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TABLE 1 DEUTERATED SOLVENTS

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D-14	Chloroform-d	CDCl ₃	99.8%	1.50	-64	62	0.740 (20)
D-21							0.611
D-122							
D-130							
D-28	Chloroform-d + 1% TMS	CDCl ₃	99.8%				
D-31							

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FORTHCOMING NMR MEETINGS

International Symposium on Solid-State NMR Spectroscopy of Polymers, Keystone, Colorado, **February 28 - March 3, 1993**; Contact: Barbara D. Hogan, Du Pont, CR&D, P. O. Box 80356, Wilmington, Del. 19880-0356; Phone: (302) 695-4394; FAX: (302) 695-8207.

PITTCON'93, Atlanta, Georgia, **March 8-12, 1993**; Contact: Dept. 60, Suite 332, Penn Center Blvd., Pittsburgh, PA 15235-5503; Phone: (412) 825-3220, Toll-free: 1-800-825-3221; Fax: (412) 825-3224.

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

In Vivo Magnetic Resonances Spectroscopy VI. Symposium on In Vivo Magnetic Resonances Spectroscopy: Focus on Spectroscopic Techniques, St. Louis, Missouri, **March 13-14, 1993**; Contact: Radiology Postgraduate Education, Room C-324, University of California School of Medicine, San Francisco, CA 94143-0628; Phone: (415) 476-5808; Fax: (415) 476-9213.

34th ENC (Experimental NMR Conference), St. Louis, Missouri, **March 14-18, 1993**; Contact: ENC, 815 Don Gaspar, Santa Fe, New Mexico 87501; Phone: (505) 989-4573; Fax: (505) 989-5073. See TAMU NMR Newsletter 408, 45.

Advanced School on Magnetic Resonance and Protein Dynamics, Erice, Sicily, Italy, **March 15-21, 1993**; Contact: Prof. Oleg Jardetzky or Stanford Magnetic Resonance Lab., Stanford Univ., Stanford, CA 94305-5055; Fax: (415) 723-2253; See TAMU NMR Newsletter 411, 50.

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

Gordon Research Conference: Magnetic Resonance, Wolfeboro, NH, **July 11 - 16, 1993**; Contact: Dr. A. M. Cruickshank, Gordon Research Center, University of Rhode Island, Kingston, RI, 02881-0801; (401) 783-4011 or -3372; FAX: (401) 783-7644.

35th Rocky Mountain Conference on Analytical Chemistry, Denver, Colorado: **July 25-29, 1993**; Contact: Patricia L. Sulik, RML, Inc., 456 S. Link Ln., Ft. Collins, CO 80524; Phone: (303) 530-1169.

12th Annual Scientific Meeting and Exhibition of the Society of Magnetic Resonance in Medicine, New York, NY, **August 14-20, 1993**; Contact: SMRM, 1918 University Ave., Suite 3C, Berkeley, CA 94704; Phone: (510) 841-1899; FAX: (510) 841-2340.

1993 FACSS Meeting, Detroit, Michigan, **October 17-22, 1993**; Contact: H. N. Cheng, Hercules, Inc., Research Center, 500 Hercules Road, Wilmington, DE 19808; Phone: (302) 995-3505; Fax: (302) 995-4117. See TAMU NMR Newsletter 411, 10.

Additional listings of meetings, etc., are invited.

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December 7, 1992
 (received 12/11/92)

Professor Bernard Shapiro
 Editor/Publisher
 TAMU NMR Newsletter
 966 Elsinore Court
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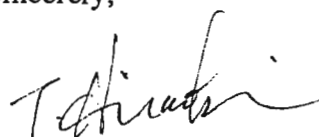
"Collective motions along the long side chain on polypeptide"

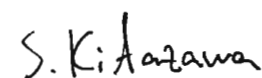
Dear Professor Shapiro,

As usual it takes your reminder notice to prompt us into submission. In our continuing studies of the side-chain dynamics of polypeptides, we have been finding the collective motions along the long side chain on polypeptides in the solid state.

The main chain of poly(γ -benzyl L-glutamate)(PBLG) forms an α -helical conformation, and this rod-like molecule packs into hexagonal in the crystalline state. On the other hand, the structure of the side chain of PBLG has not been clarified yet, and the glass-like transition is observed at above the room-temperature. We have measured T_1 and line-shape of the solid state ^2H -NMR(30.7 MHz) for three PBLGs deuterated in γ , ζ , and κ positions, respectively, in the side chain. T_1 values obtained are shown in Figure 1, with the side chain structure of PBLG. The values show almost same temperature dependence for three samples, indicating same correlation times. This finding implies that same molecular motions exist in the different positions along the side chain, for the deuteron T_1 is mainly dominated by the quadrupole interaction. The Pake powder pattern is destroyed out to a singlet, due to the fast and larger-amplitude reorientations in this temperature range.

Sincerely,


 Toshifumi Hiraoki


 So Kitazawa

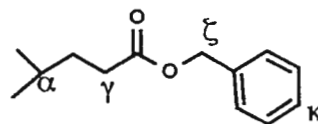
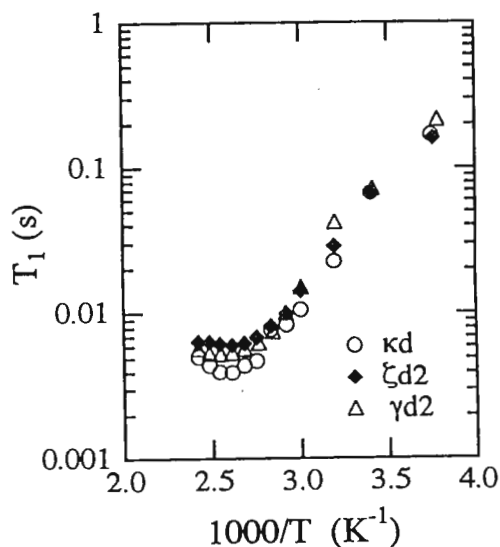


Figure 1.



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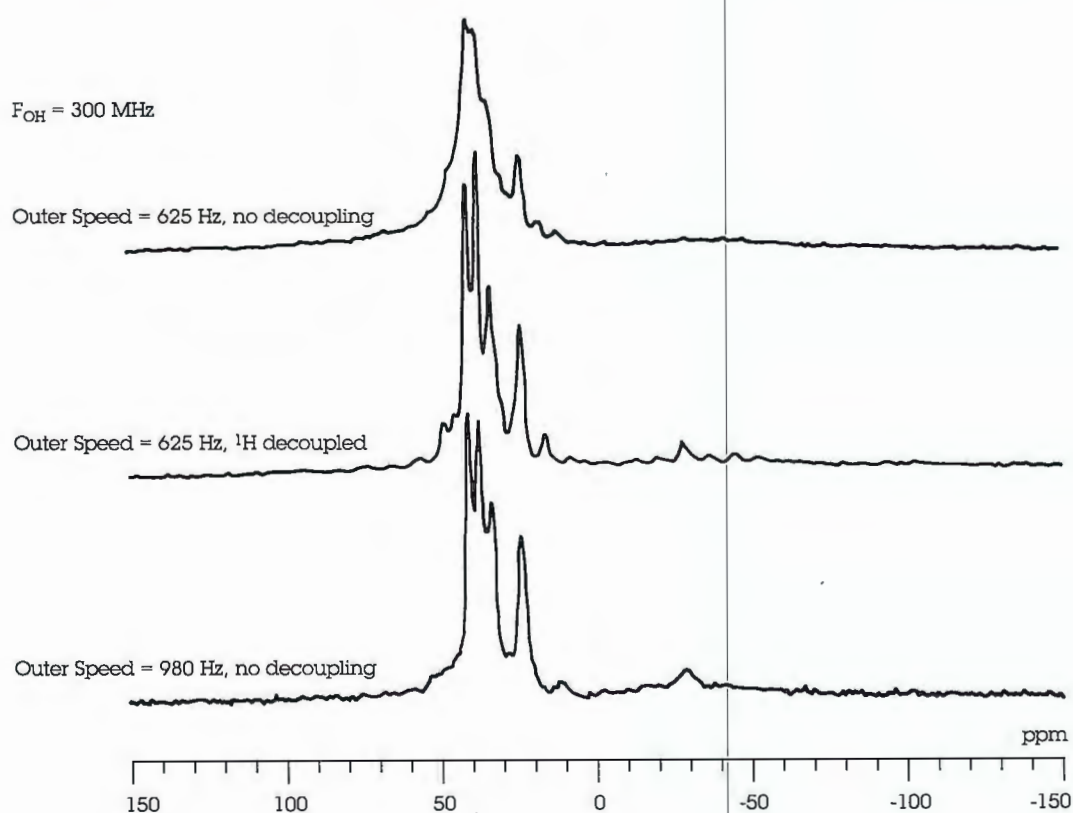
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December 8, 1992

(received 12/12/92)

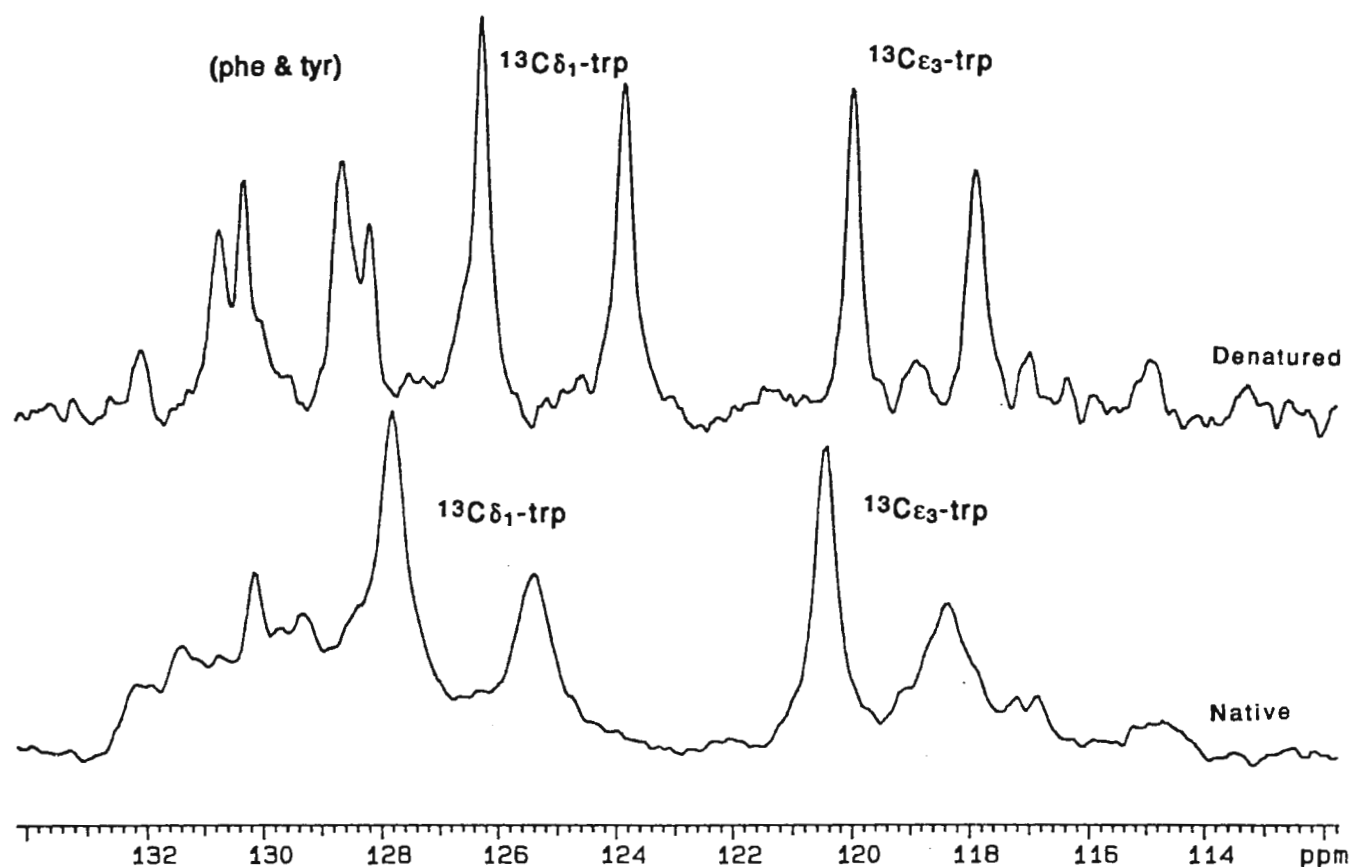
Dr. B. L. Shapiro
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto, CA 94303

^{13}C Differential Broadening of Thioredoxin

Dear Barry,

For some time we have been using ^{13}C NMR relaxation measurements to study internal motions in ^{13}C -labeled peptides and proteins in liquid solution. Normally we measure T_1 , T_2 , and the steady state NOE of ^{13}C nuclei that have attached protons. The relaxation rates are subsequently interpreted in terms of overall rotational motion and internal motion of the molecule. It is also true that useful dynamical information can be obtained from proton-coupled ^{13}C spectral lines. Consider, for example, the case of a ^{13}C nucleus with a single attached proton. Because of interference between the chemical shift anisotropy and the dipolar interactions, the individual components of the ^{13}C doublet generally will have different widths provided the CSA of the particular ^{13}C is sufficiently large. In fairly big molecules such as proteins, the more restricted the internal motion at the site of the ^{13}C , the larger this so-called differential broadening will be. The requirement on the size of the CSA is readily met by aromatic ^{13}C nuclei (for example in tryptophan) at typical frequencies of 75 MHz and above. Examples of differential broadening that we have observed in ^{13}C -tryptophan in thioredoxin (TRX) are described below.

