THE NMR NEWSLETTER

No. 460

January 1997

Cosine Division . . . . . **Bothner-By, A. A.** 2

SAR by NMR: All Ligands Are Not Created Equal . **Petros, A., Hajduk, P.,** and **Fesik, S. W.** 5

MAS ^{1}H-^{13}C-HMQC Experiment on Lipids . . . **Huster, D.,** and **Gawrisch, K.** 7

Application of Double-Filtered NMR Spectral Analysis Including Exchange for Studying Benzene in USY Zeolite . . . **Chang, W.-T., Chen, Y.-H., Jiang, B.-C.,** and **Hwang, L.-P.** 11

Effect of 2,4-Pentanedione on ^{29}Si and ^{27}Al MAS NMR Spectra of Sol-Gel Derived Aluminosilicates . . . **Miller, J. M.,** and **Wails, D.** 15

Yb(EDDS)$(D_2O)_n^-$, a Water-Soluble Chiral Chemical Shift Reagent . **Minch, M.,** and **Zhao, J.** 17

Position Available . . . . . **Spencer, R. G. S.** 18

Sofware Review . . . . . **Smith, W. B.** 21

Non-Invasive Technique . . . . . **Croquemort, Le** 22

A WISE Approach to Heterogeneous Biopolymer Mixtures: Dynamics and Domains in Wounded Potato Tissues . . . . . **Stark, R. E.,** and **Yan, B.** 25

Automated Sample Preparation with the Bruker BASP . . **Paschal, J. W.,** and **Spangle, L. A.** 29

International School of Structural Biology and Magnetic Resonance, 3rd Course: Protein Dynamics, Function and Design; Erice, Sicily, Italy; 16-28 April 1997 . **Jardetzky, O.,** and **Lefèvre, J.-F.** 30

Position Available . . . . . **Attard, J.** and **Bjørseth, H.** 33

Position Available . . . . . **Jakobsen, H. J.** 34

Unusual Bonding Mode for Chiral BIPHEP Ligands . . . . **Pregosin, P. S.** 37

High Field NMR Spectroscopy - Is It Always the Best Choice? . **Vigouroux, C.,** and **Vottéro, P.** 38

Two Different Ways to Cancel Relaxation Artifacts in 2D INADEQUATE by Using Field Gradients . . . **Bourdonneau, M.,** and **Brevard, C.** 39

1997 and 1998 ENC Dates and Locations . . . . . **Roberts, J. E.** 41

5th Annual "Advances in NMR Applications" Symposium . . **Tierney, C./Nalorac** 42

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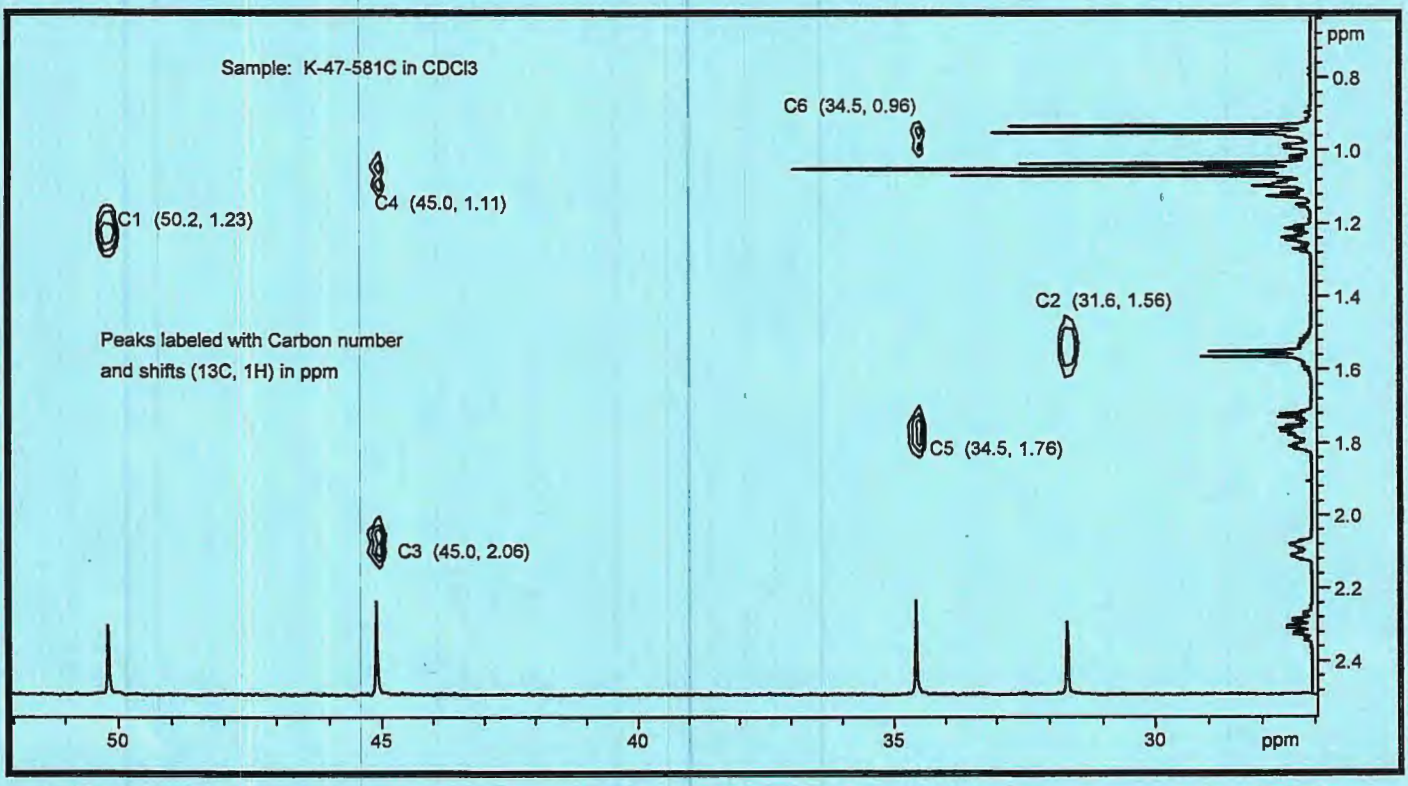

