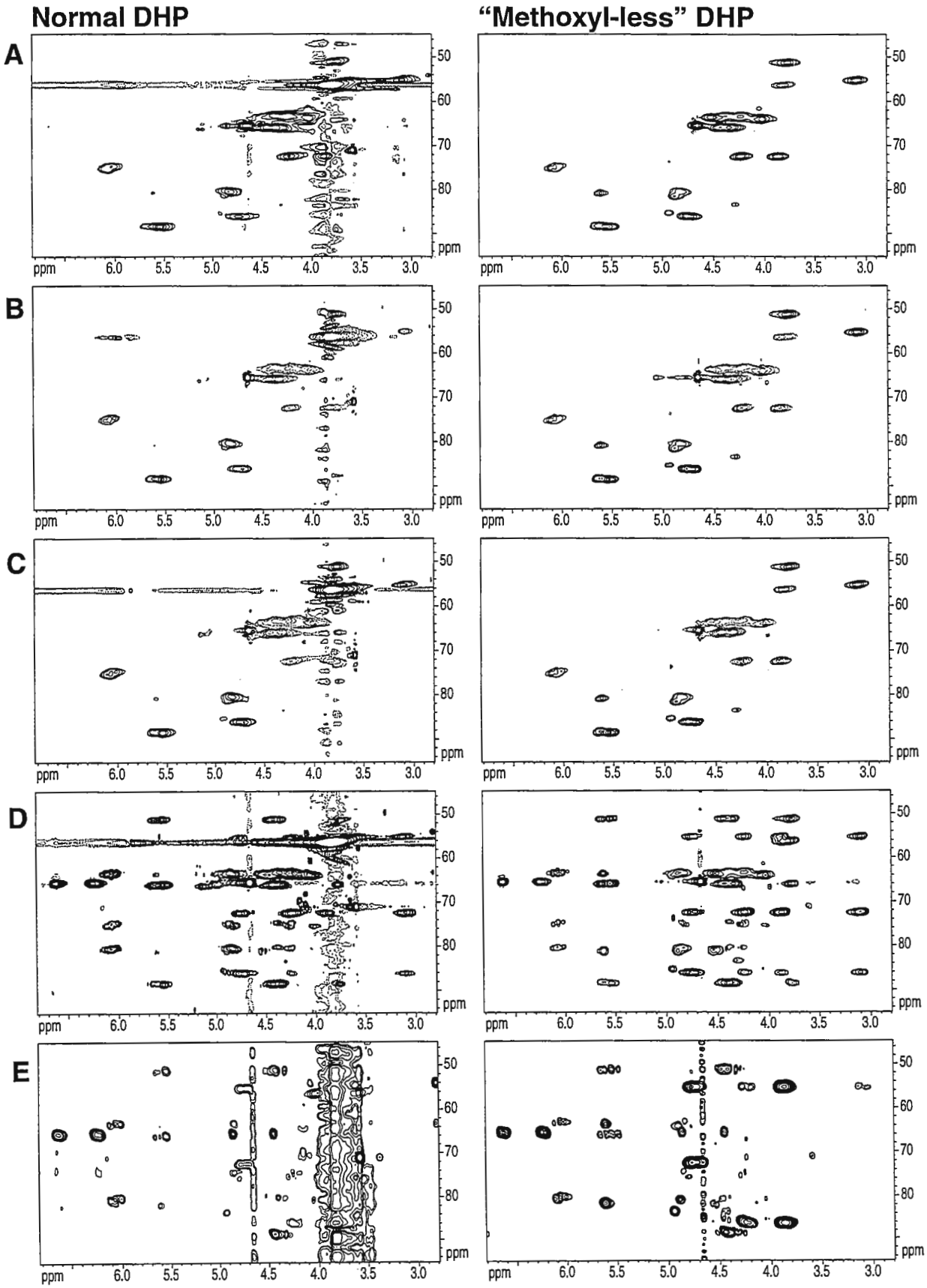
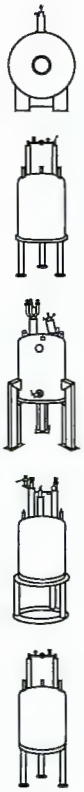


Figure 2. 2D NMR spectra of synthetic DHPs made using (left) normal coniferyl alcohol and (right) 'methoxyl-less' coniferyl alcohol. A) HMQC, B) DEPT-HMQC with a π/3 editing pulse (CH's up, CH_3's and CH_2's down), C) DEPT-HMQC with a π editing pulse (CH's and CH_3's up, CH_2's down), D) HMQC-TOCSY, E) HMBC. Light peaks are -ve.

Beware of imitations



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500	51	10	150	3.2
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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
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Dr B.L. Shapiro
The NMR Newsletter
966 Elsinore Court
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April 24, 1996
(received 4/25/96)

Dear Barry:

Progress with superconductive high resolution probes

We reported some initial results from a high resolution probe using superconductive coils about a year ago (The NMR Newsletter **439,** 41, 1995). The development of these probes is the result of a collaboration between Varian and Conductus, Inc. Since that earlier report, our Conductus colleagues have continued to make improvements in the design and performance of the probes and we have extended the operation to a greater range of experiments.

The basic design of the probe is similar to that described previously. A pair of self resonant superconductive coils is mounted on either side of the NMR sample as indicated in Fig 1. Vacuum isolation allows the coils to be cooled to 25K while the sample is maintained at room temperature. The coils are magnetically coupled to each other and and energy is magnetically coupled into and out of the system via a loop of normal metal. An adjustable conductive paddle (also made of normal metal) provides fine tuning over about a 1MHz range. A second pair of coils mounted orthogonally to the first pair is used to irradiate a second nuclear species. In the majority of experiments, this second nuclear species is deuterium for field-frequency locking. In that case, we have found that coils made from normal metal provide sufficient sensitivity.

Cooling for the coils is provided by flowing helium gas derived either form a tank of liquid helium or by a closed cycle refrigerator. The latter approach allows operation for an unlimited time. Vibrations from the cooler have been reduced to acceptable levels by using a flexible coupling between the cooler and the probe.

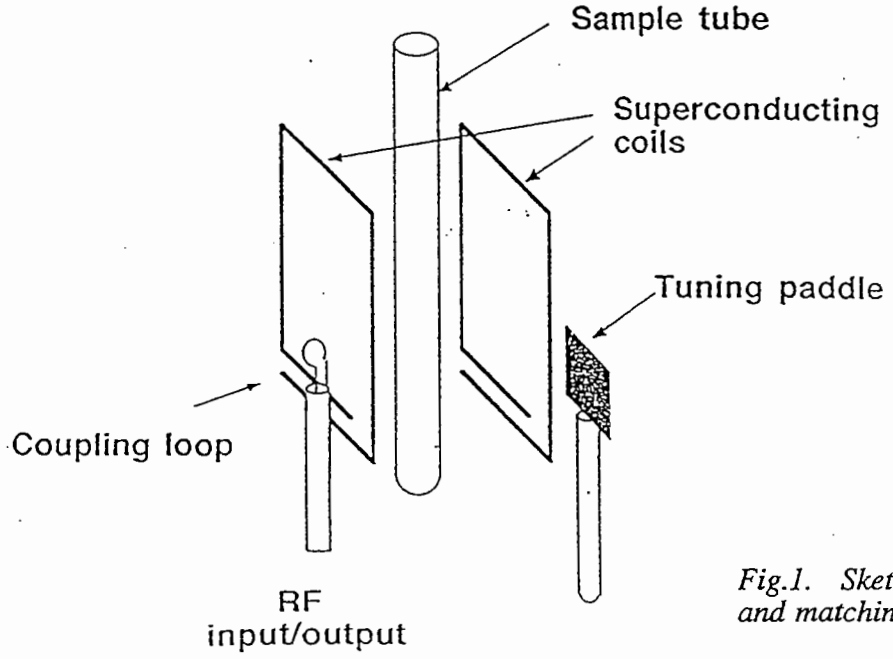


Fig.1. Sketch of coils with tuning and matching adjustments

Varian Associates, Inc 3120 Hansen Way Palo Alto, California 94304-1030 U.S.A.
415/493-4000 FAX 415/494-7186 Telex 348476

Both sensitivity and current handling have improved since our earlier report. For proton operation at 400MHz, the 0.1% ethylbenzene sensitivity is >2000:1, a factor of four improvement over normal probes, and 90° pulse widths are less than 12μsec. The current carrying capacity of the coils does saturate at high currents so that calibrations need to be carried out at a number of power levels. At low levels, the coils behave fairly linearly so that selective pulses can be applied as with normal probes.

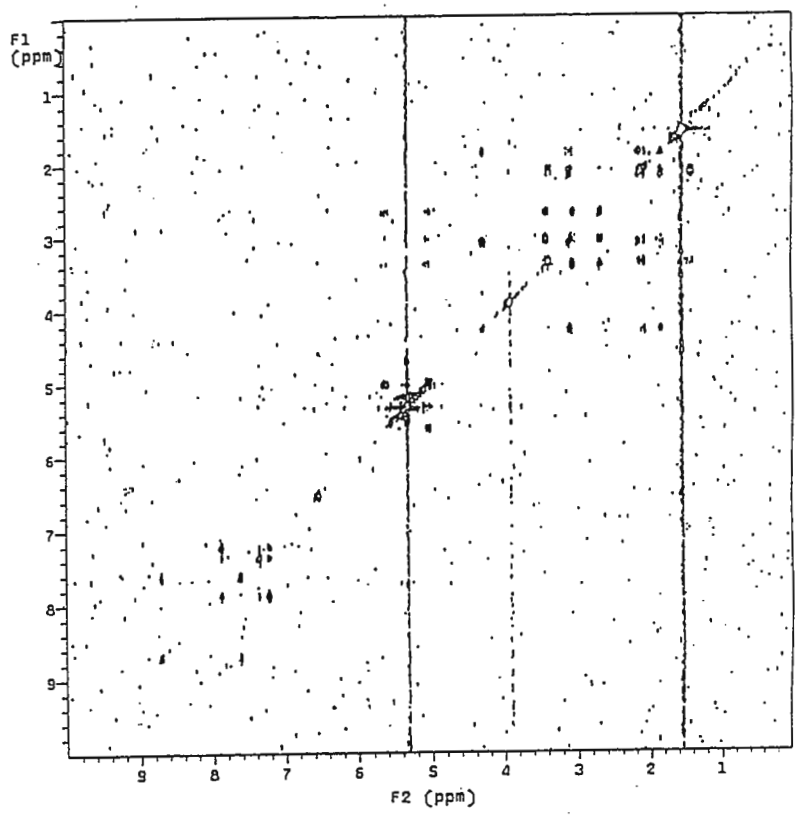


Fig.2. 400MHz TOCSY spectrum of 3mm sample of 40nmole quinine in CD_2CL_2 using superconductive probe. Mixing time = 60msec with a spin-lock field of 10kHz. Total acquisition time approx 1.5 hours.

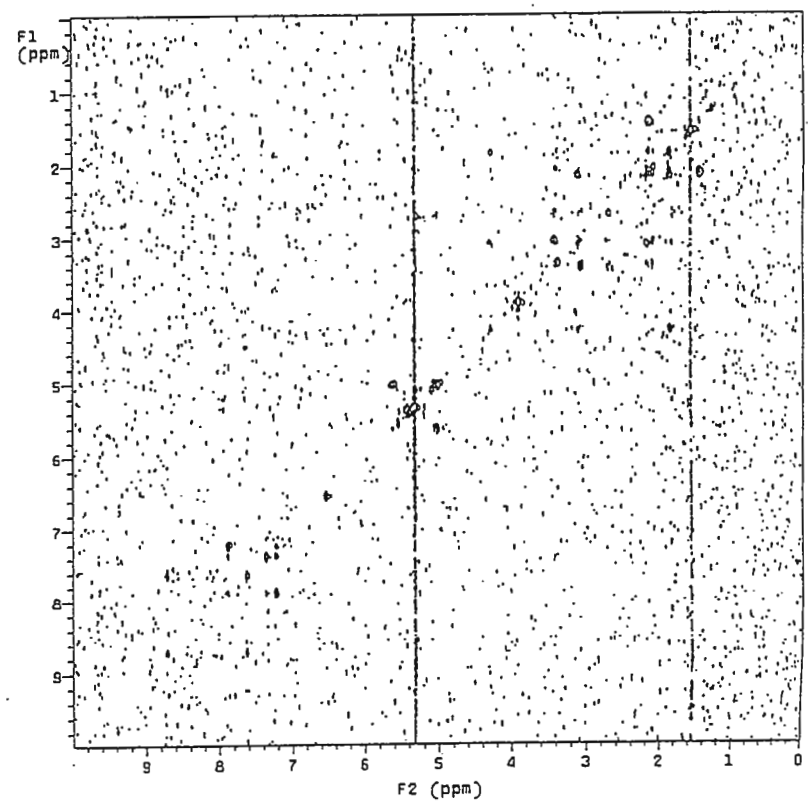
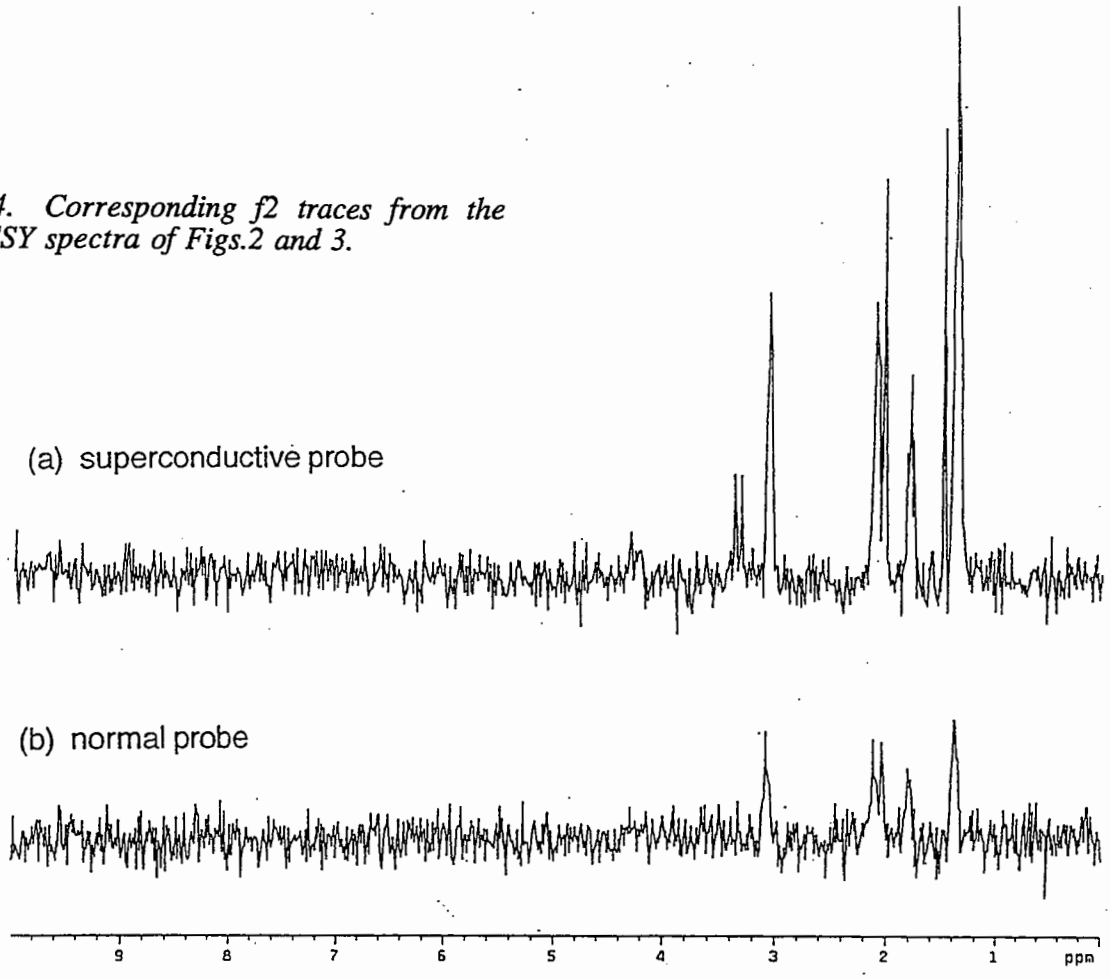


Fig.3. 400MHz TOCSY spectrum with normal probe. Same sample and parameters as for Fig.3.

