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No. 423

December 1993

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FORTHCOMING NMR MEETINGS

International Symposium on Biological NMR, On the Occasion of Professor Oleg Jardetzky's 65th Birthday, Stanford California, **March 24 - 26, 1994**; Contact: Ms. Robin Holbrook, Stanford Magnetic Resonance Laboratory, Stanford University, Stanford, California 94305-5055; Fax: (415) 723-2253; See TAMU NMR Newsletter 422, 47.

Symposium on *In Vivo* Magnetic Resonance Spectroscopy VII, Monterey, California, **April 9 - 10, 1994**; Contact: Radiology Postgraduate Education; Room C-324, University of California School of Medicine, San Francisco, CA 94143-0628; Phone: (415) 476-5731; Fax: (415) 476-9213; For registration, call (415) 476-5808; Fax: (415) 476-0318 See TAMU NMR Newsletter 422, 47.

35th ENC (Experimental NMR Conference), Asilomar Conference Center, Pacific Grove, California, **April 10 - 15, 1994**; Contact: ENC, 815 Don Gaspar, Santa Fe, NM 87501; (505) 989-4573; Fax: (505) 989-1073 See TAMU NMR Newsletter 422, 9.

8th International Symposium on Molecular Recognition and Inclusion, Ottawa, Ontario, Canada, **July 31 - August 5, 1994**; Contact: H. Morin-Dumais, Steacie Institute for Molecular Sciences, National Research Council of Canada, 100 Sussex Drive, Ottawa, ON K1A 0R6, Canada; (613) 993-1212; Fax: (613) 954-5242 See TAMU NMR Newsletter 419 34

Gordon Conference on Order/Disorder in Solids, New London, New Hampshire, **August 7 - 12, 1994**; Contact: Prof. M. A. White, Dept. of Chemistry, Dalhousie University, Halifax, Nova Scotia, Canada B3H 4J3; Tel. (902) 484-3894; Fax: (902) 494-1310. See TAMU NMR Newsletter 421, 44.

36th ENC (Experimental NMR Conference), Boston, MA, **March 26 - 30, 1995**; Contact: ENC, 815 Don Gaspar, Santa Fe, NM 87501; (505) 989-4573; Fax: (505) 989-1073

12th International Meeting on NMR Spectroscopy, Sponsored by the Royal Society of Chemistry, Manchester, England, **July 2 - 7, 1995** [sic]; Contact: Dr. J. F. Gibson or Ms. G. B. Howlett - See TAMU NMR Newsletter 415, 5; Phone: (44-71) 437-8656; Fax: (44-71) 437-8883.

ISMAR 1995, Sydney, NSW, Australia, **July 16-21, 1995** [sic]; Contact: Dr. Wm. A. Bubb, Secretary, Univ. of Sydney, Dept. of Biochemistry, Sydney, NSW 2006, Australia. See TAMU NMR Newsletter 419, 26.

Additional listings of meetings, etc., are invited.

UNIVERSITY OF TURKU
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SF-20500 TURKU, FINLAND

Professor B. L. Shapiro
TAMU NMR Newsletter
966 Elsinore Court
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Professor
Jorma Mattinen
Åbo Akademi
Institution för Organisk Kemi
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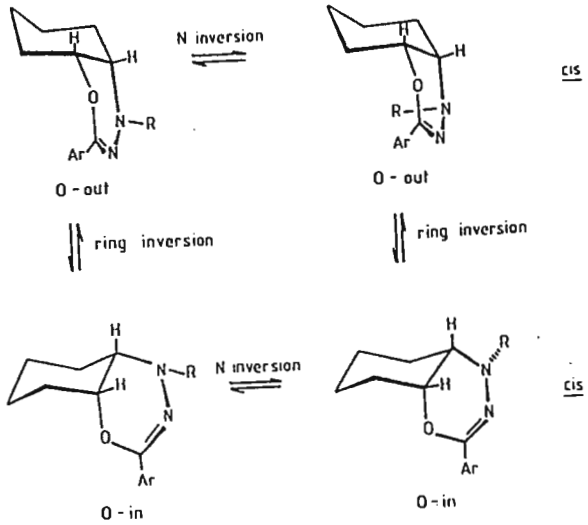
Finland
tel -21 - 654 720
fax -21 - 654 866
email jmattinen@finabo.abo.fi

(received 10/25/93)

A DYNAMIC FOUR COMPONENT EQUILIBRIUM

Recently we began studies on cyclohexane-fused 1,3,4-oxadiazanes shown in Scheme 1. The trans-fused bicycle can be regarded as anancomeric except the possible nitrogen inversion. Instead the cis-fused system (Scheme 1) exhibits an interesting four component equilibrium where both the ring and nitrogen inversions are present. Both of the latter processes can be frozen in a dynamic ^{1}H NMR experiment. However, the range of coalescence was very wide and the signals were still broad at 173 K. A more detailed analysis of this dynamic system (Scheme 1) is underway.



SCHEME 1

Jorma Mattinen
Department of
Organic Chemistry
University of Åbo Akademi
FIN-20500 Åbo,Finland

Kalevi Pihlaja
Laboratory for
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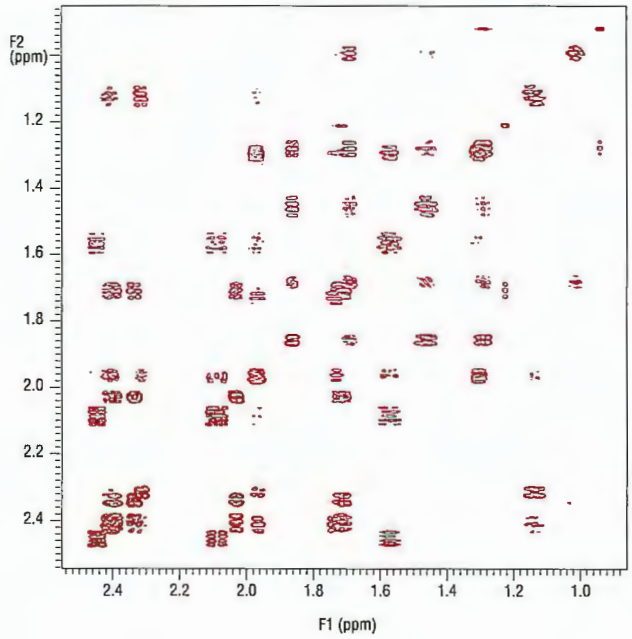

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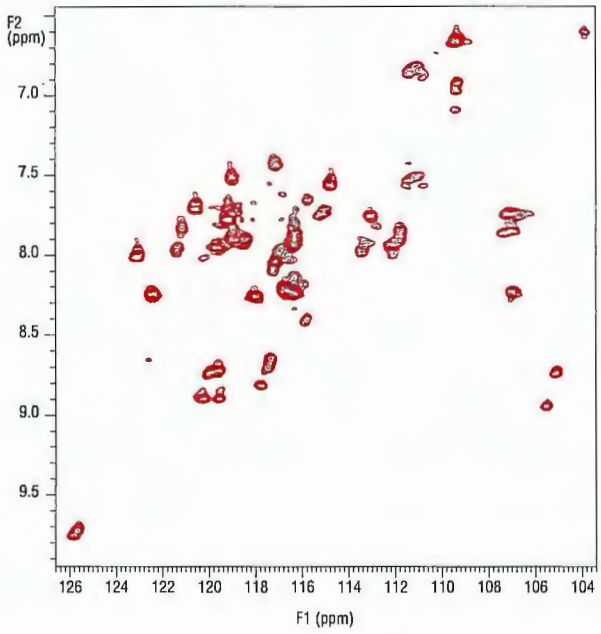

These pulsed field gradient spectra of Androstene in $CDCl_3$ and a 1 mM ^{15}N enriched protein in 90% H_2O were acquired on a 750 MHz UNITYplus™ NMR spectrometer equipped with Varian's PFG apparatus.

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November 22, 1993 (received 11/23/93)

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

Dear Barry:

The purpose of this note is several fold. First, I would like "resubscribe" to the "Newsletter." Secondly, as you can tell I have moved from the University of South Carolina to Battelle's Pacific Northwest Laboratories (PNL). The move from academics to a DOE National Lab is a transformation that I have yet to completely comprehend. However, what I would like to do is describe the staff and NMR lab that we are assembling at Battelle.

The present staff is composed of Mike Bowman, Herman Cho, Mike Kennedy, Robert Wind, and myself. We are currently seeking an additional staff person for liquid state experiments. The main thrust of the laboratory is health effects associated with the Hanford site and interfacial problems related to problems of surface chemistry, sensor development, materials chemistry, etc.

To this end the instrumentation in the laboratory is outstanding. I was able to bring my homebuilt 400-wide bore from South Carolina. To that we are about to add a suite of UNITY plus instruments from Varian, i.e. 300 and 500 MHz wide bore systems for solids and 500 and 750 MHz narrow bore systems for high resolution liquid state work. We are eagerly awaiting the 750 system. By the time you publish this note, the 750 system should be installed and in the final check-out phase. As I understand it, this will be the first 750 MHz system delivered to any customer.

We are also collaborating with Professor Gary Drobny at the University of Washington on several projects. He is constructing the console for our 1 GHz system. The magnet is being developed by Oxford Instruments. Moreover, we are also working with Gary on several DNA-protein and damaged DNA-protein interaction investigations. The 1 GHz spectrometer should be installed around the end of 1996 in our new Environmental Molecular Science Laboratory (EMSL). The purpose of this laboratory is to bring world class molecular science to environmental problems. As my program gets off the ground, I look forward to communicating some of excitement to you via future contributions to the Newsletter.

Sincerely,

Paul

Paul D. Ellis, Associate Director
Macromolecular Structure and Dynamics
Molecular Science Research Center

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Dr. B. L. Shapiro
TAMU NMR Newsletter
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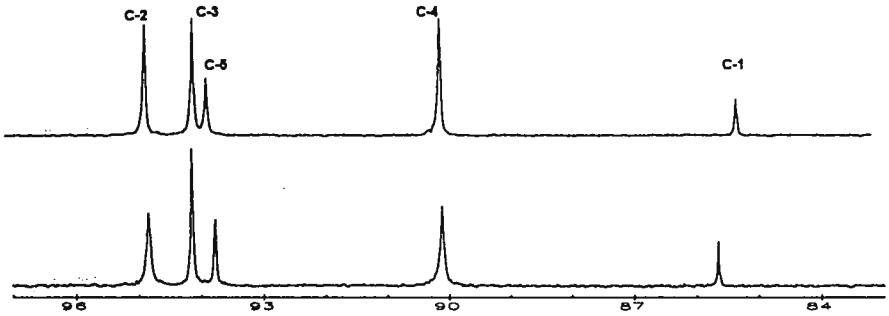
November 10, 1993
(received 11/20/93)

Differential line broadening in dianions of substituted cyclooctatetraenes

Dear Dr. Shapiro,

In collaboration with Dr. Staley's group at CMU, we are doing solution studies of intramolecular electron exchange between a doubly charged cyclooctatetraenyl (-COT) ring and a neutral -COT ring separated by a spacer group with σ or π bonds. These systems are unusual since the rate of two-electron transfer (and transfer of two counterions) can be determined from NMR lineshape analysis or magnetization transfer experiments. This is possible because the neutral ring has an unfavourable ground state conformation (boat shape) for the electron transfer and therefore the neutral and the dianion ring carbons exchange slowly on the C-13 NMR time scale.

For different COT dianions, we have at both laboratories noticed unexpected line broadenings at increased temperature when the counterion is K^+ and the solvent is THF-d_8. An example is provided in the two C-13 spectra of COT-Si$(CH_3)_2$-COT^{2-} below. In the upper spectrum (-10 °C) the intensities/linewidths are as expected for the five different carbons in the dianion ring (C_{2v} symmetry). However, in the lower spectrum (+24 °C), the C-2,8 and C-4,6 peaks are broader than the other three peaks.



The signals were assigned from 2D experiments. AM1 calculations show that the HOMO has large coefficients at the C-2,8 and C-4,6 positions. Considering that the tightness of the $-COT^{2-}/K^+$ ion pair in THF increases at higher temperature, we find it reasonable that the differential line broadening has its origin in an interaction between the K ions and the carbanion HOMO. We now undertake studies in order to reveal the nature of such an interaction and to examine other possibilities for these observations. A K-39 decoupling experiment is part of this study, and we are now able to do this with our triple resonance probe (H-1,C-13,BB) on our new Bruker AMX2 500 instrument.