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Attard, J. . . . 33
Bjørseth, H. . . 33
Bothner-By, A. A. 2
Bourdonneau, M. 39
Brevard, C. . . 39
Chang, W.-T. . 11
Chen, Y.-H. . . 11
Croquemort, Le . 22
Fesik, S. W. . . 5
Gawrisch, K. . . 7
Hajduk, P. . . 5
Huster, D. . . 7
Hwang, L.-P. . . 11
Jakobsen, H. J. . 34
Jardetzky, O. . 30
Jiang, B.-C. . . 11
Lefèvre, J.-F. . . 30
Miller, J. M. . . 15
Minch, M. . . 17
Paschal, J. W. . 29
Petros, A. . . . 5
Pregosin, P. S. . 37
Roberts, J. E. . 41
Smith, W. B. . . 21
Spangle, L. A. . 29
Spencer, R. G. S. 18
Stark, R. E. . . 25
Nalorac . . . 42
Vigouroux, C. . 38
Vottéro, P. . . 38
Wails, D. . . . 15
Yan, B. . . . 25
Zhao, J. . . . 17

Acorn NMR, Inc. . . . . inside front cover
AMT 23
Bruker Instruments, Inc. 9, 27
Broad Band Technology 35
JEOL . . . . . outside back cover
Otsuka Electronics 13
Oxford Instruments, Ltd. 19
Varian NMR Instruments 3
Wilmad Glass Company, Inc. 31

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FORTHCOMING NMR MEETINGS

5th Annual "Advances in NMR Applications" Symposium, Orlando, FL, **March 23, 1997**; Contact: Ms. Chris Tierney, Nalorac, 841-A Arnold Drive, Martinez, CA 94553;)510) 229-3501; Fax: (510) 229-1651; Email: christierney@nalorac.com. See Newsletter 460, 42.

38th ENC (Experimental NMR Conference), Orlando, FL, **March 23 - 27, 1997**; Contact: ENC, 1201 Don Diego Avenue, Santa Fe, NM 87505; (505) 989-4573; Fax: (505) 989-1073. See Newsletter 460, 41.

International Society for Magnetic Resonance in Medicine, Fifth Scientific Meeting and Exhibition, Vancouver, BC, Canada, **April 12-18, 1997**; Contact: ISMRM, 2118 Milvia St., Suite 201, Berkeley, CA 94704, USA; (510) 841-1899; Fax (510) 841-2340; Email: info@ismrm.org.

Symposium on NMR Spectroscopy of Synthetic Macromolcules, ACS National Meeting, San Francisco, **April 13-17,** 1997; Contact: H. N. Cheng or English, A. D. See Newsletter 456, 20.

International School of Structural Biology and Magnetic Resonance, 3rd Course: Protein Dynamics, Function and Design; Erice, Sicily, Italy; **April 18-28, 1997**; Contact: Ms. Robin Holbrook, Stanford Magnetic Resonance Laboratory, Stanford University, Stanford, CA 94305-5055; (415) 723-6270; Fax: (415) 723-2253; Email: holbrook@smi.stanford.edu. See Newsletter 460, 30.

