TEXAS A&M UNIVERSITY

NMR NEWSLETTER

No. 407

August 1992

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TABLE 1 DEUTERATED SOLVENTS

Cat. No.	Description	Formula	Min. Iso. (% D)	Density (g/cc)	MP (°C)	BP (°C)	$-\chi_v \times 10^6$ @ (°C)
D-11	Acetic Acid-d_4	CH_3COOD	99.5%	1.1	17	118	0.551 (32)
D-120	Acetic Anhydride-d_6	$(CD_3CO)_2O$	98%				
D-13	Acetone-d_6	CD_3COCD_3	99.9%	0.84	-94	57	0.460 (20)
D-121	Aceonte-d_6 + 1% TMS		99.8%				
D-129							
D-14							543 (20)
D-21							0.611
D-122							
D-130							
D-28	Chloroform-d	$CDCl_3$	99.8%	1.50	-64	62	0.740 (20)
D-31	Chloroform-d + 1% TMS		99.8%				

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WCV-100 (S-100A)	11" X 26"	HA-100, HA-100D	Gridded-Two Color
WCV-60 (6-60C)	11" X 26"	A-60, A-60A	Gridded-Two Color
WCV-XL (XL-100)	11" X 26"	XL-100	Gridded-Two Color
WCV-XLFT-100	11" X 26"	XL-100 (Standard 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
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FORTHCOMING NMR MEETINGS

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

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

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

Additional listings of meetings, etc., are invited.

Third and Last Notice re 1992-93 Invoices and Subscription Rates

Subscription renewal invoices for the October 1992 - September 1993 year were mailed out on 1 July. If you ought to receive such an invoice, and do not have it in your hands by the time you read this notice, please call or write me promptly. *Payment* of these invoices *must be received* by me *no later than September 10, 1992* to ensure uninterrupted mailing of the Newsletter issues. Please do not delay execution of any necessary paperwork!

Also, please be sure that the instructions on the invoice are followed precisely. In particular, *overseas subscribers* should be careful to see that their name and the invoice number appear on the payment (or, better, that the extra invoice copy which is provided is returned to me with the payment check or money order). Anonymous checks, while otherwise useful, cannot always be credited to the correct account.

The subscription rate for the October 1992 - September 1993 year has been set at US$170.00 for the twelve monthly issues, postpaid. Personal or academic subscriptions will continue to be offered at a 50% discount, at US$85.00. The inexorable rise in costs has necessitated this small increase in the subscription fee.

The new invoices contain entries for *optional* surcharges for First Class or Air Mail Printed Matter mailing. Please adjust the amount you pay accordingly (I trust no one will choose to pay both surcharges.).

Thank you for your understanding and cooperation.

B. L. Shapiro
2 August 1992

* * * * *

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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. 409 (October) -------18 September 1992
No. 410 (November) -------16 October 1992
No. 411 (December)-----20 November 1992
No. 412 (January 1993) - 18 December 1992

* * * * *

Omega PSG
Heteronuclear Triple-Resonance Three-Dimensional NMR

Heteronuclear triple-resonance NMR of a uniformly labelled serine protease (20K Dalton) is illustrated using the HNCA experiment [Kay, Ikura, Tschudin & Bax JMR v89, 496(1990)]. Sample provided by Jonathan Davis, Biophysics Group, University of California, San Francisco. Pure phase data is illustrated for a dataset featuring 32 complex points in the Nitrogen evolution period, 64 complex points in the Carbon evolution period and 512 complex points in the Proton domain. Acquisition times were 16ms, 19ms and 65ms, respectively. The C-13 pulse widths were adjusted to 65 μsec to achieve selectivity for both the carbonyl region (175ppm) and the alpha-carbon region (55ppm). Three transmitters were employed: ^{1}H, ^{15}N and ^{13}C. The carbon transmitter was set at 55ppm and carbonyl pulses were accomplished using phase ramp procedures. Data were collected in the Hypercomplex phase sensitive mode using an unmodified Omega PSG 500. TPPI 180-degree phase alternation in successive evolution dwell periods was employed to suppress axial resonances.

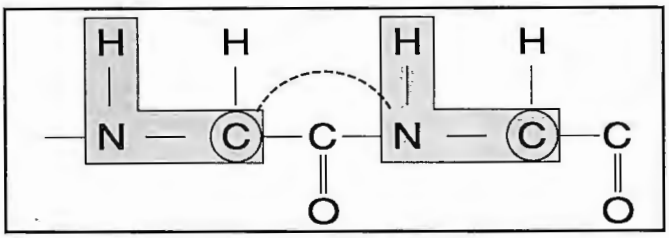

Figure 1

HNCA experiment correlates ^{15}N and NH chemical shifts with the intraresidue Cα shift. It also provides sequential connectivity information by yielding (weak) correlations between NH-^{15}N pairs and the Cα shift of the preceding residue.

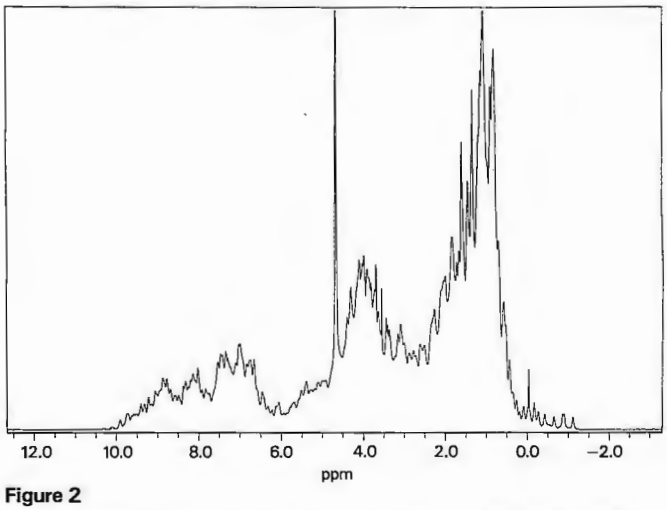

Figure 2

^{13}C, ^{15}N uniformly enriched serine protease (20K Dalton) 1.7 mM 94% H_2O 35°C.

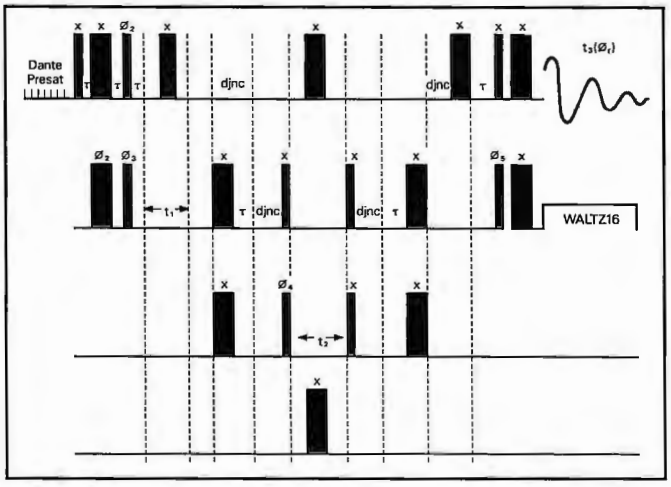

Figure 3 HNCA pulse sequence.

$\varnothing_1$ = 2X,2(−X); $\varnothing_2 = \varnothing_1 + 90$; $\varnothing_3$ = X,−X; $\varnothing_4$ = 4X,4(−X); $\varnothing_5$ = 8X,8(−X); $\varnothing_r = \varnothing_1 + \varnothing_3 + \varnothing_4 + \varnothing_5$; $\tau = 1/(4JNH)$; τ^1 = 2.75ms; djnc = $\frac{1}{9J_{NC\alpha}}$

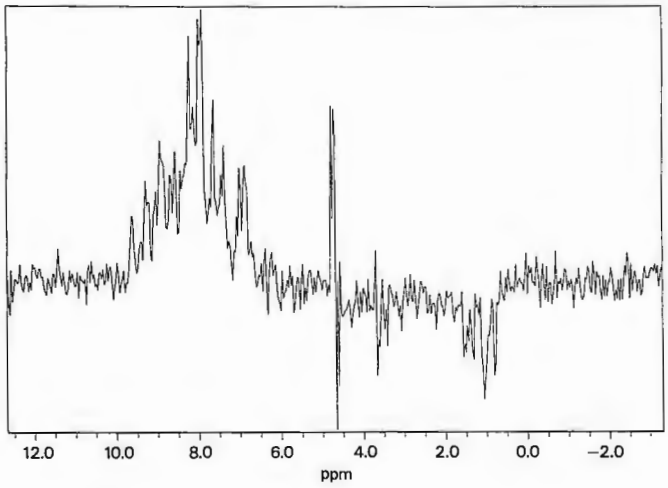

Figure 4

^{15}N, ^{13}C filtered 1D spectrum of first block.

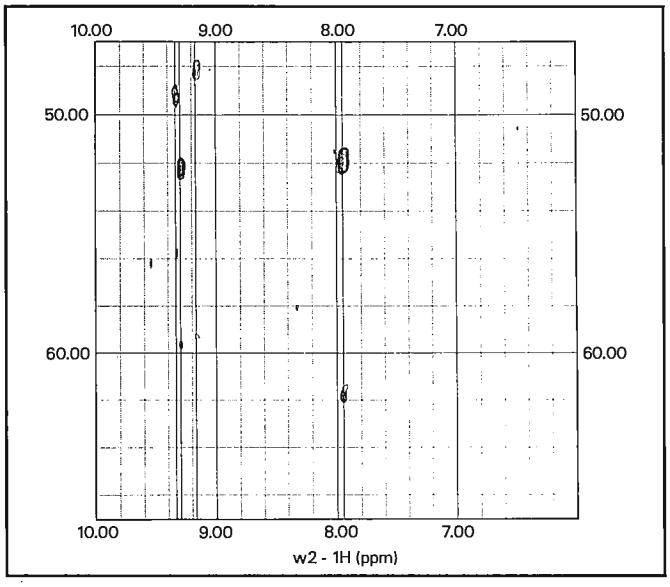

Figure 5

3D plane for $\delta^{15}N$ = 137.3 ppm showing strong and weak correlations.

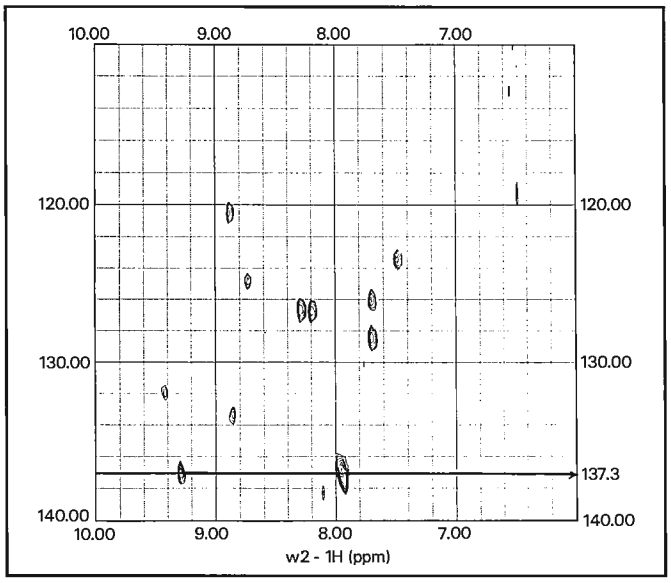

Figure 6

3D plane for $\delta^{13}C$ = 51.9 ppm showing two ^{15}N cross peaks at $\delta^{15}N$ = 137.3 ppm.

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UAB SCHOOL OF NATURAL SCIENCES AND MATHEMATICS
Department of Chemistry

June 19, 1992
(received 6/22/92)

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

Dear Barry:

We recently published the synthesis and ^{1}H and ^{13}C NMR of a series of aminoarsines, Me_2AsR (for R, see table) [1]. Also, as part of our interest in Group 13/15 chemistry, we have studied by multinuclear, VT NMR the reaction of $(Me_3Al)_2$ with Me_2AsNMe_2 [2]. In this system, an adduct is initially formed in solution, $Me_2AsNMe_2 \cdot AlMe_3$, which slowly decomposes to $[Me_2AlNMe_2]_2$ and Me_3As. Having on hand a variety of aminoarsines, we were therefore interested in studying their reactivity with $(Me_3Al)_2$ and other Group 13 Lewis acids. In particular, the choice of R might lead to interesting results from a mechanistic point of view. In fact, the reactivity of $(Me_3Al)_2$ toward the aminoarsines was quite different and dependent on the R group, although in each case the final products were $[Me_2AlR]_2$ and Me_3As [3]. For example, the time for the reaction to go to completion at RT in C_6D_6 solution depended markedly upon R: R = NMe_2, NEt_2, (1 week); R = NPr^n_2, NBu^n_2 (2 weeks); R = NBu^i_2 (1 month); R = NPr^i_2 (3 months); R = NC_5H_{10} (2 months); R = NC_6H_{12} (4 months); R = NC_4H_8 (6 months).

Particular intriguing were the results obtained for the five-, six-, and seven-membered ring derivatives. This reminded us of the classic studies of H. C. Brown and others [4,5] where, for example, Me_3B forms a stronger adduct with pyrrolidine than piperidine due to smaller steric effects upon complexation. The rationale is that the α-methylene groups in the five-membered ring are flexible and can move to avoid direct steric effects with the complexing group, whereas strong 1,3-diaxial interactions would result with the six-membered heterocycle. Since ^{13}C NMR chemical shifts are known to be sensitive to steric effects in these types of systems [6], we compared the δ_C of the parent aminoarsines with those of the initially observed adduct $Me_2AsR \cdot AlMe_3$ (See Table). In fact, the largest $\Delta\delta_C$ values for the five-membered ring are C-2,5, while those for the six- and seven-membered ring are C-3,5 and C-3,6, respectively as predicted by classical theory. Additionally, the normal alkane derivatives show large changes in δ_C at C-2 indicating their flexibility upon complexation, while the branched alkane derivatives only

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show small variations. This is reflected in relatively weak adduct formation and chemical exchange for the Pr^i and Bu^i derivatives and their subsequent longer reaction times.

