THE NMR NEWSLETTER

No. 458

November 1996

NUTS

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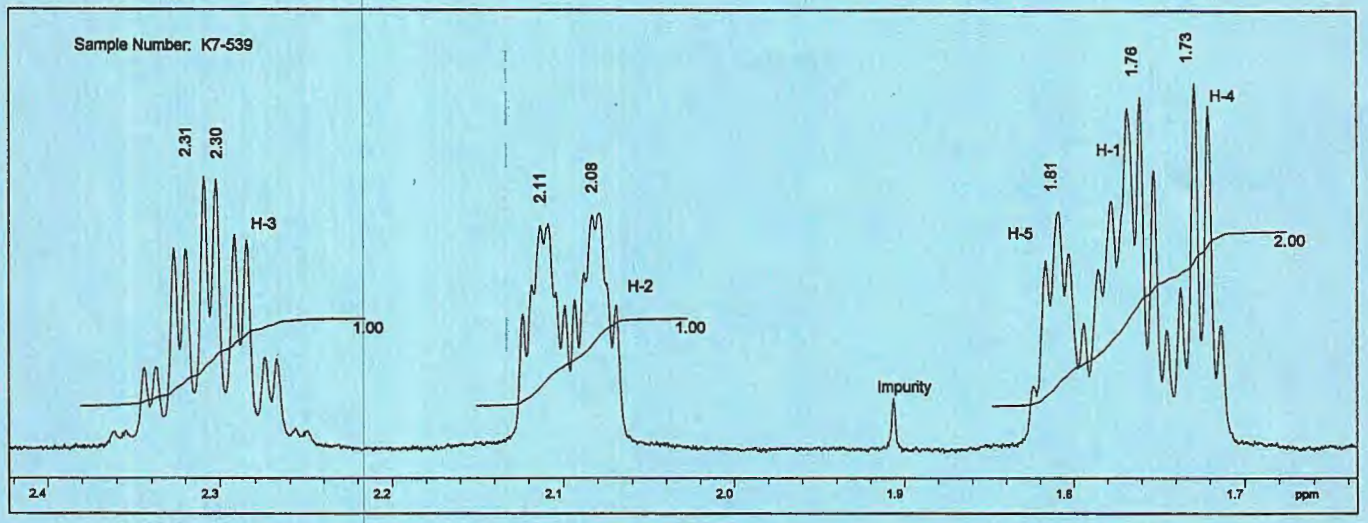

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FORTHCOMING NMR MEETINGS

38th ENC (Experimental NMR Conference), Orlando, FL, **March 23 - 27, 1997**; Contact: ENC, 1201 Don Diego Avenue, Santa Fe, NM 87505; (505) 989-4573; Fax: (505) 989-1073.

International Society for Magnetic Resonance in Medicine, Fifth Scientific Meeting and Exhibition, Vancouver, BC, Canada, **April 12-18, 1997**; Contact: ISMRM, 2118 Milvia St., Suite 201, Berkeley, CA 94704, USA; (510) 841-1899; Fax (510) 841-2340; Email: info@ismrm.org.

Symposium on NMR Spectroscopy of Synthetic Macromolcules, ACS National Meeting, San Francisco, **April 13-17,** 1997; Contact: H. N. Cheng or English, A. D. See Newsletter 456, 20.

39th Rocky Mountain Conference on Analytical Chemistry, Denver, Colorado; NMR Symposium, **August 4-7, 1977**: Contact: J. P. Yesinowski, Code 6120, Naval Research Laboratory, Washington, DC 20375-5342; 202-767-0415; fax 202-767-0594; email yesinowski@nrl.navy.mil. See Newsletter 458, 8.

4th International Conference on Magnetic Resonance Microscopy "Heidelberg Conference in Albuquerque", **Sept. 21-25, 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.

Additional listings of meetings, etc., are invited.

Duke University
Duke Nuclear Magnetic Resonance Spectroscopy Center

Leonard D. Spicer, Director
Anthony A. Ribeiro, Manager

919 684 4327
919 613 8887

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

October 22, 1996
(received 10/25/96)

Re: 19F, 13C HMQC and HMBC of Perfluoroheptanoic Acid [CF_3-$(CF_2)_5$-COOH]

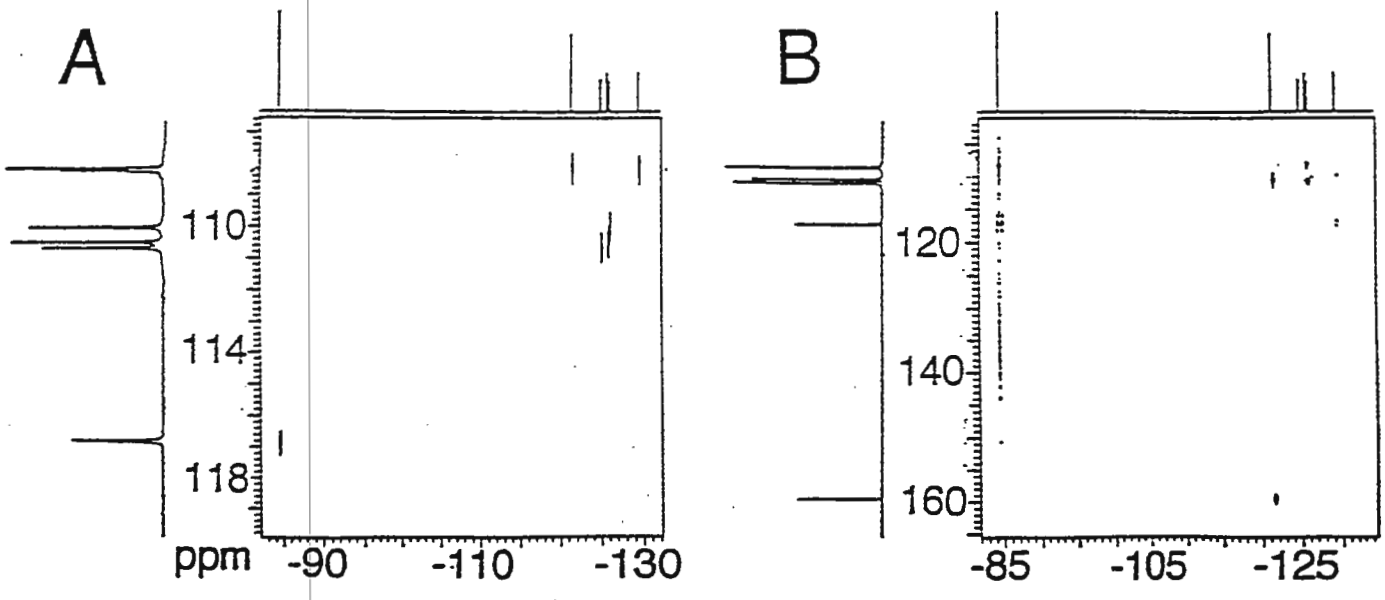
Dear Barry,

We have recently implemented a heteronuclear strategy for the analyses of perfluorinated chains whose 19F NMR spectra (top of 2D maps) usually exhibit complex multiplets with varying lineshapes due to extensive homonuclear scalar couplings. In perfluoroheptanoic acid, two resonances (- 84.5 ppm CF3; -121.6 ppm CF2) are triplet-of triplets with resolved couplings, two (-125.9 ppm and -129.6 ppm CF2) are complex multiplets with fine structure splittings, and two (-125.1 and -126.1 ppm CF2) have NMR transitions that are so closely spaced that they appear as broad, poorly resolved resonances with effective linewidths approaching 50 Hz.
The 19F, 13C single and two-bond correlation maps presented for perfluoroheptanoic acid were recorded by adapting the 1H-detected 2D HMQC and HMBC methods for 19F detection. The HMQC map with 13C decoupling gave AX-type cross peaks which correlate the 19F nuclei to the chemical shift positions of the directly bonded carbons without having to perform 13C NMR with its special challenges of wide band fluorine decoupling for fluorinated chains. The HMBC map without 13C decoupling showed multiple two-bond correlations and allowed the derivation of a complete set of 19F and 13C NMR assignments. The -121.6 ppm CF2 signal (C2), for example, yields strong two-bond connectivities to the carboxyl carbon at 159 ppm (C1) and to the 110 ppm 13C signal (C3).
We have also looked at the wide band fluorine decoupling problem. In the absence of fluorine decoupling, the 13C resonaces from the six fluorinated carbons of perfluoroheptanoic acid are split by multi-bond couplings and give rise to > 47 resolved lines in the narrow region between 106 and 121 ppm. The side plots show the spectral simplification to singlets for the carbons of perfluoroheptanoic acid achieved with simultaneous 19F-decoupling of the CF3 and CF2 regions.

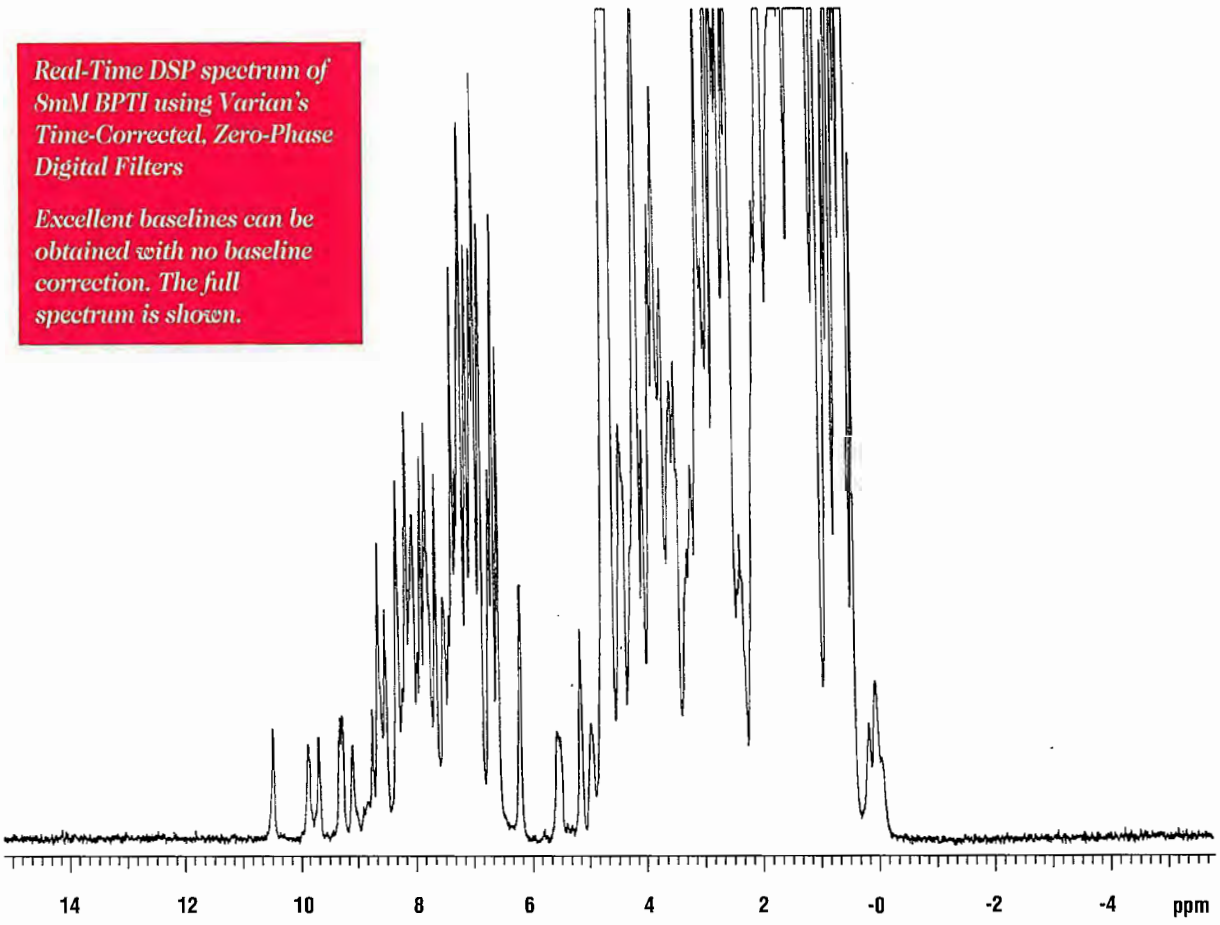
Regards,

Tony

Anthony A. Ribeiro (A^2R)



The Last Word in DSP From the First Name in NMR



Real-Time DSP spectrum of 5mM BPTI using Varian's Time-Corrected, Zero-Phase Digital Filters

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When dynamic range becomes a problem and you need to reduce the gain on your spectrometer, signal-to-noise can degrade substantially. Varian's Real-time DSP, with its high sampling rate and 20-bit precision, lets you obtain full S/N at lower gains and optimum S/N at any gain.

At Varian, we sell performance, not buzzwords. To arrange a demonstration of Varian's superior DSP capabilities, contact the Varian office nearest you.