Fig.4. Corresponding f2 traces from the TOCSY spectra of Figs.2 and 3.



The sensitivity improvement is realised for more complex experiments as shown by the 400MHz TOCSY results in Figs. 2 and 3 from the same 3mm sample of 40nmole (13.7µg) quinine in CD_2Cl_2 run in the supercon probe and a standard probe. In each case, the spin-lock rf field strength was 10kHz with a 60msec mixing time and presaturation of both the residual solvent peak and water peak was used. Corresponding traces from the two spectra are shown in Fig. 4.

We have operated other versions of the superconductive probes for proton observation at 500MHz and fluorine observation at 376MHz with similar sensitivity gains, and we have made some preliminary investigations on ^{13}C at 100MHz with promising results. For ^{13}C the coil Q cannot be as high as is used for protons because of the greater chemical shift range and the coils must carry higher currents to generate short 90° pulses. Nevertheless, we have seen significant sensitivity gains and have generated some good quality spectra.

We would like to thank our colleagues at Conductus and at Varian for their continued support with this project.

Kindest regards,

Howard. Wes

Howard Hill Wes Anderson

The University of Texas Medical Branch at Galveston

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

April 9, 1996 (received 4/20/96)

Dear Barry, **An Alternative to Adiabatic Pulses for Imaging**

We are looking forward to our INOVA upgrade on our 4.7 T horizontal bore SISCO (now Varian) system. Included in the upgrade is a gradient waveform memory which will motivate us to implement the most recent techniques for acquiring images with B1-insensitive adiabatic rf pulses. With our current configuration we have no acceptable spin echo multislice adiabatic imaging pulse sequence, which is unfortunate because we often use surface coils to acquire images of monkey brains. Surface coils provide defacto localization, improved access to the head for peripheral equipment (ventilation etc) and improved sensitivity compared to cylindrical volume coils.

In our experiments we acquire multislice spin echo images that provide full coverage of deep brain structures. In the squirrel monkey the deepest structures from the dorsal surface of the skull are 3.4 cm from the plane of our 6.5 cm surface coil. The problem with this configuration is that a single pulse power (at constant width) does not accommodate B1 drop off. Our first order solution was to calibrate the B1 field on a phantom, constructing a pulse power depth chart. We then acquire images in the plane of the surface coil utilizing an appropriate B1 power, which is tied to the slice depth in the multislice image set. Our images look great. We would show them here if it weren't for our poor luck with image reproduction in this news letter in the past. Therefore the slice profiles will have to suffice.

As you know, Dr. Leland L. Smith is retiring in August, 1996 .This means that we will have to bribe him with sherry to get him to wax scientifically, for in his words "... you cowboys are on your own, I don't have to put up with this horse$#!@ anymore", therefore please transfer his subscription to Dr. Michael Quast, University of Texas Medical Branch, Galveston, TX 77555-1143.

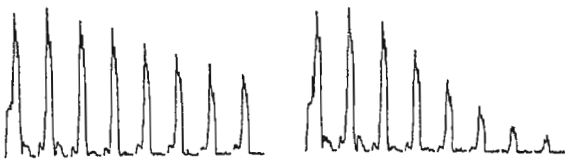

Figure: Slice profiles from two sets of a multislice spin echo images of a phantom at increasing depths from the surface coil plane. The set on the left was acquired with the pulse power incremented with increasing slice depth. The set on the right is with a constant pulse power for all slices. Each slice is 0.4 cm apart.

Mike Ed Stacy Bob L.L. Smith Fidel

M. Quast E. Ezell S. Klinke J. Gonzalez L.L. Smith G. Wei

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UNIVERSITY OF OREGON

Dr. Bernard L. Shapiro
The NMR Newsletter
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(received 4/22/96)
April 19, 1996

pH Titration "Curves" of Protein Amides

Dear Barry,

We often examine protein folding, the interaction of one protein with another protein, small molecule ligand, or hydrogen ions under conditions where fast exchange conditions hold. Under such conditions, it is sometimes important to establish if a simple two-state equilibrium holds (bound and free, folded and unfolded, etc.) or if more than two-states are required to describe the system under observation. The minimum number of rapidly equilibrating states can be easily established by examination of any two dimensional spectrum as the proportion of the molecules in the extreme states is varied. This is illustrated below for the pH titration of one domain of the chemotaxis specific protein kinase, CheA.

In the pH titration of the phosphotransfer domain $CheA_{1-134}$, we have found that changes in the backbone amide ^{1}H or ^{15}N chemical shifts parallel changes in the protonation states of nearby histidine residues. When we overlay all the ^{1}H-^{15}N correlation spectra from a titration series, most of the shifting crosspeaks follow straight lines in the overlayed spectra (For example, L30, G52, D36 or Q31 at the top of Fig. 1). This behaviour is consistent with a two-state model in which the two states represent the chemical environments of the amides at the low and high pH limits, respectively. In this model, the two states undergo fast exchange and the chemical shifts of the amide ^{1}H and ^{15}N nuclei at a particular pH are described by the following equations:

$$\delta_H = \delta_{H,1}\, 10^{-pH}/(10^{-pH} + 10^{-pKa}) + \delta_{H,2}\, 10^{-pKa}/(10^{-pH} + 10^{-pKa}) \qquad (1)$$

$$\delta_N = \delta_{N,1}\, 10^{-pH}/(10^{-pH} + 10^{-pKa}) + \delta_{N,2}\, 10^{-pKa}/(10^{-pH} + 10^{-pKa}) \qquad (2)$$

or equivalently,

$$\delta_H = \delta_{H,1} + (\delta_{H,2} - \delta_{H,1})\, 10^{-pKa}/(10^{-pH} + 10^{-pKa}) \qquad (3)$$

$$\delta_N = \delta_{N,1} + (\delta_{N,1} - \delta_{N,2})\, 10^{-pKa}/(10^{-pH} + 10^{-pKa}) \qquad (4)$$

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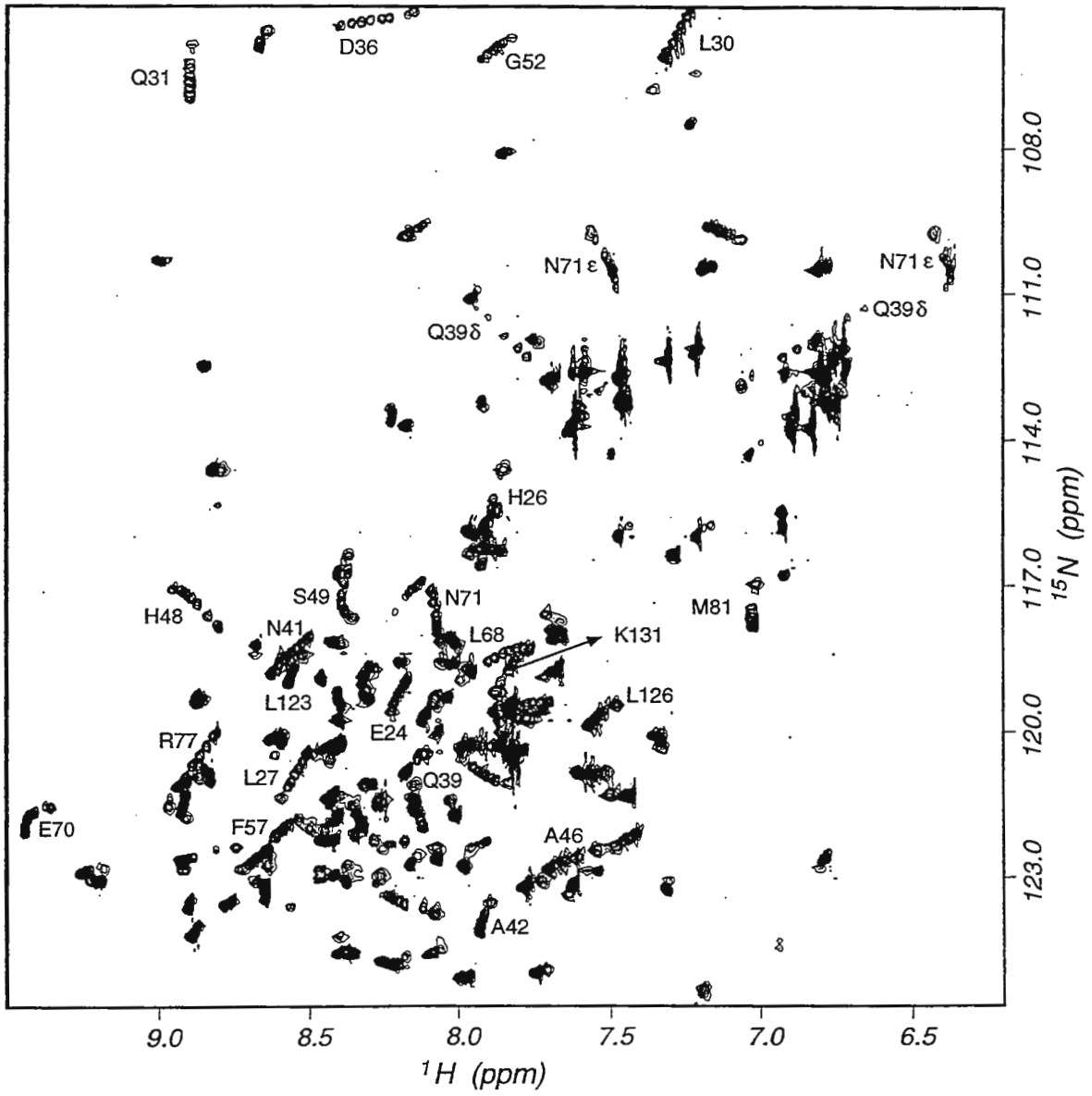

Figure 1. Overlay of 13 ^{1}H-^{15}N HSMQC spectra of $CheA_{1-134}$ representing pH range 5.2 - 9.7.

where $\delta_{H,1}$, and $\delta_{N,1}$ are the proton and nitrogen shifts at the low pH limit, and $\delta_{H,2}$, and $\delta_{N,2}$ are the proton and nitrogen chemical shifts at the high pH limit, respectively. Therefore, we have:

$$(\delta_H - \delta_{H,1})/(\delta_N - \delta_{N,1}) = (\delta_{H,2} - \delta_{H,1})/(\delta_{N,1} - \delta_{N,2}) \qquad (5).$$

This equation shows that the changes in proton and nitrogen shifts with pH are in proportion, and therefore lie along a straight line with a slope of $(\delta_{H,2} - \delta_{H,1})/(\delta_{N,1} - \delta_{N,2})$ in the overlay of ^1H-^{15}N correlation spectra.

Using the above two-state model, we have determined the apparent pKa values for some of the amides that exhibit large chemical shift changes with pH. These pKa values closely resemble the pKa values of the nearby histidine imidazole rings that were detected in another set of ^1H-^{15}N correlation spectra during the titration. By comparing the apparent pKa values of the amides with those of the imidazole rings, we have indirectly assigned the imidazole sidechain resonances to specific histidines according to the assignments of the backbone amides.

We also notice that some of the amide crosspeaks followed curved trajectories in the ^1H-^{15}N spectra (For example, H48, S49, and E70 in the lower left corner of Fig. 1). This nonlinear dependence suggests involvement of multiple titrating groups or conformational changes during the titration that cannot be simply described by a simple two-state model.

Best regards,

Hongjun Zhou Michael Strain Frederick W. Dahlquist

Worserthanwurst

From a long-forgotten source, the following is offered as an antidote to whatever cute acronyms may be encountered in the course of having one's NMR brain improved.

About 150 years ago, London was invaded by a huge and terrible monster who came upriver from the sea. Although the inhabitants were able to kill it promptly before it could do much damage, they couldn't decide what to do with the remains of the creature. At last, a butcher cut it up and made sausage from it. Thereupon, Charles Dickens, then writing for The Sun (not page 3), reported that "It was the beast of Thames, it was the wurst of Thames".

BLS

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Prof. Barry Shapiro
Editor/Publisher
NMR Newsletter
966 Elsinore Court
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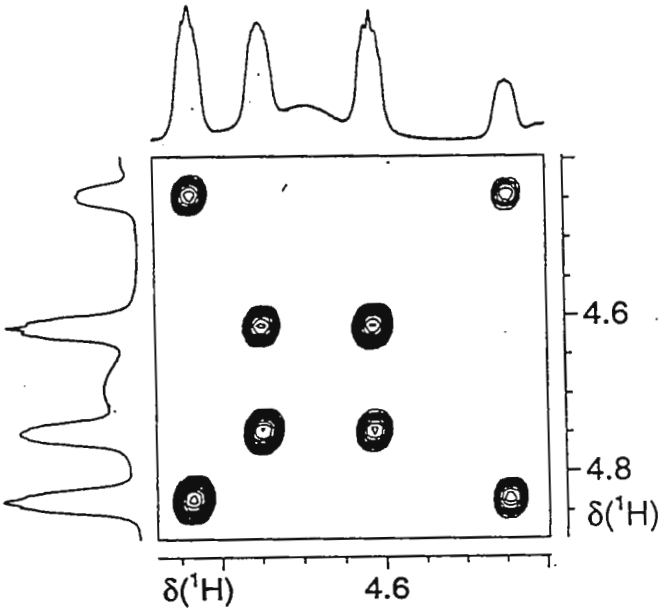
USA

April 9, 1996
(received 4/18/96)

Dear Barry,

Successful enantioselective catalysis requires that the chiral auxiliary maintain a "rigid" chiral pocket. The allyl complex $[Pt(\eta^3\text{-}C_3H_5)(2)]PF_6$, which has the P,S chelate shown, shows **four** isomers at 243K. The 2-D exchange spectrum reveals pairwise exchange for the syn allyl protons trans to the P-atom . One set arises from the two exo/endo allyl isomers and the other from inversion at the stereogenic sulfur-atom. The latter process, although slow,is still observable and suggests that thioether donors may offer "loose" chiral pockets.



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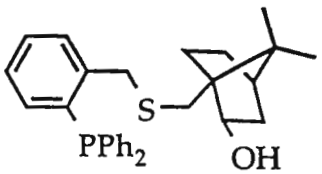

Sincerely,

Prof. P. S. Pregosin

Suggested Title: ^{1}H 2-D Exchange in a chiral Pt-allyl Complex.

