TEXAS A&M UNIVERSITY

NMR NEWSLETTER

No. 374

November 1989

Continued on page 70

TABLE 1 DEUTERATED SOLVENTS

Cat. No.	Description	Formula	Min. Iso. (D)	Density (g/cc)	MP (°C)	BP (°C)	$-X_v \times 10^6$ @ (°C)
D-11	Acetic Acid-d_4	CD_3COOD	99.5%	1.11	17	118	0.551 (32)
D-120	Acetic Acid-d_4 TMS		99.5%				
D-13	Acetone-d_6	CD_3COCD_3	99.8%	0.84	-94	57	0.460 (20)
D-121	Aceonte-d_6 + 1% TMS		99.8%				
D-129							
D-14							543 (20)
D-21							0.611
D-122							
D-130							
D-28	Choloroform-d	$CDCl_3$	99.8%	1.50	-64	62	0.740 (20)
D-31	Chloroform-d + 1% TMS		99.8%				

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Cost-conscious quality NMR solvents offered by Wilmad, such as $CDCl_3$, are frequently priced lower than more traditional sources. Included in this offering are the most common solvents, like Acetone-d_6, Benzene-d_6, D_2O, and DMSO-d_6, as well as some of the most unusual solvents for specialty applications, like 1,1,2,2-Tetrachloroethane-d_2, Octane-d_8, and Trifluroacetic Acid-d.

VARIAN

BOX/500 SHEETS

Cat. No.	Size	Instrument Model	Type
WCV-100 (S-100/4)	11" X 26"	HA-100, HA-100D	Gridded-Two Color
WCV-60 (A-60C)	11" X 26"	A-60, A-60A	Gridded-Two Color
WCV-XL (XL-100)	11" X 26"	XL-100 (CF and FT)	Gridded-Two Color
WCV-XLFT-100	11" X 26"	XL-100 (CF and FT)	Gridded-Two Color
WCV-XL-200	11" X 26"	XL-200, XL-300, XL-400	Gridded-Two Color
WCV-20 (CFT-20)	11" X 16 3/4"	CFT-20, FT-80, FT-80A	Gridded-Two Color
WC-11	X 17"	All Models	16 1/2" or 16 Blank

Use Plotter Paper and Pens? WILMAD HAS YOUR CHART!

We provide the largest variety of paper and pens for NMR recorders or plotters available anywhere. Included in these listing are the newest spectrometers from Varian, Bruker, (and IBM), General Electric and JEOL, as well as the latest models, such as the Hewlett Packard 7475A, 7550A, and Thinkjet 2225A, Zeta 8 or 8A, and Western Graphtec 4730 plotters and printers.

Searching for the Unusual Requirement?
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The most comprehensive offering of "widgets, gadgets and specials" for NMR spectroscopy, including:

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FORTHCOMING NMR MEETINGS

International Symposium NMR Spectroscopy: Structure and Dynamics of Polymeric Materials in the Solid State, Sponsored by the ACS Division of Polymer Chemistry, December 5-8, 1989; Keystone, Colorado; Contact: Mrs. Betty J. Schreiner, E. I. du Pont de Nemours & Co., Experimental Station, Wilmington, DE 19880-0356; (302) 695-4817.

Chemical Frontiers in Magnetic Resonance of Living Systems, Workshop in the Pacific Basin Congress/American Chemical Society, December 17-22, 1989, Honolulu, Hawaii; See Newsletter 372, 52.

Spatially Determined NMR, Sponsored by the British Radiofrequency Spectroscopy Group; December 17-20, 1989; Cambridge University, U.K.; Contact: Prof. L. D. Hall, Level 4 RTC, Addenbrookes Hospital, Hills Road, Cambridge CB2 2QQ, England: (44) (223) 336805.

Workshop on In Vivo Magnetic Resonance Spectroscopy III, March 29 - April 1, 1990, San Francisco, California; San Francisco, California; Contact: Dr. M. W. Weiner or Dr. G. B. Matson, Magnetic Resonance Unit, Veterans Administration Medical Center, 4150 Clement Street (11D), San Francisco, CA 94121; (415) 750-2146.

31st ENC (Experimental NMR Conference), April 1-5, 1990; Asilomar Conference Center, Pacific Grove, California; Contact: ENC, 750 Audubon, East Lansing, MI 48823; (517) 332-3667; Attendance: 1,200.

Frontiers of Polymer Characterization by NMR Spectroscopy, Symposium at the American Chemical Society National Meeting, April 22-27, 1990, Boston, Mass.; See Newsletter 372, 54.

Workshop of Special Topics in Medical Magnetic Resonance, sponsored by the Society of Magnetic Resonance in Medicine and the National Research Council of Canada, July 23-27, 1990; Whistler Mountain, BC, Canada. Contact: L. Forget - see Newsletter 374, 46.

Expanding Frontiers in Polypeptide and Protein Structural Research, sponsored by the National Research Council of Canada, July 23-27, 1990; Whistler Mountain, BC, Canada. Contact: L. Forget - see Newsletter 374, 46.

Tenth International Biophysics Conference, sponsored by the International Union of Pure and Applied Biophysics and the National Research Council of Canada, July 29 - August 3, 1990; Vancouver, BC, Canada. Contact: L. Forget - see Newsletter 374, 46.

Additional listings of meetings, etc., are invited.

All Newsletter Correspondence Should Be Addressed To:

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

(415) 493-5971

DEADLINE DATES

No. 376 (January) -----15 December 1989
No. 377 (February) ------19 January 1990
No. 378 (March)-------- 16 February 1990
No. 379 (April) ----------16 March 1990

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Mailing Address:
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Baltimore, Md. 21205

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

(received 10/11/89)
October 5th 1989

Non-Linear Least Squares fitting of the FID

Dear Professor Shapiro,

Thank you for your recent reminder. The in vivo ^{31}P NMR spectra that we record on our 1.5T GE Signa spectrometer usually have one or more of the following characteristics; low signal-to-noise ratios, overlapping peaks, or undulating baselines. Each of these features makes the measurement of peak area difficult. The traditional method of manual curve fitting in the frequency domain is both inaccurate and irreproducable under these conditions, and is certainly a time-consuming process. Consequently, we have adopted direct Non-Linear Least Squares (NLLS) fitting of the FID as a means of analyzing ^{31}P NMR spectra in an automated fashion. Time-domain fitting is especially appropriate in the case of spatial localization pulse sequences such as "DRESS" or Chemical Shift Imaging which involve delayed data acquisition.

In comparison to Linear Prediction Singular Value Decomposition (LPSVD), NLLS appears to be more stable under conditions of low signal-to-noise. This is primarily due to the ability of NLLS to incorporate prior knowledge about any spectral parameters (phase, frequency, linewidth, number of peaks, etc..) into the model. Figure 1 shows the application of NLLS to the analysis of a ^{31}P NMR spectrum recorded using a surface coil placed over the frontal lobe of the human brain. (A) is the Fourier transform of the FID, (B) is an "ersatz" spectrum produced from the parameters returned by NLLS, and (C) is the difference (A)-(B),

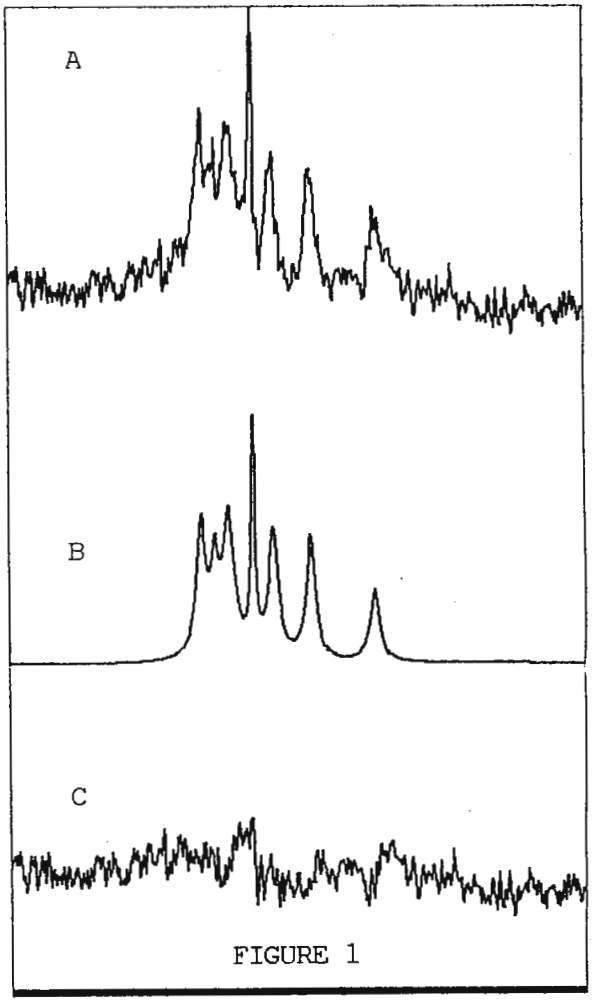

FIGURE 1

yours sincerely,

P.B. Barker

Peter B. Barker. Sibusiso Sibisi.

Fremont Magnetic Resonance
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(415)623-0722
(415)623-0851 FAX

NMR Instrument Upgrades

Installed NMR Instruments can be upgraded to do most of the NMR experiments being used today. FMR offers a variety of products and services to aid the NMR researcher in using his existing NMR instrument to solve problems. FMR can help the researcher by:

- Helping tounderstand and eliminate instrumental problems.
- Analyzing and improving RF stability.
- Finding work-around solutions for instrumental restrictions.
- Upgrading his existing NMR probes.
- Suppling accessories to expand his NMR's capabilities.
- Providing RF and probe repair services.

Noise Figure Meters

Knowing your NMR system's noise figure allows you to separate the variables of performance. Is the problem area shimming, preamp or external noise? A five minute test with an inexpensive ($500) meter can tell you where the problem area lies.

Indirect Detection

FMR provides accessories to allow excitation of an X nucleus and the observation of ^{1}H for indirect detection, 2DFT and 3DFT experiments:

Broadband Decoupler with:

- 90° and 180° phase shifts standard.
  (sub 90° phase shifts optional)
- MLEV and Waltz Modulation.
- Fast Bi-Level Switching (< 1 usec)
- Low cost / high performance.

Upgrade an existing probe to a $^{1}H\{^{31}P\text{-}^{15}N\}$ probe, or buy a new $^{1}H\{^{31}P\text{-}^{15}N\}$ probe.

Inverse ^{1}H $\{^{31}P\text{-}^{15}N\}$ 5mm Probe

	5mm	10mm
90° Pulse @ 50 Watts	15us	25us
Resolution (ODCB)	0.25	0.4
Lineshape ($CDCl_3$)	10/25	18/40
Gamma H2 (10W)		
^{15}N	2KHz	1KHz
^{13}C	3KHz	2KHz
Sensitivity (0.1%EB)		
200 MHz	60	100
300 MHz	120	200
360 MHz	170	280
400 MHz	200	330
500 MHz	300	500

Probe Upgrades

Existing probes can be upgraded at less than half the cost of a new probe. An existing unused 20mm probe, for example, can be converted to a 10mm Broadband probe. Or a 5mm ^{1}H Probe can be reworked to improved sensitivity or performance with ionic water solutions.

5mm ^{1}H Probe

90° Pulse	10 us @ 50 Watts
Resolution	0.2 Hz (ODCB)
Lineshape	10/20 ($CDCl_3$)
Sensitivity (0.1% EB)	
200 MHz	80:1
300 MHz	160:1
360 MHz	220:1
400 MHz	300:1
500 MHz	400:1

Broadband Probes

High Band broadband probes cover the range from ^{31}P to ^{15}N for solutions up to 0.2 M salt.

		Sensitivity				
5mm		**200**	**300**	**360**	**400**	**500**
^{31}P		30	60	85	100	150
^{13}C		30	60	85	100	150
^{2}H	NA=4	5	10	14	16	25
^{17}O	NA=100	20	40	55	65	80
^{15}N		4	8	10	12	16
10mm						
^{31}P		130	260	340	375	450
^{13}C		150	300	360	400	500
^{2}H	NA=4	15	30	40	50	60
^{17}O	NA=100	110	220	270	300	400
^{15}N		15	30	35	40	50
12mm						
^{31}P		150	300	400	500	550
^{13}C		180	360	500	600	700
^{2}H	NA=4	20	40	50	60	75
^{17}O	NA=100	125	250	300	350	500
^{15}N		15	30	40	50	60
20mm						
^{31}P		250	500	600	750	
^{13}C		300	600	700	800	
^{2}H	NA=4	30	60	70	90	
^{17}O	NA=100	180	350	450	550	
^{15}N		25	50	70	80	

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Instrumentation Note 13

Finished Shimming ?

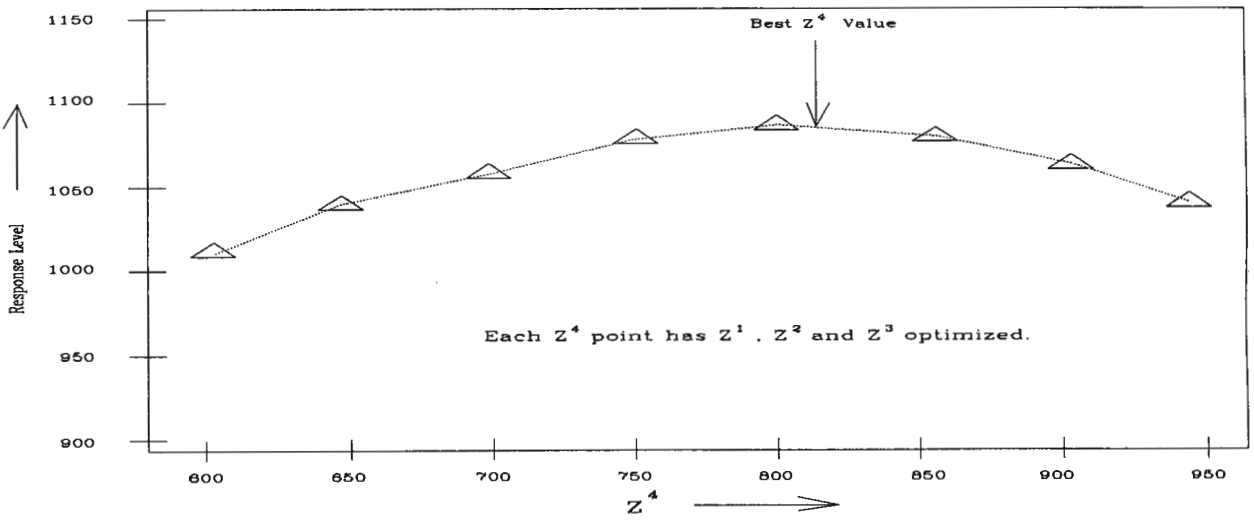
If all shim gradients were pure, the gradients they generate would be orthogonal and independent. Under these conditions, shimming would be a simple matter of optimizing the response (lock level, total area of the FID, length of ringdown of the FID or swept lock signal etc.). Newly designed shim sets are closer to this ideal, especially the matrix designs. These newer designs for shims are much easier to adjust and usually don't require the extensive procedures to optimize that the older designs often require. However, no shim set is perfect. Using imperfect shim sets of the newer or older shim designs, the NMR operator utilizes the procedure described in Note 10 or his favorite shimming procedure and ends up with a final result. The question: is this the best result possible? How does the NMR operator decide? One clue can come from lineshape distortions as described in Note 10. If further shimming efforts don't correct the lineshape, the NMR operator needs to be able to decide whether to continue shimming or run the instrument with the current shim values. A more involved procedure might be needed to make this decision. Before proceeding with these more involved measures:

- Make sure the sample is centered in the probe's receiver coil and is 2 to 3 times the length of the receiver coil.
- Make sure the sample does not have a vortex from spinning.
- Make sure the sample is free of particulate matter.

To be able to make the decision to continue shimming or not it can be very helpful to plot a given shim value versus an optimized lock level (or other response). This process can give the NMR operator confidence that the best shim value has really been obtained. The method can be used for any shim but is described for Z^4, a shim for which this approach is often used. The process is to start with a value of Z^4 and optimize, either manually or with computer shimming, the lower order shims Z^1, Z^2 and Z^3. Then record the response level and shim value on a graph. Next move Z^4 to a new value and optimize Z^1, Z^2 and Z^3 again. Record the data point on the graph and repeat the process until a graph similar to the one shown is obtained.

The graph should be continuous and describe an optimal value for the shim. Any discontinuity would indicate that the lower order shims have not been properly optimized or the shim and/or shim power supply are malfunctioning. Interpolation can be used to set the shim to a value between the points selected in the graphing process. Use of this process for any shim gives the NMR operator confidence that he has found the best value for that shim. If lineshape distortions are present after this process they are most likely due to probe material susceptibilities and not correctable by further shimming.

The graphing method described appears simple and somewhat tedious. For this reason it is often not done. The NMR operator tries to do something similar in his head. If he takes this shortcut he will not get nearly the same level of confidence that the shims are at the best values possible. If the lineshape and/or resolution is not what it should be, use the graphing method before giving up and blaming the probe or NMR instrument.



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Dipartimento di Scienze Molecolari Agroalimentari

DISMA Via Celoria, 2 I-20133 Milano Tel. 02-2663662/2365029/2362721

September 22nd, 1989
(received 10/4/89)

Prof. Bernard L. Shapiro
966 Elsinore Court, PALO ALTO, California

A QUANTITATIVE APPROACH IN CONFORMATIONAL ANALYSIS BY 2D INVERSION RECOVERY DIFFERENCE SPECTROSCOPY. DETERMINATION OF THE GEOMETRY AT THE GLYCOSIDIC SITE FOR ADRIAMYCIN AND ANALOGUES.

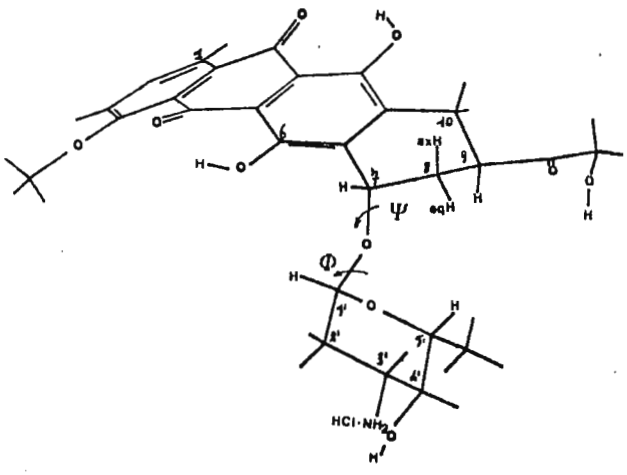
Dear Barry,

2D phase sensitive NOESY spectra are an invaluable tool in the structure determination of molecules in solution. It has been shown how, starting from a model geometry, it is possible to calculate the complete relaxation matrix Γ[1], under a suitable motional model, and then fit the molecular geometry by directly comparing experimental and calculated 2D NOESY diagonal- and cross-peak intensities.