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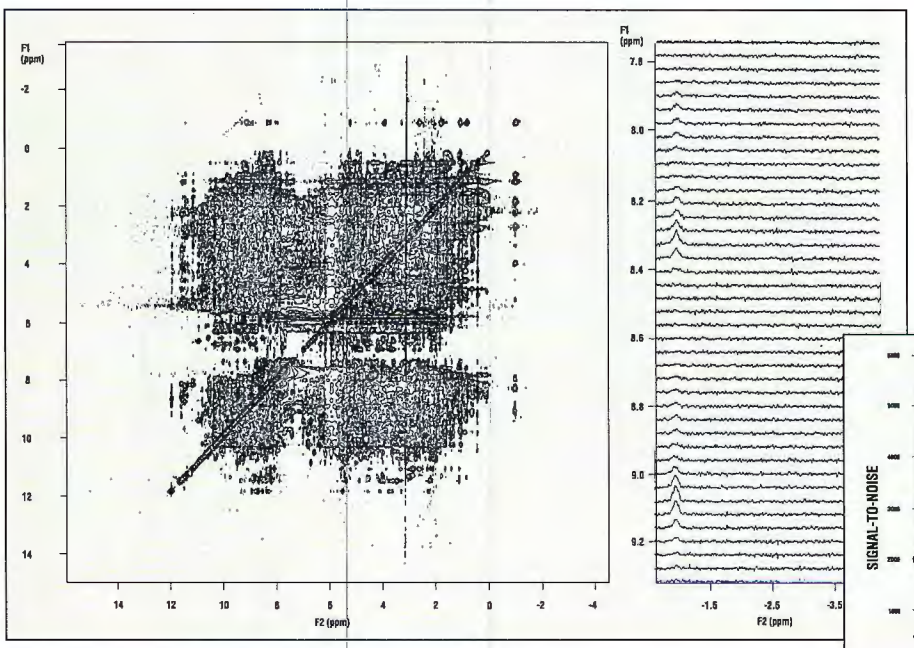
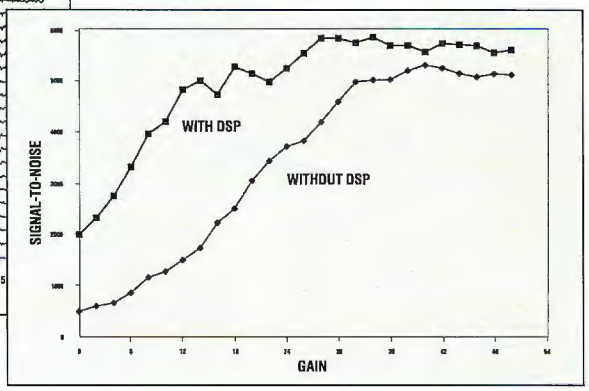
DSP from Varian–Designed for Superior Performance



2D NOESY of 10mM lysozyme using DSP with absolutely no baseline correction or linear prediction. The expanded stacked plot on the right shows some of the weakest cross-peaks with no hint of "wandering baseline".

Plot of S/N vs receiver gain with and without DSP, showing the large improvements possible with Varian's DSP.



Feature	Benefit
• Real-time digital filters (available on UNITY*INOVA*™ and UNITY*plus*™)	• Minimize data storage requirements in acquisition and host computers
• Time-Corrected Zero-Phase Digital Filters™	• Superior baseline performance
• Choice of real-time digital filters	• Optimize performance based on application
• AnalogPlus™ filter	• Improves S/N up to 10% without sacrificing baseline performance
• Brickwall filter	• Quantitative accuracy across the entire spectrum
• 400 kHz sampling rate (UNITY*INOVA* only)	• Greater oversampling brings greater S/N gains
• 20-bit precision	• Obtain the full benefit of oversampling
• No "build-up curve" at front of FID	• FIDs can be processed normally using any software
• Inline digital filters, processing data after acquisition but before storage on host computer (available on all Sun-based Varian spectrometers including UNITY*INOVA* and GEMINI 2000)	• Allows full flexibility of digital filtering without the time constraints of real-time filtering; minimizes data storage requirements on host computer
• Post-acquisition digital filtering (available on all Sun-based Varian spectrometers including UNITY*INOVA* and GEMINI 2000)	• Allows repeated digital filtering on the same data to allow optimization of parameters
• User-programmable coefficients	• Any desired digital filter can be used
• Frequency shifting	• Provides bandpass as well as lowpass filters
• Low receiver noise floor	• Allows DSP to reduce digitization noise and increase S/N

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Sept. 26, 1996 (received 9/30/96)

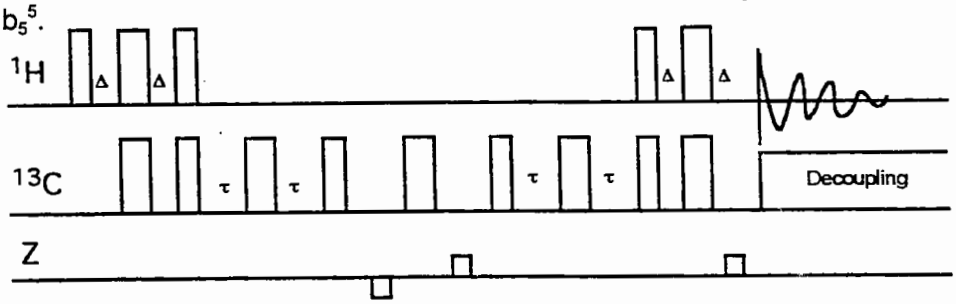
Dr. Bernard Shapiro
The NMR Newsletter
966 Elsinore Court
Palo Alto, CA 94303

A New ^{1}H-^{13}C-^{13}C Filtered ^{1}H NMR Experiment for Making Resonance Assignments in the Active Site of Heme Proteins

Dear Barry,

It is often difficult to assign resonances of heme substituents that do not have large isotropic shifts, and therefore appear under the large envelope of polypeptide resonances. Furthermore, resonances arising from heme in its reduced state are completely buried under the polypeptide envelope of resonances. We circumvented this problem by selective ^{13}C enrichment of the heme and subsequent observation with ^{13}C NMR spectroscopy[1]. Due to the low natural abundance of ^{13}C, if only the heme carbons in the protein are enriched, the resonances arising from the labeled prosthetic group can be observed with minimum interference from resonances that arise from the polypeptide.

Recently, we were interested in assigning the heme propionate carbonyl carbons in cytochrome b_5 with the aim of studying the role played by these groups in the complex formed between mitochondrial cytochromes b_5 and c. To this end, cytochrome b_5 whose heme was labeled at the positions indicated by • in Figure 1 was overexpressed in *E. Coli*, thus producing the ^{13}C-labeled fragment $\underline{C}H_2\underline{C}OO^-$. An experiment was devised for the selective ^{1}H detection of ^{1}H-^{13}C-^{13}C fragments from a large number of overlapping resonances[2]. The experiment is based on proton detected INEPT and ^{13}C double quantum coherence[3,4] (pulse sequence shown below), and takes advantage of the relatively large values of $^{1}J_{CH}$ and $^{1}J_{CC}$ (compared with $^{2}J_{CH}$), which require relatively short interpulse delays. This advantage is a requirement, due to the short T_2 values imparted on the heme resonances by delocalization of the unpaired electron. The experiment allowed us to obtain unambiguous assignments of the β-propionate methylene protons (Fig.1). These assignments proved extremely valuable in the subsequent assignment of resonances arising from the heme propionate carbonyl carbons in mitochondrial cytochrome b_5[5].



The pulse sequence was initially tested with a solution consisting of 1M sucrose and 5 mM 1,2-^{13}C sodium acetate. The efficient suppression of resonances arising from glucose and the selective detection of the methyl group in the doubly labeled sodium acetate are evident from Figure 2. Application of the experiment to ^{13}C-labeled-heme cytochrome b_5 described

above results in the spectrum shown in Figure 3. The peaks at -3.15 and 1.55 ppm arise from the diastereotopic heme propionate-7β hydrogens and the broad peaks near zero arise from heme propionate-6β hydrogens. Each set of peaks is a doublet due to the heme isomerism displayed by cytochrome b_5. Spectra were acquired on Varian Unity*plus* 600 spectrometer at The College of Staten Island.

Sincerely yours,

Feng Qiu[a,b] Ruth E. Stark[b] Mario Rivera[a]

[a] Department of Chemistry, Oklahoma State University

[b] Department of Chemistry, City University of New York, The College of Staten Island

Figure 1

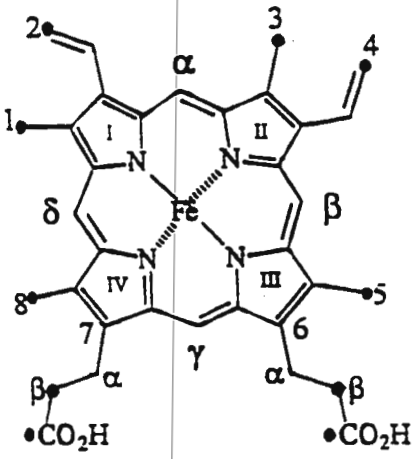
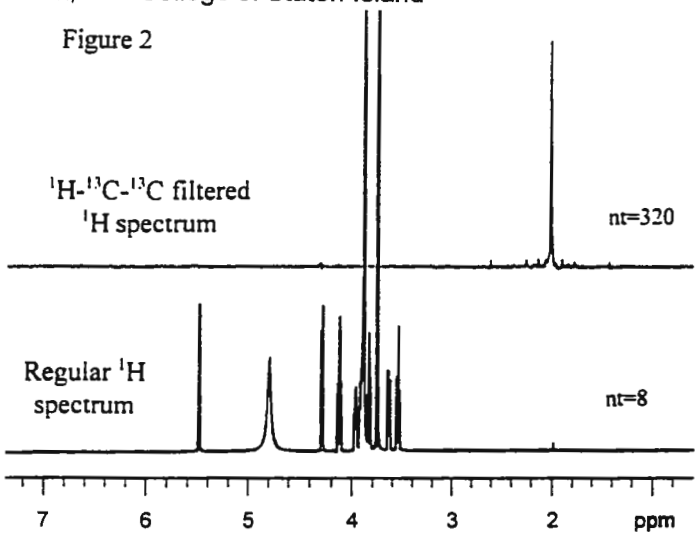
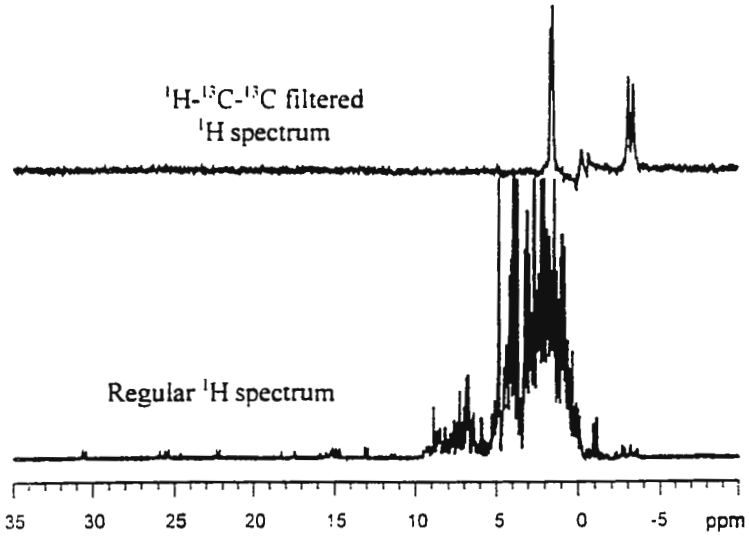


Figure 2



1 M sucrose and 5 mM ^{13}C 93% doubly labeled sodium acetate

Figure 3



Cytochrome-b_5 with $\underline{C}H_3$. $\underline{C}H_2$=CH. and $\underline{C}H_2\underline{C}OO^-$
^{13}C labeled fragments in its heme

References:

1. M. Rivera and F.A. Walker, *Anal. Biochem.*, 230, 295 (1995)
2. F. Qiu, M. Rivera and R.E. Stark, manuscript in preparation.
3. G.A. Morris and R. Freeman, *J. Am. Chem. Soc.*, 101, 760 (1979)
4. J. Weigelt and G. Otting, *J. Magn. Reson.* A, 113, 128 (1995)
5. M.J. Rodríguez-Maranón, F. Qiu, R.E. Stark, S.P. White, X. Zhang, S.I. Foundling, V. Rodríguez, C.L. Schilling, R.A. Bunce and M. Rivera, submitted to *Biochemistry*.

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UCD NMR FACILITY DAVIS, CALIFORNIA 95616

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

10/1/96
(received 10/3/96)

Lossy PC Boards

Dear Barry,

In view of a recent experience in our facility, I thought a reminder to old hands and lesson to NMR neophytes is in order.

Our users frequently design and build their own probes for in vivo spectroscopy and imaging on small animals. Part of our standard design is mounting the capacitors, which comprise the tank circuit(s) for single and double tuned probes, on a printed circuit board. It is a very nice way to mount the capacitors -- especially at high frequencies where lead inductances can be large. The coil is typically a single turn surface coil which is hung off the edge of the circuit board.