Escherichia coli TRX is a low molecular weight (11,700), almost spherical redox protein. The "active site" of *E. coli* TRX consists of cysteine residues at positions 32 and 35 while TRX has trp residues at positions 28 and 31. X-ray and NMR work have shown that trp-31 lies on the surface of the protein and that trp-28 is partially buried and spatially proximate to the pair of cysteines. We have studied two single trp variants of *E. coli* TRX, W28F and W31F, in which phenylalanine replaces trp-28 and trp-31 respectively by incorporating into those proteins two, different, isotopically labeled trp residues, one with ^{13}C at the δ_1 (or 2) position of the indole ring and one with ^{13}C at the ϵ_3 (or 4) position of the indole ring. From the relaxation measurements we found that the trp side chain motion is quite restricted in W28F and W31F. To see if the motion of the trp rings could be made less restricted, we performed relaxation measurements on urea denatured W28F and W31F. Indeed at 8-9 M urea, the internal motion of the trp side chains became substantially less restricted. As the urea concentration was titrated in W28F and W31F, two ^{13}C NMR signals in slow exchange became apparent for each label, one from the native, folded forms and one from the denatured forms. Differential broadening measurements gave dramatic evidence of the contrast in the native and denatured forms as shown in the figure (next page) for reduced W28F. In the native protein the differential broadening is substantial for both $^{13}\text{C}\delta_1$ and $^{13}\text{C}\epsilon_3$ -trp while there was essentially no differential broadening in the denatured proteins. It is clear that measurements of differential broadening can be quite informative in the study of molecular dynamics.



Proton coupled ¹³C NMR spectrum at 75.4 MHz (referenced to internal dioxane at 67.37 ppm) of reduced W28F in 50 mM, pH 6.5 phosphate buffer (below) and in 8 M urea (above) at 20 ° C.

Please credit this contribution to the account of B. D. Nageswara Rao.

Best regards,

Marvin
Marvin D. Kemple

Peng
Peng Yuan



NMR Instruments

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USA

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Our Ref: DW0625

18 December 1992
(received 12/21/92)

Dear Dr Shapiro,

Progress Towards 750 MHz - Update

Since our last communication (11 November 1991) on our 750 MHz Superconducting Magnet Development Programme we have made excellent progress towards our goal of providing a 750 MHz magnet as the flagship of our product range.

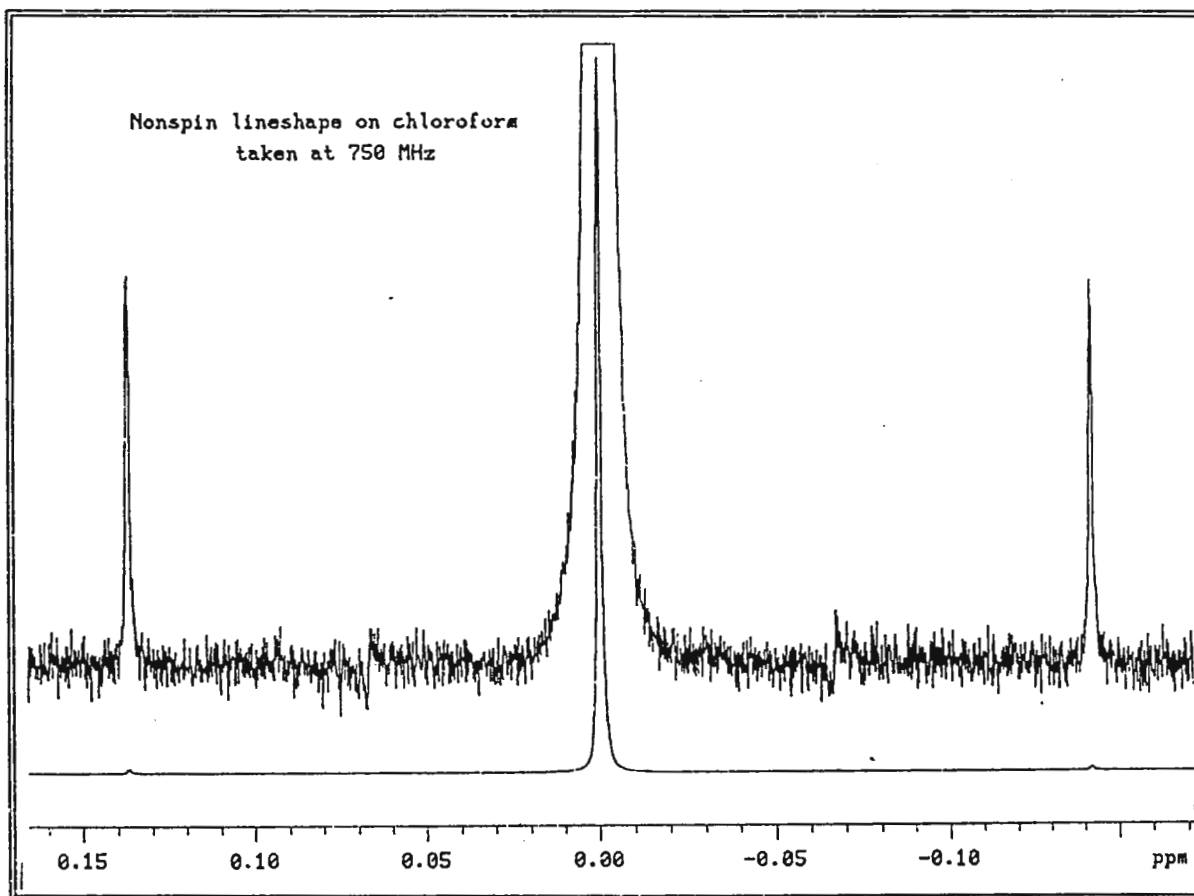
We previously reported results from Phase I of our programme which culminated in a 720 MHz prototype NMR magnet system. This system operates at a temperature of 2.2K and requires relatively complex cryogenic control. Although this was a milestone in high field NMR development we highlighted the problems associated with operating such a system as part of a routine high resolution NMR Instruments, specifically the need for highly skilled operators and the potential for NMR artefacts due to vibrational effects.

To overcome these inherent problems and offer the most user friendly system possible we have developed a 750 MHz magnet that operates at a temperature of 4.2K. Thus operating in the same convenient, well-understood way as all of Oxford's NMR magnets including the 600 MHz system.

We are pleased to announce that the prototype 750 MHz magnet has been successfully energised to field and is presently undergoing trials at Oxford's factory in the UK. Some optimisation may be required but the results obtained so far are extremely encouraging.

Although the magnet drift has not fully settled out, we have seen overnight drift rates as low as 10 proton Hz/hour and fully expect it to reduce further. The homogeneity analysis is encouraging, as can be seen from the nonspun lineshape on chloroform taken at 750 MHz shown overleaf, but some further work needs to be done to optimise the shimming. The operation of the cryostat is as simple as that of a 600 MHz cryostat, and the cryogenic performance is good, offering two months helium refill interval. The next step is to carry out some further integration and optimisation.

Continued overleaf.....



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This development could not have been carried out without the unique contributions of Bill Timms, Paul Noonan, Peter Daniels and their team. Partial financial support from the Department of Industry under the collaborative LINK scheme is acknowledged.

Yours sincerely

Bill Proctor

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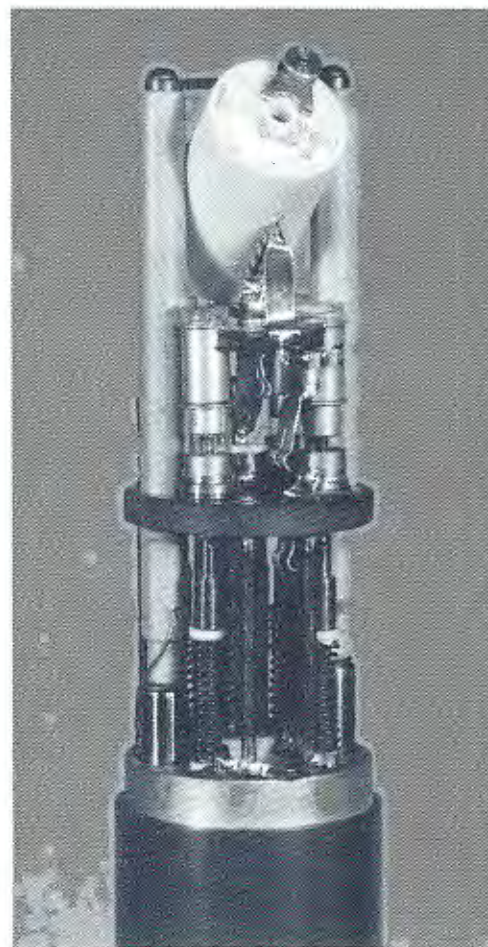
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Measurement of Long-Range ^{13}C - ^1H Coupling Constants in Carbohydrates

Rossitza Gitti and C. Allen Bush

Department of Chemistry and Biochemistry
University of Maryland Baltimore County
Baltimore, Maryland 21228

Valuable conformational information can be derived from measurements of 3-bond heteronuclear coupling constants between ^1H and ^{13}C across the glycosidic linkage in oligosaccharides, (Rosvear et al., 1982). Although the measurements are technically difficult, efforts have continued to develop suitably parameterized trigonometric formulas for correlating the coupling constants with the dihedral angles of the glycosidic linkage, (Tvaroska, 1990). Among a number of new NMR experiments recently proposed for measurement of long-range heteronuclear coupling, one by Montelione et al. (1989) involves generation of 'E-COSY' type cross peak patterns in homonuclear 2-d spectra of samples enriched in ^{15}N or ^{13}C . While most of the applications reported for this method involve ^{15}N , it should also work for ^{13}C enriched carbohydrates. Although selective or partial enrichment might avoid some spectroscopic problems such as ^{13}C - ^{13}C coupling and ^1H line broadening by ^{13}C relaxation, it should be easy to prepare uniform 100% enriched bacterial polysaccharides by growing bacteria in ^{13}C enriched media. Selective or partial enrichment could be difficult for bacteria requiring multiple carbon sources.

Therefore we have applied the method of Montelione et al. (1989) to uniformly highly ^{13}C enriched glucose. In these experiments we expect each homonuclear crosspeak to be split into four components separated in both dimensions by the one-bond ^{13}C - ^1H coupling. A phase-sensitive 2-d ^1H TOCSY spectrum of uniform 98% labeled ^{13}C glucose was recorded on a GN-500 spectrometer with MLEV-17 mixing with trim pulses. The results in Fig. 1 show the cross peaks of the upfield satellites of H2 and H4 of the β -anomer with H1. Fig. 2 shows cross peaks of H1 of the α -anomer with H2 and H3. Values of the coupling constants can be estimated from the contour plots made with high digital resolution. Using Hare's FELIX program to 'zoom' sub-blocks of our spectrum to 0.3 Hz/pt resolution, we have estimated J values to approximately ± 0.4 Hz. Measurements are in reasonable agreement with literature values for the β -anomer of 5.5 Hz for $^2J_{C1-H2}$ (Walker et al, 1976) and for the α -anomer of 5.3 Hz and 1.3 Hz for $^3J_{C3-H1}$ and $^2J_{C2-H1}$ respectively (Gidley and Bociek, 1985).

REFERENCES

- M.J. Gidley and S.M. Bociek (1985) J. Chem. Soc. 220-222.
- G. T. Montelione, M. E. Winkler, P. Rauenbuehler and G. Wagner (1989) J. Magn. Reson. **82**, 198-204.
- P. R. Rosvear, H. A. Nunez and R. Barker (1982) Biochemistry, **21**, 1421-1431.
- I. Tvaroska (1990) Carbohydrate Research **206** 55-64.
- T.E. Walker, R.E. London, T.W. Whaley, R. Barker and N.A. Matwiyoff (1976) J. Amer. Chem. Soc. **98**, 5807-5813.