Recently, difference 2D NOESY techniques were devised, in order to minimize t_1-noise and interference with weak cross-peaks of the more intense diagonal peaks. The Inversion Recovery Difference method (IRD)[2] requires the coaddition of the following pulse sequences:

I	$90(+x)-t_1-90(+x)-t_{mix}-90(+x)$	(x2 ADD)
II	$90(+x)-t_1-90(+x)-90(+x)$	(SUBSTRACT)
III	$180(+x)-t_{mix}-90(+x)-t_1-90(+x)-90(+x)$	(ADD)

The experiment leads, after Fourier Trasform, to a 2D NOESY spectrum, where the diagonal-peak intesities are the opposite of the sum of all cross-peak intensities within each row. Ratios of cross- with their own diagonal-peaks in the difference NOE spectrum provide quantities, which are relatively independent onto a wide range of experimental mixing time and onto the correlation time.



We have applied this methodology to the conformational study of Adriamycin 1, Daunomycin 2 and 9-deoxydoxorubicin 3. We were interested in the preferred orientation of the sugar moiety with respect to the aglycone, as measured by the torsional angles Φ =H(1')-C(1')-O(7)-C(7) and Ψ =C(1')-O(7)-C(7)-H(7).

Steady-state and transient NOEs of these compounds in aqueous solution display strong negative peaks and extensive spin-diffusion. This indicates the existence of non-extreme narrowing conditions ($\omega\tau_c \gg 1$) and can be explained by the great tendency of this class of compounds to aggregate, forming high molecular weight species.

The cross-peaks in the 2D NOESY difference spectrum, that correlate H1' with H7 and H5' with H8eq, H8ax and H9 for 3 , were used for the determination of the torsional

angles. Model geometries were built with standard bond lengths and valence angles and were energy minimized with Allinger's MMPI molecular mechanics program. The conformations of ring A and of the sugar were driven to a half-chair $^{9}H_{8}$ and chair $^{1'}C_{4'}$ conformations, respectively, in agreement with experimental coupling constants.

The complete relaxation matrix was constructed, as function of Φ and Ψ torsional angles, by calculating the relaxation rates from eq. 1 and 2, assuming isotropic motions, and changing systematically Φ and Ψ on the model geometry. The molecular τ_c was determined by minimizing the r.m.s. error between experimental and calculated 2D-NOESY values involving protons at fixed distances; and the validity of the assumption for isotropic motions was checked by ^{13}C relaxation time measurements.

1) $$\sigma_{ij} = \frac{\gamma_H^4 \hbar^2}{10\, r_{ij}^6}\left[\frac{6\,\tau_c}{1+(\omega_i+\omega_j)^2\tau_c^2} - \frac{\tau_c}{1+(\omega_i-\omega_j)^2\tau_c^2}\right]$$

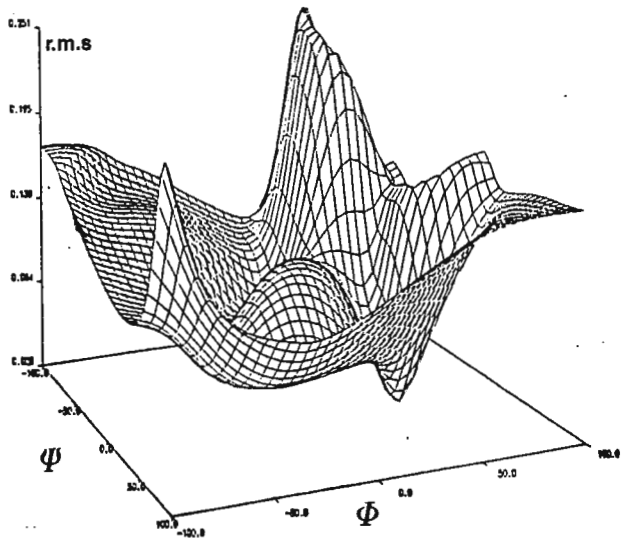
2) $$\rho_{ij} = \frac{\gamma_H^4 \hbar^2}{10\, r_{ij}^6}\left[\frac{3\,\tau_c}{1+\omega_i^2\tau_c^2} + \frac{6\,\tau_c}{1+(\omega_i+\omega_j)^2\tau_c^2} + \frac{\tau_c}{1+(\omega_i-\omega_j)^2\tau_c^2}\right]$$

The complete relaxation matrix analysis of NOESY data takes into account the marked spin-diffusion and therefore minimizes errors, likely to occur when treating data with a two-spin approximation. The 2D NOESY intensities were then calculated by means of eq. 3) and 4).

3) $$[D] = [T]^{-1}[\Gamma][T]$$

4) $$[A] = [T]\,[\exp(-D\,t_{mix})]\,[T]^{-1}$$

The calculated IRD cross-peak intensities, relative to the diagonal, I_{ij}, were compared with the experimental ones. 9-deoxydoxorubicin **3** shows in figure a global minimum of the error, between calculated and experimental intensities I_{ij}, centered at $\Psi = 18\pm10°$ and $\Phi = 48\pm10°$. In the case of **1** and **2** we obtained $\Psi = 15\pm15°$, $\Phi = 60\pm15°$ and $\Psi = [-30, 10°]$, $\Phi = 50\pm10°$, respectively.



1) J.W. Keepers and T.L. James, J. Magn. Reson. **57**, 404 (1984).
2) G. Bodenhausen and R.R. Ernst, Mol. Phys. **47**, 319 (1982).

Sincerely yours

R. Mondelli E. Ragg C. Ulbricht

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Control	VIVOMOUSE™ Control Device With Assignable Knob
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Digital Interface	Controls 2 Synthesizers: Frequency, Amplitude, And Phase For 2 RF Channels, 4 Gradient Channels, All With 12 Bit Resolution. 14 Bit A/D At 100 KHz, Audio Filter 51.2 KHz In 200 Hz Steps. Local Memory Buffer 64 KByte. External Gating Input.

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Gradient Coils	2 Gauss/cm Minimum
Rise Time (10 - 90 %)	1 msec Maximum (Uncompensated)
Power Supply	TECHRON Model 7570

HETERONUCLEAR DECOUPLER

Frequency	5 - 300 MHz
Synthesizer	PTS-500, Computer Controlled
Frequency Step	0.1 Hz
Phase Control	0 - 360 Degrees
Phase, Discrete Steps	0, 90, 180, 270 Degrees
Phase, Variable Steps	0.1 Degree Resolution
Output Power	Computer Controlled: 12 Bit DAC
Power Control	0 - 100 %
Linearity of Power Control	±1%
RF Amplifier Power Output	100W CW

STANDARD MAGNET CONFIGURATIONS

Field (T)/Bore (cm)	2/31	2/30.5	2.4/40	4.7/20	4.7/31	4.7/40	7/20
Bore With RT Shims And Gradients (cm)	26.5	26	33	15	26.5	31	15
Helium Evaporation (ml/hr)	50	50	50	50	55	60	50
Nitrogen Evaporation (ml/hr)	400	400	400	400	450	500	400
Half Length (mm)	350	275	570	396	460	735	575
5 Gauss Line — Radial From Center (m)	3.4	3.2	4.7	3.7	5.3	6.4	5.1
— Axial From Center (m)	4.4	4.0	5.9	4.7	6.7	8.1	6.4

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0426-44-4951

THE UNIVERSITY OF UTAH

David M. Grant
Distinguished Professor

3 October 1989
(received 10/12/89)

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

RE: "^{13}C Chemical Shielding of Metal-Diene Complexes"

Dear Barry:

As has long been known, upon complexation to a transition metal the isotropic chemical shift of olefinic carbons show a large upfield shift. The difference between the chemical shift in the free olefin and the complex, called the coordination shift, is a function of the metal, the olefin, any additional ligands, and has been explained by properties of all three. We have been studying how the individual components of the shielding tensor change upon metal complexation. One grouping that we have looked at is 1,5-cyclooctadiene (COD) bonded to Rh, Ir and Pd. The spectra obtained along with the principal values for the olefinic carbon tensor are shown in the attached figure.

The basic trend expected is seen, namely upfield shifts of all components with the larger changes seen in σ_{11} and σ_{22} since they are the most effected by changes in the π system. However, without any determination of the orientation of the shielding tensor it is difficult to reach final conclusions about the interaction. Two extremes in the tensorial lineshape can be described in terms of the amount of interaction between the transition metal and the olefin. In the case of a weak interaction, both the orientation and the components should be only slightly perturbed from those seen in typical olefins. On the other end of the scale, a strong interaction would result in a "metallacyclopropane" type structure, which would give an orientation and components closer to those seen in cyclopropane. In this case, a large upfield shift could be seen in σ_{33}. To aid in the analysis efforts are underway to perform *ab initio* calculations of shielding tensors for these types of complexes. The calculated orientations should be very useful in the determination of the extent and type of interaction between the transition metal and the olefinic bond.

This work was done in conjunction with Dr. Gregory M. Wallraff (currently at IBM, San Jose) and Prof. Josef Michl, at the University of Texas.

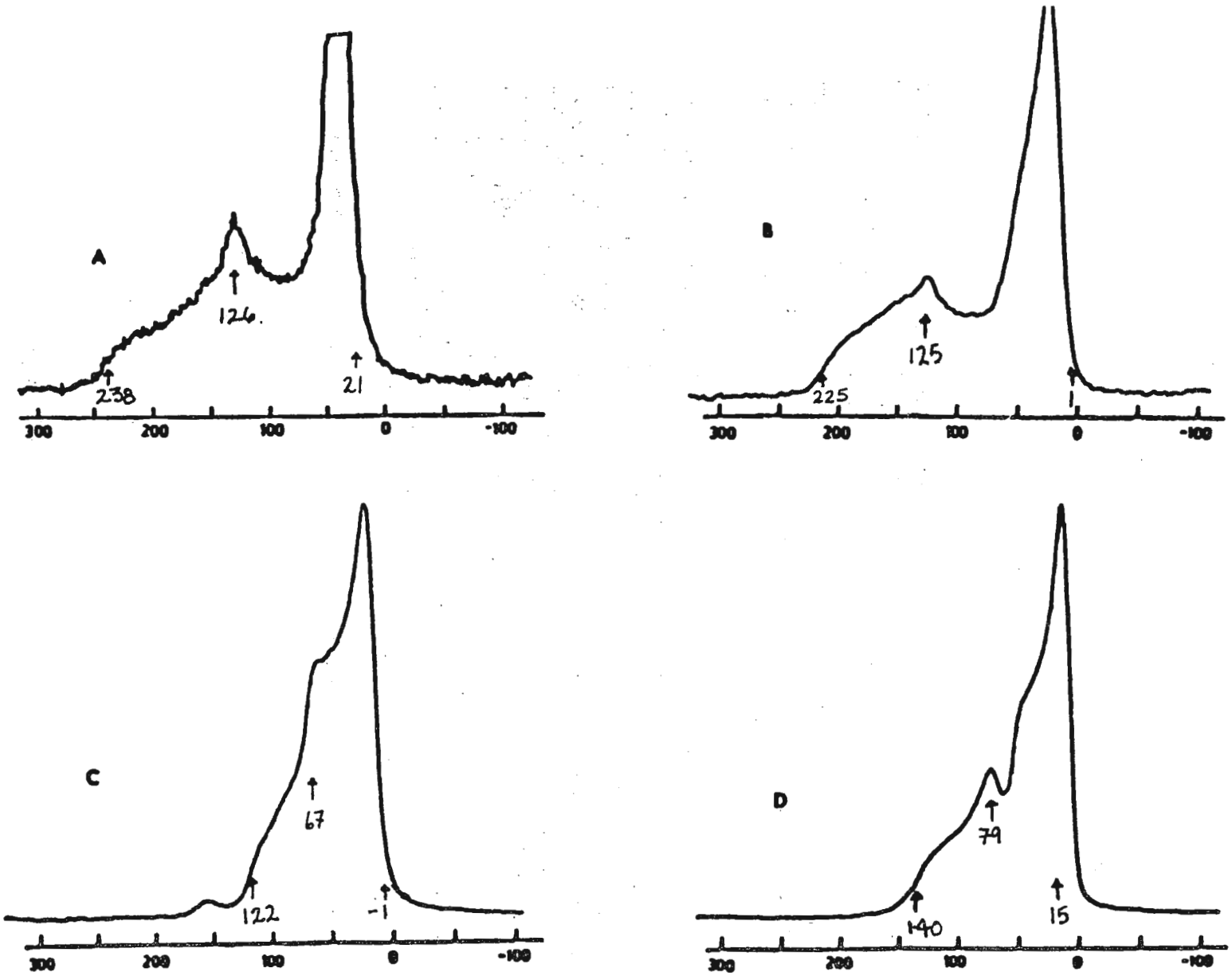
Sincerely yours,

Anita M. Orendt

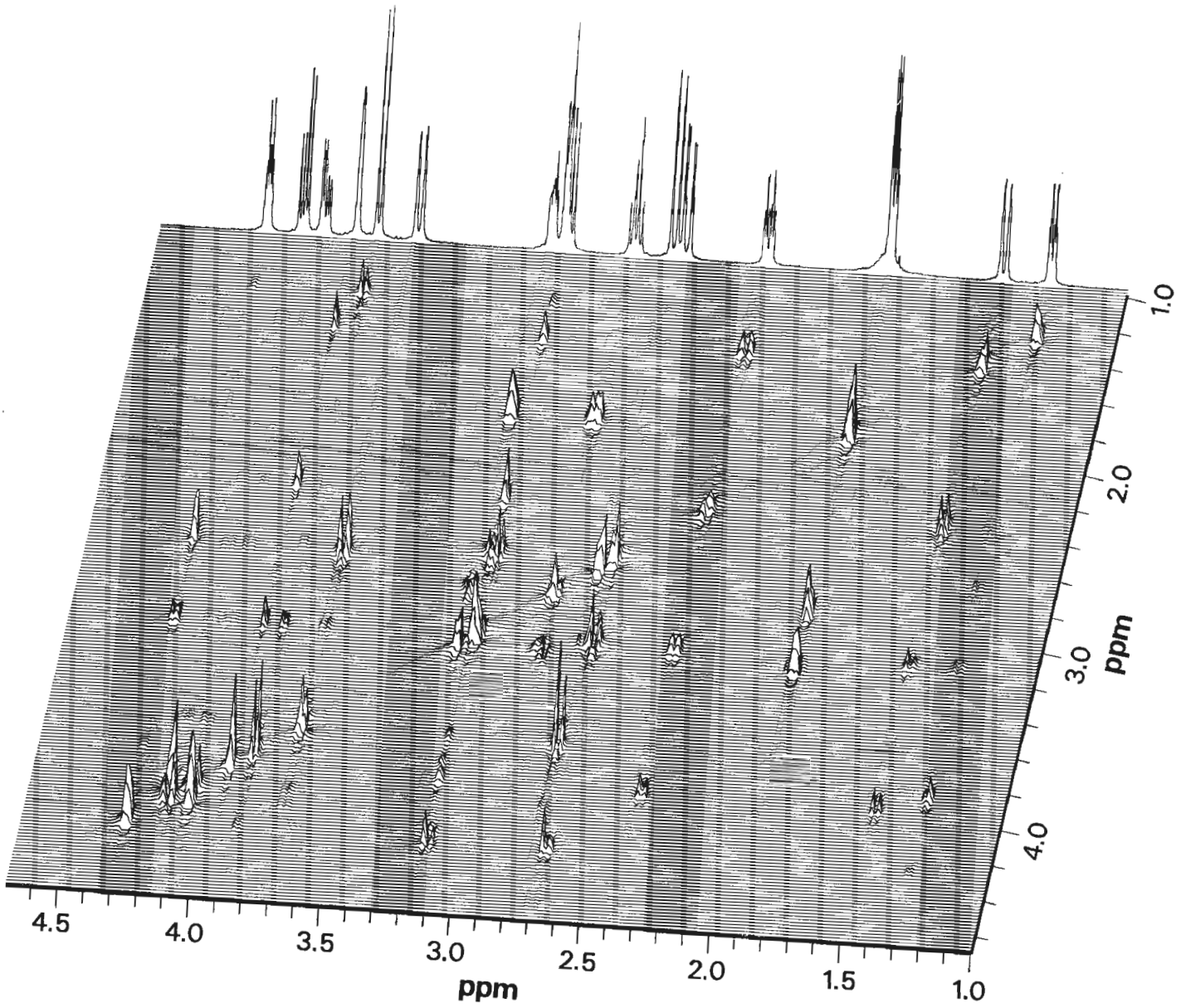
David M. Grant

DMG:lt

Department of Chemistry
Henry Eyring Building
Salt Lake City, Utah 84112
(801) 581-8854



Static solid state ^{13}C spectra of A) COD, B) $[PdCl(COD)]_2$, C) $[IrCL(COD)]_2$ and D) $[RhCL(COD)]_2$. Principal values for the olefinic carbon are given on the spectra.



