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FORTHCOMING NMR MEETINGS

International Conference on NMR Microscopy, Heidelberg, Germany, **September 16 - 19, 1991**; See Newsletter **385**, 28.

1991 Joint Meeting FACSS/Pacific Conference, Anaheim, California, **October 6-11, 1991**; NMR/EPR Program Section Chairman: Prof. Cecil R. Dybowski, Chemistry Dept., Univ. of Delaware, Newark, DE 19716. Contact: FACSS, P.O. Box 278, Manhattan, KS 66502-0003.

13th Eastern Analytical Symposium, Somerset, NJ, **November 11-15, 1991**; NMR symposia November 12-14; See Newsletter **395**, 43.

Eighth Australian NMR Conference, Lorne, Victoria, Australia, **February 2-6, 1992**; See Newsletter **391**, 38.

33rd ENC (Experimental NMR Conference), Asilomar Conference Center, Pacific Grove, California, **March 29 - April 2, 1992**; Contact: ENC, 750 Audubon, East Lansing, MI 48823; (517) 332-3667

Eleventh Annual Scientific Meeting and Exhibition, Society of Magnetic Resonance in Medicine, Berlin, Germany, **August 8-14, 1992**; Contact: S.M.R.M., 1918 University Ave., Suite 3C, Berkeley, CA 94704; (415) 841-1899, FAX: (415) 841-2340.

Additional listings of meetings, etc., are invited.

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* * * * *

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B. L. Shapiro, 19 July 1991

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. 397 (October) -----13 September 1991
No. 398 (November) -----11 October 1991
No. 399 (December)-----15 November 1991
No. 400 (January 1992) -13 December 1991

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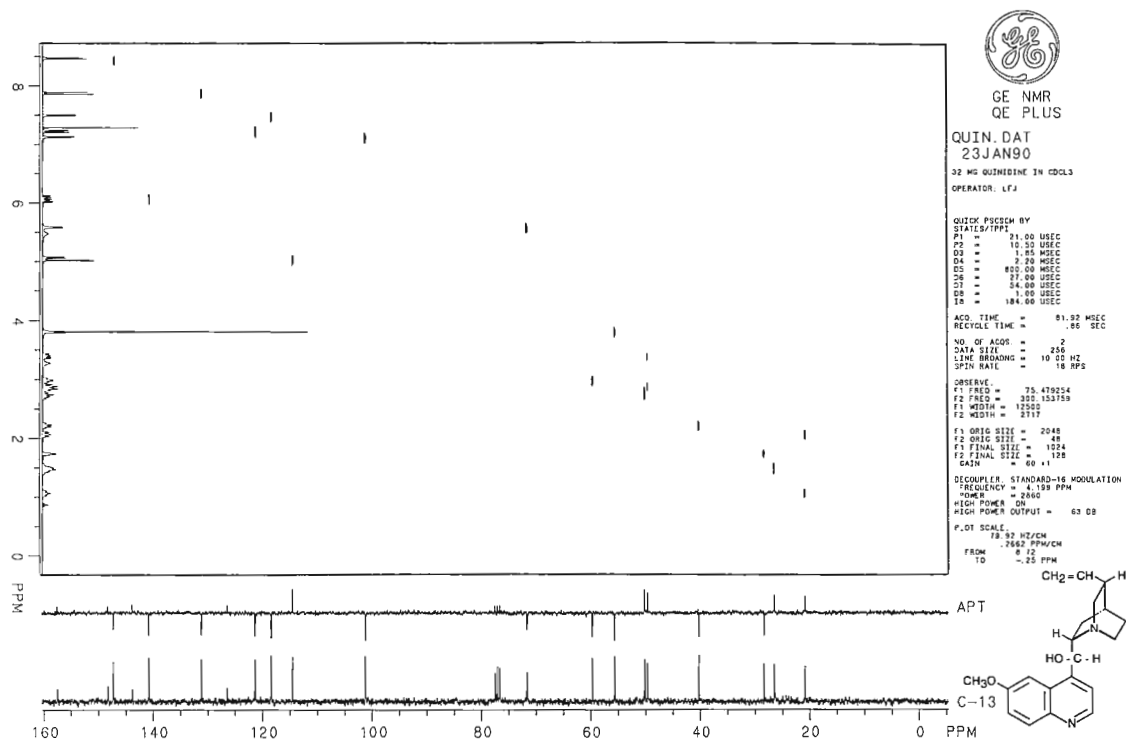
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Dr. B. L. Shapiro
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto, California, 94303

June 21, 1991
(received 6/28/91)

60 Orange Street
Bloomfield, New Jersey 07003
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Re: Measuring α - β spin-spin coupling constants ($^3J_{\alpha\beta}$) from 2D spectra of macromolecules: Is there a simple way?

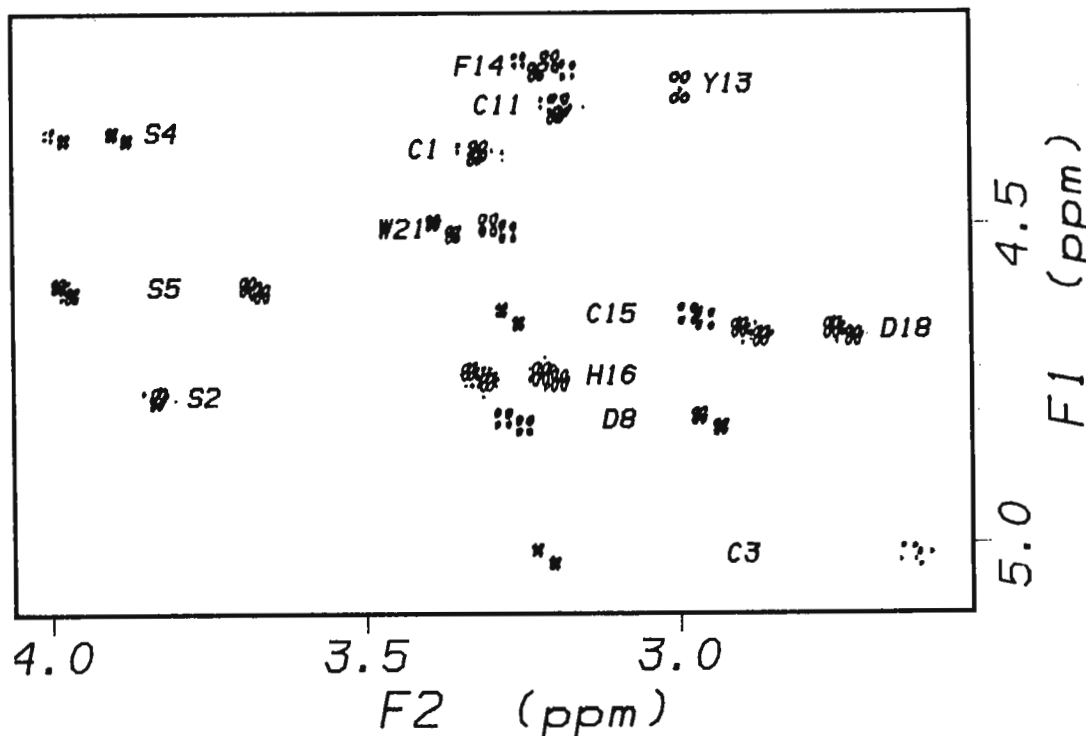
Dear Dr. Shapiro,

An integral part of a protein or peptide structure determination by NMR is the measurement of α - β sidechain coupling constants ($^3J_{\alpha\beta}$), as part of the procedure to obtain stereospecific assignments for amino acid β protons. In structure generation a knowledge of the $^3J_{\alpha\beta}$ values provides additional constraints on the χ_1 torsional value (1). There are two broad approaches for obtaining this coupling constant information from a two dimensional correlation spectrum: simulation of DQF-COSY crosspeaks using coupling and lineshape data (2), and simplification of the cross-peak structure either by combination of MQ-filtered spectra of different orders (E.COSY) (3), or by restricting coherence transfer by using a reduced flip angle mixing pulse together with a diagonal phase purging procedure (P.E.COSY) (4).

Initially, for measuring $^3J_{\alpha\beta}$ values we used the P.COSY method described by Bax et al. (5), where a suitably left-shifted one-dimensional spectrum is subtracted from a two-dimensional set to yield a P.E. COSY type of spectrum. Although this approach provided the desired data, its implementation proved cumbersome, particularly in the processing stages. A simpler approach was suggested by Dr. Luciano Mueller in his presentation at the 1990 Eastern Analytical Symposium (6). A simple COSY spectrum obtained in phase sensitive mode contains dispersive diagonal peaks and absorptive crosspeaks. Total removal of the diagonal peaks can be achieved by applying a digital filtering process which results in a "cross-peak only" COSY spectrum. We have implemented this method on our instruments and would like to describe our initial results here.

A hypercomplex phase-sensitive COSY spectrum with a 30° mixing pulse was obtained using a reduced sweep width to maximize digital resolution. For the spectrum presented in Figure 1, obtained on a GE Omega PSG-500 spectrometer, a phase roll method was used to offset the transmitter for presaturation of the residual water resonance. The dataset was reformatted then processed using FTNMR/FELIX (7). Processing in t_2 proceeds as normal, however before the transform, each column (interferogram) is shifted to place the diagonal at zero frequency using an offset dependent first order phase shift calculated from the t_1 acquisition time. This results in a shifted interferogram which is stored temporarily in a buffer. The shifted interferogram is then smoothed to remove high frequency signals then subtracted from the unsmoothed shifted interferogram. The shifted interferogram was smoothed according to Marion et al. (8) using software modified from that kindly provided by Dr. F. Ni (9) to use linear extrapolation. A reversal of the shifting procedure follows, and the data is stored back into the matrix. The t_1 transform is then performed as usual. The shifting/smoothing procedure is

convenient and fairly rapid. Elapsed time for the diagonal removal procedure is 5 minutes for 2048 columns on a Sun 4/360 system. The results from this diagonal removal protocol are shown below for the peptide endothelin (assignments are labeled).



Experimental setup for this form of COSY spectroscopy is simple, and processing requires only one additional step (diagonal removal), when compared to DQF-COSY. In our experience the major problems with this technique arise from low digital resolution, here 1.6 Hz/point, and the well-known effects of cancellation caused by broad lines in antiphase multiplets.

Sincerely

David
David C. Dalgarno

Mary
Mary M. Senior

Yu-Sen
Yu-Sen Wang

P.S., Please credit this contribution to Andy Evans' account.

- (1) Nilges, M., Clore G.M., and Gronenborn, A.M., *Biopolymers* (1990) **29**, 813.
- (2) Widmer, H., and Wuthrich, K., *J. Mag. Res.* (1986) **70**, 270.
- (3) Griesinger, C., Sorensen, O.W., and Ernst, R.R., *J. Mag. Res.* (1987) **75**, 474.
- (4) Mueller, L., *J. Mag. Res.* (1987) **72**, 191.
- (5) Marion, D., and Bax, A., *J. Mag. Res.* (1988) **80**, 528.
- (6) Mueller, L., "3D and Other Novel Approaches to Protein NMR Spectroscopy", EAS, 1990.
- (7) Hare Research, Woodinville, WA.
- (8) Marion, D., and Bax, A., *J. Mag. Res.* (1989) **84**, 425.
- (9) Ni, Feng, National Research Council of Canada, TAMU Newsletter August 1990.

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June 26, 1991
(received 7/1/91)

Professor Bernard L. Shapiro
TAMU NMR Newsletter
966 Elsinore Court
PALO ALTO, CA 94303
USA

HIGH SENSITIVITY *IN VIVO* NMR OF DILUTE CELL SUSPENSIONS

Dear Professor Shapiro,

Ideal physiological conditions never seem to be met in hitherto reported *in vivo* NMR studies on cell suspensions, in almost all cases very dense cell cultures being used without biomass control that suffer from non-uniform substrate and/or oxygen distribution over the culture volume.

Therefore, we have chosen the way to study *dilute* cell suspensions since it is relatively easy to maintain them in those ideal conditions even over several weeks of time. We must adopt an experimental setup that generates the maximal possible NMR-sensitivity. Therefore, we have developed a new NMR-bioreactor in which an almost complete elimination of saturation effects is achieved: the cell suspension in the sensitive volume of the r.f. coil is continuously replaced by suspension that has resided in a large pre-polarizing chamber for a period longer than $3 \cdot T_1$. The flow rate is adjusted such that the mean residence time t_m of cells in the r.f. coil is not less than $3 \cdot T_2^*$ to avoid severe loss of resolution. Under *in vivo* conditions, this allows for very fast 90° pulsing for utmost sensitivity. The reactor is shown in Fig. 1. It consists basically of a standard 20 mm o.d. NMR sample tube connected to a large pre-polarizing glass chamber of 7 cm diameter. It can be equipped with all monitoring and control apparatus necessary for the cultivation of both aerobic and anaerobic cells. Suspension flow, directed through a central tube, is generated by a pump positioned above the bioreactor lid. The total reactor volume is 375 ml, chosen such that nowhere over the suspension volume the magnetic field strength falls below 90 per cent of the value in the magnet isocentre. A shortened $^{31}\text{P}/^{13}\text{C}$ dual tunable 20 mm probehead is used, constructed by Bruker Spectrospin, Switzerland, to allow for maximum pre-polarizing volume.

To illustrate the high sensitivity of the system, Fig. 2 shows the *in vivo* ^{31}P NMR spectrum of a dilute cell suspension. We estimate that our continuous flow NMR bioreactor enhances sensitivity by a factor of 3 to 4. In addition, the peaks in the sugar phosphate region are better resolved and appear at lower field (indicating a higher intracellular pH) than is commonly seen in conventional spectra of this organism [1].

[1] K.D. Barrow, J.G. Collins, R.S. Norton, P.L. Rogers, and G.M. Smith, *J. Biol. Chem.* 259, 5711 (1984).

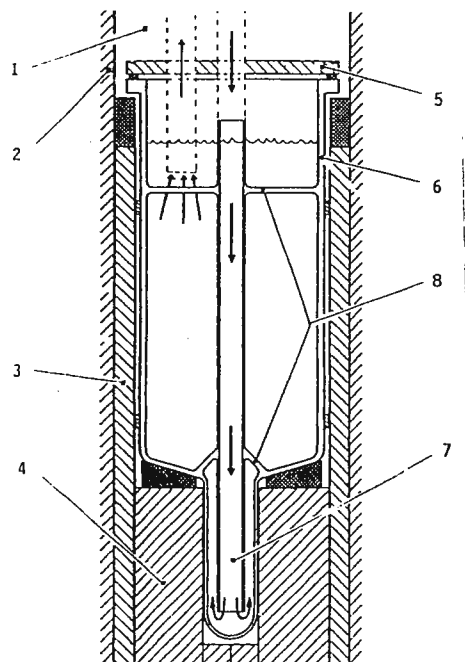


Figure 1

Schematic drawing of the newly designed continuous flow NMR bioreactor. Legend: 1. magnet bore (89 mm); 2. magnet inner wall; 3. lower section of central magnet insert holding the room temperature shim coils and with the turbine part removed; 4. upper part of the NMR probehead; 5. bioreactor lid; 6. bioreactor main section used as pre-polarizing chamber; 7. central 10 mm tube; 8. glass supports for central tube.