One of our users recently had trouble with such a circuit in that he could not get the Q of the circuit above 50. This was double sided circuit board and, in an effort to provide a nice ground plane, the ground of the component side was tied to the other side via copper tape wrapped around the edges. This, in effect, created a small (but physically large) capacitor with the board as the dielectric material. When we reproduced the circuit on Plexiglas with copper tape, we got a Q of 300! The circuit board material was a very lossy dielectric material at 300 MHz. We then went back to the circuit board, ground off the back plane and got a Q of 150. The moral of the story is that the choice of materials counts -- even on single sided circuit board. There is still stray capacitance to ground and the oscillating fields from the circuit will extend into the substrate material. If the material is lossy, it will affect your Q. This, in turn, will affect your pulse lengths and your signal to noise ratio. Dick Rosanke at Florida State has given me the name of Rogers Corporation (http://www.rogers-corp.com/index.html) as a source of high quality circuit board material.

I hope this reminder will keep others from wasting as much time as our user did. Please credit this contribution to Gerd La Mar's account.

Sincerely,

Jeffrey H. Walton

458-8

DEPARTMENT OF THE NAVY
NAVAL RESEARCH LABORATORY
4555 0VERLOOK AVE SW
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IN REPLY REFER TO:
6120:293;JPY/jpy
9 October 1996

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

NMR Symposium at the 39th Rocky Mountain Conference on Analytical Chemistry

Dear Barry:

The NMR Symposium at the 39th Rocky Mountain Conference on Analytical Chemistry will take place Monday to Thursday, August 4-7, 1997 at the Hyatt Regency Denver in downtown Denver, Colorado. The program will consist of invited lectures and contributed papers for oral and poster presentations.

Each year, we dedicate a half-day session of the NMR Symposium to the memory of Professor Robert Vaughan. We are particularly honored to have as the Vaughan Lecturer for 1997 Professor Charles P. Slichter of the Department of Physics, University of Illinois at Urbana-Champaign.

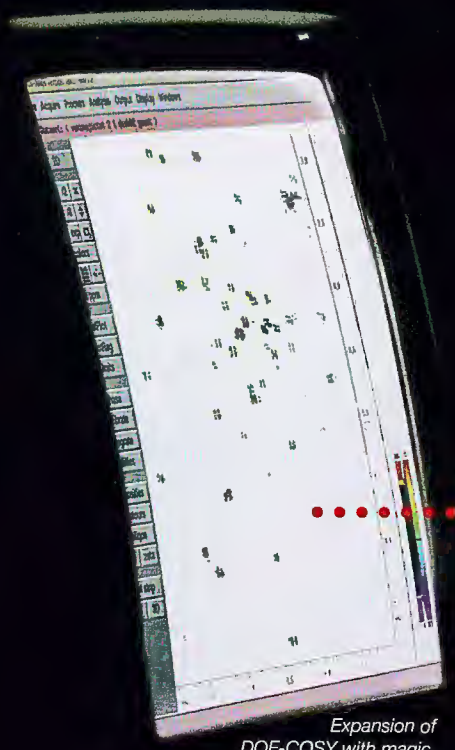
The NMR Symposium is being organized by James Yesinowski - chair, Robert Wind - co-chair, Lucio Frydman, Clare Grey, John Hanna, Jeffrey Reimer, and Steve Sinton. The sessions will include the areas of macromolecules (including bio-), inorganic materials including glasses, multi-dimensional NMR, imaging/inspection/diffusion, and new techniques and applications. Further information about the program, including the abstracts of talks and posters, will appear as available at our Web site: http://www.cchem.berkeley.edu/~jargrp/rmc.html.

Those who have attended previous meetings should be on the mailing list for the Conference, and will receive abstract forms that are due in March of 1997 (before the ENC). To be added to the mailing list, or for further information, I can be contacted at: Code 6120, Naval Research Laboratory, Washington DC 20375-5342. Phone: (202) 767-0415; FAX: (202) 767-0594; Internet: yesinowski@nrl.navy.mil.

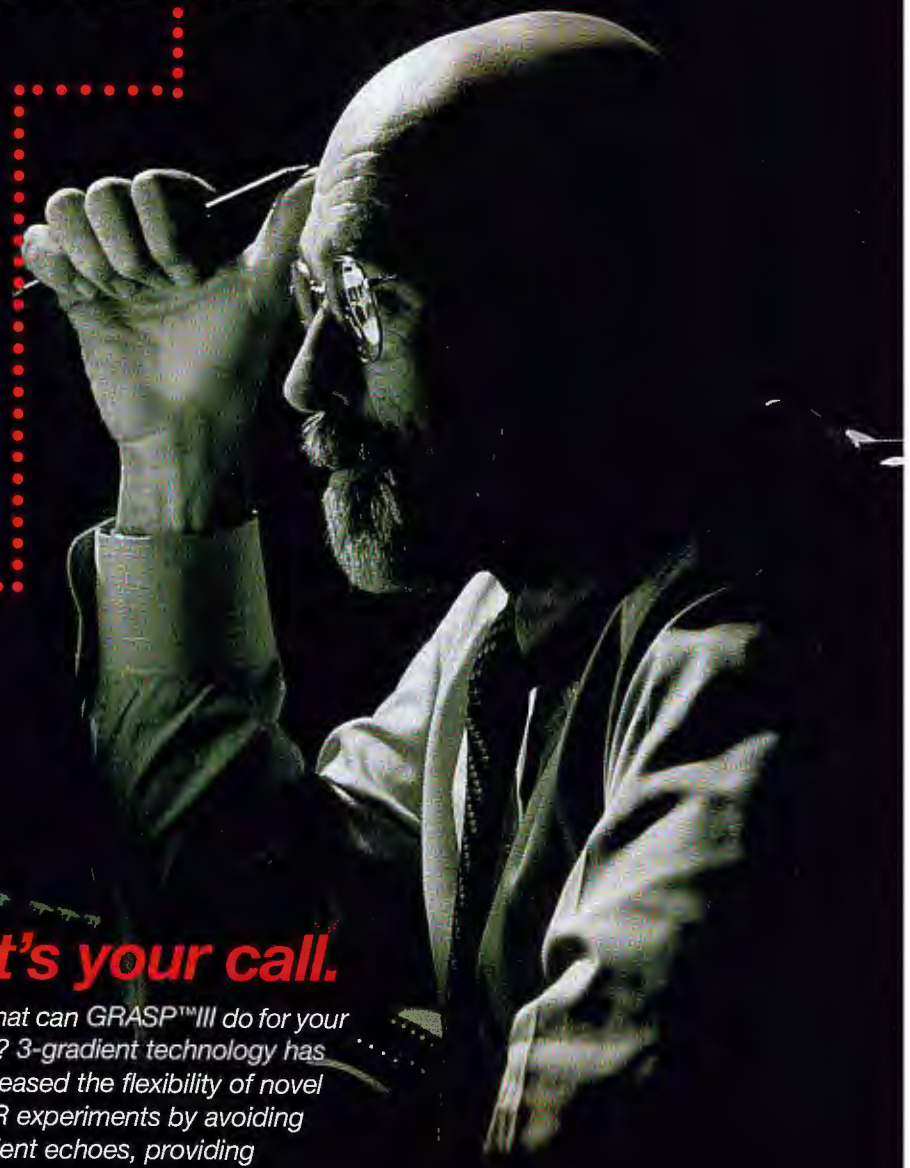
Sincerely yours,

James P. Yesinowski

Take a hard look at the difference.



Expansion of DQF-COSY with magic angle gradient, 1.5 mM BPTI in 90% H_2O/10% D_2O.



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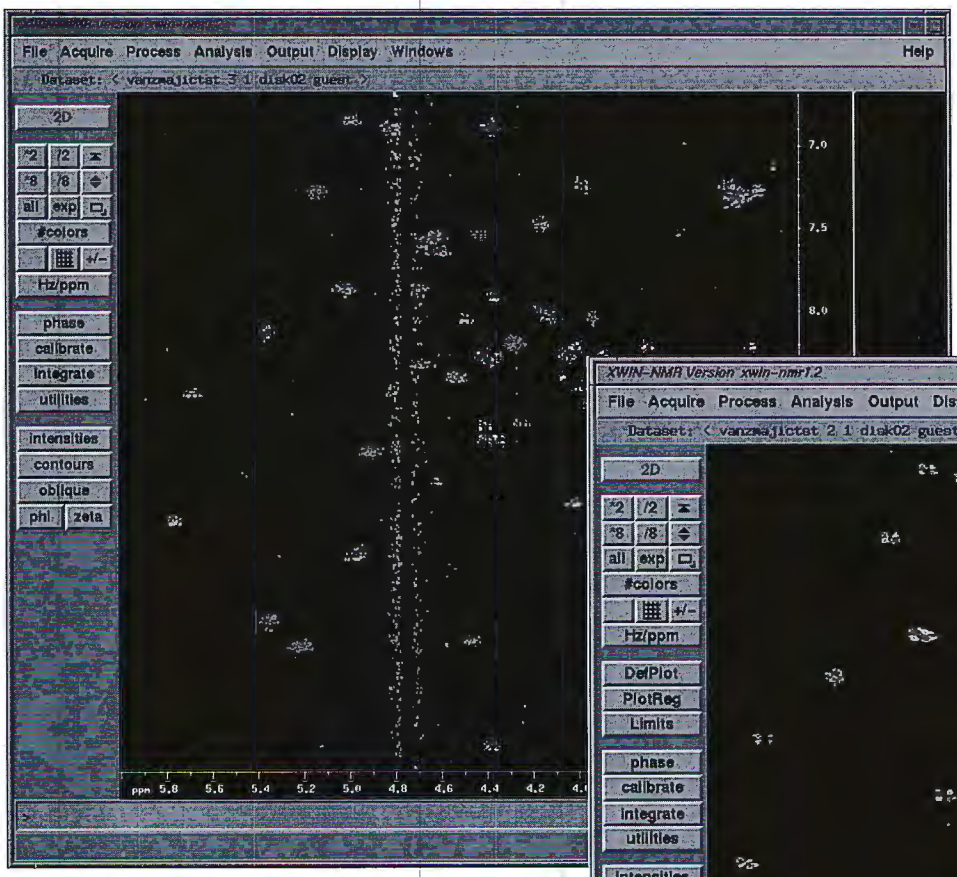
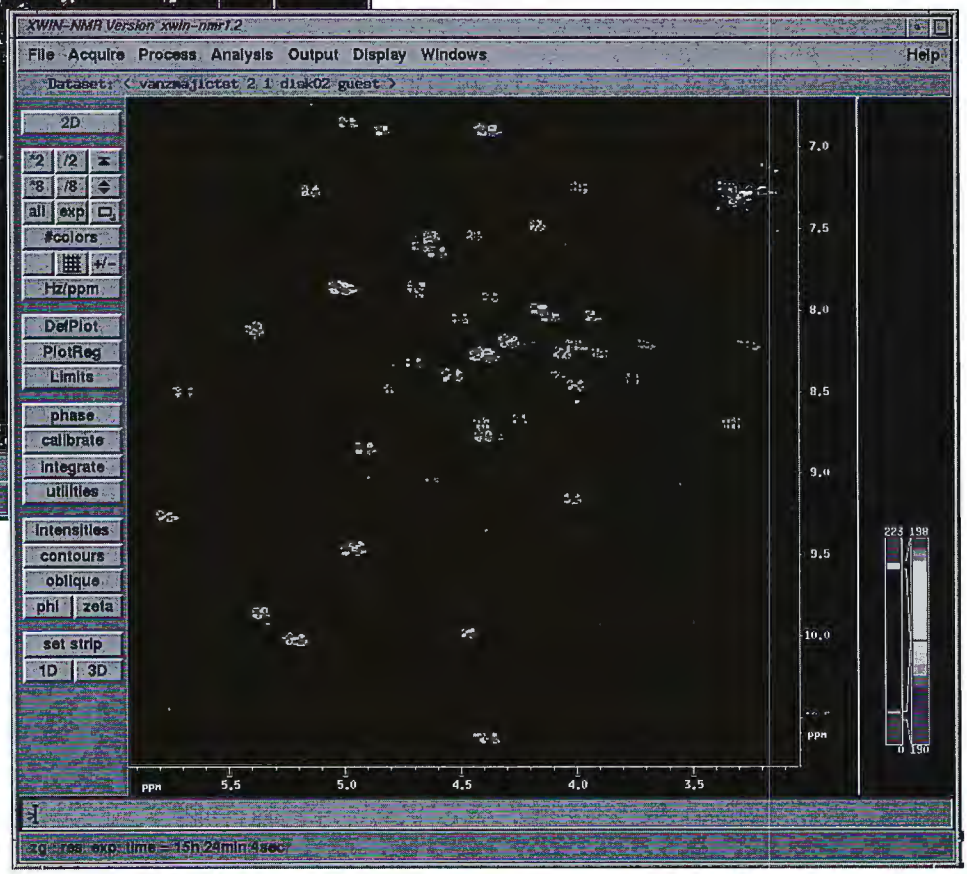


Results with the z-gradient only... residual water ridge is clearly visible and overlaps crosspeaks of interest.