HOHAHA of Strychnine on an Omega 600

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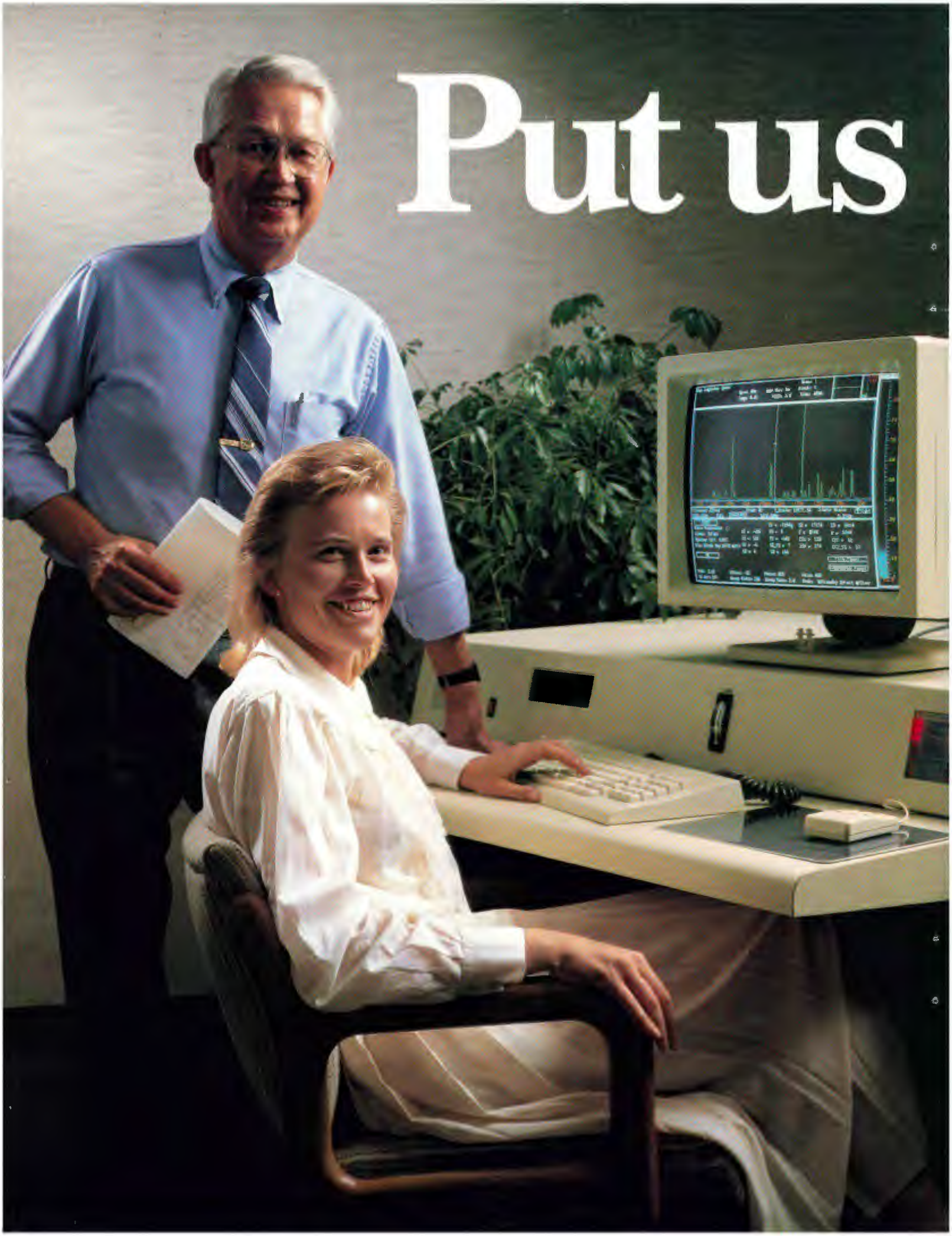
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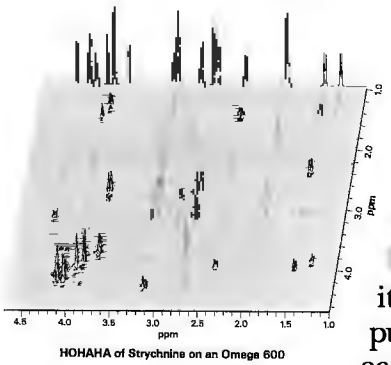

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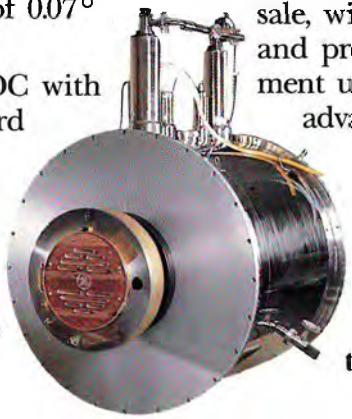

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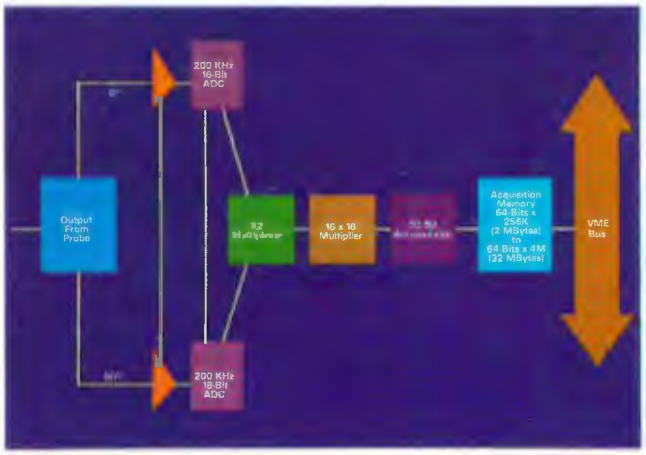

Fig. 1

The Alpha HDR digitizer.

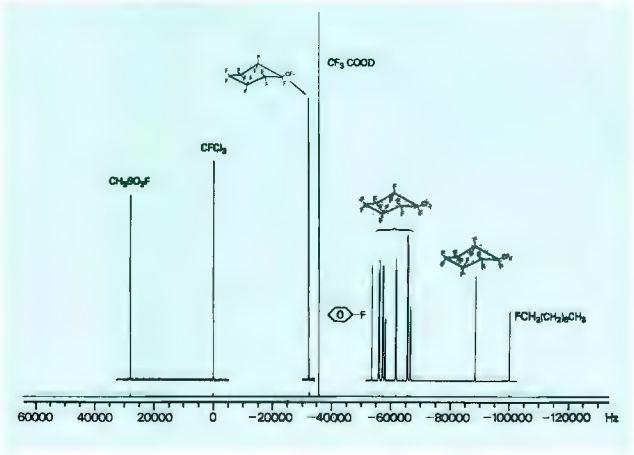

Fig. 2

200 KHz spectral width ^{19}F spectrum acquired on a GN-500 Omega System. Note the extremely flat baseline obtained with the Alpha HDR.

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September 29, 1989
(received 10/5/89)

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

Dear Barry:

Progress Reports

I thought it might be interesting to give some updates on several solid state NMR projects, previously reported:

Superconductors: Linda Reven, Shengtian Yang, Jay Shore and Todd Duncan have been obtaining VT shift, T_1 and T_2 results on most classes of oxide superconductors. All show Korringa-like behavior in their normal states, and preliminary results indicate BCS behavior in T_c vs $1/\lambda$ plots, in the lower T_c systems. For the cuprates, there are additional effects, perhaps not inconsistent with various spin-fluctuation results. No coherence peaks for anything yet. Does anyone believe in coherence peaks in the oxides (including the authors of the three published accounts)?

Brains: Cindy Husted, Ben Montez and Xi Shan have now obtained ^{1}H and ^{13}C solution and solid state spectra of just about all lipids, which has permitted assignment of all the ~ 65 resonances in the spectra of human myelin. With the exception of C^{ζ} of arg (basic protein), only the lipids are seen. Good quantitation has been obtained, using only decoupled Bloch decays. There are clear changes in PS/CER level with brain development, but spectra from different species are, basically, all over the place. Currently emphasis in moving towards detailed dynamics studies of normal and abnormal myelins.

Proteins: Foluso Adebodun, H. C. Lee, Chris Coretsopoulos and K. D. Park have obtained reasonable ^{17}O spectra now of several proteins -including hemoglobin and myoglobin (there have been attempts at this for ~20 years - success at last). We find spectra very similar to those of $^{17}O_2$ in picket fence porphyrin, at low T, indicating somewhat ozone-like bonding, but with, we guess, "bigger orbitals" (smaller QCC).

Zeolites: K. Guo and K. Park have been pushing forward with H. K. Timken's ^{17}O work on zeolites, using a more multinuclear approach. T_1/T plots show interesting differentiation can be made between say (A,X,Y) and ZSM-5 zeolites, possibly due to hydration effects. So far, it seems there are remarkable mappings of T_1/T behavior for the different nuclei (^{17}O, ^{23}Na and ^{27}Al) in that T_1 minima appear at the same temperature, but the absolute magnitudes are all very different, with ^{17}O being the longest. Presumably, water is playing an important role in modulating the quadrupolar interaction, as noted for ^{27}Al previously by others.

Yours sincerely,

Eric Oldfield
Professor of Chemistry

EO:kk

374-16

PROFESSOR DAVID M. DODDRELL

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DEPARTMENT OF CHEMISTRY

UNIVERSITY OF QUEENSLAND

4th October 1989 (received 10/13/89)

Professor B.L. Shapiro,
Editor/Publisher,
TAMU NMR Newsletter,
966 Elsinore Court,
Palo Alto, CA 94303,
U.S.A.

Dear Barry,

Slice Selection with a 2π Selective Pulse

Much has been written about selective self-refocusing pulses - these are rf pulses which have negligible phase roll across their excitation bandwidth. An instrumentally stable self-refocusing pulse has use in high-resolution NMR as well as for slice selection in magnetic resonance imaging and *in vivo* spectroscopy.

To determine the slice selection characteristics of such a pulse, an incorrect approach has been to solve the Bloch equations at discrete frequency points and examine the excitation profile for phase coherency. The problem is that a discrete calculation does not model correctly the continuous distribution of frequencies generated by a field gradient (1).

As one extreme example of this breakdown, note the discretely calculated profiles (Fig. 1) obtained by solving the Bloch equations following the pulse sequence $2\pi(+y) - \tau$ - Acquire, where $2\pi(+y)$ is a 2 cycle sinc pulse of phase y having length 2.048 msec and τ = 280 μsec. The profiles for $Sx(\Delta\omega)$ and $Sy(\Delta\omega)$ would suggest that the slice generated using such a pulse train applied in the presence of a gradient would be weak. The actual slice Fig. 2(A) is very good and "only" 50% reduced compared to that obtained using a π pulse to recall the phase memory (2).

Best wishes.

Yours sincerely,

David

David M. Doddrell

1. J.M. Bulsing, G.J. Galloway, I.M. Brereton and D.M. Doddrell, Magn. Reson. in Med., 5, 478 (1987).

2. S. Fernbach and W.G. Proctor, J. Appl. Phys., 26, 170 (1955).

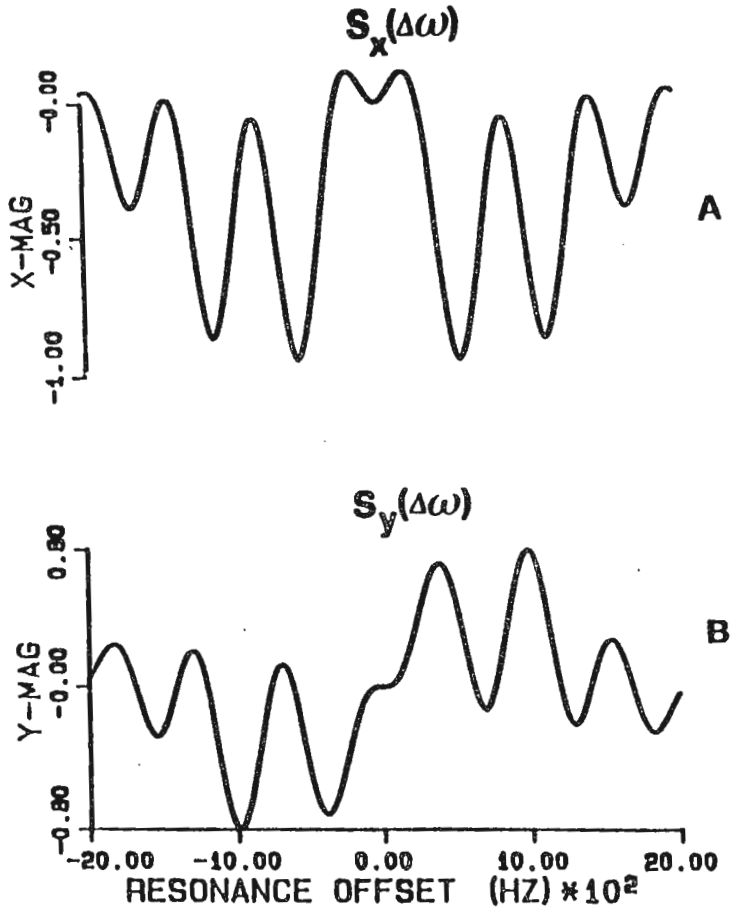

Fig. 1 : Magnetization profiles (A) $S_x(\Delta\omega)$ and (B) $S_y(\Delta\omega)$ calculated for a 2.048 msec 2-cycle sinc pulse. The on-resonance rotation angle was set at 2π.

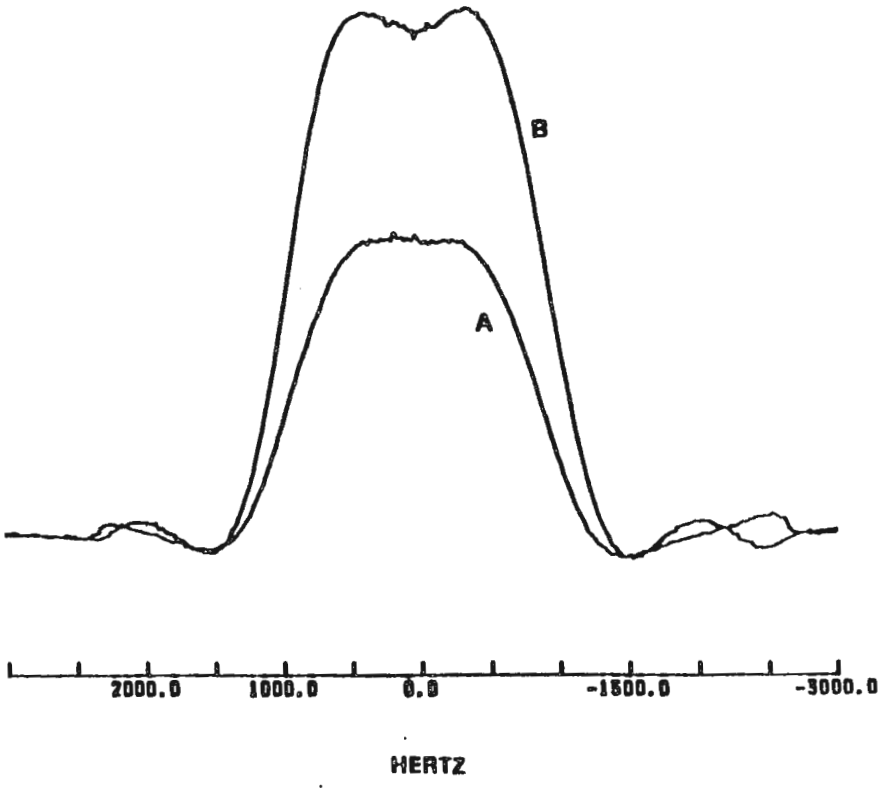

Fig. 2 : Measured slice profile following pulse sequence (A) compared to (B) for a 2.048 msec $\frac{\pi}{2}$ sinc pulse refocussed with a hard π pulse.

374-18

INSTITUT FÜR PHYSIK DER UNIVERSITÄT BASEL
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Prof. Dr. P. Diehl

CH-4056 Basel (Schweiz) Sept.28,1989
(received 10/2/89)

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

Title: Indirect ^{13}C-^{13}C couplings in hexachlorobenzene

Dear Barry,

As a by-product of our studies on the transferability of bond contributions to the degree of order of oriented chlorobenzenes [M. Kellerhals and P. Diehl, Mol.Cryst.Liq. Cryst., to be published] we have lately determined the ^{13}C-^{13}C indirect coupling constants, which, of course, cannot be obtained in the isotropic phase. Their values (measured in the LC-solvent ZLI 1132) are:

$$J^{o}_{cc} = 76.30\ (13)\ \text{Hz}$$

$$J^{m}_{cc} = 6.92\ (11)\ "$$

$$J^{p}_{cc} = 4.26\ (16)\ "$$

The positive value of J^{m}_{cc} is surprising because predicted values [REX NMR, P. Pyykö and L. Wiesenfeld, Mol.Phys. 43, 557(1981)] are consistently negative for various Cl-substituted benzenes.

We have also tried to measure the anisotropies of these J's but had to give up, because the precision was too low. After having corrected the measured direct couplings for harmonic and anharmonic vibration as well as for molecular deformation by the solvent, the residual couplings which must stem from the J-anisotropy are of the order of 1 Hz, whereas the error of the sum of all the corrections will be of a similar magnitude.

With best wishes,

Yours sincerely

(Peter Diehl)

M. Kellerhals
(Martin Kellerhals)

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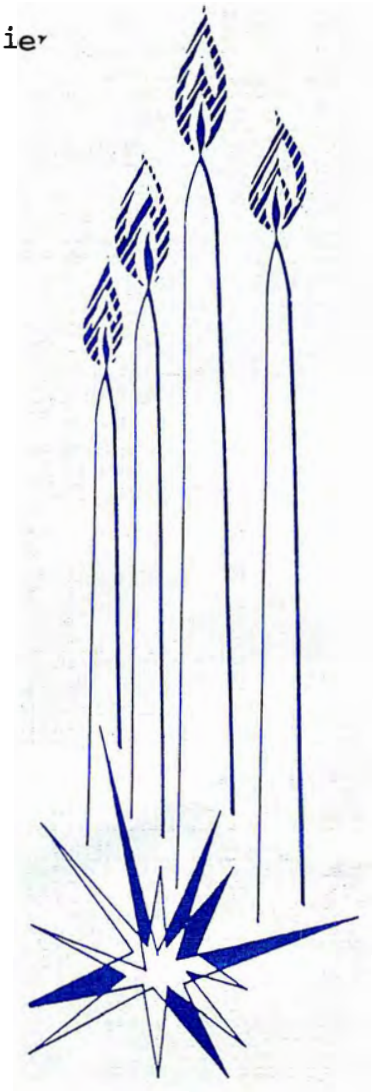

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(415) 952-1366

Re-projecting > 1D Data Sets; an imaging case. October 10, 1989

(received 10/11/89)

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

Dear Dr. Shapiro,

In imaging applications, three dimensional FT acquisition and reconstruction promise to replace the currently popular clinical strategy of performing separate protocols along multiple axes. The total acquisition time for 3D is no longer than 2 of the 2D variety and the growing presence of fast graphics workstations will keep hungry eyes soothed with lightning-fast reconstruction. However, creating views of the images from arbitrary angles seems to imply an interpolation and loss of resolution. I would like to describe a versatile and efficient algorithm for calculating images from generalized oblique planes and curved surfaces that preserves the volume resolution of the original data set. Some images are included of oblique planar slices as well as slices along curved surfaces.