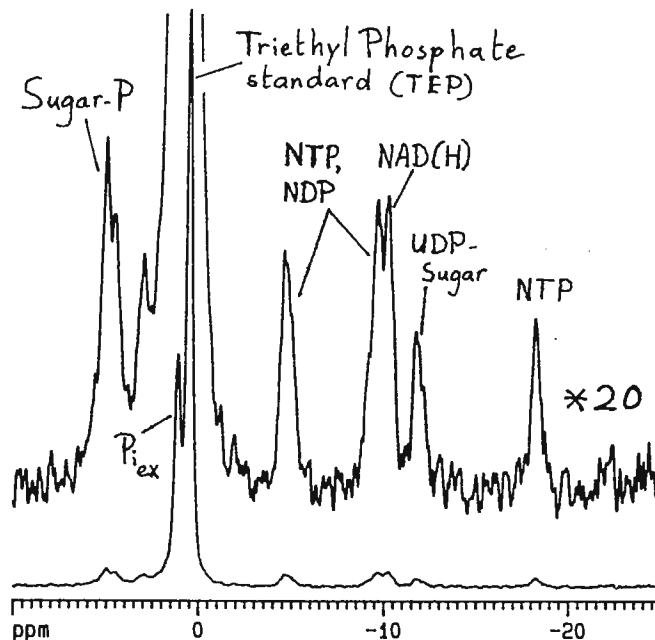


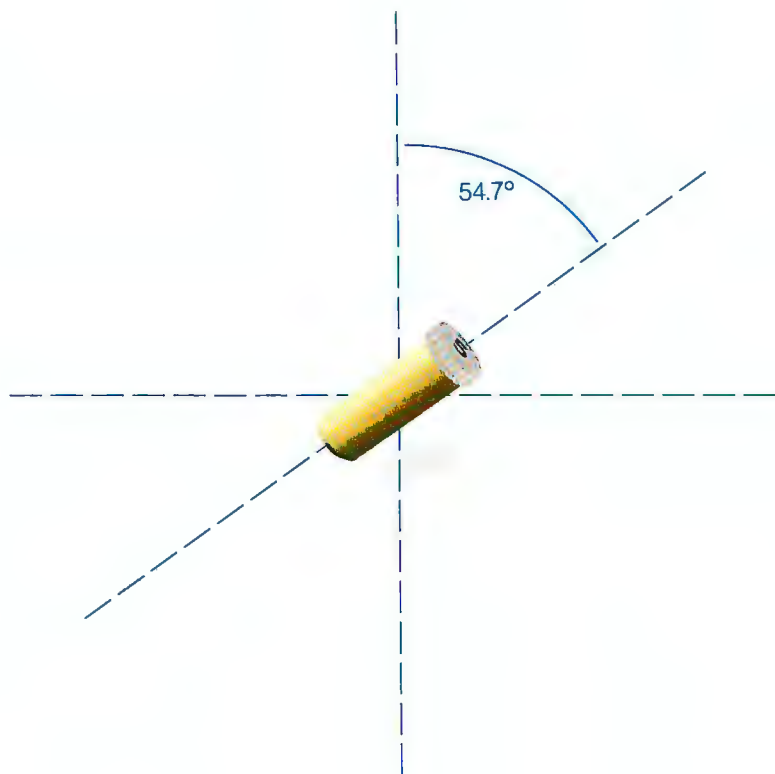
Figure 2

In vivo ^{31}P (162 MHz, Bruker AMX-400 WB spectrometer) spectrum of a dilute suspension (2 mg dw/ml) of fermenting *Zymomonas mobilis* ZM4 bacteria (ATCC 31821), obtained with the continuous flow NMR bioreactor. Peaks of extracellular P_i and TEP appear strongly enhanced relative to intracellular peaks due to the low relative intracellular volume (0.5 %). Experimental parameters: flow 600 l/h, 90° pulses, 25,000 scans, T_r 72 ms (30 min signal accumulation), 30°C , acquisition started 1 minute after addition of 13% glucose.

Sincerely,

Albert A. de Graaf

Rolf M. Wittig



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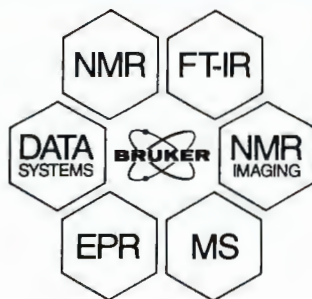


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May 30, 1991
(received 6/27/91)

BRUKNET-DECnet to a PC

Dear Dr. Shapiro:

PC's have sufficient speed and mass storage capacity for archiving spectroscopic data and for off-line data processing. Unfortunately, it is relatively difficult to transfer files from many existing NMR instruments to PC's. The Kermit and FASTRAN protocols that Bruker supports for direct communications between the MSL computer, an ASPECT 3000, and a PC are relatively slow and tedious to use. One alternative that we have found to circumvent this data transfer bottleneck is to connect these nodes over the Research Center Ethernet network. BRUKNET provides ASPECT 3000 to VAX/VMS file transfer capabilities. Files on the VAX are downloaded to the PC with DEC PATHWORKS for DOS which provides a copy utility between a virtual disk on the VAX and the PC's local disk. For small amounts of data the direct transfer with Kermit is considerably simpler than this approach, but for large transfers it is a real time saver.

One problem with the use of BRUKNET is that it adds a header to each packet that is sent over Ethernet which changes the format of the original file. Thus, the drivers that software vendors provide to convert Bruker binary files (e.g. sent via Kermit) to their format will not work on files transferred by BRUKNET. Because we do not always know each vendors data format and because we always want to minimize the number of different data formats on our systems, we have written a MS-DOS utility that strips the header information from BRUKNET files. Once stripped these files are identical to files sent with Kermit and have been successfully tested with WIN-NMR (Bruker) and Spectra Calc (Galactic Industries, Corp).

After the initial time investment was made to treat data files on a PC, other features were added to the program. The Bruker acquisition parameters are available on a text screen and the data can be viewed graphically with PAN and ZOOM capabilities. More recently we have incorporated a feature to read T2 relaxation data and generate time, intensity lists directly from the parameters stored with the files. The output can be written in ASCII and ASYSTANT (Asyst Software Technologies) formats. We would be glad to provide this program to your readers.

p.s. We are expecting delivery in the near future of an AMX spectrometer. Because the Bruker X-32 supports both BRUKNET communications to an ASPECT computer and TCP/IP communications to a PC, we anticipate that the X-32 will soon become the file server for our local area PC network in the NMR laboratory.

Regards,

Mark Sullivan

Wayne Duncan

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Dr. Bernard Shapiro
 TAMU NMR Newsletter
 966 Elsinore Court
 Palo Alto, CA 943030, USA

June, 21th 1991
 (received 6/27/91)

1D NOE $^1\text{H}/^1\text{H}$ SPECTROSCOPY

Dear Dr. Shapiro

Recently we found that the application of a series of selective (Gauss-shaped) 180° pulses at a given frequency (Fig. 1) is a valuable alternative to the usually used techniques with CW irradiation for 1D NOE spectroscopy. The saturation degrees of the protons selected for selective perturbation should be close to the maximum and as uniform as possible in order to achieve highest sensitivity and directly comparable NOE data respectively. On the other hand highest selectivity is a prerequisite for the unambiguous interpretation of NOE data. Excitation windows of CW and pulsed decoupling as incorporated in the basic and the modified pulse sequence have different profiles. Fig. 2 shows the experimentally determined excitation z-profiles for CW irradiation with decoupler powers of 0.08 mW (A) and 0.02 mW (C) respectively and the excitation z-profile of a train of 180° Gaussian pulses (B) of 80ms duration and a pulse interval of 95ms. The profile was recorded by stepping the decoupler frequency in 1Hz increments through resonance. The profile of a train of Gaussian 180° pulses (B) is very similar to the profile of a single Gaussian 180° pulse (not shown) and only differs in the weak additional modulation visible within the shadowed region and a minor broadening of the window. It is characterized by homogeneity close to resonance and a more or less sharp falling off at a frequency determined by the length and the interval of the shaped pulses. This attractive profile contrasts with the profiles of CW irradiations. Whereas for comparable selectivity a rather inhomogeneous profile results (A), wide wings and hence reduced selectivity is obtained on the other hand for similar homogeneity close to resonance (C).

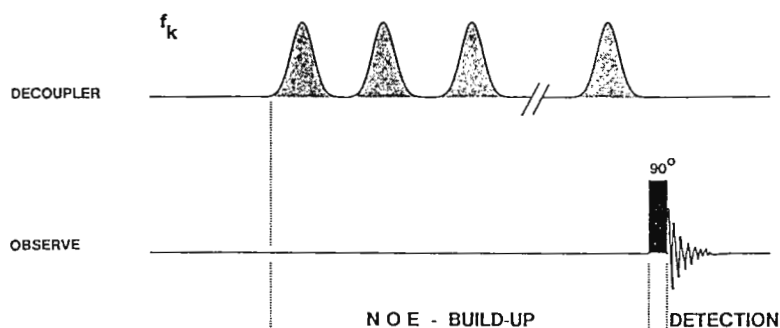


Fig. 1

Yours sincerely
 Dr. P. Bigler

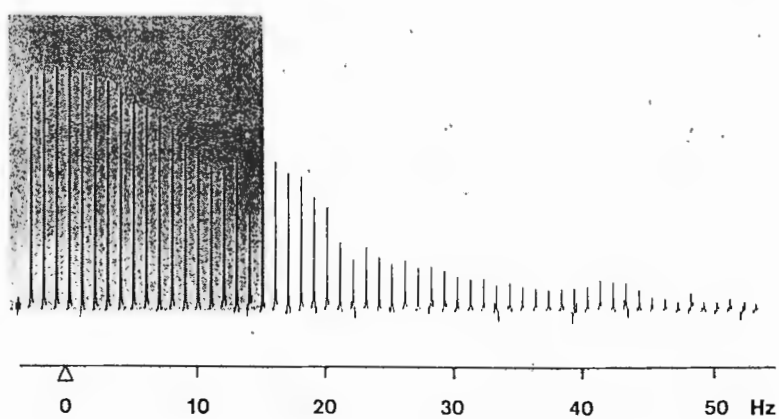
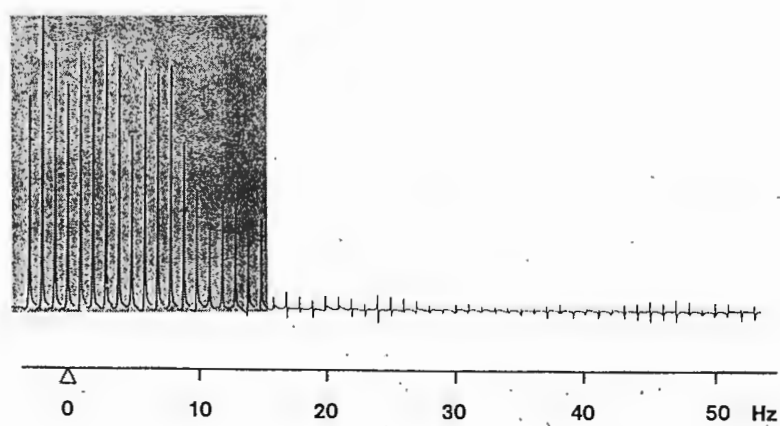
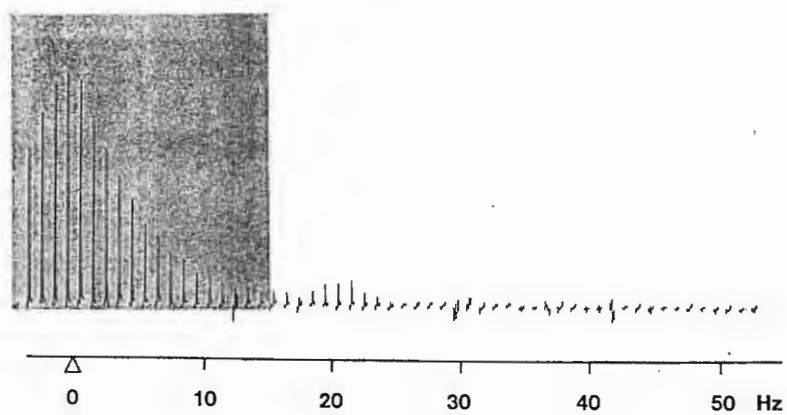


Fig. 2

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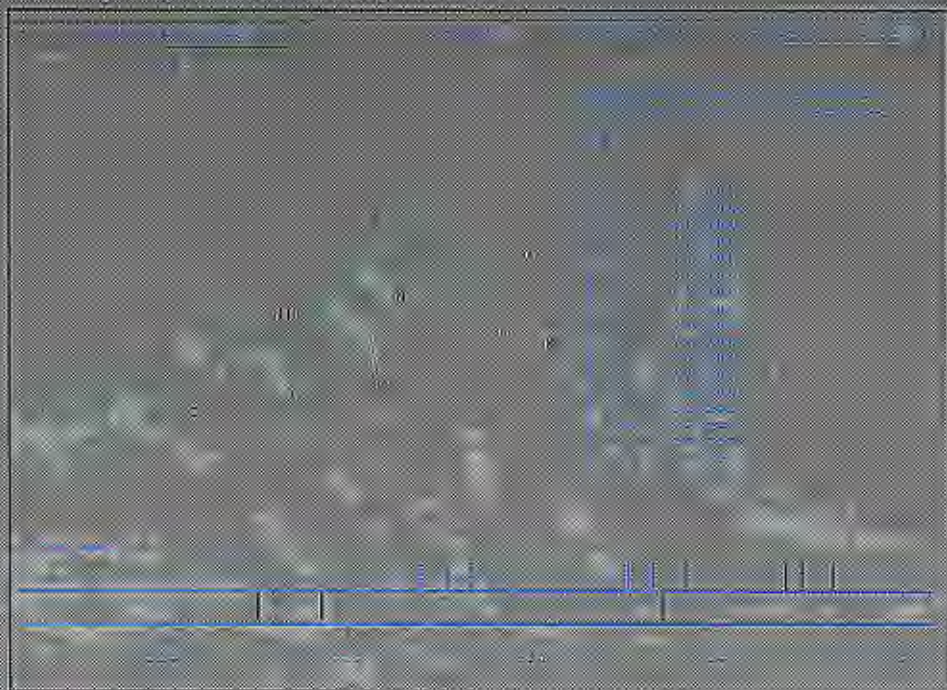
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NMR Research and Development
July 12, 1991
(received 7/15/91)

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

Spectral Editing by Peak Aliasing

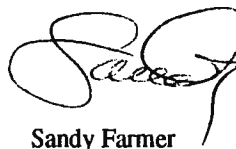
Dear Barry,

Folding (or aliasing) has become increasingly more popular in multi-dimensional heteronuclear NMR experiments for reasons of instrumental efficiency. Bax and co-workers have recently described a technique whereby 2D peaks folded an odd number of times in F_1 have opposite phase to those either not folded or folded an even number of times in that dimension (I). They have applied this technique to a 2D [^1H , ^{13}C]-HMQC experiment on ^{13}C -enriched calmodulin for which the $^1\text{H}_\alpha$ chemical shifts are sufficiently distinct from the sidechain (sc) ^1H chemical shifts that the F_1 folding of the ^1H - $^{13}\text{C}_{\text{sc}}$ peaks does not lead to spectral overlap. In general, such good fortune does not exist.

It is quite easy to perform a 2D experiment as an arrayed experiment in which the second set of FIDs is uniformly incremented in t_1 by half of a dwell time over the first set. Processing the first set, which should be acquired so that no linear F_1 phase correction is needed, yields 2D peaks which all have the same phase regardless of the level of folding in F_1 . Processing the second set with a 180° linear F_1 phase correction yields 2D peaks which have phase $(-1)^m$ where m represents the number of times the peak has been folded in F_1 . If the two 2D spectral data sets are added together, only peaks for which m is an even number are retained; if one 2D spectral data set is subtracted from the other, only peaks for which m is an odd number are retained. Unless more than one level of F_1 folding is used, accidental spectral overlap is not an issue with this method regardless of the experiment or the sample. Figure 1 presents an [^1H , ^{15}N]-HSQC experiment, with ^{13}C decoupling during τ_{nh} and t_1 , applied to [^{13}C , ^{15}N]-enriched human carbonic anhydrase. Figure 1A contains only those 2D peaks which have not been folded in F_1 ($m = 0$); Fig. 1B, only those peaks which have been folded once in F_1 ($m = 1$).