Readers are most welcome to send comments regarding this type of line broadening.

Please credit this contribution to Dr. Ulf Edlund's account.

Sincerely,

Patrik Boman and Bertil Eliasson
Postal address: S-901 87 Umeå
Sweden

Russell A.Grimm and Stuart W. Staley
Dept. of Chemistry
Carnegie Mellon University
4400 Fifth Avenue, Pittsburgh, PA 15213

e-mail to B.E. : bertil@picea.chem.umu.se

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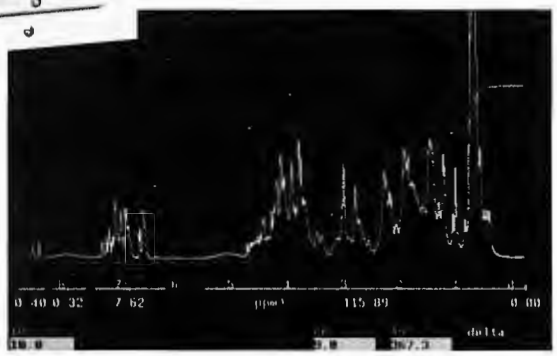

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Dr. B. L. Shapiro
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto
California 94303
U.S.A.

10 November 1993
(received 11/15/93)

Dear Barry,

Protein Kinase C Inhibitor Dynamics

Protein Kinase C (PKC), an enzyme which phosphorylates serine or threonine hydroxyl groups, has a pivotal role in signal transduction, and is therefore involved in the pathogenesis of numerous disease states such as cancer, rheumatoid arthritis, asthma, AIDS and psoriasis. Synthetic manipulations of the potent but non-selective PKC inhibitor, staurosporine (I),[1] led to a number of interesting lead compounds, e.g. the bisindolylmaleimide (II).

Broad peaks in the ^{1}H or ^{13}C spectra of (II) suggested restricted rotation about one or both of the bonds shown by arrows. Apart from the headaches caused by this broadening for the spectroscopists trying to characterise compounds of this type, the dynamic behaviour is quite interesting. The indole rings, precluded from coplanarity for steric reasons, can exist syn or anti to each other relative to the plane of the maleimide ring (Scheme). Non-planarity is confirmed by large upfield chemical shifts for H-4 of the indole relative to staurosporine. Low temperature ^{1}H nmr spectra of (II) show two forms in the ratio 2:1. Analysis of the ring current shifts allows assignment of the major form as syn, with a conformation close to that observed in the x-ray of related bisindolylmaleimides. The rotational barrier is approximately 15.6 Kcal/mole.

NMR studies of the dimethylated model compound (III) were quite surprising. Again two forms were present at lower temperatures, and the major form is again syn, with a rotational barrier of 13.0 Kcal/mole. The indole rings in (II) and (III) are not stacked parallel to each other but rather in positions as close as possible to orthogonal (IIIA) (see Scheme below). At lower temperatures (<213°K) peaks due to the syn form start to broaden further indicating a second rate process (Fig). Our best guess so far is that the syn form indulges in a flip-flop exchange between two dissymetric mirror image forms. The anti form does not appear to show this phenomenon.

I hope this reinstates us on your Newsletter circulation list.

Yours sincerely,

Dr W A Thomas

Mr I W A Whitcombe

1. R A Bit et al, J.Med.Chem., 1993, 36, 21-29.

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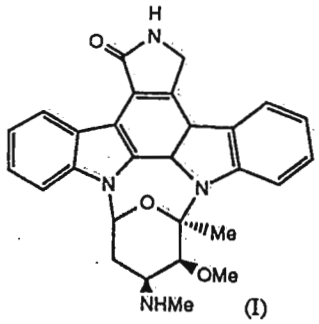
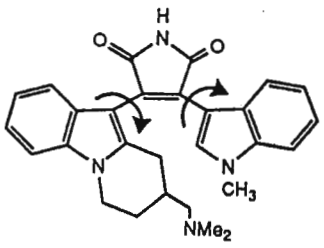
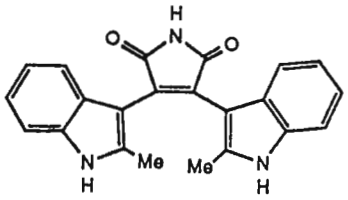


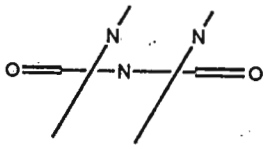
(I)



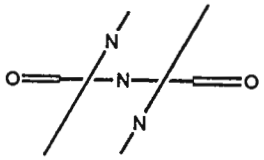
(II)



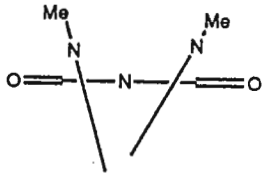
(III)



(II) SYN

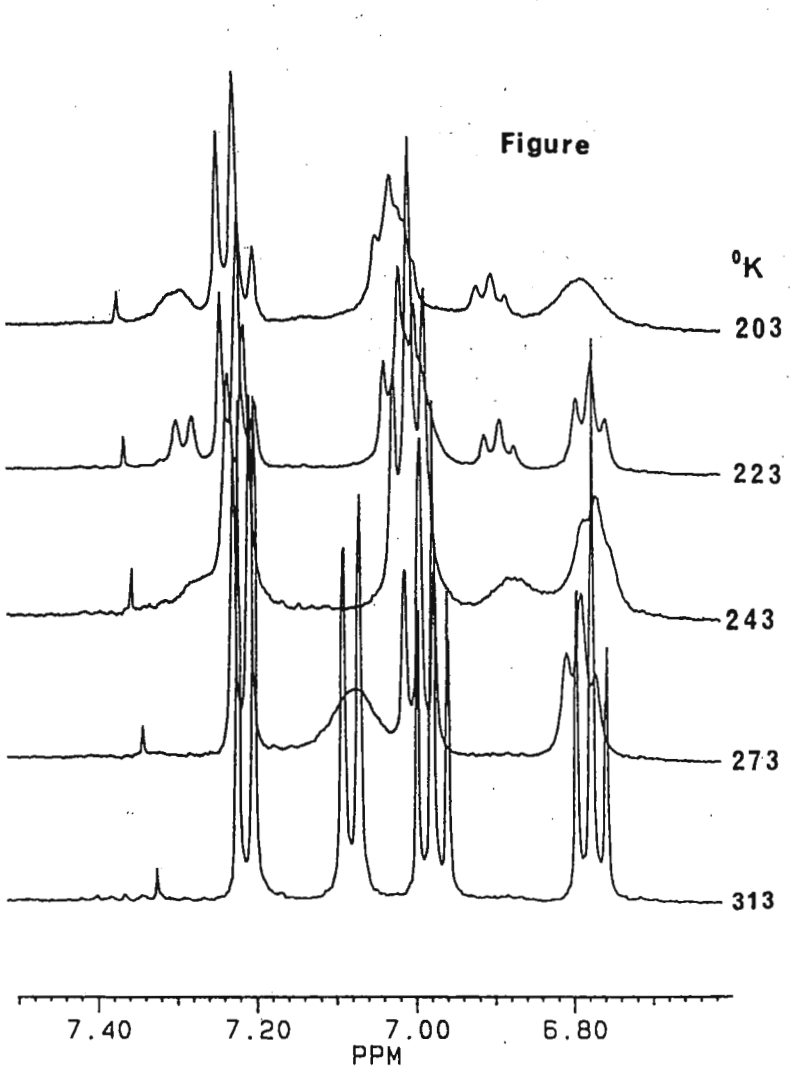


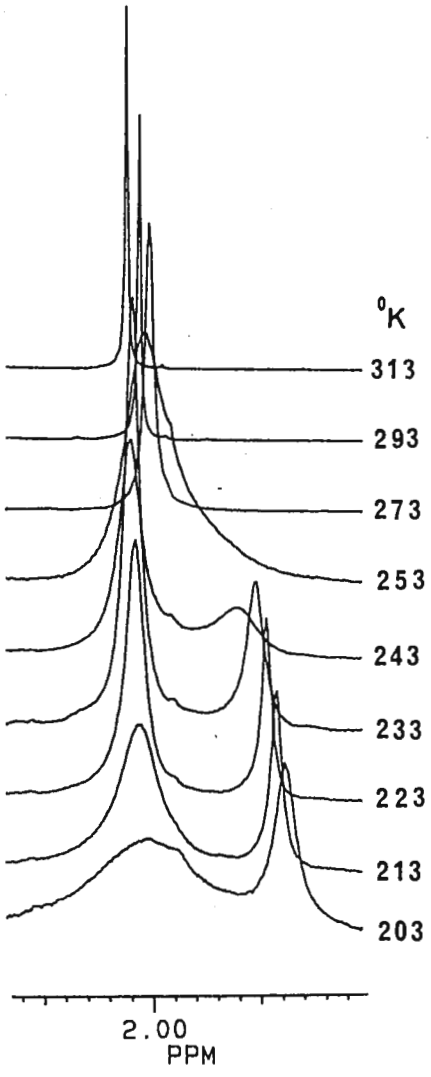
(II) ANTI



(IIIA) SYN

Figure





BRUKER

OVERSAMPLING AND DIGITAL FILTERING

IN MULTI-DIMENSIONAL NMR SPECTROSCOPY

Oversampling and digital filtering in NMR has many advantages, which are well documented and proven in the NMR literature:

- **improvement of dynamic range**[1]
- **elimination of baseline artifacts**[2]
- **improved S/N, sharp filter-cutoffs, absence of folded peaks**[3]

Here we wish to report the utility of digital filters to acquire a "fingerprint" region in a 2D NOESY experiment on 2 mM BPTI. Only the region from 5.0 to 11.4 ppm is acquired (1 hour 50 minutes), and all other spectral regions, including water, are filtered out. In figure 1, traditional analog filters were used, and the aliased water signal is clearly visible folded around the negative axis at 11.4 ppm, since simultaneous acquisition mode was employed. In figure 2, the water is completely filtered out, and not folded back into region above 10 ppm. The "fingerprint" region acquired here with oversampling and digital filtering in F2, has considerably less artifacts and contains more structural information than the equivalent experiment with analog filters. Moreover, due to the ideal phase linearity of digital filters, the baseline is extremely flat.

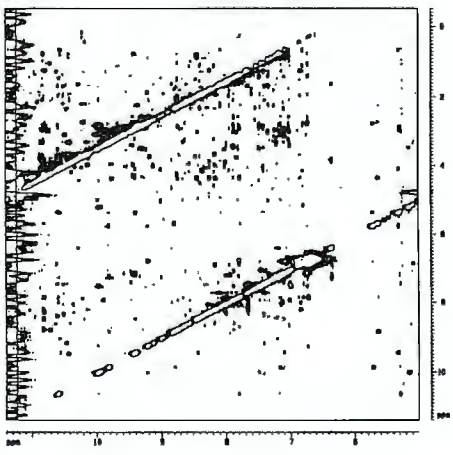


Figure 1: Analog Filter

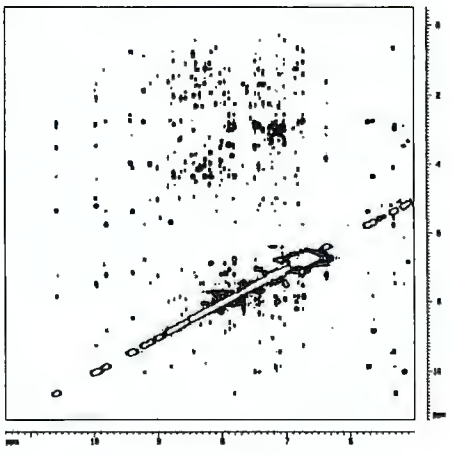
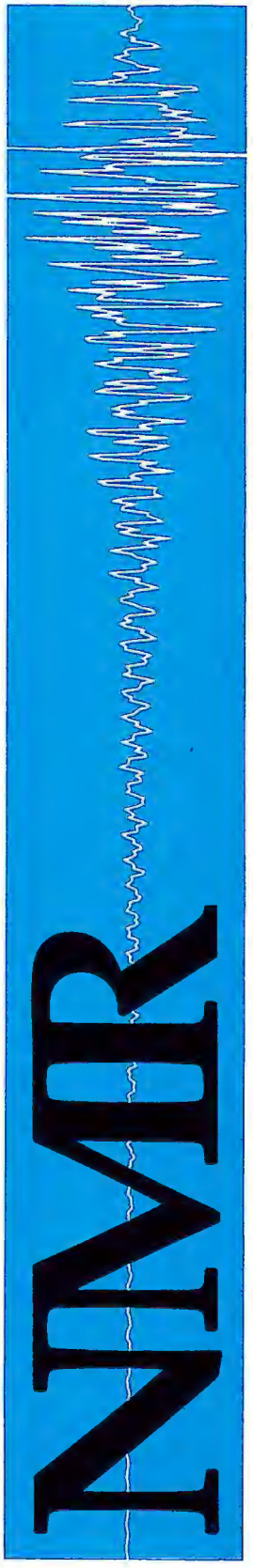