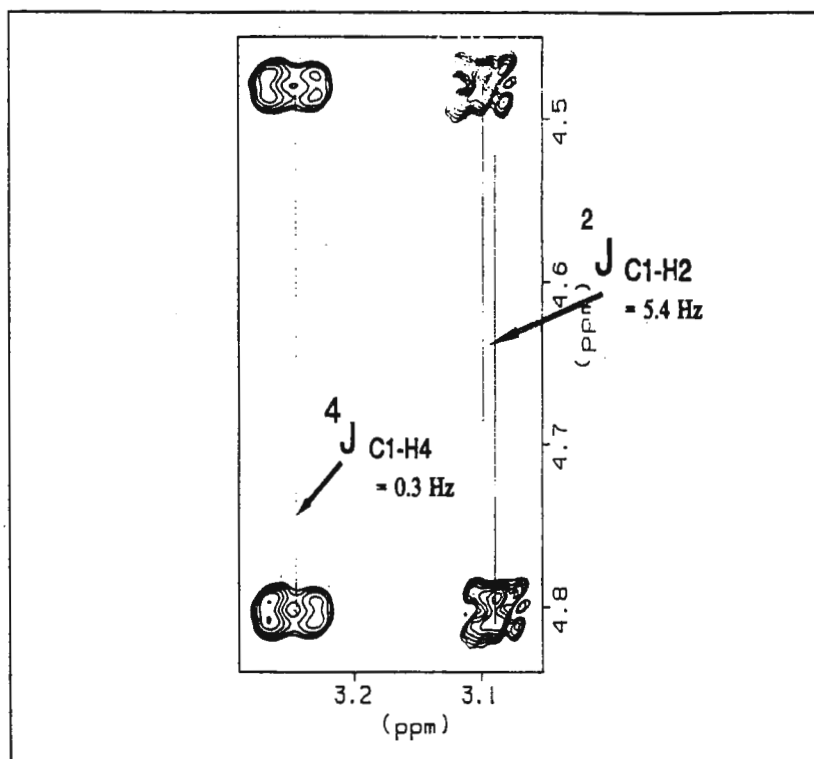


Figure 1. TOCSY cross peaks of β -H1 with H2 and H4 yield estimates of $^2J_{C1-H2}$ and of $^4J_{C1-H4}$.

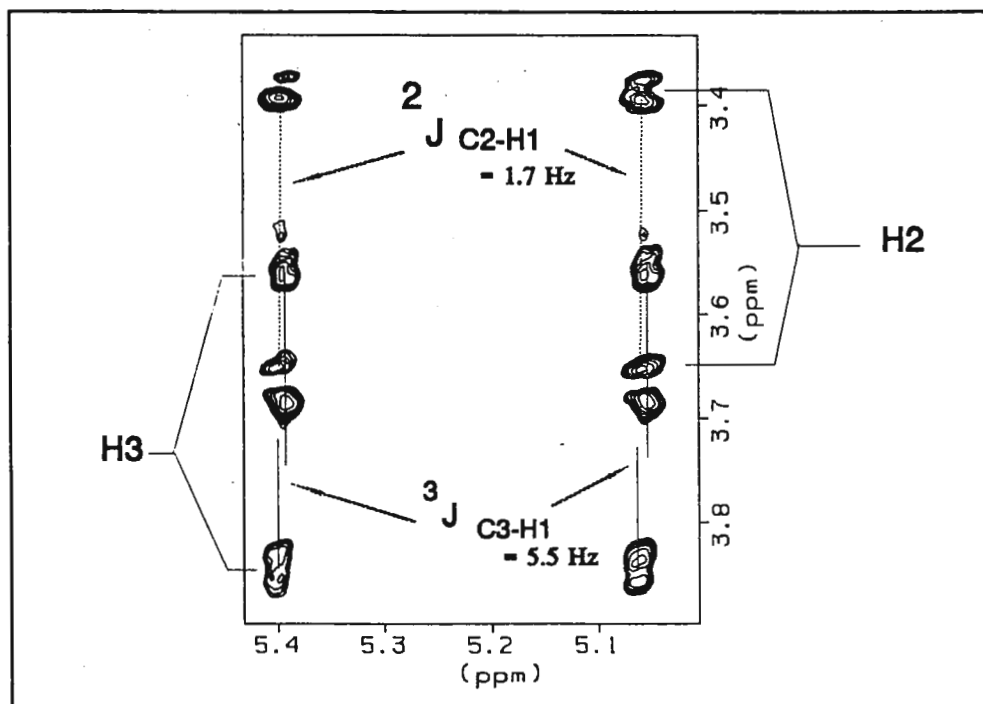


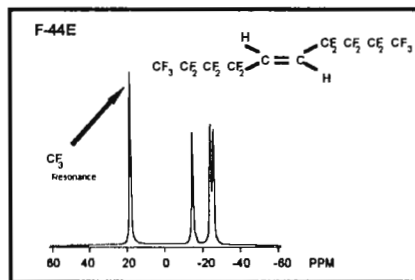
Figure 2. TOCSY cross peaks of α -H2 and H3 with H1 yield estimates of $^2J_{C2-H1}$ and $^3J_{C3-H1}$.



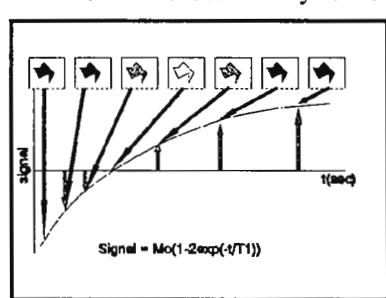
Professor Bernard L. Shapiro
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto, CA 94303

Dr. Shapiro:

Measuring tissue oxygenation non-invasively *in-vivo* is a daunting task. However, it is well known that the spin-lattice relaxation rate of ^{19}F in perfluorocarbon (PFC) emulsions is linearly related to dissolved oxygen content.¹ Spatial mapping of oxygen tension *in-vivo*, using sequestered PFC emulsions, has also been published.² Producing pO_2 maps can take an extraordinary amount of time because of long spin-lattice time constants of ^{19}F PFC emulsions at normal oxygen tensions. We propose a technique to map oxygen tension *in-vivo* by employing ^{19}F echo planar inversion recovery imaging of sequestered PFC emulsions. An inversion recovery method is more accurate than a progressive saturation technique because it provides twice the dynamic range. Since echo planar



imaging also produces an entire image in less than 100 milliseconds, more images can be obtained or averaged at different inversion times making spin echo techniques time prohibitive.



Female, C3H mice are administered a 10g/kg dose of F-44E PFC emulsion (see spectrum in Figure 1) by tail vein injection. In three to five days most of the PFC emulsion is sequestered in the reticuloendothelial system of the liver and spleen. At this time the animals are anesthetized with 1.5% isoflurane in air or pure oxygen. Measurements are performed on a 2.0T GE-CSI-II imaging spectrometer with 20g/cm self-shielded gradients. The transmitter frequency is selectively set on the CF_3 resonance and a 64×64 ^{19}F projection echo planar image, instead of a FID, is acquired at each inversion recovery time (Figure 2). The following TI intervals were used: (0.08, 0.2, 5, 1, 2, 4, 8) seconds at a TR of 10 seconds. At a spectral width of 70KHz, the acquisition time was 28.6msec. With $\text{NA}=2$, the total acquisition time was approximately 2.5 minutes.

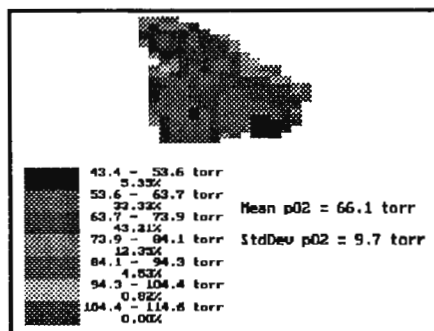
Biomedical Engineering Department

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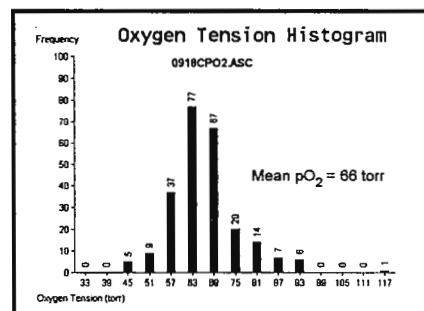
November 18, 1992

Oxygen Tension Mapping with ^{19}F Echo Planar Inversion Recovery Imaging



The oxygen tension maps are then produced by calculating the relaxation rate, R_1 , on a pixel by pixel basis, using a nonlinear least squares algorithm. Each pixel is then assigned a pO_2

value, in torr, from calibrations obtained from phantom experiments. A pO_2 map of the liver and spleen of a mouse breathing 21% O_2 is shown in Figure 3. The corresponding histogram with bin widths of 6 torr is shown in Figure 4. Oxygen tension maps were also produced for the same animal breathing 100% O_2 and post-mortem with mean pO_2 's of 115 and 8.7 torr, respectively. We hope to extend this technique to spatially map tumor oxygenation *in-vivo* to study the effects of adjuvants on radiotherapy.



¹P. Parhami and B.M. Fung, *J. Phys. Chem.*, 87, 1928(1983).

²S.K. Holland, R. Soto, C.C. Jaffe, E. Gordon, and J.C. Gore, *Magn. Reson. Imaging*, 6, (Supplement 1), 72(1988).

Bernard J. Dardzinski
Bernard J. Dardzinski

Christopher H. Sotak

Chris Sotak



Oklahoma State University

DEPARTMENT OF CHEMISTRY
COLLEGE OF ARTS AND SCIENCES

Dr. B. L. Shapiro
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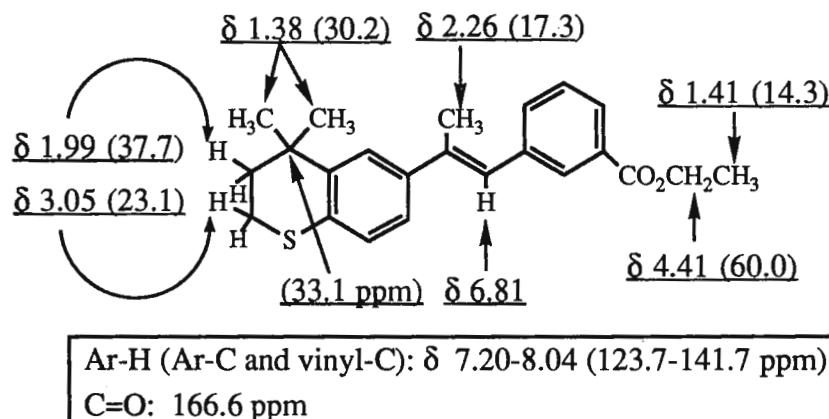
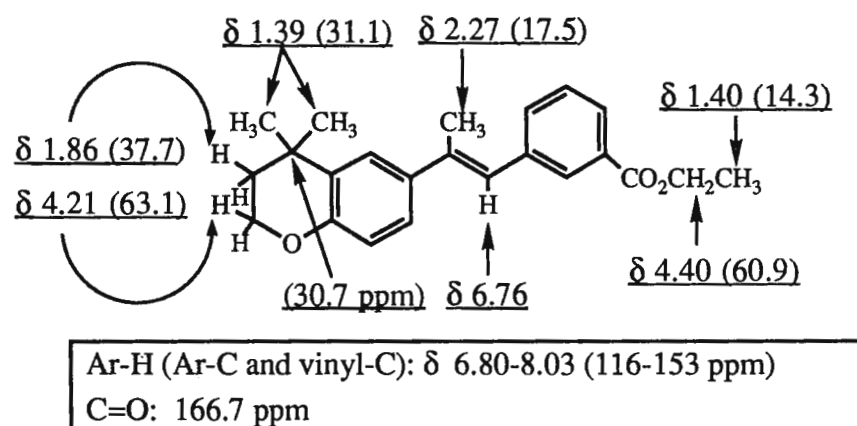
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PHYSICAL SCIENCES 106
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11/24/92
(received 11/27/92)

SUBJECT: Novel Heteroarotinoids

Dear Dr. Shapiro:

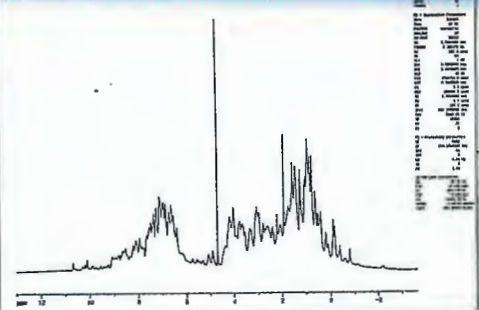
Our contribution to the Newsletter is concerned with some heteroarotinoids (peripherally related to certain natural retinoids). The examples prepared recently were interesting in that at 300 MHz most of the signals were visible. The proton signals are followed by the signals for C-13 in ppm (all from TMS). What



is interesting is that the NMR signals for the oxygen compound (top) in DCCl_3 do not appear to correlate well with what is present in the solid state analysis. The two aryl rings are more twisted (they are tilted approximately 45° with respect to each other) in the solid state than anticipated from the NMR analysis. We are looking at several other examples in this novel family.

Sincerely yours,
Darrell

K. Darrell Berlin
Regents Professor

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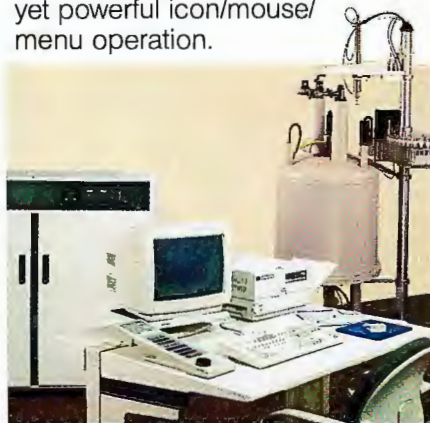
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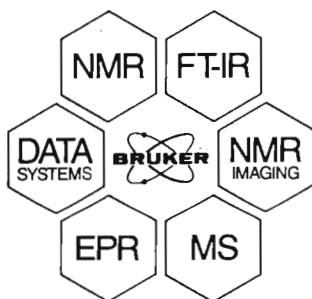
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Performance Data

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

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

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



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

December 14, 1992
(received 12/17/92)

First High Resolution Spectrum at 750 MHz

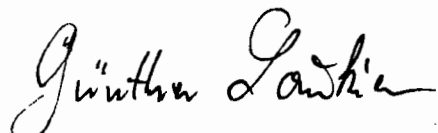
Dear Barry:

BRUKER is proud to present the first high resolution spectrum at 750 MHz. This spectrum was obtained within a few days after energizing our AMX 750 in its final dewar. We first successfully energized a 750 MHz persistent superconducting magnet, which BRUKER built in cooperation with the Kernforschungszentrum Karlsruhe, more than one year ago, and reported it in the TAMU NEWSLETTER in October 1991. We expected then, that the development of a complete 750 spectrometer would take several years, but progress has been more rapid.