The algorithm is based on the Fourier shift theorem. Simply stated the theorem says that one can produce a shifted data function through a convolution with a delta function that is displaced from the origin by the amount of the desired shift. Let us limit our discussion to shifts in the slice (or z–) direction. That can be used to "reconstruct" images between the original slices in a way that is equivalent to zero-filling the FT in the slice direction. To produce an oblique cut through the volume one can apply a shift that varies linearly with distance (say, along the x–axis). To align image pixels from an arbitrary surface onto a plane one needs only a simple description of the surface. A set of image features of interest can be brought into a single viewing "plane" through the use of a different 1D shift for each ray in the z–direction. The axis to which one applies the shifts is that which is closest to the desired viewing axis.

An example of a reconstruction of a tilted plane is shown in Figure 1. Figures 1a-c are the original axial slices; d-f are the calculated obliques. An example of a curved reconstruction can be seen in Figure 2. The phantom consisted of two annular rings of lexan bridged by empty glass tubes laying along the inside surface of the annular supports. It was placed in a bath of aqueous Mn^{++} on its side with its axis along the imaging z-axis. A coronal 3D image was reconstructed in the usual manner; 3.5 mm thick sections with 0.7 mm resolution in-plane. In Figure 2a are 8 of the 32 conventional planes. In Figure 2b is a slice along a parabolic surface. This reconstructed slice contains nearly all of the tubes.

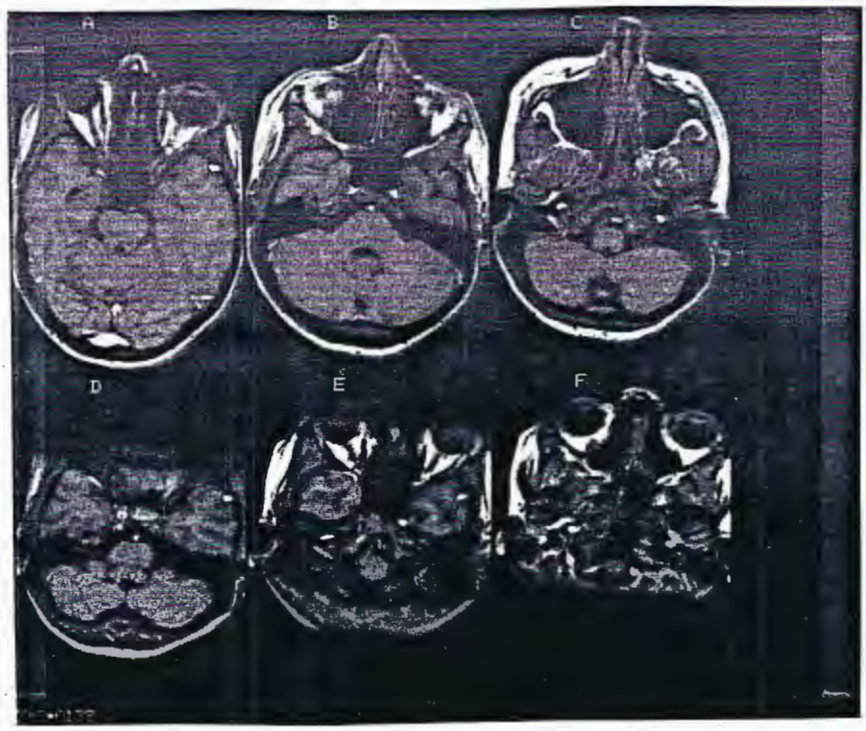
This general obliquing algorithm is easy to program if the argument of the shift convolution is expressed as a polynomial of sufficient order to represent warping of the desired complexity. It can be done efficiently using FFTs for large numbers of planes, and most importantly, it is preserves the resolution of the pixels in the original images. It will not have escaped the attention of your readers that this trick may improve the appearance of off-axis plots from 2D and 3D spectral experiments.

Sincerely,

David M Kramer

David M. Kramer

(a-c) Standard axial planes (2mm thick) through the head of a normal volunteer. (d-f) Oblique planes parallel to the ones normally acquired in x-ray CT through the orbits and auditory apparatus. The tilt corresponds to 54 planes over the field of view in the vertical direction; 5 horizontally. Notice that spatial resolution is maintained.



(a) Standard 3DFT (coronal) images of the phantom described in the letter (b) A parabolic "slice" through the region containing the tubes.

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October 10, 1989 (received 10/12/89)

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

VAPOR FOR LOCALIZED PROTON SPECTROSCOPY IN HUMANS

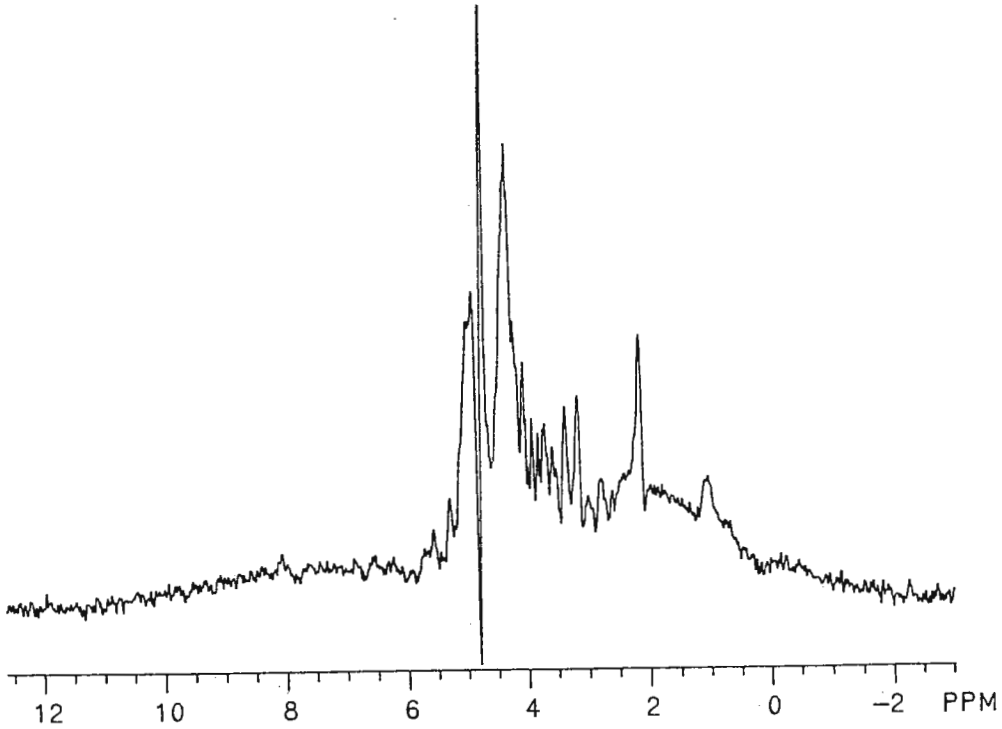
Dear Dr. Shapiro:

After years of effort, preliminary results suggest that proton spectroscopy has diagnostic utility in medicine.(1) Progress has been slow, as researchers have investigated how to best solve the problems of having adequate spatial localization, adequate solvent suppression, and worked around problems with generation of multiple quantum coherence, phase problems with first-order proton spectra, etc. The votes seem to be in, and STEAM or PRESS-based sequences are the choice of the majority of investigators for performing localized high-resolution proton spectroscopy in humans.

We find that combining a technique for solvent suppression published by Doddrell et al. several years ago (SUBMERGE, ref. 2) with a short-echo STEAM-type sequence gives a superior sequence, dubbed **VAPOR.**(3) The **VAPOR** technique is a "rocket", and can be used by MRI technicians to acquire localized proton NMR spectra in patients. Our clinical technicians can achieve solvent suppression factors of >800 routinely. Technician adjustment of the magnetic field homogeneity usually gives water linewidths of 8-12 Hz over an 8-16 cc voxel. The entire sequence of events requires 15-25 minutes, and makes single voxel proton spectroscopy an "add-on" exam comparable in length to other MRI procedures such as GdDTPA contrast-enhanced exams. We have incorporated **VAPOR** as an optional add-on component to our clinical exams of the brain at CNID.

An example of a **VAPOR** spectrum from an 8 cc voxel located in parietal white matter is shown below. The repetition time was 1.5 sec, and the echo time was 10 msec. A total of 128 averages were summed, treated with 0.5 Hz of exponential broadening, and Fourier transformed. No baseline correction was applied. The usual resonances are present between 1.0 and 4.0 ppm. Two "new" features appear at the 10 msec echo time compared to longer echo times. First, a broad hump is centered around 1.2 ppm in all patients studied to date. In addition, a more intense resonance. possibly from mobile glycerol head groups is observed at 4.2 ppm. Our preliminary studies of MS, stroke, and tumor

have shown that variations in the relative intensities of these resonances may also be of diagnostic utility.



Sincerely,

Richard H. Griffey
CNID

Duane Flamig
Department of Radiology
Baylor University Medical
Center, Dallas TX

1. H. Bruhn, J. Frahm, et al. Radiology 1989; 172:541-548.
2. D. Doddrell, G. Galloway, et al., J. Magn. Reson. 1986; 70:176.
3. R. Griffey and D. Flamig, J. Magn. Reson. 1989; in press.

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TAMU NMR Newsletter,
966 Elsinore Court,
Palo Alto, CA 94303, U. S. A.

Dept. of Molecular Physics,
Agricultural University,
Wageningen 6703 HA,
The Netherlands
05-10-1989 (received 10/16/89)

Actual and measured quadrupole splittings

Dear Dr. Shapiro,

By using specifically deuterated phospholipids we have been studying the interactions of various phospholipid types in multilamellar bilayers with the coat protein of the bacteriophage M13 by deuterium NMR. From the recorded powder spectra the residual quadrupolar anisotropy, as measured from the quadrupole splitting, can be determined. However the inhomogeneous linebroadening of the resonances that make up the powder spectrum causes the measured quadrupole splitting to be smaller than the actual quadrupole splitting. This can be shown by computer simulations (Fig. 1) where with increasing linebroadening the measured quadrupole splitting eventually collapses to zero and the spectrum loses the appearance of a typical powder pattern. For these simulations a relative linebroadening σ is used with the inhomogeneous spin-spin relaxation time T_2^* being given by $1/\sigma\,\Delta\nu_Q$ where $\Delta\nu_Q$ is the actual quadrupole splitting. This effect of the linewidth can pose a problem as in many lipid protein systems including the M13 system the presence of protein in a lipid bilayer causes an increase in the linewidth and hence a decrease in the measured quadrupole splitting. To determine whether a decrease in the quadrupole splitting in the presence of protein is due solely to linebroadening effects or actually reflects a true decrease in the order of the system the actual quadrupole splitting must be extracted from each spectrum. One approach is the procedure of depaking, a numerical technique developed by Myer Bloom and his group in Vancouver[1,2]. This technique calculates the orientated spectrum from the powder spectrum from which the quadrupole splitting can also be measured. The quadrupole splittings from both the simulated and depaked spectra (Fig. 1) are listed in Table 1. For values of σ up to a value of 1 the depaked spectra give a quadrupole splitting very close to that used in the simulations. However there is an artifact in the depaked spectra at twice the resonance frequency of the orientated component. At values of $\sigma > 1$, this artifact interferes with the deconvoluted signal due to its linewidth, giving a splitting greater than that actually used in the simulations. These artifactual depaked spectra also correspond to spectra in which the measured splitting is zero.

Acknowledgements

We thank Myer Bloom at the University of British Columbia, Vancouver for providing us with the depaking program.

T.W. Poile

Trevor W. Poile

Johan C. Sanders

Marcus A. Hemminga

1 M. Bloom, J. H. Davis & A. L. Mackay, Chem. Phys. Lett. **80** 198 (1981)
2 E. Sternin, M. Bloom & A. L. Mackay, J. Magn. Reson. **55** 274 (1983)

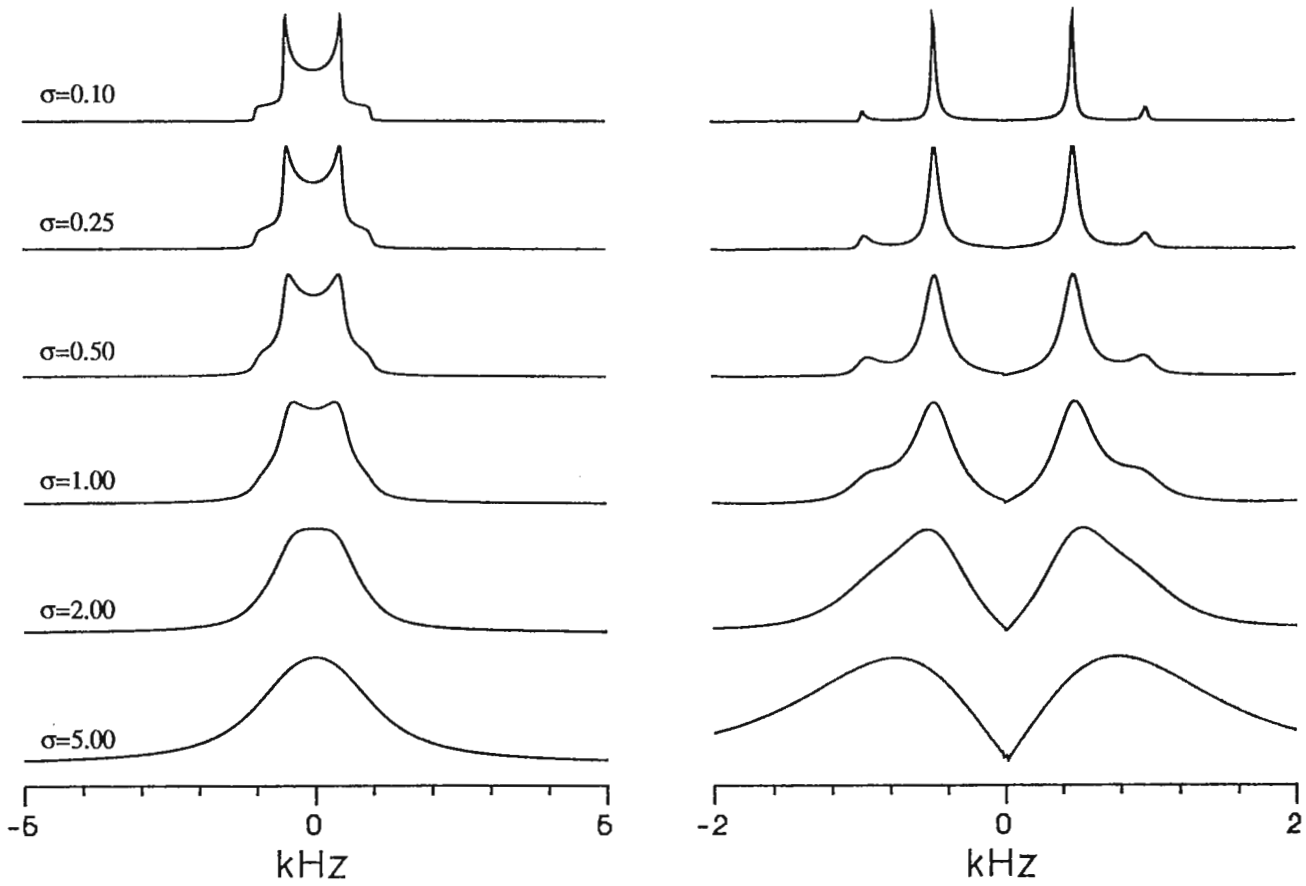

Fig 1. Computer simulations of deuterium powder spectra using Lorentzian linebroadening and various linewidths σ (left) together with the oriented spectra as calculated by the procedure of depaking (right).

Table 1. The measured quadrupole splittings from the simulations ($\Delta\nu_{Q,measured}$), those obtained from the oriented spectra ($\Delta\nu_{Q,depaked}$) and the relative linebroadening used in the simulations (σ). The actual quadrupole splitting used in the simulations was 1.

σ	$\Delta\nu_{Q,measured}$	$\Delta\nu_{Q,depaked}$
0.10	0.95	0.97
0.25	0.91	0.97
0.50	0.85	0.97
1.00	0.71	0.97
2.00	0.00	1.09
5.00	0.00	1.55

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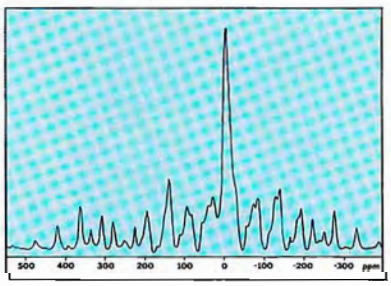

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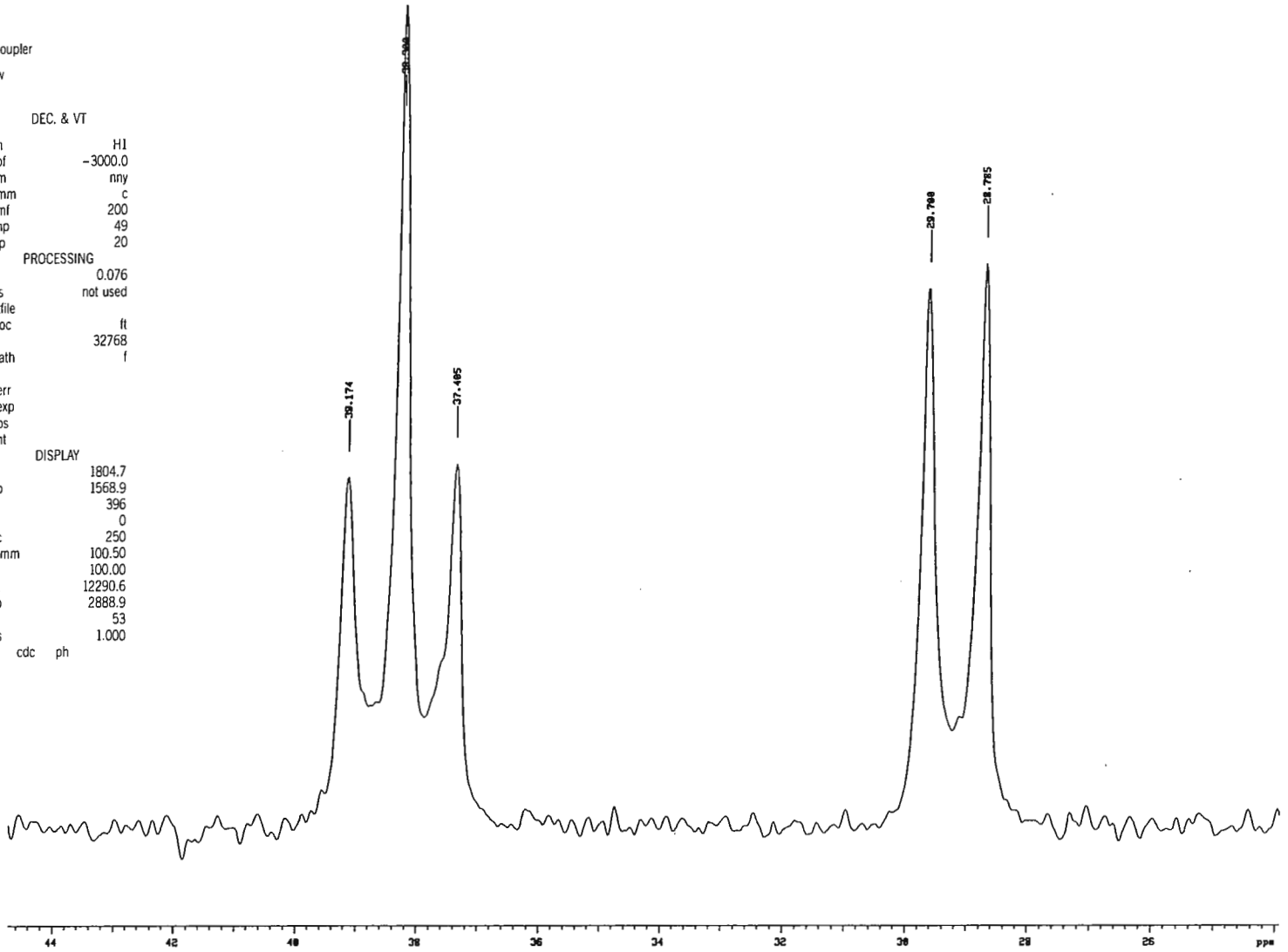
Normally the high power decoupling used to remove dipolar coupling also removes J_{CH} coupling. On a UNITY NMR Spectrometer, this can be retained while still removing the dipolar coupling by using one of a number of multiple pulse methods. In this example, a semi-windowless MREV8 sequence on the decoupler, with acquisition of each point occuring in a 2τ window, gives a J coupled ^{13}C CP/MAS spectrum. Note that J is scaled as predicted for MREV8.