Using the Bruker GRAdient SPectroscopy III (GRASPTM-III) accessory with x,y,z-gradients, an effective gradient at the magic angle can be easily produced by applying three gradients simultaneously. This greatly improves the elimination of residual water by coherence selection in multiple-quantum filtered COSY experiments.

Both experiments were acquired on the Bruker AVANCE 500 equipped with a 5 mm inverse triple resonance (TXI) probe with GRASPTM-III. The sample is 1.5 mM BPTI in 90% H_2O/10% D_2O. The experiment is DQF-COSY.



Results with magic angle gradient... residual water ridge is eliminated! Crosspeaks previously overlapped by the water can be observed and used for correlation assignment. The elimination of the water signal is achieved by coherence selection. No presaturation is used in either experiment!

For complete details or to arrange a demonstration please contact your nearest Bruker representative.

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October 15, 1996

(received 10/21/96)

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

Dear Barry,

Isotope Effects on ABX - Type Spin Systems.

Hardly any of us need a tutorial on the ABX spin system, apart from the optimists who use product operators in arriving at its 2-D responses. Yet, a 'spin on this spin system' goes as follows.

The X region yields the chemical shift of X, $J_{AX} + J_{BX}$ and the positive quantities $\{[(\upsilon_A - \upsilon_B) \pm \frac{1}{2} (J_{AX} - J_{BX})]^2 + J_{AB}^2\}^{\frac{1}{2}}$, as shown in A of the diagram. Now, suppose that A and B are ^{19}F nuclei, you do not own a fluorine probe, but that you have molecules containing a mixture of isotopic substituents, say ^{35}Cl and ^{37}Cl; these cause isotopic perturbations of υ_A and/or υ_B. Further, suppose that no isotopic perturbation of the J's occurs: the only known secondary isotope effects on J's arise from $^1H/^2H$ or $^1H/^3H$ substitution. Finally, suppose that X is a proton. Your sample will display two X spectra, one for each of the chlorine isotopes. Hence four C values are now available. From these, you may deduce J_{AB}, J_{AX}, J_{BX}, the relative signs of the latter two; the two values $\upsilon_A - \upsilon_B$. This assumes that the six peaks of an X component are of detectable intensity.

Generalization to $ABM_nR_o X_p\cdots$ or, indeed, $AB_2M_nR_oX_p\cdots$ spin systems is straight forward. The number of C values is 2^n, where n is the number of M, R and X nuclei. The spectral quantities within the surds increase only as (n + 2). For an ABMX spectrum, for instance, one may deduce $\upsilon_A - \upsilon_B$ and J_{AB}, J_{AX} and J_{BX}, together with the relative signs of the latter two. If an isotope effect on $\upsilon_A - \upsilon_B$ exists, then its magnitude is also available from the X spectrum, say; the number of C's becomes 2^{n+1}, the number of quantities within the surd is n + 3.

As an example, B in the diagram shows the nmr spectrum at 300 MHz and 300K of H-5 of 1-chloro-2,4-difluorobenzene, as a 5 mol % solution in acetone-d_6. Beneath it, in C, is shown the simulated spectrum. The subspectra corresponding to various spin of H-3 and H-6 are indicated by stick spectra. Each subspectrum contains a more intense set, corresponding to ^{35}Cl, of six peaks and a less intense set, corresponding to ^{37}Cl, of <u>four</u> peaks. Because no detectable isotope effect on the coupling constants exists (linewidths of 0.04 Hz), the peaks separated by $J_{AX} + J_{BX}$ (X = H-5) coincide for the molecules containing ^{35}Cl or ^{37}Cl.

The combined ^1H and ^{19}F nmr spectra do not correspond exactly to a spin system, ABMRX, for a given isotope of chlorine in the sense that the internal proton chemical shifts are not all exceedingly large compared to $^nJ(^1H, ^1H)$; that is, ^nJ (^1H, ^1H) on the off-diagonals of the Hamiltonian matrix do somewhat effect the spectrum, the intensities more than the frequencies, as expected from perturbation theory. However, treatment as an ABMRX situation leads to a clear decomposition of the spectrum of H-5 and a set of reasonably accurate constants, C, from which the internal fluorine chemical shifts of the isotopic molecules can be derived; as can all the spin-spin coupling constants involving H-5; also J_{AB}. Note that, in each subspectrum, the C values are C(r,m, ± ½).

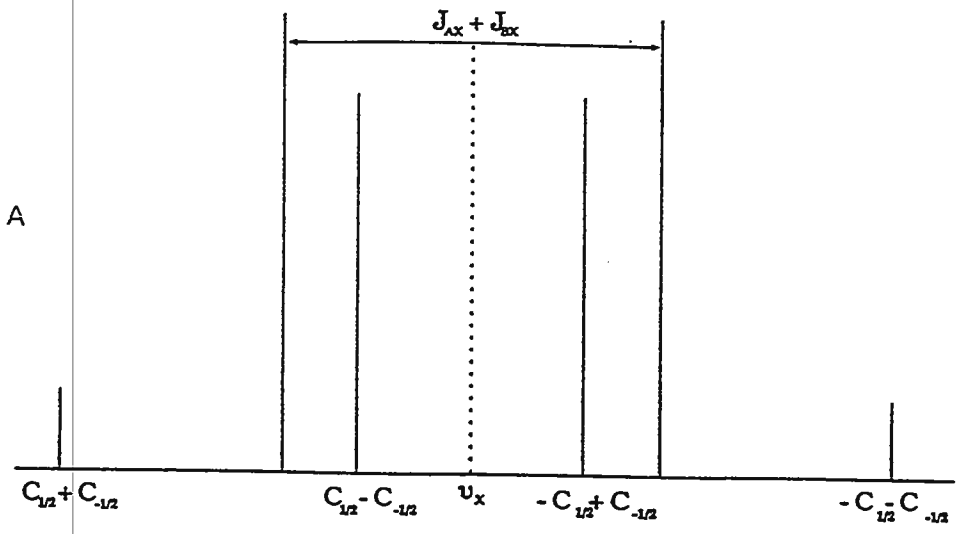
The internal chemical shifts of the fluorine nuclei, obtained from the H-5 spectrum, differed by 0.31(2) Hz or 1.1(1) ppb, being larger in the presence of ^{37}Cl. The known range of $^3\Delta F$ in fluorobenzene derivatives is 1.58 to 1.61ppb (Magn. Reson. Chem. <u>33</u>, 879 (1995)) and implies $^5\Delta F$ as about 0.5 ppb, a value <u>three</u> times larger than $^4\Delta F$ in 2,6-dichloro-4-fluorophenol (see reference above). Trust me!

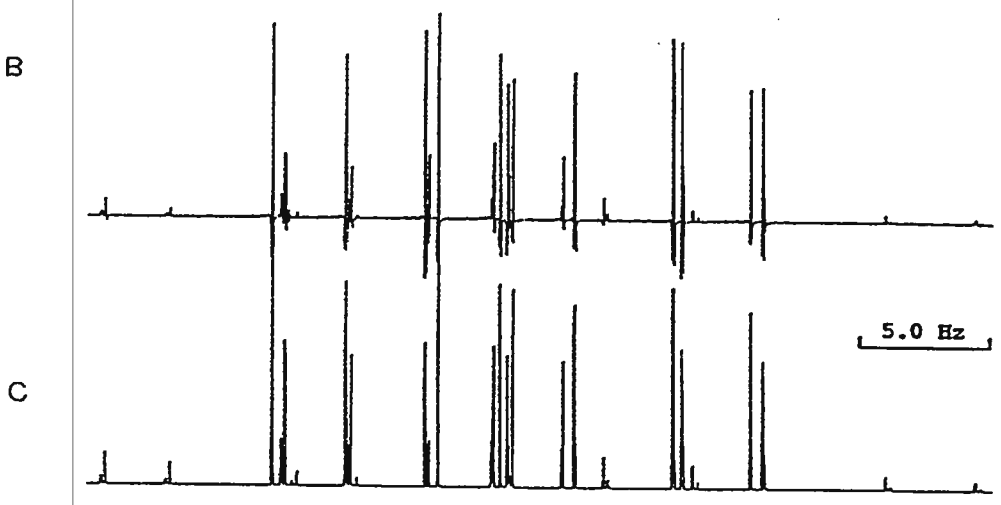
This work was done by Guy Bernard - a patient fellow.

Best wishes from

Ted Schaefer

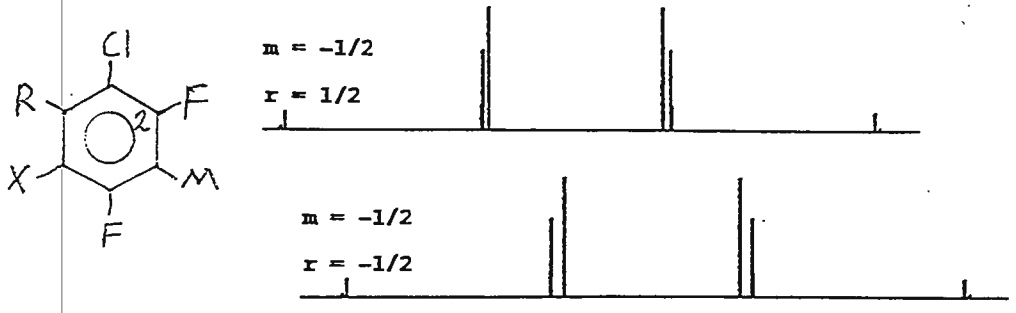
/dg


A
$J_{AX} + J_{BX}$
$C_{1/2} + C_{-1/2}$
$C_{1/2} - C_{-1/2}$
υ_X
$-C_{1/2} + C_{-1/2}$
$-C_{1/2} - C_{-1/2}$


B
C
5.0 Hz


m = 1/2
r = 1/2


m = 1/2
r = -1/2


Cl
R
F
X
M
F
m = -1/2
r = 1/2
m = -1/2
r = -1/2

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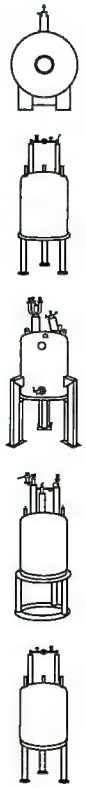

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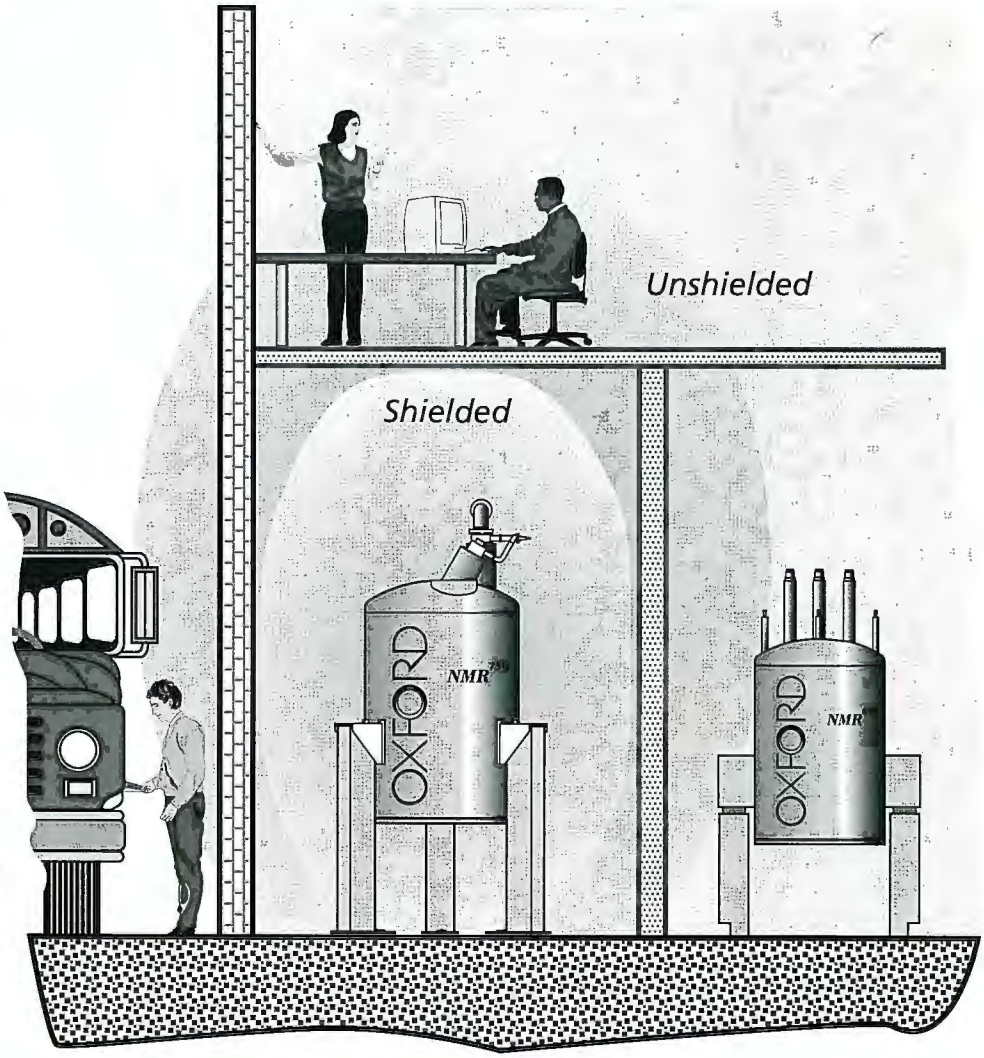

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750	51	15	60	3.8
600	51	10	120	3.4
500	51	10	150	3.2
400	54	8	365	2.8
360	54	8	365	2.8
300	54	3	365	2.8
270	54	2.7	365	2.8
200	54	2	365	2.8
100	54	1	365	2.8
600	89	12	90	3.4
500	89	15	120	3.4
400	89	10	180	2.8
360	89	10	365	2.8
300	89	3	365	2.8
270	89	2.7	365	2.8
200	89	2	365	2.8
100	110	1	119	2.8

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

October 10, 1996
(received 10/15/96)

Re: Gradient-selected HoMQC experiments

Dear Barry,

Hope my subscription renewal has arrived in time and I have not been deleted from the mailing list...