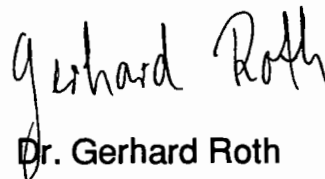
The first 750 MHz spectrum shown in Fig. 1 was obtained from a cyclic tripeptide (Cyclo [L - Pro - L - Pro - L - Thz]), using a 5 mm proton selective probe (with lock) and BOSSTM shims on an AMX 750 with SE-451TM receiver. For comparison, the same spectrum is also shown using an AMX 600 spectrometer. Besides the 25 % higher dispersion, which is clearly evident, the absolute resolution of the 750 MHz spectrum is essentially the same as at 600 MHz. Due to the higher dispersion, the two spin systems which at 600 MHz are still partially overlapping, are now well resolved at 750 MHz.

We hope that this first spectrum gives a good indication of the future potential of this new class of high-resolution, solid-state and microimaging NMR spectrometers. As progress continues, we will be happy to keep you informed about the latest developments with this new research spectrometer.

Sincerely,

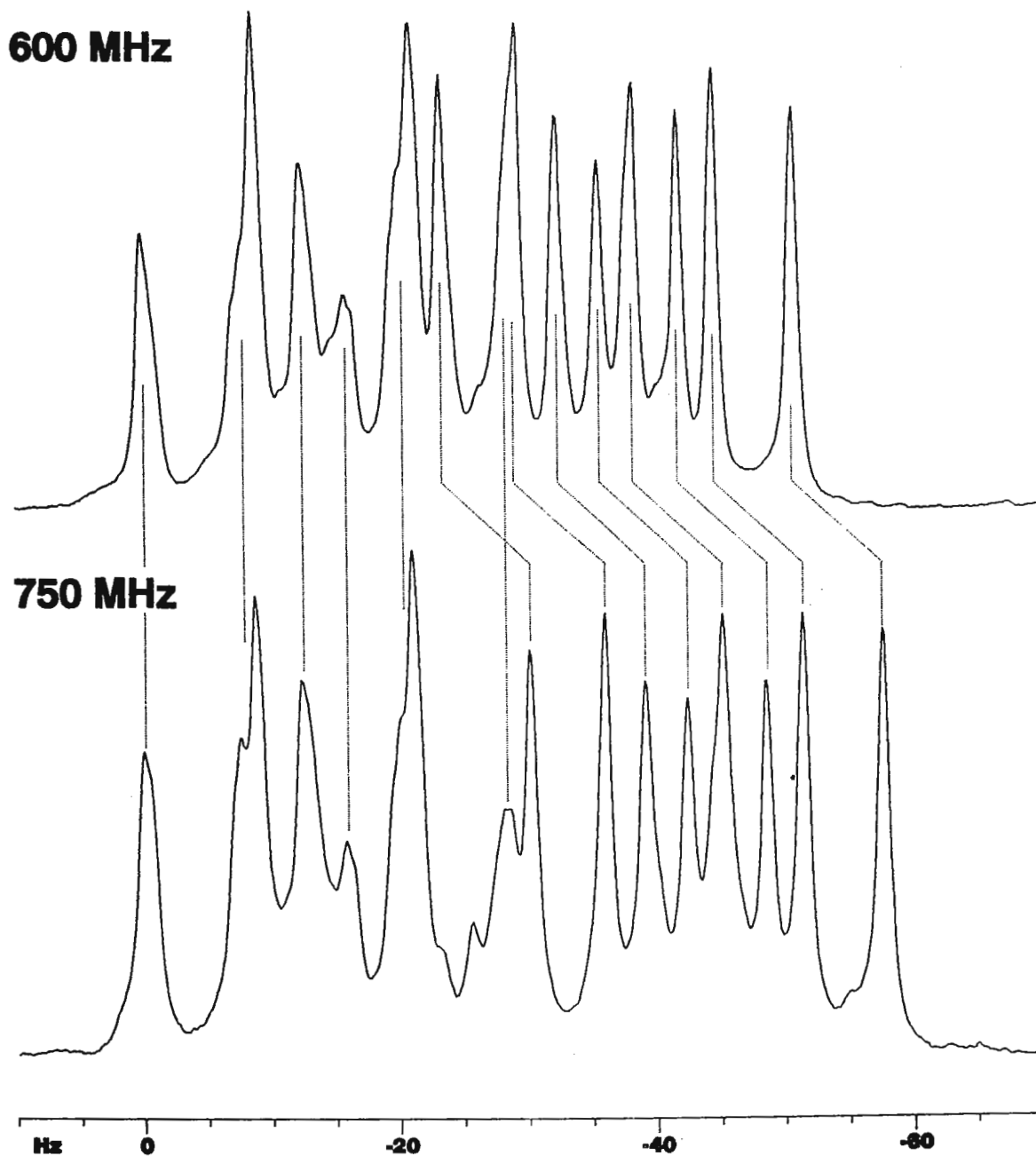


Prof. Dr. Günther Laukien



Dr. Gerhard Roth

**Fig.1: 600 and 750 MHz spectra of cyclo[L-Pro-L-Pro-L-Thz] in CDCl₃.
The expansion shows the signals of the cis- δ proton of proline
1 and 2. Both spectra are plotted on the same Hz/cm scale.**



We acknowledge Dipl. Phys. R. Ruin for quickly shimming the magnet and Dr. W. Bermel for recording this first spectrum.

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Dr B. L. Shapiro
TAMU NMR Newsletter
966 Elsinore Court
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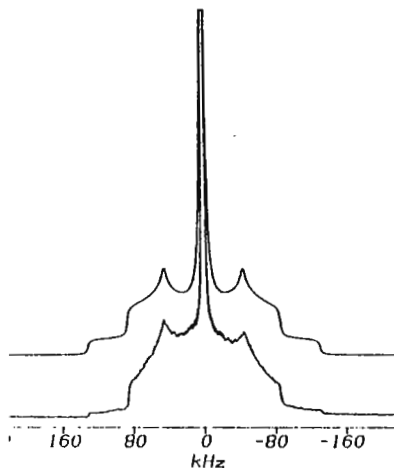
Dec 1 1992 (received 12/10/92)

Dear Barry,

QCC and Asymmetry Parameters of Phenyllithiums Using Solid State ^7Li NMR.

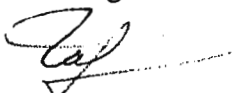
For many organolithium compounds it is possible to obtain a detailed information of the state of aggregation in solution by measuring lithium relaxation, provided that there exists a reliable probe for the effective rotational correlation time of the studied complex. However, this latter requirement is quite critical and the obtained quadrupolar splitting constants (QSC) may be far from reliable.

To circumvent this problem and to compare the solution and solid state data, we have studied microcrystalline samples of variously complexed phenyllithiums using static and MAS ^7Li NMR. A representative experimental and simulated powder spectrum is shown below for the PMDTA complex yielding a QCC of 260 kHz as expected for a monomer. For the TMEDA adduct, being a dimer according to X-ray, we obtained a QCC of 147 kHz, which affords a QSC of 161 kHz as compared to the solution value of 162 kHz. Similar agreement between solid and solution data was noted for tetrameric complexes. So solid state studies can be quite useful to characterize reactive organolithium species and to connect X-ray and solution experiments. However, keep your polymeric rotors away from these species. Organolithium compounds eat them.



All solid state studies so far have been performed on our old Bruker MSL-100 instrument. We will soon have solid facilities on our 500 MHz system also together with an upgrade from AM to a rack version of the new "X32-free" AMX.

Best regards


Ulf Edlund

Dan Johnels



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Prof. B.L. Shapiro
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Prof. P. Diehl
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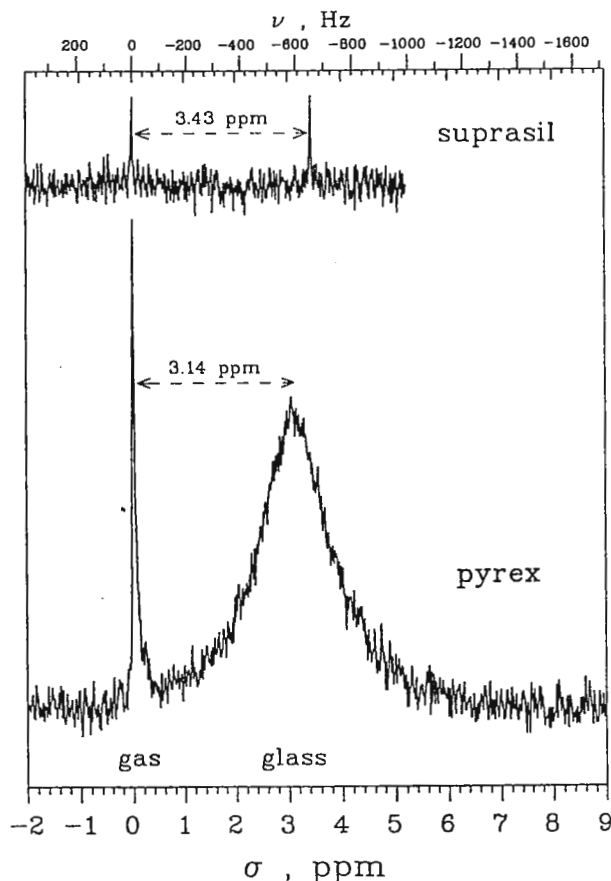
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diehl@urz.unibas.ch

December 14, 1992
(received 12/18/92)

NMR of ^3He in vitreous silica

Dear Barry,

We have lately been interested in measuring solvent effects on the chemical shift of ^3He in liquids. During these studies we observed that ^3He can also be detected if it has penetrated into the glass of the sample wall (see the Fig.). So far we have been able to obtain signals of ^3He in quartz (suprasil) (one FID) and in pyrex (several thousand FID's). In suprasil the diffusion is rather fast (half time of ≈ 2 days); in pyrex it is slower. We have studied the ^3He relaxation and have been able to attribute it to the dipole-dipole interaction in pyrex with Fe^{3+} impurity ions and in suprasil with unpaired electrons of defects. Presently we are studying ^3He in various solids.



With best regards

P. Diehl
Prof. P. Diehl,

M. Mazitov
Dr. R. Mazitov,

R. Seydoux
R. Seydoux

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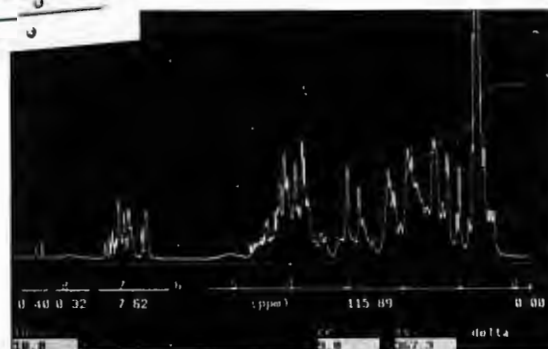
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Input/Output impedance	50 ohms
Input VSWR	$< 2:1$
Pulse width	20 ms
Duty cycle	Up to 10%
Amplitude rise/fall time	250 ns typ.
Amplitude droop	5% to 20 ms typ.
Phase change/output power	10° to rated power, typ.
Phase error overpulse	4° to 20 ms duration, typ.
Noise figure	11 dB typ.
Output noise (blanked)	< 20 dB over thermal
Blanking delay	$< 2 \mu\text{s}$ on/off, TTL signal
Protection	1. VSWR: infinite VSWR 2. Input overdrive: up to 10 dB 3. Over duty cycle/pulse width 4. Over temperature

Supplemental characteristics:

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Indicators, front panel	1. AC power on 2. Peak power meter 3. Over pulse width 4. Over duty cycle 5. Over temperature 6. Over drive 7. CW mode
System monitors	1. Forward/Reflected RF power 2. Over pulse width/duty cycle 3. DC power supply fault 4. Thermal fault
Front panel controls	1. AC power 2. Pulse width 3. Duty cycle
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Operating temperature	$+10$ to 40°C
AC line voltage	208/230 VAC, $\pm 10\%$, 50–60 Hz
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THE UNIVERSITY OF NORTH CAROLINA
AT
CHAPEL HILL

Department of Chemistry

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

CB# 3290, Venable and Kenan Laboratories
The University of North Carolina at Chapel Hill
Chapel Hill, N.C. 27599-3290

(received 12/15/92)

"Squeezing more out of FT-PGSE self-diffusion experiments - blood from a turnip (?)"