Figure 2: Digital Filter



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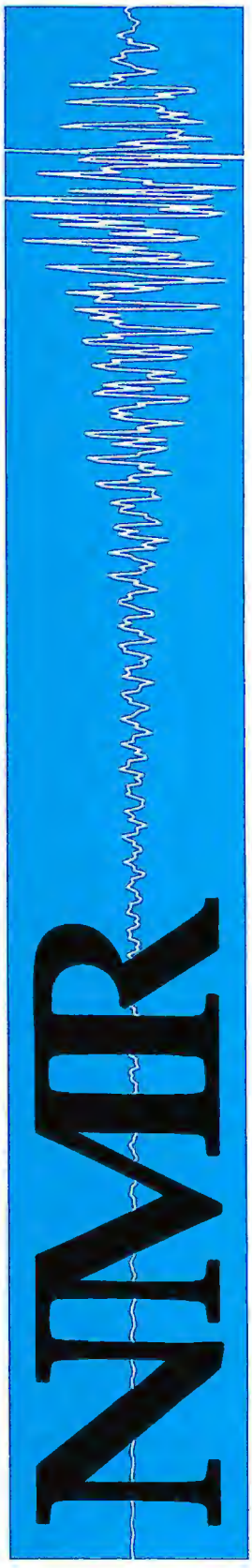

For many years NMR spectroscopists have expressed the desire to acquire small "fingerprint" regions within a multi-dimensional NMR experiment, with the flexibility to match the spectral width to the region of interest without concerns over aliasing and artifacts. This type of customer requirement is typical in modern NMR structure determination, where the major features of the homonuclear and heteronuclear spectra often are well known, but repeated experiments or experimental modifications are made to extract the maximum information content from selected "fingerprint" regions. With the availability of modern high-fidelity, digital signal processors (DSP), Bruker could finally meet these customer requests when designing the revolutionary AVANCE™ series of NMR spectrometers.

In a previous AVANCE application note, the extremely steep cut-off of digital filters has been demonstrated in a 1D NMR spectrum of ethylbenzene[3]. In the example shown, an AVANCE DMX400 was used to acquire proton homonuclear data with a 16 bit digitizer in oversampling mode at sampling rates up to 400 kHz (200 kHz bandwidth). The oversampled FID is immediately digitally filtered by DSPs, so that the total amount of data which needs to be stored is not greater than in a conventional NMR experiment, but has a greater dynamic range.

Oversampling and digital filtering fulfills a longstanding requirement by NMR spectroscopist to be able to acquire selected "fingerprint" regions within multi-dimensional NMR spectra within a shorter time period, but without folding (or aliasing) problems for more efficient structural NMR research. This application note demonstrates that as predicted in the literature, oversampling and digital filtering indeed provide the expected benefits in terms of suppression of artifacts, and improved spectral quality.

References:
1. M.A. Delsuc, J.Y. Lallemand, J.Magn.Res. **69**, 504-507 (1986).
2. G. Wider, J.Magn.Res. **89**, 406-409 (1990).
3. Bruker Application Note "Oversampling and Digital Filtering", July 1993.

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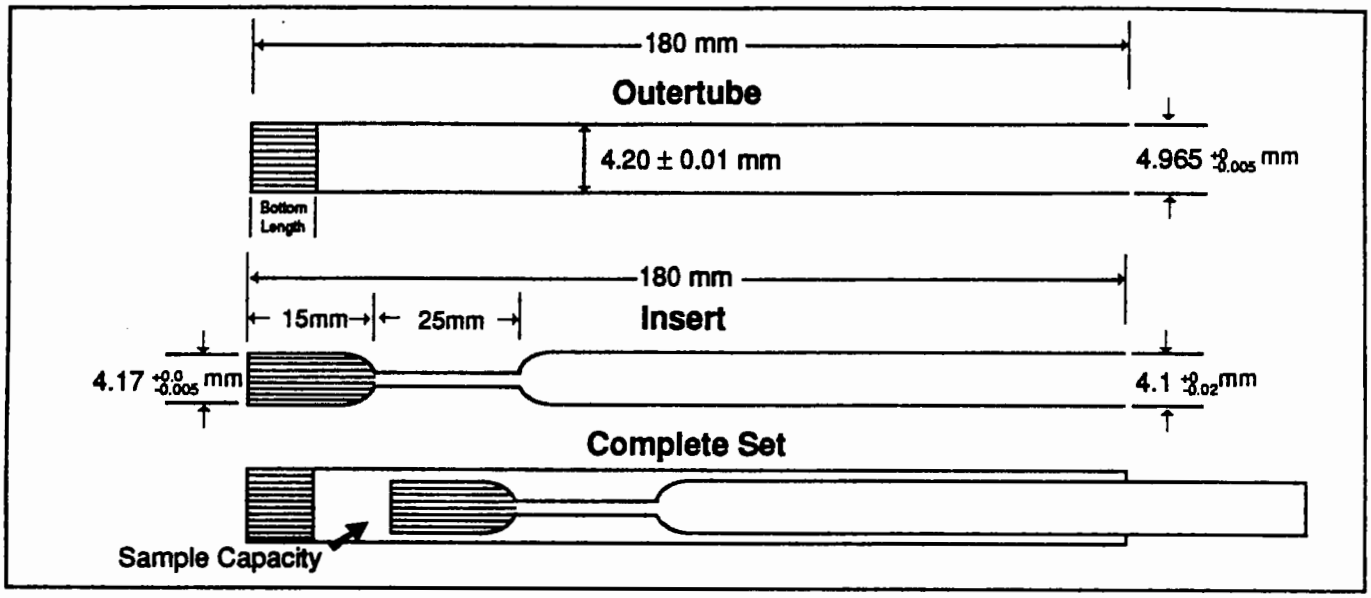

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Complete Set	Insert length (mm)	Insert ID (mm)	Insert OD (mm)	Outertube length (mm)	Outertube ID (mm)	Outertube OD (mm)	Bottom* length (mm)
BMS-005B	180	2.6	4.1	180	4.2	4.965	8
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***For best results, choose the one that matched your probe coil height most closely.**

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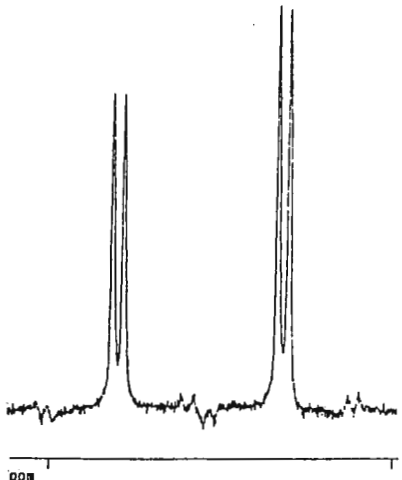
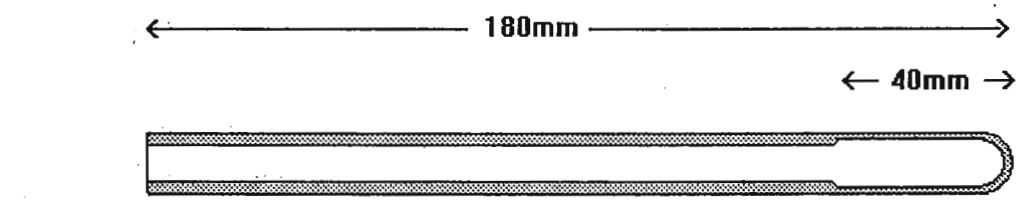
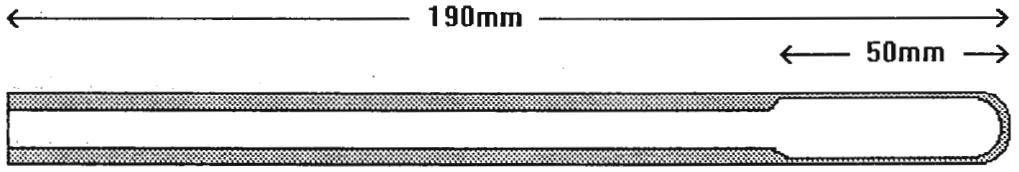


Fig.1a Fig.1b

The spectra of 20mm sucrose in D_2O were obtained with a single scan without apodization prior to Fourier transformation on a Bruker AMX-600 spectrometer at 298 K. By using Shigemi high quality 5mm standard tube (Fig.1a) and the Shigemi highly sensitive thin wall 5mm tube (Fig.1b), the spectra confirms a sensitivity enhancement of about 10%.



PST-001 and PST-002



ST8-001,ST8-002, ST10-001, and ST10-002

O.D. (mm)	Product Number	Wall (mm)	Concen - tricity/ Camber (μ)	OD (mm/in)	ID (mm/in)
5.0	PST-001	0.21	20/ 8	4.96 + 0.00 - 0.01	4.54 ± 0.01
	PST-002	0.21	40/15		
8.0	ST8-001	0.25	40/ 8	8.00 + 0.00 - 0.01	7.52 ± 0.01
	ST8-002	0.25	50/15		
10.0	ST10-001	0.25	40/ 8	9.98 + 0.00 - 0.01	9.52 ± 0.01
	ST10-002	0.25	50/15		

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P.O Box. 23-34
TAIPEI, TAIWAN, REPUBLIC OF CHINA
PHONE: 3635357
FAX: 886-2-3636359
886-2-3630290

Professor B.L. Shapiro
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto, California 94303
U. S. A.

October 18, 1993
(received 10/26/93)

Dear Prof. Shapiro,

"Resolution of ^{25}Mg NMR Resonance in ATP Solution"

Many ions of biological significance are lack of convenient physical and spectroscopic properties to study their binding to specific sites on biomolecules. Therefore, NMR studies provide a unique technique to observe such interaction. Magnesium ion is certainly among the most important inorganic ions in most living systems. However, progress in the applications of ^{25}Mg NMR spectroscopy has been much retarded primarily because of experimental inaccessibility due to poor chemical shift separation blurred further by low sensitivity, acoustic ringing, and sizable quadrupole relaxation effects. Furthermore, spectral estimation of many kinds of spectroscopic signals is usually based on the Fourier transform. This method is computationally very efficient but suffers from some well-known drawback such as sinc wiggling artifacts by zero filling, phase distortion due to windowing or apodization processes, its inability to distinguish between signal and noise, and particularly, the limited resolution constrained by strongly damped or truncated data.

To overcome the above problems inherent in ^{25}Mg NMR as well as many other FT spectroscopies, we present a novel approach to achieve resolution and accurate determination of NMR spectral parameters. By applying the matrix property mappings (1) and minimum-norm linear prediction with singular value decomposition (2) directly to the time domain signals, we can significantly suppress the noise and achieve a resolution far exceeding the conventional Fourier transform. Monte Carlo simulations for resolving two very closely-spaced exponentially damped sinusoids have verified that the present method provides not only higher estimation accuracy for the spectral parameters but also lower S/N threshold. We have further verified the feasibility of this method by applying it to study the chemical shifts of $^{25}Mg^{2+}$ on binding to ATP. The result is shown in Fig(1). The broad line shown in (B) corresponds to Mg-ATP species.