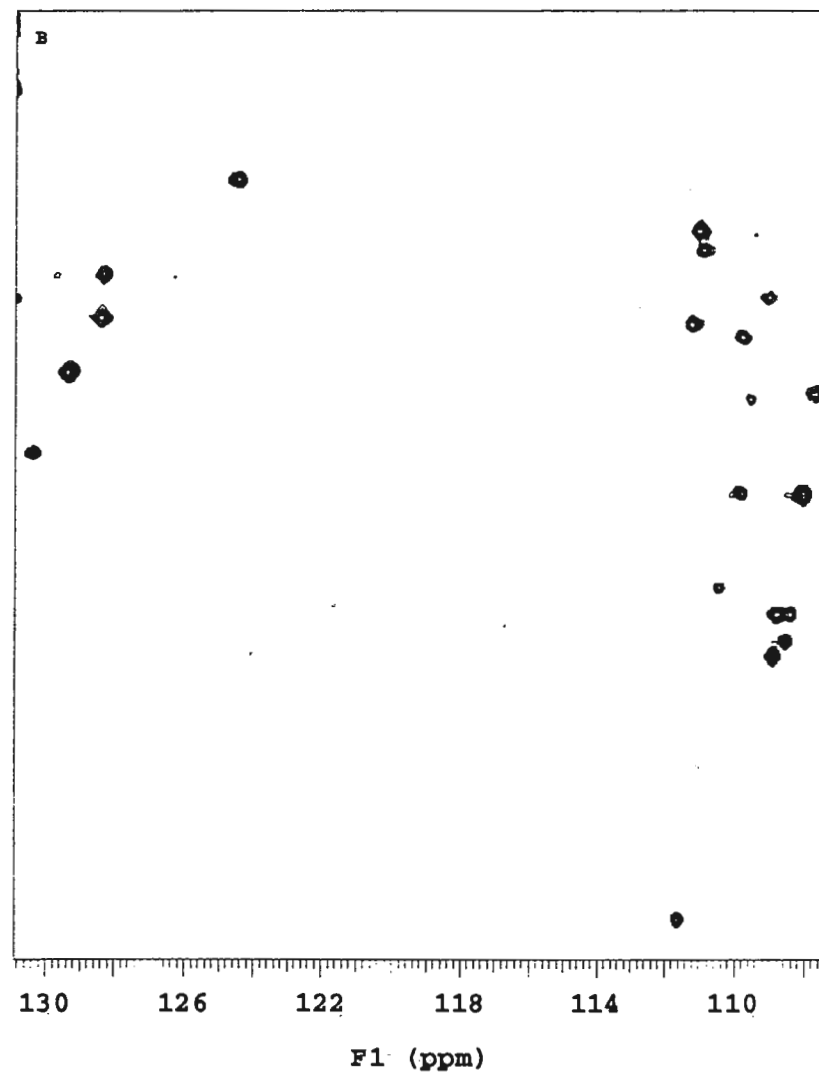
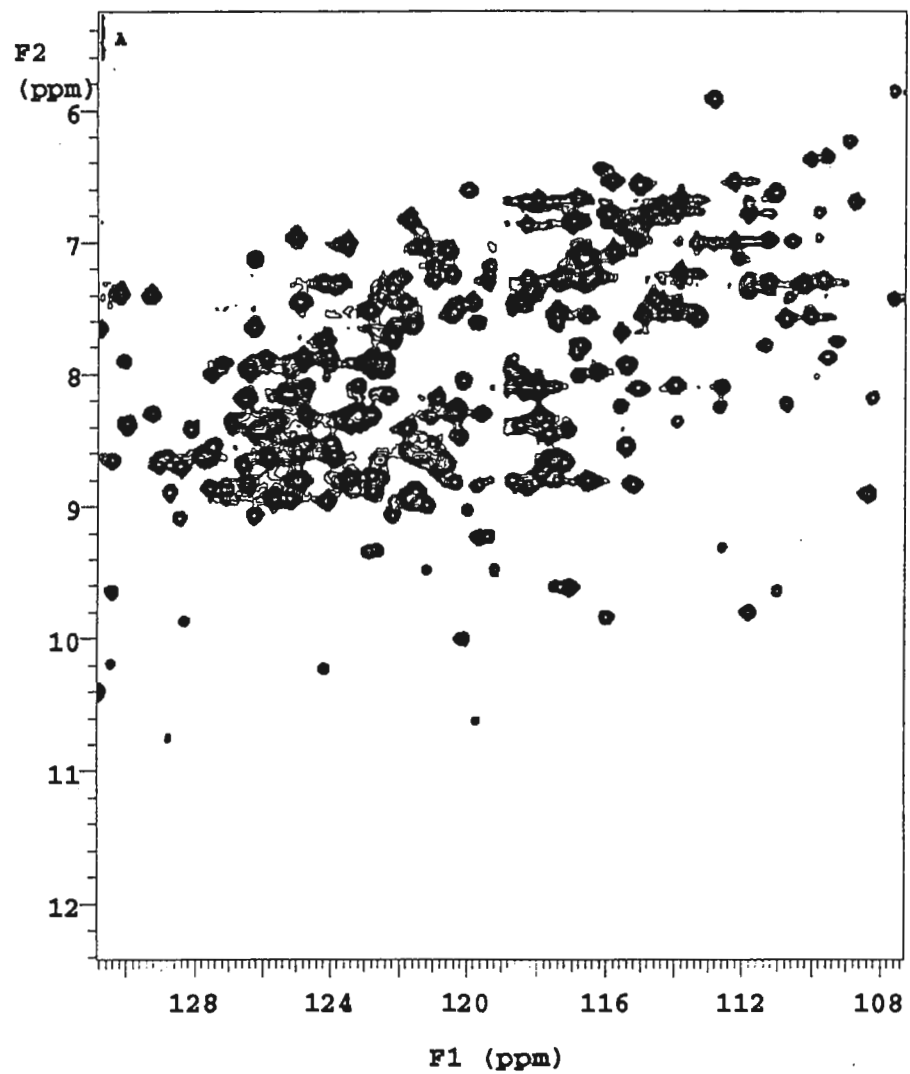
This method is easily generalizable to $n\text{D}$ experiments. For an $n\text{D}$ experiment in which folding is used in the $n-1$ indirect dimensions, $2(n-1)$ sets of FIDs must be collected. There is no loss in sensitivity per unit acquisition time. There is, however, a $2(n-1)$ decrease in the size of the phasecycle which can be used for a given acquisition time. The method will be described in more detail in a forthcoming publication. Sorry, no acronym at this time.

Sincerely yours,



Sandy Farmer

1. A. Bax, M. Ikura, L.E. Kay, and G. Zhu, *J. Magn. Reson.* **91**, 174 (1991).



PTS 040		
Range: 0.1-40 MHz Resolution: 0.1Hz-100KHz (opt) Switching: 1-20 μ s	Output: +3 to +13dBm; 50ohm Spurious Outputs: -75dBc Phase Noise: -75dBc (0.5Hz-15KHz)	Freq. St'd: OCXO, TCXO, Ext. Interface: BCD par. or GPIB Price: \$5,125.00*
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PTS 160		
Range: 0.1-160 MHz Resolution: 0.1Hz-100KHz (opt) Switching: 1-20 μ s	Output: +3 to +13dBm; 50ohm Spurious Outputs: -75dBc Phase Noise: -63dBc (0.5Hz-15KHz)	Freq. St'd: OCXO, TCXO, Ext. Interface: BCD par. or GPIB Price: \$6,245.00*
PTS 250		
Range: 1-250 MHz Resolution: 0.1Hz-100KHz (opt) Switching: 1-20 μ s	Output: +3 to +13dBm; 50ohm Spurious Outputs: -70dBc Phase Noise: -63dBc (0.5Hz-15KHz)	Freq. St'd: OCXO, TCXO, Ext. Interface: BCD par. or GPIB Price: \$7,155.00*
PTS 300		
Range: 0.1-300 MHz Resolution: 1Hz Switching: 1-20 μ s Phase Continuous: 1Hz-100KHz steps	Output: +3 to +13dBm; 50ohm Spurious Outputs: Type 1 -70/65 (typ/spec) Phase Noise: -68dBc (0.5Hz-15KHz)	Freq. St'd: OCXO, TCXO, Ext. Interface: BCD par. or GPIB Price: Type 1 \$6,175.00* Type 2 \$5,625.00*
PTS 500		
Range: 1-500 MHz Resolution: 0.1Hz-100KHz (opt) Switching: 1-20 μ s	Output: +3 to +13dBm; 50ohm Spurious Outputs: -70dBc Phase Noise: -63dBc (0.5Hz-15KHz)	Freq. St'd: OCXO, TCXO, Ext. Interface: BCD par. or GPIB Price: \$8,385.00*
PTS 620		
Range: 1-620 MHz Resolution: 0.1Hz-100KHz (opt) Switching: 1-20 μ s	Output: +3 to +13dBm; 50ohm Spurious Outputs: -70dBc Phase Noise: -63dBc (0.5Hz-15KHz)	Freq. St'd: OCXO, TCXO, Ext. Interface: BCD par. or GPIB Price: \$8,675.00*
PTS 1000		
Range: 0.1-1000 MHz Resolution: 0.1Hz-100KHz (opt) Switching: 5-10 μ s	Output: +3 to +13dBm; 50ohm Spurious Outputs: -70dBc (0.1-500 MHz), -65dBc (500-1000 MHz) Phase Noise: -60dBc (0.5Hz-15KHz)	Freq. St'd: OCXO, TCXO, Ext. Interface: BCD par. or GPIB Price: \$11,275.00*
PTS x10		
Range: 10 MHz band, selected decade 0.1-100 MHz Resolution: 1Hz Switching: 1-5 μ s Phase Continuous: 2 MHz band, even or odd steps	Output: +3 to +13dBm; 50ohm Spurious Outputs: -65/-60dBc (typ/spec) Phase Noise: -70dBc (0.5Hz-15KHz)	Freq. St'd: OCXO, TCXO, Ext. Interface: BCD par. or GPIB Price: \$2,575.00*



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

Prof. J. Lugtenburg
Gorlaeus labs.
Leiden University
Einsteinweg 5, Leiden
The Netherlands

MAKE YOUR OWN ¹H, ¹³C, ³¹P TRIPLE PROBE

(received 7/12/91)

Dear Dr Shapiro,

In this technical contribution we will show you a cheap way of making a triple resonance probe out of a dual probe.

In the old situation we measured ¹H and ¹³C in a 5mm CH-Dual probe and had to change the probe to measure ³¹P in a BB-probe, which occurred more than once daily. Of course the RF-unit and shim-values had to be changed as well. For reasons of convenience we made a modification in our CH-Dual probe, which took our electronic engineer about one morning of work. In the new situation we don't need to change the probe and shim-values. We just pull out or push in a rod and change the RF-unit and we can measure ¹H, ¹³C or ³¹P.

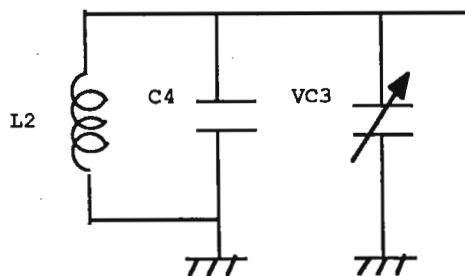
We needed (1) a non-magnetic switch, (2) some capacitors (we used a combination of 30 pF, 3.3 pF and a variable 1-6 pF capacitor, HIGH Q from ATC) and (3) a plastic rod (3mm thick, 38 cm length).

Some technical data:

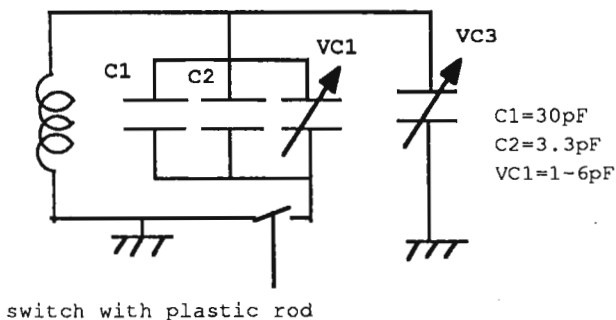
Spectrometer: JEOL FX-200

Frequency		original CH-probe: 90 -pulse	new made CHP-TRIPLE-probe 90 -pulse
¹ H	200 MHz	14.0 μsec	14.0 μsec
¹³ C	50.1 MHz	6.9 μsec	7.8 μsec
³¹ P	80.7 MHz	-	9.2 μsec

Original situation:



New situation:



Starting from the original situation we first removed capacitor C4 and tuned VC3 to 80.76 MHz (³¹P frequency) using a WOBL sweep generator. Then we made a switchable circuit to get resonance at 50.1 MHz

Sincerely:

A.W.M. Lefeber

G. van Kampenhout

C. Erkelens

J. Lugtenburg

21/6/91

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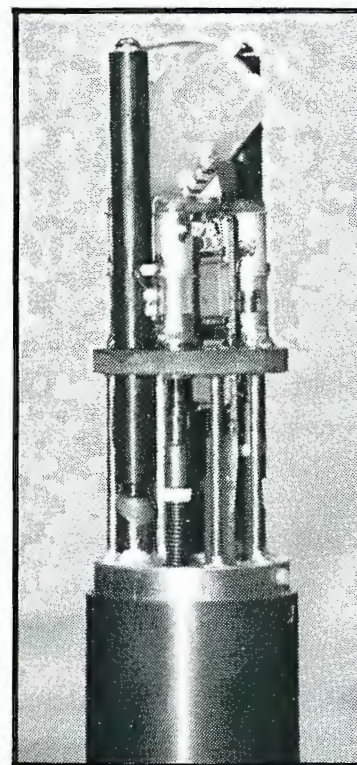
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Sample volume - 70 μ l to 110 μ l

7 MM STANDARD

The 7 mm standard speed spinner is available for supercon probes 40 mm in diameter or larger. It provides the largest filling factor and highest sensitivity of the 7 mm probes.

Spinning speed - 5 kHz to 6 kHz

Sample volume - 200 μ l to 350 μ l

7 MM HIGH SPEED

The 7 mm high speed spinner is available for probes 44 mm in diameter and larger.

Spinning speed - 9 kHz to 11 kHz

Sample volume - 250 μ l to 370 μ l

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Sample volume - 2.8 ml

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

Your reference and date

Our reference

Office telephone

Date

JAP/mk/668

(015) 78 5892

July 4, 1991
(received 7/19/91)

Subject

Sub-division

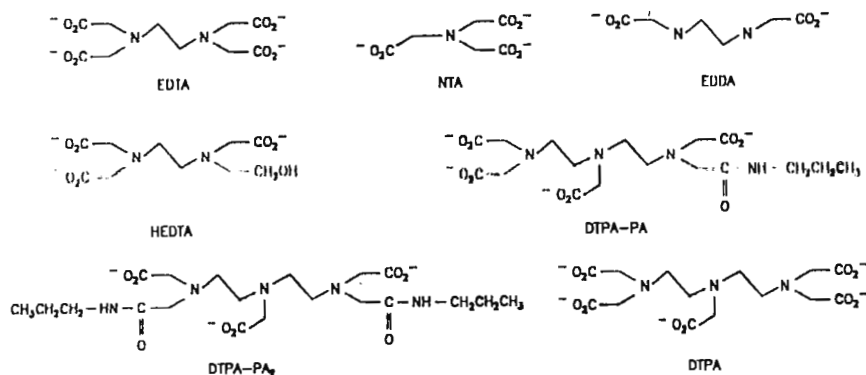
**Determination of the Number of Inner-sphere
Water Molecules in Lanthanide(III)-Poly-
aminocarboxylate Complexes**

Dear Dr. Shapiro:

The exchange between water bound in Dy(III) complexes and the bulk is fast on the ^{17}O NMR time scale. Previous studies suggest that the Dy(III)-induced ^{17}O shift (DyIS) of a Dy(III)-bound ^{17}O nucleus (Δ) is predominantly of contact origin and almost independent of the nature and the stoichiometry of the complex in question.¹ The experimental induced ^{17}O shift of water in the presence of a highly stable Dy(III) complex is given by:

$$\text{DyIS} = q \cdot \Delta \cdot [\text{Dy}(\text{ligand})_n(\text{H}_2\text{O})_q] / [\text{H}_2\text{O}] \quad (1)$$

For low complex concentrations a plot of DyIS versus $[\text{Dy}(\text{ligand})_n(\text{H}_2\text{O})_q]$ should give a straight line with slope $q \cdot \Delta / [\text{H}_2\text{O}]$ and then q can be determined. In this way we have determined the number of coordinated waters in a series of Dy(III)-polyaminocarboxylates. In Table I the results are compared with those of other methods (NMRD and luminescence spectroscopy).