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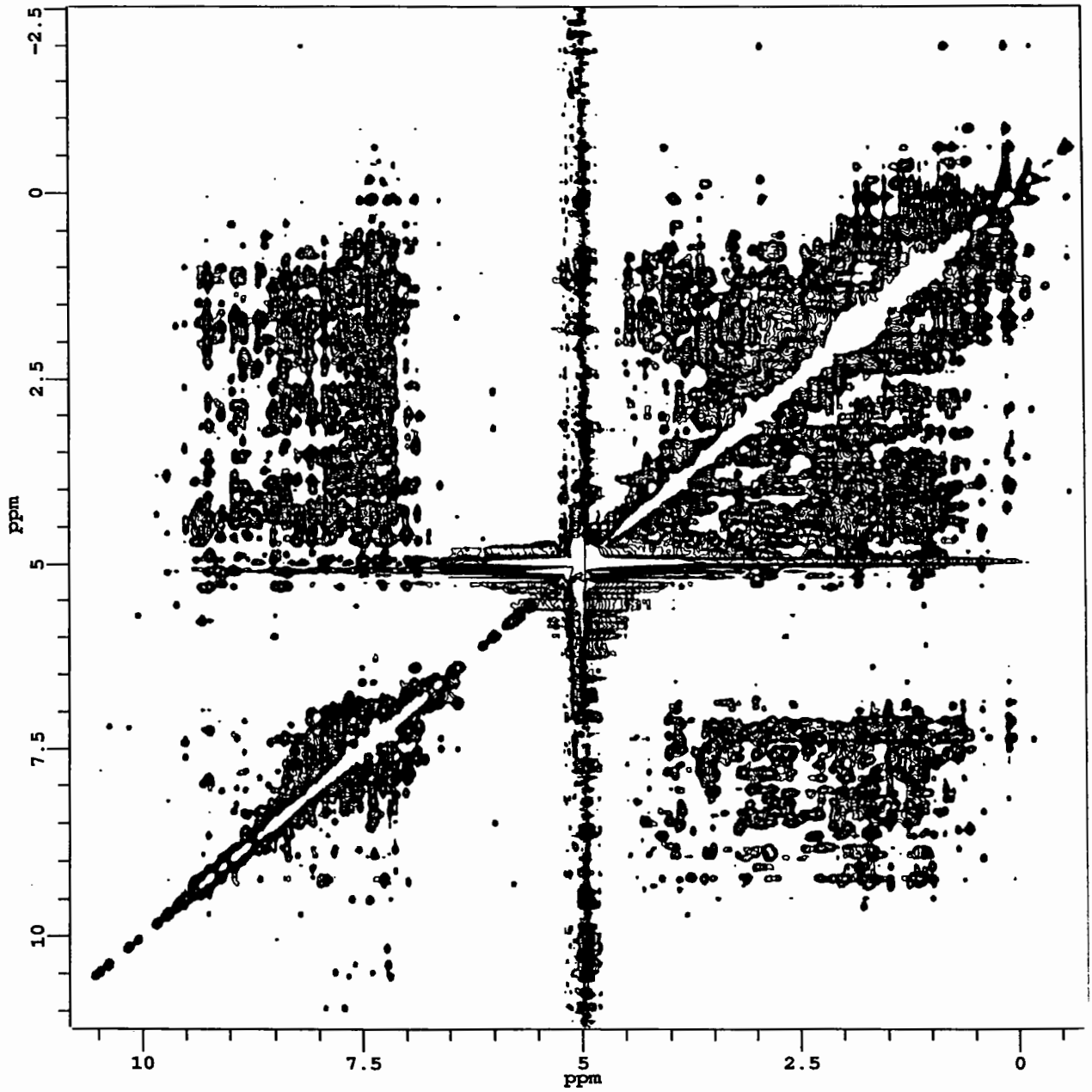

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April 15, 1996
(received 4/18/96)

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

Re: **MRI in the Environmental Sciences**

Dear Barry:

Hydrocarbon mixtures that constitute spills often exhibit low aqueous solubilities and persist in groundwater as separate, immiscible phases. Hydrocarbon liquids can be classified as non-aqueous phase liquids (NAPLs) or as DNAPLs, which are contaminants that are more dense than the aqueous phase. Of particular concern is the dispersion of DNAPLs, such as trichloroethylene (TCE) and carbon tetrachloride (CCl_4), or localized polyaromatic hydrocarbon (PAH) sludges scattered in the saturated zone. Hydrocarbon contaminants adsorbed to the matrix of soil or present as globules in the subsurface environment can persist for an indefinite period of time, serving as a source of persistent contamination even after many times the volume of water required to flush or dissolve the least soluble pollutant has been processed in a conventional pump-and-treat operation.

We have demonstrated that proton magnetic resonance imaging (MRI) techniques can be used to distinguish between hydrogen environments of aqueous and NAPL phases in-situ within core specimens, see Figure 1. The NAPL contaminant appears as a bright spot in a series of 2D slices from a water saturated core specimen, where experimental techniques have been implemented to contrast hydrogen in different environments on the basis of NMR relaxation time differences. In this case shown, contrast is based on differences in the proton spin-lattice relaxation times, T_1s. Using T_2 weighting protocols in spin-echo imaging experiments, we have been able to distinguish and visualize DNAPL nodules, see Figure 2. We also have been able to establish DNAPL migration patterns in core specimens, see Figure 3, during flushing with a variety of effluent phases, including water, foams and aphron systems. In addition, we have also designed new chemical-shift selective excitation pulse techniques to distinguish materials on the basis of differences in chemistries (*The NMR Newsletter*, **1994**,*435*, 17-18).

Sincerely,

Robert E. Botto
Chemistry Division

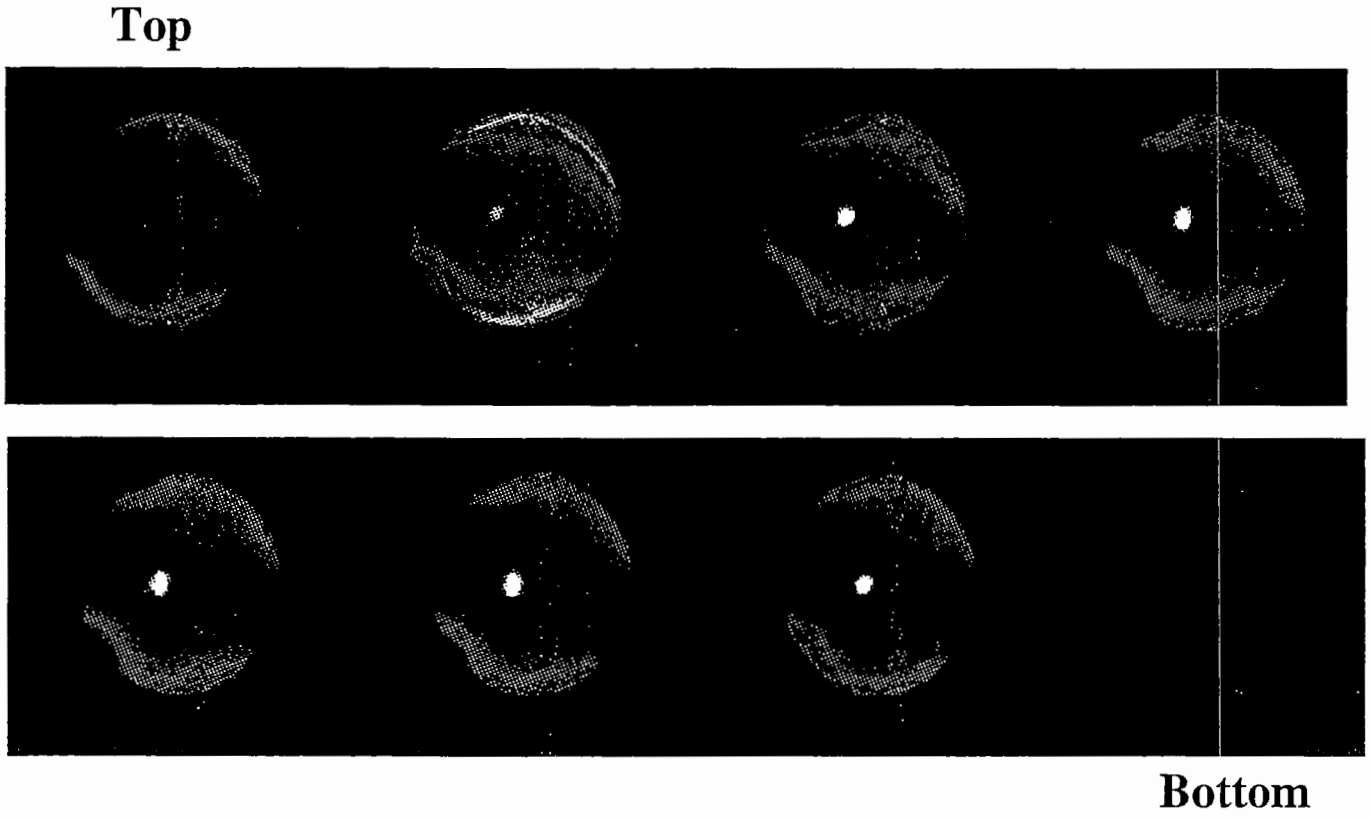

Figure 1. Typical 3-D MRI slices of NAPL containing sand core saturated with water; protons from NAPL appear bright as a result of T_1 contrast (Botto et al 1994).

Figure 2. Proton localized NMR spectroscopy of DNAPL ganglia (Botto et al. , 1994)

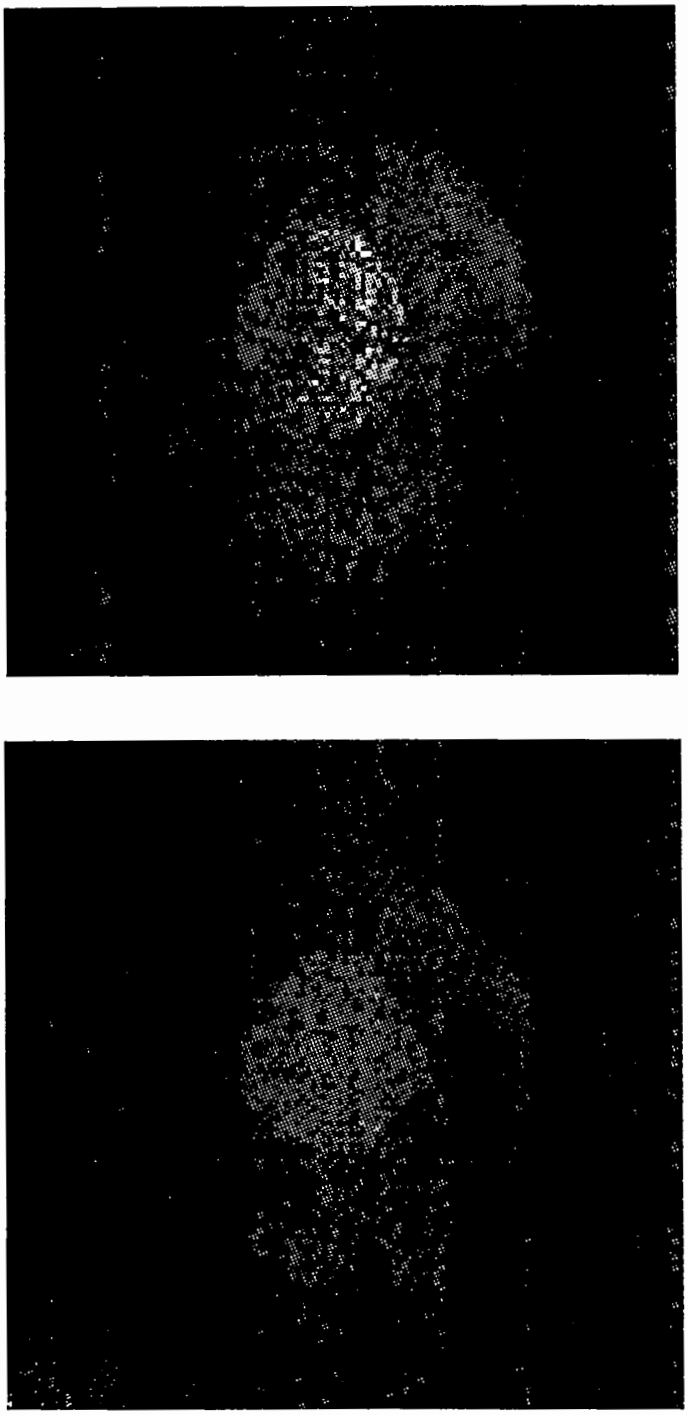

Figure 3 Flow MRI study of NAPL globule in 20-mm X 25-mm diameter cylindrical specimen of sand saturated with water; (top) MRI slice of initial specimen, and (bottom) identical MRI slice of specimen flushed with 1.3 liters of water for 1 hour.

Brock University

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Thu, Apr 11, 1996
(received 4/16/96)

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

^{27}Al MAS NMR of Synthetic Aluminosilicate Catalyst Supports

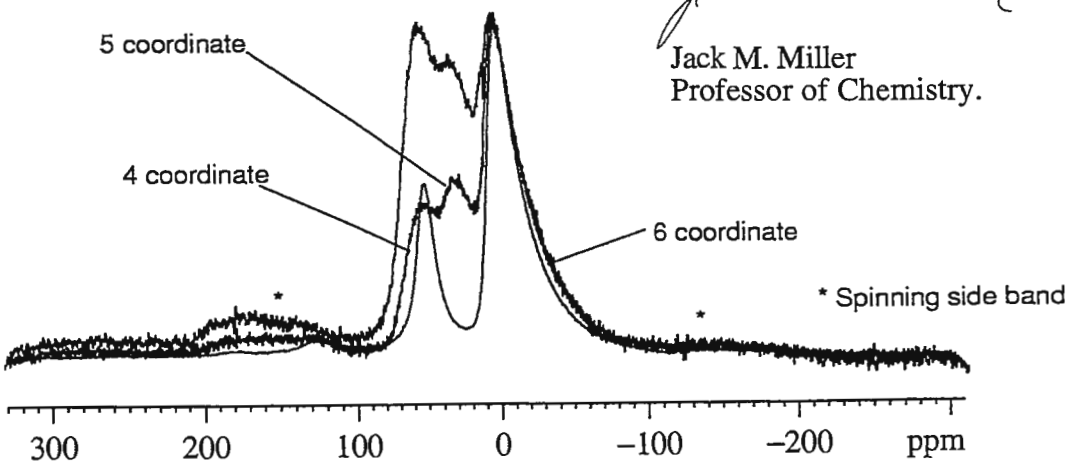
We have been learning the capabilities of our new narrow bore solutions/solids Bruker DPX 300 over the past few months. The instrument is basically a DRX but limited to only three channels. Having both a small solutions and solids sample changer on the system has tremendously increased the throughput on the system.

It is quite the change moving from our home-built Andrew-Beams type MAS probes with 3 KHz capability run on our old AC200 to a Bruker 4 mm CPMAS probe capable of 14-15 KHz on the 300. As we learn the trick of the trade, the reliability of the solids auto-sample-changer are improving.

As an example of some of our MAS work run by Dr. David Wails on supported catalysts the ^{27}Al spectra at 78.2 mHz are illustrated. When we have a metal halide supported on an synthetic Aluminosilicate, the material obtained at room temperature shows 4 and 6 coordinate aluminum. As the catalysts are heated to activate them for Friedel Crafts Alkylations five coordinate aluminum appears, initially at the expense of 4 coordinate, but then both 4 and 5 coordinate species increase at the expense of 6 coordinate aluminum. ThesE species are clearly seen at a 10kHz rotation rate for 4000 scans, taken with a 25 ms acquisition, 50 MS delay and 45° pulse. The traces starting from the bottom have been activated at room temperature, 200 and 500°C.The next step will be to try some of the new pulse sequences designed for quadrupolar nuclei..

Yours Sincerely,

Jack M. Miller
Professor of Chemistry.