Yours sincerely,

Lian-Pin Hwang

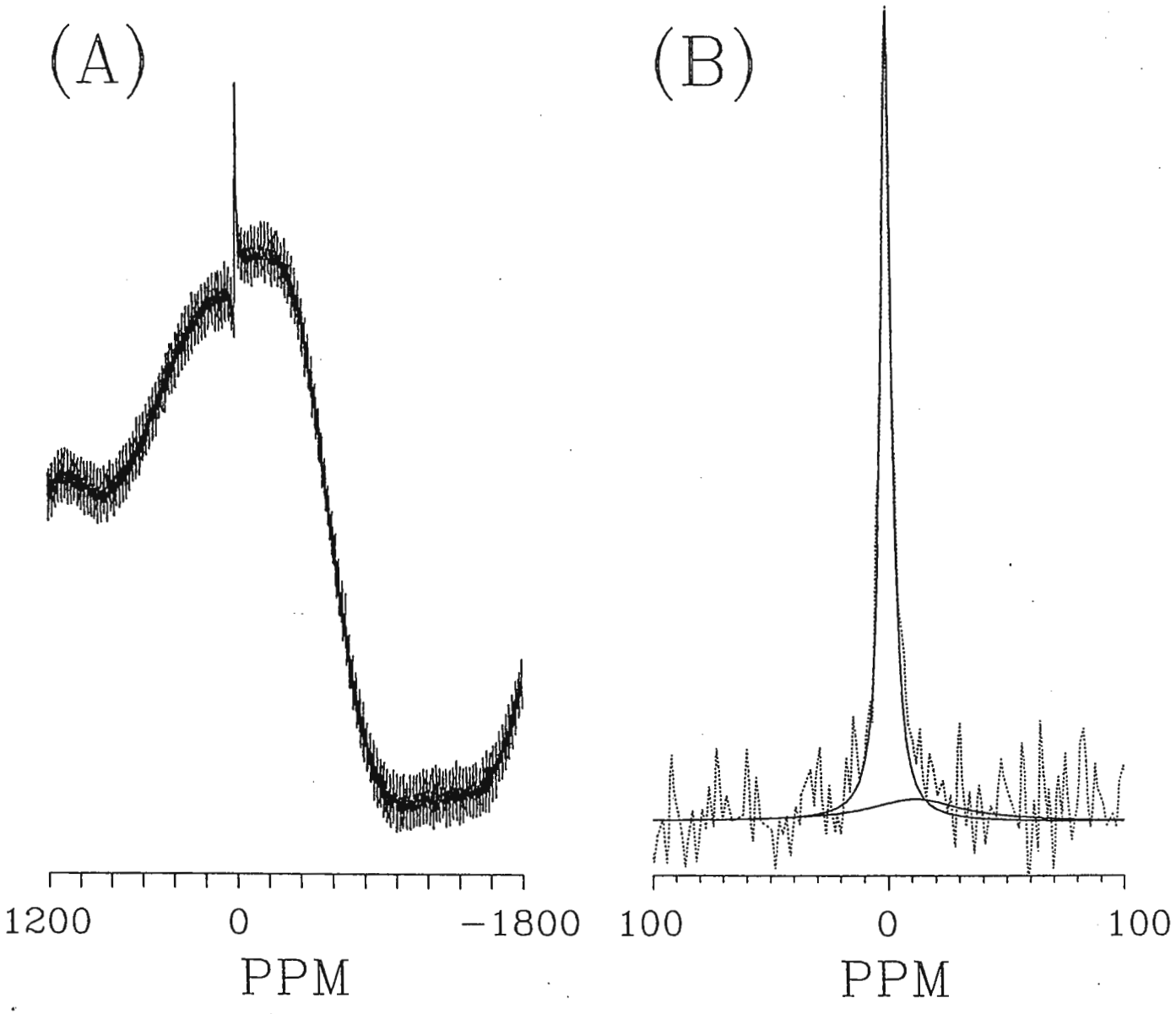
Yung-Ya Lin Nien-Hui Ge Lian-Pin Hwang
Professor of Chemistry

Refferences :

(1) Y.Y. Lin and L.P. Hwang, *J. Magn. Reson.* **103A**, 109(1993)

(2) R. Kumaresan and D. W. Tufts, *IEEE Trans. Aerosp. Electron. Syst.* **AES-19**,134(1983)

Fig(1): Lin et al.



Fig(1) : The nature abundant ^{25}Mg Spectra obtained at 273 K and 30.62 MHz in an aqueous solution containing 0.2 M $MgCl_2$ and 0.05 M ATP at pH = 6.9 with 0.8 M Tris buffer. Initial 128 data points in the FID were used in the analysisof spectral lines. Other parameters used are NS = 240000, DW = 5μs and D3 = 50 μs. The spectrum shown in (A) is obtained with no left shift from Fourier transform of FID in which the ^{25}Mg resonance appears at 0 ppm. The resolved spectrum after removing the acoustic ringing components are shown in (B). The $[Mg^{2+}]$ /$[Mg^{2+}$-ATP] ratios before and after correcting the effect of initial deadtime relaxation are 6.1/1.0 and 2.9/1.0, respectively.

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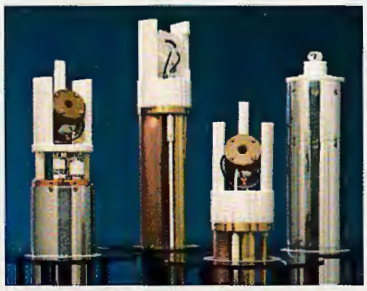

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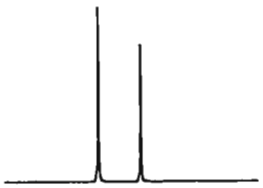
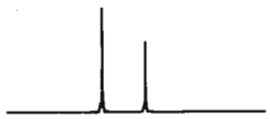
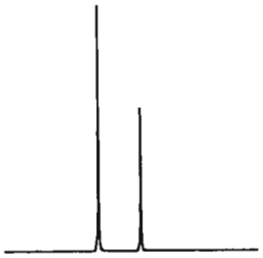


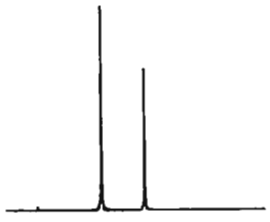
Variable amplitude cross polarization (VACP) *under* −2.6 KHz *mismatch condition*



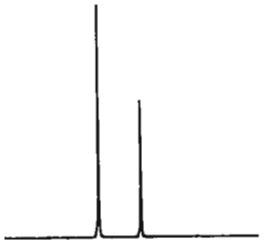
Deliberate "mismatch" of −1.9 KHz



VACP *under normal match condition*



Normal "ideal" matched conditions



VACP *under* +2.0 KHz *mismatch condition*

Deliberate "mismatch" of +1.4 KHz

Chemagnetics would like to thank Professor Steve Smith for suggestion of this work and useful discussions during its implementation.

United States Department of Agriculture
Agricultural Research Service
Midwest Area National Center for Agricultural Utilization Research
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Peoria, Illinois 61604
Telephone: 309-685-4011
FAX: 309-681-6686

October 19, 1993
(received 10/25/93)

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

RE: Aluminum-27 NMR of "Aluminum-Free" Ceramic Rotors, Update

Dear Barry:

In 1988 we wrote about an Aluminum-27 NMR signal from our SiN rotors (1). This year the manufacturer of my 5mm high speed probe is providing SiN rotors without this signal. The new rotors are a darker shade and can be readily distinguished from the old rotors when Aluminum-27 is to be observed. The spectra I show for comparison employ the same single crystal of zunyite (aprox. 5.5 mg) centered in both kinds of rotors. I felt this was an easy way to guarantee that the same amount of "sample" Aluminum was in the same part of the coil. The centering was acomplished by placing the crystal in a rotor which had been filled with a loosely packed powder and previously spun to create a small cylindrical cavity. 10 kHz MAS with a single air supply was sufficient due to the relatively small quadrupolar coupling constants. The F.W. of zunyite is about 1250, depending on the number of OH- groups vs. F-, with only one tetrahedral Al per F.W. and as can be seen from the spectra below there is no trace of a rotor signal at 108 ppm or anywhere else with the new rotor (I also ran an empty rotor, noise and roll not shown).

(1) A. R. Thompson and R. E. Botto private communication. (1988).

78 MHz Aluminum-27 spectra with old SiN rotor left and new rotor right.

Sincerely,

Arthur Thompson

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Department of Chemistry
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FAX: (0203) 524112
DR. OLIVER W. HOWARTH

University of Warwick
Coventry CV4 7AL

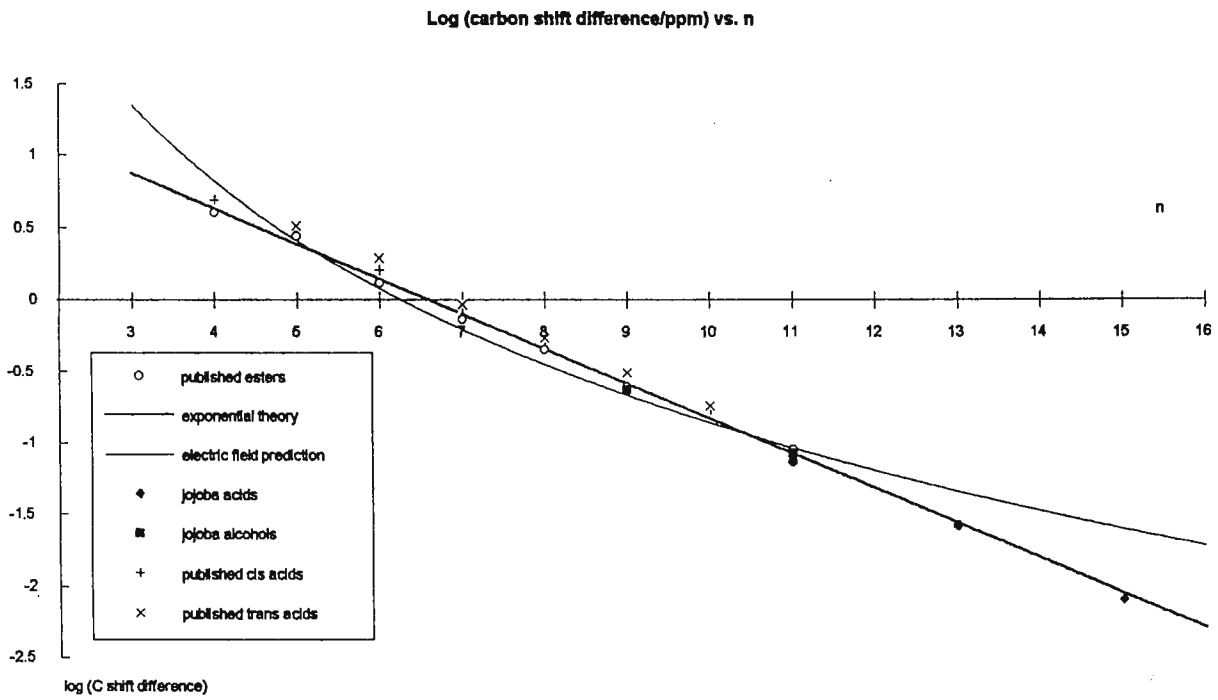
Department of Physics
Tel: (0203) 523403
FAX: (0203) 692016
PROF. RAY DUPREE

1st November 1993

Sigma-inductive interaction through up to 14 C-C single bonds

Dear Barry,

In the course of a collaboration with the Italian Olive Oil Research Institute, we believe have found our way to a disproof of a longstanding theory about the ^{13}C chemical shifts of the double bond carbons in the chains of monounsaturated fatty acid and alcohol esters. The current wisdom is that these resonances separate, almost symmetrically, from their 129.73δ value in very long chains, because of the electric field that arises from the ester moiety. The original (1973) proposers of this theory predicted an $n^{-3.5}$ falloff of the resulting splitting with n, the position of the double bond. However, we now have much better resolution available to us, down to ca. 3 ppb, and so we can test this theory to larger vales of n. It fails. But, as the log plot below shows, a 1.75^{-n} dependence fits remarkably well. In other words, each intervening methylene group, beyond the first few, provides a ÷1.75 attenuation of the influence of some bond dipole. This argues strongly for a σ-inductive mechanism for the transmission of the electric field.