Best regards.

Charles L. Watkins
Professor

Larry K. Krannich
Professor and Chairman

REFERENCES:

1. C. J. Thomas, L. K. Krannich, C. L. Watkins, *Synth. React. Inorg. met.-Org. Chem.*, 21, 427 (1991).
2. L. K. Krannich, C. L. Watkins, D. K. Srivastava, *Polyhedron*, 9, 289 (1990).
3. C. J. Thomas, L. K. Krannich, C. L. Watkins, *Polyhedron*, submitted.
4. H. C. Brown, M. Gerstein, *J. Am. Chem. Soc.*, 72, 2926 (1950).
5. M. L. Newnam, Ed., Steric Effects in Organic Chemistry, Wiley, New York, 1956.
6. A. Flores-Parra, et. al., *Tetrahedron*, 47, 6903 (1991).

Table. Differences in ^{13}C NMR chemical shifts -- $\delta(Me_2AsR\cdot AlMe_3) - \delta(Me_2AsR)$

R	Me_2As	C-1	C-2	C-3	C-4	C-5	C-6	C-7
Me_2N	0.50	-1.02	—	—	—	—	—	—
Et_2N	-0.51	0.72	-4.06	—	—	—	—	—
$Pr^n{}_2N$	-0.58	0.58	-3.66	0.03	—	—	—	—
$Pr^i{}_2N$	-1.63	-0.40	0.71	-0.40	—	—	—	—
$Bu^n{}_2N$	-0.48	0.95	-4.38	0.41	-0.38	—	—	—
$Bu^i{}_2N$	-1.57	-0.34	-0.19	0.17	0.17	—	—	—
NC_4H_8	2.07	—	2.12	0.44	0.44	2.12	—	—
NC_5H_{10}	1.82	—	-0.99	-4.87	-1.90	-4.87	-0.99	—
NC_6H_{12}	1.04	—	-0.21	-3.19	-1.33	-1.33	-3.19	-0.21

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

(received 6/26/92)

Dear Dr. Shapiro,

Title: The softening of decoupler pulses on a VXR-400

A radiofrequency transmitter with a large dynamic range is essential for NMR experiments which are used to produce weak irradiating fields for selective excitation, moderate power levels for broadband decoupling or spin-locking, and an intense RF field for π and $\pi/2$ pulses. For example, a B_2 field of 10 Hz for selective perturbation and a non-selective π pulse may require 10 μW and 10 W, respectively, for proton experiments at high field strengths. Pulse sequences which require rapid and frequent switching between power levels should employ solid-state control circuits to preclude the slow switching speed (10-25 ms) and limited life expectancy of electromechanical relays.

SEARLE

In our Varian VXR-400, the full range of B_2 power from the decoupler transmitter is only available by using a combination of low (DLP) and high (DHP) decoupler levels. Since the VXR-400 uses electromechanical relays to change between the low and high power levels, it is unsuitable for experiments which require extensive use of the full range of B_2 fields. The DHP mode satisfies the intermediate and high power requirements, but the decoupler power regulation scheme stops working at a power level of approximately 1 mW (see Figure 1) which gives a B_2 field of 100 Hz.

We have modified the DHP transmitter circuit to incorporate a computer controlled 30 dB attenuator between the transmitter output and the probe. Figure 2 is a block diagram of the VXR-400 transmitter section (the attenuator is shown in a dashed block). The modification (Figure 3) comprises solid-state control using PIN diodes to either pass the transmitter power directly to the probe or to route the power through a resistive network making up the 30 dB attenuator. The switching time between the normal and low (attenuated) power mode is 5 μs. This switching is controlled by one of the spare computer-control lines on the VXR-400 Interface Board and is controllable in pulse sequences (1). With this modification we now have control of a continuous range of B_2 fields from 3 Hz to > 8 KHz.

Sincerely,

Robert W. Dykstra

(1) E. Johnson and S. Patt, Magnetic Moments, Vol. 1, No. 4 (1985), Varian Assoc., Palo Alto, CA.

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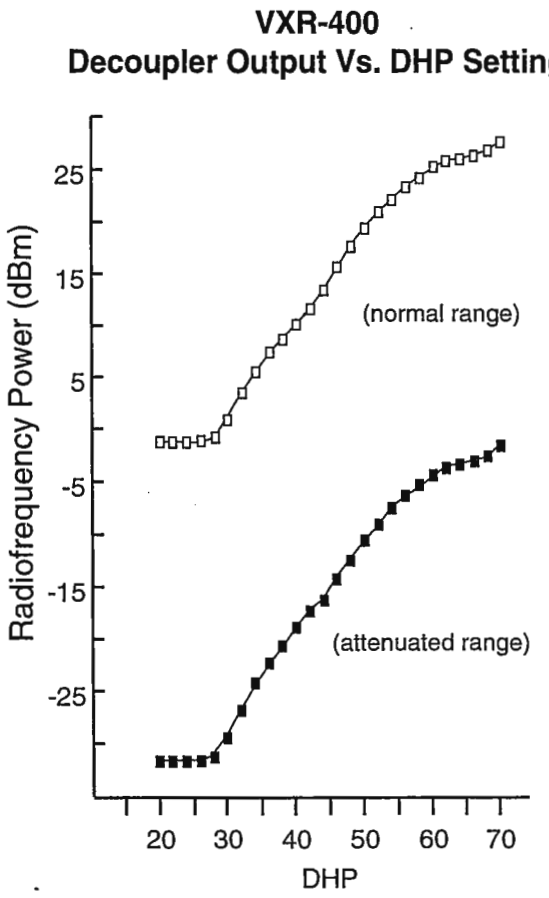


Figure 1

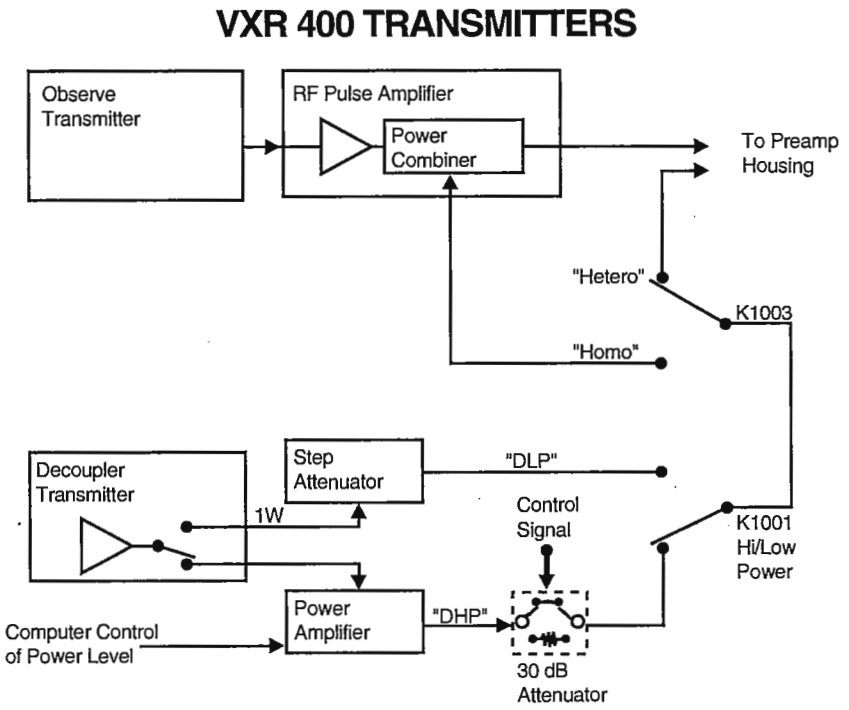
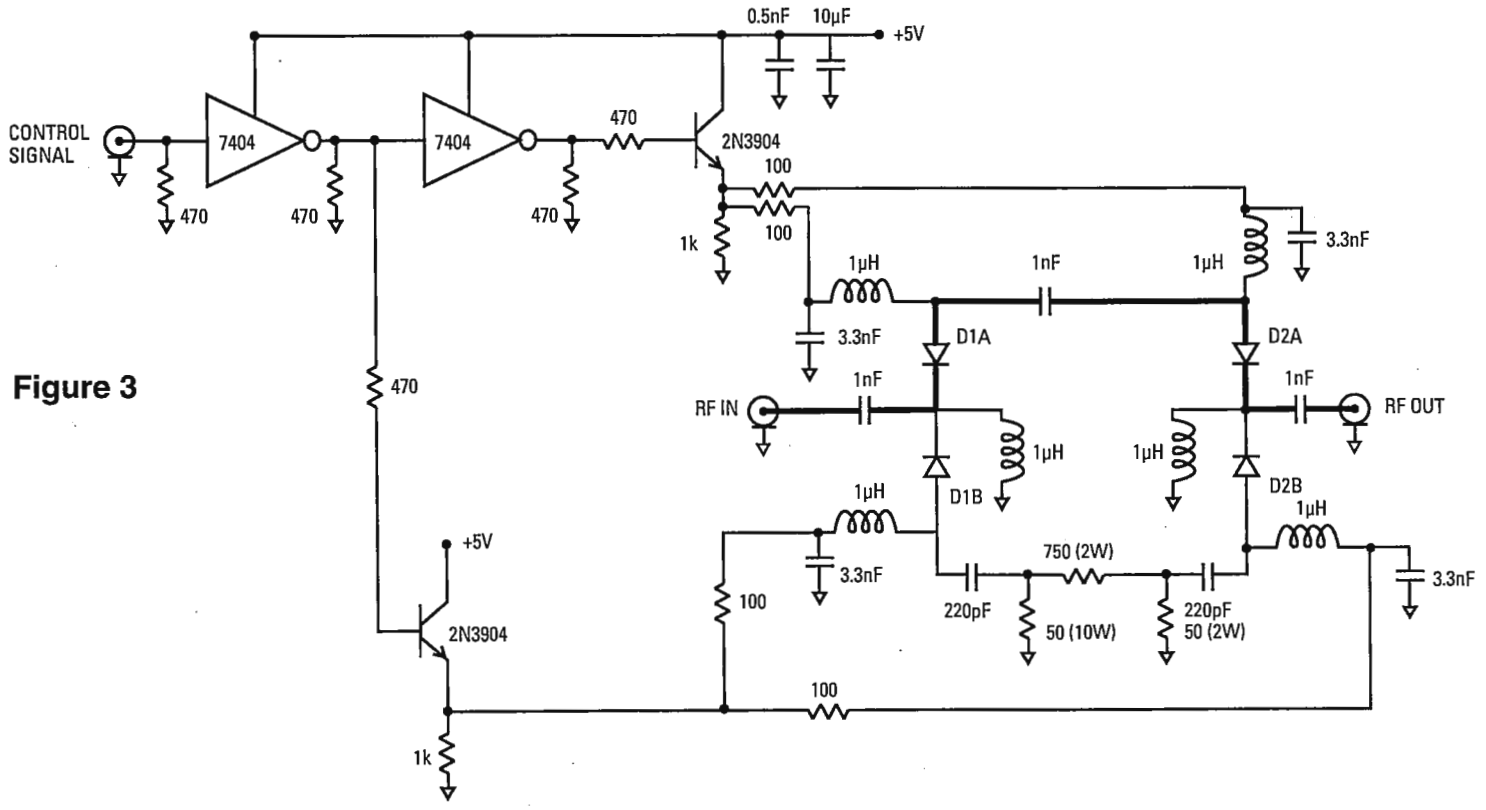


Figure 2

Figure 3



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June 9, 1992 (received 6/29/92)

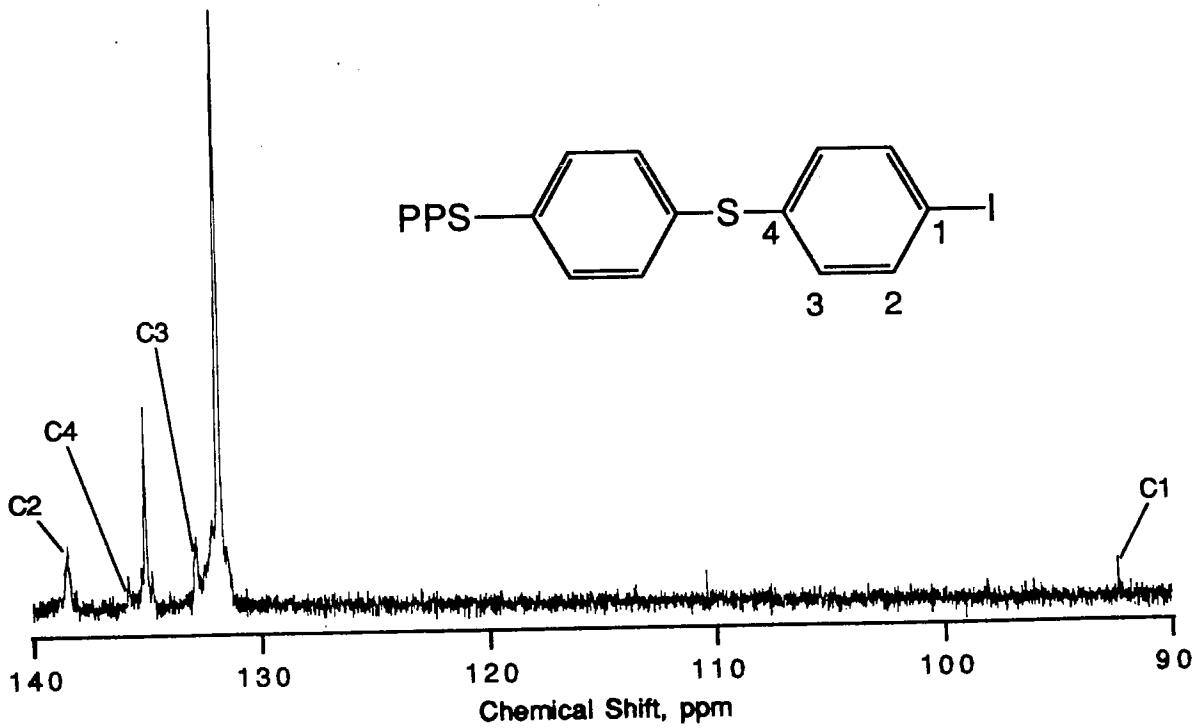
Prof. Bernard L. Shapiro
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto, CA 94303