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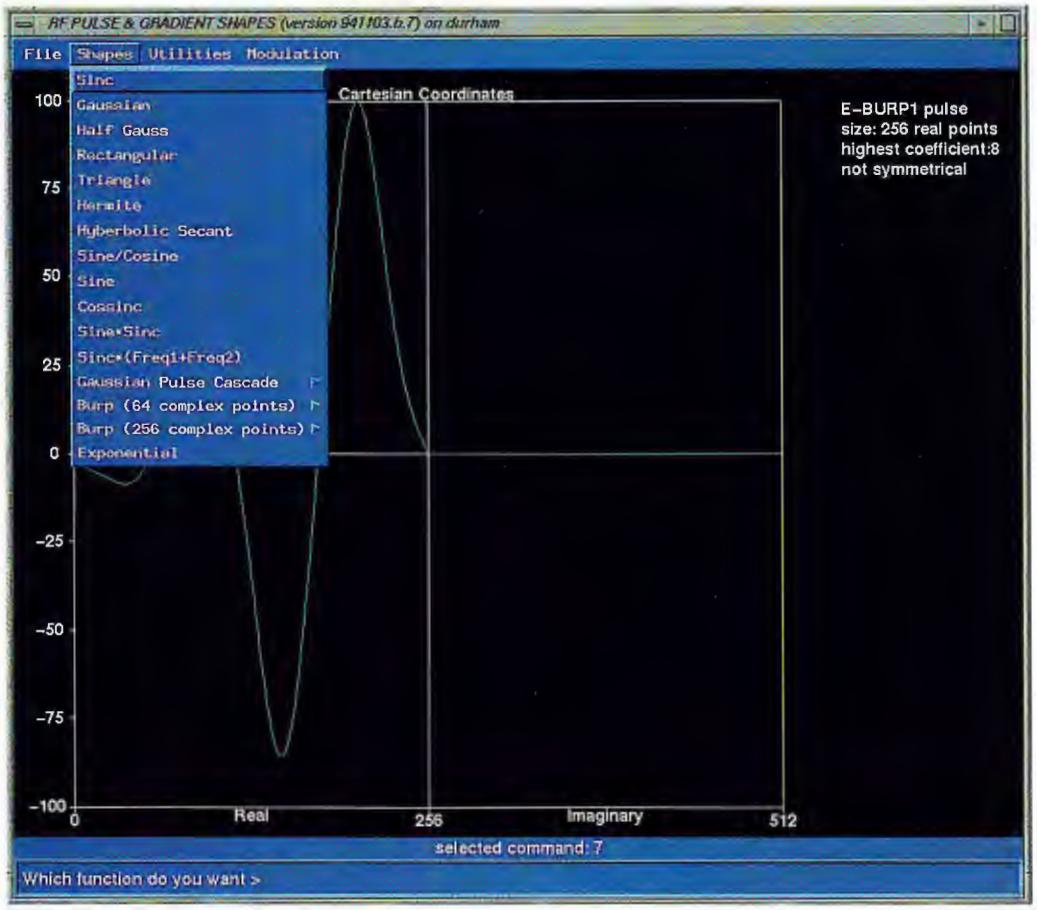

Figure 1: Select a shape and specify the parameters

Bruker's new NMR software package, *XWIN-NMR*™, offers a windows-based utility for creating RF pulse shapes, called *xShape*.

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- The program prompts you to enter appropriate parameters to completely define the shape.
- Write the shape to a file for use by the *XWIN-NMR*™ software.

- Finish by inserting the shape into your pulse sequence using the file that was written in *xShape*. This is easy and convenient for both routine and advanced applications.

Figure 2: Enter the optimized shape into pulse sequence

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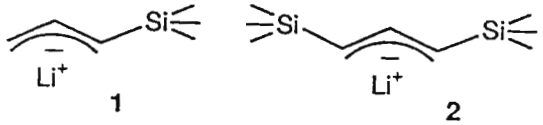
April 17, 1996
(received 4/22/96)

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

Allylic Lithiums,
Dynamics,
Partial Delocalization

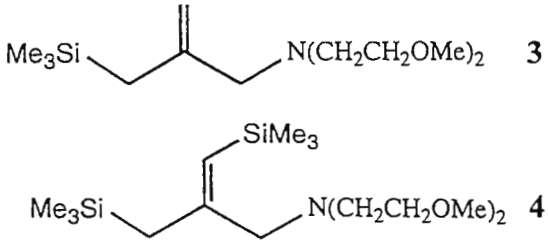
Dear Barry:

Allylic lithium compounds are ordinarily considered to be ion-pairs in which allyl is delocalized.



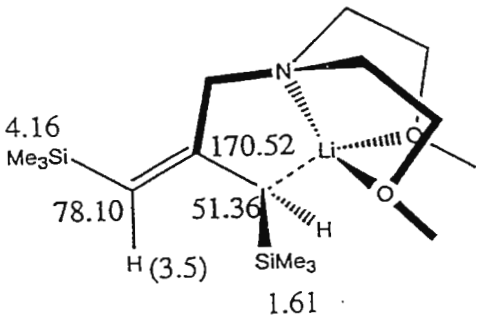
1

2

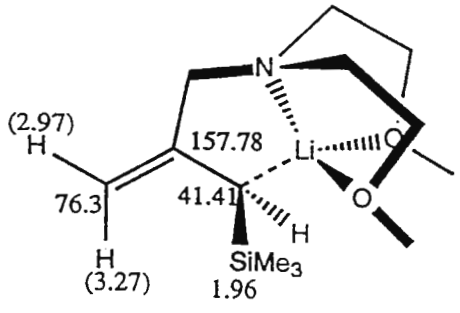


3 4

Compounds **1** and **2**, drawn without solvation, have been shown to be ion-pairs containing delocalized allyl anions. As expected so far allylic lithium compounds have not exhibited coupling between ^{13}C and directly bonded lithium.



5

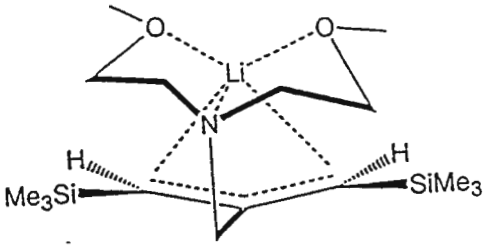


6

In contrast the products **5** and **6** of deprotonating **3** and **4**, respectively, using n-butyllithium in THF appear for the first time in such systems to be partly delocalized. Carbon 13 and (proton) shifts are indicated on the structures. Both **5** and **6** exhibit $^1J(^{13}C_1, ^6Li)$ values of 2.3 Hz and 3 Hz, respectively. Carbon-13 shifts of C_1 and C_3 in **5** (THF solution) 51 δ and 78 δ, respectively lie between those for known delocalized **2**, both at 67 δ and unsolvated localized neopentylallyllithium, C_1 at 20 δ and C_3 at 100 δ; there are small differences between the values for the cis and trans forms. These shift values imply that the allyl moiety in **5** is only partly delocalized. For lithium to be tridentately complexed to ligand

requires that N and Li lie in the allyl plane with methoxy's normal to the plane on two sides, as proposed in **5** (the numbers are ^{13}C shifts).

Above 270 K with increasing temperature the methyl silyl ^{13}C resonances of **5** begin to signal average as do the peaks for C, with C_3, the result of a progressively faster 1,3-lithium sigmatropic shift, the first observed. Lineshape analysis establish ΔH^* and ΔS^* to be 18 kcal/mol and 7.5 eng respectively.



7

We regard this process as taking place via a symmetrical transition state, **7**, in which the allyl moiety is more delocalized than in the ground state. This is the inverse of the process proposed to account for the influence of lithium ligand on the barrier to rotation of allyllithium in which case the transition state is more localized than the ground state with some C, Li covalence developed.

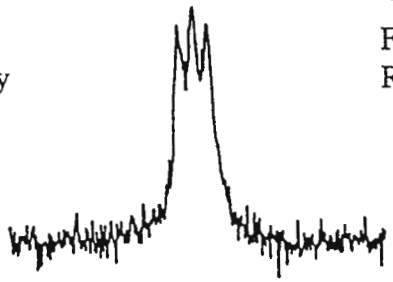
The structure of deprotonated **4**, proposed as **6**, is similar to **5**. All carbons on the pendant ligand are magnetically non-equivalent at 200 K. The ^{13}C, ^{6}Li coupling constant is 3 Hz. With increasing temperature three ^{13}C doublets due to O-methyl's, OCH_2's and N-CH_2 C's each progressively average to single lines at their respective centers. Lineshape analysis of each collapsing doublet gives rise to similar activation parameters ΔH^* and ΔS^* of 8 kcal/mole and -4.7 eu, respectively. This process, phenomenologically is the result of inversion at carbon bonded to lithium.

That **5** and **6** are so different in structure and behavior compared to their externally solvated ion-paired analogs is entirely due to their (**5** and **6**) stereochemistry of solvation.

Best regards.
Yours Sincerely,

Gideon Fraenkel
Professor of Chemistry

Fayang Qiu
Research Associate



C13 NMR C1
Cpd 6
J(C13, Li6)
= 3 Hz

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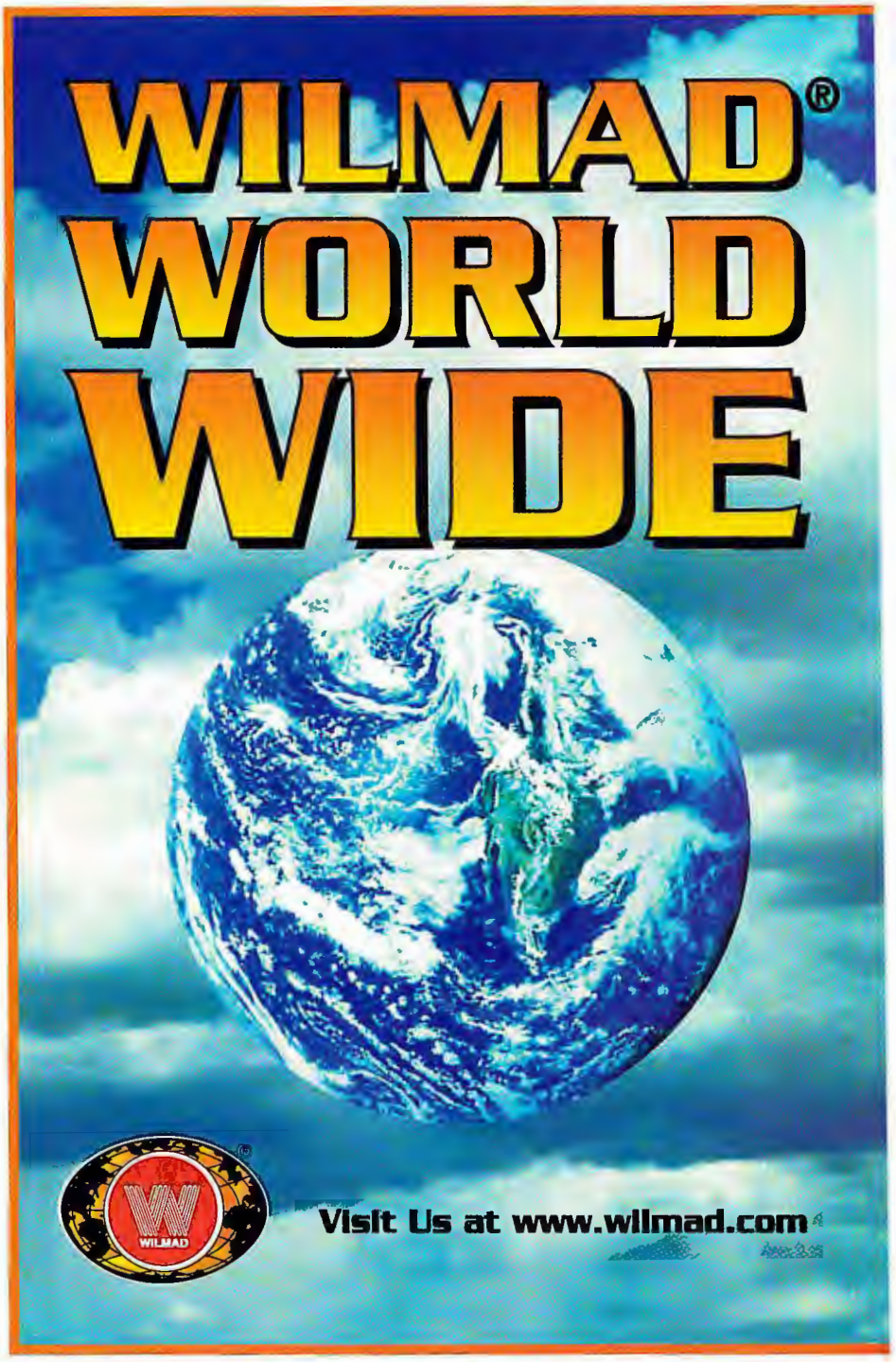

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Determination of J_{PH} in DNA (& Adobe Illustrator Update)

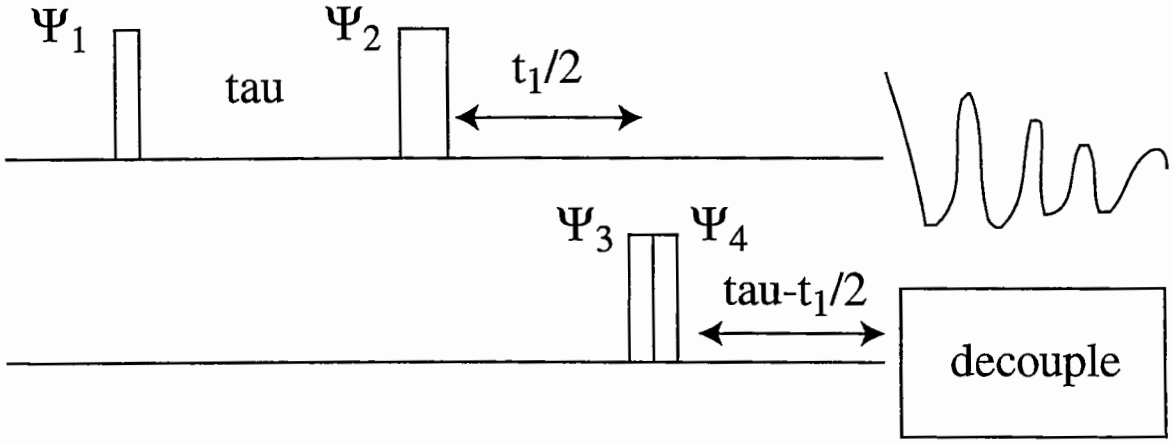
Tuesday, April 16, 1996
(received 4/18/96)

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

Dear Barry:

For sometime now we, and many others, have been interested in the heteronuclear J_{PH} of DNA. These couplings can be quite useful in structure refinement and we have found that the refinement of duplex DNA structure is an erratic process when these couplings are not used. A number of methods have been proposed for determining these heteronuclear couplings and these couplings have been reported for a number of DNAs by us, the groups of Feigon, Gorenstein, James, Kessler, Reid and others. The basic problems with the detemination of these couplings are that the proton-phosphorus couplings are small, the phosphorus is coupled to many protons and the dispersion of the phosphorus spectrum is typically not very impressive. Kessler and co-workers have recently shown how to determine these couplings using ^{13}C-^{1}H ECOSY type correlation experiments at natural abundance but we can not do these types of experiments until our new magnet with its 8 mm pfg probe arrives in a little while. These natural abundance experiments are of fairly low sensitivity.