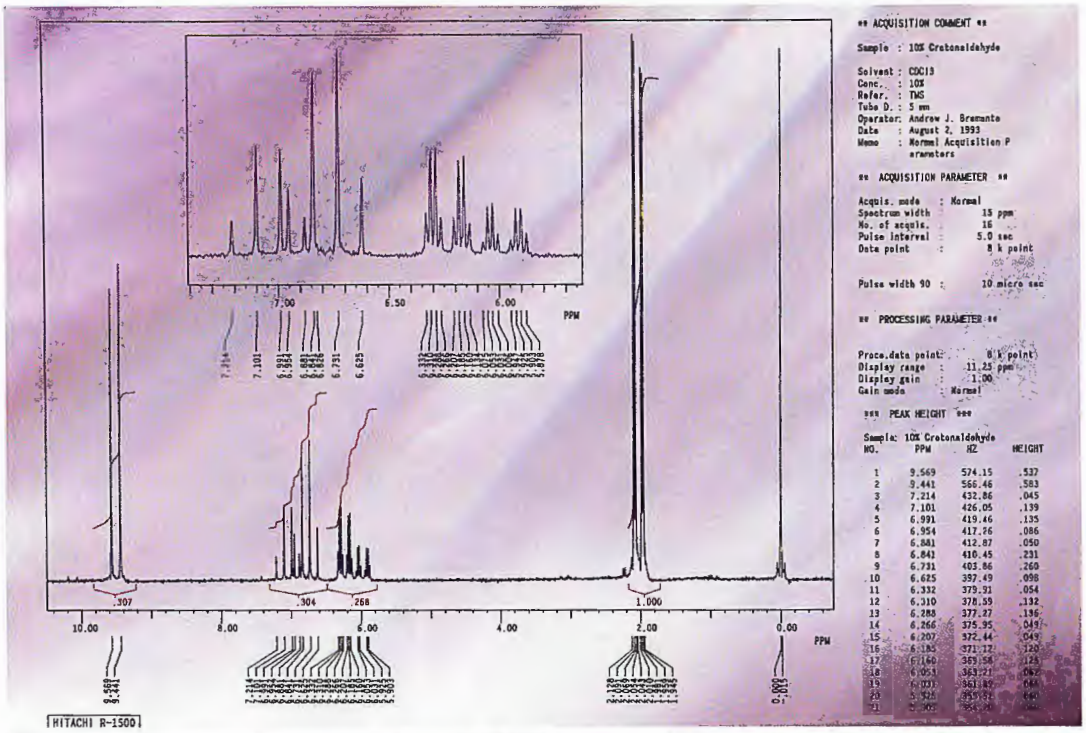
Much of the new data in the above figure comes from a high-resolution study of jojoba oil. This is chemically unusual, in being composed of double (acid + alcohol) *cis*-monounsaturated long-chain monoesters. The shifts from either chain fit the above loglinear relationship nicely, and there is not too much difference between the two chains. Even *trans*-acids show the same gradient, though with somewhat larger shift separations.

We may also apply a further test. Because alkyl esters are known to have mainly linear conformations, the sign of the C=C shift separation should reverse between the acid and the alcohol chains, on the electric field theory, but probably not on the inductive theory. We have therefore performed complete C + H assignments for some reasonable (and assignable!) model esters, such as ethyl petroselinate (n = 6), and *cis*-5-decenyl acetate. We find no reversal of sign; the carbon nearer to the ester moiety is always the more shielded. The same conclusion is supported by the details of the asymmetry of the jojoba data. So it looks as if the through-space electric field is not very important for the shifts in these long chains.

Very best wishes,

Oliver Howarth

(received 11/8/93)



This R-1500 FT-NMR spectrum of crotonaldehyde represents a 16 pulse acquisition; each pulse was 10 µsec with a pulse interval of 5 seconds.

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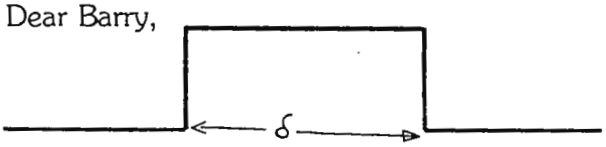
THE ROYAL INSTITUTE OF TECHNOLOGY
Department of Chemistry, Physical Chemistry
Professor Peter Stilbs

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

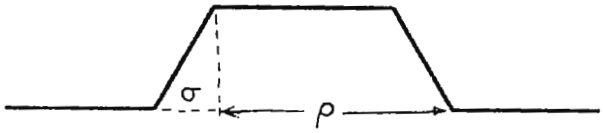
November 2, 1993
(received 11/8/93)

Re: Ultimatum, shaped field gradient pulses, equipment sought

Dear Barry,



$$A(2\tau) = A(0) \cdot e^{-\gamma^2 G^2 \delta^2 D(\Delta - \delta/3)}$$



$$e^{-\gamma^2 G^2 D(\Delta\rho^2 - (\frac{\rho^3}{3} - \frac{1}{6}\rho\sigma^2 + \sigma^3))}$$

Thank you for your Ultimatum. Royal Institute of Technology has now reorganized (or rather disorganized) so that we are no longer a separate department, hence the slight address change above. Like all bureaucrat-induced changes, it has proven to be for worse.

Anyway, we persist in trying to do some work, and are still very much focussed on pulsed-gradient spin echo work on self-diffusion. We have recently started to routinely use shaped field gradient pulses instead of the traditional rectangular, as generated by a gradient driver designed by W.S. Woodward at University of North Carolina, Chapel Hill. The effect of non-rectangular pulses on the Stejskal-Tanner pulsed-gradient experiment was already discussed by Gross and Kosfeld (Messtechnik 7-8 (1969) 171). Some later studies are those of Price and Kuchel (JMR 94 (1991) 133) and Merrill (JMR Ser. A 103 (1993) 223), but actual use of non-rectangular pulses has been rare in practice, primarily because their generation is more complicated than that of rectangular ones. A benefit of modern current-regulated gradient drivers is the inherent ability to control the pulse rise and fall times to some extent, so perhaps common practice will change.

Why use shaped pulses ? Anyone who has tried high-gradient work has probably noted the ticking sound upon applying gradient pulses, and nervously wondered whether the winding and probe

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Electronic mail:
peter@physchem.kth.se

will withstand the strain. Needless to say, mechanical vibrations are also a highly undesirable phenomenon in attempts to monitor molecular displacement over small distances. Eddy currents in the probe housing and magnet will also be very much lessened by reducing the gradient rise and fall times. For pulse shapes like the trapezoidal one above, we find that a risetime of 0.1 ms or so reduces 'ticking sounds' to almost nothing, for a 15 A millisecond gradient pulse. Also the quality of the measurements is significantly improved, as a combined result of reduced vibrations and eddy currents. One should note that the echo attenuation effect is *not* the same for pulses of the same area that differ in shape (see the Figure above). The modified Stejskal-Tanner equation given was derived using the symbolic algebra package Maple V for Windows.

Yours Sincerely

Kim Paulsen Peter Stilbs

PS: **Equipment sought:** We are looking for spare boards and plug-in units for JEOL FX-60, FX-90Q and FX-100. In particular, we are interested in external lock units (probe plug-in and oscillators), computer boards and probes. I also have an old Varian E3 EPR spectrometer, with a dead klystron high voltage power supply. If anyone has any supplies of the mentioned items that they want to get rid of, please contact me.

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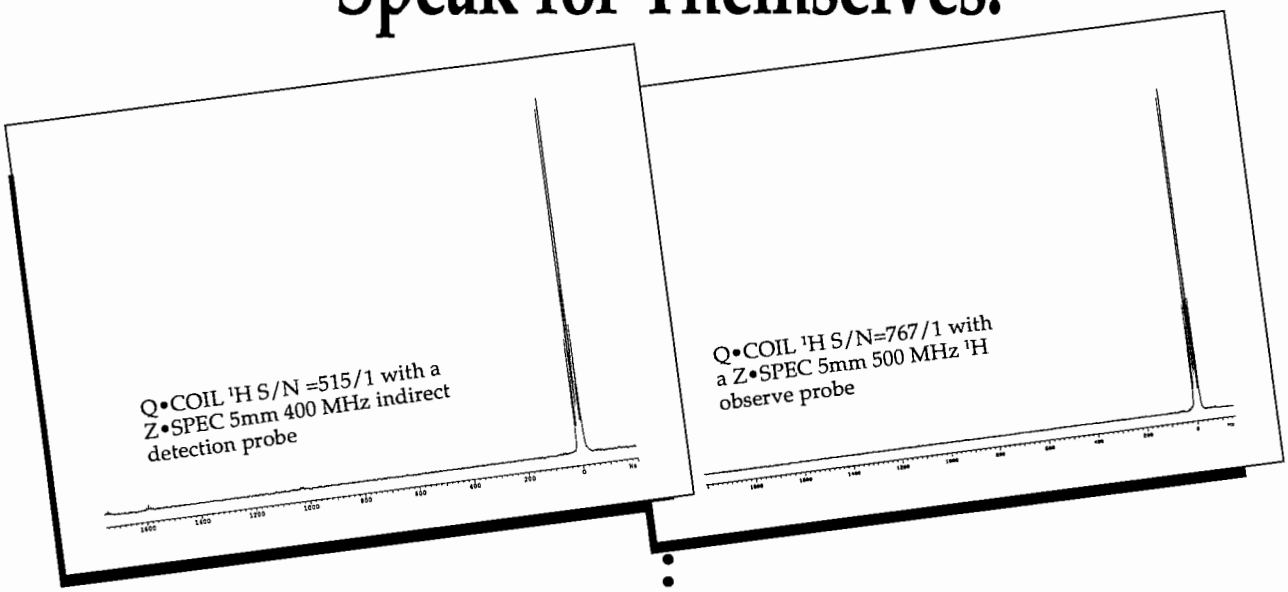

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Figure 1: ^1H line shape determination on 1% $CHCl_3$ for Z•SPEC H 500-5 probe at 500 MHz. Resolution = 0.24 Hz.

Figure 1

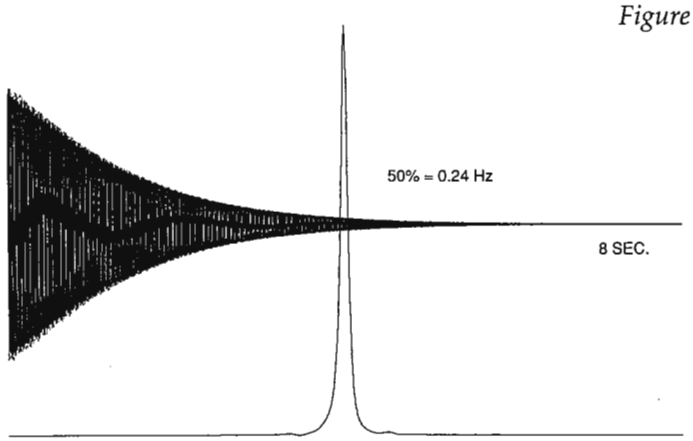


Figure 2

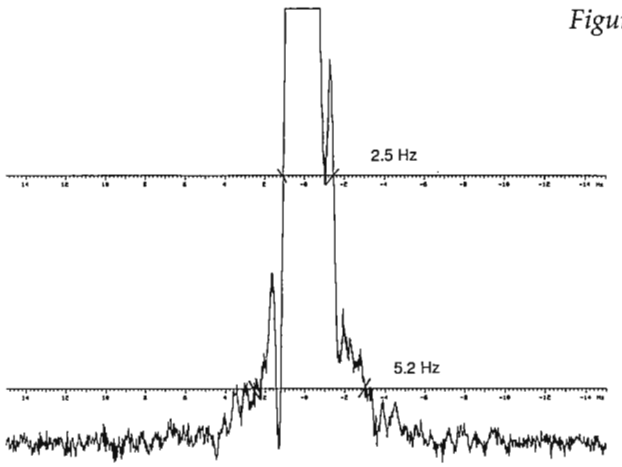
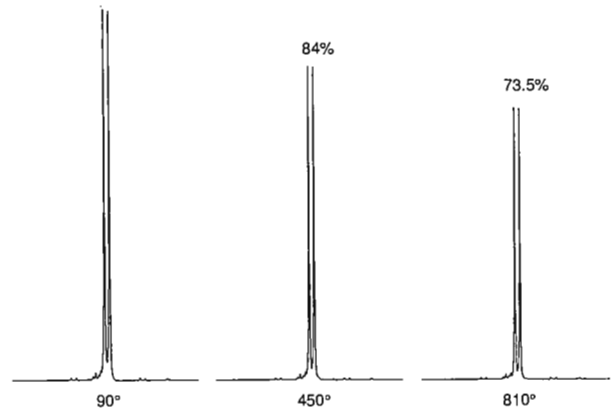


Figure 2: ^1H line shape determination on 1% $CHCl_3$ for Z•SPEC H 500-5 probe at 500 MHz, 2.5 Hz at 0.55% and 5.2 Hz at 0.11%.