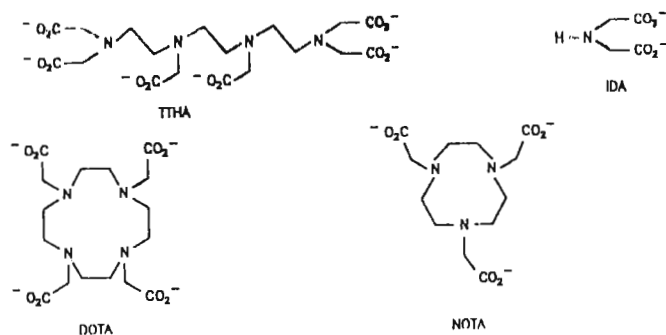


Figure 1. Structures of ligands

Table I. q Values for Dy(III)-polyaminocarboxylate complexes at 21 °C derived from ^{17}O chemical shifts; comparison with q values of corresponding Eu(III), Gd(III), and Tb(III) complexes obtained from the literature.

Ligand ^a	ρ^b	pH	q (NMR)	q (fluorescence)	q (NMRD)
			This work	Literature values	
H ₂ O		3.5	9.0	9.0	9
edta	1.00	7.0	2.3	2.5; 2.6	2.6-3
dtpa	1.00	7.0	1.3	1.1; 1.2	1
dtpa-pa	1.00	6.8	1.7	1.1	1
dtpa-pa ₂	1.00	6.5	1.7	1.0	1
ttha	1.00	7.0	0.2	0.2	0
hedta	1.00	7.0	3.2	2.9; 3.2	3.2
hedta	1.00	10.0	1.0	1.2	1
edda	1.00	6.2	5.2		
edda	1.00	5.3	6.0		
edda	0.50	5.7	2.6		
edda	0.25	6.2	0.6		
nota	1.00	7-8	2.5 ^c	3.3	2
dota	1.00	7-8	1.9 ^c	1.2	1

^aStructures, see Figure 1; ^bMolar ratio Dy(III)/ligand; ^cData from ref. 2.

In these cases the agreement is very good. For the Dy(III)-edda system, the stability constants of the various complexes are too low to allow a direct use of eq (1). However, with the use of speciations calculated from reported stability

constants and assuming $q = 5$ and 1 for the $1:1$ and $1:2$ species, respectively, "averaged" q values could be calculated, which agree well with the experimental ones.

A separation of contact and pseudocontact contributions to the experimental DyIS is not needed to obtain reliable coordination numbers. From cases where such a separation is performed, it is concluded that for $q \geq 2$, the contact contribution is larger than 78%. Only when $q = 1$, the contribution of the pseudocontact shift may be appreciable, because then the averaging out of the geometric term via exchange is less efficient. The maximum absolute error in q with this method is ± 1 .

For Dy(III)-nta and Dy(III)-ida the results were dependent on the sample preparation. For Dy(III)-ida, samples prepared from stock solutions with high concentrations (≥ 150 mM) and at $\text{pH} \geq 7$ were not stable with time. The pH was decreasing gradually and after readjustment gels were obtained after some hours. This oligomerization process was irreversible. Correspondingly, the plots according to eq (1) are not linear, but they suggest a decrease of q with concentration, when the samples were prepared by increasing the concentration of the sample. However, when the samples were prepared by dilution of a 100 mM stock solution of the complex, a straight line was obtained

^{139}La NMR showed that during the oligomerization of La(III)(nta), a disproportionation of La(III)(nta) ($\delta = 106$ ppm) to La(III)(nta) $_2$ ($\delta = 202$ ppm) occurred.²

Sincerely,



M.C. Alpoim



A.M. Urbano



C.F.G.C. Geraldes

(Chemistry Department, University of Coimbra, 3000 Coimbra, Portugal)



J.A. Peters

(Delft University of Technology)

1. Chen Zhi, J. van Westrenen, H. van Bekkum and J.A. Peters, *Inorg. Chem.*, 1990, **29**, 5025 and references cited therein.
2. C.C. Bryden, C.N. Reilley and J.F. Desreux, *Anal. Chem.*, 1981, **53**, 1418.
3. J. van Westrenen, J.A. Peters, H. van Bekkum, E.N. Rizkalla and G.R. Choppin, *Inorg. Chim. Acta*, 1991, **181**, 233.

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Frequency range	6 - 220 MHz	6 - 220 MHz	
Pulse power (min.) into 50 ohms	1000 W	300 W	
CW power (max.) into 50 ohms	100 W	30 W	
Linearity (± 1 dB to 200Mhz)	0-800 W	0-250 W	
(to 220MHz)	0-600 W	0-200 W	
Gain (typ.)	65 dB	60 dB	
Gain flatness	± 4 dB	± 3 dB	
Input/Output impedance	50 ohms		
Input VSWR	< 2:1		
Pulse width	20 ms		
Duty cycle	Up to 10%		
Amplitude rise/fall time	200 ns typ.	150 ns typ.	
Amplitude droop	5% to 10 ms typ; 7% max		
Phase change/power output	10° to rated power, typ.		
Phase error overpulse	4° to 10 ms duration, typ.		
Noise figure	11 dB typ.	8 dB typ.	
Output noise (blanked)	< 20 dB over thermal		
Blanking delay	< 2 μ s on/off, TTL signal		
Protection	1. VSWR: infinite VSWR at rated power 2. Input overdrive: up to +10 dBm 3. Over duty cycle/pulse width 4. Over temperature		
<u>Supplemental characteristics:</u>			
Connectors, rear panel	1. RF input: BNC (F) 2. RF output: Type N (F) 3. Noise blanking: BNC (F) 4. Interface: 25 pin D(F), EMI filtered		
Indicators, front panel	1. Peak power meter 2. Over temperature 3. Over duty cycle/pulse width 5. CW Mode 6. Overdrive		
System monitors	1. Thermal fault 2. DC power supply fault 3. Over duty cycle/pulse width 4. Forward/Reflected RF power		
Front panel controls	1. AC power 2. Pulse width 3. Duty cycle		
Cooling	Internal forced air		
Operating temperature	+10 to 40°C		
AC line voltage	120/240 VAC, $\pm 10\%$, 50-60Hz (3200, 220/240V only)		
AC power requirements	2000 watts 700 watts		
Package	Rack mount		
Size (HWD, inches)	12.25x19x24 5.25x19x24		
Net weight	165 lbs 42 lbs		

July 15, 1991
(received 7/19/91)Professor B. L. Shapiro
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto, CA 94303**Measurement of Relaxation Time Constants for Methyl Groups
by Proton-Detected Heteronuclear NMR Spectroscopy**

Dear Professor Shapiro,

The advent of proton-detected two-dimensional NMR methods for determining heteronuclear relaxation time constants [1-4] has led to a renewed interest in heteronuclear ^{13}C and ^{15}N NMR relaxation in biological macromolecules; however the DEPT and INEPT sequences commonly used for magnetization transfer can give erroneous results for I_2S and I_3S spin systems, such as ^{13}C methylene and methyl groups, respectively [1]. We have developed a method for obtaining accurate spin-lattice relaxation time constants for methyl groups that uses a double polarization transfer and is sufficiently sensitive for use with natural abundance samples. By using magic angle pulses in DEPT-based sequences, or analogously tuned delays in INEPT-based sequences, contributions to the observed NMR signal are eliminated that would otherwise affect the measurement of the desired time constants.

Figure 1 shows the pulse sequence for a one-dimensional proton-detected inversion recovery experiment for measurement of heteronuclear spin-lattice relaxation time constants utilizing DEPT polarization transfers. If the effects of cross-correlation between different IS bond vectors are significant, this sequence will yield identical results as a conventional inversion recovery experiment for an I_3S spin system if $\theta = \theta' = 54.7^\circ$. If cross correlation effects are not significant, only θ' need be set to 54.7° and θ can be set to $30\text{--}35^\circ$ to maximize magnetization transfer. For each angle that is set to 54.7° , rather than $30\text{--}35^\circ$, the sensitivity of the experiment is reduced by a factor of approximately 0.71.

Conventional and proton-detected inversion recovery spin lattice relaxation measurements were performed on a 0.1 M solution of $^{13}\text{CH}_3\text{CO}_2\text{Na}$ at 298 K. Proton-detected relaxation measurements were also performed on a 10 mM solution of the Xfin-31 peptide (Ac-YKCGLCERSFVEKSALSRHQVRHKN-NH₂, ^{13}C at natural abundance) [5] at 303 K. Conventional, direct ^{13}C detection experiments used a $\pi\text{--}\tau\text{--}\pi/2\text{--}\text{acquire}$ sequence.

The phenomenological time constants, T_1 , for $^{13}\text{CH}_3\text{CO}_2\text{Na}$ and for a $\gamma\text{--}^{13}\text{CH}_3$ of Val 22 in Xfin-31, obtained from a single exponential fit to the experimental data, are shown in Table I. As can be seen for $^{13}\text{CH}_3\text{CO}_2\text{Na}$, the sequence of Fig. 1 results in a lower estimate of T_1 when $\theta = \theta' = 30^\circ$, as compared to the result of the conventional direct-detected experiment; however, when $\theta = \theta' = 54.7^\circ$, the results of the proton detected and conventional experiments are identical. Similarly, the T_1 value for the $\gamma\text{--}^{13}\text{CH}_3$ of Val 22 in Xfin-31 is significantly shorter for $\theta = \theta' = 30^\circ$ than for $\theta = \theta' = 54.7^\circ$.

A more complete account of this research will appear in *Chemical Physics Letters*. Please credit this contribution to the account of Peter E. Wright.

Sincerely yours,



Arthur G. Palmer, III

Dr. Angelika Sebald
Dr. Lawrence Merwin

Bayerisches Geoinstitut, Universität Bayreuth
Postfach 10 12 51, 8580 Bayreuth, FRG

Tel. 09 21/55 21 64 (Sekretariat)
55 21 65 (Seifert)

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

Bayreuth, July 3rd, 1991
(received 7/12/91)

Cross polarisation ^{19}F to ^{29}Si in partially protonated systems

Dear Dr. Shapiro,

recently we have become involved with MAS NMR investigations (including cross polarisation from ^{19}F to ^{29}Si) of fluorine-doped aluminosilicate glasses [1, 2]. In such anhydrous systems $^{19}\text{F} \rightarrow ^{29}\text{Si}$ cross polarisation is very efficient, even if such systems contain no more than 1 - 5 % fluorine. It is straightforward to set the $^{19}\text{F}/^{29}\text{Si}$ Hartmann-Hahn matching condition, using e. g. Na_2SiF_6 or topaz, $[\text{Al}_2(\text{F},\text{OH})(\text{SiO}_4)]$ as set-up samples. It is important to verify ^{19}F on-resonance conditions, so preliminary ^{19}F CRAMPS (or fast MAS ^{19}F) experiments are highly recommended. In anhydrous systems ^{19}F turns out to be a promising magnetisation source, as the ^{19}F $T_{1\rho}$'s tend to be fairly long.

We noticed that investigations of such aluminosilicates (containing 1 - 5 % fluorine plus 1 - 5 % H_2O) by means of $^{19}\text{F} \rightarrow ^{29}\text{Si}$ cross polarisation techniques are severely hampered: the CP efficiency is strongly decreased by the presence of water, the ^{19}F $T_{1\rho}$'s are very short under such circumstances. Short ^{19}F $T_{1\rho}$'s have also been reported for the combination $^{13}\text{C}/^{19}\text{F}/^1\text{H}$ in mixed organic polymer blends [3]. Now we can report a similar finding (i. e. short ^{19}F $T_{1\rho}$) for a molecular crystalline compound, $\text{F}_4\text{Si}-\overset{\ominus}{\text{CH}_2}-\overset{\oplus}{\text{CH}_2}-\overset{\oplus}{\text{CH}_2}-\text{NH}_3$. There is no problem to obtain the ^{29}Si MAS spectrum with ^{19}F high-power decoupling but $^{19}\text{F} \rightarrow ^{29}\text{Si}$ cross polarisation is again not very efficient (see figure 1).

Clearly, all the mutual $^1\text{H}/^{19}\text{F}/\text{X}$ -nucleus interactions have to be taken into account. We would be most interested to hear if others have come across similar findings for such double or triple resonance CP MAS experiments.

Best regards from Bayreuth,

Angelika Sebald

Dr. Angelika Sebald

- [1] A. Sebald, L. Merwin, T. Schaller and W. Knöller; J. Magn. Reson., submitted
- [2] T. Schaller, D. Dingwell, H. Keppler, W. Knöller, L. Merwin and A. Sebald; Geochim. Cosmochim. Acta, submitted
- [3] C. H. Klein Douwel, W. E. J. R. Maas, V. S. Veeman, G. H. Werumeus Buning and J. M. J. Vankan; Macromol., 23, 406 (1990)