Very High-Temperature Carbon-13 NMR

Dear Prof. Shapiro:

As we continue to investigate means of enhancing our knowledge of the chemistry of poly(p-phenylene sulfide/disulfide), PPS/DS, we find that very high-temperature solution-state carbon-13 NMR is very useful. PPS/DS is a high temperature thermoplastic made from the reaction of p-diiodobenzene (DIB) and sulfur. During this reaction, both sulfide and disulfide linkages are formed, thus our designation of PPS/DS.[1]

Iodo-terminated oligomers of PPS/DS would be envisioned from this reaction mix as the polymer builds up. In the attached figure is



presented a spectrum of an iodo-terminated oligomer containing only sulfide linkages and 9 repeat units. This oligomer was prepared in the same manner as PPS/DS except that an excess of DIB was employed. For the carbon-13 NMR spectrum acquired at 230°C in a 10-mm NMR tube, the oligomer was dissolved in N-cyclohexyl-pyrrolidinone and used glyme-d_6 held in a concentric 5-mm NMR tube for field/frequency stabilization. From the spectrum, it is clear that detailed information about the nature of iodinated end groups can be obtained from a carbon-13 NMR spectrum of this material. Carbon resonances of the terminal phenyl group as well as the internal phenyl groups are clearly evident and assignable. Chemical shift assignments in ppm are as follows:

Terminal Group		Internal Group	
C1	92.3	Protonated C	131.8
C2	138.4	S-Substituted C	135.1
C3	132.7		
C4	135.7		

It is clear from this spectrum that carbon-13 NMR spectroscopy at very high temperature in solution offers extremely exciting possibilities for studying this polymer system.

[1]M. Rule, D. R. Fagerburg, J. J. Watkins, P. B. Lawrence; Makromol. Chem., Rapid Commun. **1991**, 12, 221

Sincerely yours,

Douglas W. Lowman
Principal Research Chemist
(615) 229-4728
Research Laboratories
P. O. Box 1972

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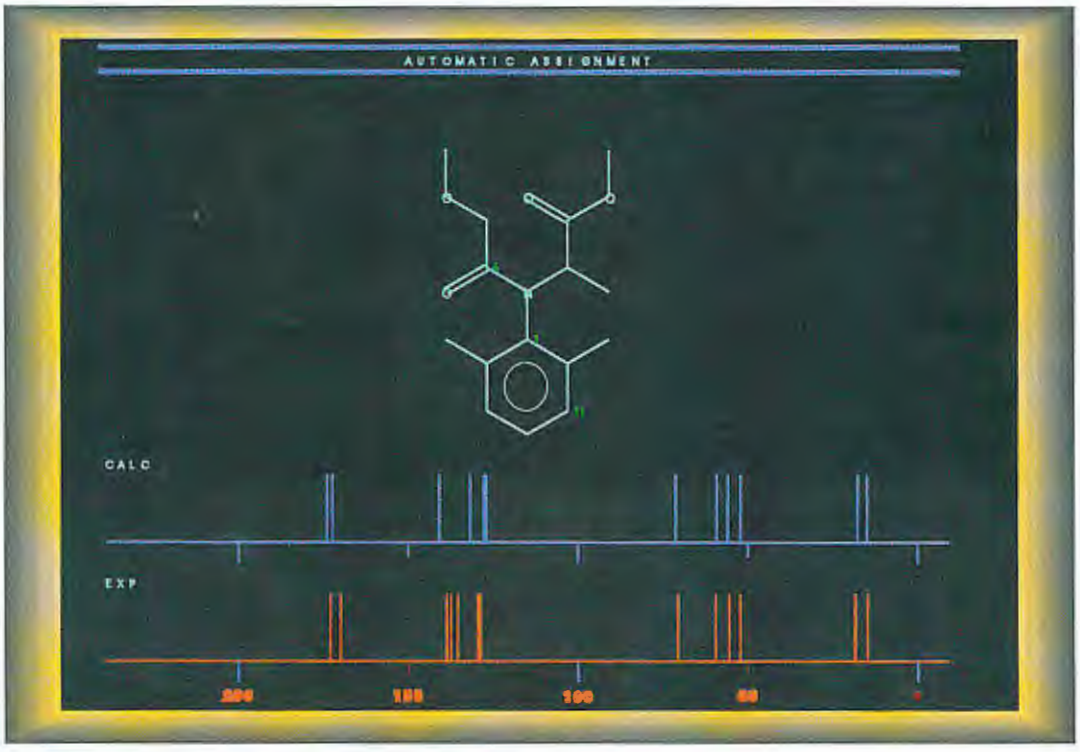

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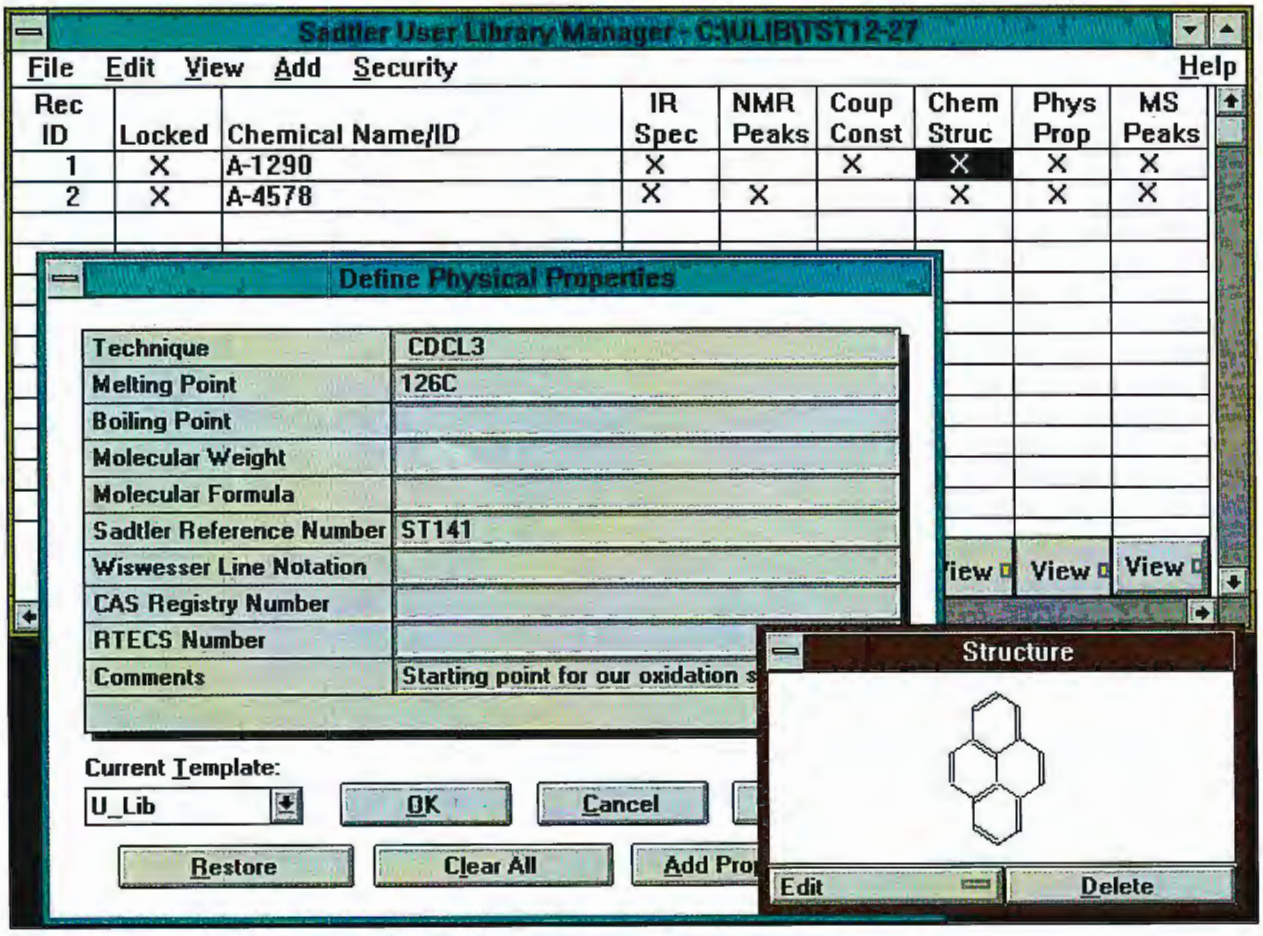

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The College of Staten Island

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OF NEW YORK

ST. GEORGE CAMPUS
130 STUYVESANT PLACE
STATEN ISLAND, NEW YORK 10301

July 1, 1992
(received 7/7/92)

Professor Bernard L. Shapiro
966 Elsinore Court
Palo Alto
CA 94303

Dear Professor Shapiro:

Aggregation phenomena are often of central importance to the function of macromolecules, from industrial applications such as tertiary oil recovery to physiological events that include the biliary secretion of membrane lipids and the intestinal digestion of acylglycerols. Micellar aggregates are thought to play a role in these latter processes, both as substrates for lipolytic enzymes and as vehicles for transport of digestive products to the absorptive mucosa of the small intestine.

We have measured ^{13}C NMR relaxation times and NOEs in both CD_3COD and D_2O for model digestive mixtures containing a bile salt (taurocholate, TC), a monoglyceride (1-monocapryloyl-rac-glycerol, MC), and a fatty acid (caprylic acid, CA) in proportions roughly matching those found physiologically in the aqueous contents of the upper small intestine during fat digestion.

We first found contrasting ^{13}C spin-lattice relaxation behavior for designated hydrophilic, interfacial and hydrophobic groups. Hydrophilic moieties (TC SO_3^{2-}, CA COO^-, MC CHOH, MC CH_2OH) exist in a substantially aqueous environment, and their dynamic behavior may be complicated by interactions with a fluctuating water structure. Interfacial groups (TC C_{26}, MC CH_2OR, CA C_2) are expected to exhibit common motional behavior that is dominated by the aggregate as a whole, with no additional internal chain reorientation. Hydrophobic moieties (alkyl chains, steroid skeleton) have a less polar environment, with motions influenced by the local segmental motions as well as by the overall reorientation of the aggregate.

Table 1 demonstrates that very similar values of NT_1 and NOE are indeed found for the interfacial groups in aqueous TC/MC/CA mixtures. No such equality holds for the spin-relaxation parameters of this same set of carbons in a "nonaggregating" solvent such as CD_3COD.

Table 1 Boundary-Carbon Relaxation Parameters in two Solvents

	CD_3OD				D_2O			
	NT_1, s		NOE		NT_1, s		NOE	
Magnetic field (MHz)	50	100	50	100	50	100	50	100
MC, CH_2OH	3.0	3.0	2.5	3.0	0.90	1.32	2.0	2.4
CA, (2)	6.0	5.8	2.7	3.0	0.92	1.32	2.0	2.4
TC, 26	1.64	1.74	2.3	1.9	0.92	1.22	2.0	2.7

Using these data, the two-step model[1,2] may be used to obtain best-fit values for the dynamic parameters τ_f, τ_s and a local order parameter S^2. All values of NT_1 and NOE calculated from the best-fit dynamic parameters are in excellent agreement with the experimental spin-relaxation data. Both the rate of local motions and the angular excursion of the MC/CA chain segments are diminished upon formation of mixed micelles with TC, suggesting highly organized packing of the alkyl chains. Preferential immobilization of the MC backbone is also consistent with an aggregate structure that has a belt of polar groups at its aqueous interface.