Figure 3: ^1H RF homogeneity determination for Z•SPEC ID 500-5 probe at 500 MHz, 84% at 450° and 73.5% at 810°.

Figure 3



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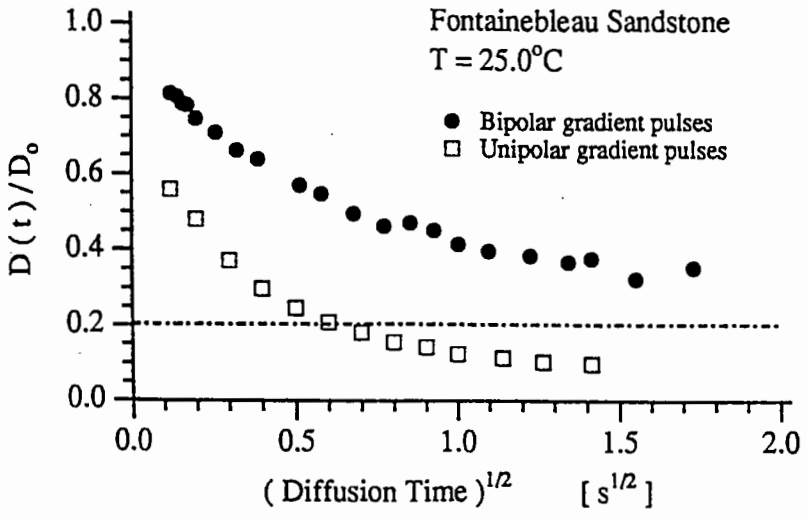
November 10, 1993 (received 11/13/93)

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

Re: The Importance of Internal Field Gradients in Porous Media

Dear Dr. Shapiro,

The theoretical study of internal magnetic field gradients has a long history in NMR (1) and experimental work has recently been done on both biological and non-biological systems (2). Pulse sequences have been developed (3) to negate the effects of these gradients that arise from the difference in magnetic susceptibility in differing regions of the sample. In rock, for example, the gradients arise from the susceptibility difference between the rock grain and the water-filled pore space. These gradients can range, in rock, from a few to hundreds of Gauss/cm. Below we present data on Fountainebleau 2, a clean sandstone with grain sizes of approximately 200μm, as an example of the importance of correcting for these gradients. We plot the normalized, time-dependent diffusion coefficient, D(t),versus the square root of the diffusion time, $\sqrt{t_d}$. D(t) is normalized to the free-diffusion coefficient, D_0, of the brine that is saturating the pore space of the rock. There are data from two pulse sequences depicted here. The pulse sequence used to generate the solid circles is that of Latour *et al.*, which uses alternating, signed, gradient pulses to cancel the effect of the internal gradients. The data denoted by the open squares is generated with a normal Stejskal-Tanner (S-T) pulse field gradient sequence.



At long diffusion times the spins in the saturating fluid sample many pores and therefore give a measure of the tortuosity of the rock. Tortuosity, a quantity that characterizes the "tortured" path of the spin as it wends its way through the pore space, has been independently measured for this rock and is shown by the dashed line. Thus, as t_d is increased, $D(t)/D_o$ should approach the measured value of tortuosity. In the case of the S-T data there is a rapid decrease in $D(t)/D_o$ until it falls below the tortuosity at a $\sqrt{t_d}$ of approximately 0.6 $\sqrt{s}$. The data collected with the sequence of Latour *et al.*, on the other hand, shows the correct behavior. This example nicely illustrates the importance of correcting for internal gradients as Fountainebleau 2 is known to be a clean rock, *i.e.*, one with relatively small internal gradients.

1. J.E. Tanner and E.O. Stejskal, J. Chem. Phys. 49, 1768 (1968).
2. J. Zhong, R. Kennan, and J.C. Gore, JMR 95, 267 (1991); L.L. Latour, P. P. Mitra, R.L. Kleinberg, and C.H. Sotak, JMR A 101, 342 (1993).
3. L. L. Latour, L. Li, and C. Sotak, JMR B 101, 72 (1993) and references therein.

Sincerely,

Karl G. Helmer Martin Hürlimann Larry L. Latour Chris Sotak

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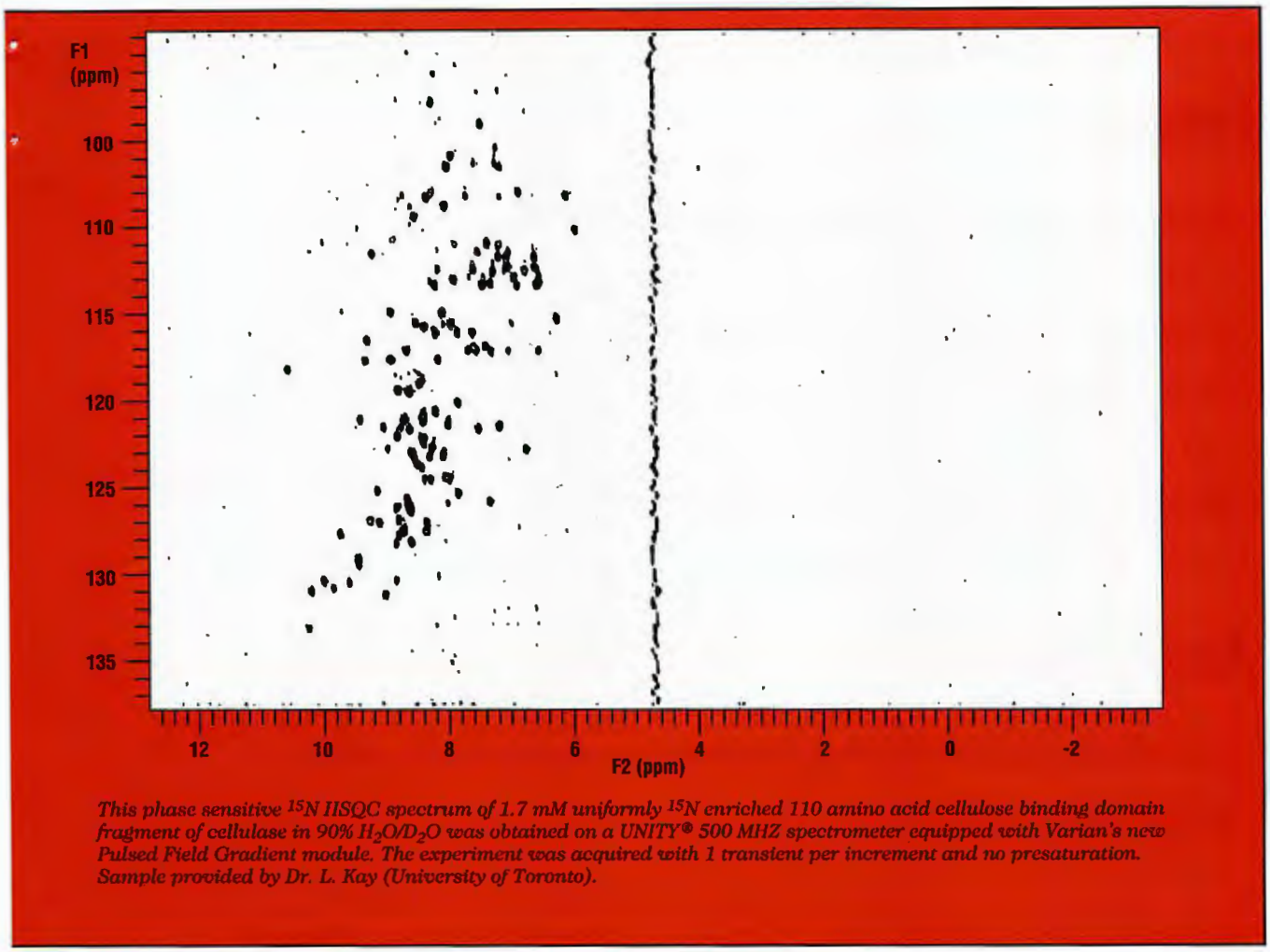

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November 4, 1993
(received 11/8/93)

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

Motional narrowing of second order shift

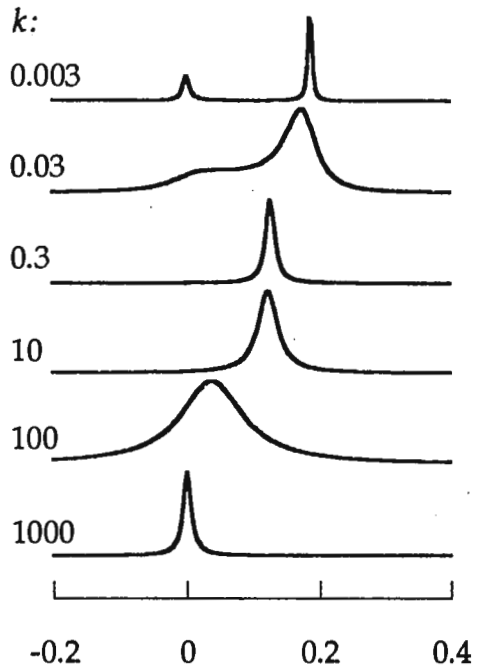
Dear Barry:

The central 1/2 ↔ -1/2 transition of semi-integral quadrupolar nuclei has peculiar properties. While in many respects it can simply be treated as a pseudo-two-level spin system, its behavior often deviates from that expected of a plain spin 1/2, reminding us that the two states $m = 1/2$ and -1/2 are not completely isolated from the rest of the spin system. This is, for instance, demonstrated by the fact that the frequency shift of the central-transition due to the quadrupole interaction arises from matrix elements of the quadrupolar Hamiltonian that fall outside the 1/2 ↔ -1/2 manifold and therefore perturb the Zeeman energies to second order only. Thus the shift is of the order of ω_Q^2/ω_L, where ω_Q is the quadrupole frequency and ω_L is the Larmor frequency.

This letter deals with the question of what happens to this shift when the quadrupole interaction becomes time dependent, either by random molecular motion or by MAS, DAS, or DOR sample spinning. To determine the average shift under fast motion we must choose between two approaches: On the one hand we know that in the case of rapid isotropic motion in liquids every quadrupolar matrix element is averaged to zero, leaving nothing to induce a shift in either the central or the non-central transitions. We also know from the way that MAS and DOR work that the spectra are just determined by the time average of the second-order shift of the central transition only. In other words, in one approach we average the time-dependent matrix elements before we square them to calculate the shift, while in the other we average the time-dependent shift calculated from te squares of instantaneous off-diagonal matrix elements. Since the square of the average differs from the average of the square, the outcomes are not the same. To be sure, the answer to this dilemma is found in the literature. For instance, Baram et al.[1] calculated lineshapes for isotropic motion, and Goldman et al. [2] provided the rationale for the theory of sample-spinning line shapes. But wanting to get hands-on experience, I decided to

[1] A. Baram, Z. Luz, and S. Alexander, J. Chem. Phys. **58**, 4558 (1973).

[2] M. Goldman, P. J. Grandinetti, A. Llor, Z. Olejniczak, J. R. Sachleben, and J. W. Zwanziger J. Chem. Phys. **97**, 8947 (1992).



do my own calculation on a simple model that would bring out the essential points.The model is a spin 3/2 subjected to a Zeeman field along z and an axially symmetric field gradient whose principal axis spends equal time along the x, y, and z axes in a random jump motion. The motion is akin to isotropic, because it averages every matrix element of the quadrupole Hamiltonian to zero. The Larmor frequency is ω_L = 100 and the quadrupole frequency is ω_Q = 10, both in arbitrary angular frequency units. The rate of jumps from one site to either other site is denoted by k, corresponding to a correlation time $\tau_c = 1/3k$. The figure shows central-transition lineshapes calculated by step-wise integration of the full time-dependent density matrix equation in the rotating frame, followed by FT. In very slow motion, we see the peak for the z spins at zero shift and the overlapping peaks for x and y at $-(3/16)\omega_Q^2/\omega_L$. When k increases to values larger than the shift difference, the peaks merge into a peak at the average shift like any spin 1/2 would do. But when k increases further beyond the Larmor frequency, the peak moves to the zero-shift position, where it should be according to the vanishing average Hamiltonian. In the intermediate range, where $k \approx \omega_L$, the line broadens.