Unity-300
CPMAS C13
Adamantane
J coupled, using mrev-8 on decoupler

exp6 pulse sequence: xpoljnew

SAMPLE		DEC. & VT	
date	Aug 30 89	dn	H1
file /usr/usr2~		dof	-3000.0
/vnmr1/j_resolved_~		dm	nny
adamantane/adam_mr~		dmm	c
	ev_1.fid	dmf	200
ACQUISITION		dhp	49
sfrq	75.429	dlp	20
tn	C13	PROCESSING	
at	0.199	gf	0.076
np	9984	gfs	not used
sw	25125.6	wtfile	
fb	13900	proc	ft
bs	256	fn	32768
tpwr	51	math	f
pw	4.8		
p2	5000.0	werr	
d1	15.000	wexp	
xpol	y	wbs	
srate	2007	wnt	
loss	n	DISPLAY	
pdp	n	sp	1804.7
tau	3.400	wp	1568.9
pp	3.275	vs	396
level 1	55	sc	0
level 1f	100	wc	250
level 2	63	hzmm	100.50
level 2f	4095	is	100.00
tof	-3393.7	rf1	12290.6
nt	1	rfp	2888.9
ct	1	th	53
a lock	n	ins	1.000
gain	20	ai cdc ph	
FLAGS			
il	n		
in	n		
dp	y		
hs	nn		



MAG-5185/296

UNIVERSITY OF CAMBRIDGE
DEPARTMENT OF CHEMISTRY
Lensfield Road
Cambridge CB2 1EP

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

20 September 89
(received 10/4/89)

"Saint-Exupéry Rides Again"

Dear Barry,

There are several experiments in two- and three-dimensional NMR where a "top-hat" excitation function (Fig. 1) would be useful. But as Saint-Exupéry (1) pointed out, a top-hat is not always quite what it seems (Fig. 2), and we may need more fitting parameters (2).

Fig. 1 

Fig. 2 

So Helen Geen in my group set out to design an amplitude-modulated pulse shape that would excite a uniform absorption mode signal across a restricted band of frequencies with essentially zero excitation outside the prescribed band. She also set the constraint that dispersion-mode signals should be minimized at all frequencies. The pulse shape was defined as a Fourier series

$$A_0 + \sum_{n=1}^{m}[A_n\cos(n\omega t) + B_n\sin(n\omega t)].$$

and we found that the series could be safely truncated at m=8. Frequency-domain excitation patterns were calculated through the Bloch equations, neglecting relaxation. Of course if we start with a simple pulse shape, the excitation profile is not quite what we want, so an iterative optimization program is needed. Helen chose to use simulated annealing (3) since it holds out the promise of achieving a *global* solution in the presence of many *local* minima. We were pleasantly surprised by the results, although it does gobble up computer time. No room here to show you the shape; send cheque or money order for complete set of Fourier coefficients. Tested experimentally, it can pick out individual spin multiplets from a reasonably crowded high resolution spectrum (Figure 3). The band-selective pulse was implemented as a DANTE sequence with modulated flip angles. Where the flip angles get close to zero, we used a forward/backward pair, a trick we learned from Howard Hill.

Kindest regards,

Ray Freeman

(1) A. de Saint-Exupéry, *Le Petit Prince*, Gallimard 1946.
("*Un serpent boa qui digère un elephant*")
(2) *Mit vier parameteren kann man ein elephant beschreiben.*
(3) Metropolis *et al. J. Chem. Phys.* **21**, 1087 (1953).

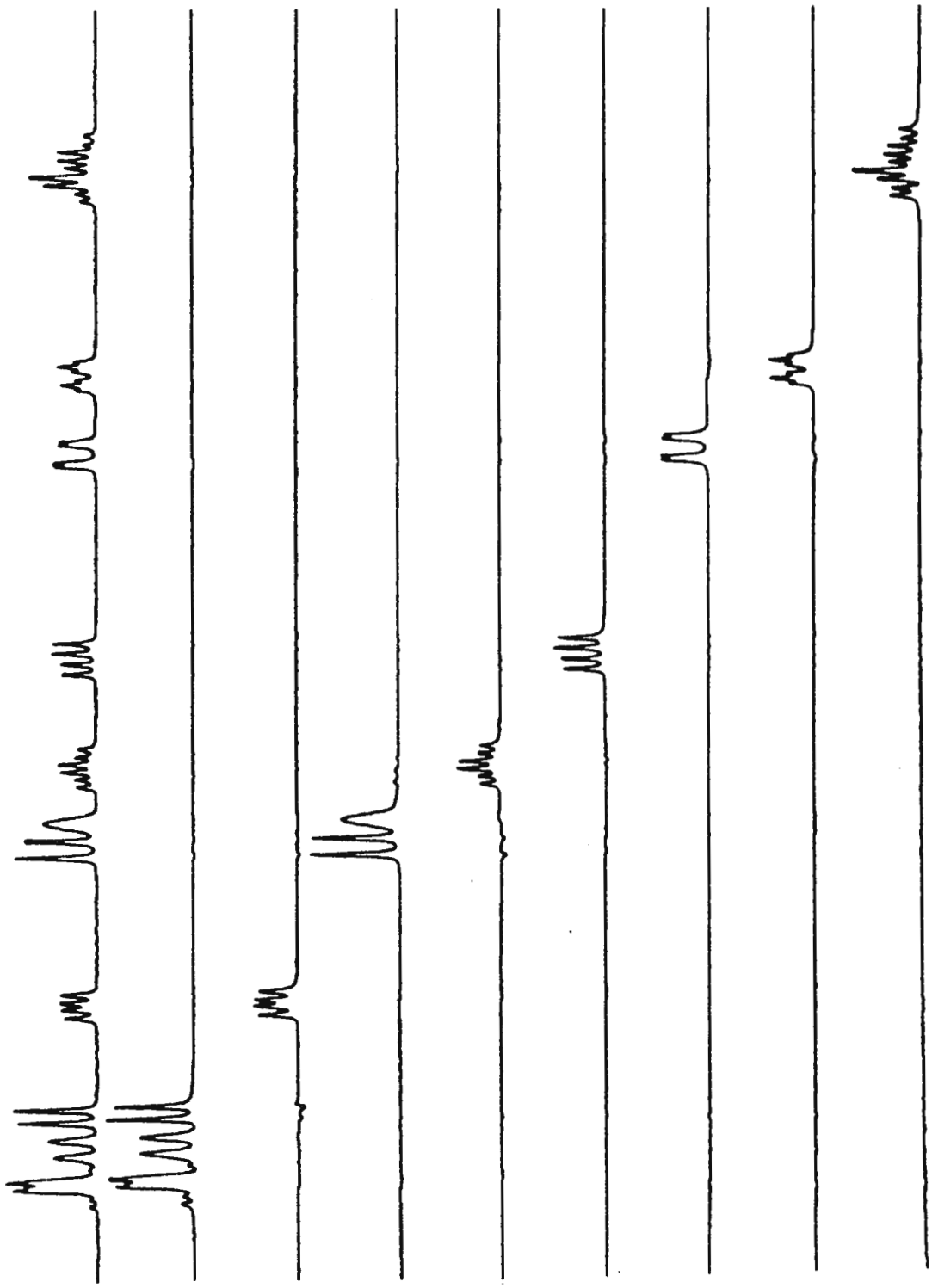


Fig. 3. 400 MHz spectra of an unidentified sample found in a closet. Individual spin multiplets excited by a "top-hat" band selective pulse. No phase corrections applied after the conventional spectrum had been phased.

PHILIPPS-UNIVERSITÄT MARBURG

FACHBEREICH CHEMIE

Prof. Dr. S. Berger

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MARBURG, DEN 29.9.89 (received 10/7/89)
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Prof. Dr. B. L. Shapiro
TAMU-NMR Newsletter
966 Elsinore Court
Palo Alto, Cal. 94303
USA

Rapid Injection and COSY speed up

Dear Barry,

We have been involved in rapid kinetic measurements on our Bruker AM-400. For this purpuse a double syringe unit was constructed according to the description in the literature[1] and a pneumatic unit which drives the syringes. The main trick is the mechanical alignment of the whole system to provide spinning of a 5 mm nmr tube, even though two capillaries from the syringes are ending in the tube. An optical device triggers the computer to start the measurement after the solutions have been injected into the nmr tube. The microprogram listed below has the feature that during the measurement of subsequent spectra no unnecessary time is spent for disk transfer to provide real time attention of the computer for the evolving chemistry within the nmr tube.

```
; RAPID.AUR
; FAST KINETIC PROGRAM
; SET SI=TD !
; W.BOCK/M.OCHS 07.89

1 ST0                    ; SET START ADRESS
7 ST                     ; INREMENT ADRESS POINTER
8 ZE
9 LO TO 7 TIMES L1       ; L1 = NUMBER OF FILES
10 TRIG                  ; WAIT FOR TRIGGER
13 D5 STA0               ; SET START ADRESS, D5 = 0.4 msec
17 AQ
19 D4 STA                ; INCREMENT ADRESS POINTER, D4 = 0.4 msec
22 L1 TO 17 TIMES UPR
23 RCYC=23
24 D2                    ; D2 = 10 msec
```

```
26 ST0                    ; SET START ADRESS FOR STORING
28 WR #1.001
30 IF #1
32 ST                     ; INCREMENT ADRESS POINTER
40 EXIT

;D4 = DELAY BETWEEN SUBSEQUENT RUNS, MINIMUM 0.4 msec
;L1 = NBL = NUMBER OF EXPERIMENTS
;D2 = 10 msec
;D5 = DELAY FOR LOCK STABILIZATION AFTER INJECTION,
;TD MUST BE EQUAL SI!
```

The same principle can be used if one wishes to omit the dummy scans in normal COSY sequences. The time saving for a short "quick and dirty" COSY is rewarding. To save your precious newsletter space we are happy to provide all interested parties with a listing of the program.

Sincerely yours

(Prof. Dr. S. Berger) (Willi Bock)

1) McGarrity, J. F., Ogle, C. A., J. Am. Chem. Soc. **107**, 1805 (1985)

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

October 6, 1989
(received 10/13/89)

Dear Barry,

^{35}Cl NMR Determination of Membrane Potential in Red Blood Cells

A ^{31}P NMR method for measurement of membrane potential in red blood cells has been recently described (1). The method requires the introduction of a probe (hypophosphite ion) and suspension of cells in artificial media. Furthermore, the probe must be present in millimolar concentration and the transmembrane chemical shift is small. Because changes in the membrane potential, E_m, parallel changes in E_{Cl} (2), it should be possible to determine the membrane potential by measuring the intra- and extracellular concentrations of $^{35}Cl^-$. It has previously been shown that the intracellular $^{35}Cl^-$ resonance is very broad and not detectable using small (1000 Hz) spectral widths (3,4). Recently we re-examined the $^{35}Cl^-$ resonance of intact packed red cells in plasma (approx. 99% hematocrit) using a 100 KHz spectral width. We have found that a broad intracellular $^{35}Cl^-$ resonance is detectable using this spectral width, the linewidth at half height being approx. 1130 Hz (T_2^*=280 μs, see upper trace of Fig. 1). The linewidth of the ^{35}Cl resonance from plasma of this blood was approx. 260 Hz (T_2^*=1.2 ms, lower trace of Fig. 1). We were able to quantitate the intracellular and plasma $^{35}Cl^-$ concentrations by comparing the integrated areas of resonances of the above samples with a calibration standard consisting of a plasma sample spiked with 50 mM NaCl. The intracellular chloride concentration $[Cl^-]_{in}$ was calculated to be 74 mM, assuming the intracellular water volume to be 0.7 (2), and the serum chloride concentration $[Cl^-]_{out}$ was calculated to be 110 mM. The membrane potential was determined to be -10.5 mV using the Nernst equation, $E_m = RT/F \ln [Cl^-]_{in}/[Cl^-]_{out}$ (at 37° RT/F = 26.7 mV). This value is similar to that reported for washed human red cells, -9.9 mV (2). ^{35}Cl NMR offers a noninvasive method for studying red cell membrane potential, which may be altered in some disease states, in intact blood.

Yours sincerely,

Linda A. Jelicks Joseph C. Veniero Raj K. Gupta

1. Kirk, K., Kuchel, P. W., and Labotka, R. L. *Biophys. J.* 1988, 54 241-247.
2. Hoffman, J. F. *Chapter 13* in "Physiology of Membrane Disorders", Andreoli, T. E., Hoffman, J. F., Fanestil, D. D., and Schultz, S. G., eds., 1986, Plenum Pub. Corp., NY.
3. Hoffman, D. and Gupta, R. K. *J. Magn. Reson.* 1986, 70 481-483.
4. Brauer, M., Spread, C. Y., Reitmeir, R. A. F., and Sykes, B. D. *J. Biol. Chem. 1985, 260 11643-11650.*

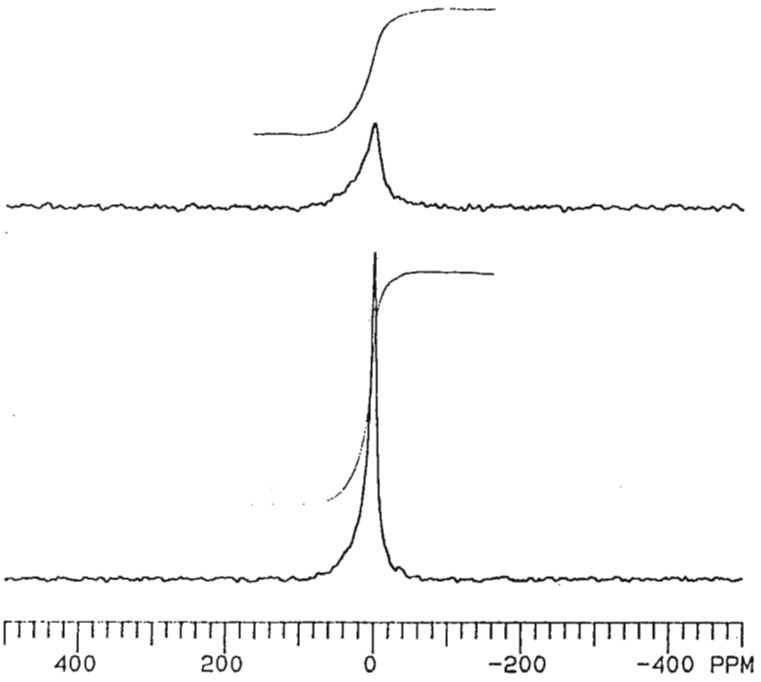
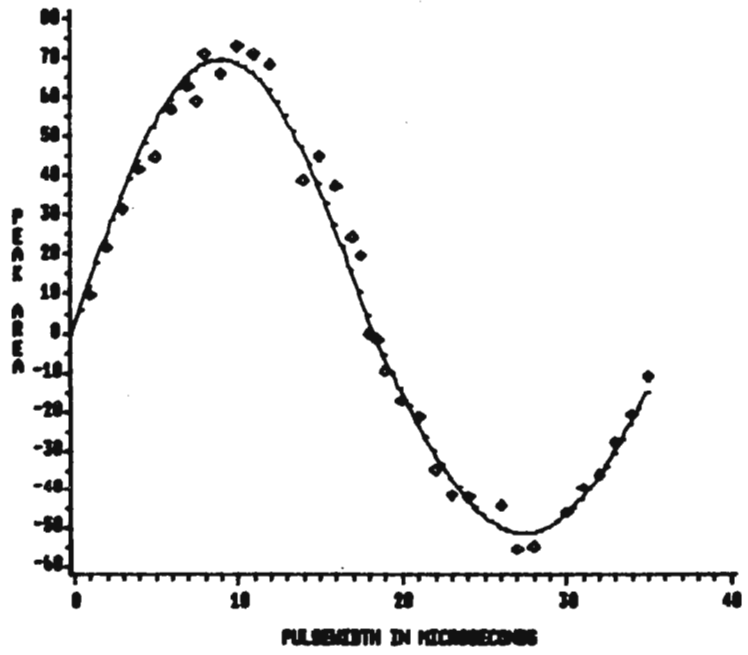


Figure 1: ^{35}Cl NMR spectra of packed human red cells (upper trace) and plasma (lower trace) acquired at 48.9 MHz. The spectral width was 100 KHz, acquisition time was 0.08 s, 90° pulse width was 55 μs, 10000 transients were signal averaged, and 200 Hz. of line braodening was applied.