1 H. Wennerstrom, B. Lindman, O. Soderman, T. Drakenberg and J. B. Rosenholm, J. Am. Chem. Soc., **101**, 6860(1979).

2 H. Walderhaug, O. Soderman, P. Stilbs, J. Phys. Chem. **88**, 1655(1984).

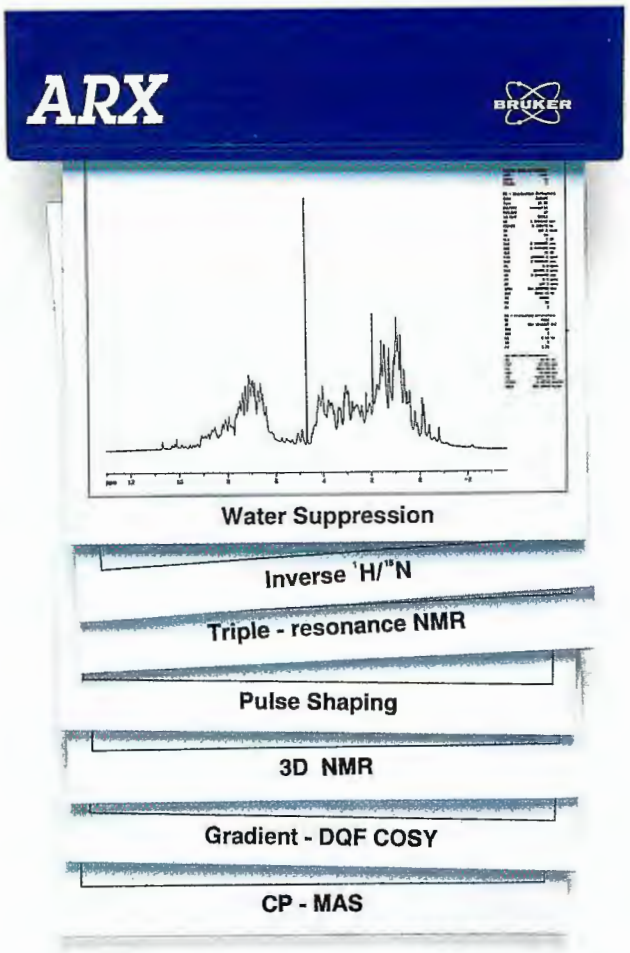
Sincerely

Dehua Wang

Dehua Wang

Ruth

Ruth E Stark



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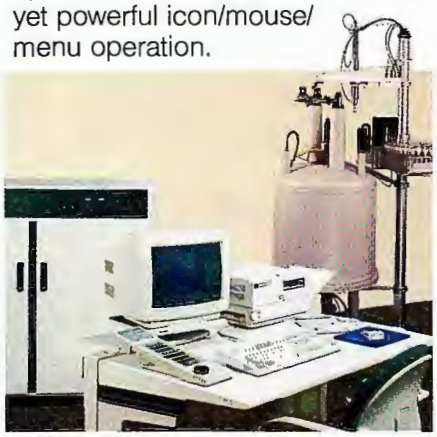

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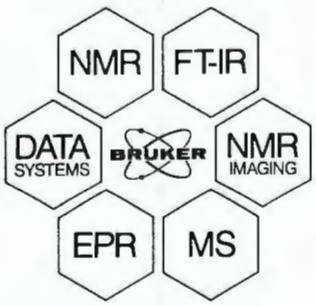
Performance Data

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

EB = ethylbenzene (for ^{13}C with ^{1}H-dec.);
ASTM = 60% C_6D_6 in dioxane
Form. = formamide (^{1}H-dec. without NOE)
lineshape: ^{1}H = $CHCl_3$ linewidth at ht. of ^{13}C-satellites/at 20^6 this level
^{13}C = C_6H_6 linewidth at 0.55% / 0.11 level (^{1}H-dec.)
SSB = Spinning sidebands measured with 8 transients
QNP: 5 or 10 mm ^{1}H, ^{31}P, ^{13}C, ^{15}N
VSP: 5 mm ^{15}N – ^{13}P; 10 mm ^{109}Ag – ^{31}P.

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



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June 30, 1992 (received 7/10/92)

Prof. B. L. Shapiro
968 Elsinore Court
Palo Alto, CA 94303

Baseline Distortions due to Corrupted FIDs

Dear Prof. Shapiro:

It is generally well known that distorted baseline results in the Fourier transformed spectrum when initial few points of the FID get corrupted due to nonideal audio filter response or transmitter breakthrough. We have found this to be the limiting factor in the quantitative accuracy of NMR measurements. Software packages available on most modern spectrometer provide numerical schemes that attempt to fit the baseline as a polynomial of some specified order to correct the baseline. In order for us to understand what baseline correction entails, we needed an equation of the sort that expressed a quantitative relationship between distortion of any FID data point and intensity of any frequency domain data point. Not being able to find such an equation in any standard books on NMR (which mostly treat this subject qualitatively), we derived the following expression to relate deviation of the intensity of any frequency data point when some phenomena causes the time domain data to be corrupted:

$$\Delta I(F) = \sum_j A_j \left((1-\Delta_1) + \sum_{K=2}^{N} 2.(1-\Delta_K) \cos\left(2\pi . K . \frac{F-F_j}{SW}\right) \right)$$

where $\Delta I(F)$ is the deviation from ideal intensity of a data point with frequency "F" in real part of the spectrum that contains "j" lines with A_j and F_j as their respective intensity and frequency. The total number of points transformed is "N" (including any zero filling) and "sw" is the spectral width. Frequency and "sw" are in units of Hz. Δ_K represents the factor by which the K^{th} point of the FID is multiplied. In an ideal case all Δ_K are equal to 1 and intensity deviation of any frequency point from ideal intensity is zero. Corrupting only the first point results in all frequency points shifted by the total intensity of all the lines times $(1.-\Delta_1)$, i.e., a dc offset (note that actual intensity of a resonance line in frequency domain spectrum is $N*A_j$). Corruption of subsequent FID points will generate a frequency dependent intensity variation in the baseline resulting in "rolling baseline". The interesting fact to note is that such baseline roll depends on the relative frequency and intensity of all the lines in the spectrum. For a spectrum containing numerous lines with different frequencies and intensities, corruption of even the second point in the FID, i.e., all Δ_K are equal to 1 except Δ_2, can generate a very complicated baseline.

This is illustrated in the figure by the simulated spectra for a system containing only two lines with equal intensity but varying frequency. The spectra were calculated in the time domain and convoluted with a filter response function with all points equal to 1 except the second point which was set to zero, i.e., Δ_2=0, before FT. Note how the character of the baseline changes when the relative frequency of the two lines is varied.

Hopefully, your readers will find the equation above useful when trying to understand spectral baseline distortions or when attempting to decipher what the "blackbox" baseline correction numerical routines on commercial spectrometers are doing.

Best regards,

Naresh K. Sethi

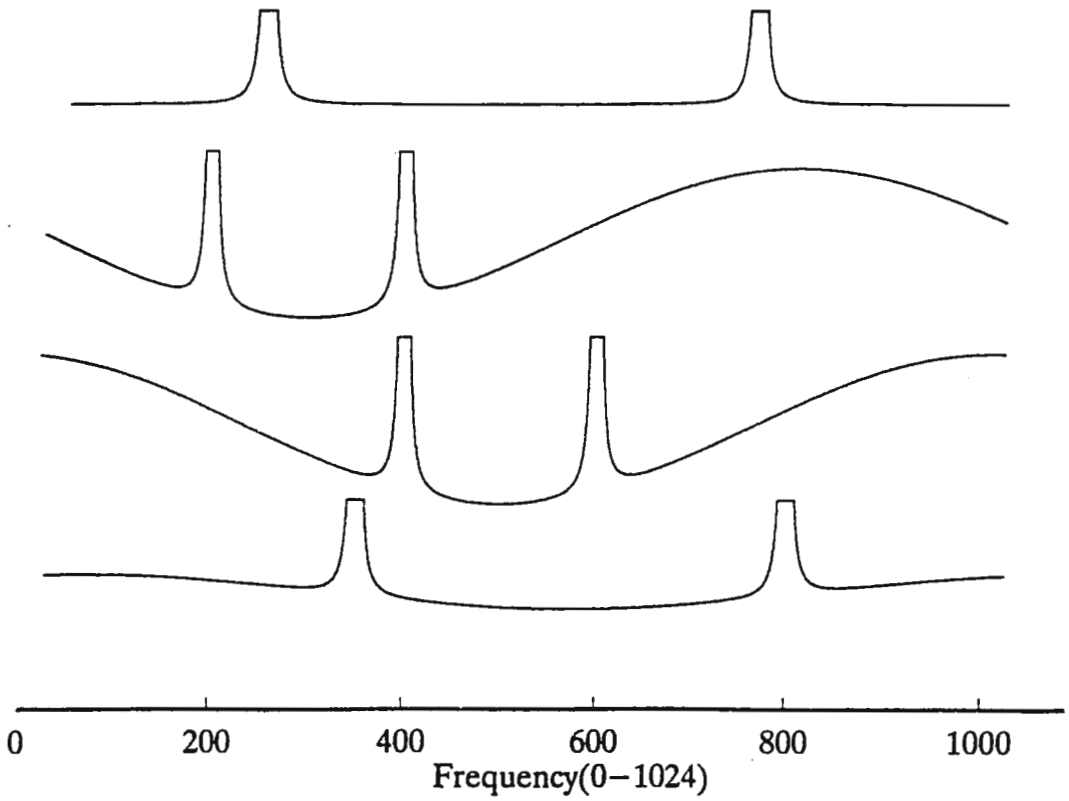

Fig: Distorted baselines when the second point in the FID is zero for a two line system with equal intensity but different relative frequency.

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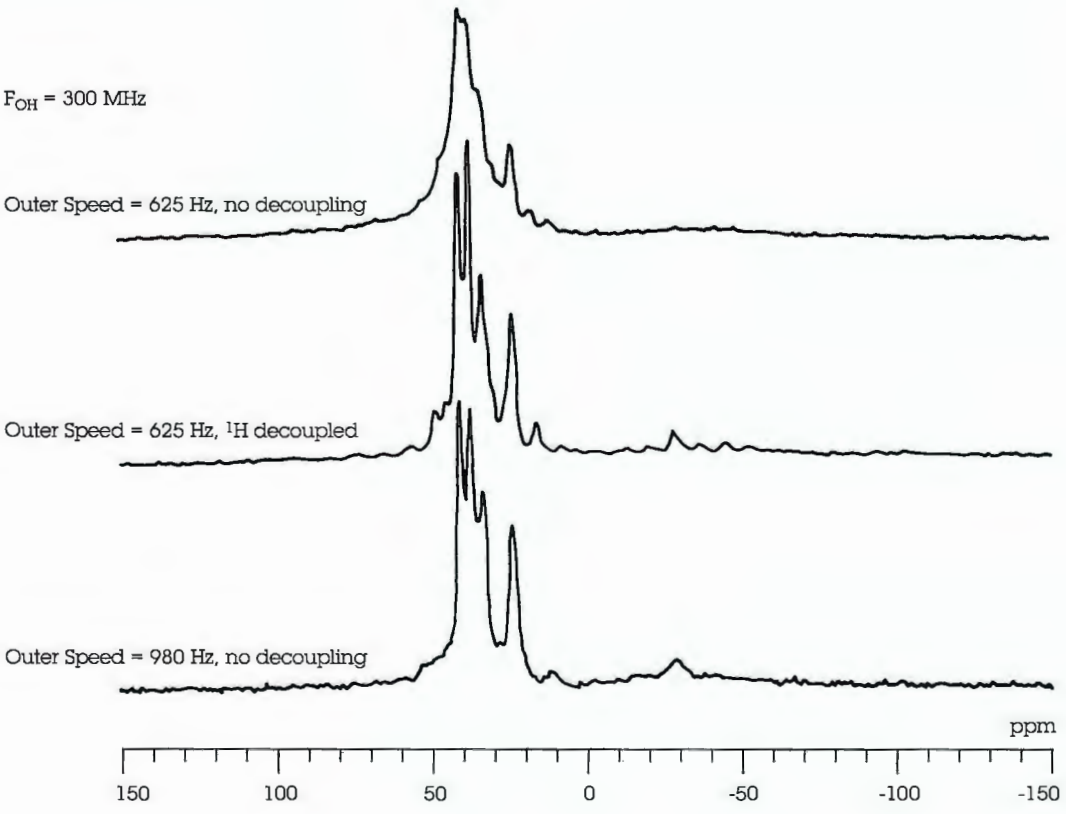

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Sandia National Laboratories

Albuquerque, New Mexico 87185

July 22, 1992
(received 7/24/92)

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

Intercalation of Molecular Species into the Interstitial Sites of Fullerene

Dear Barry,

As part of our program to prepare polymers using "bucky balls" as a building block, we had occasion to record the high resolution ^{13}C NMR spectra of solid C_{60} under MAS conditions. Fig. 1 shows the fully relaxed spectrum of C_{60} purified in our laboratory. In addition to the primary resonance at 143.7 ppm, there is a minor resonance at 144.4 ppm with 4.5 % of the total intensity. The spin-lattice relaxation time of the primary peak is 71±7 s while the spin-lattice relaxation time of the minor peak is 0.26±0.03 s. The intensity of the minor peak was shown to depend on sample history. Pure C_{60} purchased from Texas Fullerenes had a minor resonance with an intensity of 2.5 % of the total intensity while its minor peak intensity was reduced to 0.6 % after the sample was annealed for 1 hr at 225 C under dynamic vacuum. We conclude that the minor resonance is attributable to C_{60} molecules which are associated in some way with molecular oxygen.

Since molecular oxygen is paramagnetic, the chemical shift of the minor resonance is believed to be caused by the Fermi-contact interaction between the unpaired electron spin of the oxygen and the ^{13}C nuclear spin. To establish this relationship, we varied the temperature to determine if the shift obeyed Curie's law. Fig 2 shows the chemical shift of the minor resonance, referenced to that of the primary resonance, as a function of 1/T. The least-squares fit of the data yields an intercept of 0, within experimental error, and provides additional support that the shift is caused by the Fermi-contact interaction of oxygen with C_{60}.