Without reference to philosophical arguments concerning the deeper meanings of motional narrowing, we can read the solution to our averaging dilemma directly from the figure: When $k << \omega_L$ the second-order shifts are averaged and when $k >> \omega_L$ the shift is determined by the average Hamiltonian. MAS, DAS, and DOR lineshapes are what they are because the sample spinning rates are always smaller than the Larmor frequency. One could contemplate the effects of MAS at rates higher than ω_L and hope that it would completely narrow the line, rendering DAS and DOR obsolete. This expectation will, however, not be met, since the off-diagonal elements do not average to zero under MAS and thus continue to broaden the line.

The lineshapes in the figure demonstrate another important aspect of central-transition dynamics. The exchange process does not broaden the spectrum far outside the boundaries of the static spectrum. Some of us may have expected that to happen. After all, the motion mixes various parts of the Hamiltionan and could perhaps impose satellite-like broadening on the central transition. It turns out that exchange between closely spaced NMR-observable lines will never cause the second-order spectrum to broaden beyond detection.

Please consider this a contribution to P. L. Watson's account.

Best wishes,

Alex

Alexander J. Vega

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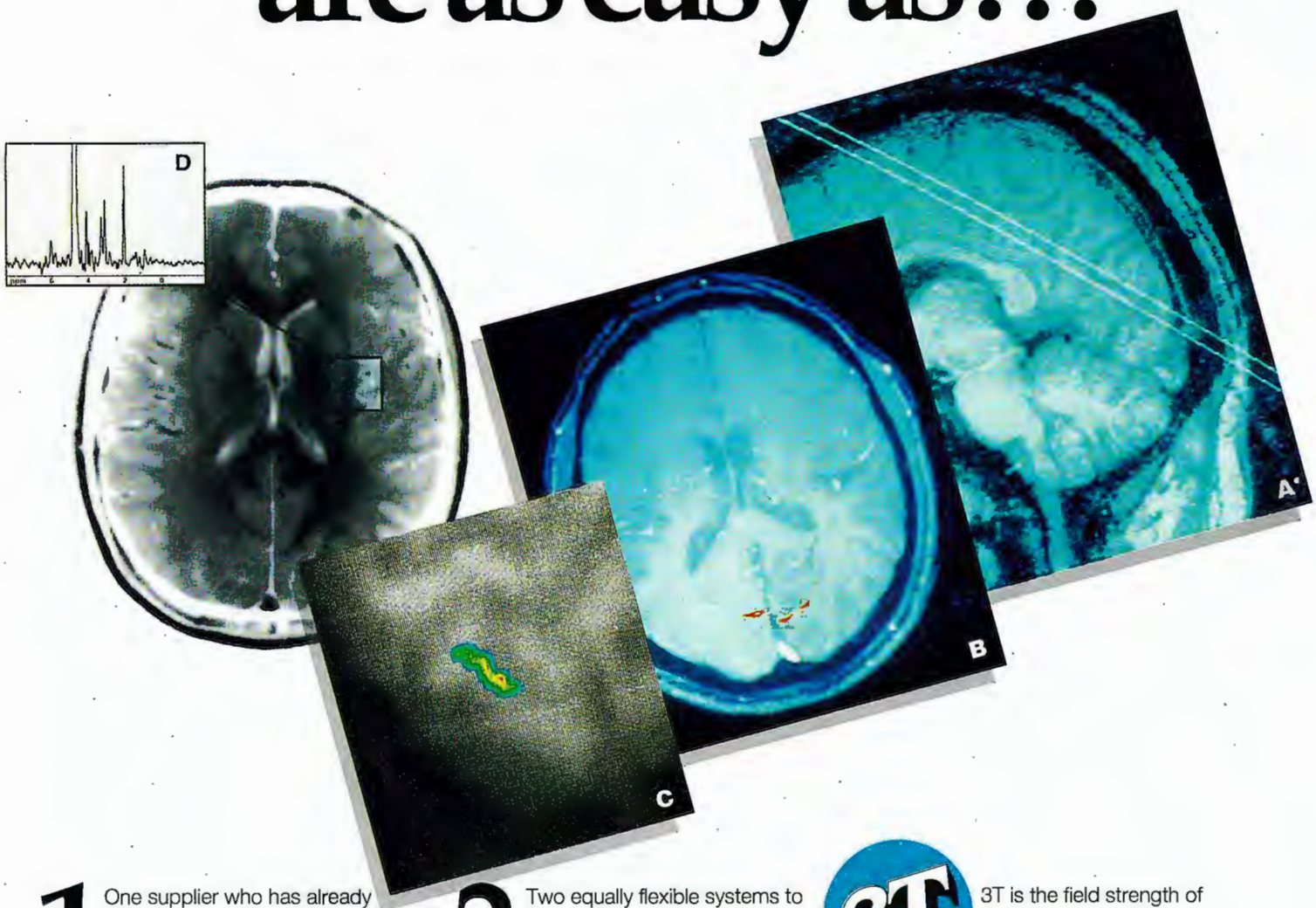

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C: Functional correlation image of stimulation of the motor cortex (EPI, 64 x 64, TR = 1s, TE = 25 ms, slice = 10 mm) Courtesy of Biophysics Res. Inst., Med. Coll. of Wisconsin, Milwaukee, Dr. J. S. Hyde
D: Localized proton spectroscopy: Spin-echo technique. Bruker Medizintechnik, Applications Laboratory

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TAMU NMR Newsletter - Book Reviews

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

"*In Vivo* Magnetic Resonance Spectroscopy I: Probeheads and Radiofrequency Pulses, Spectrum Analysis"

"*In Vivo* Magnetic Resonance Spectroscopy II: Localization and Spectral Editing"

"*In Vivo* Magnetic Resonance Spectroscopy III: *In Vivo* MR Spectroscopy: Potential and Limitations"

Edited by **M. Rudin**

NMR Basic Principles and Progress, (P. Diehl, E. Fluck, H. Günther, R. Kosfeld and J. Seelig, eds.), 1992; Springer-Verlag New York, Vol. 26 - ISBN 0-387-54547-6, 284 pp.; Vol. 27 - ISBN 0-387-55022-4, 296 pp.; Vol. 28 - ISBN 0-387-55029-1, 190 pp.

This three-volume set summarizes the current state of the art of *in vivo* spectroscopy in a series of articles by acknowledged experts on each aspect of the field. Edited by M. Rudin, the first volume is devoted to the design of probeheads and rf pulses, the second to localization and spectral editing and the third to the potential and limitations of the method.

In a collection of this kind, it is not surprising to find a great deal of heterogeneity both in the quality of presentation and scope of coverage of each topic. This brief review cannot do justice to every article. Its focus will be on the major unsolved problems of *in vivo* spectroscopy, noting that van Zijl and Moonen spoke for many authors in the opening sentence of their chapter on solvent suppression (v. I, p. 68): "Despite its obvious potential for assessing *in vivo* metabolism, NMR spectroscopy has yet to acquire a significant place in clinical practice." The three reasons they give - low sensitivity, low spatial resolution and low specificity due to tissue heterogeneity - are certainly among the main reasons, though there are some others.

Not all of the authors, especially those writing on engineering subjects, are adequately aware of these reasons. The field in these three volumes, as in real life, appears strictly compartmentalized among experts - rf experts, coil experts, data analysis experts, computer experts, pulse sequence experts - each seemingly convinced that if he solves the problems he can identify with his expertise, all problems of *in vivo* NMR will be solved. Not a single article in this collection looks at the field in its entire perspective and asks the basic question: Why is it that after 20 years of intensive effort by scores of - largely - very talented physicists and engineers, so little new or at least clinically useful information has come from *in vivo* NMR? This to my mind is the greatest weakness of the series. Volume III purports by its title to discuss the potentials and limitations of the method, but it does not. Instead it presents a series of expert reviews on a few specific applications.

Most progress in *in vivo* NMR has been in the development of techniques for just getting better spectra on living specimens - constructing better coils, implementing better pulse

sequences, better data analysis. It is not surprising, therefore, that the best, most rigorously reasoned articles are those discussing the principles of such techniques. The articles in Volume I describing these principles - be it probe design in the articles by Link and Schnall, frequency selective excitation by Morris, or time domain fitting procedures by de Beer and van Ormondt - are of uniformly high quality, except for one feature: the absence of significant biological results. The techniques are a prerequisite for doing meaningful *in vivo* spectroscopy, but they are also quite generally applicable. The first volume could have been an excellent general text on NMR techniques. It is spectroscopy saying little about the *in vivo* part.

A word of caution has to be said about the article by Cady on the "Determination of Absolute Concentrations of Metabolites," which discusses standardization of signal intensities and ends with the claim that MRS techniques can provide absolute metabolic concentrations. The claim is false. One can get an estimate of the amount of a metabolite in a gram of tissue, but to calculate a true concentration, as needed by the biochemist or the clinician, one would have to know the size of the compartment in which it is distributed. We have no way of measuring that in a heterogeneous tissue. The problem of measuring *concentrations* by MRS remain unsolved. It cannot be solved by any procedure discussed in these volumes and is one of the reasons for the limited usefulness of MRS in either biological research or in clinical practice.

Volume II is devoted in its entirety to one of the most active areas of *in vivo* research - localization and spectral editing - though again one finds here mostly the basic physics and engineering of NMR and little interesting *in vivo* information. The editor's opening statement, "A crucial step in *in vivo* NMR is the proper definition of the region-of-interest (ROI)," is true, but incomplete, because of what just has been said about measurement of concentration. There are good reviews of most of the currently used localization techniques - from surface coils by Bosch and their inventor, J. J. Ackerman, to spectroscopic imaging by P. Styles and Hollander, Luyten and Marien. Regrettable is the absence of one of the leading experts on such techniques, J. Frahm, from the list of authors.

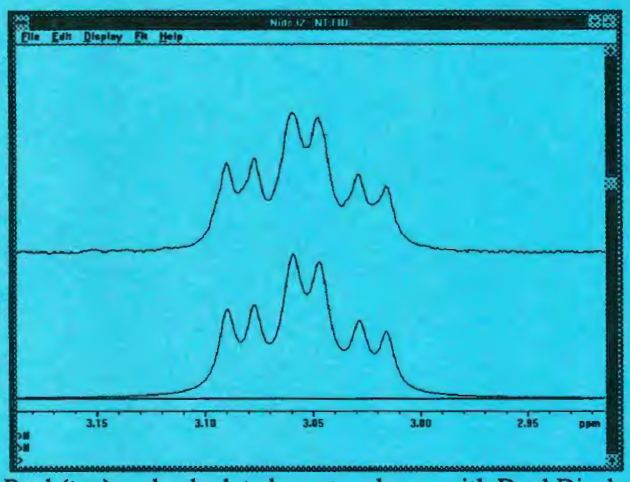
Oddly irritating is the high sales pitch tone of the article by P. Bottomley on DRESS. It reminds us that this tone, used by the early proponents of *in vivo* NMR, grossly oversold the potential of the method in medicine. As a result, we are now suffering from a backlash of skepticism and even rancor from serious biologists and clinicians for making false claims. It does not help the understanding of the subject to pepper articles on what is essentially physics with phrases like "highly relevant to ischemic disease" or "may ultimately be of significant value to patient management" when neither has been demonstrated. Unfortunately, such phrases abound throughout the volumes.

The articles on spectral editing and 2D methods describe standard universally applicable NMR methods, saying more what the methods *should* do than what they *have* done for *in vivo* spectroscopy. This reviewer, with some experience in these methods, fears that their even lower sensitivity compared to 1-dimensional spectroscopy will severely limit their usefulness *in vivo*. One wonders what the article by Rudin and Sauter is doing in this Volume, but it aptly summarizes the kinetic equations useful in studying enzyme kinetics *in vivo*.

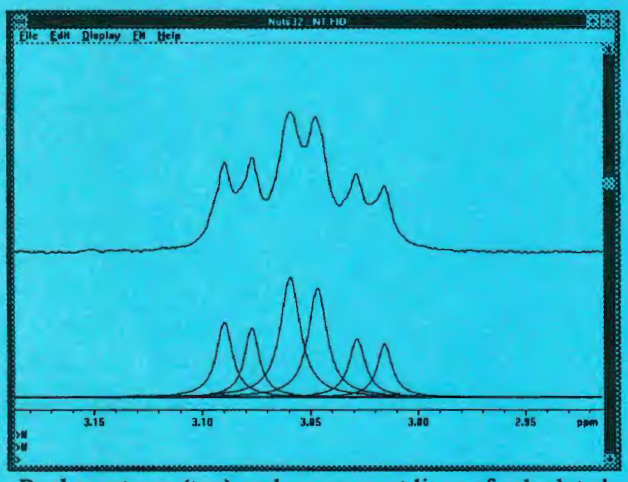
Of Volumes I and II it can be said: what is good about them is the physics, and what is *in vivo* is either absent or weak.