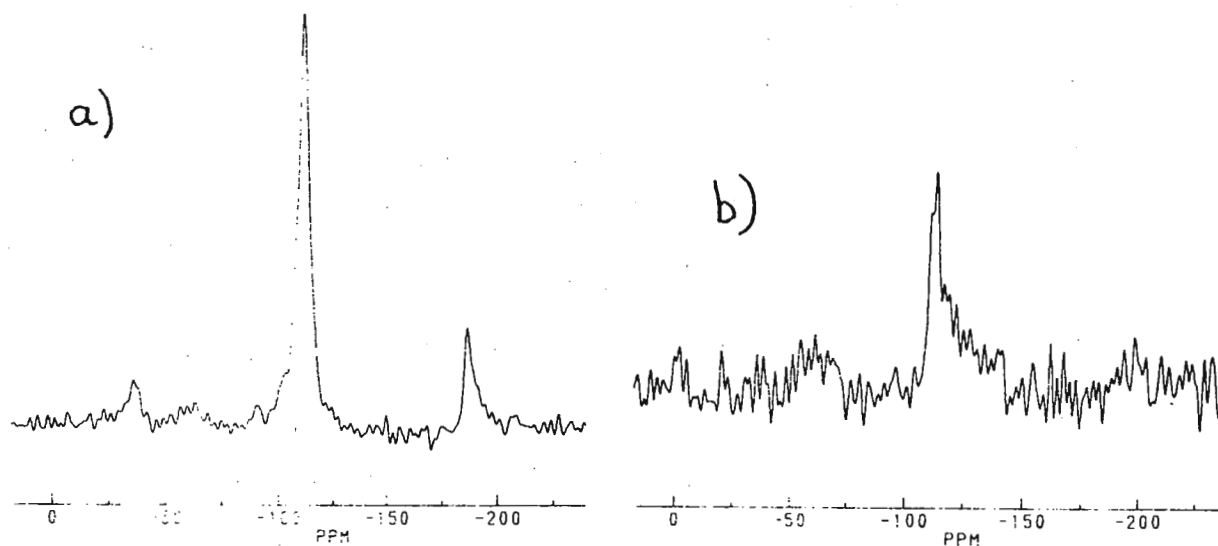
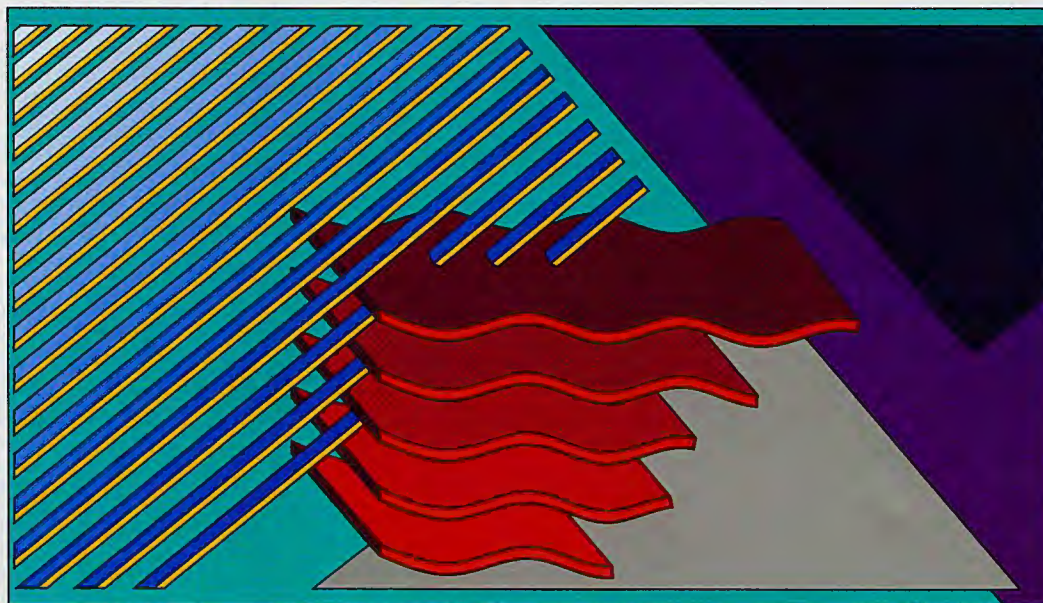


Fig. 1 Single pulse, ^{19}F high-power decoupled ^{29}Si MAS (a) and $^{19}\text{F} \rightarrow ^{29}\text{Si}$ CP

⊖
MAS (b) spectra of $\text{F}_4\text{Si}-\text{CH}_2-\text{CH}_2-\text{CH}_2-\text{NH}_3$.
⊕

Rotation rate 4.5 kHz, 2000 transients (a) and 1100 transients, 10 ms contact time (b), respectively

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Professor B. L. SHAPIRO
966 Elsinor Court
Palo Alto
California 94303 - Etats Unis

Dear Dr Shapiro

Theix, 1991 july 8
(received 7/13/91)

STUDY OF COLLAGEN USING SOLID ^{13}C NMR

The collagen protein is almost completely resistant to solubilization with non denaturing solvents. Cross-link variations were determined using two ^{13}C NMR approaches : spectroscopy and measurements of relaxation times. Our investigation was performed on three connective tissues : control intramuscular connective tissue purified with trypsin, the same tissue depolymerized with penicillamin, and the third one more cross-linked with borohydride.

High-resolution ^{13}C solid-state NMR spectra (CP/MAS) of the three collagens are compared after treatment by the Maximum Entropy Method. Differences between solid-state NMR spectra of the three polymerization states of collagen are observed. The lines which are significantly dependent of crosslinking degree are not yet assigned and have low intensities.

To study the influence of cross-linking on the mobility of the various carbons, the spin-lattice (T_1) and the cross-polarization (T_{CH}) times are measured. The cross-polarization time T_{CH} does not vary with cross-link degree. On the other hand, T_1 increases with polymerization, specially for the well resolved line of the C γ of hydroxyproline ; so the correlation time is longer when the crosslink degree of the solid sample increases.

High-resolution ^{13}C solid-state NMR can inform on the aging of collagen. The relaxation time T_1 values of carbons are consistent with the stabilizing effect of crosslinks on the mobility of the collagen molecule.

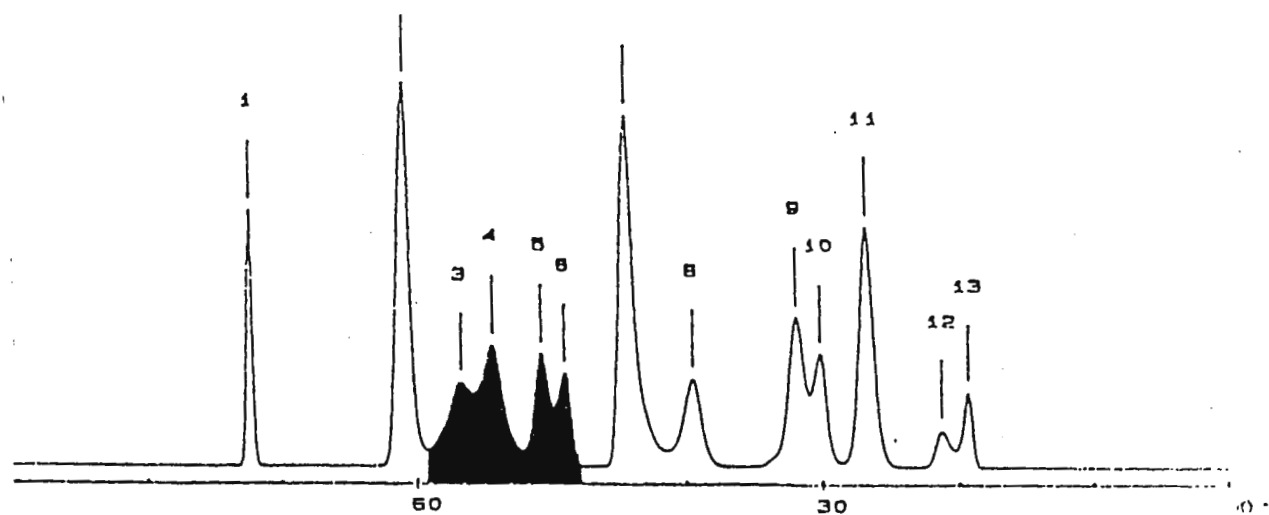
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M. BONNET

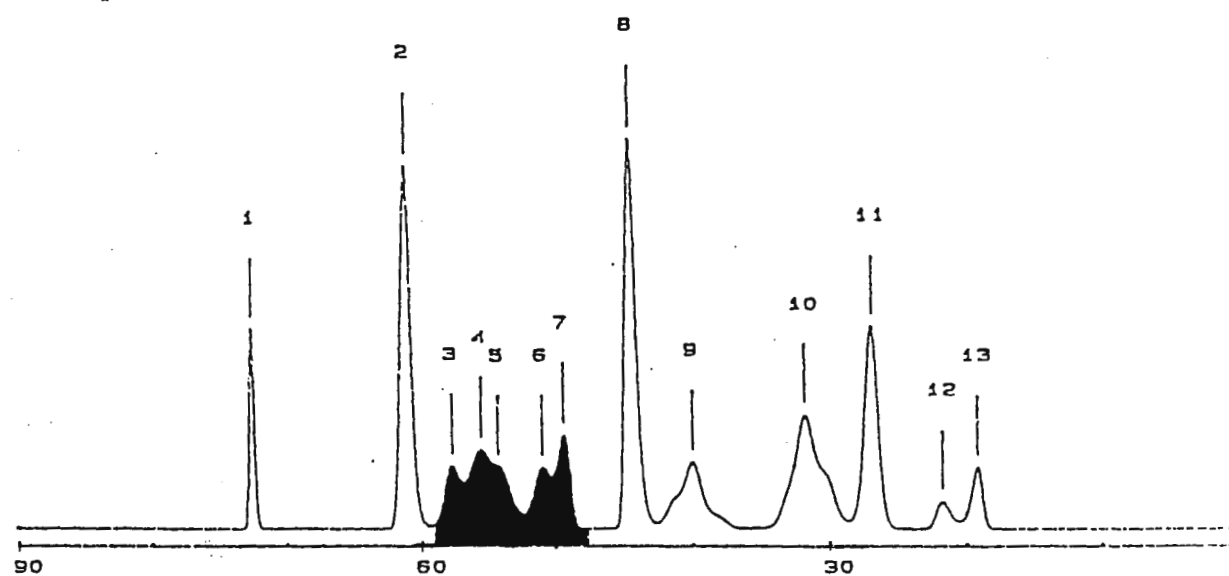
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J. DUPUIS

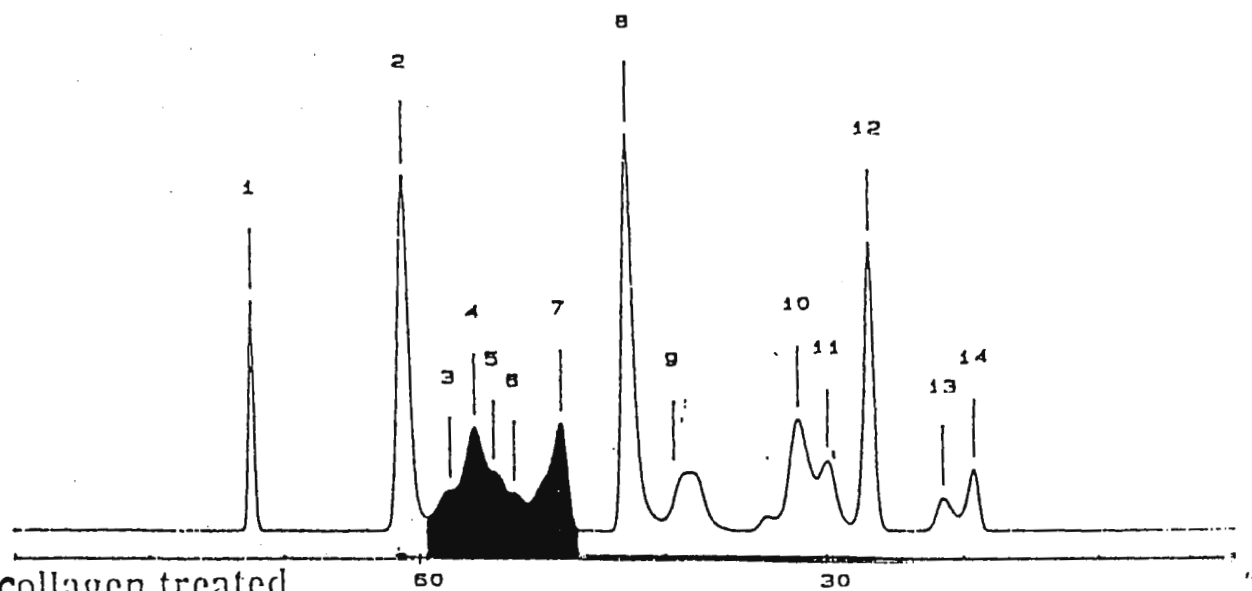
J.P. RENOU



collagen treated
with penicillamin



Control collagen



collagen treated
with borohydride

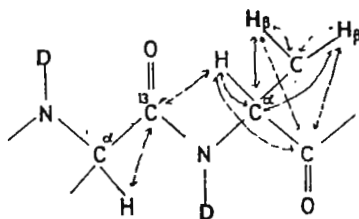
Prof. B.L.Shapiro
TAMU NMR Newsletters
966 Elsinore Court
Palo Alto. CA 94303
USA

Roskilde July 12 1991
(received 7/18/91)

Natural abundance ^{13}C spectra of proteins.

Dear Prof. Shapiro,

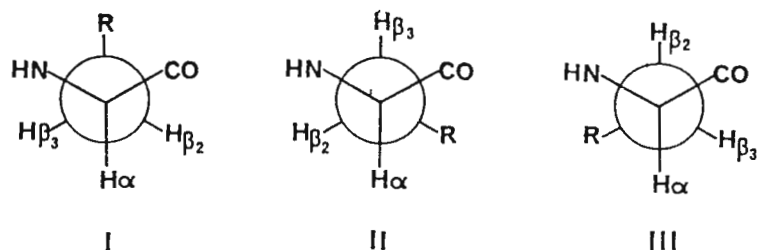
With the help of Ad Bax we have managed to record and analyse the natural abundance ^{13}C spectrum of BPTI (bovine pancreatic trypsin inhibitor), MW 6500, using the HMBC (heteronuclear multiple bond correlation) technique¹. This technique utilizes long-range heteronuclear (^{13}C) proton couplings. The strength of the method is the observation of a large number of correlations per carbon and per hydrogen leading to a network of correlations (see Fig.1) crosslinking carbon and hydrogen assignments. The only weak spot of the method is the absence of a resonance in case of small couplings.



A complete analysis of the natural abundance ^{13}C spectrum is achieved together with the complete assignment of the ^1H spectrum. The latter was done beforehand², but a few new assignments were made. The analysis of peak intensities leads to estimates of coupling constants. Three-bond, $^3\text{J}(\text{CO}, \text{H}\beta)$ couplings are used in stereospecific assignments of the $\text{H}\beta$ protons. It is furthermore demonstrated that two-bond carbon-hydrogen couplings, e.g. $^2\text{J}(\text{C}\alpha, \text{H}\beta)$ can give additional information about χ_1 angles.

The two-bond coupling depends on the orientations of electronegative substituents on the coupling path. For rotamer II the position of the NH group gauche to both $\text{H}\beta$ leads to more negative two-bond couplings for both $^2\text{J}(\text{C}\alpha, \text{H}\beta_2)$ and $^2\text{J}(\text{C}\alpha, \text{H}\beta_3)$ and to observable resonances, whereas for rotamers I and III only one coupling is large enough for observation. The observation of two-resonances as seen in Fig. 2 is hence a direct proof of the presence of rotamer II.

The overall experience is that the HMBC technique is very powerful in the analysis of ^{13}C spectra of medium size proteins and that carbon-hydrogen couplings add extra structural information.



The coupling constants have so far mostly been used in the analysis of χ_1 angles, but can most likely also be used to establish e.g. glutamic acid hydrogen-bonding to the back-bone and other patterns found in proteins. The full story will appear in Biochemistry.