Fig 3 shows the MAS ^{13}C NMR spectrum of an annealed sample which has been exposed to 1.0 kbar oxygen for 1 3/4 hr, removed from the pressure chamber and then placed in a MAS rotor. The spectrum was accumulated using a recovery of 1 s so the primary resonance at 143.7 ppm was not fully recovered and its equilibrium intensity is much larger than that represented by its resonance in Fig. 3. We see 6 secondary resonances of comparable intensities and no evidence of a seventh resonance. A maximum of 6 minor resonances were also recorded for samples which had been exposed to 1 kbar of oxygen for several days. Even in this instance, when the sixth minor resonance had the greatest intensity, there was no evidence of a seventh resonance. We conclude that a C_{60} molecule associates with a maximum of 6 oxygen molecules. The fact that the minor resonances are well defined suggests that the oxygen interacts equally with all of the carbons in a C_{60} molecule. This would be expected if the C_{60} rotates rapidly so that all of the carbons sample the oxygen environment equally. Rapid rotation of the C_{60} molecule in the solid at room temperature has been reported [1].

These results are consistent with the oxygen molecule occupying the octahedral interstices in the C_{60} crystal lattice. Each C_{60} molecule is surrounded by 6 equivalent octahedral sites. Previous work [2] as well as our own single crystal studies have shown that the lattice constant equals approximately 14.19 Å. The van der Waals radius of a C_{60} molecule is then 5.01 Å and the minimum octahedral site spacing is 4.12 Å. The interatomic separation of oxygen, 1.2 Å, together with a van der Waals radius of 1.4 Å suggest that the molecule could tumble freely within the octahedral interstices. The limit of 6 minor resonances denotes that only one oxygen molecule occupies a site under the conditions employed in this study.

A full report of this work has been accepted for publication by the Journal of Materials Research. Please credit this contribution to the account of P. Cahill.

Sincerely,

Roger A. Assink, James E. Schirber,
Douglas A. Loy, Bruno Morosin and Gary A. Carlson

REFERENCES

1. R. Tycko *et al.*, *J. Phys. Chem.* **95**, 518 (1991); C. S. Yannoni, R. D. Johnson, G. Meijer, D. S. Bethune, J. R. Salem, *J. Phys. Chem.* **95**, 9 (1991).

2. P. Heiney *et al.*, *Phys. Rev. Lett.* **66**, 2911 (1991); S. Liu, Y. Lu, M. M. Kappes, J. A. Ibers, *Science* **254**, 408 (1991); W. I. F. David *et al.*, Nature **353**, 147 (1991).

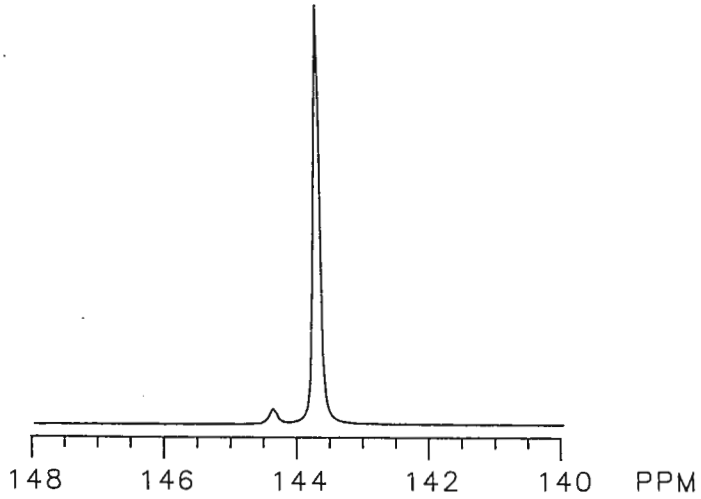

Fig. 1. High resolution ^{13}C NMR spectrum recorded at 50.2 MHz using MAS.

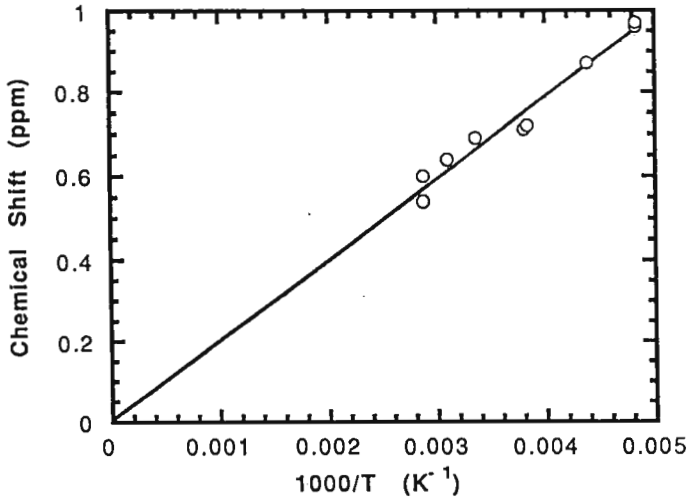

Fig. 2. The chemical shift of the minor resonance obeys Curie's law.

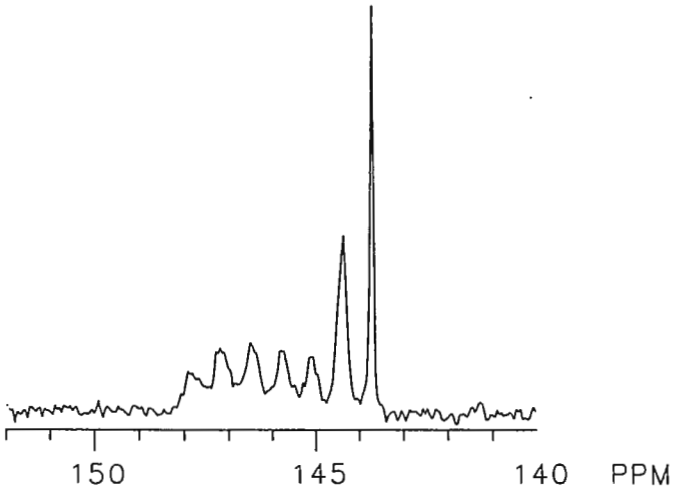

Fig. 3. Spectrum of a sample which was exposed to 1.0 kbar oxygen for 1 3/4 hr.

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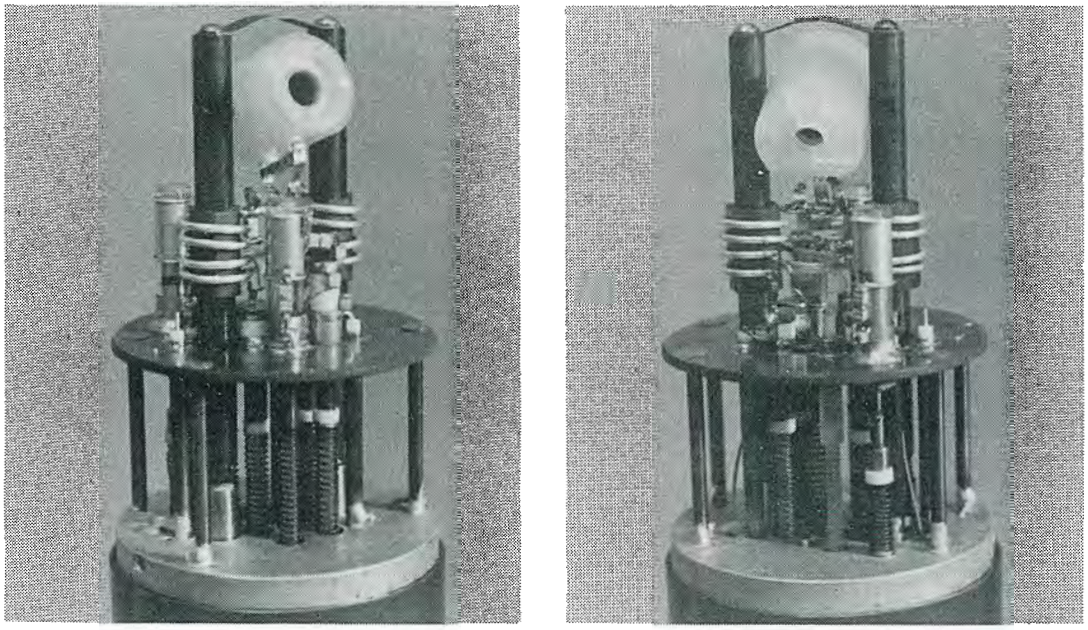

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Abbott Park, IL 60064

(received 7/25/92)

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

RE: MEASURING SMALL J-COUPLINGS FROM ECOSY-LIKE SPECTRA

Dear Barry,

Recently, several NMR methods have been proposed for measuring homonuclear and heteronuclear three-bond coupling constants in large systems from ECOSY-like experiments.[1] In these experiments, small three-bond couplings are measured from the frequency difference between two cross-peaks in one dimension that are separated by a large one-bond coupling in another dimension. In practice, it is very difficult to reproducibly measure these coupling constants from the spectra. In our experience, a good method for measuring these J-values is by superimposing 1D traces extracted from the spectra, shifting one spectrum relative to the other, and measuring the frequency shift required to obtain the peak overlap.[2] This method has the advantage of utilizing the peak lineshapes which should be the same for each of the peaks.

The approach is illustrated in Figure 1 for the measurement of the $^{3}J_{H\alpha,H\beta}$ coupling constants from a coupled 3D HCCH experiment for Phe15 of [U-15N,13C]FKBP when bound to the immunosuppressant, ascomycin.[2] Figure 1A depicts traces through the center of the two peaks from which the couplings are measured. In Figure 1B, the lower trace has been shifted to superimpose the peaks on the right, and in Figure 1C, the lower trace has been shifted to superimpose the peaks on the left. The shift required to overlap the peaks corresponds to the coupling constant.

Sincerely,

Ed Olejniczak Robert Xu Stephen Fesik

[1] Montelione, G.T., Winkler, M.E., Rauenbuehler, P., and Wagner, G., J. Magn. Reson., **82**, 198-204 (1989).

[2] Xu, R.X., Olejniczak, E.T., and Fesik, S.W., FEBS Lett., **305**, 137-143 (1992).

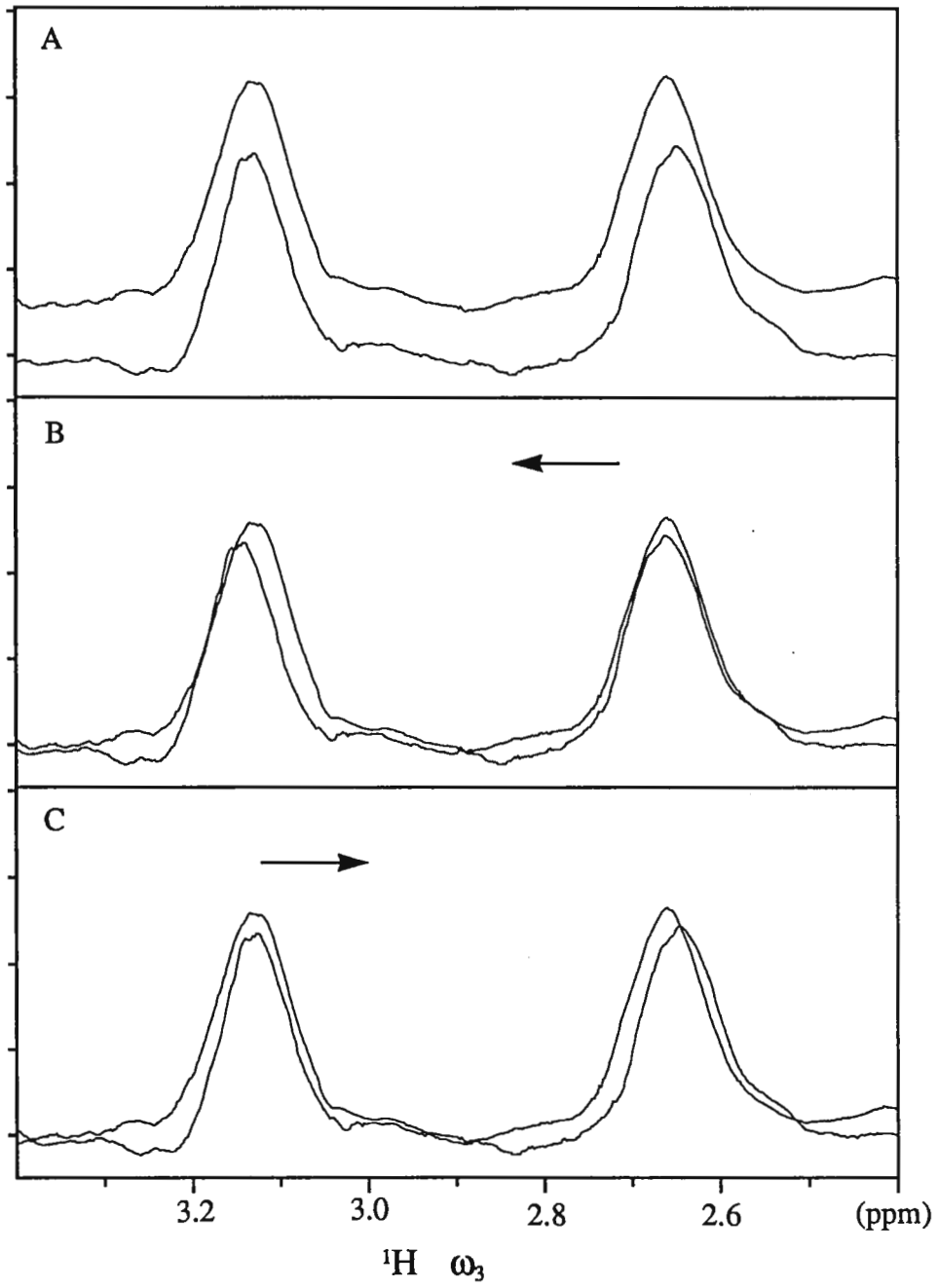

Figure 1. Traces from a coupled 3D HCCH spectrum extracted at the center of the two ^{1}H-coupled C$^\alpha$ peaks of Phe15 for [U-15N,13C]FKBP when bound to the immunosuppressant, ascomycin.[2]