-continued on p. 41

Multiple Line Deconvolution with NUTS!



Real (top) and calculated spectra shown with Dual Display



Real spectrum (top) and component lines of calculated spectrum shown using Dual Display

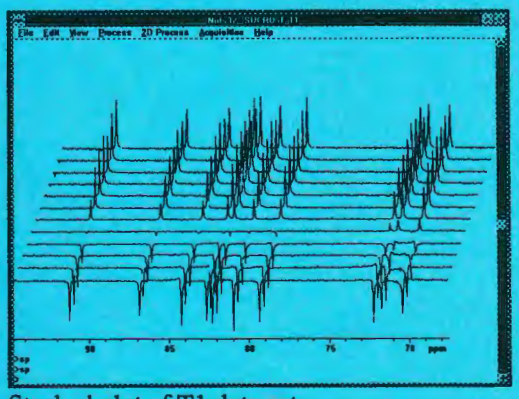
NUTS Line Fit features:

- Simplex fit on a set of Lorentzian lines
- No limit on the number of component lines
- Easy "point-and-click" input and adjustment of component lines using mouse
- Input starting set of lines with single command using NUTS peak pick function
- Display individual lines, sum or difference
- Multiple options for displaying real and calculated spectra
- Save calculated spectrum and/or "template" for repeated matching to series of spectra
- Printout of all parameters for calculated spectrum

Deconvolution module included in both 1D and 2D versions of NUTS

New NUTS Features:

- Stacked plots of 2D data or series of 1D spectra
- Macros to perform operations such as 2D processing with a single command
- "Smart" file import command auto-detects data format and reads data directly into NUTS
- 32-Bit operation for increased speed


Stacked plot of T1 data set

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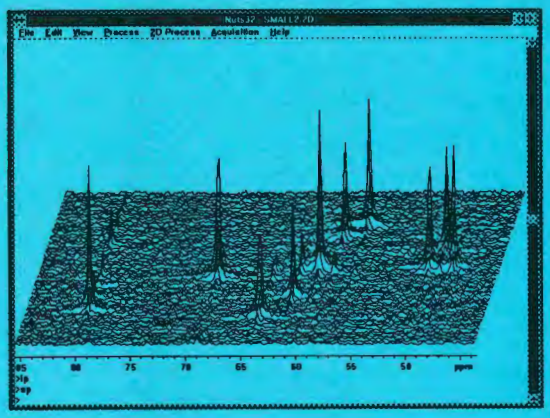


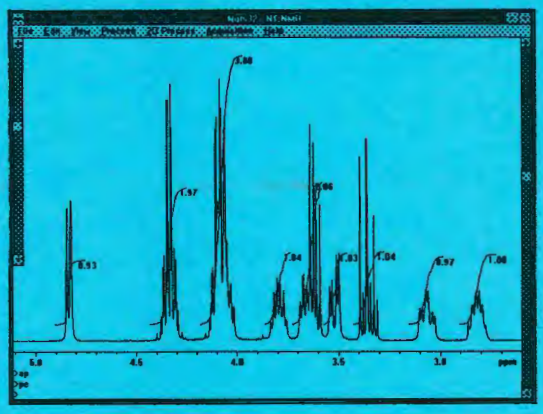
Stacked plot of 2D HETCOR spectrum

NUTS Features

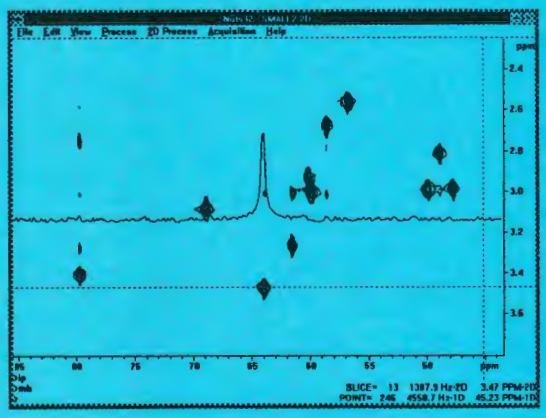
- 16K complex FT in 1.5 sec on 486DX2/50 PC
- Automatic and real-time phasing
- Spectrum addition / subtraction
- Dual display of 2 spectra
- Multiple baseline correction options
- Integrals labelled on the screen and plots
- Intelligent peak-picking
- Acquisition parameters translated & printed on plots
- Configuration file lets you customize NUTS for your preferences

Easy to learn and use Windows format

- **On-line Help** saves searching through manuals
- Menus and command line interface active at all times for maximum flexibility
- Command sequences automated with customizable quick keys
- ****New**** Macros automate complex operations
- Copy spectra and paste into reports



Integration with relative values displayed on screen



Real-time display of slices under mouse control

Data Translation from most spectrometer models

- **Bruker** Aspect 3000 data files
- **Varian** XL, Gemini and older VXR files
- **Nicolet** Dexter and TMON files
- **GE** Omega and TMON files
- **JEOL** *.GXD/*.GXP data files
- **ATI** ASCII and binary files

Other data file translations will be added on request if file format information can be supplied.

NUTS 1D version 3.0 for IBM PC (math coprocessor required) on 3.5" HD floppy .....$ 499.00
NUTS 2D version 2.0 for IBM PC (math coprocessor required) on 3.5" HD floppy .....$ 750.00

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Volume III is a series of articles on the application of specific nuclei: ^{19}F, ^{13}C, ^{1}H. There is a good survey of NMR studies of perfused cells and organs by Kaplan, van Zijl and Cohen, but then such studies are *in vivo* in a limited sense. The biochemistry of such preparations can be studied by many less expensive techniques. The main attraction of *in vivo* spectroscopy in medicine is its non-invasiveness - the ability to observe metabolism without disturbing the intact organism. Its status on this front is honestly summarized in the last article of this volume by Rudin (the series editor) and Sauter: "The value of ^{31}P NMR...for diagnosis and better understanding of human disorders remains to be demonstrated." This statement justifies the title of the Volume, but those seeking more detailed insight on the potential and limitations of *in vivo* NMR in this volume will seek it in vain.

In summary, if one approaches a specific article in this series with the question: How do I do a particular thing? - one may find useful information. If one approaches it with the question: What can *in vivo* MRS do for me? - or less selfishly: What has *in vivo* MRS done for humanity? - one will find next to nothing. The Editor is to be commended for the honesty of his own writing and for presenting an honest collage of snapshots of the field. He can hardly be blamed that the collage reflects an abundance of technical ingenuity coupled with somewhat tubular vision, good physics coupled with mostly empty biological phrases, and an inability of the field to solve its fundamental problems. If the overall impression about its future one gets from these volumes is pessimistic, one is reminded of the first book on the subject by D. Gadian, severely criticized for its pessimism at the time. Although shouted down by more vociferous enthusiasts, Gadian is being proven right. One cannot define a field by what a technique *might* do for it, but only by what it *has* done.

Oleg Jardetzky
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Supervisor, NMR Spectroscopy Facility.(anticipated). The Department of Chemistry/Center for Photochemical Sciences at Bowling Green State University seeks to fill a full-time staff position in modern high-field NMR spectroscopy starting July 1, 1994. The successful candidate should be experienced in the techniques of modern multidimensional, and multinuclear NMR spectroscopy. Responsibilities will include supervising the daily operation and maintenance of the Department's new 400 MHz Varian Unity*plus* and 200 MHz Gemini systems. Responsibilities will also include user training and supervision. An advanced degree in Chemistry is required. Send CV and 2 letters of recommendation by February 15: NMR Search Committee, Department of Chemistry, Bowling Green State University, Bowling Green OH, 43403. Bowling Green is an equal opportunity/affirmative action employer.

Postdoctoral Opportunities in Biomedical Materials

We anticipate two postdoctoral openings in the Biomedical Materials Laboratory of the MGH NMR Center. The first, with an expected starting date around April, 1994, is for an NIH-funded project involving *in vivo* solid state NMR of bone mineral. The second, starting **July,** 1994, and funded by NIH and other sources, involves the study of silicone-gel implants, including animal and human subjects. Postdoctoral fellows will have Harvard Medical School academic appointments. The MGH NMR Center encompasses over 50 researchers, with backgrounds in chemistry, physics, biology, medicine and engineering, including graduate students from Harvard and MIT. It is the focus of many cutting edge developments in clinical and basic science research. Please reply to Prof. Leo Garrido, 617-726-5822, for the silicone project, and Prof. Jerry Ackerman, 617-726-3083, for the bone project. Mailing address: NMR Center, Room 2301, Massachusetts General Hospital, 149 13th Street, Charlestown, MA 02129. Fax: 617-726-7422.

All Newsletter correspondence should be addressed to

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

(415) 493-5971 - *Please call only between 8:00 am and 10:00 pm, Pacific Coast time.*

Fax: *Not for submitting technical contributions:* (415) 493-1348.

Deadline Dates

No. 425 (February)	21 January 1994
No. 426 (March)	18 February 1994
No. 427 (April)	25 March 1994
No. 428 (May)	22 April 1994

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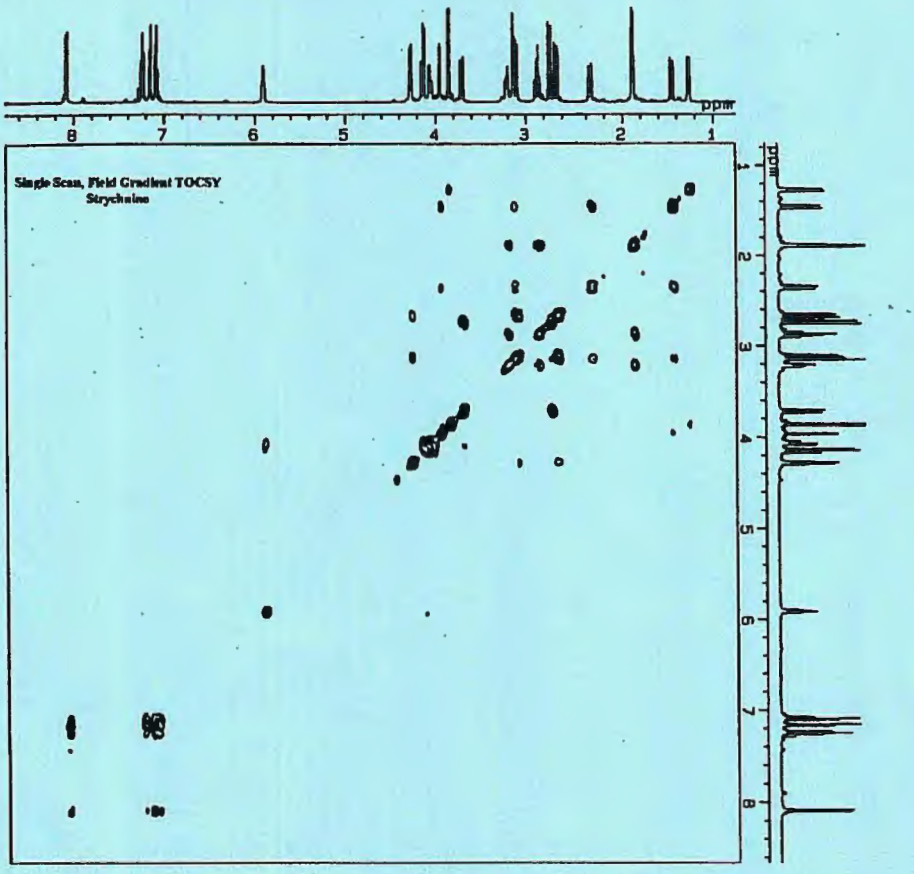
If the mailing label on your envelope of this issue is adorned with a large **red dot** or circle: this decoration means that you will not be mailed any more issues until a technical contribution has been received by me.

NMR

FROM JEOL USA

Gradient Probes



The above data is a 512 by 512 *TOCSY* spectrum and was obtained on an **Alpha**- 500 in less than 5 minutes with **JEOL**'s gradient probe. This same probe is also designed to do reverse and water suppression experiments. Pulse shaping, three channel operation, dynamic Rf control, matrix shims, as well as unsurpassed field reliability are all standard with the **Alpha**.

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