Thus, we have been examining the utility of carrying out constant time, heteronuclear, difference J experiments. The pulse sequence for such an experiment is quite straightforward as shown below.



Ψ_1 0 Ψ_3 0 obs 0 2 2 0
Ψ_2 0 0 1 1 Ψ_4 0 2 0 2

The sensitivity is high since only proton magnetization is excited and detected. The F1 traces can be considered to be 1:-2:1 pseudo-triplets with the spacing between the lines giving the heteronuclear coupling. The pulse sequence as written is not efficient since half the time is spent obtaining the same reference spectrum. We are now working on the variation of obtaining a single reference spectrum, as can be done in ECOSY experiments, and subtracting this from all of the CT free induction decays.

Figure 1 shows typical data obtained on a nucleotide and on this sample very high resolution data can be readily obtained quite easily and the heteronuclear coupling determined to a high level of precision. The application of the method to DNA is primarily limited by the T_2s of the protons. The use of a tau of more than 70-80 msec leads to quite weak signals.

Sincerely,

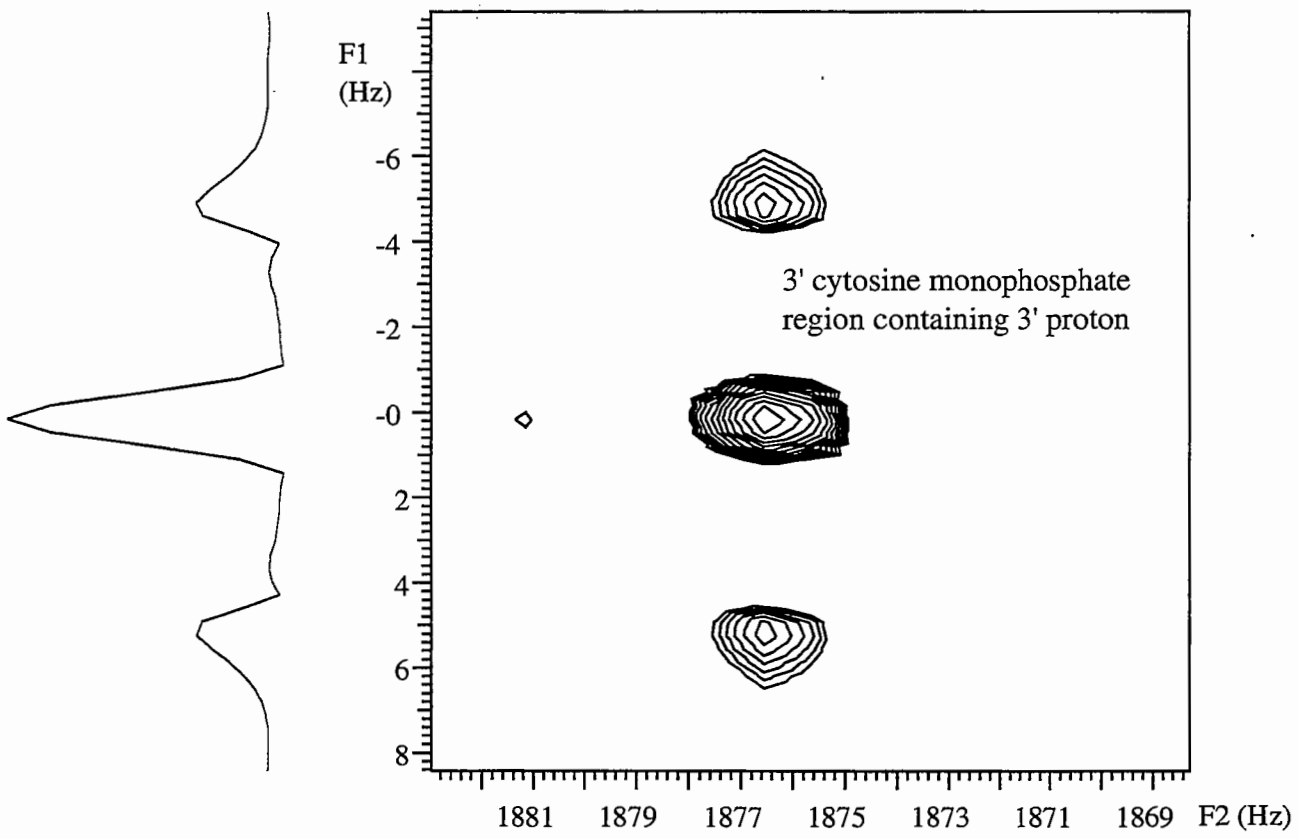
Ke Yu Wang

Philip Bolton

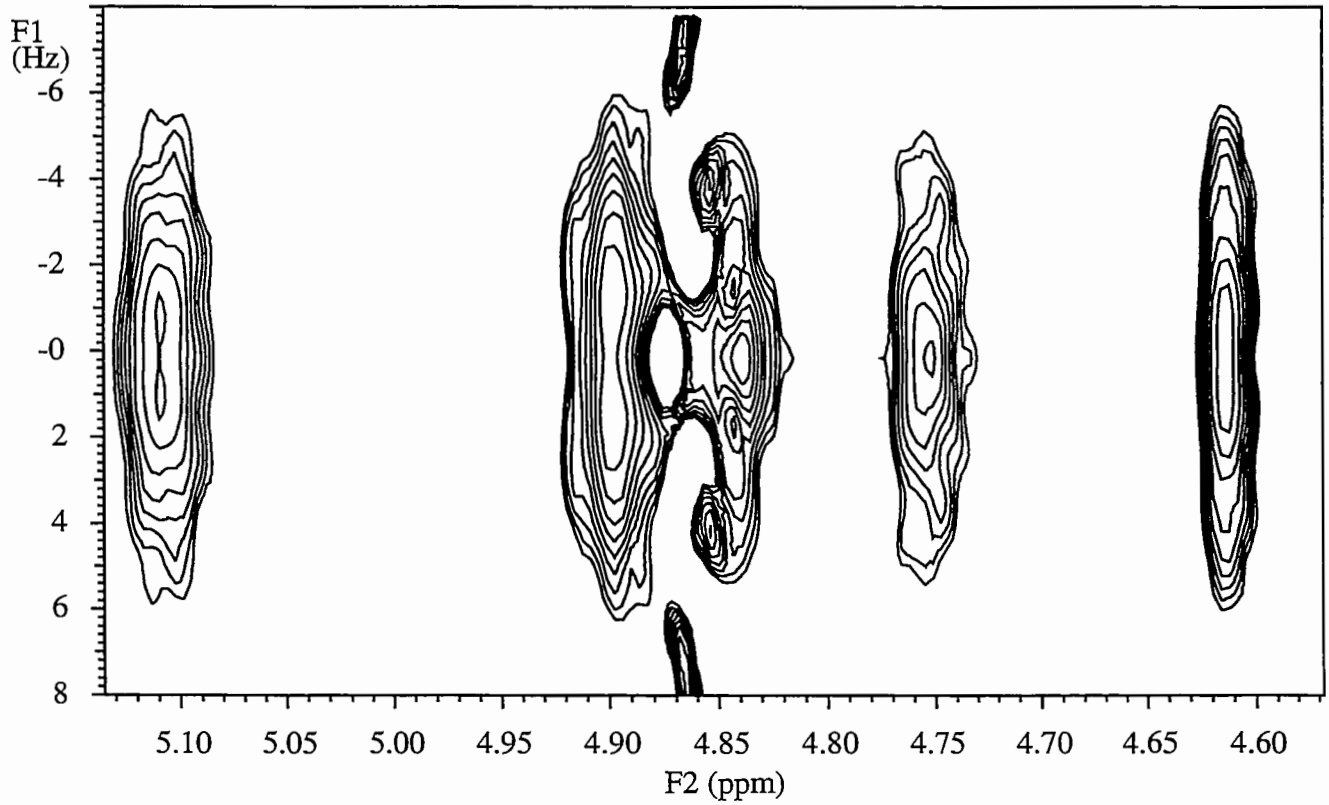
ps. Adobe Illustrator 6.0 opens postscript files directly avoiding those nuisance conversion steps. Also, version 6.0 allows converting Illustrator documents to JPEG so the Illustrator rasterizer can be used in the preparation of slides rather than relying on the lousy one that came with our Polaroid slide machine.

Position Available

Ke Yu Wang has just accepted a position at Gilead Sciences and will be joining them in the very near future. Thus, there is a post-doctoral position available to study the structures, metal ion binding and dynamics of aptamer, telomere and other quadruplex DNAs in an NIH funded study. We have a Varian Unityplus 400 which is being upgraded to include 5/8 mm pfg probes and a Varian Inova 500, three channel spectrometer with pfg 5/8 mm probes whose installation begins next week. The group has extensive computational support including IBM and SUN workstations.



DNA Aptamer (dGGTTGGTGTGGTTGG):
sw=2994.0 sw1=16.0 CT=1024 ni=8 fn=np=4096 fn1=128 d1=1.0 temp=15.0



The NMR Newsletter - Book Reviews

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

Multidimensional NMR in Liquids:

Basic Principles and Experimental Methods

by

Frank J. M. van den Ven

VCH Publishers, Customer Service Center, 303 Northwest 12th Avenue,
Deerfield Beach, FL 33442-1788; 1995
ISBN 1-56081-665-1 (Hbk). 399 pages. $55.00

Persons contemplating NMR textbooks at the graduate level will wish to examine this text carefully. The first impression is of a highly math-oriented venture into the physics of spin systems. The reader is pointed to a series of appendices - Linear Algebra, Recipes of Quantum Mechanics, and Angular Momentum - not to provide remedial instruction, but rather to apprise the reader of what is going to be required to get the most out the text.

Chapter 1, Basic Principles, ranges from the usual description of an NMR spectrometer, the vector system for picturing the behavior of spin systems in electromagnetic fields, and Fourier transformations, to the more rarefied air of the quantum mechanical descriptions of Hilbert space and Liouville space. Chapter 2 follows a brief introduction of chemical shifts and J-coupling with two sections on product-operator formalism, a section on relaxation of coherences and another on chemical exchange. Chapters 3 and 4 deal with one- and two-dimensional NMR, respectively. Chapter 5 extends the treatment to n-dimensional NMR, while the final chapter (6) concludes with more on relaxation processes. There is an extensive list of items for additional reading, concluding with a comprehensive four-page index.

I am not sufficiently into the mathematical side of NMR to present a critique of the many equations in this book. However, the author's approach often seemed quite novel. Furthermore, the explanations accompanying the equations make abundantly clear the meaning and significance of each. One can literally muddle through much of the math and still emerge with a better understanding of what is going on in these systems than with some more descriptive explanations. As I settled into my reading, I became progressively amazed how matters which had been hitherto obscure became increasingly clearer. There are a number of informational nuggets among the equations, and a sense of delight settles in on the reader as he begins to discover these. In other words, this volume is worth the effort.

We are sorry to note here that Dr. van den Ven died a few months ago.

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

Old Tricks for (sort of) New Kinds of Samples.
Nuclear Magnetic Resinance

April 23, 1996
(received 4/25/96)

Dear Barry,

Just like everyone else in the pharmaceutical industry (or so it seems) we are interested in crosslinked polystyrene resins to be used for solid phase combinatorial syntheses. For our part, we would like to develop NMR tricks which will aid in structural characterization of solvent swollen, resin bound organic structural units. By now, everyone knows that the place to start is with a magic angle spinning probe. We agree and have ordered one from Doty Scientific. However, we are receiving samples now and need to make do with the conventional probe technology we currently possess. In order to better serve my clients, I decided to modify some of the techniques that we use to analyze small molecules to make them more suitable for the analysis of resin bound moieties. The techniques I wanted to adapt first were those designed to determine the number of hydrogens bound to a carbon. As described below, the implementation of some of these was more of a challenge than I had bargained for.

I first took a look at the DEPT sequence. The sample used here was a CD_2Cl_2 swollen Knorr MBHA resin available from Advanced ChemTech, the structure for which is shown at the bottom of this letter. All spectra in this note were collected on a seriously aging Varian XL-200. Not surprisingly, the proton spectrum (**1**) obtained on this sample is essentially useless, showing broadening due primarily to motionally attenuated proton-proton dipolar interactions. On the other hand, the ^{13}C spectrum (**2**) contains many nicely resolved resonances in spite of residual chemical shift anisotropy effects. The quality of this spectrum offered the hope that proton multiplicities could be readily determined by conventional techniques. Sadly, however, DEPT spectra (**3**, $\theta = 45^{\circ}$ and **4**, $\theta = 135^{\circ}$) are singularly uninformative due to the poor signal to noise of these samples.

Undaunted, I pressed on. Because the poor DEPT signal to noise is undoubtedly due to the short proton T_2's of the sample, I considered that an alternative polarization transfer technique might be more successful. Unfortunately, I am old enough to remember that before there was DEPT, there was Refocussed INEPT. R-INEPT has the advantage of using shorter delays following the magnetization creating pulse than does DEPT. For example, R-INEPT produces its FID only 5.25 ms (for all protonated carbons in phase, tuned to J=142 Hz) after the initial pulse in contrast to DEPT which requires 14 ms. Encouraged by this realization, I hastened to reimplement R-INEPT on the XL-200 (it had disappeared who knows when or to where), checked out the sequence with standard samples (data not shown, but it looked fine), and tried it out on the MBHA resin sample. As predicted, the signal to noise of the R-

INEPT for Δ = 1.75 ms (**5**) was far superior to the corresponding DEPT display (θ = 45°). However, the Δ = 5.25 ms spectrum (**6**) gave a surprising result in that at least some of the aromatic CH's were out of phase with the Δ = 1.75 ms spectrum. Examination of the Δ = 1.75 ms spectrum reveals that this effect is also present here; some of the aromatic peaks are about 90° out of phase with the others. These effects are probably due to perturbations of scalar couplings by incompletely averaged proton-proton dipolar splittings. If this is the case these interactions could be eliminated by the imposition of any one of a number of multiple pulse decoupling schemes during the delay periods. Whatever the cause, it appears that polarization transfer experiments are not well suited for characterization of gel phase resin samples in a conventional probe. These problems should be eliminated by carrying out the experiments in a MAS probe.

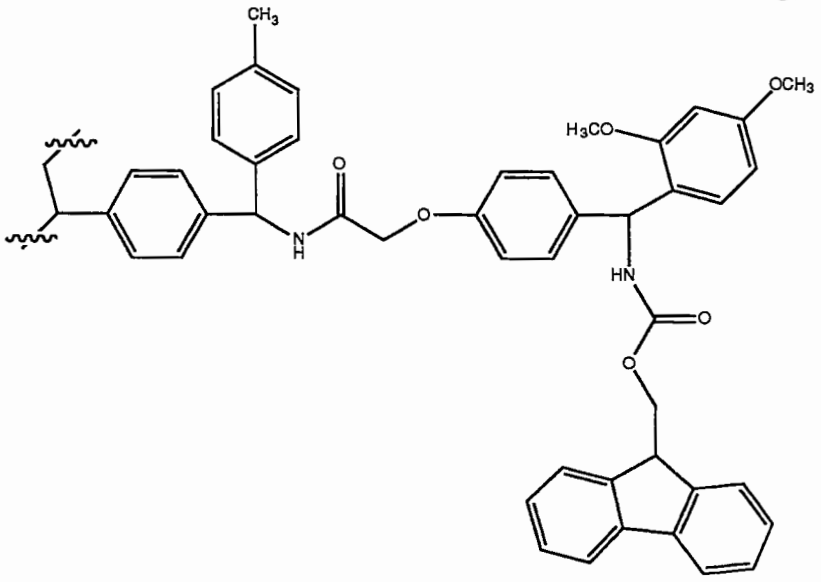
Happily, the APT technique seems to work well in these systems (spectrum **7**). As traditionally implemented, APT allows the use of less than a 90° signal generating pulse, a necessity for general application to small molecules. This is achieved by an additional 180° pulse, a couple of 1 ms delays, and some phase cycling. For gel phase resin samples, T_1's are typically pretty short, so it is usually possible to use a 90° pulse for survey purposes. Hence, a standard interrupted decoupling spin echo sequence (no additional 180° pulse) should provide a sensitivity advantage over APT, but only for the very broadest lines. The spectrum (**8**) collected with such a sequence is virtually identical to the APT spectrum.