As most installation procedures have been finished for our Varian Unity/INOVA 600 MHz and 500 MHz instruments it is time to report some applications. The main focus is on biomolecular structures, but I would like to show here an implementation of a quick and easy *ho*monuclear *m*ultiple-*q*uantum *c*orrelation (HoMQC) experiment which can be useful for studying both smaller and larger molecules.

The pulse sequence is shown on Figure 1. Multiple-quantum coherence is selected by the pair of gradients, the ratio of which determines the number of quanta passing through. Other than setting the pulse width and choosing the number of quanta there is nothing much else to do when starting this experiment. As soon as we use the gradients for coherence pathway selection magnitude presentation is required. The disadvantage of such presentation is compensated by the simplicity of experiment setup, data processing and visualization. All these make this approach suitable for automation in industrial applications, for example.

We loose sqrt.2 sensitivity by gradient pathway selection/sign discrimination in comparison with that done by phase cycling. However, selection of *n*-quanta itself would require $n * 2$ steps in phase cycling with alternating addition-subtraction in the receiver which usually introduces increasing t_1 noise. This process can be reduced to a single acquisition step using gradients if the concentration is high enough, cutting short experiment time and leading to nice and clean correlation maps.

Figure 2 presents a set of such HoMQC spectra of a *ca.* 20 mM cholesteryl-acetate sample at 600 MHz. 2Q, 3Q, 4Q and 5Q-HoMQC spectra are shown, which were acquired with 100 ms excitation delay each. Overall acquisition time was ca. 5.5 min, 11 min, 22 min, and 44 min, respectively. One times of aliasing was used for the first three spectra, while four times of aliasing for the 5Q-HoMQC.

Sincerely, with my best regards,

Istvan

(István Pelczer)

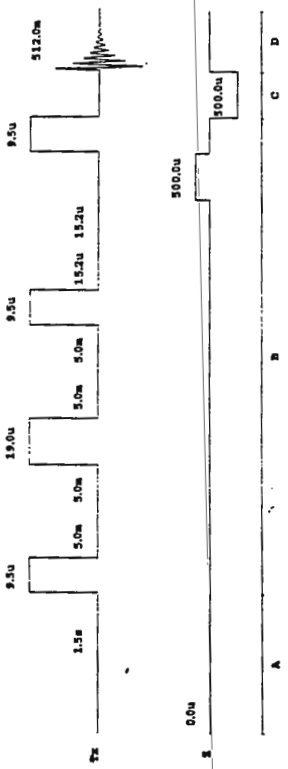

Fig. 1.

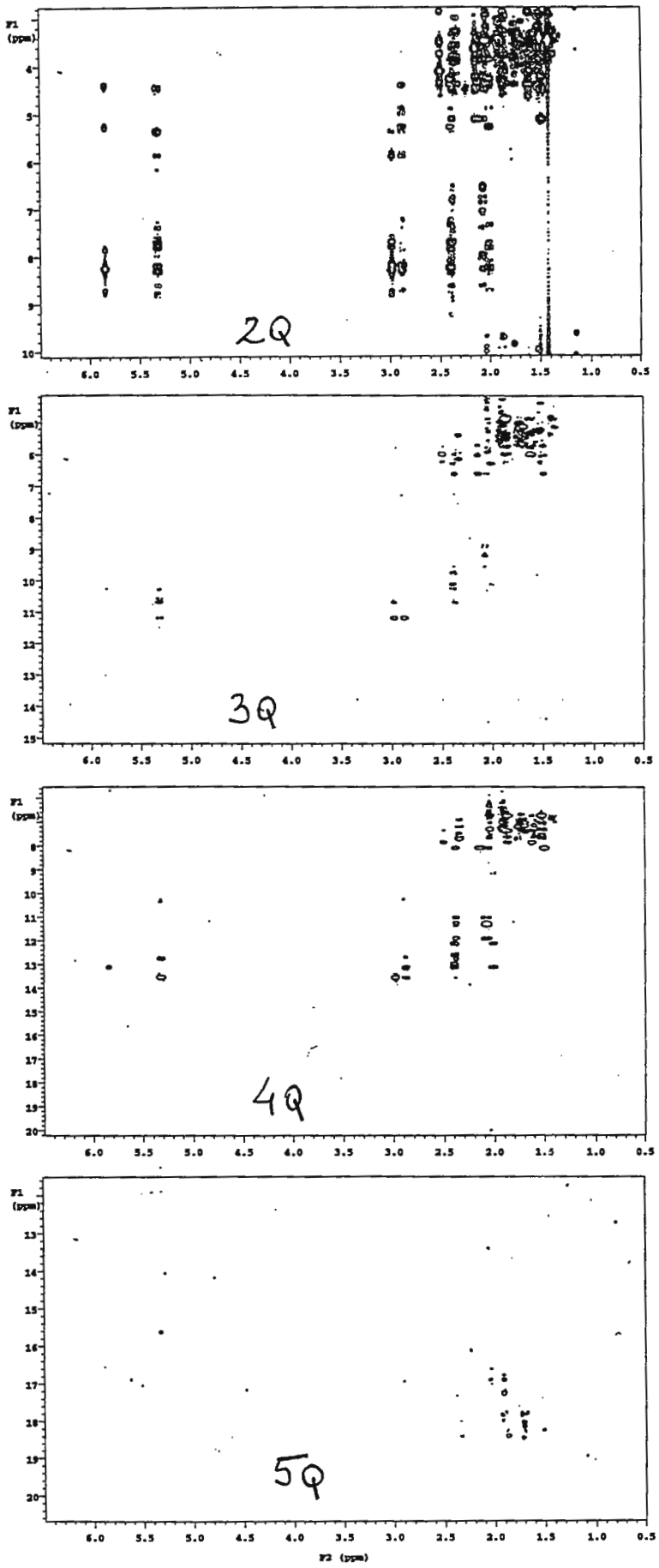

Figure 2.

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Pulse width	20 ms	20 ms
Duty cycle	Up to 10%	Up to 10%
Amplitude droop	5% to 20 ms typ.	5% to 20 ms typ.
Harmonics	Second: -25 dBc max. Third: -24 dBc max.	
Phase change/output power	10° to rated power, typ.	
Phase error overpulse	4° to 20 ms duration, typ.	
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Blanking delay	< 1 µ s on/off, TTL signal	
Blanking duty cycle	Up to 100%	
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Washington
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Department of Physics

1 October 1996
(received 10/5/96)

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

Magnetization Recovery in Rigid Solids Without Spin Diffusion

Dear Dr. Shapiro:

Dipolar relaxation to dilute relaxation centers can proceed by spin diffusion in rigid solids.[1] When that spin diffusion is removed (for example by quadrupolar broadening or by MAS) the residual magnetization recovery process reflects the dimensionality of the nuclear spin array and the nature and concentration of the relaxation centers.

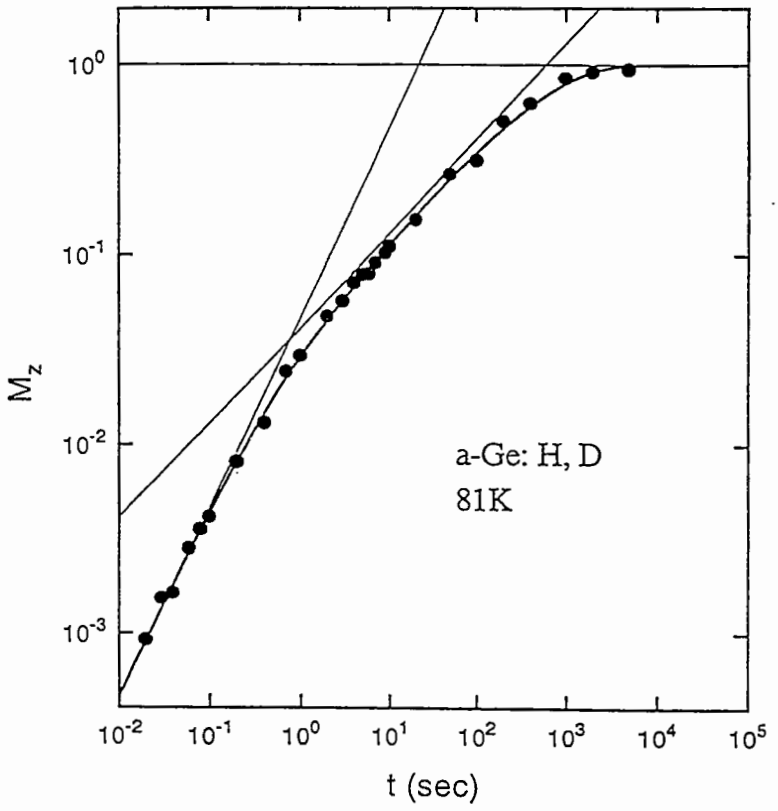


Figure 1: Deuteron magnetization recovery for Ge-bonded D in a-Ge:H,D at 81 K. The heavy solid line shows the fit of the three-dimensional expression.

Washington University
Campus Box 1105
One Brookings Drive
St. Louis, Missouri 63130-4899
(314) 935-6276
FAX (314) 935-6219

Figure 1 shows the transient recovery of magnetization of Ge-bonded deuterons over three orders of magnitude after saturation in deuterated amorphous germanium. Each data point is the average of 40,960 acquisitions. As is evident in the figure, the evolution begins linear in time, switches to $\sqrt{t}$ (for a three dimensional matrix) and then bends over towards the equilibrium magnetization M_o. The curved solid line shows the predicted recovery[2]

$$\frac{M_o - M_z(t)}{M_o} = \frac{1}{1-c}[F(c^2\alpha t) - cF(\alpha t)]$$

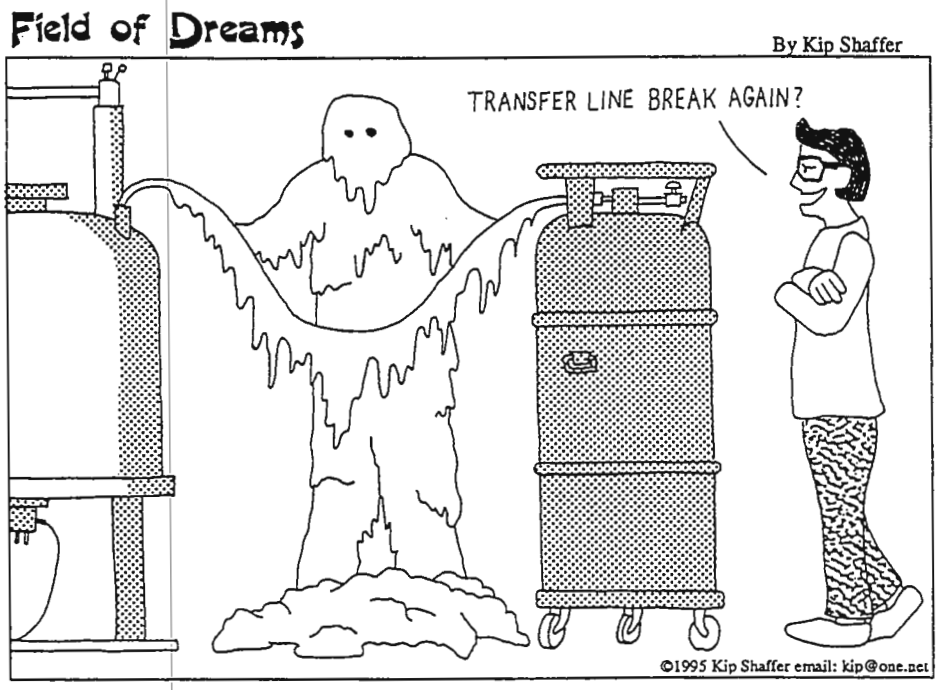
where $F(x) = e^{-x} - \sqrt{\pi x}\ \mathrm{erfc}\sqrt{x}$. Here erfc is the complementary error function, c is an effective concentration of relaxation centers, and α is the dipolar relaxation rate of nuclei adjacent to the relaxation center. For the data of Fig. 1 α is 3.7 sec^{-1} and c is 0.012. The anticipated dipolar rate for a deuteron adjacent to a paramagnetic dangling bond is about 10^4 sec^{-1} and the anticipated dangling bond density is 6×10^{-7}. On the other hand secular flip flop D relaxation to molecular para-D_2 relaxation centers is more likely since there one estimates $\alpha = 1$ sec^{-1} and $c = 1.2\%$ is a reasonable concentration.