Dear Barry,

Thank you for the final ultimatum to TAMU NMR Newsletter subscriber PS (normal address RIT, Stockholm, but presently having a short sabbatical in the laboratory of Charles S. Johnson Jr., at UNC). We have done some independent work along the lines of improved data evaluation in the case of FT-based pulsed-gradient NMR spin-echo experiments for the determination of self-diffusion coefficients on multi-component or polydisperse samples in solution. Self-diffusion coefficients contain a wealth of highly relevant and easily conceived physico-chemical information, especially on colloidal or macromolecular systems in solution, and NMR is undoubtedly the method of choice for accessing this type of information, as compared to radioactive tracer methods, or optical techniques.

The traditional approach of measuring NMR spin-echo intensities or peak integrals for subsequent fitting to the Stejskal-Tanner equation is normally sufficient for the majority of needs. However, if there is extensive peak overlap between components, or if the sample contains some polydisperse polymer, one must turn to more advanced data processing. With present-day instrumentation, it is natural then to try to use the complete experimental information available in bandshapes already stored on the spectrometer disk, and to utilize the really enormous computing power available on the on-line or network-connected workstations typically available.

Details of full FT-PGSE spectral data processing through deconvoluting routines like CONTIN, SPLMOD or DISCRETE (see e.g. S.W. Provencher, *Comput. Phys. Commun.*, 27 (1982) 213 and 229), adding some extra "decision rules" for the deconvoluting process was recently submitted for publication (Morris and Johnson, see also previous communications in *JACS* 114 (1992) 3139 and 776). Figure 1 illustrates the power of that approach, and the information content that can be achieved. The 2D variant of the techniques is named DOSY (Diffusion Ordered NMR Spectroscopy). Together with former Stockholm postdoc Dirk Schulze (now at Shell Laboratories, Amsterdam), PS developed a complementary approach, that enables one to directly deconvolute spectral bandshapes, contributing to severely overlapping bands in FT-PGSE data sets, even in the case of very similar self-diffusion coefficients (Figure 2; the paper will be submitted when this appears in print).

We believe that procedures like these will be of significant value in future FT-PGSE work on more complex systems than previously considered feasible. Please credit this contribution to Peter Stilbs subscription.

Best regards,

Kevin Morris

Charles S. Johnson, Jr

Sincerely Yours

Peter Stilbs

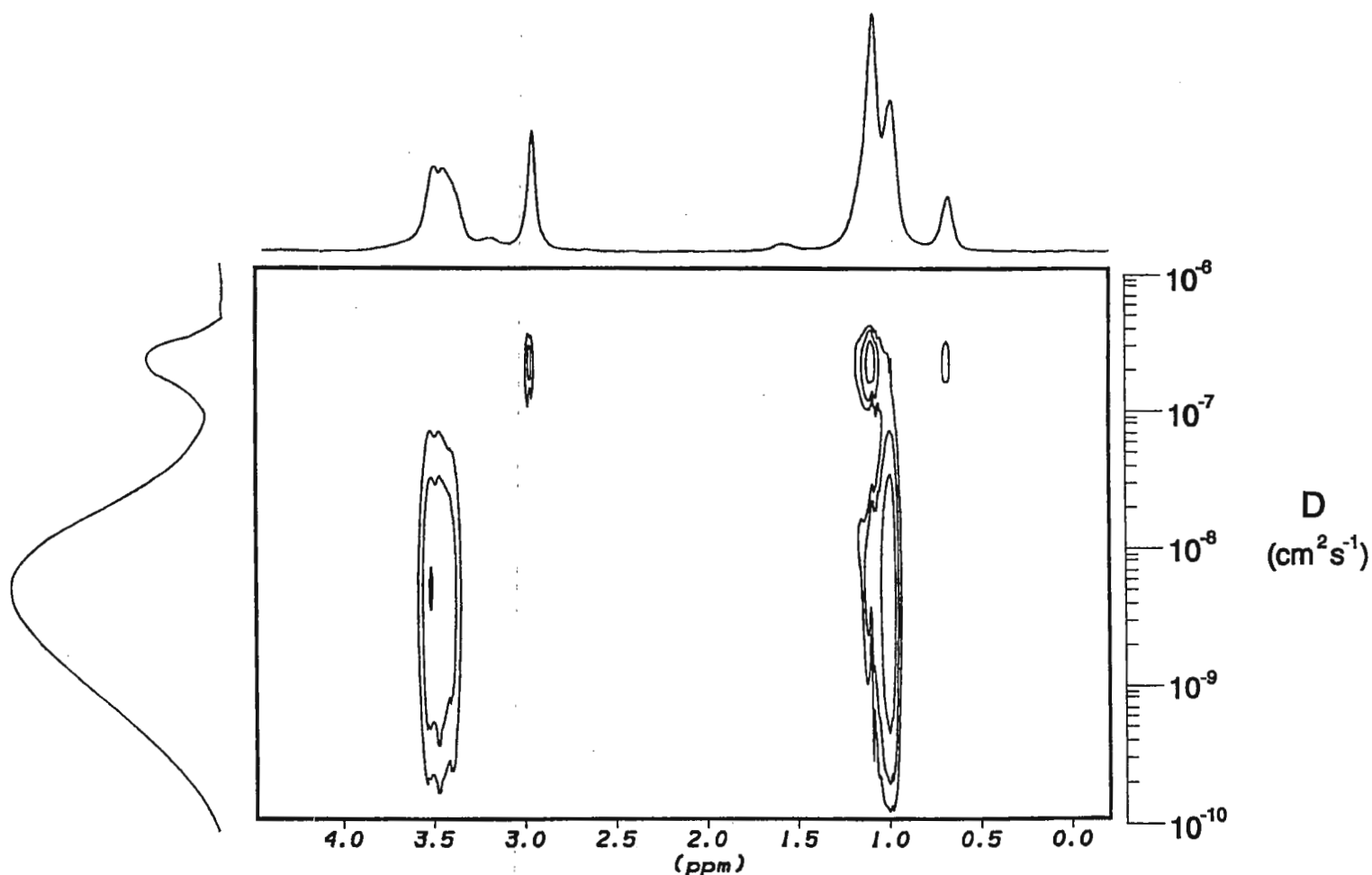


Figure 1: A 250 MHz Proton DOSY spectrum of 1% Ethyl(hydroxyethyl)cellulose (EHEC) and 0.34% cetyl trimethylammonium bromide (CTAB) in heavy water. Such systems exhibit highly unusual rheologic behaviour. Note the very wide (polydisperse) EHEC diffusion distribution, and the very much narrower one for CTAB.

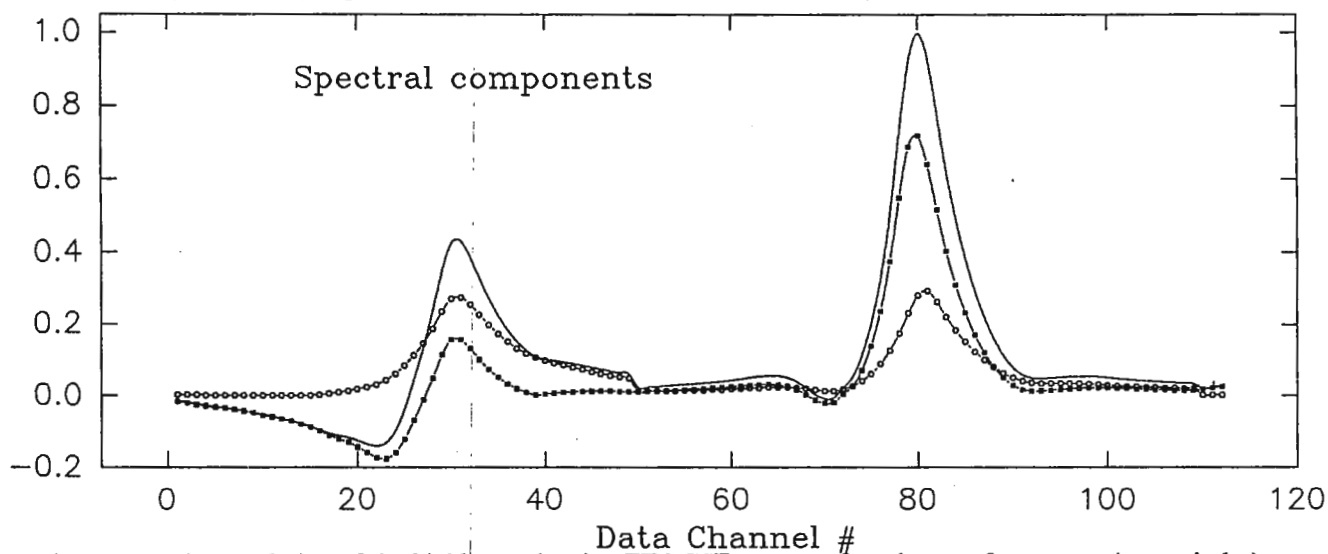


Figure 2: A deconvolution of the highly overlapping FT-PGSE spectra of a mixture of n-octane (open circles) and n-heptanol (filled squares); as based on differential signal attenuation effects on the combined bandshape (solid line) during FT-PGSE experiments.



CENTRO DE INVESTIGACION Y DE ESTUDIOS AVANZADOS DEL I.P.N.

APARTADO POSTAL 14-740

MEXICO 07000, D.F.

November 6, 1992.

(received 11/21/92)

Dr. Bernard L. Shapiro
Editor/Publisher
TAMU NMR Newsletter
966 Elsinore Ct.
Palo Alto, CA 94303 USA

¹H NMR Studies of E and Z 2-hydroxyindolenines

Dear Professor Shapiro:

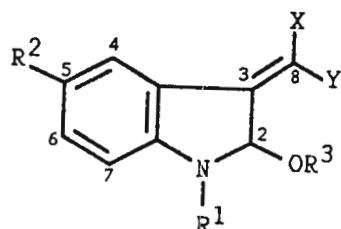
The proton signals of 2-hydroxyindolenines 1-4 and of the 2-methoxy derivative 5 were assigned after first-order analysis of the 300 MHz spectra measured in CDCl₃ and DMSO-d₆ solutions from mixtures of E and Z isomers. The data are listed in the Table.

In the chloroform spectra, the proton signals from position 2 owing to the E and Z isomers (E/Z ratio ca 1:10) appear as broad doublets resulting from the interaction with the 2-OH group. For the Z isomer the H-2 signal appears at lower fields (0.41-0.47 ppm), compared with that of the E isomer, as a result of a selective anisotropic effect exerted by the carbonyl moiety of the ester group at C-8. Accordingly, the relative shieldings of the H-4 signals in the E,Z pairs vary in opposite directions, with H-4 in the Z isomer resonating at higher fields (0.30-0.41 ppm) than the E isomer. In the later form, H-4 is coplanar with the 8-carbonyl moiety^{1,2}. Consequently the chemical shifts owing to H-2 and H-4 can be used as key arguments for E,Z assignments. Although all other aromatic protons are less affected by the configuration of the C/3=C/8 double bond, they were easily discerned from the proton-proton coupling patterns.

On changing the solvent from CDCl₃ to DMSO-d₆ some chemical shift variations were observed. As expected, the most severe change occurs in the 2-OH chemical shift, which was considerably more shielded in CDCl₃ (by ca 3 ppm) than in DMSO-d₆.

Contrarily to observations for the related 2-oxoindolenines³, the E/Z ratio for 1-5 were found to be solvent independent.

- 1.- G. Jones and W.J. Rae, Tetrahedron, 22, 3021 (1966).
- 2.- R.L. Autrey and F.C. Tahk, Tetrahedron, 23, 901 (1967).
- 3.- M.S. Morales-Ríos, Y. Mora-Pérez and P. Joseph-Nathan, Magn. Reson. Chem., in press.



		X	Y	R ¹	R ²	R ³
<u>1</u>	E	CO ₂ Me	CN	CO ₂ Me	H	H
	Z	CN	CO ₂ Me	CO ₂ Me	H	H
<u>2</u>	E	CO ₂ Et	CN	CO ₂ Et	H	H
	Z	CN	CO ₂ Et	CO ₂ Et	H	H
<u>3</u>	E	CO ₂ Me	CN	CO ₂ Me	NO ₂	H
	Z	CN	CO ₂ Me	CO ₂ Me	NO ₂	H
<u>4</u>	E	CO ₂ Et	CN	CO ₂ Et	NO ₂	H
	Z	CN	CO ₂ Et	CO ₂ Et	NO ₂	H
<u>5</u>	E	CO ₂ Me	CN	CO ₂ Me	H	Me
	Z	CN	CO ₂ Me	CO ₂ Me	H	Me

Table.- Chemical shifts in CDCl₃ (and DMSO-d₆).