As you can see, my attempts to improve these techniques didn't work out very well on a conventional probe. However, even on a MAS probe, T_2's for these resin samples are shorter than for small molecules, and many of the suggestions proffered here will be valid. We look forward to carrying out these (and other) experiments on our MAS probe. I want to thank Jim Prestegard and Joel Tolman of Yale University for helpful discussions regarding this contribution.

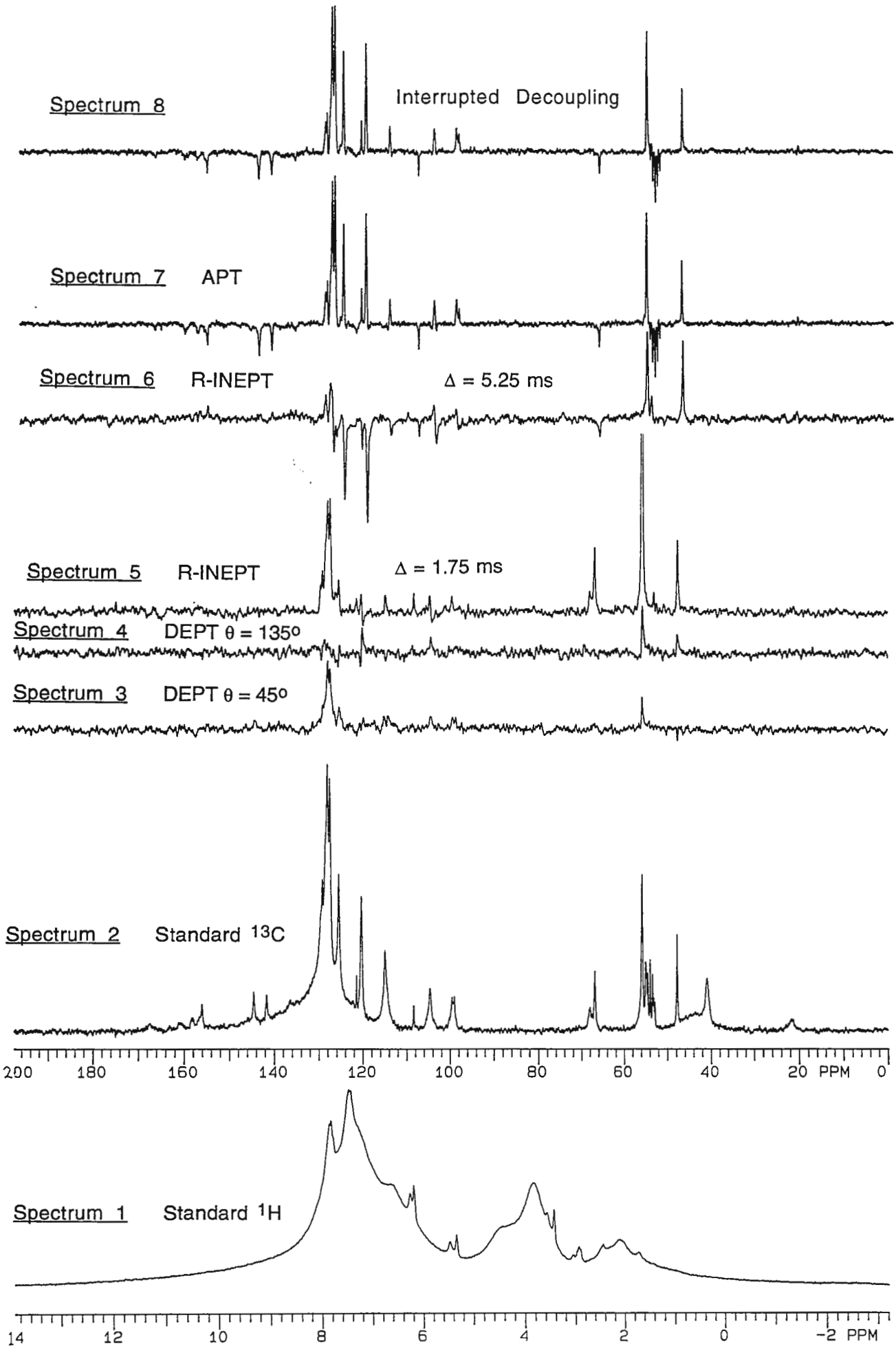
Sincerely,

Andy

C. Anderson Evans



Knorr MBHA resin


Spectrum 8
Interrupted Decoupling
Spectrum 7
APT
Spectrum 6
R-INEPT
Δ = 5.25 ms
Spectrum 5
R-INEPT
Δ = 1.75 ms
Spectrum 4
DEPT θ = 135°
Spectrum 3
DEPT θ = 45°
Spectrum 2
Standard 13C
200
180
160
140
120
100
80
60
40
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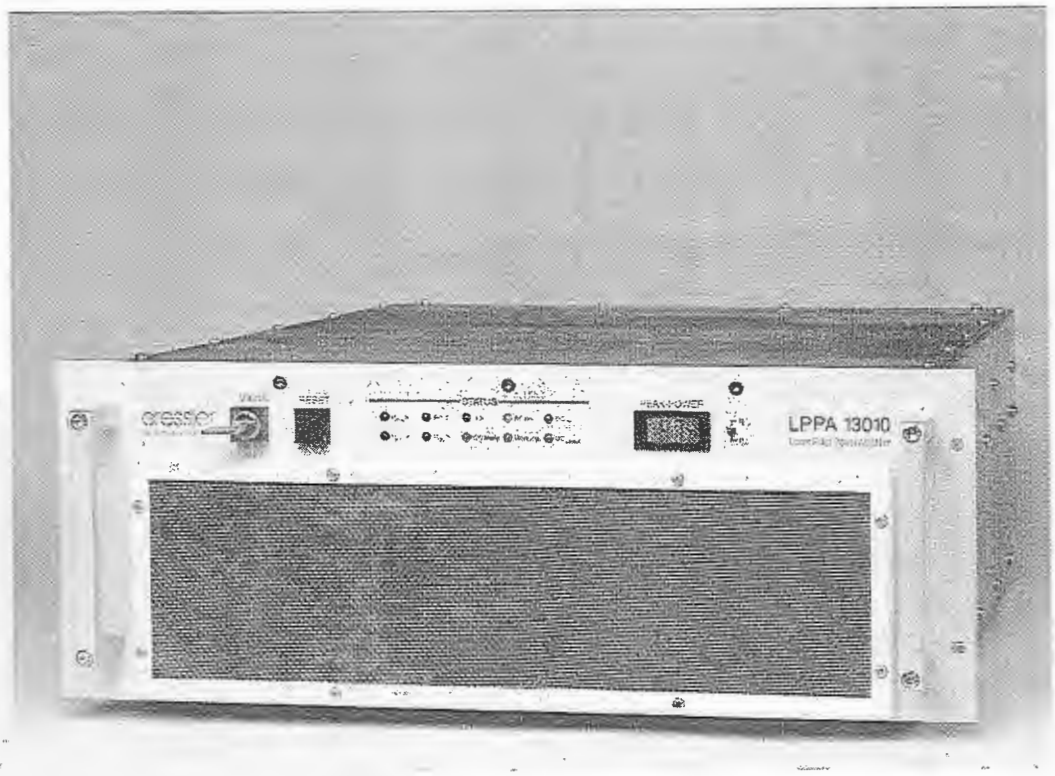
Pulse Power 400 - 8000 W

Frequency ranges within 10 - 500 MHz



Linear RF amplifiers for NMR/MRI applications

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hochfrequenztechnik gmbh

dressler USA
Lowell A. Beezley
2501 North Rose Drive
Placentia, CA 92670
Phone (714) 528-7217
Fax (714) 528-3513

Technical performance:

	LPPA 13010	LPPA 13020	LPPA 20010	LPPA 5004	LPPA 20080	
Frequency	10 – 130 MHz	10 – 130 MHz	175 – 205 MHz	200 - 500 MHz	175 – 205 MHz	
RF pulse power	1000 W	2000 W	1000 W	400 W	8000 W	into 50 Ohms
	700 W	1400 W	–	–	–	(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					
	● RESET push button and indicator					
	● RF on	● Blanking	● P forw.	● DC RF	● CW Mode	
	● P*T	● P DC	● P reflected	● DC control	● Thermal overload	
Interface	Description see text above					
	Input levels 5 – 24 V DC. Outputs 5 V TTL					
	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

dressler
hochfrequenztechnik gmbh

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Phone (+49)24 02/7 10 91 + 7 10 92
Fax (+49)24 02/7 10 95 • Germany

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PO Box 218, Lindfield, NSW 2070
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Facsimile: (02) 413 7631
International Facsimile: (612) 413 7631

4th April 1996
(received 4/20/96)

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

RE: The effect of electric field on the nmr spectra of lipid dispersions.

Dear Dr. Shapiro:

We have been using solid-state NMR to study the effect of electric fields on dimyristoylphosphatidylcholine (DMPC) deuterated at the choline methyls and the headgroup α and β methylenes.

Hydrated DMPC dispersions and mixtures of DMPC with egg yolk phosphatidylcholine (EYPC) were placed between gold coated glass coverslips and subjected to fields ranging between 1 MV m^{-1} and 16 MV m^{-1}, using biphasic pulses which were generated either continuously or synchronously with the NMR pulse program [1]. Continuous frequencies were varied between 100 Hz to 10 kHz, and the synchronous pulse lengths from 1.0 ms to 100 ms. The effects observed include an irreversible collapse of the deuterium quadrupolar splitting and the appearance of a reversible isotropic line. ^{31}P NMR was used to monitor the phase of the lipid. Sample hydration and power dissipation were assessed using electrical impedance spectroscopy. The resulting ^{2}H spectra were consistent with the formation of partially reversible powder like lamellar structures from aligned samples, in agreement with the results of ^{31}P NMR spectra.

The headgroup did not appear to move in response to the fields, which were estimated to be up to 16 MV m^{-1}. Field focussing within the sample may have increased the field intensity beyond this value by up to a factor of x5 (reference 1). This suggests that the long range fields due to action potentials in natural membranes do not directly affect the headgroup component of lipid bilayers although they may have an effect by modulating the surrounding ionic milieu.

Shorter electric field pulses are highly desirable because they would allow the use of well hydrated membrane samples and would more effectively express the field across the membrane

while permitting much more intense field pulses to be generated without sample breakdown. Typically 0.1ms to 1ms pulses would be appropriate. Short field pulses synchronised with the FID acquisition should be achievable, without broadening the NMR spectral response, by experiments in which the free induction decay is time sliced and synchronised with the electric field pulse. It is felt that this technique will enable observations of electric field induced conformational changes at the molecular level using NMR spectroscopy.

1. Osman, P. and Cornell, B. (1996) *Biochim. Biophys. Acta* **1278**, 160--168.

Sincerely,

Peter Osman

Frances Separovic

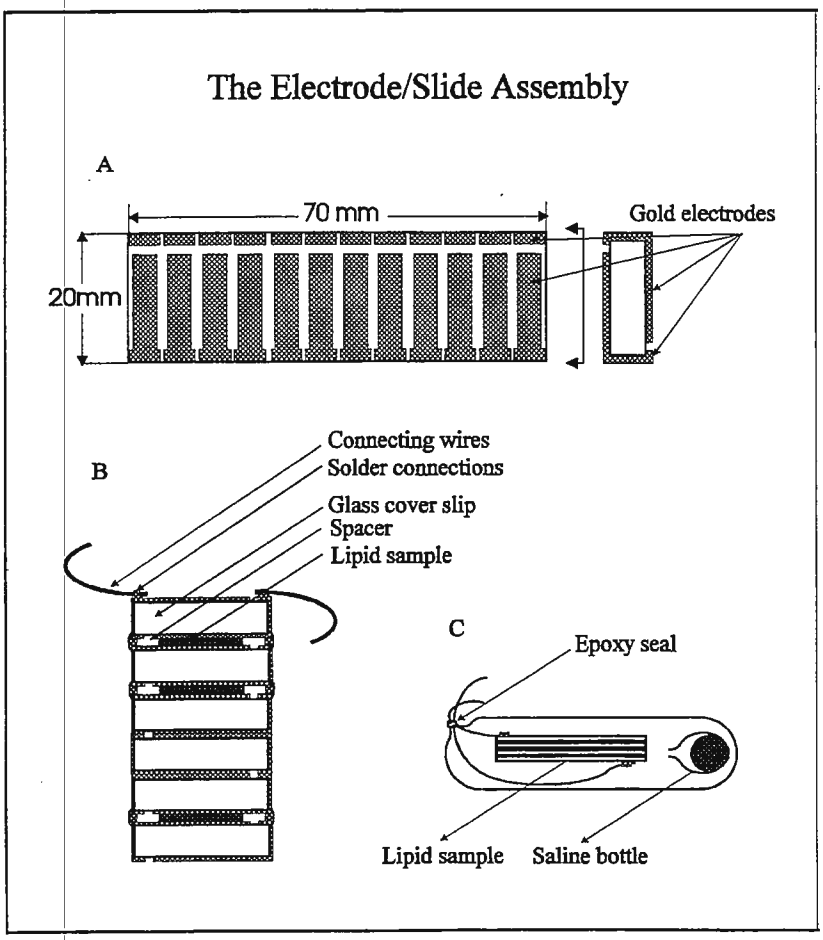

Figure 1. Schematic of the slide assembly. A) shows a single glass slide with the gold electrode pattern, including a section view (not to scale). B) shows a stack of glass slides spaced at 100μm with the lipid sandwiched between electrodes and in which alternate electrodes are connected to one or other excitation wires. C) shows the assembled slide sample encased in a hermetically sealed glass tube containing a vial of saline solution or water.

An Example of The Electrode/Sample Assembly

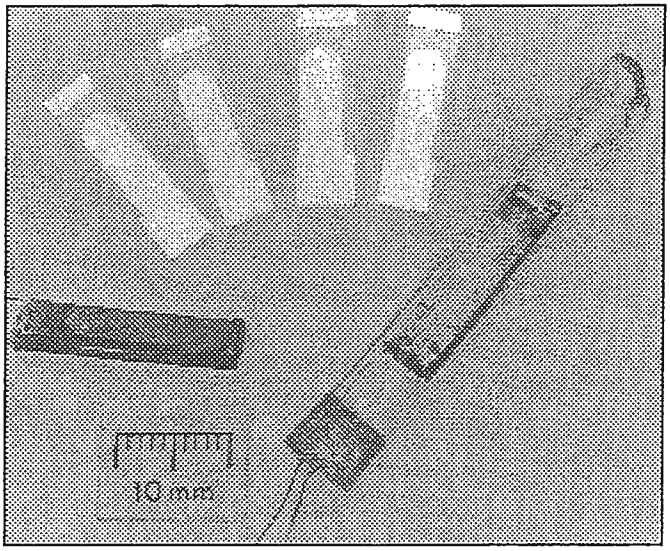

Figure 2. Three items are shown: on the right is an assembled NMR sample tube containing an electrode stack and a vial of saturated salt solution, in the top centre are four 5 mm x 20 mm electrode strips and to the left is a stack of electrode strips which have been assembled and which have connecting wires attached.