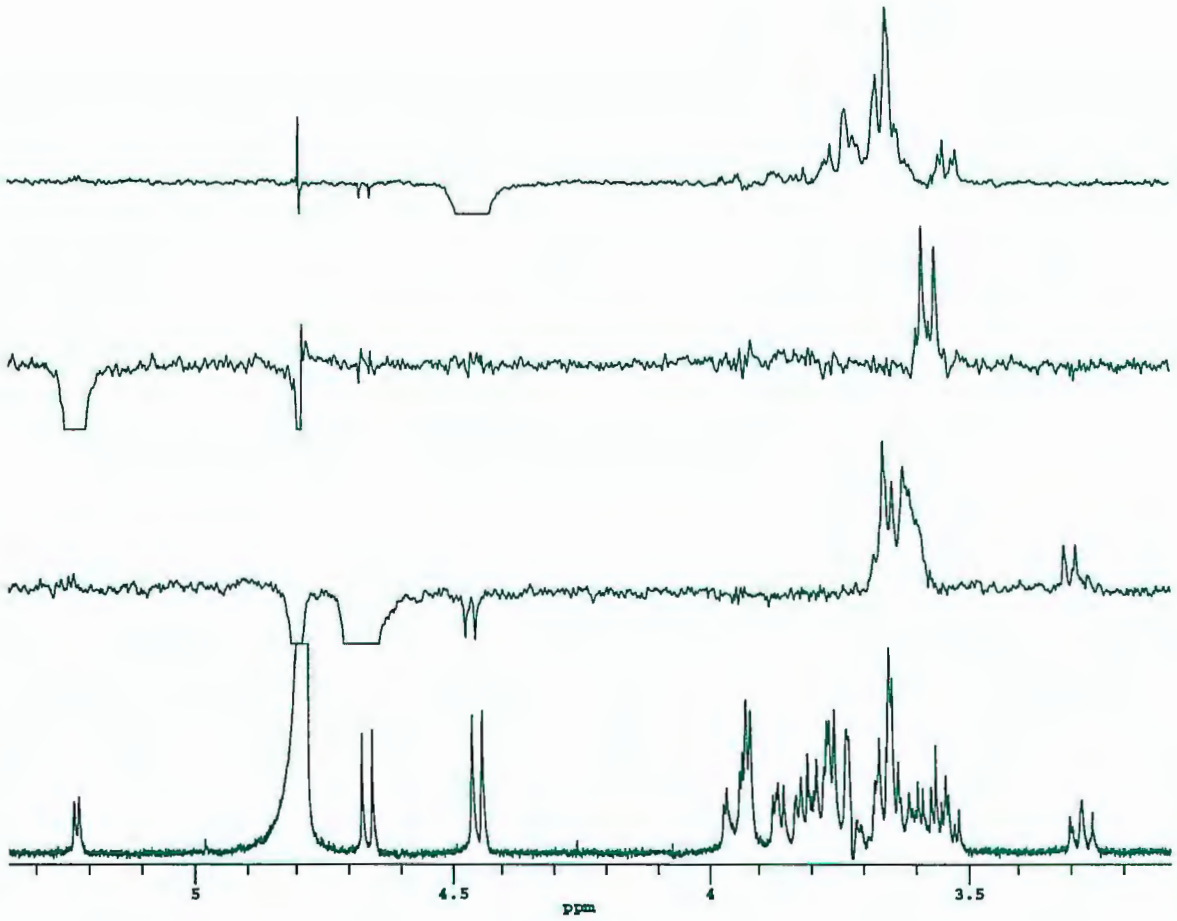
The relaxation model also applies to two- and one-dimensional arrays of spins and these cases involve incomplete gamma functions rather than error functions.

1. N. Bloembergen, *Physica* **15**, 386 (1949).
2. J. R. Bodart, V. P. Bork, T. Cull, H. Ma, P. A. Fedders, D. J. Leopold, and R. E. Norberg, *Phys. Rev.* **B**, in press.

Tom Cull
T. Cull

R.E. Norberg
R. E. Norberg





This spectrum was collected in 128 acquisitions

NOE DIFFERENCE SPECTRA OF LACTOSE

Shown Here are NOE Difference Spectra of Lactose Collected on the Chemagnetics™ 400 MHz CMX Infinity Spectrometer.

Successful nOe difference experiments are critically dependent on an instrument's amplitude and phase stability, as well as lock and temperature stability.

NOe difference spectra of 1.0 mM lactose in D_2O are shown here. The bottom trace is the normal ^1H spectrum acquired in a single acquisition. Upper traces are the nOe spectra acquired with presaturation of different resonances. Note the excellent cancellation of unperturbed peaks.

Data were acquired on a CMX Infinity 400 MHz spectrometer equipped with a Nalorac™ 5 mm indirect detection triple resonance gradient probe. The nOe spectra were collected in 128 acquisitions.

Departments of Radiology & Pathology
NMR LABORATORY

4301 West Markham, Slot 582
Little Rock, Arkansas 72205-7199
501/686-6105
Fax 501/686-5406

Richard A. Komoroski, Ph.D.
Professor

UAMS MEDICAL CENTER

October 8, 1996 (received 10/11/96)

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

Title: Distribution of Lithium in Rat Brain and Muscle by ^{7}Li NMR *In Vitro*

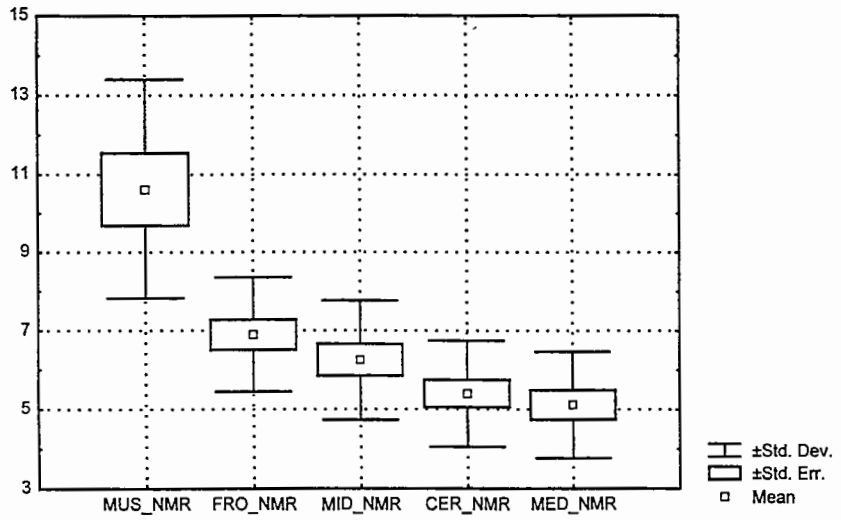
Dear Barry:

Lithium (Li), which is normally present only at trace levels in the body, is efficacious in the treatment of manic-depressive illness. There has been considerable interest in the concentration and regional distribution of Li in animal brain and other tissues. In collaboration with Dr. J. Newton, we have obtained results on the Li distribution in brain and muscle using high resolution ^{7}Li NMR spectroscopy *in vitro* on excised tissue samples from rats which were dosed intraperitoneally with LiCl (3 doses of 5 meq/kg over 1.5 days). The figure is a plot of mean Li concentrations (mM) for rat muscle (MUS_NMR, N=9), forebrain (FRO_NMR, N=14), midbrain (MID_NMR, N=14), cerebellum (CER_NMR, N=15), and medulla (MED_NMR, N=13) determined by ^{7}Li NMR *in vitro* at 116.8 MHz. Extract signal intensities were compared to that of a spherical microcell containing a shifted (with $TmDOTP^{5-}$) Li standard. Each region was statistically ($p<0.05$) different from all others, except for medulla and cerebellum, by t-test for dependent samples. These results serve as a basis for comparison to *in vivo* results in the same animals using ^{7}Li NMR imaging, which we will report later.

Sincerely,

Richard A. Komoroski

John M. Pearce



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Slobodan I. Macura, Ph.D.
Biochemistry and Molecular Biology

October 18, 1996
(received 10/21/96)

CARVE: Frequency Selective Excitation of Arbitrary Shapes

Dear Barry:

Recently we have proposed a new technique for the excitation of arbitrary shapes[1]. It is based on a parallel sequence of small tip angle RF pulses and gradient pulses. The small tip angle rotations co-add yielding a 90° excitation within the selected profile while outside the profile, the rotations cancel each other. In CARVE, *k-space* is discrete because the RF is applied in pulses. The discrete character of k-space permits an arbitrary trajectory for the k-space walk. The optimal random trajectory is found by minimizing the gradient load using simulated annealing. The method is based on the Fourier series expansion of desired profile. Each Fourier coefficient of the expansion defines one event in the pulse sequence. Fourier coefficient position in the *k-space* defines the profile shape; the *k-space* trajectory (each point is visited only once) defines the gradient pulse sequence. The Fourier coefficients amplitude defines RF pulse tilt angle and the phase is equal to the RF pulse phase.

Besides the spatial selectivity, CARVE is also frequency selective. This selectivity makes CARVE very attractive for selective chemical shift imaging of an arbitrarily shaped region. CARVE is relatively easy to implement even on high resolution spectrometer equipped with the three axis gradient probes.

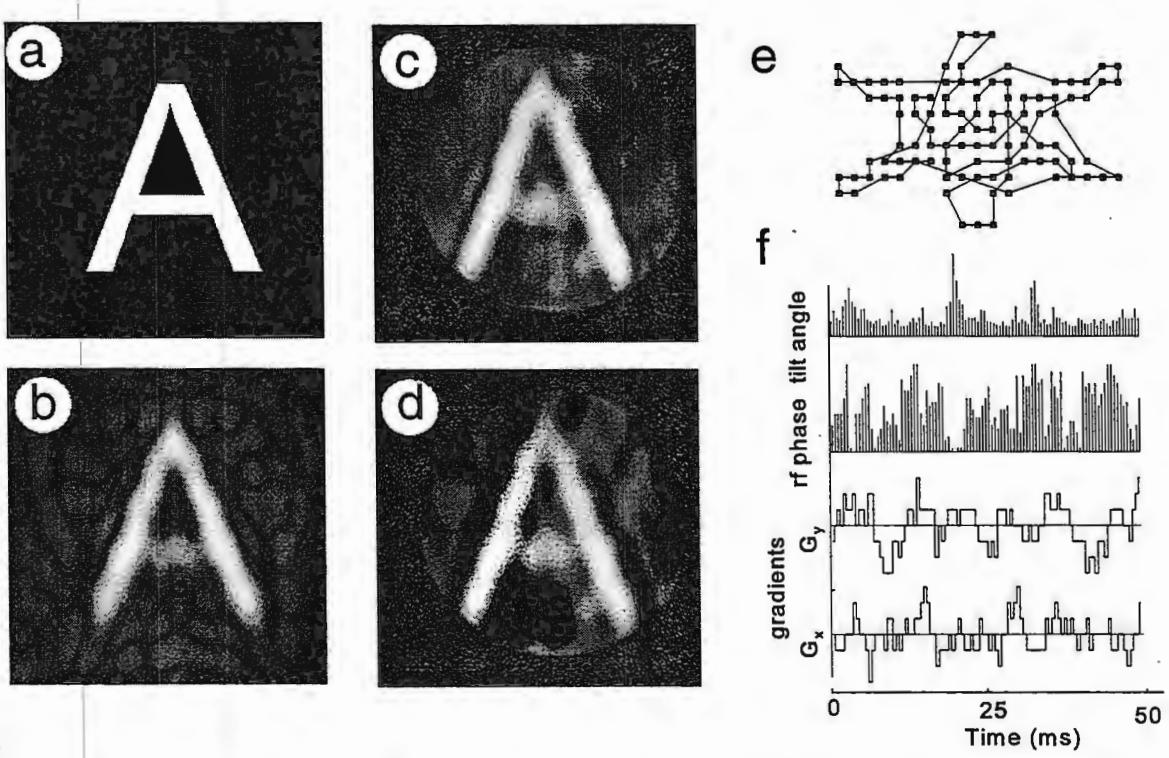


Figure 1 shows 2D CARVE images of character A: a) an ideal theoretical image of 256×256 points is defined with 65,000 (256^2) Fourier coefficients; then its excitation profile would require a sequence with 65,000 pulses; b) the same theoretical image defined with 100 largest coefficients; c) experimental image (absolute value mode) of profile b) excited in a 5 mm tube filled with water; d) experimental image in phase sensitive representation; e) *k-space* trajectory: points represent positions of 100 largest Fourier coefficients of character A; f) pulse sequence consists of 100 events. Each event of 0.5 ms consists of a small tip angle RF pulse with amplitude proportional to the amplitude of respective Fourier coefficient and phase equal to the phase of the coefficients. Gradient sequence is defined by the *k-space* trajectory.