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DEPARTMENT OF CHEMISTRY

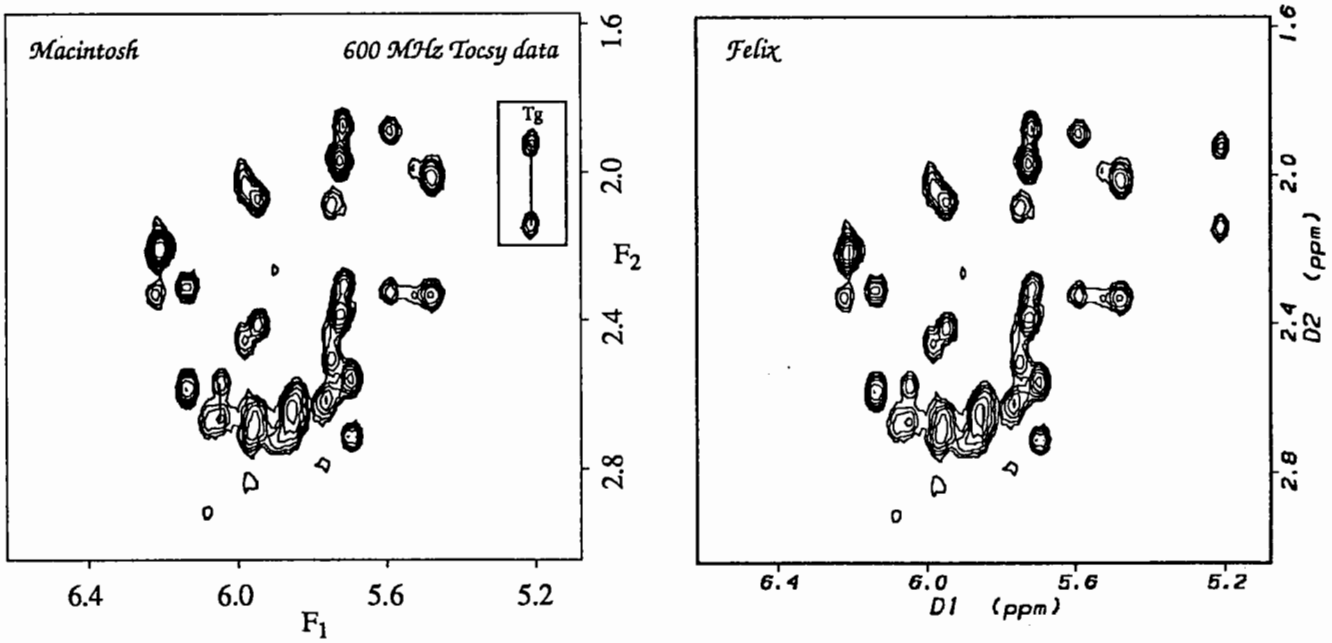
From Felix to Mac

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

Tuesday, July 14, 1992
(received 7/20/92)

Dear Barry:

We have recently begun to use Felix to process NMR data. While we have found the 2.05 version to be quite useful the plots can not be readily annotated and the HPGL format gives rise to rather poor quality letters and labels. To convert the Felix output into Macintosh format we capture the HPGL output file(s); use a network to take the files to an IBM compatible; the HPGL files are converted into Adobe Illustrator (our personal favorite drawing program) format files by the program Corel Draw (Corel Draw and the other available PC converters can take HPGL into PICT and other formats suitable for use in MacDraw and so on. There are no Macintosh HPGL interpreters that we are aware of.); and finally put the files on a Macintosh. This allows the spectra to be annotated by the usual Macintosh procedures. A comparison is given below, at the size of a typical *Biochemistry* figure, for data on a DNA duplex which contains a single thymine glycol whose H1'-H2' and H1'-H2" connectivities are in the box.



Sincerely,

JungYie Kao

Philip Bolton

Department of Pure and Applied Chemistry
University of Strathclyde
Glasgow G1 1XL Scotland UK

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

(received 7/17/92)
11th July 1992

NMR Spectra of δ-Toxin and Analogues Delimit Helical Structures

Dear Barry,

δ-Toxin (1) is a 26-amino acid peptide from *Staphylococcus aureus* which can adopt an amphilphilic structure and form cation channels consisting of a hexameric cluster of helices.

```
M  A  Q  D  I  I  S  T  I  G  D  L  V  K  W  I  I  D  T  V  N  K  F  T  K  K
1           5              10              15              20              25
```

(1)

To investigate the effects of modifications to the structure on the propensity to form helices, δ-toxin itself and six analogues were prepared by solid state synthesis. The analysis of the nOe results (NOESY, plus DFQ-COSY and TOCSY) was done initially using DGEOM [1]. Further refinement of initial structures was done using the molecular mechanics program PIFF [2]. The results for δ-toxin itself confirm the helical structure found by Tappin [3] for the natural material; the length of the helix being somewhat solvent dependent. The extent of the helices in the analogues is shown in the table.

Compound	**α-Helical Region***	**Comments**
δ-Toxin (a)	Thr^{8}–Lys^{22}	
δ-Toxin (b)	Gln^{3}–Lys^{26}	
Ac-δ-toxin (b)	Met^{1}–Lys^{22}	
Glu^{7}-δ-toxin (a)	Thr^{8} or Ile^{9}–Lys^{22} or Phe^{23}	
Glu^{8}-δ-toxin (a)	Glu^{8} or Ile^{9}–Lys^{22} or Phe^{23}	
Pro^{14}-δ-toxin (a)	Trp^{15}–Lys^{22}	Bend preceding helical region
des Lys^{14}-δ-toxin (b)	Ser^{7}–Lys^{22}	
Glu^{8},Pro^{14}-δ-toxin (b)	No well-defined helix	

* (a) solvent=CD_3OH; (b) solvent=CD_3OH/H_2O (2:1)

[1] J. M. Blaney G. M. Crippen, A. Dearing, and J. S. Dixon. *QCPE Bulletin*, 1990, **10,** 37. (Program 590).
[2] PIFF was written by Dr. Armin Widmer of Sandoz AG. Basel, Switzerland.
[3] M. J. Tappin, A. Pastore, R. S. Norton, J. H. Freer, and I. D. Campbell. *Biochemistry,* 1988, **27,** 1643.

Yours sincerely,

Christine Bladon Peter Bladon John Parkinson

Christine Bladon* Peter Bladon John Parkinson*

* Department of Chemistry, University of Edinburgh, West Mains Road, Edinburgh, EH9 3JJ

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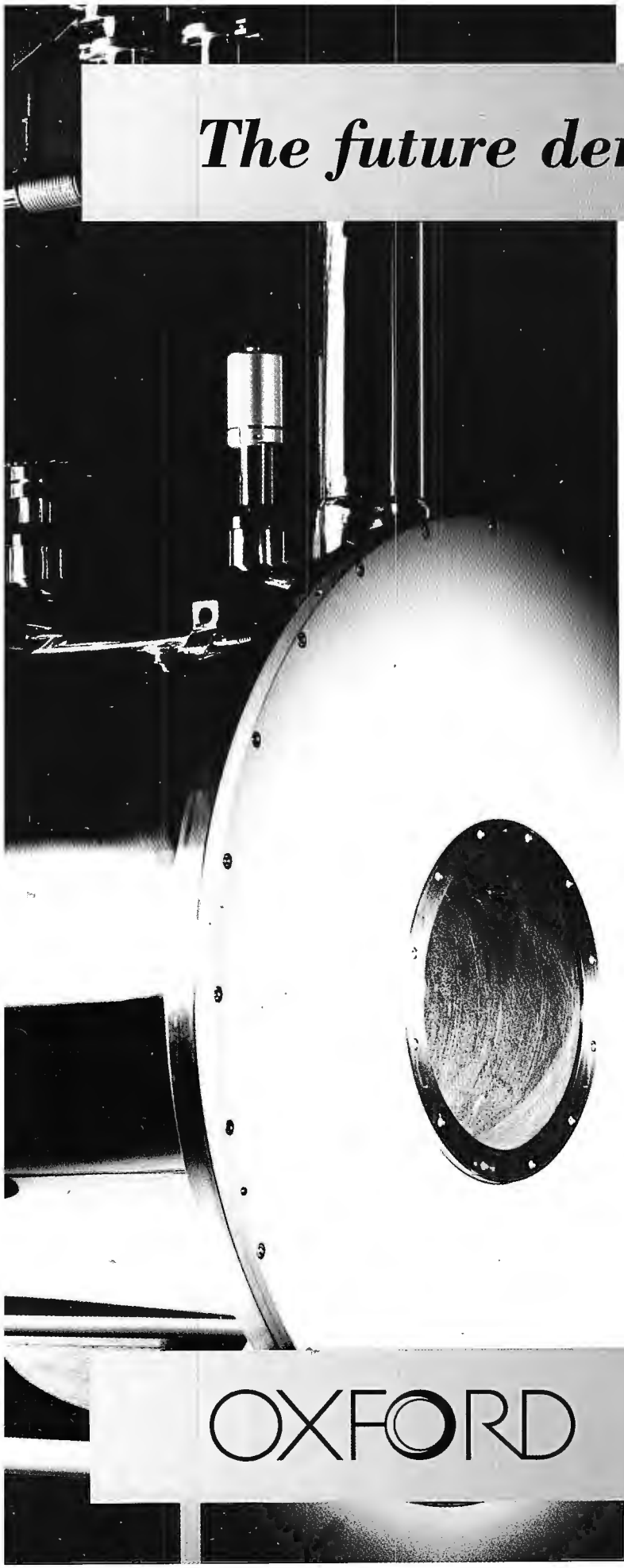

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DEPARTMENT OF HEALTH & HUMAN SERVICES
Public Health Service

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

July 1, 1992
(received 7/9/92)

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

Dear Barry:

Title:Protein-Receptor Interations at 600 MHz

As usual it takes your reminder notice to prompt us into submission (pun intended). We have recently taken delivery of our new AMX600 and AMX360. Our AMX600 will be used primarily to study proteins in solution whereas the AMX360 will be used to study peptides and small proteins bound to membrane surfaces and large proteins. Following the example of Kurt Wüthrich, we have installed the 600 magnet on a vibration table in a pit which is 5 feet deep. Indeed shimming is very easy. Furthermore, we see no effects of movement of metal objects in the corridor approximately 10 feet from the magnet.

We have started studies of some DNA binding proteins in the free and in the bound state. Some of these proteins represent fragments of the immunodominant region of the envelope glycoprotein gp120 of HIV-1, the etiologic agent of AIDS. Until now we have found that the AMX is capable of performing any pulse sequence we have tried. The phase stability of the spectrometer is very good and t1 signal/noise is approximately 800:1. Water suppression is spectacular and we use a software procedure developed by Ad Bax to subtract away low frequency oscillations from the FID.

We are also initiating the study of peptide antigens bound to monoclonal antibodies using specific site enrichment. In this context anybody interested in a postdoctoral fellowship at NIH to work on these or related projects should write to me and include a cv and the names of three references.

Please credit this subscription to Bob Highet.

Best regards,

James A. Ferretti
Laboratory of Biophysical
Chemistry
Bldg. 3, Room 418

CALIFORNIA INSTITUTE OF TECHNOLOGY

DIVISION OF CHEMISTRY AND CHEMICAL ENGINEERING
THE CHEMICAL LABORATORIES

July 10 1992
(received 7/17/92)

Dr. Bernard Shapiro:
TAMU NMR Newsletter
966 Elsinore Ct.
Palo Alto, CA 94303

Conformational Effects on ^{13}C Shifts of Nucleotide Sugars

Dear Barry:

In our continuing studies of oligonucleotides with heteronuclear NMR we have been finding distinct deviations in ^{13}C shifts of the sugar moieties of DNAs that adopt alternative structures compared to B DNA. This has been particularly pronounced in studies of so called G-tetrads which have planar associations of four G bases. Models of these systems from dimeric association of two oligomers, GGTTTTCGG, and GGTCGG, respectively have been examined. From ^{1}H NMR data the sequential ^{1}H assignments have been obtained in Dinshaw Patel's lab, and it has been established that the G bases in a tetrad plane alternate between *syn* and *anti* orientations relative to their respective sugars for these models. In B DNA only the *anti* orientation is present. The change in orientation of the base relative to the sugar is expected to effect the sugar conformation. Using the ^{1}H assignments and ^{1}H detected ^{13}C 2D NMR methods, the carbon shifts of the sugar carbons in these tetrad systems have been examined. The C1′ carbons for the G residues in the *syn* orientation resonate at distinctly lower field, between 89.3 and 90.0 ppm from TMS, than do those associated with *anti* bases at 83.6 to 85.1 ppm. The trend extends to the C3′ and C4′ carbons as well with the *syn* C3′ sites between 80.5 and 81.8 ppm relative to the *anti* site values between 72.8 and 78.1 ppm and C4′ *syn* sites 89.0 to 89.5 ppm relative to *anti* at 87.1 to 88.7 ppm. The shifts cannot be due to any significant degree to ring current effects induced by reorientation of the bases since the ^{1}Hs attached to these carbons show no major comparable trends. The shifts for C1′ and C3′ carbons with *syn* bases are distinctly lower than those reported for respective sites in several studies on B DNA. Although we have been unable to correlate these shifts to a specific sugar pucker as yet, clearly these shifts can be used as convenient markers for significant changes in the sugar conformation.