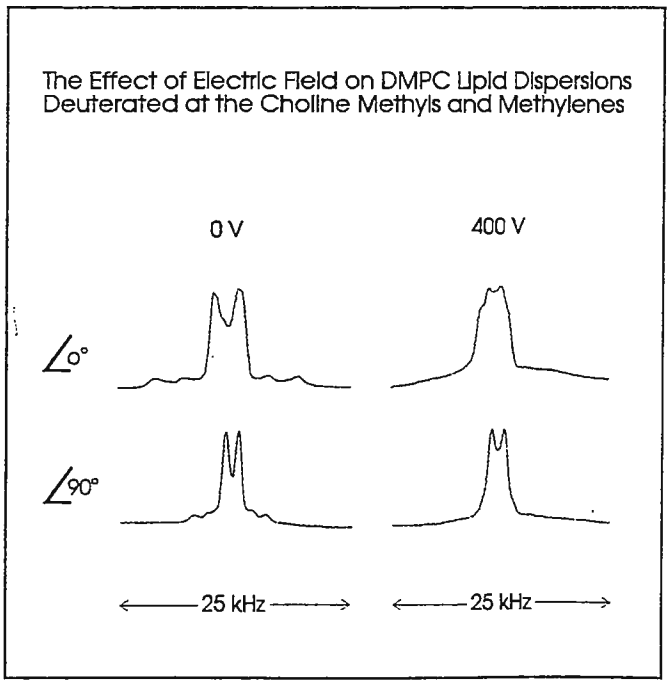

Figure 3. These spectra were acquired before and after application of a biphasically pulsed electrical potential across a sample of hydrated DMPC. The applied potential was 400V with a pulse duration of 200ms. The ^2H NMR spectra were typically acquired using a solid echo sequence with a delay of 20μs between pulses, a sweep width of 125 kHz, a 90° pulse width of 6 μs, and a repetition rate of 0.5s with a line broadening of 100 Hz.

DEPARTMENT OF MEDICAL GENETICS
GRADUATE DEPARTMENT OF MOLECULAR & MEDICAL GENETICS

University of Toronto

April 25, 1996

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

WWW Electronic Poster Session Announcement: EPS-2
June 17-21, 1996 • http://bellatrix.pcl.ox.ac.uk/nmr96/Welcome.html

Dear Barry:

We'd like to announce the The Second Electronic NMR Poster Session (EPS-2), scheduled to run this year from June 17-21. This session follows on our first effort last summer, which won a "Chemistry On The Internet: Best of the Web 1995" award (http://www.ch.ic.ac.uk/infobahn/boc.html) and featured 43 posters from all areas of NMR spectroscopy. Most of last year's session is still available on the net; stop by our site at http://bellatrix.pcl.ox.ac.uk/nmr/poster.html or any of the mirror sites indicated there for more details.

This year's session will offer more posters along with discussions of various topics through both mailing lists and real-time interactive forums. We will format Web pages that will link posters together at various sites around the world including Europe, North America and Japan. To participate in this session:

1. Subscribe to the mailing list associated with the conference; this will allow you to receive information as the conference approaches. Email majordomo@bellatrix.pcl.ox.ac.uk with a message:

subscribe nmrlist@bellatrix.pcl.ox.ac.uk your_email_address your_name

2. If you would like to display a poster as part of the session, please send a note to the organizers (nmrorg@bellatrix.pcl.ox.ac.uk) and we'll send you a brief form to fill out.

That's it! If you do not have the ability to mount a poster onto the WWW from your location or have any other questions, please email the organizers (nmrorg@bellatrix.pcl.ox.ac.uk or any of the individual addresses listed below). Thanks!

Kevin Gardner

Kevin Gardner, on behalf of the EPS-2 Organizing Group:
Bryan Finn (bryan@freja.fkem2.lth.se)
Kevin Gardner (gardner@bloch.med.utoronto.ca)
Barry Hardy (barry@bellatrix.pcl.ox.ac.uk)

Telephone: (416) 978-0642 Fax: (416) 978-6885 Faculty of Medicine Toronto Ontario M5S 1A8

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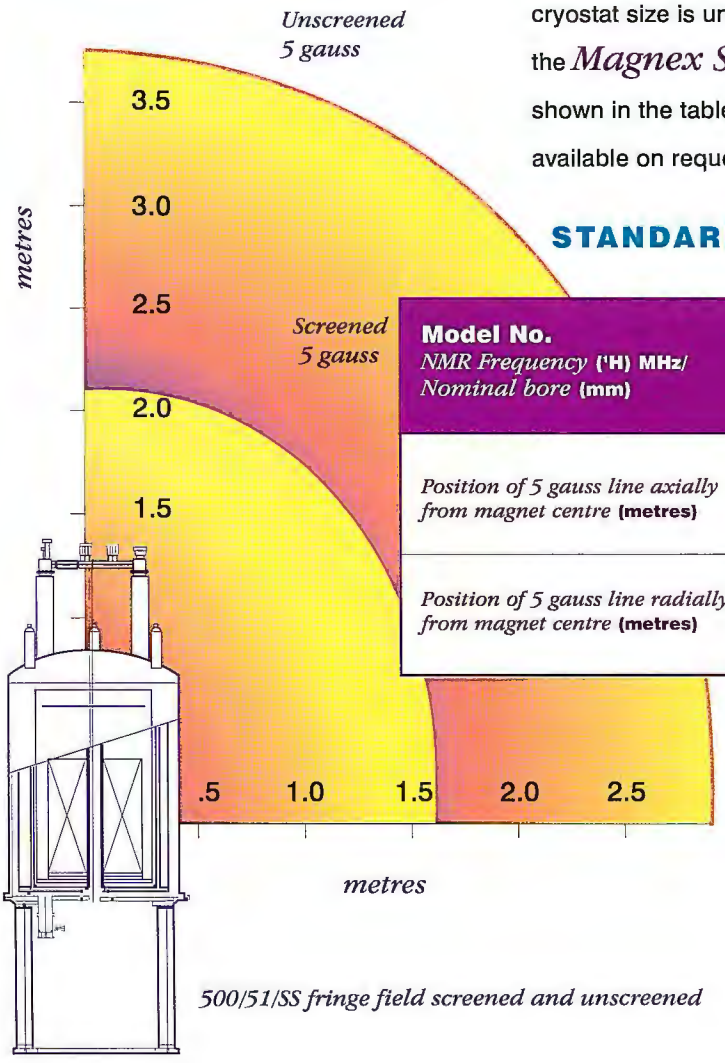
Model No. *NMR Frequency* (¹H) MHz/ *Nominal bore* (mm)	MRCA 400/54/SS	MRCA 400/89/SS	MRCA 500/51/SS	MRCA 500/89/SS	MRCA 600/51/SS
Position of 5 gauss line axially from magnet centre (metres)	2.0	2.1	2.1	3.1	3.0
Position of 5 gauss line radially from magnet centre (metres)	1.5	1.6	1.6	2.4	2.4



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SPECIFICATIONS FOR VERTICAL BORE MAGNET SYSTEMS

Model No. NMR Frequency ('H) MHz/Nominal bore (mm)	MRCA 200/89	MRCA 300/54	MRCA 300/89	MRCA 400/54	MRCA 400/62	MRCA 400/89	MRCA 400/104	MRCA 500/51	MRCA 500/62	MRCA 500/89	MRCA 500/104	MRCA 500/120	MRCA 550/89	MRCA 600/51	MRCA 600/89	MRCA 750/54	MRCA 750/62
Room temperature bore minimum access (mm)	88.8	53.8	88.8	53.8	62.1	88.8	104.4	51.2	62.1	88.8	104.4	120.0	88.8	51.2	88.8	53.8	62.1
Minimum helium hold time between refills (days)	160	135	160	160	190	170	190	150	125	96	100	100	100	120	90	75	63
Minimum nitrogen hold time between refills (days)	12	15	12	14	16	16	17	18	17	18	18	18	18	18	18	18	18
Minimum homogeneity volume : diameter (mm) x length (mm)	20x20	10x20	20x20	10x20	10x20	20x20	20x25	10x20	10x20	20x20	20x25	25x25	20x20	10x20	20x20	10x20	10x20
Homogeneity specification for given sample volume HHLW* with supercon shims	0.2	0.1	0.2	0.1	0.1	0.2	0.2	0.1	0.1	0.2	0.2	0.2	0.2	0.1	0.2	0.1	0.1
HHLW* with supercon and RT shims	0.02	0.01	0.02	0.01	0.01	0.02	0.02	0.01	0.01	0.02	0.02	0.02	0.02	0.01	0.02	0.01	0.01
HHLW* with supercon and RT shims/spinning	0.002	0.001	0.002	0.001	0.001	0.002	0.002	0.001	0.001	0.002	0.002	0.002	0.002	0.001	0.002	0.001	0.001
Spinning side-bands for carbon 13: less than (%)	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1
Field stability Better than (ppm/hour)	0.03	0.02	0.02	0.02	0.02	0.03	0.03	0.02	0.02	0.03	0.03	0.03	0.03	0.02	0.03	0.03	0.03

* Half Height Line Width

Notes

1. Extended bold-time cryostats are also available for all magnet types.

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21 Blacklands Way
Abingdon, Oxon OX14 1DY
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Tel: (44) 01235 534488
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Japan
Tel: (81) 078 991 9445
Fax: (81) 078 991 9446

DEPARTMENT OF HEALTH & HUMAN SERVICES

Public Health Service

National Institutes of Health
National Heart, Lung, and
Blood Institute
Bethesda, Maryland 20892

April 10, 1996

James A. Ferretti
Bldg. 3, Room 412
MSC-0380
NIH
Bethesda, MD 20892-0380

SOLUTION NMR STUDIES ON PROTEIN-DNA RECOGNITION

(received 4/15/96)

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

Dear Barry,

Sorry that you had to remind me about my contribution to the newsletter. I will try to be more prompt next time. For the past several years we have been interested in the basis of DNA recognition by homeodomain proteins. The investigations are a collaborative effort with Dr. Marshall Nirenberg of the Laboratory of Biochemical Genetics of the NHLBI here at NIH.

Homeodomains are the part of a protein that is encoded by the homeobox. Genes that specify positional information and segmental identity in development of the body plan have the homeobox in common. The homeobox contains approximately 180 base pairs so that the homeodomain is a protein segment of approximately 60 amino acid residues. An interesting feature of the homeodomain is that it appears to be conserved in evolution in species from yeast through humans. Functional studies suggest that the corresponding mechanism of gene regulation might be conserved throughout evolution and many homeodomain proteins have been shown to be important regulators of gene expression during development.

Our current interest includes the *vnd* (ventral nervous system defective) gene, where mutations lead to loss of function in early neurogenesis. Expression of the *vnd* gene initiates the neural pathway of development in part of the ventral nerve cord of the Drosophila embryo[1]. A mutation of the *vnd* gene that disrupted the transcriptional unit resulted in the loss of neuroblasts in the ventral nerve cord[2].

The homeodomain recognizes specific nucleotide sequences in DNA and in many cases is responsible for the DNA binding of the entire protein encoded by the gene. This specificity of the homeodomain-DNA interaction and the resulting high affinity binding must be important in transcription regulation and thus in the cascade of events associated with developmental control. Because of the size of the homeodomain (with flanking residues the molecular weight is around 9000), it is ideal for NMR studies. Also, the minimum size of the DNA is in the range of 14 -

16 base pairs (molecular weights around 10000). In fact, we recently have published two papers on the structure of the protein free in solution[3,4]. The coordinates are available by contacting me. An additional manuscript on the homeodomain-DNA binding was submitted recently to Structure.

The strength and specificity of homeodomain-DNA binding is linked closely to the three-dimensional structure of the homeodomain, since the secondary and tertiary structures adopted by the protein produce the amino acid sidechain orientations necessary for specific interactions. The vnd homeodomain binds to the unusual nucleotide segment AAGT, as the core consensus sequence. We have been able to describe the nature and relative importance of the various atom contacts involved in the vnd-DNA complex in the context of our three-dimensional structure (of the complex).

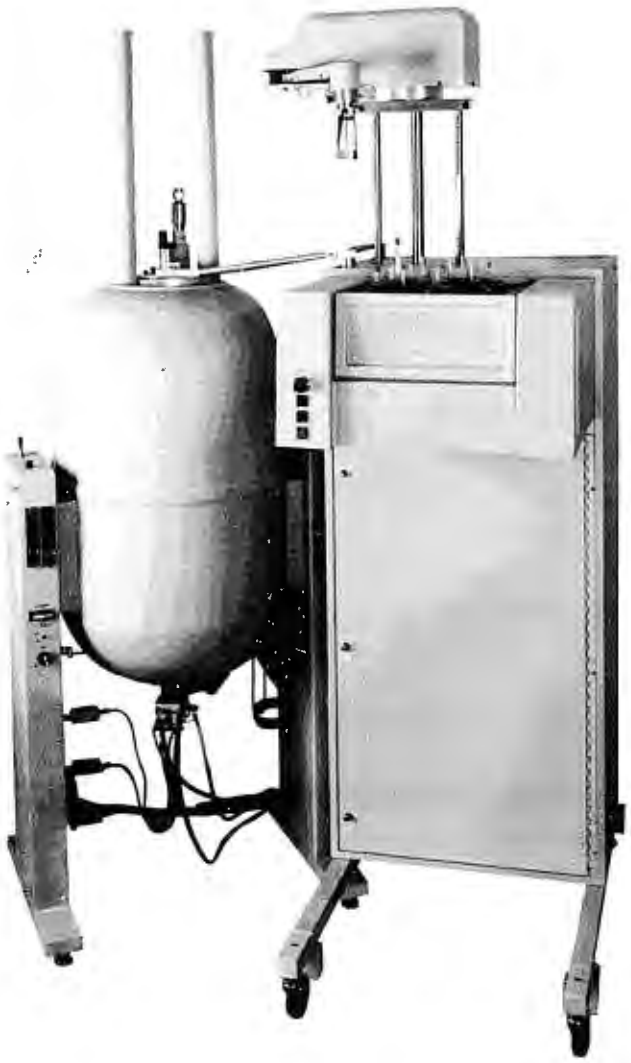
One of the outstanding questions in this field concerns the ability of the developing organism to turn on and off the appropriate gene at the appropriate time and position. An important part of this development cascade involves the homeodomain-DNA interaction. However, the degree of specificity required in development would appear to greatly exceed that amount of binding specificity afforded by this interaction. Some of our recent mutational studies show that we can alter binding affinities by one or two orders of magnitude at most in the selection of a given homeodomain for a particular sequence of DNA.

This question of binding specificity raises another difficult issue which is dear to my heart. Variations in binding specificity of one or two orders of magnitude (in terms of binding affinities) represents only a few Kcals that must be distributed over all of the atom contacts. It is questionable (from my point of view) whether NMR (or Xray for that matter) will ever become sufficiently sophisticated to be able to make such distinctions, even though our technique distinguishes individual atoms so exquisitely.

Best regards,

James A. Ferretti

1.Nirenberg & Lad (1995). Ann. N. Y. Acad. Sci. **758**, 224-242.
2.Jiminéz & White (1995). EMBO J. **14**, 3487-3495
3.Tsao, Gruschus, Wang, Nirenberg, & Ferretti (1994) Biochemistry **33**, 15053-15060.
4.Tsao, Gruschus, Wang, Nirenberg, & Ferretti (1995) J. Mol. Biol. **251**, 297-307.