1. I. Sersa, S. Macura, *J. Magn. Reson. Ser. B.* **111,** 186-188 (1996).

Sincerely yours,

Igor Sersa

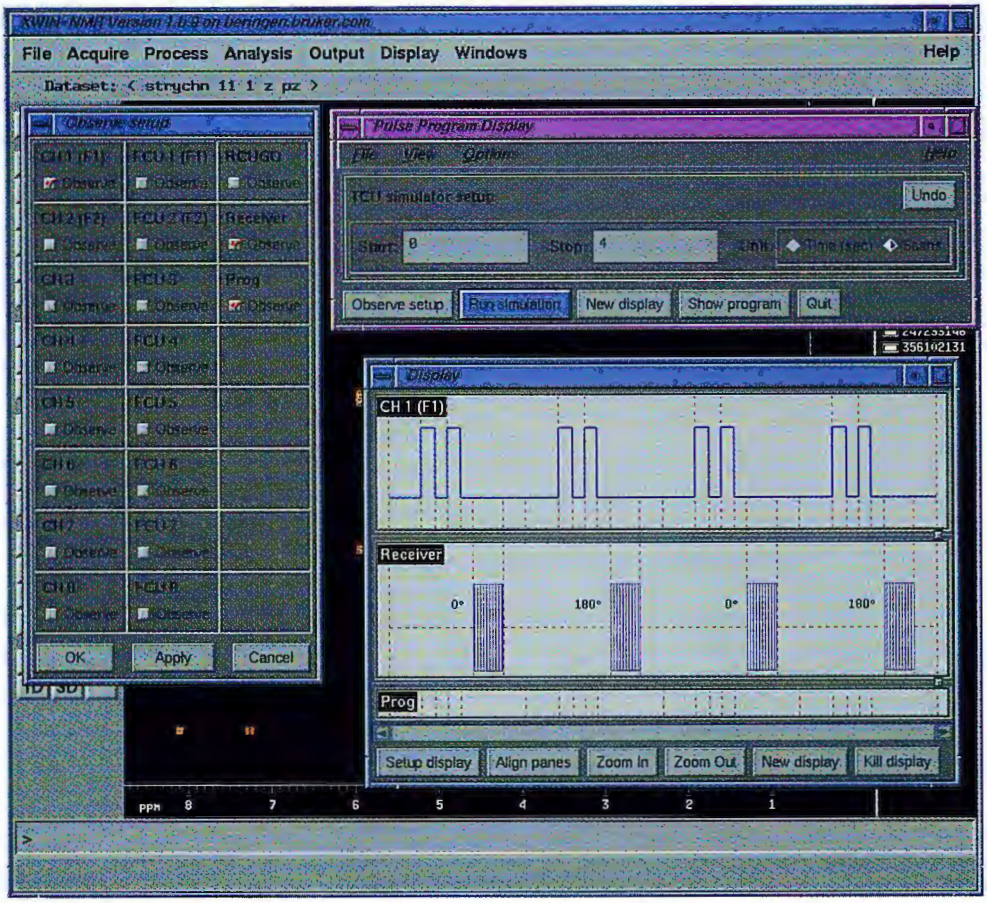
Slobodan Macura
macura@mayo.edu

The NMR Revolution advances...

BRUKER

XWIN-NMR™ Software:

Pulse Program Display



The *Pulse Program Display* module in XWIN-NMR, Bruker's new NMR software package, provides **exact** graphical visualization of a pulse sequence on the *AVANCE* spectrometers. This includes illustration of the amplitude, timing and phase for all RF and gradient channels. All this to make even the most complicated experiment look easy!

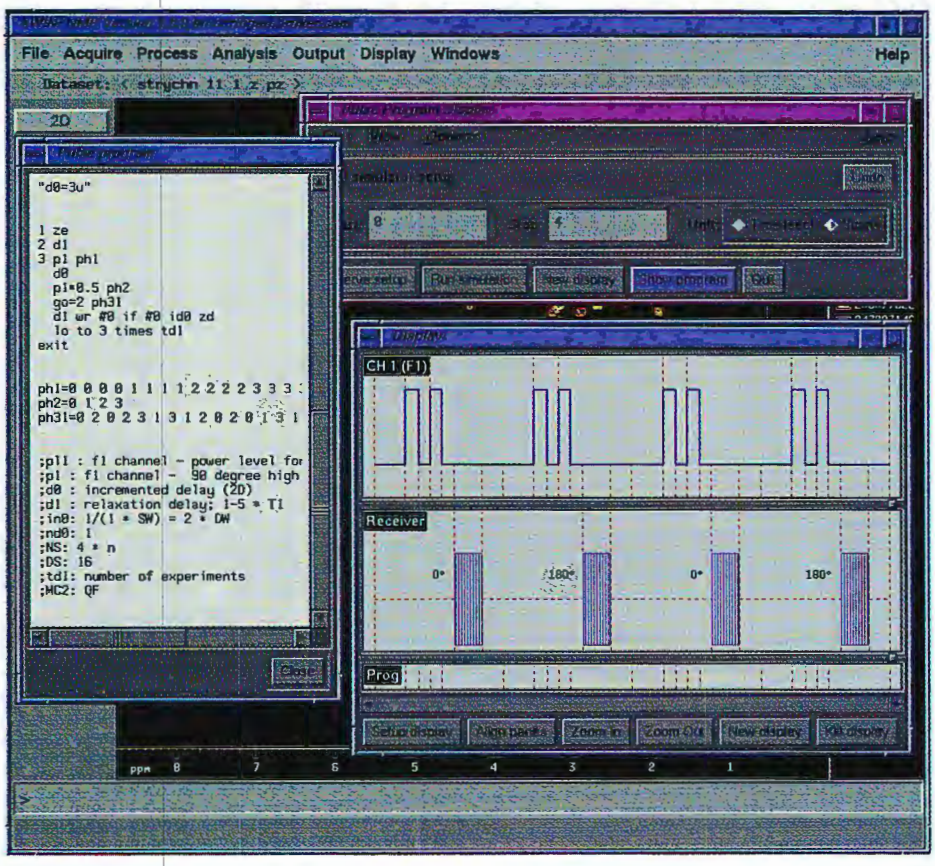
The *Pulse Program Display* uses the *same* XWIN-NMR pulse program compiler to create the graphical display *and* to control the hardware. Other graphical display programs use a separate interpreter to create the sequence display. This latter approach introduces possibilities for error, leading to frustration in debugging the pusle sequences.

BRUKER

...The NMR Revolution advances

Features of the *Pulse Program Display*:

- easy-to-use graphical X11/Motif user interface,
- simple scrolling through the displayed sequence,
- easy set-up and navigation,
- zoom from 12.5 nsec to entire experiment,
- multiple displays in independent windows,
- number of channels, pulse-gating, phase and amplitudes individually selectable,
- simulation in units of time or scans,
- simulation of multidimensional sequences including evolution.



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Stockholm, 17 October 1996

(received 10/25/96)

ROYAL INSTITUTE OF TECHNOLOGY

KTH

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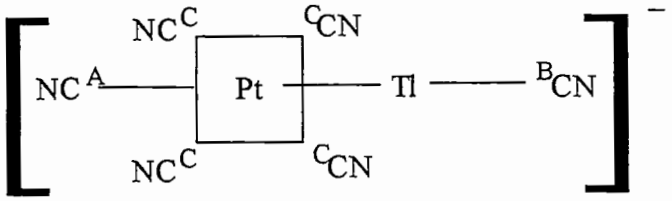
Dr. B.L. Shapiro
The NMR Newsletter
966 Elsinore Court
Palo Alto, CA 94303, USA

First 2-Dimensional ^{205}Tl - ^{195}Pt Spectra

Dear Dr. Shapiro,

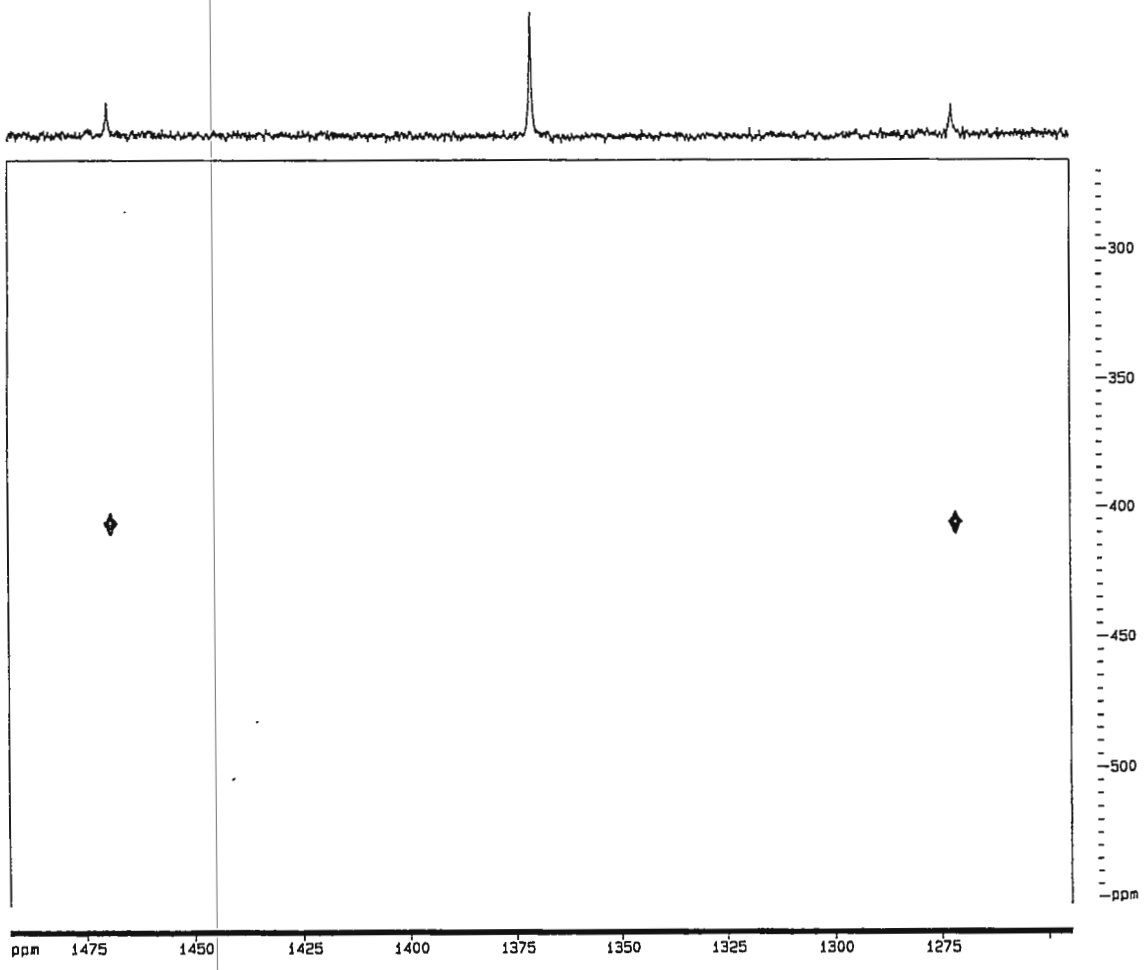
Recently, we have shortly described the formation and characterisation of the first representative of a new class of Pt-Tl cyanide species containing a strong unsupported metal-metal bond, namely the complex ion



which is formed in aqueous solution.[1] Now, we set out to investigate the structure of this class of compounds in solution using 2D methods. To this end, we are fortunate to have a Bruker-made ^{205}Tl-X probe, when X can be any nucleus in the broadband region. This probe takes 5 mm o.d. samples and was specially optimized to give short pulses in our "lossy" solutions. In fact, we observe a 90° pulse for ^{205}Tl NMR below 10 μs, whereas for the same solutions it was 70-100 μs on our old (but still going strong) AM400. Interestingly, also pulses for the X-nuclei (outer coil of this probe) are unexpectedly short, in the range of 10 μs, or even below for ^{13}C NMR.

The Figure shows the first inverse-detected heteronuclear 2D experiment using a thallium nucleus. This experiment is also the one using the greatest coupling constant for coherence tranfer; 57 kHz! The (^{205}Tl,^{195}Pt) HMQC spectrum was recorded using the automated Bruker pulse sequence *invnd*.[2] We recorded 512 FIDs and no zero filling (512W) in the F_1 dimension with a spectral width of 62 kHz; 1K real data points, no zero filling, a spectral width of ~100 kHz (acquisition time 10 ms), 16 scans and a relaxation delay of 180 ms

(T_1 for Tl was determined as 28.9 msec) were used in the F_2 dimension. The delay for inversion recovery was set to 484.0 msec. The experiment time was under two hours.



(^{205}Tl,^{195}Pt) HMQC spectrum of a 50 mM aqueous solution containing $[(NC)_5Pt\text{-}Tl(CN)]^-$ and some other monomeric Tl- and Pt-species

Mateus W. da Silva *Julius Glaser*

References

1. K.E Berg, J. Glaser, M.C. Read, and I. Tóth, *J. Am. Chem. Soc.*, 1995, 117, 7550.
2. A. Bax and S. Subramanian, *J. Magn. Reson.*, 67 (1986) 565.