Sincerely yours,

David Live

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Triple Resonance Indirect Detection: ^{1}H, (^{13}C, ^{15}N)				IDT300	IDT360	IDT400	IDT500	IDT600
Microsample Indirect Detection				MID300	MID360	MID400	MID500	MID600
Broadband ^{15}N-^{31}P Observe	BB200	BB250	BB270	BB300	BB360	BB400	BB500	BB600
Dual Broadband ^{1}H/^{15}N - ^{31}P Switchable	DB200	DB250	DB270	DB300	DB360	DB400	DB500	DB600
Dual ^{1}H/^{13}C Switchable	D200	D250	D270	D300	D360	D400	D500	D600
^{1}H/^{19}F Observe BB Decoupling of ^{1}H or ^{19}F	H-F200	H-F250	H-F270	H-F300	H-F360	H-F400	H-F500	H-F600
^{1}H/^{19}F Observe	HF200	HF250	HF270	HF300	HF360	HF400	HF500	HF600
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Equipment Needed. We need a V-4230B sample probe or other type that is compatible with a V-4210A variable frequency rf unit. Also needed is a probe to sense changes in the magnetic field between the magnet's pole pieces and send a signal to the flux stabilizer to stabilize the magnetic field. The magnet is a Varian electromagnet, model V-3800-1. If you have either one of these to spare, call Dr. D. W. Sheppard at (304)-473-8023 or write him at West Virginia Wesleyan College, Box 68, Buckhannon, W. Va. 26201.

We are looking for a fluorine or proton 5mm probe for a Nicolet NT300 narrow bore system with an Oxford magnet. The system was purchased in 1980. It is not necessary that the probe be in working condition. Please contact Maria Palasis at the University of Cincinnati, Department of Chemical Engineering, 697 Rhodes Hall ML 171, Cincinnati, Ohio, 45221 (513) 556-2761.

NMR Spectroscopist in the Molecular Spectroscopy Laboratory, School of Chemical Sciences, University of Illinois-Urbana. Requirements for this position include a Ph.D. in chemistry or extensive equivalent experience with state-of-the-art NMR spectrometers; a thorough familiarity with modern FTNMR spectroscopy; and the ability to work independently and collaboratively in a stimulating research environment. Experience in devising or implementing new FTNMR experiments (e.g., new 2D NMR pulse sequences), a working knowledge of organic chemistry, as well as experience with General Electric (Nicolet) and Varian FTNMR instruments (General Electric GN500, Varian Unity 400) is desired. Duties will include the supervision of instrument operators and the instruction of graduate students and post-doctoral fellows in NMR techniques.

The starting date is January 6, 1993, but other starting dates could be negotiated. This is a regular, full-time position. The starting salary is commensurate with experience. To ensure full consideration, submit resume and three letters of recommendation by November 4, 1992 to Dr. Vera V. Mainz, School of Chemical Sciences, Box 34 Noyes Laboratory, University of Illinois, 505 S. Mathews Avenue, Urbana, IL 61801 (Tel: (217) 244-0564). The University of Illinois is an affirmative action/equal opportunity employer.

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Postdoctoral Positions Available

Two postdoctoral positions are available at the MRS Unit, VA Medical Center and the University of California San Francisco for the development of clinical applications of *in vivo* MR spectroscopy and spectroscopic imaging.

I. A **scientific programmer** is being sought for the development of spectral data analysis routines and image processing and display software. Applicants should have a strong background in mathematics, statistics, engineering or related field, with experience in scientific computer programming. Knowledge of UNIX and X-windows is desirable.

II. A **MR spectroscopist** position is available to carry out clinical MR studies of Alzheimer's and other neurodegenerative diseases. Applicants should have a background in Chemistry, Biochemistry, Physics or related field, and preferably have some experience with MR imaging or *in vivo* MR spectroscopy.

These positions will continue with ongoing research projects using spectroscopic imaging, while the development of independent research projects is also encouraged. Applicants should send resume and names of references to:

Michael Weiner, M.D.,
MRS Unit, DVA Medical Center
4150 Clement St, (11M)
San Francisco, CA 94121.

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July 15, 1992
(received 7/16/92)

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

In Vivo Microimaging Methods for Mouse Studies at 9.4 Tesla

Dear Barry:

We presently are developing *in vivo* imaging methods using mice in our 9.4T 89-mm vertical bore spectrometer, which is otherwise occupied with protein and peptide structural projects. Some of our microimaging work was reported recently at a local meeting (1). We previously have described imaging studies of pills (2,3) using this and other NMR systems kindly made available to us by GE NMR Instruments (Fremont, CA). In setting up *in vivo* experiments for mouse studies we wanted to run through various MRI methods to assess their applicability at high field using a 26-mm i.d. bird-cage imaging probe designed for our 9.4T instrument. In particular, because we expected to encounter lower contrast-to-noise ratios due to T1 and T2 leveling at high field, we were interested in applying the technique of magnetization transfer contrast (MTC) to regain some of the lost contrast (4). We report herein the results of a few of our experiments.

Figures 1 and 2, obtained with a standard proton-density spin-echo imaging sequence, illustrate the spatial resolution attainable using our instrument. Image 1 is a transverse slice of the lower abdomen of a healthy mouse. The bright spot at top right is a water-filled 1-mm capillary serving as a slice-location marker. Note how easily the marrow of the toe bones can be resolved. The smallest structures easily identified in the image are about 100 microns across. Image 2 is an expanded sagittal view of a mouse kidney in which vascular components, cortex, and medulla are readily visualized.

The MTC technique involves irradiation of soft-tissue protons which are in chemical and/or magnetization exchange. Our first *in vivo* implementation of this technique at 9.4T was on a healthy C57BL/6 mouse. Figure 3A is a normal ^1H NMR spectrum (i.e., from a simple one-pulse sequence, no gradients employed) showing clear resolution of the water and fat resonances. Figure 3B is the spectrum obtained with the same sequence except for the incorporation of low-power irradiation offset 10 kHz from the water resonance. The decrease in signal intensity of the water resonance with respect to the fat resonance corresponds to the transfer of magnetization from immobile protons (i.e., membrane-, protein-bound) to mobile water protons. The fat protons do not exchange magnetization with macromolecular-associated protons, hence the fat signal is unaltered by off-resonance saturation.

The influence of magnetization transfer on image contrast varies for different physiological compartments according to the degree of ^1H-^1H dipolar relaxation enhancement as well as the number of macromolecules contributing to the chemical exchange between pools of restricted and free protons. Thus, tissues which have higher overall exchange rates will show diminished signal intensity relative to those which have slower exchange rates. By using a spin-warp imaging sequence modified to include a very short period of off-resonance presaturation (5), we have demonstrated the effect of this exchange phenomenon on image contrast. Figure 4 is a gradient-recalled-echo (GRE) image which shows good resolution of the kidneys, spinal cord, and cecum, as well as other features of mouse anatomy. Figure 5 is an MTC GRE image scanned at the same transverse slice position. The effect of MTC is dramatically portrayed by the virtual disappearance of signal from water protons in magnetization exchange with macromolecular protons present in the cerebrospinal fluid and the kidneys. The extreme reduction in signal intensity observed for these regions reflects a predominance of short-T2 components in those tissues that are able to transfer the saturation.

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Perkin Elmer Sigma 2000 with HS-100 auto-injector
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Tekmar LSC II Purge and Trap
Tracor 540 with ECD, FID
Varian 6000 FID, FPD capillary and packed column with model 8000 autosampler

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Dionex 2000i/sp ion chromatograph
Hewlett Packard diode array detector 1040M + HP 9000 (9843A), 20 Meg drive and printer
Perkin Elmer LC-75 UV detector
Perkin Elmer LC-85 UV detector
Varian Varichrome UV detector
Varian 5060 + UV-100 and Vista 401
Waters 6000A pump
Waters 440 detector
Waters 441 detector
Waters 710B wisp Autosampler

MASS SPECTROMETERS:

Finnigan 4021
Finnigan 1020/1050
Hewlett Packard 5987 with 5890 GC with RTE-6 operating system

UV-VIS:

Bausch & Lomb Spectronic 2000
Beckman DU-7
Hitachi 100-80
Perkin Elmer Lambda 3B
Varian 2290

ATOMIC ABSORPTION:

Instrumentation Labs 457 with D2 and 440 AVA
Jarrell Ash 951 with D2
Jarrell Ash 655 Atomizer
Jarrell Ash EDL Power Supply
Jarrell Ash Smith-Hieftje Video 12E with 188 Furnace Atomizer and 440 AVA
Leeman Labs Plasma Spec III simultaneous and sequential
Perkin Elmer EDL Power Supply
Perkin Elmer 403
Perkin Elmer 373 with D2
Perkin Elmer 560 with D2
Perkin Elmer 703 with D2
Perkin Elmer 2380 with printer
Perkin Elmer 5000 with D2
Perkin Elmer HGA-400
Perkin Elmer HGA-500
Perkin Elmer HGA-2200 furnace
Perkin Elmer AS-40 Autosampler
Perkin Elmer AS-50 Autosampler
Perkin Elmer Data System 10 for AA
Varian 875 with graphite furnace and computer

DISPERSIVE IR:

Beckman Acculab 2
Perkin Elmer 1330 Perkin Elmer 297

FT-IR:

Digilab FTS-15B +10 cm-1 option
Nicolet 20SX Nicolet 20DX

RESONANCE SPECTROMETERS:

Bruker WM-360 NMR
Bruker CXP-200 NMR
JEOL FX-90Q NMR
Varian CFT-80 NMR
Varian EM-360L NMR

ELECTRON MICROSCOPES:

Cambridge 120B SEM
Hitachi H-300 TEM
ISI SS-60 SEM + Kevex 8000 III EDS
ISI 100
JEOL 35C SEM
Philips 400 STEM with EDS

NUCLEAR INSTRUMENTS:

Beckman 5000 scintillation counter
Tracor Gamma Trac 1290

THERMAL ANALYZERS:

Dupont 1090 with 910 DSC

BIOTECHNOLOGY

Applied Biosystems (ABI) Protein Sequencer 477A with 120A
ABI Peptide synthesizer 430A
ABI Analytical HPLC 150A
ABI DNA synthesizer 380B-02
ABI Separation system 130A
ABI HPLC 151A
Beckman J2-21M Centrifuge
FTS Systems freeze-dryer

MISCELLANEOUS:

A.O. model 820 microtome
Aminco SPF-500 spectrofluorometer
Best Power 3KVA UPS
Bicron M92, M95, M86 surveyors
Blue M deluxe floor oven with Touch-Matic controls
Hiac Royco 4100 with 3200 sampler 346 BCL sensor
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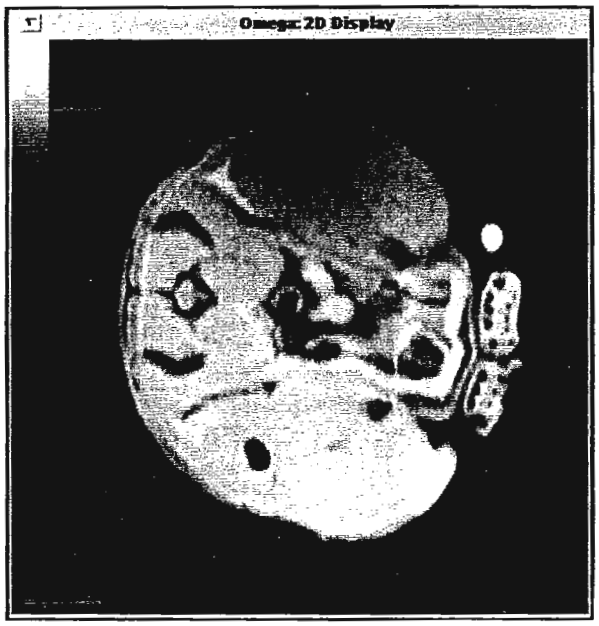
Figures

1



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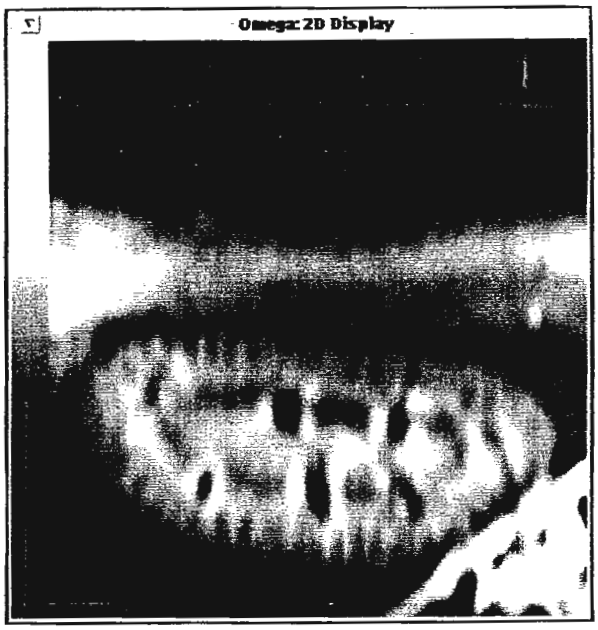

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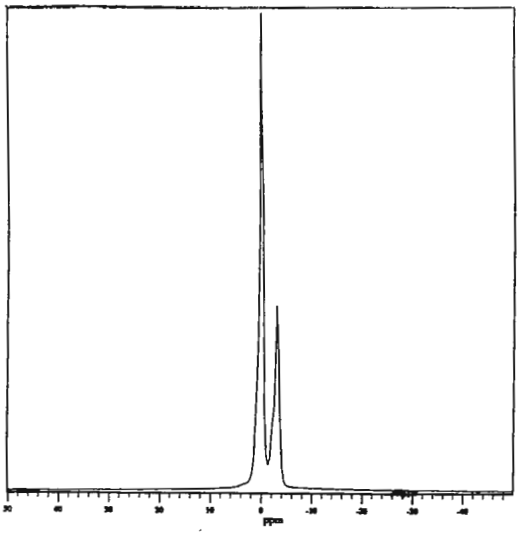