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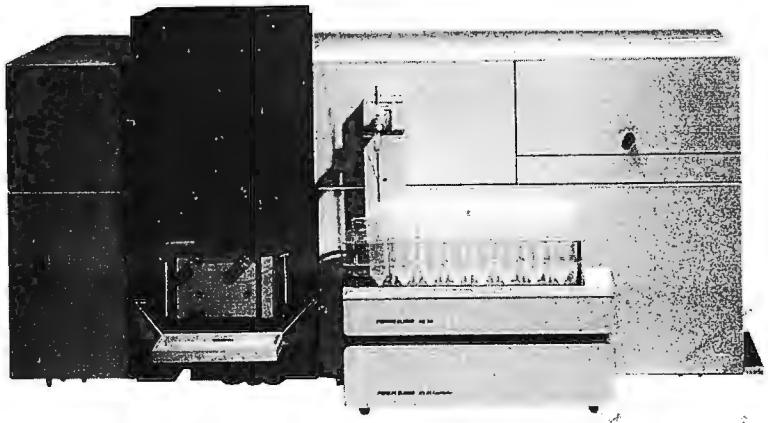

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XVIIth International Conference on Magnetic Resonance in Biological Systems
August 18-23, 1996 - Keystone, Colorado, USA

Conference Office
1201 Don Diego Avenue
Santa Fe, NM 87505, USA

Phone: (505) 989-4735
Fax: (505) 989-1073

The 17th in the series of biennial International Conferences on Magnetic Resonance in Biological Systems will be held at the Conference Center in Keystone, Colorado, August 18 - 23, 1996. This meeting has not been held in North America since 1988, and we anticipate a large and dynamic attendance, hence we encourage you to make your plans and register early.

A broadly based program has been planned, covering new magnetic resonance methods, applications of high resolution NMR to structural biology, application of solid state NMR techniques, use of EPR in studying a variety of paramagnetic systems, *in vivo* spectroscopy and imaging, and a minisymposium on Biomolecular NMR and Drug Discovery.

The conference will provide a unique opportunity for biological, medical, and technique oriented practitioners of magnetic resonance to discuss common goals and problems. To this end, aspects of magnetic resonance are combined and focused on specific topics in the program, resulting in a balance of NMR, EPR, and *in vivo* applications presented jointly throughout the program.

The conference will feature 12 plenary lectures, along with about 100 invited lectures and 25 short presentations in three parallel sessions. Three poster sessions, each accommodating up to 200 posters, will permit participation by a large number of workers active in many aspects of biological magnetic resonance. You are invited to submit an abstract of your work on the form in this announcement.

In recognition of the breadth of this community, we encourage you to share this announcement with your colleagues and inform them of the various vehicles to obtain further information on the conference as detailed within this announcement

PRELIMINARY PROGRAM

Additional speakers will be added and the order of presentations has not been finalized.

SUNDAY	MONDAY	TUESDAY
	Plenary Session **8:30 - 10:00 AM** *Pines* *Thomas*	***Plenary Session*** **8:30 - 10:00 AM** *Cates* *McDermott*
	Plenary Session **10:30 AM - 12:30 PM** *Weisskopf* *Wuthrich*	**10:30 AM - 12:30 PM** *Session A* **Protein Structure III** *Campbell* *Wagner* *Prestegard* *Sykes* *Session B* **MR Microscopy** *Haase* *Neeman* *Session C* **New Methods II** *Meier* *Freed* *Gullion* *Fujiwara*
	2:00 - 4:00 PM ***Poster Session I***	**2:00 - 4:00 PM** ***Poster Session II***
4:00 PM ***Welcome to ICMRBS*** ***Registration*** Keystone Conference Center	**4:00 - 5:15 PM** *Session A* **Protein Structures I** *Laue* *Poulsen* *Chazin* *Session B* **Gradients & Coils** *Brey* *Cory* *Session C* **Enzymes & Catalysis** *McDowell* *Graslund* *Bowman*	**4:00 - 5:15 PM** *Session A* **Nucleic Acids I** *Pardi* *Reid* *Kainosho* *Session B* **Dynamics II** *Jardetzky* *Drobny* *Palmer* *Session C* **Proteins & Membranes** *Smith* *Cross* *Eaton*
	5:30-7:00 PM *Session A* **Extremes in EPR** *Yannoni* *Brunel* *Session B* **New Methods I** *Montelione* *Mueller* *Kupce* *Session C* **Dynamics I** *Peng* *Robinson* *Zuiderweg* *Marion*	**5:30 - 7:00 PM** *Session A* **Cell Metabolism I** *Gilles* *Glickson* *Degani* *Session B* **New Methods III** *Kay* *Griesinger* *Sessiom C* **Proteins & Membranes II** *Tycko* *Gawrish* *Marsh*
6-8 PM ***Welcome Mixer***	**7:00 PM - midnight** ***Corporate Hospitalities***	**7:00 PM - midnight** ***Corporate Hospitalities***

Consult the conference web page for program updates: http://nmrsgi1.ncifcrf.gov/icmrbsxvii

The web page will be updated periodically with the complete program including titles of all talks and posters. The program of invited speakers should be complete by April 1, 1996, and the program of posters will be listed by July 1. The program posted on the Web page will replace the mailing of a preliminary program. Also, authors of talks and posters will be invited to post their abstracts on the web. Instructions will be included with acceptance notices. Information on Keystone and the surrounding area is also available via the web.

PRELIMINARY PROGRAM

Please consult the conference web page for updates: http://nmrsgi1.ncifcrf.gov/icmrbsxvii

WEDNESDAY

Plenary Session
8:30 - 10:00 AM
Lubitz
Varani

10:30 AM - 12:30 PM

Session A	*Session B*	*Session C*
Nucleic Acids II	**Cell Metabolism II**	**Protein Folding**
James	*Shulman*	*Dobson*
Patel	*Radda*	*Serrano*
Williamson	*Ugurbil*	*Christofferson*
Feigon	*Lewandowski*	*Imperiali*
Tinoco	*Kalyanaraman*	*Rico*

Afternoon Free

Afternoon Free

Free Time

7 PM - midnight
Corporate Hospitalities

THURSDAY

Plenary Session
8:30 - 10:00 AM
Fesik
Inagaki

10:30 AM - 12:30 PM

Session A	*Session B*	*Session C*
Biomolecular NMR & Drug Discovery Symposium	**Paramag. Proteins**	**Diffusion & Perfusion**
Falke	*Markley*	*LeBihan*
Grzesiek	*Bertini*	*Moseley*
Summers	*Hoffman*	*Moonen*
Wittekind		*Gore*

2:00 - 4:00 PM
Poster Session III

4:00 - 5:15 PM

Session A	*Session B*	*Session C*
Symposium Continued	**Electron Transfer**	**Inorganic Ions**
Kessler	*Britt*	*Navon*
Stockman	*Huber*	*Ackerman*
O'Connell	*Antholine*	*Sherry*

5:30-7:00 PM

Session A	*Session B*	*Session C*
Symposium Continued	**Metals**	**Chemical Shifts**
Cowburn	*Vogel*	*Oldfield*
Fry	*Gerothanassis*	*Case*
Ni	*Ikura*	*Williamson*

7:30 - 9:30 PM
Western Barbeque

FRIDAY

Plenary Session
8:30 - 10:00 AM
Clore
Thornton

10:30 AM - 12:30 PM

Session A	*Session B*	*Session C*
Protein Nucleic Acids	**Proteins & Membranes III**	**Water Macro-molecules**
Wright	*Opella*	*Springer*
Kaptein	*Griffin*	*Bryant*
Arrowsmith	*Fillingame*	*Jeener*
Nishimura		

Conference Adjourned

GENERAL INFORMATION

CONFERENCE REGISTRATION. The deadline is June 21. Contact the ICMRBS office for details.

SCHEDULE of SPECIAL EVENTS.

Sunday, 4 pm **Registration**

6-8 pm **Welcome reception.** Informal refreshments and beverages will be served on the outdoor patio overlooking the Lake. The reception is open to all registrants and registered companions.

Wednesday afternoon Free time. There are many recreational opportunities at Keystone which you may pursue on your own or choose from the following planned activities:

Guided Trail Rides of two hours through the Arapaho National Forest. Children must be 8 years old and have riding experience. Departure times will be 2 pm or 4:30 pm. *Cost: $29 per person.*

18-Hole Golf Scramble at the Robert Trent Jones Jr. designed course at Keystone. This high altitude course features spectacular mountain vistas. It is rated among the top 50 resort courses in the country by *Golf Digest*. Tee off will be at 2 pm. *Cost: $91 per person, includes green fees and golf cart.*

White Water Rafting Excursion will provide an enchanting three hours on the Colorado River. Experienced guides will relate the lore of these beautiful canyons. This excursion is ideal for beginners and families. Buses will depart at 1:30 pm. A box lunch will be provided on the bus. You will be returned to Keystone at 7 pm. *Cost: $70 includes box lunch and transportation.*

Thursday, 7:30-9:30 pm **Western BBQ and Barn Dance** will feature assorted salads and relishes, barbecued chicken and ribs, corn on the cob, baked beans, iced watermelon and fruit pies. Wear your blue jeans and plan to make the mountains rock! *Cost: $25 per person.*

COMPANION REGISTRATION. Companion registration is offered for adults accompanying conference registrants and includes a designated name badge and the conference opening reception. A lounge will be provided 9-11 am, Monday - Wednesday to meet and greet other accompanying persons. Companions and children are welcome to register for the planned activities offered on Wednesday afternoon above.

Cost: $25 for companion registration.

ADDITIONAL INFORMATION. Contact ICMRBS, 1201 Don Diego Avenue, Santa Fe, NM 87505.

Telephone: (505) 989-4735. Fax: (505) 989-1073. E-mail: 70404.2407@compuserve.com.

ICMRBS web page address: **http://nmrsgi1.ncifcrf.gov/icmrbsxvii**

OTHER USEFUL NUMBERS.

Lodging Reservations **(800) 258-0437 or (970) 468-4242 (press 2 at the auto menu). Fax: (970) 468-4343**

Keystone Information **(800) 222-0188 or (970) 468-4242** and make selections from the automated menu

Airport Shuttle Bus **(800) 334-7433 or (970) 468-7600** to make a reservation for the shuttle bus between Denver International Airport and Keystone. **Fax: (970) 468-4142**

High Country Travel **(800) 367-1654 or (970) 468-1080 Fax: (970) 468-8030**

Keystone web page **http://www.csn.net/resorts/keyst.html**

CONFERENCE CALENDAR.

May 1 Deadline for young scientist travel stipend applications and abstracts

June 1 Abstract Deadline

June 21 Registration Deadline

June 21 Deadline for Keystone Room Reservations

July 18 Deadline for Cancellation of Room Reservation. Contact Keystone directly.

Address all Newsletter correspondence to:

Dr. B. L. Shapiro
The NMR Newsletter
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. 453 (June)	24 May 1996
No. 454 (July)	28 June 1996
No. 455 (August)	26 July 1996
No. 456 (Sept.)	23 Aug. 1996
No. 457(Oct.)	27 Sept. 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.]

http://www.nmrnewsletter.com

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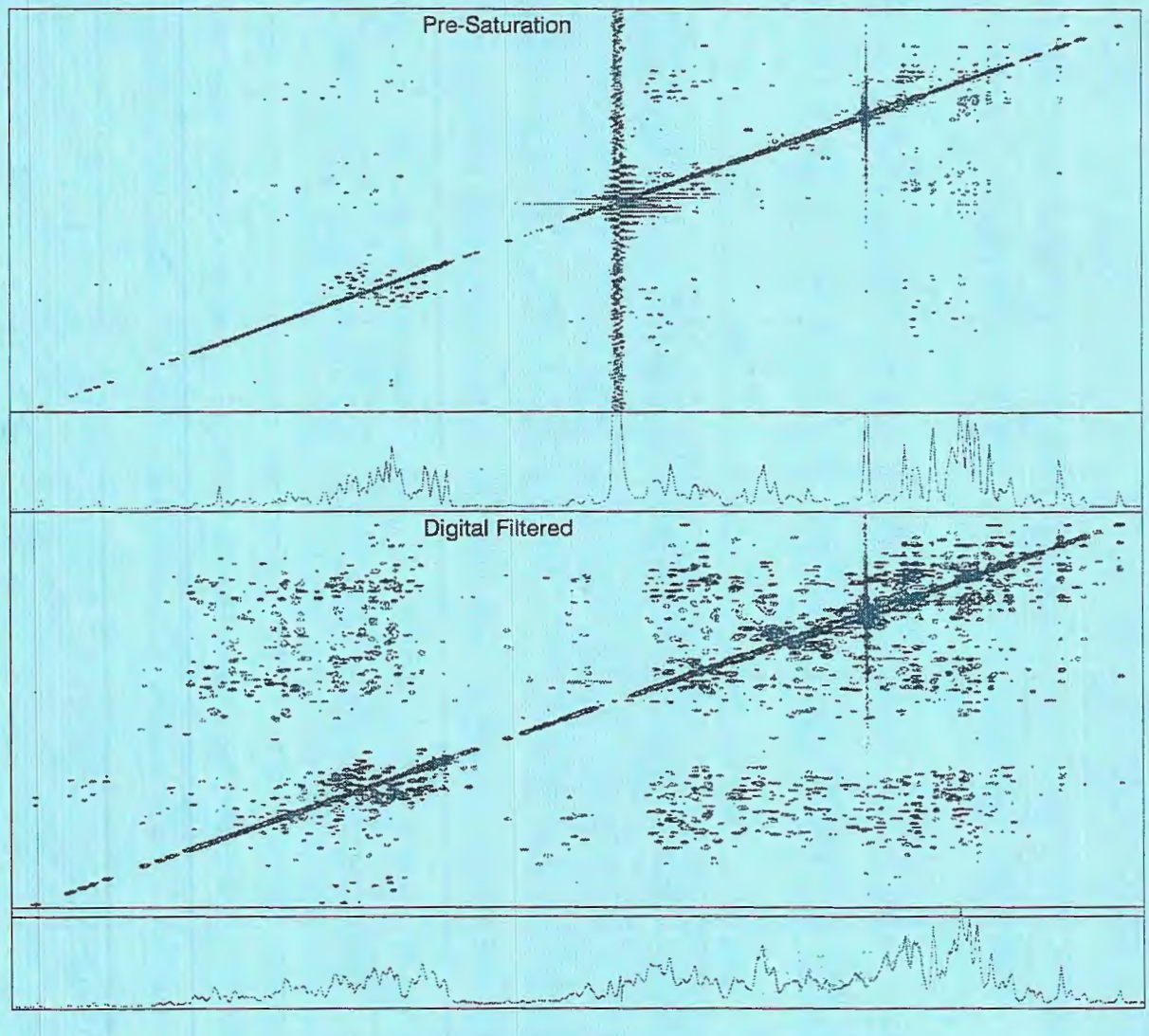
ECLIPSE NMR Advantage: Digital Filtering



Eclipse NMR *The Better Way!*

This data shows the digital filtering capability of JEOL USA's ECLIPSE NMR workstation. Eclipse does digital filtering via software after the data is acquired, not via hardware during acquisition. This offers a significant advantage because with software digital filtering the acquisition is completed before you filter the data.

JEOL feels spectrometer time is best spent acquiring new data rather than repeating experiments because conditions were not optimized. It takes more time to write this kind of software, but JEOL took the time. Now you can use the ECLIPSE NMR Advantage to your advantage.

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