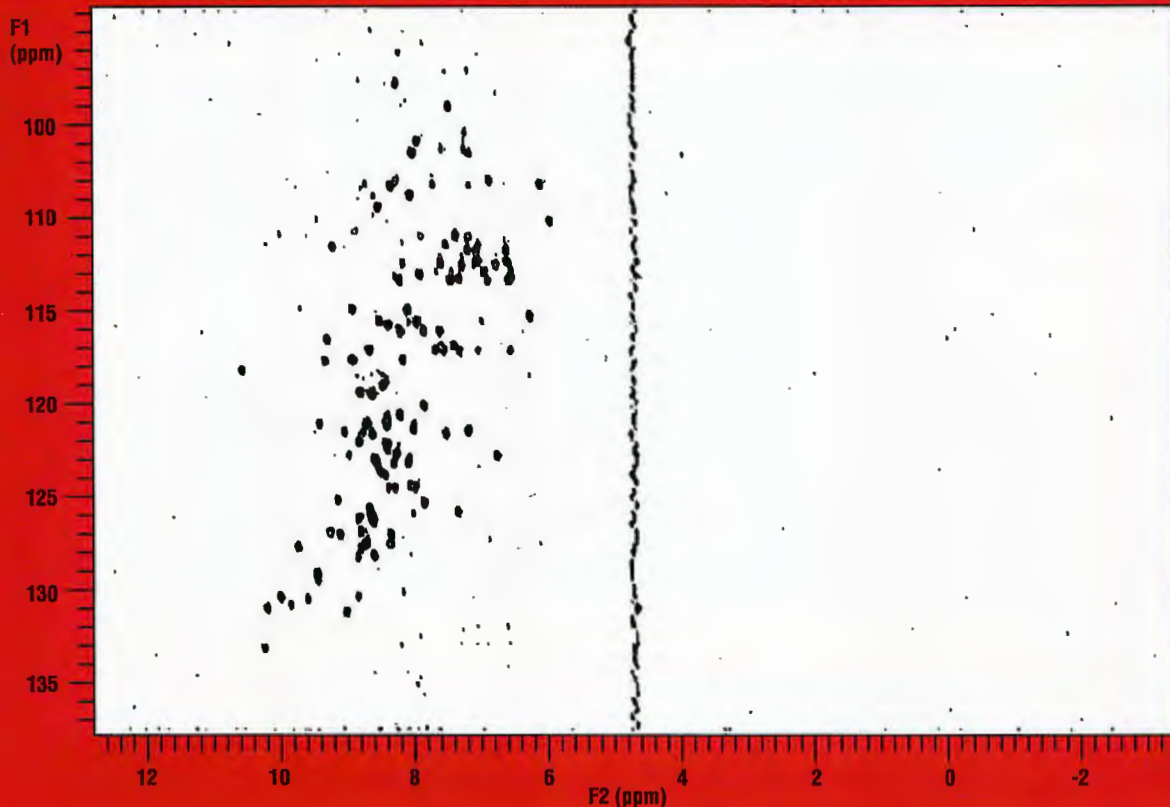
	H2	H4	H5	H6	H7	OH
<u>1</u>	E 6.33(6.16)	8.76(8.57)	7.11(7.24)	7.53(7.65)	7.98(7.88)	4.65(7.69)
	Z 6.80(6.71)	8.46(8.29)	7.18(7.27)	7.60(7.69)	7.98(7.88)	4.50(7.40)
<u>2</u>	E 6.39(6.13)	8.75(8.57)	7.12(7.15)	7.52(7.63)	7.95(7.88)	4.68(7.66)
	Z 6.80(6.69)	8.46(8.27)	7.16(7.23)	7.59(7.66)	7.95(7.88)	4.52(7.32)
<u>3</u>	E 6.50(6.25)	9.76(9.56)	-	8.44(8.49)	8.13(8.01)	4.74(8.05)
	Z 6.92(6.76)	9.33(9.05)	-	8.48(8.52)	8.13(8.01)	4.48(7.77)
<u>4</u>	E 6.51(6.24)	9.74(9.51)	-	8.43(8.46)	8.12(8.00)	4.69(8.03)
	Z 6.92(6.72)	9.33(9.03)	-	8.47(8.50)	8.12(7.98)	4.54(7.70)
<u>5</u>	E 6.19(6.15)	8.72(8.57)	7.12(7.20)	7.55(7.66)	7.94(7.86)	-
	Z 6.88(6.66)	8.46(8.27)	7.18(7.28)	7.58(7.70)	7.94(7.86)	-

Sincerely yours,

Martha S. Morales-Ríos

Pedro Joseph-Nathan

Varian's Pulsed Field Gradients Provide Enhanced Sensitivity.



This phase sensitive ^{15}N HSQC spectrum of 1.7 mM uniformly ^{15}N enriched 110 amino acid cellulose binding domain fragment of cellulase in 90% $\text{H}_2\text{O}/\text{D}_2\text{O}$ was obtained on a UNITY[®] 500 MHz spectrometer equipped with Varian's new Pulsed Field Gradient module. The experiment was acquired with 1 transient per increment and no presaturation. Sample provided by Dr. L. Kay (University of Toronto).

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December 16, 1992
(received 12/18/92)

Varian Obtains NMR Results at 750 MHz

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

Dear Barry,

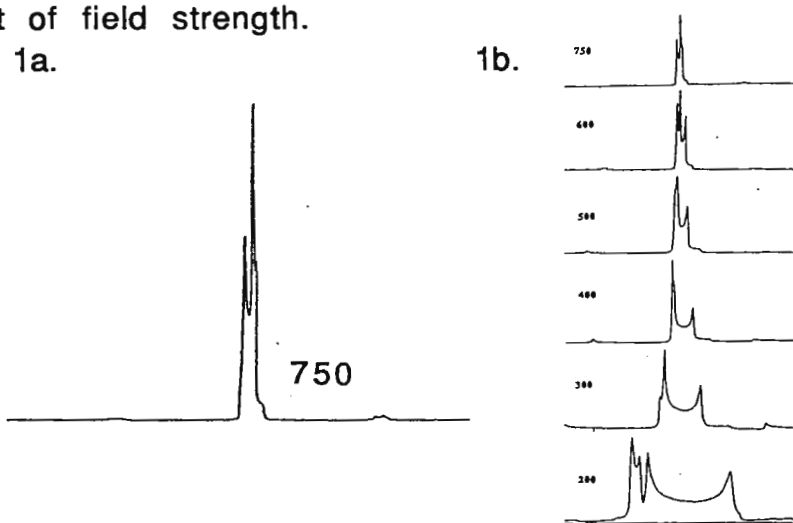
We are currently acquiring data on our new Varian UNITYplus™ NMR console connected to a prototype Oxford 750 MHz superconducting magnet! We believe these are the first reported high resolution NMR results at 750 MHz for chemical and biomolecular applications which further demonstrates Varian's continuing commitment to high field NMR.

Varian has been developing probes and spectrometer consoles at 750 MHz in anticipation of the Oxford 750 MHz magnet. The spectra below demonstrate that today's 750 MHz NMR systems provide excellent results in a variety of experiments with the resolution and sensitivity enhancements that are expected.

Results

1.a. The ^{23}Na MAS spectrum of Na_2MoO_4 continues to narrow as the data is acquired at higher field strengths.

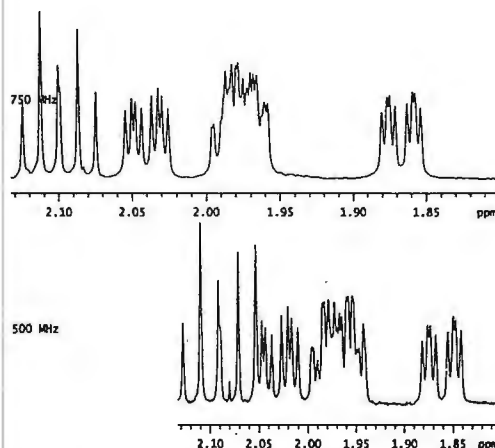
b. The series of spectra plotted from 200 to 750 MHz shows the effect of field strength.



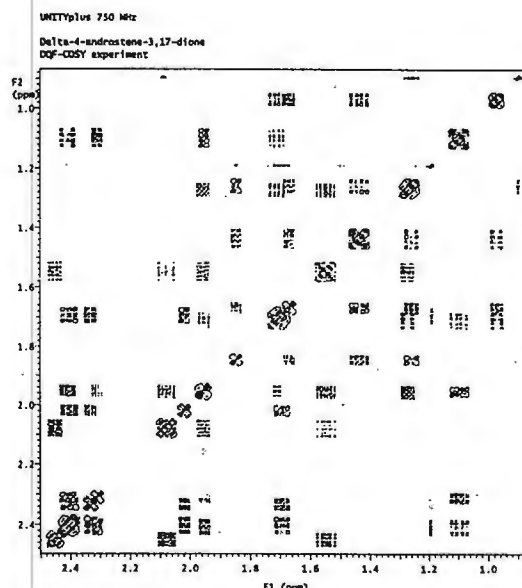
2.a. The second result is a comparison of resolution on a 750 MHz spectrometer with a 500 MHz spectrometer. The sample is delta-4-androstene-3,17-dione. The gain in resolution due to chemical shift dispersion is quite significant.

b. The DQCOSY on the same sample demonstrates that the magnet and spectrometer technology available today is capable of providing long term two dimensional results.

2a.

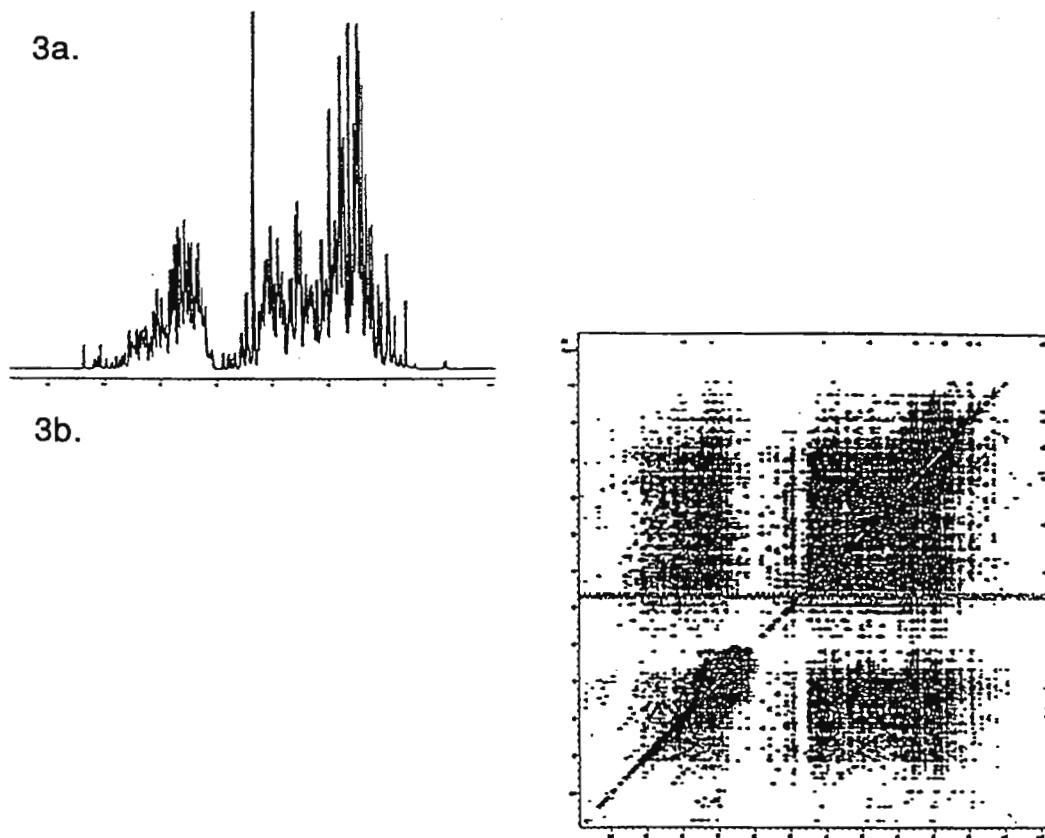


2b.



3.a. The third result is a water presaturation experiment on 5mM Lysozyme in 90% H₂O/D₂O. The result shows that excellent lineshape can be obtained on the 750 MHz system and biomolecular studies are feasible today. The peaks next to the residual water in the 1D spectrum are not suppressed.

b. The NOESY on 5mM Lysozyme in 90% H₂O/D₂O has a narrow water stripe with resolution and sensitivity superior to a 600 MHz spectrum.



We are extremely excited about these results and are looking forward to working with scientists worldwide on additional applications at 750 MHz.

Sincerely,

Timothy Saarinen

Timothy Saarinen
Product Manager

Mark van Criekeing

Mark van Criekeing
R&D Engineer

PETER SANDOR

Peter Sandor
Applications Chemist

George Gray

George Gray
Applications Chemist

Ali Behbin *Ali Behbin*
Probe Engineer

Ronald Haner
Ron Haner
Probe Engineer



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This position requires an advanced degree in Applied Physics or Chemistry and 5 years' experience in biomolecular NMR techniques and applications. A thorough understanding of present day high-resolution NMR methods is also required. Familiarity with modern NMR instrumentation is desirable and good communication skills are essential.

Senior Applications Chemist

You will be responsible for conducting demonstrations of NMR equipment, running customer samples, presenting workshops, and evaluating new equipment. You'll also write manuals, assist in product development, participate in research programs, and provide post-sales customer support and training.