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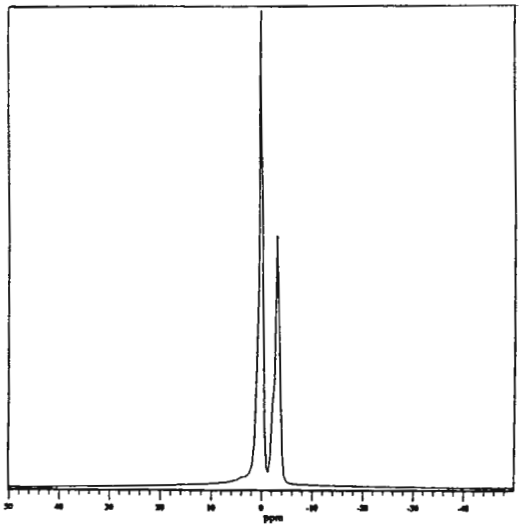
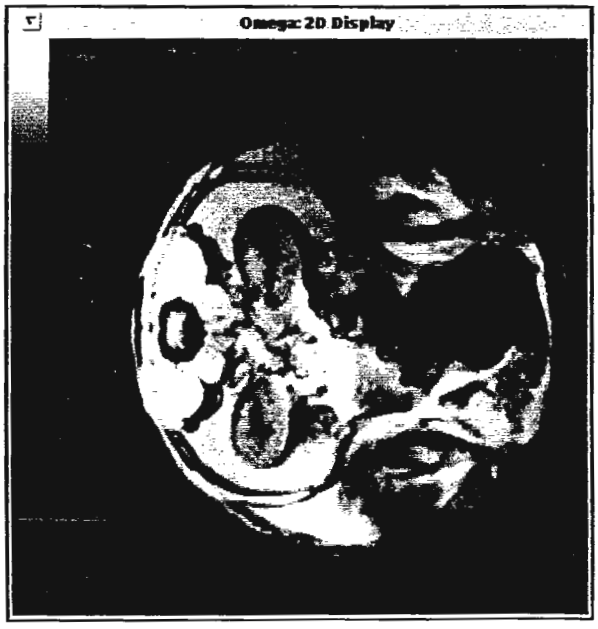


4



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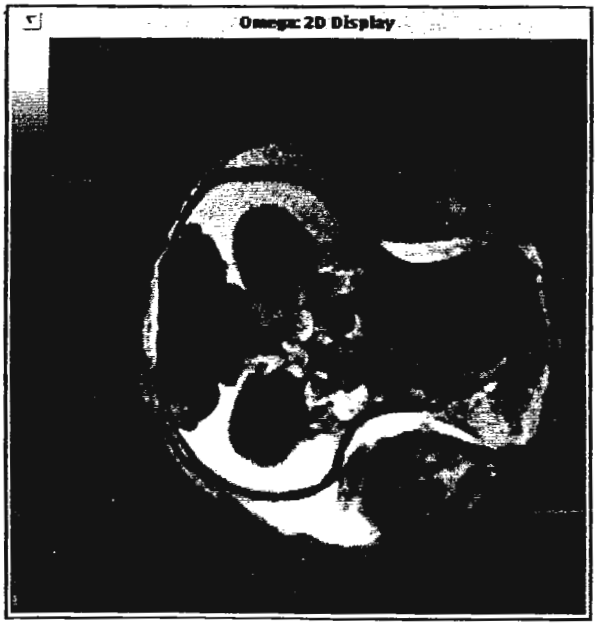


We have carried out a number of other standard MRI experiments (e.g., FLASH, EPI, ISIS) in addition to those described here. In general, we find that high-field imaging gives high-quality results with excellent tissue discrimination. In fact, the predicted loss of contrast due to T1 and T2 leveling is less severe than we had feared. Encouraged by these results, our immediate objective is to use our 26-mm i.d. RF coil to explore *in vivo* magnetic resonance spectroscopy and its applications to small animal models.

Sincerely,

Virginia L. Iuorno T. M. Chan C. Anderson Evans

---

(1) VL Iuorno, TM Chan, and CA Evans. **Microimaging methods and strategies for *in vivo* studies at 9.4 Tesla.** The Fourth Annual Poster Symposium of the North Jersey American Chemical Society Magnetic Resonance Topical Group, Princeton, NJ, May 1992.

(2) RE Hurd, F Alvarez, and CA Evans. **High field (9.4T) short TE microimaging: Application to water absorption in tablets.** The 31st Experimental NMR Conference, Asilomar, CA, April 1990.

(3) M Ashraf, VL Iuorno, D Coffin-Beach, CA Evans, and LL Augsburger. **A novel NMR imaging method for measuring the rate of penetration of the water front in hydrophilic polymer matrix capsule plugs and its role in drug release.** Submitted to *Pharmaceutical Research.*

(4) SD Wolff, J Eng, and RS Balaban. **Magnetization transfer contrast: Method for improving contrast in gradient-recalled-echo images.** *Radiology* 179: 133-137, 1991.

(5) We modified the usa (universal spin-warp acquisition) imaging sequence supplied with GE Omega 6.0.2 software so that a cycle of short off-resonance saturation pulses (20 consecutive sinc pulses each of 1-ms duration) was applied +10 kHz from f1 immediately prior to on-resonance image scanning.

Postdoctoral Research Associate Position Available

The Departments of Biomedical Engineering (Worcester Polytechnic Institute) and Radiology (University of Massachusetts Medical School) are seeking a postdoctoral research associate to participate in *in vivo* NMR spectroscopy and imaging studies. The candidate will have half-time responsibilities as the lead NMR scientist in a project evaluating the efficacy of pharmaceutical interventions in the treatment of stroke using diffusion-weighted imaging and multiple-quantum NMR spectroscopy. For the remaining portion of the appointment, the candidate will be free to participate in a variety of other ongoing projects in the laboratory. The individual will also help supervise graduate students. Qualifications for the position include a doctoral degree in a basic science and hands-on experience with *in vivo* NMR spectroscopy and imaging. Salary is commensurate with experience.

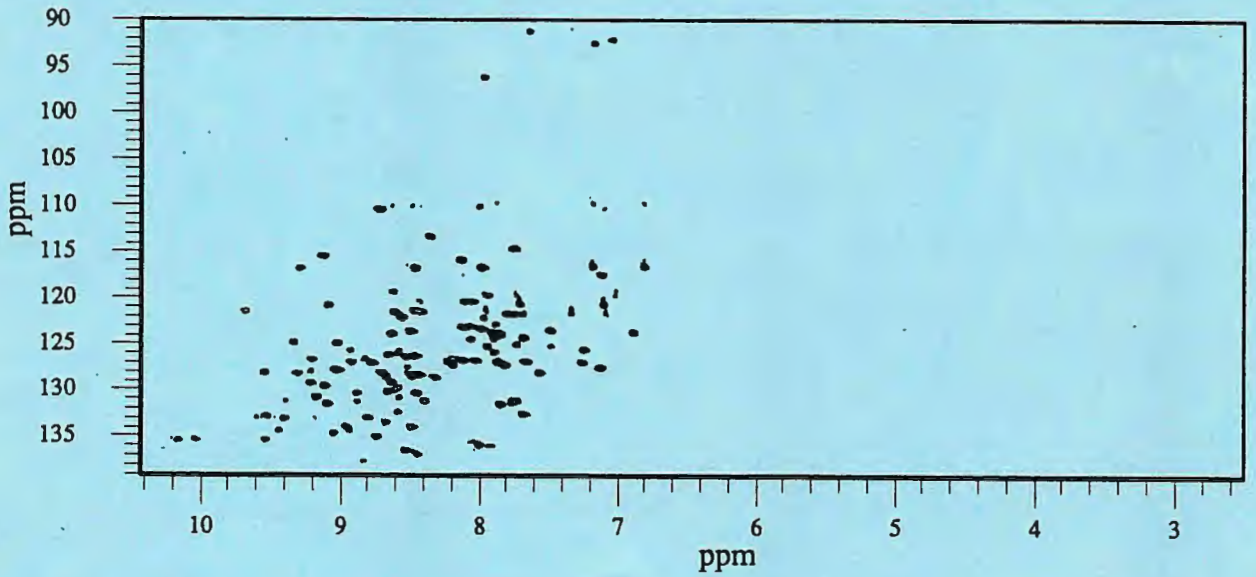
The joint program in NMR spectroscopy and imaging is located in the Massachusetts Biotechnology Research Park, adjacent to the campus of the University of Massachusetts Medical School and a five minute drive from Worcester Polytechnic Institute. Equipment includes a 2.0T/45 cm GE CSI-II imaging spectrometer, exclusively for research, and two, 1.5T GE Signa clinical instruments (one of which is equipped for clinical spectroscopy), available for both clinical and basic research.

Worcester is located less than one hour from Boston. The beaches of Cape Cod, the Rhode Island shore, and Maine are 1.5 hours away, and the best skiing in the East is within 2 hours. Interested candidates should contact Prof. Chris Sotak, Department of Biomedical Engineering, Worcester Polytechnic Institute, 100 Institute Rd., Worcester, MA 01609.

Gradient Techniques for Large Proteins

Inverse detected ^{15}N–^{1}H NMR techniques in conjunction with ^{15}N labelling have proved to be an invaluable tool for assigning the ^{1}H resonances of proteins in solutions. However, the solvent saturation used in the phase cycled versions of these experiments often results in a decreased peak intensity due to exchange processes. Gradient selection of multiple quantum pathways eliminates the water signal and provides an attractive alternative to traditional phase cycled methods for the following reasons:

- ***Superior Water Suppression*** – Single quantum peaks such as the water resonance are eliminated while correlations which occur at the frequency of the solvent resonance are routinely observed. The undesirable effects of traditional presaturation are also avoided.
- ***Fewer Artifacts*** – Gradient selection conveniently avoids the t_1 noise and cancellation artifacts commonly found in phase cycled experiments.
- ***Greater Sensitivity*** – Since the suppression of undesired signals occurs *prior* to acquisition the receiver gain may be increased.



A phase sensitive GE–HMQC spectrum of a ^{15}N enriched protein of approximately 27 kDa (0.1 mM in 90% H_2O). The data were collected on an Omega 500 system equipped with the S–17 Gradient Enhanced Spectroscopy accessory using a 5 mm inverse probe. The S–17 GES accessory is a three–axis actively shielded gradient set. This spectrum demonstrates that gradient techniques can be successfully applied to relatively large proteins in dilute solution.

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α NMR

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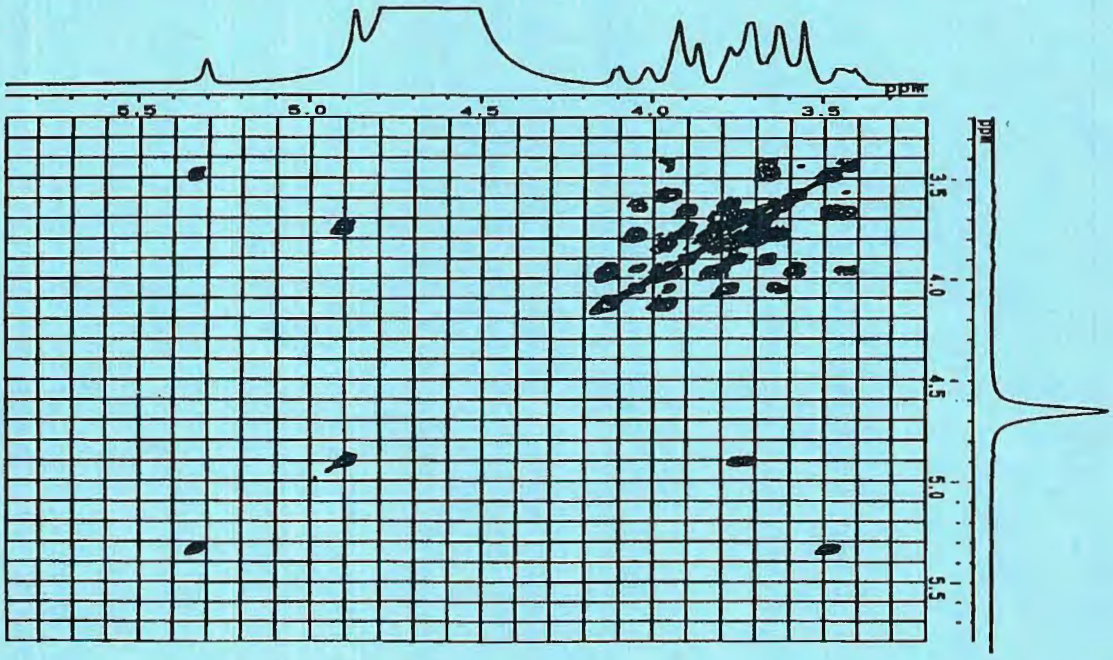

JEOL's gradient probe is tuned to observe ^{1}H with ^{15}N to ^{31}P decoupling and is designed to be used for reverse detection experiments. The above data were collected on an **ALPHA** 500 with the pulsed field gradient accessory. The sample is stachyose in 90% H_2O. By comparing the 1D spectrum plotted at the top with the 2D data, one can see the magnitude of the water suppression afforded by the gradient probe. It is also worthwhile to note that each slice was obtained in one scan and that the double quantum filtering was accomplished with the gradients. This 512 by 512 matrix was obtained in approximately ten minutes.

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