Yours sincerely

Poul Erik Hansen

Poul Erik Hansen

1. A. Bax and M.F. Summers, *J. Am. Chem. Soc.* 107, 2821 (1986)
2. G. Wagner et al., *J. Mol. Biol.* 196, 611 (1987).

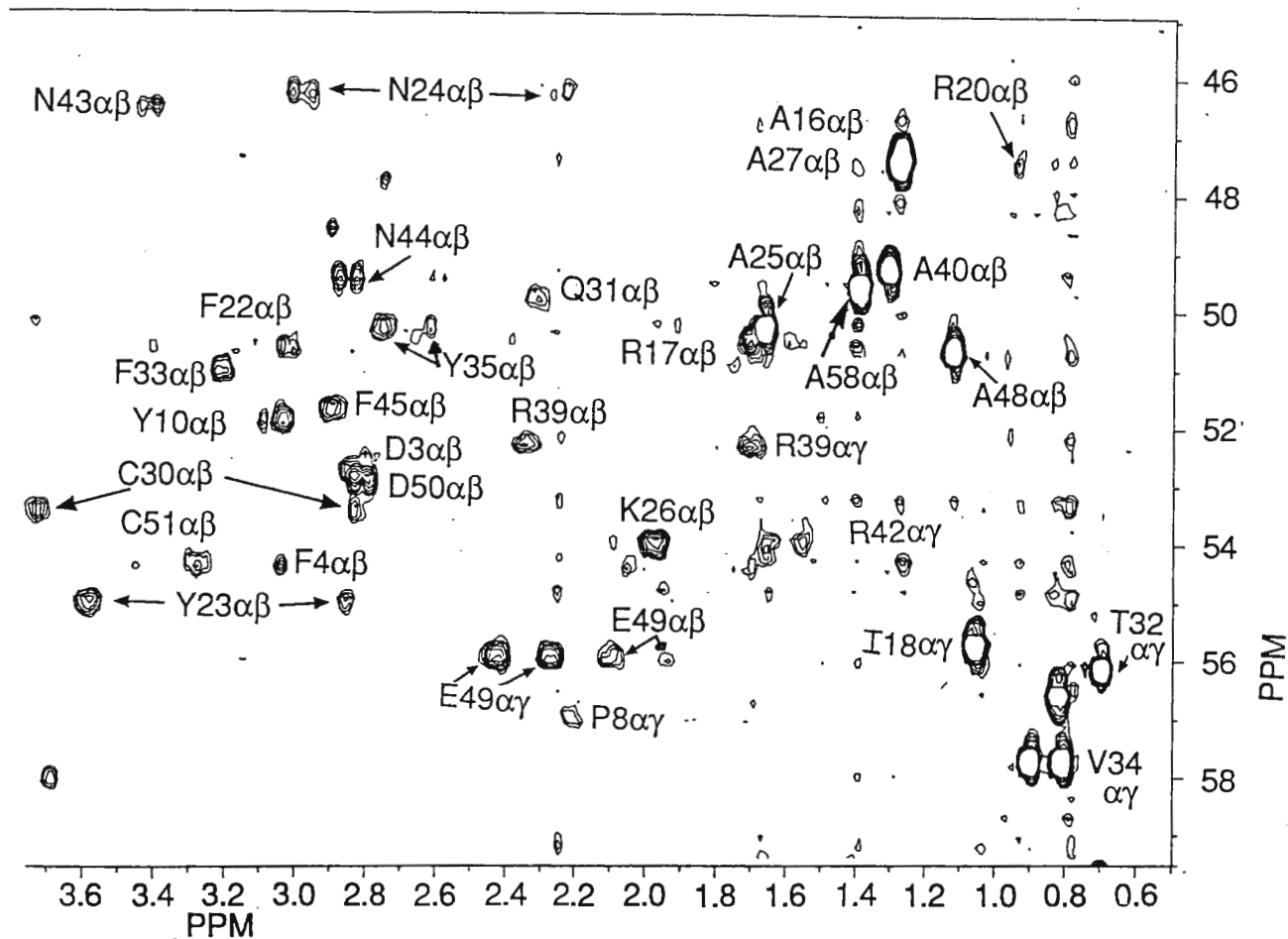
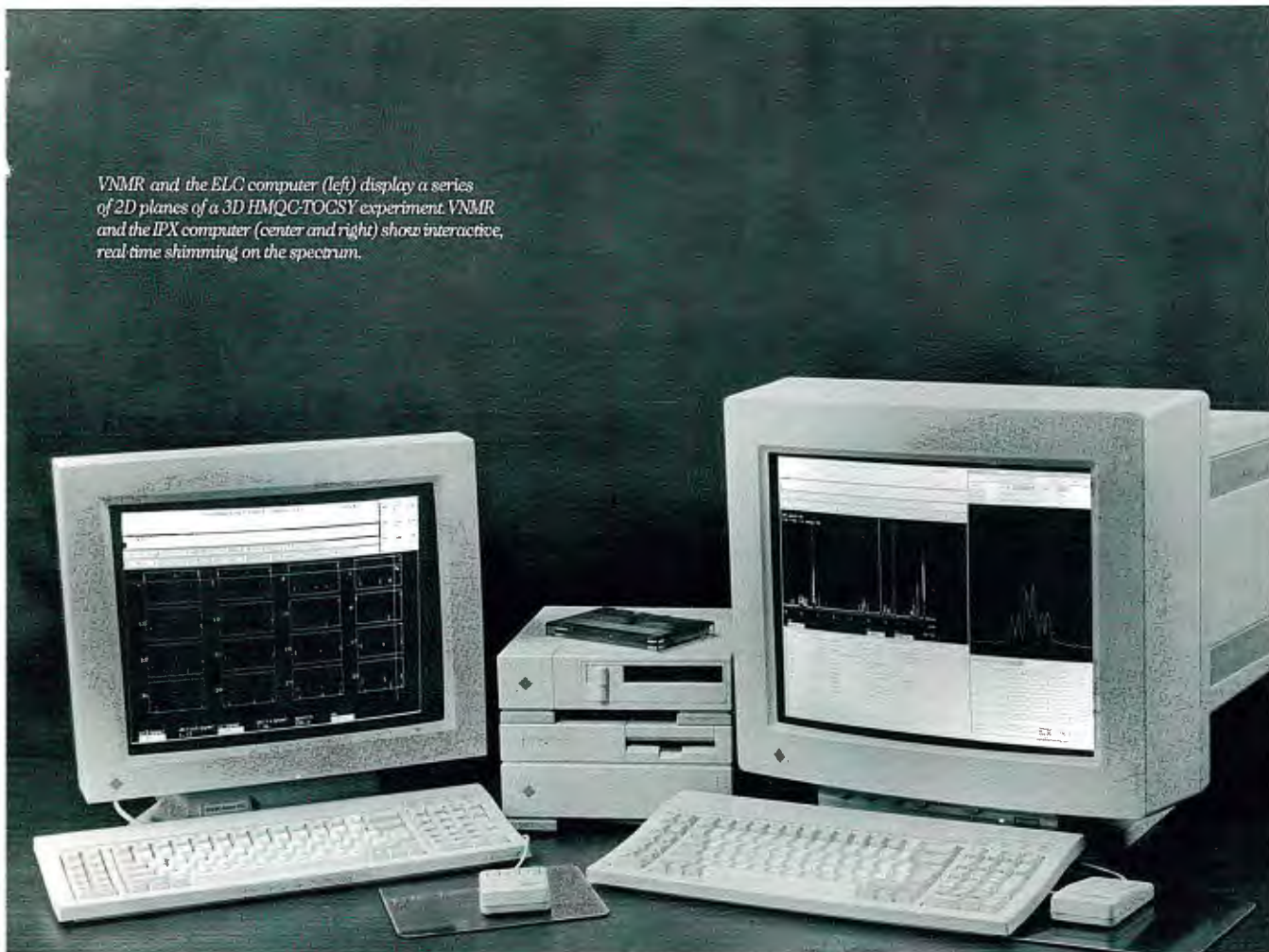


Fig.2 Part of the $C\alpha, H\beta$ region. show residues with two $C\alpha H\beta$ resonances

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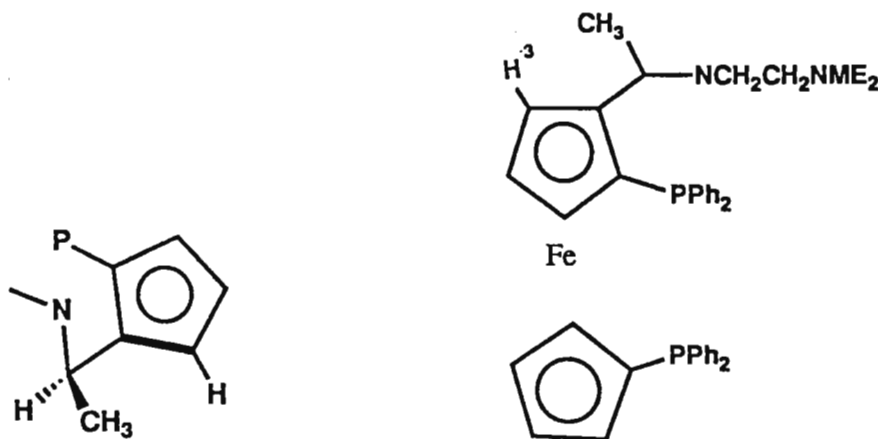
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(received 6/26/91)

Dear Barry,

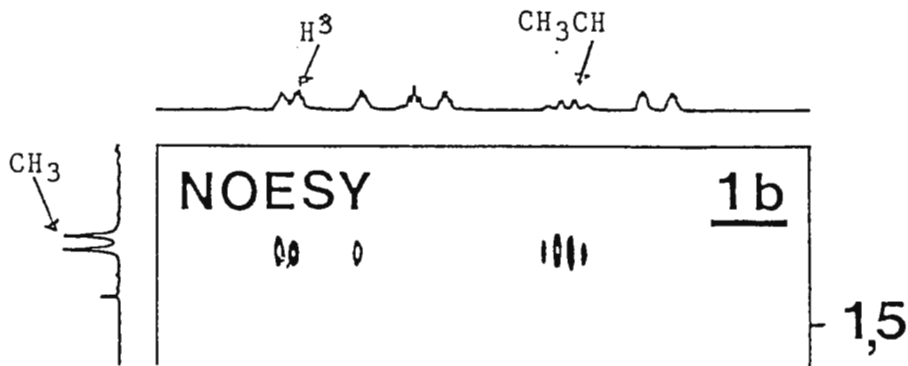
Optically active soluble transition metal catalysts are in increasing use in synthetic chemistry.

For the complexes based on the ferrocene structure shown below, it is of interest to relate the conformation of the side-chain to the ferrocene moiety. One approach to this problem involves Overhauser effects between protons on the side-chain and one-or both- of the cyclopentadienyl moieties.



A section of the ¹H-NOESY spectrum from one such derivative shows that the methyl protons on the side-chain develop substantial NOE's to both the cp proton indicated, as well as to cp protons of the ring below. This information, when combined with some P,H coupling constant data, allows us to postulate that this chiral complex has the conformation shown. A similar approach allows us to define the 3-D structure of complexes in which a second transition metal is coordinated to the two PPh₂ moieties.

Please credit this contribution to the account of Luigi Venanzi.



Suggested title: ¹H NOESY on chiral complexes.

Best wishes

Paul



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

(received 7/18/91)

Simulated Annealing in Time-domain NMR Signals

Dear Dr. Shapiro,

One of the major problems encountered in Fourier transform NMR is that any noise sampled in the time-domain is necessarily transformed into the frequency domain. This is not the case for parametric estimation algorithms such as simulated annealing, which is actually a global minimization algorithm. It is well-known by now that there are two major classes of algorithms used to minimize functions; approximation and optimization, with definite benefits and drawbacks to each one. Approximation algorithms are usually very fast and yield results in polynomial time. The drawbacks are that there is no guarantee that the results are the optimal ones, and the results are very dependent on the initial conditions. Optimization algorithms are independent of initial conditions, and usually yield globally optimal results, but at the expense of astronomically large amounts of CPU time in cases of large problems. We have found that it is possible to have the best of both worlds so to speak, by writing an algorithm that combines both these properties.

Intrinsic to the annealing algorithm, there are a large number of acceptances by the Metropolis algorithm (1-3) early on, with the number approaching zero as the control parameter approaches zero. While this criterion is imperative in the early stages, it can be both detrimental to the final results and extremely time consuming in the later stages. We saw that if the algorithm was going to fall in relative minima, it would do so very early on, usually after 10-15 "temperature" cycles. If, after say 15 "temperature" cycles, we switched over to an iterative approach, i.e., by setting the control parameter to zero, we not only achieved results at least as good as the strict annealing approach, but also realized as much as a one hundredfold speedup in computational time. We currently have a note in press in *J. Magn. Res.* that describes our algorithm in full.

Sincerely,

DiGennaro

Frank S. DiGennaro

- 1) K. Sekihara and N. Ohyama, *Magn. Res. Med.* **13**, 332 (1990).
- 2) R.E. Hoffman and G.C. Levy, *J. Magn. Res.* **83**, 411 (1989).
- 3) W.H. Press, B.P. Flannery, S.A. Teukolsky, and W.T. Vetterling, "Numerical Recipes," Cambridge Univ. Press, New York 1989.

P.S. Please credit this to David Cowburn's account.

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 CUNY, Staten Island
 130 Stuyvesant Pl.
 Staten Island, NY 10301

Gwendolyn N. Chmurny
 PRI/DynCorp, NCI-FCRDC
 P.O. Box B, Bldg. 469, Rm. 162
 Frederick, MD 21702-1201

Tuesday, November 12, 1991, p.m.

NMR Award to Professor John D. Roberts, David M. Grant, U. of Utah, Salt Lake City, UT 84112, Chairman

"Three Dimensional World of Chemistry Viewed Through Chemical Shift Tensors", David M. Grant, U. of Utah, Salt Lake City, Utah 84112

"Perturbation of Conformational Equilibria by Deuterium and Tritium Isotope Effects", Frank A.L. Anet, UCLA, Los Angeles, CA 90024

"From the Rediculous to the Sublime. The Last Forty Years of NMR", James A. Shoolery, Varian, Inc., Palo Alto, CA 94303

"NMR - An Endless Frontier", John D. Roberts, Cal. Tech., Pasadena, CA 91125

Wednesday, November 13, 1991, a.m.

NMR in Pharmaceutical Research, Stephen Fesik, Abbott Labs., Abbott Park, IL 60064, Chairman

"Cyclic Peptide and Protein-Ligand NMR Studies for Pharmacophore Identification", Kenneth Kopple, Smith Kline Beecham Pharmaceuticals, King of Prussia, PA 19406

"NMR Approaches to Simplify Receptor/Ligand Interactions", David Delgarno, Schering-Plough Res., Bloomfield, NJ 07003

"NMR Investigations of Wild Type and Oncogenic Ras Proteins", Sharon Campbell-Burk, Du Pont Merck Pharmaceutical Co., Wilmington, DE 19880-0328

"NMR Studies of Phospholipases", Stephen Brown, Glaxo, Inc., Morrisville, NC 27560

"Multi-Dimensional NMR Studies of an Isotopically Labeled BPTI Mutant", Sally Heald, Miles, Inc., Research Division, West Haven, CT 06516

"Characterization of the A Chain RNP Protein C Using Heteronuclear Multidimensional NMR Techniques", Luciano Mueller, Squibb Institute for Medical Research, Princeton, NJ 08543

"The Interactions of FK-506 with its Binding Protein by Multidimensional NMR Spectroscopy", Allen Kline, Lilly Research Labs., Indianapolis, IN 46285-0403

"Structural Studies of Immunosuppressants and Their Binding Proteins", Stephen Fesik, Abbott Labs., Abbott Park, IL 60064

Wednesday, November 13, 1991, p.m.

NMR Methods for Peptides and Proteins, Sharon Campbell-Burk, Du Pont Merck Pharmaceutical Co., Wilmington, DE 19880-0328, Chairman

"An NMR Structure of a Protein in Dynamic Equilibrium - Acyl Carrier Protein", James H. Prestegard, Yale University, New Haven, CT 06511

"Heteronuclear 3D NMR and Triple Resonance Studies of Larger Proteins: Ras p21", Peter Domaille, Du Pont Merck Pharmaceutical Co., Wilmington, DE 19889-0328

"Multiple Resonance Experiments on Proteins", Gerhard Wagner, Harvard Medical School, Boston MA 02115

"Alternative Procedures for Obtaining Structural Information from NMR Data", Ad Bax, National Institute of Health, Bethesda, MD 20892

Thursday, November 14, 1991, a.m.