We require a PhD in Chemistry or Physics, or equivalent, and 3-5 years' experience with state-of-the-art biomolecular NMR. You'll also need a background developing and/or implementing NMR pulse sequences, an understanding of NMR phenomenon sufficient to implement and test new experiments, and familiarity with UNIX*. Excellent communication and presentation skills essential. NMR hardware design or troubleshooting experience desirable.

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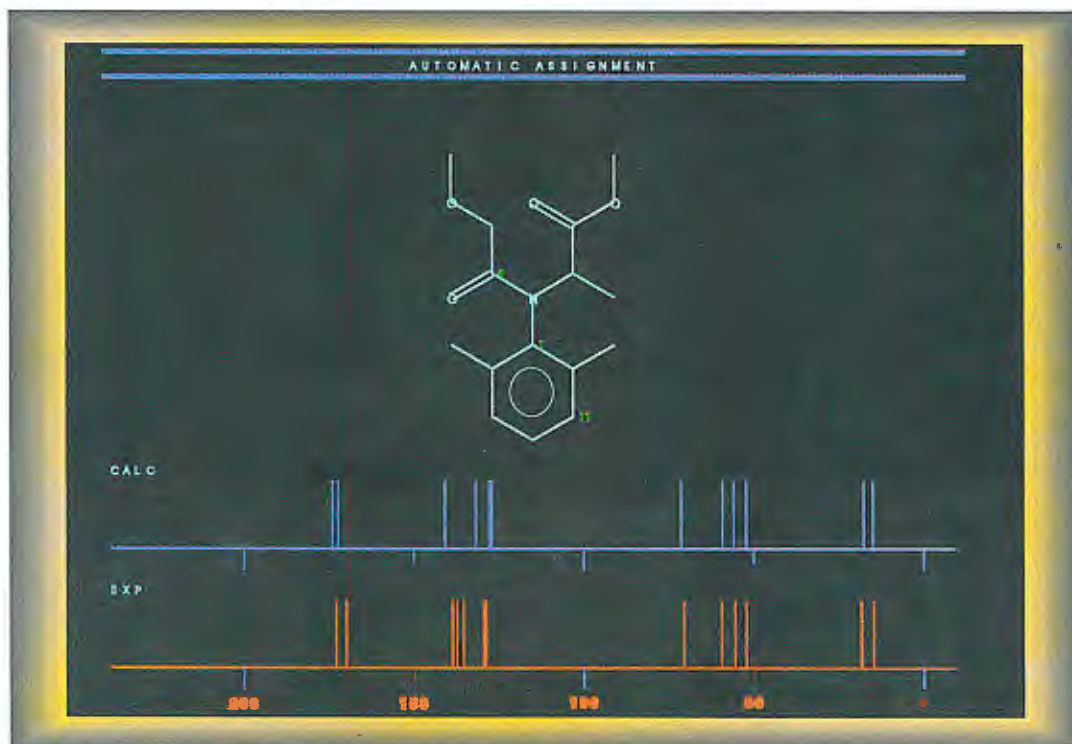
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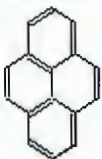
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Boiling Point	
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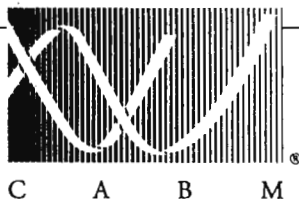
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CENTER FOR ADVANCED BIOTECHNOLOGY AND MEDICINE

679 Hoes Lane • Piscataway, NJ 08854-5638 • 908/463-5300

November 23, 1992
(received 11/28/92)Triple Resonance Experiments for Determining Resonance Assignments
in Small Proteins

Dear Dr. Shapiro,

During the last several months we have developed an improved strategy for determining sequence-specific NMR assignments in small isotope-enriched proteins. In our approach, the ^1H - ^1H planes from a set of ^{15}N -resolved 3D-NMR experiments are analyzed to provide both spin system identification and sequential connectivity information. These pulse sequences are extensions of the basic HCNH triple resonance^{1,2} experiment, modified to provide HCCH TOCSY transfer from sidechain protons, through sidechain and backbone carbon nuclei, to backbone amide nitrogen and protons. Multidimensional $\text{H}_2\text{C}_2\text{CA}_2\text{N}_2\text{H}_2$ TOCSY³ experiments are first used to connect together the resonances of each amino acid spin system and to provide unique identification of many spin system types, including those of asparagine and glutamine which exhibit cross peaks between sidechain aliphatic and sidechain amide nuclei. Next, sequential connections between amino acid spin systems are established using multidimensional $\text{H}_2\text{C}_2\text{CO}_2\text{N}_2\text{H}_2$ TOCSY⁴. The $\text{H}_2\text{C}_2\text{CA}_2\text{N}_2\text{H}_2$ TOCSY³, or "CA-TOCSY", spectra provide an extensive set of intraresidue connections between proton, carbon, and nitrogen resonances of each amino acid in the sequence, while the complementary $\text{H}_2\text{C}_2\text{CO}_2\text{N}_2\text{H}_2$ TOCSY⁴, or "CO-TOCSY", spectra provide sequential connections between aliphatic α , β , γ , and δ proton and carbon resonances of residue i and the amide proton and nitrogen resonances of residue $i+1$. These experiments are related to CBCANH and CBCA(CO)NH triple experiments recently described by Grzesiek & Bax^{5,6}.

Representative data from triple-resonance TOCSY spectra are shown in Figs. 1 and 2. Together, these multidimensional CA- and CO-TOCSY experiments provide the information needed to determine most of the sequence-specific backbone and sidechain resonance assignments in small proteins. Details of these pulse sequences and their application in determining sequence specific resonance assignments in proteins will be presented elsewhere^{3,4}.

Barbara A. Lyons

Mitsuru Tashiro

Gaetano T. Montelione



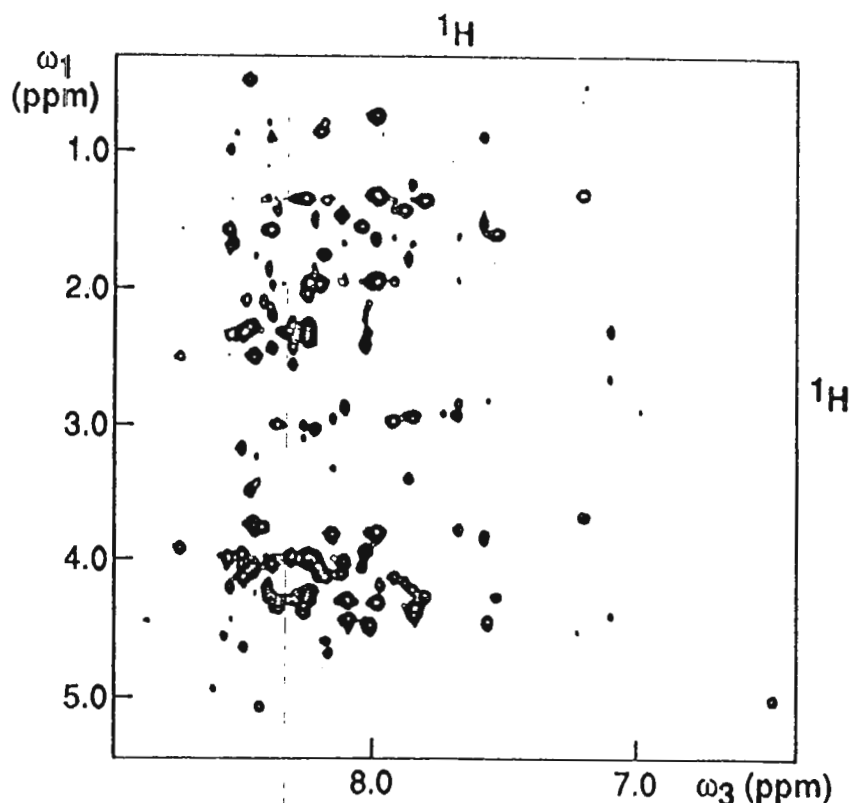


Fig. 1. 2D CO-TOCSY spectrum of a 100% uniformly ^{13}C - and ^{15}N -enriched sample of a modified 8.2 kDa domain (called Z-Domain⁷) of the immunoglobulin-binding Protein A from *Staphylococcus aureus* at protein concentration of 2 mM. These data were obtained at pH 6.5 and temperature of 30° C. in 24 hours using a Varian Unity 500 NMR spectrometer. Detailed analysis of the spectrum reveals that most of the cross peaks are sequential correlations between the sidechain aliphatic protons of residue i and backbone amide protons of residue $i+1$. In addition, for asparagine and glutamine residues intrasidue cross peaks are observed between aliphatic and sidechain amide protons. Connections between sidechain aliphatic and amide protons of asparagine and glutamine are also observed in CA-TOCSY spectra.

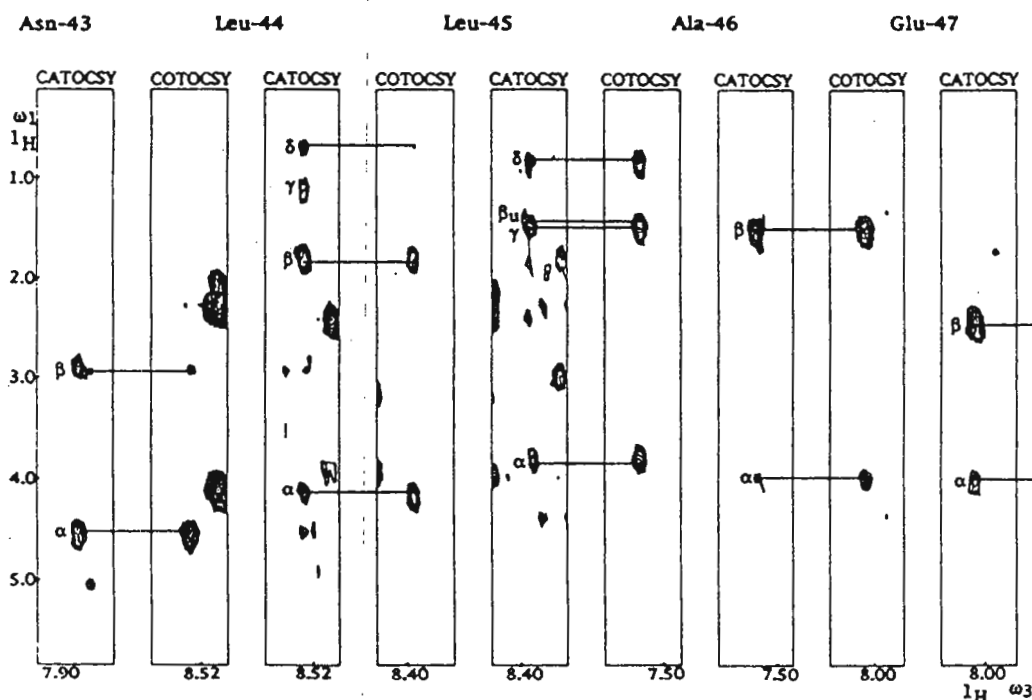


Fig. 2. Illustration of our strategy for correlating intrasidue cross peaks of residue i from the 3D CA-TOCSY spectrum with the corresponding sequential cross peaks at the amide frequency of $i+1$ in the 3D CO-TOCSY spectrum. This panel shows $\omega_2 = ^{15}\text{N}$ strips from 3D data sets establishing connections within the polypeptide segment Asn-43 to Glu-47 of the Z-Domain of Protein A⁷. Sample conditions are the same as in Fig. 1. Although this Z-Domain is highly α -helical and exhibits highly degenerate ^1H and ^{13}C spectra, the 2D and 3D CA- and CO-TOCSY experiments have enabled us to determine an extensive set of sequence-specific ^1H , ^{15}N and ^{13}C resonance assignments without recourse to NOESY.

(1) Montelione & Wagner, 1989 *J. Am. Chem. Soc.* 111, 5474. (2) Montelione & Wagner, 1990 *J. Magn. Reson.* 87, 183. (3) Lyons & Montelione, submitted. (4) Montelione, et al. *J. Am. Chem. Soc.* (in press). (5) Grzesiek & Bax, 1992 *J. Am. Chem. Soc.* 114, 6291. (6) Grzesiek & Bax, 1992 *J. Magn. Reson.* 99, 201. (7) Nilsson et al., 1987 *Protein Engineering* 1, 107.



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

(received 12/2/92)
26 November 1992

Accurate Multiexponential Analysis of Relaxation Decays Based on Linear Prediction and Singular Value Decomposition

Dear Prof. Shapiro,

An important element in relaxation-time analysis is the fitting of a decay curve to the sum of a series of exponential functions. An examination of past literature clearly reveals that such multiexponential decays function is particularly difficult to analyse using nonlinear least squares fitting¹. The difficulty lies in the fact that a decay of this type is particularly insensitive to changes in the parameters defining the exponential components, or even to change in the number of components assumed to represent the decay. Besides, in such a fitting process, the experimental error has a strong influence on the accuracy of the determined amplitudes and damping factors (inverse time constants). Thus conventional nonlinear least squares analysis is sensitive to noise and gives poor estimates when the signal-to-noise ratio is low. Hence, a very accurate analysis of multiexponential decays requires exceedingly, and sometimes impossibly, accurate data extending over a very large part of the decay curve.