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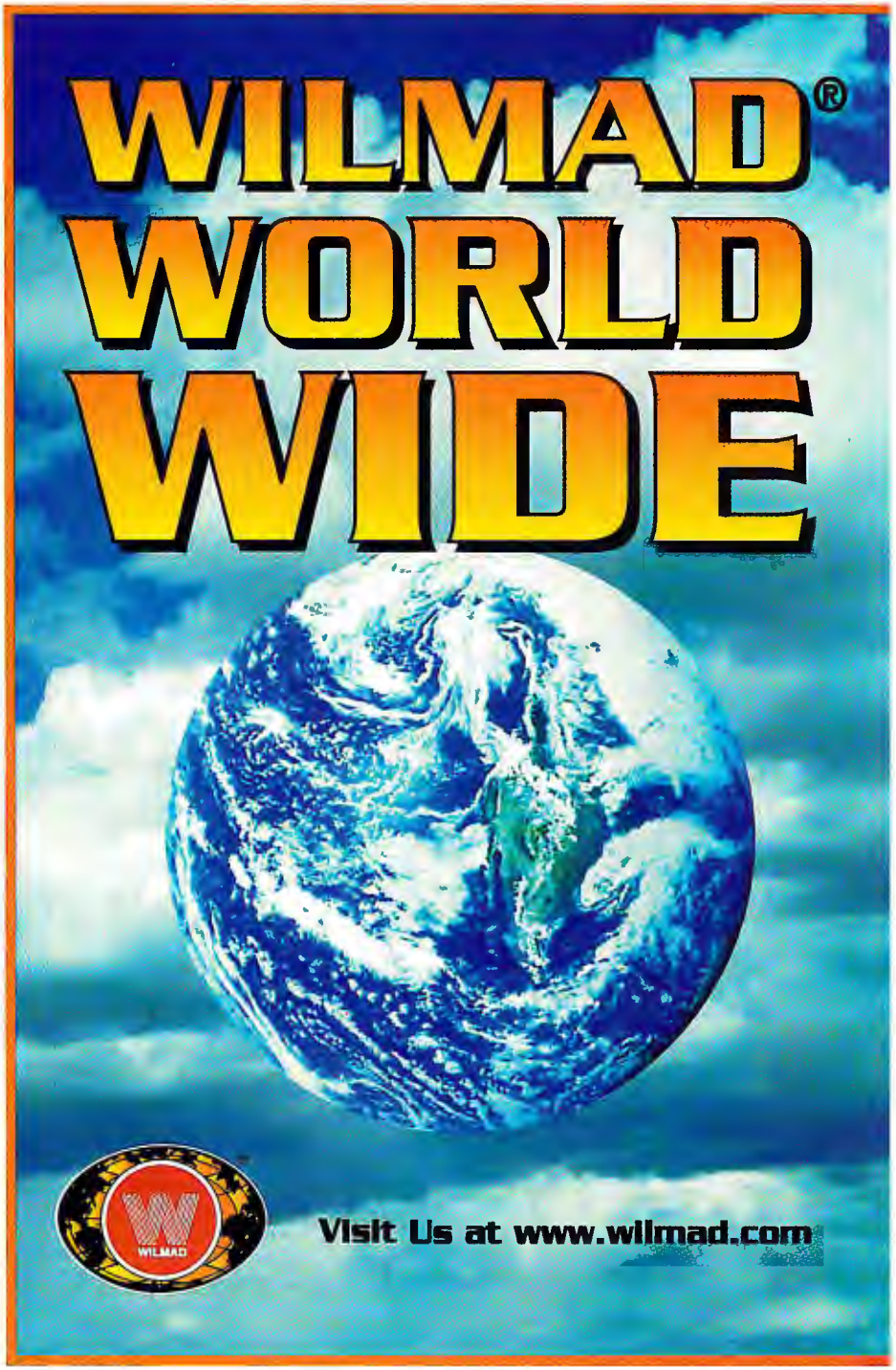

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Sept 30,1996
(received 10/9/96)

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

Title: ^{13}C Chemical Shift Tensors for Solid Veratrole (1,2-dimethoxybenzene).

A great deal of literature has appeared regarding the solution conformation of the title molecule. Due to its low melting point (15°C) however, no X-ray crystal structure has been published and there are no solid state NMR data. After a great deal of meticulous effort, PhD student Marielle Gerzain has succeeded in growing a crystal at low temperature and obtaining both an X-ray structure and a series of low temperature ^{13}C CPMAS spectra at various spinning rates. The latter set of experiments have permitted a measure of the ^{13}C shielding tensors using the Herzfeld and Berger method (J. Chem. Phys. 73, 6021 (1980)).

Below are tabulated the results of this work along with a comparison of the calculated values using the LORG approach with a D95V basis set. The X-ray crystal structure was used for the geometry. CM1 and CM2 are the methyl groups attached to C1 and C2 respectively. It can be seen that there is reasonable agreement between theory and experiment.

		C_1	C_2	C_3	C_4	C_5	C_6	CM1	CM2
solution	σ_{iso}	149.1	149.1	111.4	120.9	120.9	111.4	55.7	55.7
solid	σ_{iso}	149.4	149.4	112.0	122.8	121.1	110.4	55.0	56.9
	σ_{11}	209	209	185	215	210	184	82	84
	σ_{22}	168	168	137	139	134	134	68	68
	σ_{33}	71	71	14	14	19	13	15	19
LORG, D95V	σ_{iso}	157.4	158.4	117.9	128.7	122.4	112.7	52.8	56.8
	σ_{11}	236.9	240.0	218.9	246.2	237.2	213.6	79.6	81.1
	σ_{22}	167.8	170.3	130.7	132.4	129.6	125.5	70.1	75.5
	σ_{33}	67.4	64.8	4.2	7.4	0.4	-0.9	8.8	13.9

G.W. Buchanan, Professor of Chemistry

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Edmonton

Canada T6G 2G2

Department of Chemistry
Faculty of Science

Chemistry Building, Telephone (403)492-3254
FAX (403)492-8231

October 3, 1996
(received 10/9/96)

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

Dear Barry :

RE: PHASE AND AMPLITUDE MODULATION IN APT SPECTRA

As Alex (1) recently pointed out, every so often we rediscover the effects of strong coupling. In our case it occurred when we were obtaining the normal (A) and APT(B) ^{13}C spectrum of the compound shown below. The compound contains two chemically equivalent but magnetically non-equivalent phosphorus nuclei so that the methylene carbons form the X part of an ABX spin system. There are two nearly coincident ABX spin patterns in A because the methylene carbons above the plane of the paper have a slight chemical shift from those below the plane. The APT spectrum shown in B shows phase and intensity modulation of the inner and outer transitions. A full explanation of these results will be reported elsewhere.

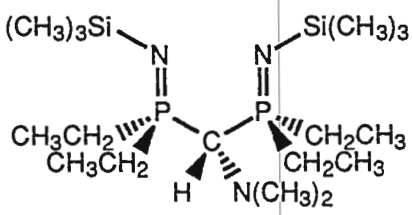
Sincerely yours,

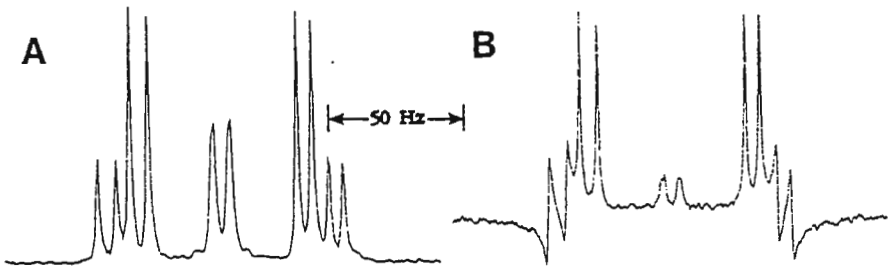
Tom

Tom Nakashima

1. A.D. Bain, *The NMR Newsletter*, 455-11, 1996.

TTN:dd





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2. Biophysical Chemistry - Candidates with strong backgrounds and expertise in biophysical chemistry are sought. The successful candidate must establish an independent research program to investigate an important biological problem that involves macromolecular interactions. Center facilities include analytical ultracentrifugation, electrospray mass spectrometry and dynamic light scattering apparatus.

Candidates for both positions must have outstanding potential in research with a record and stature in their field of expertise. They are expected to attract funding from national agencies and to fully participate in teaching and service with the Department of Human Biological Chemistry and Genetics. Affiliations with The Sealy Center for Structural Biology, other UTMB Centers and relevant Departments are possible.

Structural Biology currently consists of 10 biophysical faculty members in areas of NMR spectroscopy, x-ray crystallography, computational biology, and biophysical chemistry. Facilities in areas of NMR spectroscopy, computation, x-ray crystallography, and biophysical chemistry are directed by Drs. David Gorenstein, Werner Braun, Robert O. Fox and James Lee, respectively. Structural Biology received financial support from UTMB and endowed support from the Sealy Center for Structural Biology. Facilities consist of a newly renovated building and additional laboratories, along with excellent computational, NMR, crystallographic, and biophysical equipment.

Applications should include a complete curriculum vitae, a description of research interests and names and addresses of at least three references. Review of applications will continue until the positions are filled. Applications should be addressed to:

Dr. James C. Lee
Structural Biology Search Committee
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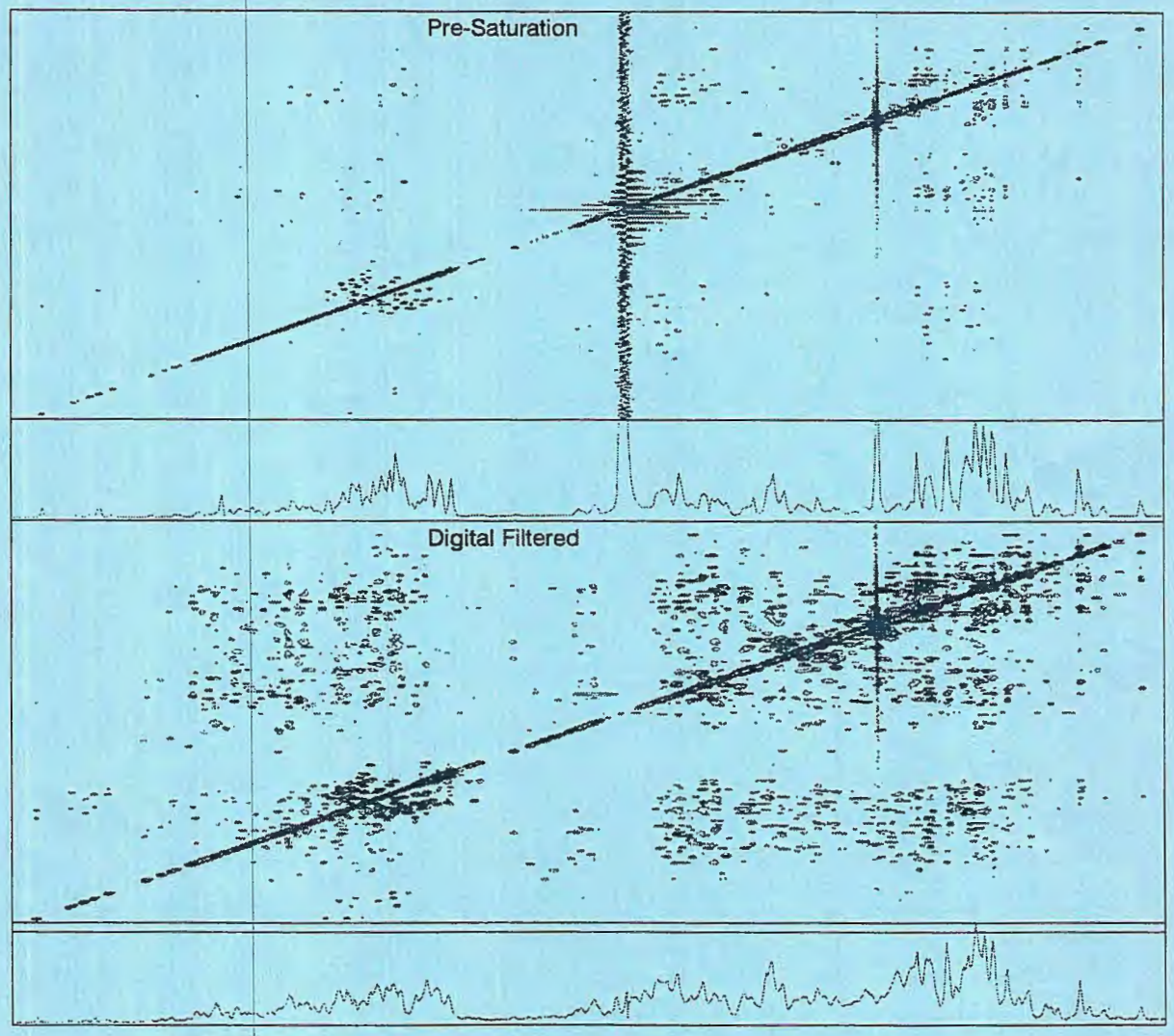

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