Experimental NMR Techniques, Ruth E. Stark, CUNY, Staten Island, NY 10301, Chairman

"Adventures in Selective-Excitation Technique Applications", William C. Hutton, Monsanto Corp. Research, Monsanto Co., St. Louis, MO 63198-0001

"1D and 2D NMR Methodology for Studying Small-Molecule Dynamics", Eric R. Johnston, Central Research & Development, Du Pont Co., Wilmington, DE 19880

"Solid-State NMR Strategies for Agricultural Materials", Walter V. Gerasimowicz, US Department of Agriculture, Philadelphia, PA 19118

"Analytical Applications of Relaxation Coupling in Heterogeneous Systems", Robert G. Bryant, U. of Rochester Medical Center, Rochester, NY 14642

Wednesday, November 13, 1991 all day. Poster Sessions

Advances in NMR in Biological Chemistry, David Fry, Hoffmann-La Roche, Nutley, NJ 07110, Chairman

Advances in NMR - Polymers and Chemical Sciences, Peter Mirau, AT&T Bell Labs., Murray Hill, NJ 07974, Chairman

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The NMR Data

Processing

Software

MAGFIELD

for the

Macintosh II

tecThe logo for tecmag, featuring the word 'tec' in a bold, lowercase sans-serif font, followed by 'mag' in a similar font. A thick black arrow starts from the bottom of the 'g' and points diagonally down and to the right.

MacFID 1D

SPECIFICATIONS

- Data format translation:
 - Bruker
 - Chemagnetics
 - GE
 - JEOL
 - Varian
 - MacNMR
 - FTNMR/Felix
 - Varian
 - Native parameter import
- 1D processing:
 - Baseline correction
 - Apodization functions
 - Complete FFT modes
 - ICON driven Integrals
 - Peak Picking
 - Line fitting functions
 - T1 and T2 calculations
 - S/N calculation
 - User Macro files
 - Integral Setup SAVE files
 - DSP array processor support
- 1D Display:
 - Real/Imaginary/Magnitude
 - Difference display mode
 - Axis/grid
 - Stack plot display
 - Display color selection
 - Copy to Clipboard
 - Lines/Points mode
 - Point shift left/right
- 1D Printing:
 - All display modes
 - Integrals and tables
 - Peak tables
 - Parameter block
 - Logo (PICT format)
 - SAVE as PICT document
 - Print Setup SAVE files

MacFID 2D

SPECIFICATIONS

- 2D processing:
 - Baseline correction
 - 2D apodization functions
 - 2D FFT macro command
 - DSP array processor support
 - Transpose functions
 - Projections & Cross-Sections
 - Symmetrization functions
- 2D Display:
 - Mouse driven Stack plot
 - Thin/Fat Contour lines
 - Pseudo-color map
 - 256 Gray scale
 - Zoom, Cursors, Axis
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SB
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Pharmaceuticals

July 12, 1991
(received 7/15/91)

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

ZETA Connection

Dear Dr. Shapiro,

Despite all the fancy computational tools currently available, some of us still use large plots (not to see the bigger picture, of course). We came across some sticky "Handshake" problems when we tried to plot spectra from FELIX on a ZETA 936 plotter. FELIX does not explicitly support Zeta plotters. However we can use HPGL on Zeta plotters. Here is a simple procedure to connect ZETA 936 plotters to SUN 4/260. We have connected the ZETA plotter to the "ttya" port of the SUN. Essentially we use the plotter as a spooled device (named nicolet). We need to create an entry (corresponding to the plotter) in the "printcap" file which is located in the /etc directory. The entry is as follows:

```
nicolet:\n      :lp=/dev/ttya:sd=/var/spool/nicolet:br#4800:fs#0000:\n      :fc:#0300:ms=ixon,ixoff:lf=/var/spool/nicolet/error_log:
```

Switch settings on the plotter are as follows:

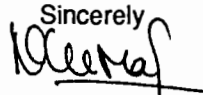
SW01: 2 & 3 down

SW02: 3, 4, 5 & 7 down

SW03: 1, 3, 7, 8 down

Rest of the switches are up.

We create a HPGL plot file from FELIX (define hpm=31 and define plot sizes by hsz) and then print this file using lpr command (eg. lpr -Pnicolet -s filename). The symbolic link (-s option in lpr command) is used to get around the occasional problem of large file size. Please remember to use -P (CAPITAL P) option if you don't want to jam your default printer.

Sincerely

N. Vasant Kumar

Please credit this contribution to Susanta Sarkar's account.



UNIVERSITY OF OREGON

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July 15, 1991
(received 7/19/91)

Dr. Bernard L. Shapiro,
TAMU Newsletter

AQ11/FELIX: NMR SPECTROMETER CONTROL BASED ON THE IBM-COMPATIBLE PC

Dear Barry:

We have developed a PC-based NMR data system to replace the Nicolet 1180 computer of our multinuclear NTC-360 spectrometer at the University of Oregon. A key feature of our system is that instrument control has been incorporated into the standard FELIX NMR processing program of Hare Research. The IBM-compatible PC provides an economical, but powerful, upgrade alternative for multichannel Fourier-transform spectrometers. A particularly attractive feature of the PC is the abundance of low cost peripherals and software, in addition to the ability to rapidly transfer data to other networked computer systems. Spectra are stored in an exchangeable binary-format that can be directly read by FELIX (or other programs) running on UNIX or VMS workstations sharing the ethernet network.

Our use of generic, modular software and hardware components, would permit the updating virtually any spectrometer to perform modern multiple-pulse, Fourier-transform NMR experiments. At the same time the system can be extended to almost any hardware and experimental configuration without the need to modify the underlying architecture.

While our development work has been primarily aimed at updating spectrometers here at the University of Oregon, we believe that the results of our efforts may be of interest to others and we would welcome to opportunity to share our experience.

Sincerely yours,

Michael Strain,
Research Associate

SOFTWARE: "FELIX" (Hare Research) with integrated "AQ11" instrument control developed at the U. of Oregon. Provides full-function FELIX 1-D or 2-D data display and processing with concurrent interrupt-driven data acquisition in background.

PULSE PROGRAMMER: 32K program steps/15 TTL output lines, 0.1 usec resolution, 2 loop counters. (U of Wisconsin "Enhanced state-machine pulse programmer", T.C. Farrar *et al.*, Rev. Sci. Instr. **59**:2285-2289, 1988). Pulse programs are entered interactively or with FELIX command macros. Offers gating, phase, and level control of the f1 (observe) and f2 (decoupler) channels, and provides sweep flag, digitizer clock, and CPU interrupt lines.

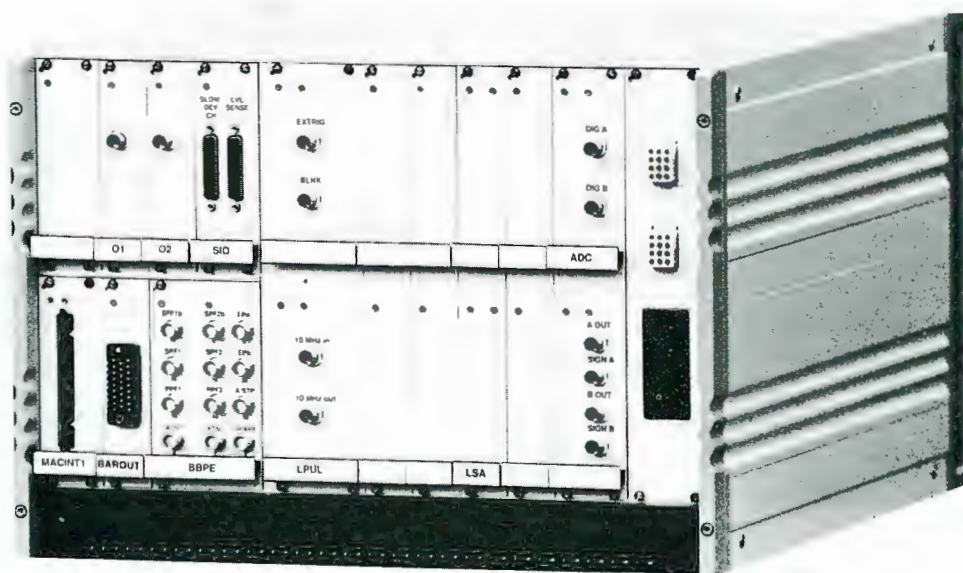
DIGITIZER: 500 kHz, 12-bit, 128 KB buffer; interrupt service routine sums the new data (with quadrature-phase cycling) into a 32-bit array in main program memory. Data tables may be of arbitrary size from 1 to 16384 words (complex).

SPECTROMETER CONTROL LINES: Program control of F1 & F2 synthesizers (PTS-160), audio filters, decoupler modes, and other console functions is provided by generic 8255-type digital I/O controllers. Additional controllers can be easily added.

(Please credit this contribution to F.W. Dahlquist)

MacSpect

Aspect 2000 Replacement for Bruker Spectrometers



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RESOURCES

The MacSpect™ is a Real Time NMR Station. Coupled with a Macintosh™ II computer, the MacSpect has been specifically designed to replace the Aspect™ 2000A computer used on Bruker™ NMR spectrometers. Replacing an Aspect 2000A with a MacSpect can be accomplished in minutes with no special tools.

The Back Panel Emulator (BPE), an integral part of the MacSpect system, allows the Macintosh II computer to take control of all the peripherals normally attached to a Bruker spectrometer. Two DB25 connectors on the BPE unit replace the Slow Device Channel and the Level/Sense connectors located on the Aspect computer. Observe and decoupling frequencies and Fourier filters can be set directly from the "dashboard" of the Macintosh II computer. Temperature, receiver gains and decoupling modes can be set as well.

MacSpect also has sixteen BNC connectors to emulate the signals found on the Aspect computer, such as PULSE's, PF's and SPF's signals, and offset oscillators O1 and O2. These two offset oscillators are now locked on the 10 MHz Master Clock.

Based on the design used in the Tecmag LIBRA™ system, the MacSpect incorporates the following:

- one 100 ns resolution pulse programmer with 5 loop counters, 2048 steps and 70 control lines.
- one 128K x 32-bit of memory signal averager.
- two 2µs per complex point, 12-bit simultaneous sampling Analog-to-Digital Converters (standard).
- one Back Panel Emulator to control the peripherals already present on the Bruker spectrometers.

MacSpect has been designed for any type of experiments and can be expanded to accommodate such options as a 100 MHz pulse programmer, a pulse programmer extension, up to 4-channels of waveform generators, different types of ADC.

MacSpect uses MacNMR™, the same powerful software that has made the success of LIBRA. Regular upgrades insures the user of always having state-of-the-art software applications.

Specifications

PULSE PROGRAMMER

Minimum Pulse Width: 100 ns
Maximum Pulse Width: 430 s
Time Resolution: 100 ns
Loop Counters: 5
Memory Size: 2048 x 128 bits

SIGNAL AVERAGER

ADC Resolution: 12 bits
No. of Channels: 2
Min. Sampling Time: 2 µs /channel
Max. Bandwidth: 500 kHz (± 250 kHz)
Memory Size: 128 k-Word x 32 bits



DEPARTMENT OF HEALTH & HUMAN SERVICES

Public Health Service

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

July 10, 1991
(received 7/15/91)

National Institutes of Health
National Institute on Aging
Gerontology Research Center
4940 Eastern Avenue
Baltimore, Maryland 21224

Spillover and Incomplete Saturation in Transient Saturation-Transfer Experiments

Dear Dr. Shapiro:

We have been fascinated for quite some time with the use of saturation-transfer techniques to derive apparently precise information about rate constants in in-vivo systems. We are most interested in transient saturation transfer, in which one of two resonances in chemical exchange is saturated for varying amounts of time and the amount of saturation transferred to the other is measured. This gives a reaction rate and the T_1 of the non-saturated resonance.

Among other problems, such as appropriate selection of time-points and errors due to noise¹ (particularly in systems with poor signal-to-noise), are the problems of spillover saturation and incomplete saturation. Spillover is generally measured, judged inconsequential (e.g., with the words "...less than 10%"), and neglected, or is accounted for by a baseline saturation. The latter method assumes a symmetry of the saturating rf, in addition to being potentially time-consuming. Incomplete saturation (as evidenced by the ability to readily point to a purportedly saturated resonance) is, in general, ignored, after attempts have been made to minimize it. Of course, there is frequently a trade-off between the problems of spillover and incomplete saturation, as attempts to minimize one of these tend to exacerbate the other.

While we are Believers in saturation transfer, we nonetheless were interested in determining how these (frequently unavoidable) errors propagate into rate constants. For exchange between sites A and B with saturation at A, the ideal Bloch equation, with the usual notation, is:

$$\frac{dB}{dt} = \frac{B_0 - B}{T_1} - k_{BA} B. \quad (1)$$

For incomplete saturation, i.e., residual A of amount \hat{A} , and spillover saturation, i.e., zero-exchange time amplitude of B of amount \hat{B} , under the (reasonable) assumption that \hat{A} and \hat{B} are independent of saturation time, the modified equation is

$$\frac{dB}{dt} = \frac{\hat{B} - B}{T_1} - k_{BA} B + k_{AB} \hat{A}, \text{ with } k_{AB} = k_{BA} \left(\frac{B_0}{A_0} \right). \quad (2)$$

Both \hat{A} and \hat{B} are readily measurable, and of course, the equation (2) is equi-trivial to equation (1), so that knowing the (measured) time-course of saturation, one can fit data to the solution of (2) and obtain k_{BA} .

We have performed this analysis with simulated data, and found, not surprisingly, that $\hat{A} \neq 0$, $\hat{B} \neq B_0$, can lead to non-negligible errors. For example, with numbers appropriate for the creatine kinase reaction in heart, a error of greater than 10% in k_{AB} and 25% in T_1^{CP} results from $\hat{A} = 0.1$, $\hat{B} = 0.9$. This emphasizes the necessity of a) minimizing the spillover and incomplete saturation errors experimentally, and/or b) analyzing the data according to the correct model, eq. (2), and/or c) being very careful in the interpretation of the results of magnetization transfer results. Of course, similar considerations apply to, for example, measurement of T_1 's in exchanging systems by performing an inversion recovery experiment with one resonance saturated.

Please credit this contribution to Gunther L. Eichhorn's subscription.

Yours sincerely,

Richard G. S. Spencer

Richard G. S. Spencer
NIH/NIA; GRC 4-101

James A. Ferretti

George H. Weiss

1. M. Rydzy, R. Deslauriers, I.C.P. Smith, and J.K. Saunders, *Mag. Res. in Medicine* **15**, 260 (1990).

UNIVERSITÉ CLAUDE BERNARD LYON I

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July 2, 1991

(received 7/10/91)

Prof. Bernard L. Shapiro
TAMU NMR News Letter
966 Elsinore Ct.
Palo Alto, CA 94303

Linear prediction and gaussian lines

Dear Professor Shapiro,

It is well known that linear prediction (LP) method works well when NMR spectra can be fairly approximated by lorentzian lines. For K such resonances one may write in the time domain :

$$x(t) = \sum_{k=1}^K A_k \exp(i\phi_k) \exp((-b_k + i\omega_k)t) \quad [1]$$

where $x(t)$ is the NMR signal in the time domain and where A , ϕ , b and ω are respectively amplitude, phase, transverse relaxation rate and angular frequency. R. De Beer and D. Van. Ormondt [in "In vivo Magnetic Resonance Spectroscopy", M. Rudin and J. Seelig, Eds. Springer 1991 - in press] showed that Singular Value Decomposition (SVD) decomposes a gaussian signal according to :

$$\alpha \exp(i\omega t - \beta^2 t^2) \simeq \sum_{n=1}^N A_n \exp(i\phi_n) \exp((-b_n + i\omega_n)t) \quad [2]$$

where α is a particular amplitude and β a relaxation rate. So Eq.[2] shows that SVD considers one gaussian line as a superposition of N lorentzian lines. At low signal to noise ratio (SNR), the SVD technique leads to $N = 1$, and it gives a value for A which is about 30 % larger than α .

Before performing the SVD method, we generally use an enhancement procedure (J.A. Cadzow, IEEE Trans. ASSP, 36, 49 1988) that uses the fact that the number K of spectral lines is perfectly known. The enhanced signal involves K spectral lorentzian components and has the lowest distance from the noisy signal.