- continued from page 45.

PULSEWIDTH DETERMINATION JULY 89



THE UNIVERSITY OF CONNECTICUT

College of Liberal Arts and Sciences
Department of Chemistry
Box U-60, Room 151
215 Glenbrook Road
Storrs, CT 06269-3060

October 12, 1989
(received 10/16/89)

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

Pulsewidth on a Dual-Probehead

Dear Dr. Shapiro,

We have been experiencing problems with proton pulsewidth determinations on a $^{1}H/^{13}C$ Dual-Probehead. We are interested in hearing if some of your readers have encountered the same difficulties.

Our experimental conditions are as follows: The sample used is 50 % $CHCl_3$ in acetone-d_6. The relaxation period between pulses is 150 seconds. At first we determine a 180° pulse according to the procedure recommended by Hull.[1] We then determine peak areas for times up to a 360° pulse. To avoid possible systematic errors the times are scrambled. On these data, we perform a three parameter non-linear regression based on the following equation:

Peak area = (A_1 or A_2) • sin(A_0 • t), where A_1 and A_2 are the amplitudes for the first and second half-period, respectively. The resulting fit is shown in the figure.

The 180° pulsewidth derived from this fit is in excellent agreement with that determined by the procedure recommended by Hull. However, the figure indicates a systematic deviation in the first half-period between experimental data and the estimated fit.

As a consequence, the value for a 45° pulsewidth based on the first half-period alone differs by approximately 20 % from the value based on the full period. We presume this uncertainty to be too large to perform e.g. a COSY-45 experiment in a satisfactory manner. We are interested in hearing if this simply represents the "facts of life" with the Dual-Probehead.

Please credit this contribution to the account of Dr. Thomas K. Leipert.

Best regards,

Timo V. Ovaska

Lyn M. J. Zarcone

[1] William E. Hull, in "Two-Dimensional NMR Spectroscopy. Applications For Chemists and Biochemists". William R. Groasmun and Robert M. K. Carlson, Eds., p 87-88.

374-46

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Lectures focussed on topics for discussion will be presented in the mornings. A tentative programme follows. Afternoons will be free for spontaneous discussions. Panel discussions on topics generated during the morning sessions, as well as poster sessions, will be held in the evenings. Special efforts are being made to facilitate contact and discussion among the participants.

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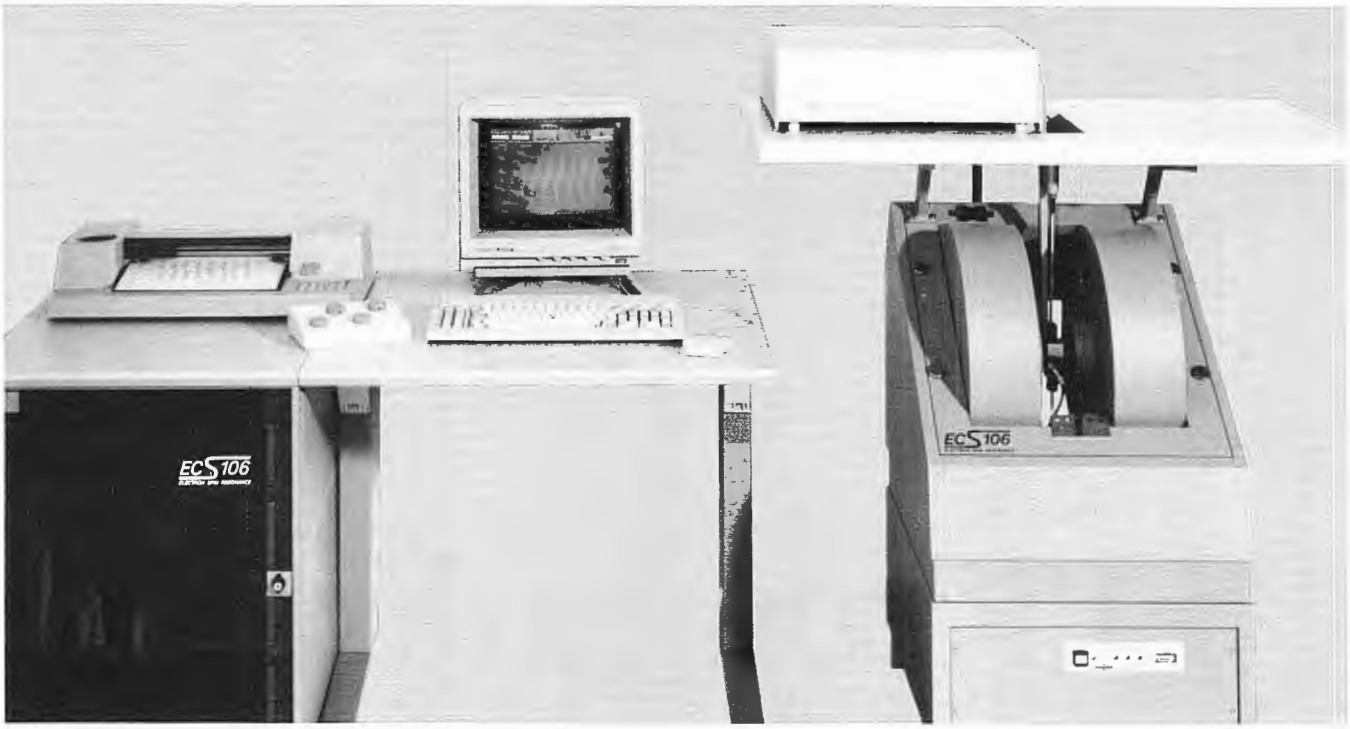

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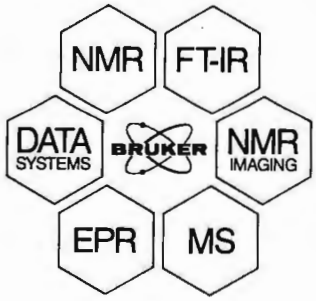

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Dr. B.L. Shapiro
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto, CA 94303

(received 10/19/89)
October 2, 1989

Dear Barry:

Fast 2D and 3D on an AM

Our first work with 3D NMR was done on an old NT-500 spectrometer that has been heavily modified during its 8-year lifetime. Although this spectrometer gave beautiful results (JACS 111, 1515-7, 1989), a major problem was that one had to come in every few hours to change disks. This destroyed the dreams of Dominique Marion and Lewis Kay who first developed 3D NMR in our lab with the main goal of getting the weekend off while letting the spectrometer do its 3D thing. With the arrival of a new Bruker AM-500, we figured it would be a piece of cake to implement the 3D on the new AM and thus solve the disk storage problem. One thing we had not counted on however, since it was no problem on the old NT, was the overhead time required on the Bruker before skipping to the next (t_1,t_2) increment of the 3D experiment. Typically our 3D experiments require only 4-8 scans per (t_1,t_2) increment (5-10 sec) and after this time Bruker takes a 4-15 sec break (depending on the complexity of the sequence). After this break, we would need to waste even more time for regenerating an acceptable steady state (dummy scans). The bottom line was that a weekend experiment required almost a week of measuring on the new instrument, a clearly unacceptable situation.

To solve the overhead problem mentioned above, Rolf Tschudin designed and built an extension to the Bruker process controller (plug-in device) that takes care of the t_1/t_2 incrementation and requires zero overhead. For the past four months this device has functioned very reliably and it has never interfered with the "regular" Bruker mode of doing 2D or 1D. The only disadvantage of the "3D box" is that the number of t_1 and t_2 increments has to be a power of 2 (a Bruker software problem), and that it requires some skill to peek at the data while they are coming in. Using the device, we have been able to record 2D spectra in as little as five minutes (Fig.1), and we're back to recording a 3D experiment per weekend.

As Fig.1 shows, even for 2D the "3D box" can be very useful. For example, to study which amide protons exchange rapidly with solvent, one can record a 2D spectrum immediately after the protein is dissolved in D_2O. In the spectrum of Fig.1, all but ten of the amide protons of BPTI can be observed in the spectrum that is recorded between two and seven minutes after dissolving the protein. The spectrum is of high quality, despite the fact that we used a sample concentration of "only" 4.5 mM and a recording time of 5 minutes.

Kindest regards,

Ad Bax

PS. For those who want to know more about the box, please call Tschudin (301 4962692), not me.

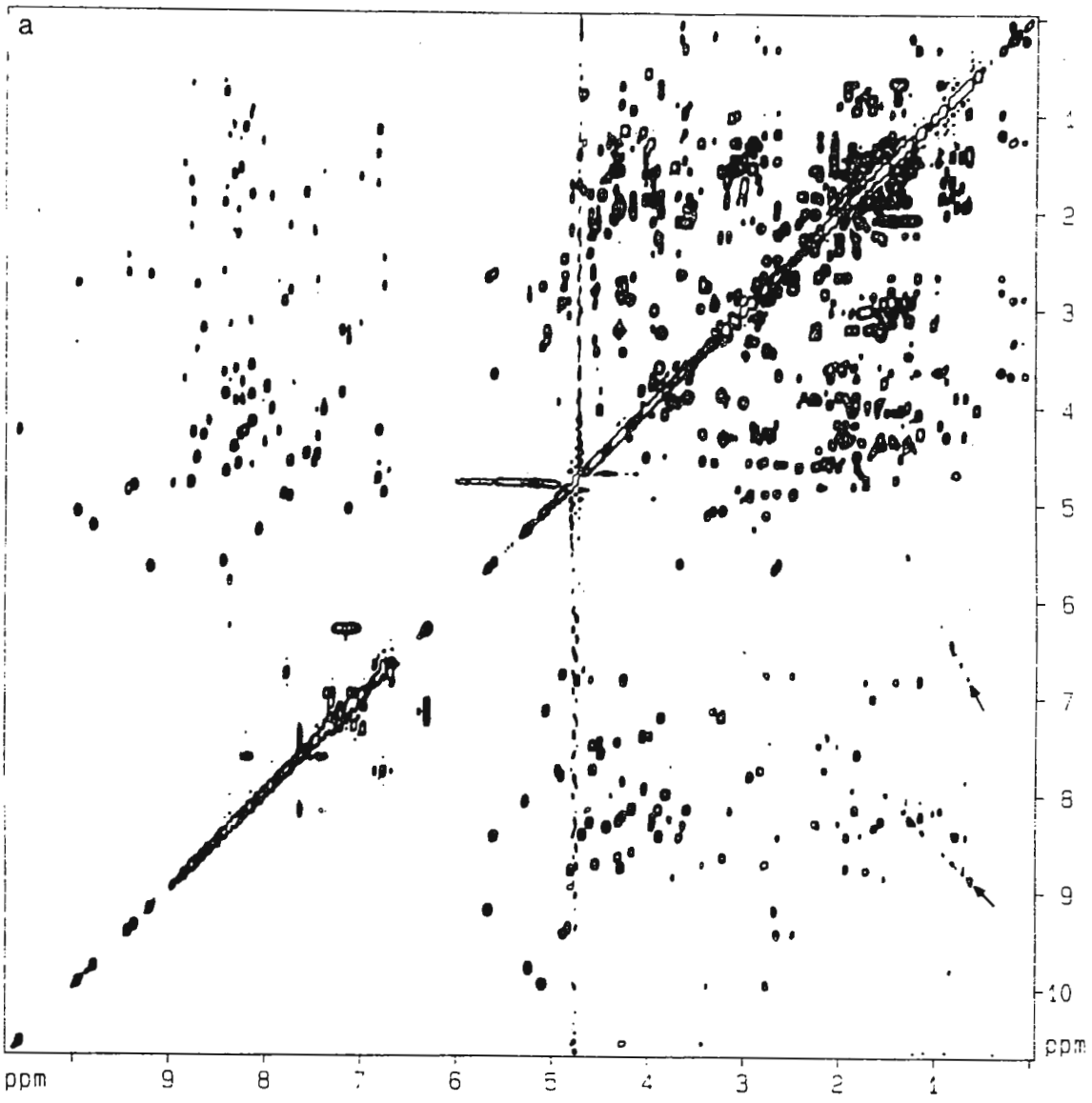

Fig.1. 500 MHz 2D HOHAHA spectrum of 4.5 mM BPTI, freshly dissolved in D_2O, p^2H 3.7, 24°C. The experiment was started 2 minutes after dissolving the sample, and the total experiment time was 5 minutes (1.2 sec per scan). The spectrum results from a 128(complex)x1024(real) data matrix with 2 scans per complex t_1 value. To minimize artifacts in the absence of phase cycling, we used a TPPI-States hybrid method of data acquisition (Marion, Ikura, Tschudin & Bax, JMR in press).

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The Squibb Institute
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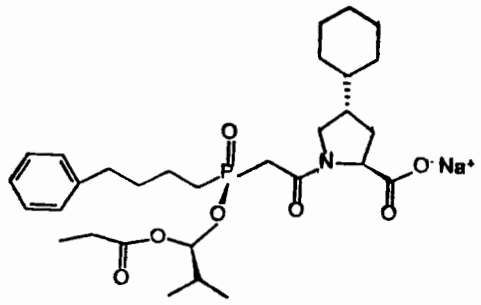
October 17, 1989
(received 10/21/89)

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

Polymorphic Characterization by ^{31}P Solid State NMR

Dear Dr. Shapiro:

In continuing efforts to characterize pharmaceutical polymorphs by solid state NMR, we have recently studied the A and B polymorphs of fosinopril sodium. Fosinopril sodium is a second generation angiotensin converting enzyme inhibitor used in the treatment of hypertension. It has been demonstrated that different polymorphs may have different solubility, bioavailability, chemical stability, and processing characteristics [1]. Therefore, it is necessary to fully characterize polymorphic systems associated with a given drug molecule.



The A/B polymorphic system of fosinopril sodium was hypothesized to be enantiotropic on the basis of the ordering of the melting points, heats of fusion, and the solution mediated transformation of B to A at room temperature [2]. Table I and Figure 1 display the various properties and solid state ^{31}P NMR spectra for each polymorph, respectively. The enantiotropic relationship of the polymorphs was substantiated by the solid state conversion at room temperature from B to A observed by solid state ^{31}P NMR (Figure 2).

Table I

Polymorphic Form	Melting Point (°C)	Heat of Fusion (J/g)
A	199.6	61.2
B	203.4	54.3

Due to the substantial chemical shift difference of the phosphorus resonance for each polymorph, a quantitative method may be developed. The spin-lattice relaxation time for each polymorph was determined by the inversion-recovery pulse sequence. It is interesting to note that the T_1 time for polymorph A, the more thermodynamically stable form, was 126.7 sec. as compared to 5.6 sec. for polymorph B. Further experimentation is planned in order to fully determine the extent of molecular motion within the solid state and its effect upon conversion from one polymorphic form to another at various temperatures.

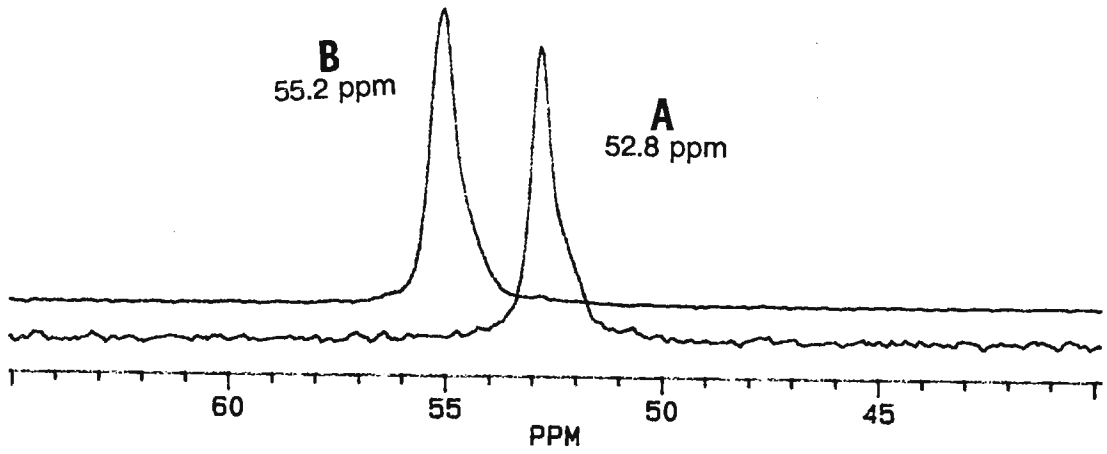


Figure 1. Solid state ^{31}P NMR spectra for A-phase fosinopril sodium (bottom trace) and B-phase fosinopril sodium (upper trace).

Figure 2. Solid state ^{31}P NMR spectra of fosinopril sodium displaying the partial conversion from the B-phase to the A-phase after storage at room temperature and humidity conditions for three months (relative intensity scale).

Such studies, in conjunction with x-ray crystallography, represent a powerful tool for characterizing closely related polymorphs within a given system. In addition, non-intrusive quantification by solid state NMR has the distinct advantage of preserving the pharmaceutical sample which is typically expensive and in extremely short supply within our industry.

Sincerely,

Kenneth R. Morris

David E. Bugay

1. J. Haleblian and W.C. McCrone, *J. Pharm. Sci.*, **58**, 911 (1969).
2. D.E. Bugay, K.R. Morris, A. Serajuddin, H.G. Brittain, A. Thakur, S.J. Bogdanowich, and J. DeVincentis, "Characterization of the Polymorphic System of Fosinopril Sodium and Transformation under Stressed Granulating Conditions", **Fourth National AAPS Mtg.**, 10/22/89-10/26/89, Atlanta, GA, Abstract PD895.