Recently, we proposed a novel method based on linear prediction and singular value decomposition for the parameters of superimposed exponentials corrupted by random noise. By invoking the principle of linear prediction the fitting can be carried out by a linear least squares procedure, and therefore needs no subjective initial guess. The least squares procedure is based on singular value decomposition, which enables one to distinguish between signal and noise subspaces. This method yields a list comprising the damping factor and amplitude of each distinct exponential decay. The mathematical method used in this work was originally proposed by Kumaresan and Tufts (KT)² for estimating the parameters of damped exponential sinusoids in additive white noise. While KT's method performs well for spectrum analysis applications, it is shown here that it provides poor estimates of damping factors when directly applied to the multiexponential decay cases without any modification. This failure is because the bias in the damping factor and amplitude become significant at low signal-to-noise ratio. This type of bias is common in linear prediction methods and is due to the noise variance contributing significantly to the signal subspace. To overcome this problem, we present an empirical formula to subtract the noise contamination from the signal singular values. In addition to the normal effective singular values, we have also found it useful for further enhancing the accuracy to include the two sequential smaller singular values and their corresponding singular vectors such that the smaller signal components remain present in the signal subspace.

Intensive Monte Carlo simulations for Kumaresan and Tufts's two-exponential test sample² and Clayden and Hesler's three-exponential test sample¹ are used to exhibit the improved robustness of this method compared to conventional nonlinear least squares analysis. A typical result of Kumaresan and Tufts's two-exponential test sample compared to nonlinear least square analysis is shown in Fig. A and B respectively with the true values of (damping factor and amplitude) located at (0.1,1) and

(0.2,1). Experimental data of ^7Li longitudinal and transverse relaxations are also used which contain only 14 data points. Since ^7Li is an $I=3/2$ quadrupolar nucleus, the relaxations exhibit two-exponential behavior when the extreme narrowing condition is not satisfied. The longitudinal relaxation has the function form $\left(\frac{1}{5}e^{-r_1t} + \frac{4}{5}e^{-r_2t}\right)$, and the transverse $\left(\frac{3}{5}e^{-s_1t} + \frac{2}{5}e^{-s_2t}\right)$, respectively. The damping factors of both longitudinal and transverse relaxations estimated by our new method, our previously proposed null point spectrum analysis³, and the conventional nonlinear least squares analysis are listed in the following table. It should be noted that our new results are consistent with the null point spectrum analysis and need no subjective initial guess.

Method	$r_1, r_2 \text{ (s}^{-1}\text{)}$	$s_1, s_2 \text{ (s}^{-1}\text{)}$
our work	2.560, 4.848	3.661, 6.150
null point spectrum analysis	2.173, 4.824	3.498, 6.479
nonlinear least squares analysis	2.339, 4.446	3.144, 5.390

We are now trying to extend this method to more accurate quantification of NMR spectral parameters. Further modification and application shall be reserved for a later occasion.

References

1. N. J. Clayden and B. D. Hesler, *J. Magn. Reson.* **98**, 271 (1992). (and references therein)
2. R. Kumaresan and D. W. Tufts, *IEEE Trans. ASSP-30*, 833 (1982).
3. (a) W. S. Price, N. H. Ge, L. Z. Hong, and L. P. Hwang, *J. Am. Chem. Soc.* (in press).
 (b) N. H. Ge, W. S. Price, L. Z. Hong, and L. P. Hwang, *J. Magn. Reson.*, **97**, 656 (1992).
 (c) W. S. Price, N. H. Ge, and L. P. Hwang, *J. Magn. Reson.*, **98**, 134 (1992).
 (d) T. S. Lee and L. P. Hwang, *J. Magn. Reson.*, **89**, 51 (1990).

Sincerely,

Yung-Ya Lin

Yung-Ya Lin

Nien-Hui Ge

Nien-Hui Ge

Lian-Pin Hwang

Lian-Pin Hwang

Fig. A: Our Method

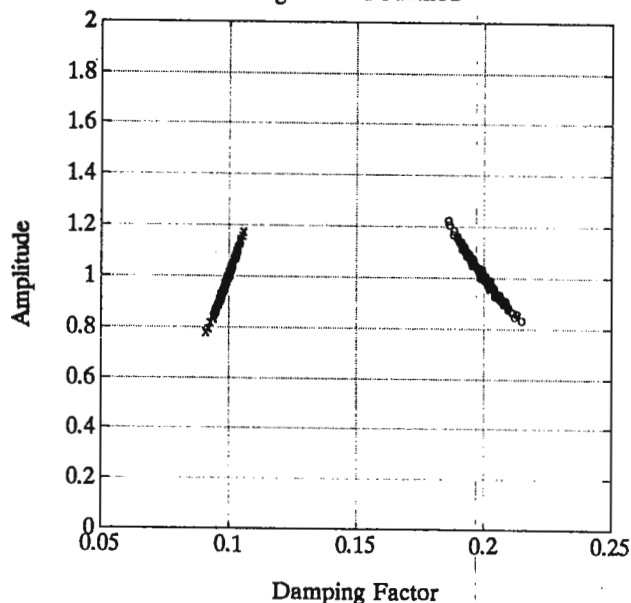
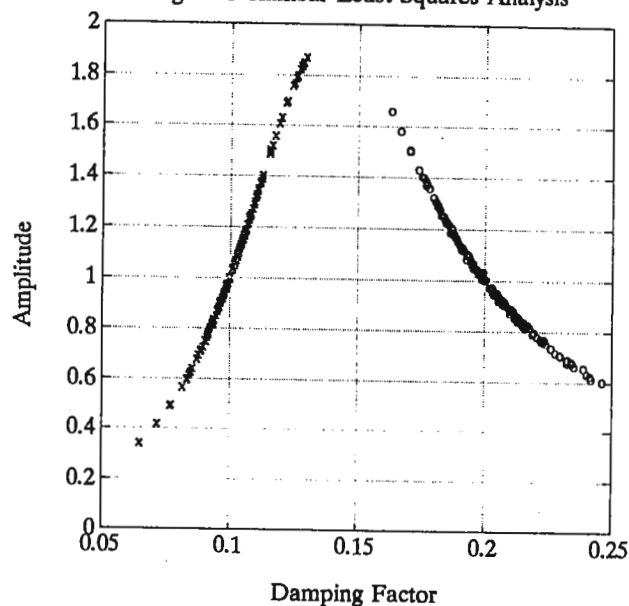


Fig. B: Nonlinear Least Squares Analysis



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PTS 160			
Range: 0.1-160 MHz Resolution: 0.1Hz-100KHz (opt) Switching: 1-20μs	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: 1-20μs	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 310			
Range: 0.1-310 MHz Resolution: 1Hz Switching: 1-20μs Phase Continuous: 1Hz-100KHz steps	Output: +3 to +13dBm; 50ohm Spurious Outputs: Type 1 -70/65 (typ/spec) Phase Noise: -68dBc (0.5Hz-15KHz)	Type 2 -65/60dBc -63dBc	Freq. St'd: OCXO, TCXO, Ext. Interface: BCD par. or GPIB Price: Type 1 \$6,175.00* Type 2 \$5,625.00*
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Range: 1-500 MHz Resolution: 0.1Hz-100KHz (opt) Switching: 1-20μs	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: 1-20μs	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: \$9,255.00*	
PTS 1000			
Range: 0.1-1000 MHz Resolution: 0.1Hz-100KHz (opt) Switching: 5-10μs	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: \$11,375.00*	
PTS x10			
Range: 10 MHz band, selected decade 0.1-100 MHz Resolution: 1Hz Switching: 1-5μs 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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FACULTY POSITIONS IN MAGNETIC RESONANCE. The Department of Chemistry, University of Florida, invites applications for two tenure-track positions which are available in connection with the National High Magnetic Field Laboratory. The positions will be filled at any level, depending upon the qualifications of the successful candidates, who will be expected to conduct innovative programs of research which will emphasize the high-field capabilities of the instrumentation being developed at the NHMFL. Researchers in NMR will have access to existing new Varian Unity 500 MHz and 600 MHz spectrometers in the Department of Chemistry and the University of Florida Health Center, respectively. Additional support will be provided through the NHMFL facilities, including use of magnets and spectrometers operating at 17 tesla and higher fields as they are developed. In addition, attractive startup packages are associated with the positions. Applicants should have extensive experience at the post-doctoral or more advanced levels in NMR or EPR spectroscopy, and skill and interest in developing and expanding experimental techniques. Collaborative research with investigators in fields such as the study of biological macromolecules, polymers, or inorganic solids will be an important aspect of the two positions. The Department of Chemistry of the University of Florida is a leading chemistry department with a faculty of 50 including an established group of senior investigators as well as a number of well-qualified young scientists. There are 80 post-doctorals, 200 graduate students and 45 support personnel, and an average of 30 doctoral degrees are awarded annually. Computer, X-ray, mass spectrometer and EPR facilities are excellent.

Applications should be submitted by February 1, 1993. Applicants should include names of three or more references and will facilitate consideration by requesting references to send supporting letters. All correspondence should be sent to Wallace S. Brey, Chair, Search Committee, Department of Chemistry, University of Florida, Gainesville FL, 32611-2046. The University of Florida is an Affirmative Action-Equal Opportunity employer.

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Abbott Laboratories is a global leader in the development of advanced health care products and services. Abbott is an Affirmative Action Employer and Smoke-Free Environment. For consideration, please send your resume to: **Dr. Merrill Nuss, D418, Abbott Laboratories, Abbott Park, IL 60064.**

The National Institute on Aging of the National Institutes of Health wishes to recruit a Ph.D. level NMR spectroscopist for the In-vivo NMR Unit. Present research activities include studies of peripheral muscle metabolism in humans and experimental animals, and methodologic studies not directly related to metabolism. Current instrumentation is a Bruker Biospec 1.9T/31 cm horizontal bore spectrometer, which will be upgraded to a Bruker AMX system in the Spring of 1993. A wide-bore Bruker AMX 400 system is also on order for delivery this coming Spring. In addition, the NMR facilities of the main NIH campus in Bethesda, as well as instrumentation at the nearby Johns Hopkins Medical School, are available. The appointment will be as an IRTA Postdoctoral Fellow or Staff Fellow for US citizens, and as a Visiting Fellow or Visiting Associate for non-citizens, depending on experience and qualifications. In-vivo experience or a background in biochemistry/physiology is preferred but not required. Interested individuals should send a curriculum vitae and bibliography to: Dr. Richard Spencer, NIH/NIA, GRC 4D-13, 4940 Eastern Avenue, Baltimore, MD 21224, or telephone directly at 410-558-8226.



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* * * * *

All Newsletter Correspondence

Should Be Addressed To:

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Palo Alto, CA 94303, U.S.A.
(415) 493-5971

DEADLINE DATES

No. 414 (March)-----19 February 1993
No. 415 (April) -----19 March 1993
No. 416 (May) -----23 April 1993
No. 417 (June)-----21 May 1993

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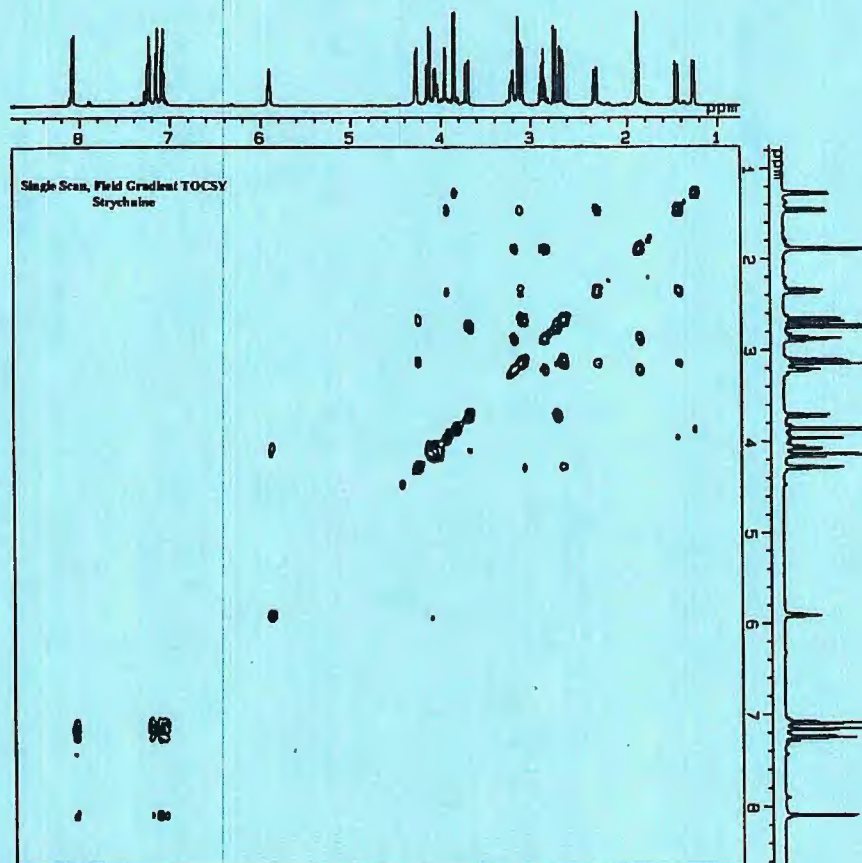
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