So we investigated by simulations the capability of this procedure to quantify a "gaussian signal". If we are dealing with a spectrum made of K gaussian lines, the preceding enhancement procedure considers that any gaussian line is a lorentzian one. This leads to the following approximation :


$$\alpha \exp(i\omega t - \beta^2 t^2) \simeq A \exp(-b + i\omega)t \quad [3]$$

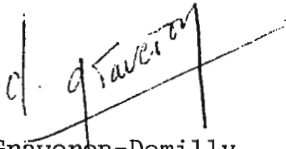
In fact we are looking for the lorentzian line which is the closest to the gaussian line.

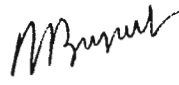
At low SNR, simulations show that A is still 30 % larger than α . We took also $\text{SNR} = \infty$ and we found that Eq.[3] was able to give A with a 5 % amplitude error only.

Finally, since the error is almost independent of the spectral position, we conclude that for a given experiment, it is feasible to determine the relative intensities of the spectral lines without knowledge about the model function of the signal.

Sincerely Yours.


A. Diop


D. Gräveron-Demilly


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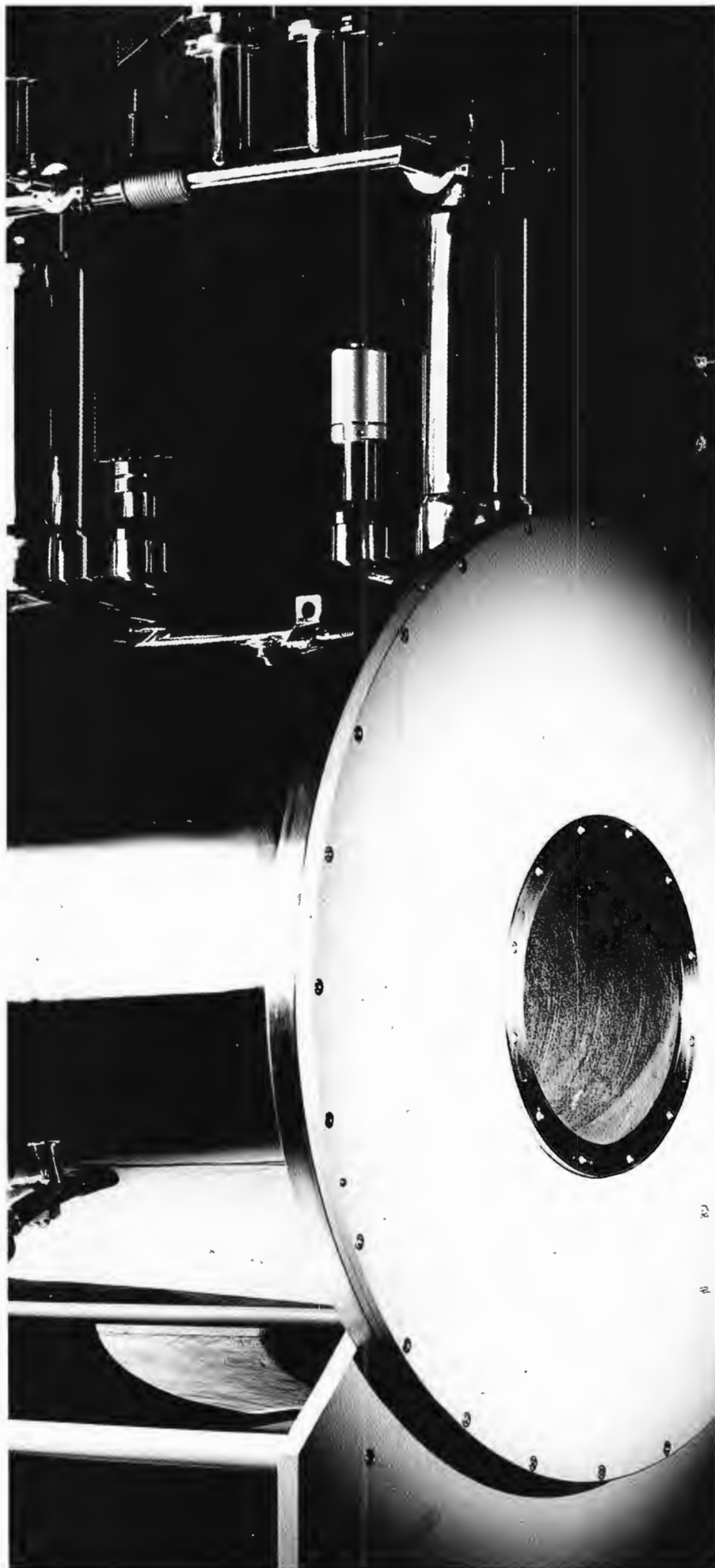
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Dr. Bernard Shapiro
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto, CA 94303

July 10, 1991
(received 7/13/91)

SPREADSHEET FOR FITTING DAMPENED SINUSOIDAL FUNCTIONS

Dear Barry:

It occasionally is necessary to measure effective H_1 power to determine 90° and 180° pulse widths and, for CP/MAS work, to determine accurately the Hartmann-Hahn match parameters. For solids, the 1H power is measured easily by varying the width of the 1H excitation pulse which immediately precedes the cross-polarization period. The plot of signal vs. pulse width is a dampened sine wave with well-behaved parameters; i.e., $PW_{90^\circ} = PW_{180^\circ}/2 = PW_{270^\circ}/3$ within reasonable error (say, $< 0.5 \mu s$). Thus, $\gamma_H H_{1H}$ is known accurately.

The carbon power is measured by putting a variable ^{13}C pulse *after* the CP period, with the pulse phase shifted 90° from the spin-lock pulse phase. One would expect this sequence to produce a nice dampened cosine wave (maximum intensity at zero pulse width) but, at least on my spectrometer, this is not the case. The figure shows a typical data set. The apparent PW_{90° , the point where intensity is zero, is longer than one-half the apparent PW_{180° , where intensity is most negative, and PW_{180° is longer than two-thirds the apparent PW_{270° , and so forth. The reason for this is a shifting of the time base by some uncompensated delay (phase shifting time, perhaps) in the experiment as performed; the data resemble a phase-shifted cosine.

The broken line in the figure is the least-squares 3-parameter fit to the dampened cosine function $y = A \cdot \exp(-\tau/T_d) \cos(2\pi \nu \tau)$, where τ is the variable pulse width, T_d a time constant of dampening, and ν the effective power, $\gamma_C H_{1C}$. The solid line is the fit when the cosine function is allowed to shift along the τ axis: $y = A \cdot \exp(-\tau/T_d) \cos[2\pi \nu (\tau + \delta)]$, where the fourth parameter, δ , is the shift in μs . As judged by the eye and by s , the standard deviation of fit, the shifted function predicts the data very well. More importantly, the $\gamma_C H_{1C}$ value from the 4-parameter fit is much more realistic in establishing Hartmann-Hahn match; the two fits give widely different $\gamma_C H_{1C}$ values.

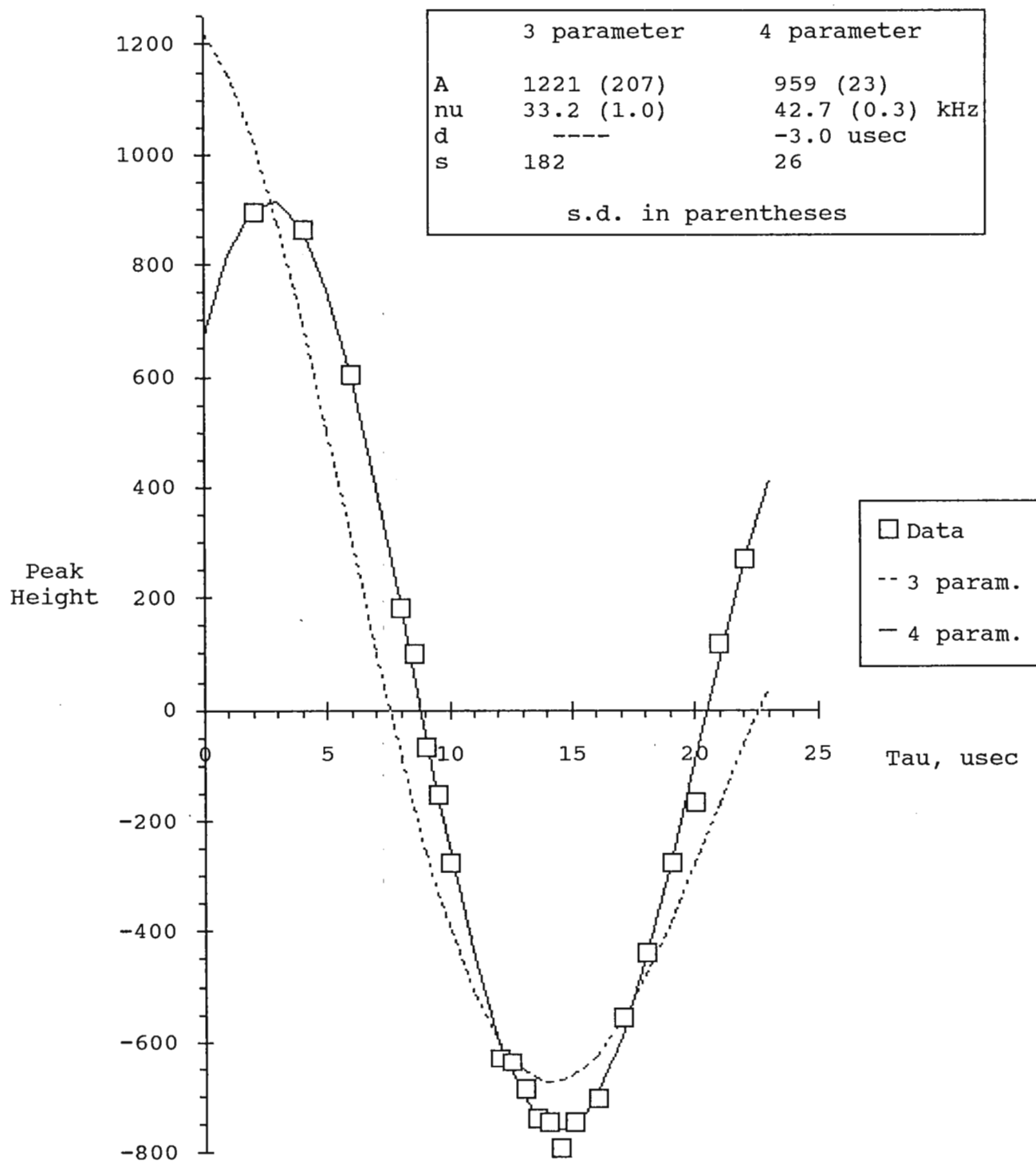
I created this 4-parameter fit by applying the iterative method of Gerhards and Dietrich (*1*). Calculations are conducted easily in a Microsoft® Excel spreadsheet, which I will copy for interested parties who send me a 5 $\frac{1}{4}$ " 2S/2D floppy in a prepaid return-mail envelope. The regression should prove useful in describing the numerous dampened sinusoidal behaviors germane to NMR.

*1. R. Gerhards and W. Dietrich,
J. Magn. Reson. 23, 21 (1976).*

Best regards,

Larry Amos

Aromatic Signal of HMB.
Carbon Gamma Experiment.



Gradient Enhanced Spectroscopy SWAT

GE introduces the use of Switched Acquisition Time (SWAT) gradients to achieve pure phase 2D spectra with quadrature detection in both the acquisition (ω_2) and evolution (ω_1) dimensions without any phase cycling and without an additional set of t_1 data.

One example of a pure phase gradient enhanced COSY spectrum of a solution of 2,3-dibromopropionic acid in benzene- d_6 is shown in Fig. 1. SWAT gradients and a single acquisition per block were used. Data was collected on an Omega 300WB with Microstar actively-shielded gradients. A 5mm inverse probe was built for use within the gradient coils.

Digital resolution of 1.2 Hz in ω_1 and 2.4 Hz in ω_2 was achieved by collection of a 512 x 512 matrix with t_1 evolution time of 840ms and a t_2 acquisition time of 420ms. A single acquisition per t_1 evolution data block and an average recycle time of 1.84s resulted in a 15 minute total collection period.

Since the SWAT gradient method encodes the necessary information in a single t_2 acquisition time, it avoids the collection of additional data blocks required by traditional pure phase methods. This time efficiency is especially important for collection of large multidimensional data sets.

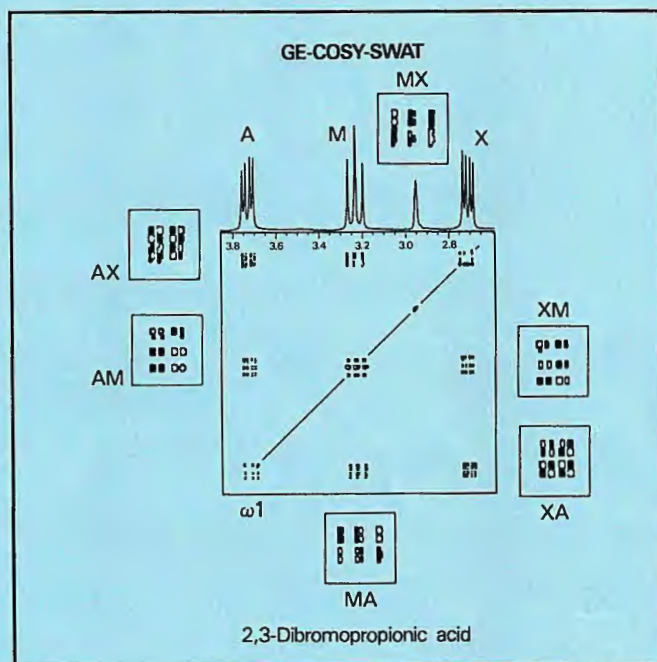


Fig. 1

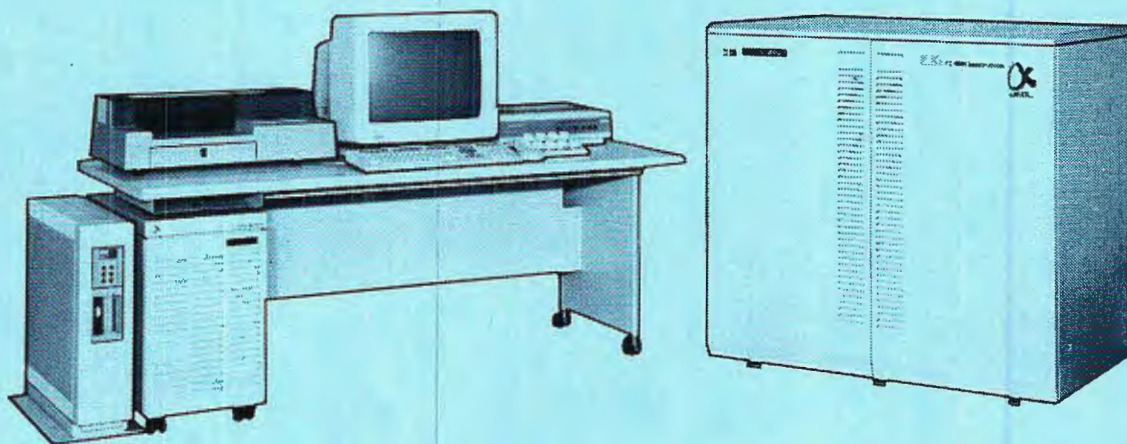
Contour plot of a 300 MHz pure-phase COSY spectrum of a solution of 2,3-dibromopropionic acid in benzene- d_6 acquired with only a single acquisition per t_1 evolution time increment using the GE-COSY-SWAT method. Cross peaks are shown in expanded insets with positive peaks as darkened contours and negative peaks as open contours. A one dimensional spectrum is plotted across the top of the 2D spectrum.



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