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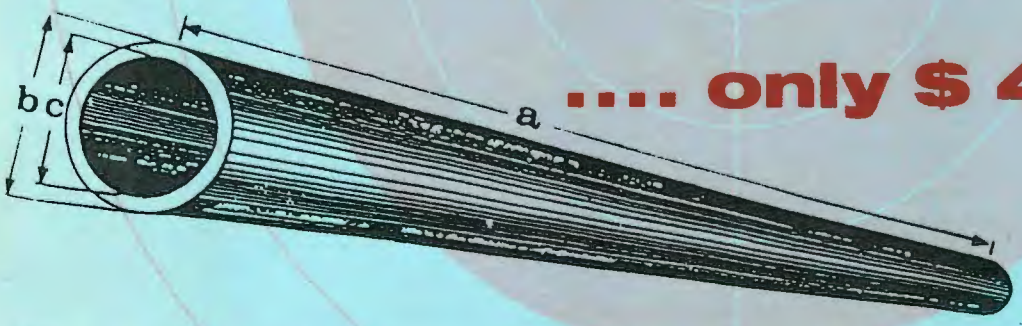

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Barry Shapiro, Ph.D.
TAMU NMR NEWSLETTER
966 Elsimore Ct.
Palo Alto, CA 94303

9/27/89
(received 9/30/89)

^{31}P SPECTROSCOPIC IMAGING OF ISCHEMIC RAT BRAIN

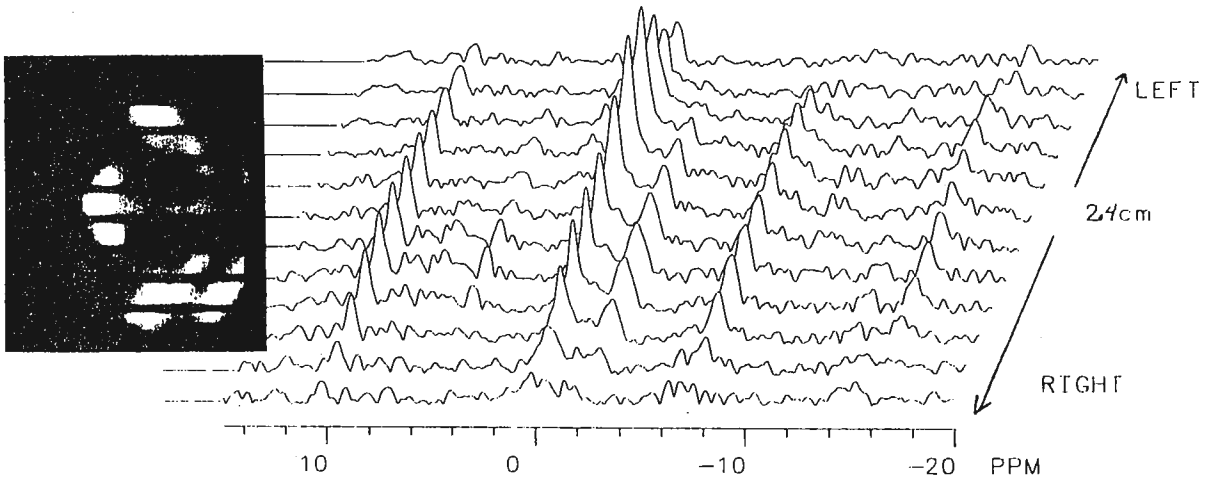
Dear Barry,

We have recently been doing ^{31}P spectroscopic imaging as a means of localizing signals from phosphate metabolites in our experimental model of focal cerebral ischemia. Rats were prepared by occlusion of the right middle cerebral and common carotid arteries which produces infarction in the right cortex. The spectroscopic images were acquired with one spacial dimension from right to left across the transverse axis producing spectra corresponding to contiguous 2 mm (with one zero-fill in the spacial dimension) sagittal slices. A 1.8 cm surface coil was attached to the top of the intact scalp. Along the phase encode axis, above the surface coil, was a 5 mm NMR tube filled with a dilute solution of potassium phosphate which served as an intensity standard. At pH 10 the phosphate signal is about 10 ppm, well outside the typical in vivo chemical shift range. Non-selective composite 90 degree rf pulses were followed by a 2 ms phase encode gradient. Acquisition was preceded by a 1 ms delay to allow eddy currents to die down.

The figure shows one of the spectroscopic images we obtained. Along the left axis is a transverse proton spin echo image in register with the ^{31}P spectroscopic image. The top of the brain is on the right. The brain is divided into the sections from which the corresponding ^{31}P spectra originate. The major peaks observed in the ^{31}P spectra are 1) potassium phosphate (external standard), 10.0 ppm; 2) inorganic phosphate (Pi), ca. 5 ppm; 3) phosphocreatine (PCr), 0 ppm; 4) gamma-ATP, 2.4 ppm; 5) alpha-ATP, 7.3 ppm; 6) beta-ATP, 16 ppm. In these data we observed the expected accumulation of Pi and reduction of PCr in the area of the bright ischemic lesion seen in the right cortex. Still in the early acute stage, we do not observe reduction in the ATP peak intensities. In the same experiment performed at times greater than two hours post-occlusion we observe a return of the PCr/Pi ratio towards normal but a global reduction of phosphate metabolites in the ischemic hemisphere.

Please credit this letter to the account of Leland L. Smith, Ph.D.

Figure. ^{1}H spin echo and ^{31}P spectroscopic images of a rat brain with an ischemic lesion in the right cortex.



Best wishes,

Michael Quast, Ph.D.

Mike Q.

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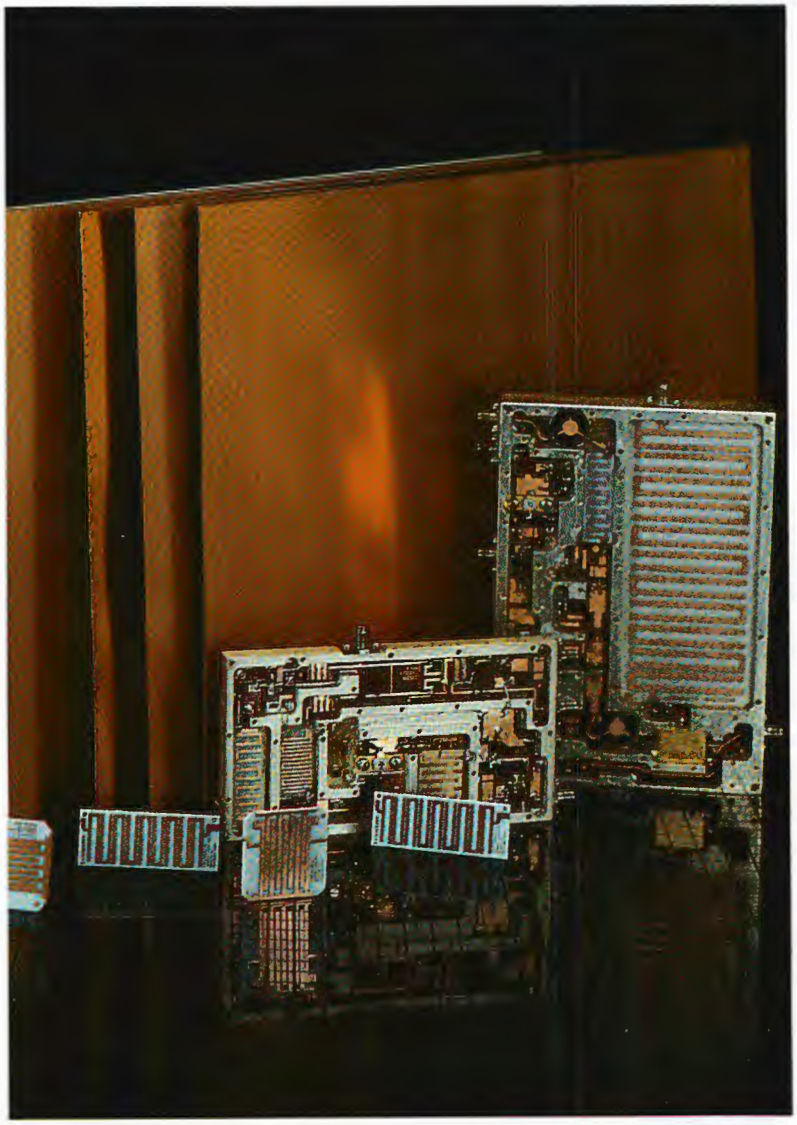

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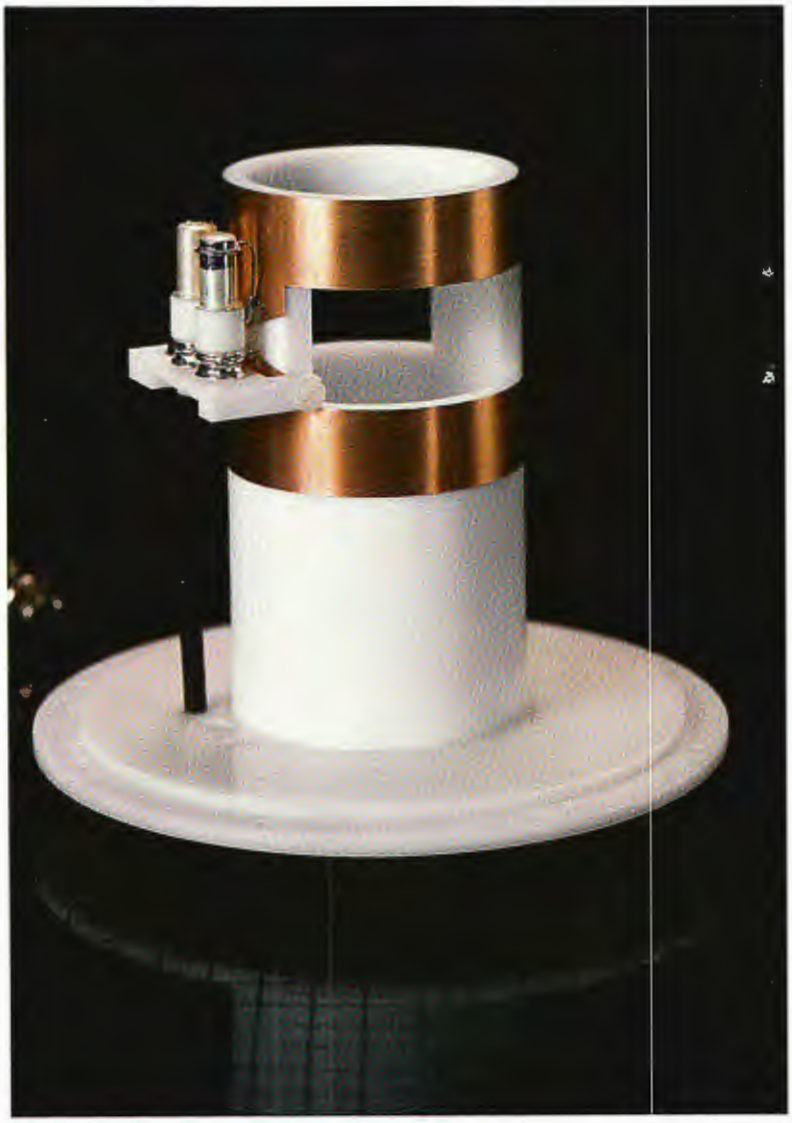

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Prof. B. L. Shapiro
TAMU NMR Newsletter
966 Elsinore Court

Palo Alto, CA 94303
USA

19891004/kt
(received 10/10/89)

NMRSAW

Dear Prof. Shapiro,

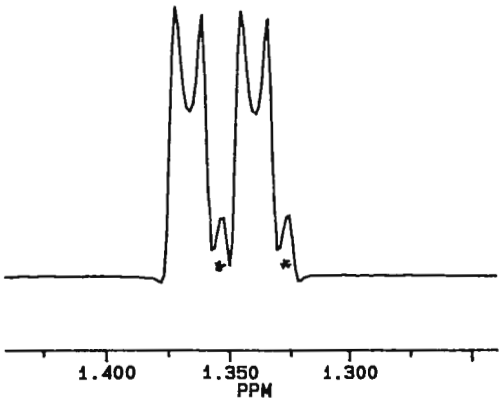
Last year Dr. Lech Kozerski visited Roskilde and we engaged in mapping the behaviour of NMR spectra of small amphiphiles in water (SAW). The idea to this project emerged some years ago in Canada, when we observed that the ^{19}F resonance of o-fluoroanisole in H_2O/D_2O with a little deoxycholate moved (changed chemical shift) in time.[1]

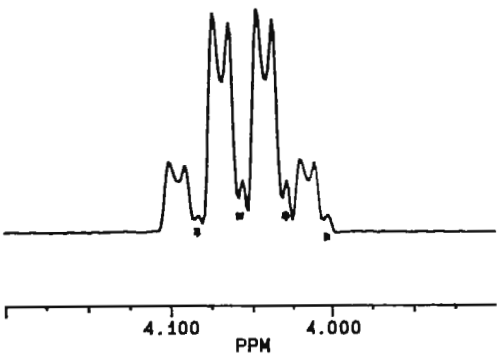
The compounds that we have investigated are α-phenylethylamine (both the - and the ± forms) plus other similar small amphiphiles. The ^1H resonances change position in time, but not very much and long periods, several days, may be needed to reach the final position. The final position is normally to high field of the original one. The ^1H spectras seen in the figures show also some interesting splittings. In the case shown (140 mM) the splittings appear after 24 hrs. At other concentrations the splittings appear immediately. The splittings are identical for the CH_3 and the CH (methine) ^1H resonances. The aromatic region cannot be unravelled. A third species is also observed marked with an asterisk. The splittings could possibly be concentration dependent.

We are continuing these investigations, but would be interested to hear if others have observed similar phenomena or have some good explanations.

1. P.E.Hansen, H.D.Dettman and B.D.Sykes, J.Magn.Reson. **62**, 487 (1985).

Yours sincerely,
Poul Erik Hansen





International Meeting 8.-9.2.1990
25 YEARS OF STRUCTURAL CHEMISTRY IN TURKU UNIVERSITY:
- FROM 1,3-DIOXANE TO NATURAL PRODUCTS -
Kalevi Pihlaja, Prof.

FIRST ANNOUNCEMENT

The Physical and Organic Chemistry Laboratories of Turku University will organize in February 8-9, 1990, an international meeting entitled:

"25 YEARS OF STRUCTURAL CHEMISTRY IN TURKU UNIVERSITY: FROM 1,3-DIOXANE TO NATURAL PRODUCTS".

The meeting will be held in the DataCity Center of Turku. Its scope will be the structure and energetics of organic compounds, including fundamental research as well as its applications to the investigation of natural products and environment, for instance, research on peat, humus and sediments.

The following scientists will give plenary and keynote lectures in the meeting:

Prof. William F. Bailey, USA	Dr. Eino Lappalainen, Finland
Prof. Gabór Bernáth, Unkari	Dr. Bo Nordén, Sweden
Prof. Charles Fuchsman, USA	Prof. Jaakko Paasivirta, Finland
Dr. Ferenc Fülöp, Unkari	Prof. Erkki Rahkamaa, Finland
Dr. György Göndös, Unkari	Prof. Frank Riddell, Scotland
Prof. David Jordan, USA	Prof. Janusz Szafranek, Poland
Prof. Erich Kleinpeter, GDR	Dr. Pirjo Vainiotalo, Finland

Those interested in participation can present short oral contributions (á 15 min including discussion) or posters. The registration fee including lunch and coffee on the meeting days is 60 US$.

The registration starts on Wednesday afternoon, February 7, continuing with a city reception later in the evening. The social program consists of the conference dinner on February 8 and a farewell reception at the end of the meeting.

The second Announcement, including the "final" program and information about accommodations, will be sent to all those who have returned the early registration form appended, preferably before November 15, 1989 to:

Riitta Kremer, Department of Chemistry, University of Turku, SF-20500 Turku, Finland. Tel. 358-21-6335724/Telex 62683 tyf sf/ Fax 358-21-319836

On the behalf of the organizing committee
Chairman, Dr. Harri Lönnberg

Preliminary Registration Form

Name: .....

Address: .....

.....

Tel. .....**Telex.** .....

Fax. .....**(Please print or type legibly)**

Title of the intended presentation.....

.....

.....**I prefer oral/poster presentation.**

Name(s) of accompanying person(s): .....

.....

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Professor Bernard Shapiro
Editor/Publisher, TAMU NMR Newsletter
966 Elsinore Court
Palo Alto, Califoirnia 94303
U. S. A.

October 2, 1989
(received 10/10/89)

Dear Dr. Shapiro:

Determination of Tensorial Orientation of Chemical Shift Anisotropy Interaction by Relaxation Studies of $2I_zS_z$ Spin Order in Solution

We have been re–investigating the relaxation behavior of $2I_zS_z$ spin order to determine the tensorial orientation of chemical shift anisotropy (CSA) interaction for methyl formate ($HCOOCD_3$) in methanol–d_4 solution[1].

The pulse sequence used for measuring the relaxation of the $2I_zS_z$ spin order basically invokes coherence transfer sequence as the initial preparation of the spin system:

$$(\pi/2)^{H}_{x} - 1/(2J_{C-H}) - (\pi/2)^{H}_{\varphi} - t - (\pi/2)^{C}_{y} - (FID)$$

The first two proton 90° pulses are used for generating $^1H \rightarrow ^{13}C$ coherence–transfer and the last 90° pulse of ^{13}C that follows converts it into the signal of two spin order for observation. Difference spectra of ^{13}C with φ=+y and –y are recorded for each value of the evolution time t in the pulse sequence. An anti–phase doublet spectral lines are observed. The difference in the intensity of the two (anti–phase) lines is related to the evolution of $2S_zI_z$ terms.

The description of the relaxation processes during t period may be interpreted due to cross correlation of dipole–dipole interaction (^1H–^{13}C) and CSA interaction of carboxyl carbon[1]. The effect of spin rotation interaction is also corrected in order to determine the tensorial orientation of CSA interaction[2]. The experimental data and theoretical calculations at various temperaturs are shown in the figure. The ordinate scale is related to the intensity of two spin order.

Truly yours,

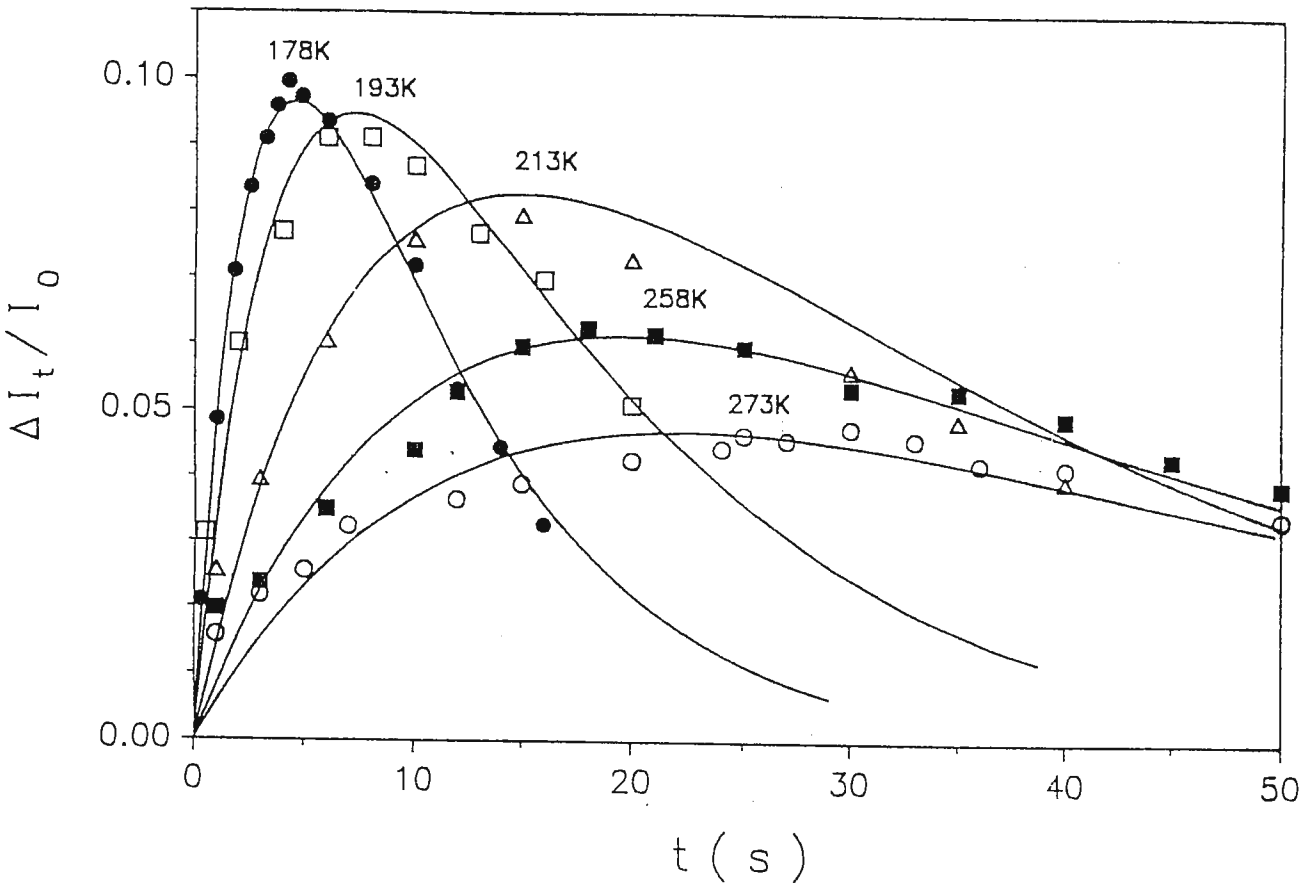
Wen–Tsung Chang Pei–Lein Wang Der–Ming Duh Lian–Pin Hwang

Lian-Pin Hwang

Professor of Chemistry

References:

1. Jaccard, G.; Wimperis, S.; Bodenhausen, G. *Chem. Phys. Letters* **1987**, *138*, 601.
2. Chang, W. T.; Wang, P. L.; Duh, D. M.; Hwang, L. P. *J. Phy. Chem.* (accepted for publication)



SPECTROSCOPIST

Kimberly-Clark Corporation has an immediate opening for a Spectroscopist in our Analytical Laboratory located in an Atlanta suburb. The ideal candidate is a problem solver who will communicate well with other scientists, engineers and plant personnel; plan experiments; interpret spectra; and maintain/operate the instrumentation. The main instrumentation consists of a Bruker AC250 MHz FTNMR with multi-nuclear capability and solids accessory and a Nicolet SX-740 FTIR with IR-PLAN microscope attachment. A B.S. or M.S. with up to five years of experience or Ph.D. in the physical sciences is required.

In addition to a modern facility in beautiful surroundings, K-C also offers an outstanding benefits program and salary commensurate with experience. For immediate consideration, respond in strictest confidence with your resume or letter of qualifications to: **Kimberly-Clark Corporation, Recruiting/MAK/MLD, 1400 Holcomb Bridge Road, Roswell, GA 30076-9701.** An Equal Opportunity Employer M/F.

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Amoco Research Center
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312-420-5111

October 2, 1989 (received 9/29/89)

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

Dear Dr. Shapiro:

New Life for an Aging Spectrometer and Expanded Capabilities for a New One

We recently installed a custom-made 20mm broadband probe in our 1980 generation Nicolet 200 spectrometer. The probe was designed and constructed by Craig Bradley of Cryomagnet Systems, Inc. The probe was purchased to upgrade the signal to noise performance of the spectrometer for routine polymer analysis from the original specifications of 200:1 on ASTM standard sample to a quoted 350:1 in the large 20mm NMR tube.

The probe is designed to be a bottom loading type which required complete replacement of the upper stack and the VT unit of the Nicolet design. Our magnet had already been placed on longer legs to accommodate bottom entry solids probes and therefore was easily compatible with the new design. The performance of the new probe has far exceeded the quoted specifications. We routinely observe between 450 and 475:1 on the ASTM sample, linewidths of 2Hz at the 0.55% level and 4Hz at the 0.11% level of decoupled dioxane, and the 90° pulse width is typically around 23 usec. The probe has been in continuous service for about 6 months with excellent performance at both room temperature and at +130°C. The increased sensitivity has allowed us to reduce the accumulation times and increase the number of samples we are able to process in a normal working day. The improved performance achieved by this probe has generated revised interest in expanding the use of this spectrometer with more new probes and automation equipment rather than completely retiring the spectrometer as a whole.

In the spirit of fair play and stimulating competitive marketing, we are also evaluating a 20mm BB probe designed by Toby Zens of Nalorac Cryogenics Corporation on one of our VXR 300 spectrometers. The evaluation is not yet complete, but initial testing indicates that this probe is performing very well and we expect to achieve at least double the 10mm sensitivity of 300:1 for the ASTM standard sample and reliable performance from this probe. Installation of these valuable upgrades require consultation with the various probe designers and patience to allow the probes to be built to the proper specifications. Our experience suggests that the manufacturers of these items are always willing to discuss your needs and build a suitable system to accommodate your specific requirements.

Sincerely,

R. W. Dunlap

R. W. Dunlap, F-9
708-420-5154

RWD/TAMU89/jh

Department of Chemistry
LOUISIANA STATE UNIVERSITY AND AGRICULTURAL AND MECHANICAL COLLEGE
BATON ROUGE · LOUISIANA · 70803-1804

October 11, 1989 *504/388-3361*
(received 10/14/89)

Dr. B. L. Shapiro, Editor
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto, CA 94393

RE: Dear Santa, I would like an EM360 LX-Si-*Turbo*

Dear Dr. Shapiro:

The Varian EM360 has been a highly popular undergraduate instrument. But it's dated and it's time to start making a new instrument for community colleges, four year colleges, and universities.

I want an NMR **DESIGNED** for use by undergraduates and that really lets students open it up and see how it works. I want the NMR built so that at least these concepts are clearly seen:

- There are rf pulses going into the probe. "See them on the scope?"
- There is a modulated rf signal coming back from the probe. "See it on the scope?" (Can be done with fast digital scope and Larmor frequency of 50 MHz)
- RF mixing to audio frequencies. "Now look at the scope."
- The difference between detection in the laboratory and the rotating frames. (FT both rf and audio signals and compare)
- Data analysis: Perform non-linear least squares and f-test on T_1 data for a multicomponent system without using "canned software packages".
- Solution of eigenvalue/eigenvector problem and on-screen comparison to experimental spectrum using a brief, understandable simulation program.

The NMR must have these features for the teaching laboratory:

- Use no cryogens; no water if possible.
- Has an open rf path.
- Has open instrument control software like LabView on a Macintosh.
- Has a common computer and interface system like a Macintosh with an IEEE-488 bus.

A suggested instrument:

- A permanent magnet or a Walker/Magnion electromagnet.
- An HP digital oscilloscope (54201A, 200 megasample/second, 1000 samples)
- A Macintosh IIx, several National Instruments interface boards, LabView, Matlab, and Mathematica (Statview, CricketGraph, and Excel not allowed)
- A single broadband rf system with multiple pulse capability: 180°-τ-90°, CPMG, and quadrupole nutation.
- A single channel 10-60 MHz probe. VT optional.

Other comments:

- Linewidth isn't so important. The organic labs will either use the department's larger NMRs, or a simple $50,000 GC/MS. Isn't it obvious to everyone that GC/MS is a much more cost effective chemical analysis tool than is an EM360?
- There is considerable discussion regarding the style undergraduate chemical laboratory instruction. Some think it is time to focus on a single advanced instrument (the alternative, an array of modern instruments, seems to be both exceedingly expensive and leads to black-box level of instruction). I think NMR should be the central instrument that illustrates computer interfacing, data analysis, and application of quantum mechanics.

Sincerely

Les Butler
Associate Professor of Inorganic Chemistry

UNIVERSITÀ DEGLI STUDI DI FIRENZE

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Via Gino Capponi 7
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RESEARCH GRANTS IN NMR

A **Bruker AMX600** is being installed in Florence, at the Department of Chemistry, for the Italian community of NMR spectroscopists.

Within the frame of a scientific collaboration between **Bruker** and the **University of Florence** two positions are opened. They aim at the development of applications centered in the field of **paramagnetic macromolecules.**

One position is oriented towards the instrumental developments. The salary is **24.000.000 It. Lire for the first year.**

Another position is oriented towards the development of the potentialities of the NMR techniques in the chemical investigation. The salary is **18.000.000 It. Lire for the first year.**

The positions are renewable for a second year. High power MSL200 and CXP90 are available in the same lab.

Candidated are required to possess a title corresponding to at least four years of University level education in the areas of Physics, Chemistry or Engineering.

Contact: Prof. Ivano Bertini
Department of Chemistry
University of Florence
Via G. Capponi 7 – 50121 Florence
Italy

Phone: (003955)2757549; Telex: 570123 CHIMFI I
Fax: (003955)244102; Bitnet: BERTINI AT IFICHIM

TEXAS A&M UNIVERSITY
NMR
NEWSLETTER

No. 374
November 1989

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* * * Help ! Help ! Help ! **Help !** **Help !** * *

Mailing Label Adornment: Is Your Dot Red ?

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

Page Length Request

Attention overseas subscribers: If you must use paper which is longer that 11", *please* take care that nothing appears below 10" (25.5 cm) on your pages. It is costly to make reductions. Thank you.

* * **Thank you !** **Thank you !** Thank you ! * *

CSI 2T Applications

Shielded Gradients: Theory and Design

NMR imaging and localized spectroscopy depend on the use of pulsed magnetic field gradients. As these techniques have grown more complex, it has become apparent that eddy currents created in the magnet cryostat and other structures by pulsed gradients have become the chief limitation to many sophisticated applications.

Figure 1a illustrates the design problem for unshielded gradients. Figure 1b illustrates the shielded gradient arrangement. Figures 2 and 3 show the contours of constant flux for an unshielded and shielded Z gradient coil, respectively. This demonstrates that, for the shielded gradients, most of the flux has been kept away from the magnet bore.

The dramatic reduction of eddy currents which can be made over the conventional, unshielded gradients is shown in Figures 4a, b. These graphs show frequency as a function of time following the application of a long, constant amplitude gradient pulse which is suddenly cut off. Soon after cut off, a 90° pulse is applied and the complex FID recorded. The instantaneous frequency is then obtained from the FID and normalized by dividing by the frequency offset at the sample during the gradient pulse.

Figure 4a shows a typical decay of extra magnetic fields in a CSI 2T instrument caused by eddy currents in the conventional, unshielded gradient set with compression.

Figure 4b shows the decay of the uncompensated shielded Z gradient and Figure 4c shows the Z gradient decay with compensation. Note that the time scale for 4b and 4c is five times shorter than that for the unshielded gradients.

Fig. 1a.

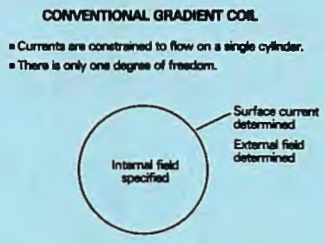


Fig. 1b.

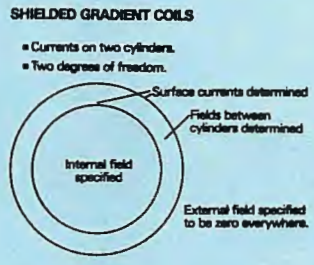


Fig. 1a—Design problem for unshielded gradients. The field inside the winding is specified to be a linear gradient and the current pattern on the cylinder is determined. ***Fig. 1b****—Design arrangement for shielded gradients. The field inside the inner cylinder is specified to be a linear gradient and the field beyond the outer cylinder is specified to be close to zero. The current patterns on both inner and outer cylinders are then determined.*

Fig. 2.

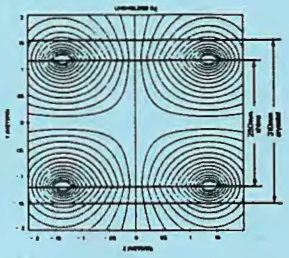
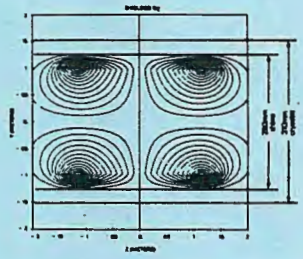


Fig. 3.



Lines of constant flux for Z-gradient. ***Fig. 2****—Unshielded gradient. Note that flux lines extend well beyond the cryostat bore.* ***Fig. 3****—Shielded gradient. Flux lines are kept within the outer gradient cylinder.*

Fig. 4a.

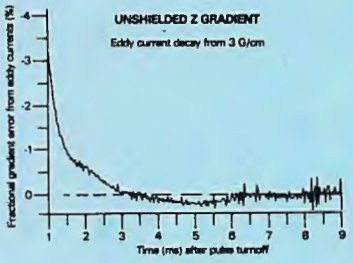
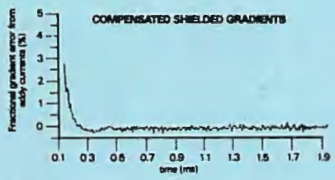


Fig. 4b.

Fig. 4c.



Decay of field following application of square gradient pulse. ***Fig. 4a****—Unshielded gradients.* ***Fig. 4b****—Shielded S150 gradient with no waveform compensation.* ***Fig. 4c****—Shielded S150 gradient with waveform compensation.*

GE NMR Instruments

VPLX

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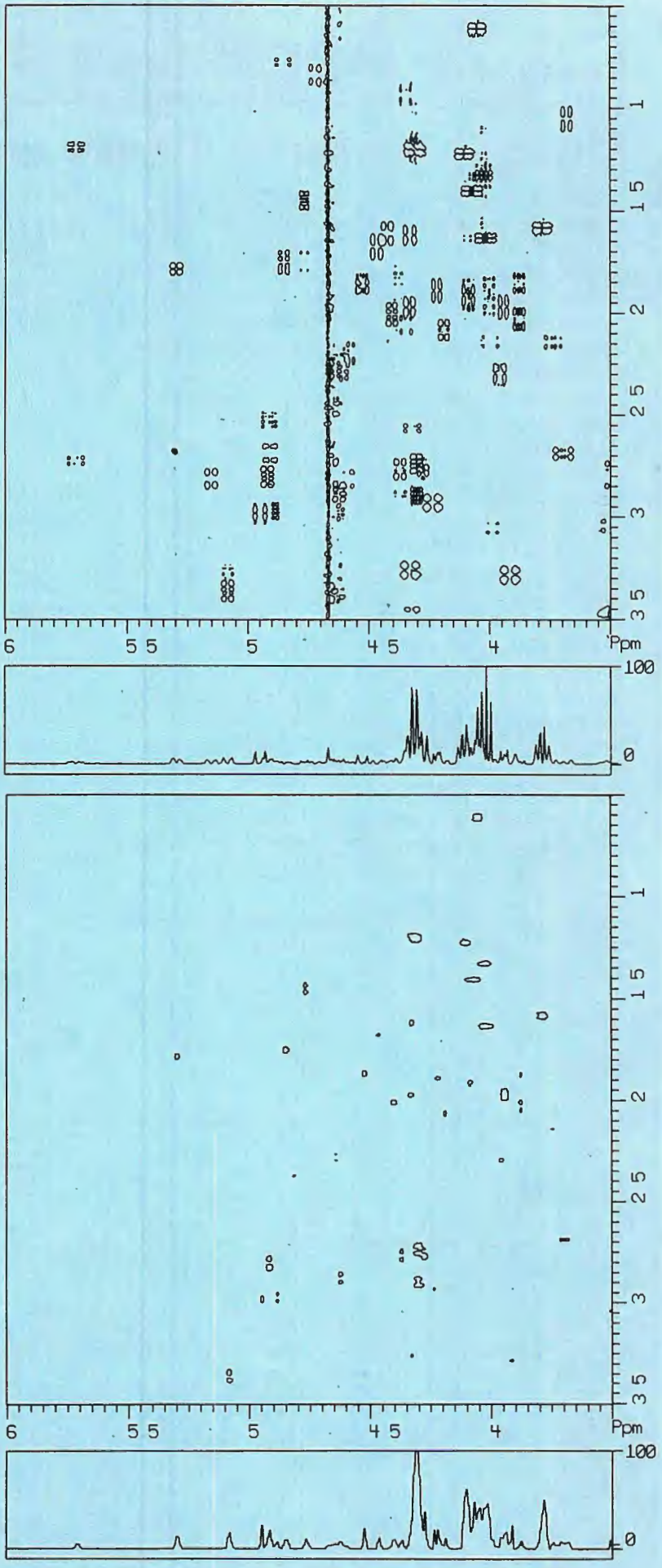
JEOL USA introduces the VPLX data processing package, our latest upgrade to the GX and GSX NMR spectrometers. When used with the latest network options of Multi-PLEXUS, VPLX provides the power and speed of a VAX™ and eliminates the need to learn a new set of software commands.

In addition to allowing for off-line processing, VPLX offers advanced functionality such as MEM/LPZ and Symmetry Filtering. The top data shows the normal NH to alpha region in a double quantum filtered COSY of BPTI in water. This matrix was produced on a GSX-400, processed on VPLX, and printed on a laser printer. The bottom data is identical to the first with the exception that a symmetry filter has been applied to the matrix. This symmetry filter discriminates on the basis of the known phase relationship of true COSY peaks. Each of the COSY peaks that passes through the filter is reduced to a centroid representation.** This filtering allows for the rapid elimination of spurious cross peaks and is the first step necessary for computer based spectral interpretation.

For more information, contact JEOL.



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**JC Hoch, S Hengyi, M Kjaer, S Ludvigsen, and FM Pousen, "Symmetry Recognition Applied to Two-Dimensional NMR Data", Carlsberg Res., Commun., Vol. 52, p.111, (1987).