6th Meeting of AUREMN (NMR Users Association of Brazil), Rio de Janeiro, Brazil, **12 - 16 May, 1977**; Contact: Snia Maria C. de Menezes, Petrobás/Cenpes/Diquim/Radial 2, Quadra 07 - Ilha do Fundão, 21949-900 Rio de Janeiro, Brazil; Tel. +55 21 598-6171 and 598-6914; Fax. +55 21 598-6296; Email; sonia@cenpes.petrobas.gov.br.

Continued on page 16

Aksel A Bothner-By
6317 Darlington Rd.
Pittsburgh, PA 15217

7 December 1996
(received 12/12/96)

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

COSINE DIVISION

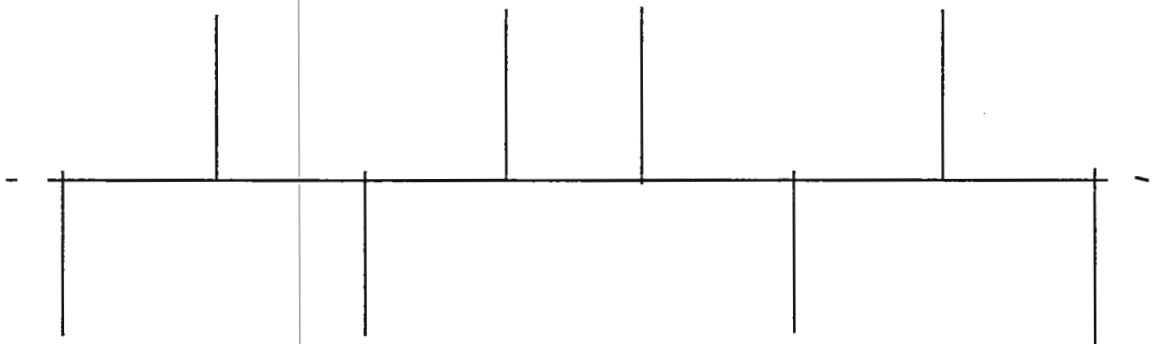
Dear Barry,

OK, here it is (slavedriver!). A few years ago, Joe and I developed a method for determining doublet splittings accurately. It involved dividing the fid by $\cos(\pi Jt/2)$. Since the fid of a doublet consisting of two identical lines is $Ae^{-i\omega t}\cos(\pi Jt/2)S$, with A the amplitude, ω the chemical shift, J the splitting, and S a shape factor, the division simply removes the splitting, and Fourier transformation yields a spectrum consisting of one line of the same shape as the original two. If the trial value chosen for J is not equal to the actual splitting, Fourier transformation gives a spectrum with a repetitive pattern of interference peaks in the base line and a broadened and distorted peak.

There are two troubles with this method. (1) Division by $\cos(\pi Jt/2)$ gives spikes as a result of dividing noise by very small numbers, whenever πJt is close to π. (2) The shapes and amplitudes of the two lines are often not the same, producing unwanted noncancelled signals in the base line of the transformed spectrum.

I have found that it is possible to overcome both of these difficulties in the following way. First shift the doublet to the center of the spectrum, which can be done by multiplication by $e^{i\omega t}$. It is not important to have the exact value of ω but it is more convenient if the error is less than the linewidth. The fid is now $A\cos(\pi Jt/2)S$. The information about the different line shapes and amplitudes is in the S function, imaginary part, and this is the only complex component of the fid. Zero it. We now have a completely real fid, yielding a centered symmetrical doublet with splitting equal to J.

Finally, instead of dividing by $\cos(\pi Jt/2)$, multiply by $\cos(\pi Jt/2) - \cos(3\pi Jt/2) + \cos(5\pi Jt/2) - \cos(7\pi Jt/2)$ ... These are the first terms of a series expansion of $1/\cos(\pi Jt/2)$. The Fourier transform of this series looks like this:



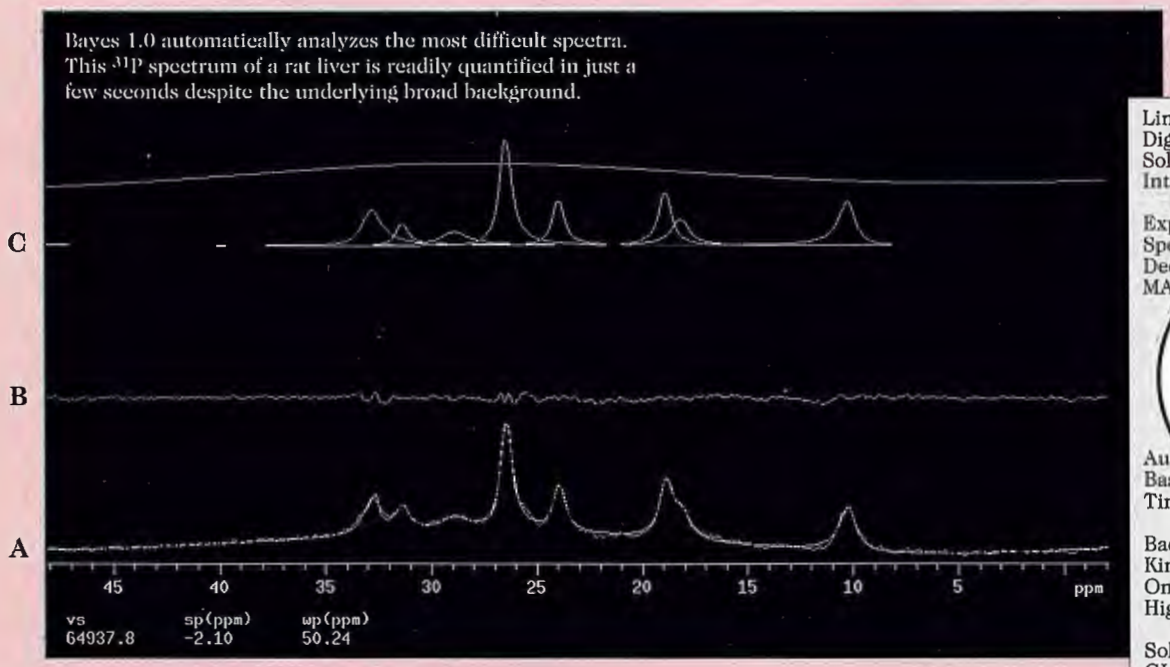
The convolution of this function with the transformed doublet spectrum is what you get. If there are infinitely many terms in the series expansion, the peak you see results from the convolution of the doublet and the central two lines above. Somewhere out at infinity there are also uncancelled signals. If you limit the number of terms in the series expansion so that the residual uncancelled terms are close to the right and left spectral limits, you will now get a spectrum which looks like this:

(Well, with some noise, of course) Now one can apply whatever criterion one wishes to the central peak and baseline to guide the choice of the best value for J.

Best wishes to you and Lee for the Holidays and New Year. Long live the Newsletter.

Der Alte *Aksel*

Results You Can Believe In



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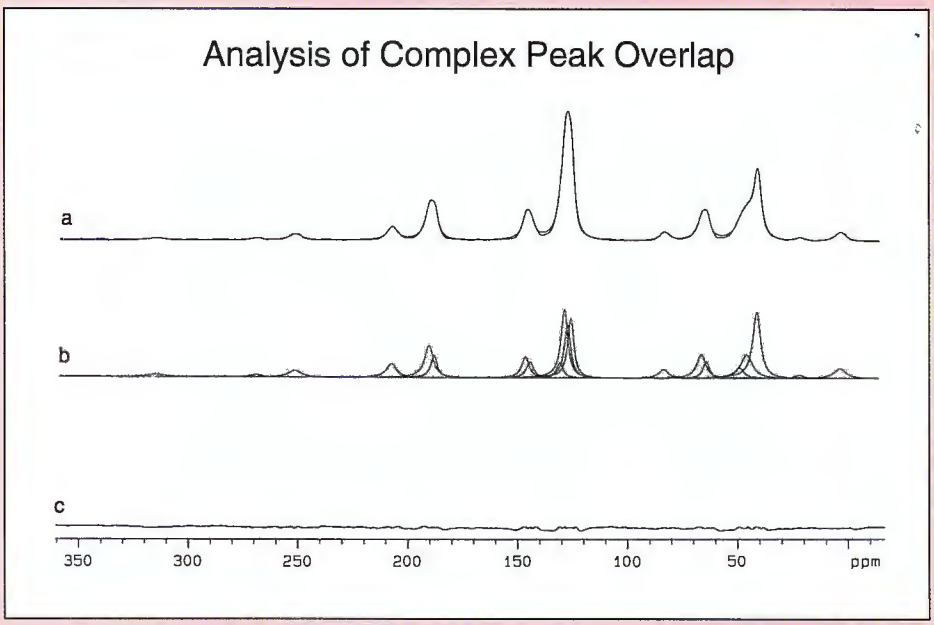
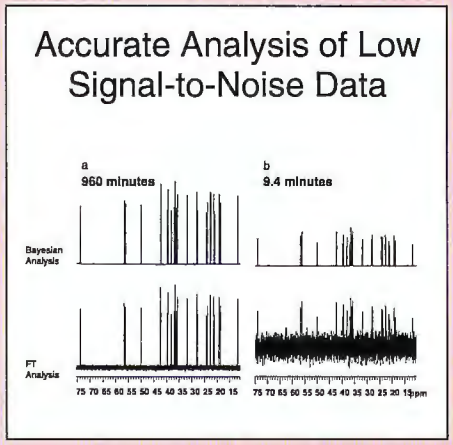
Bayes 1.0 Applications

I.
a. Overlay of Bayesian-modeled and experimental ^{13}C CP/MAS spectra of polystyrene, b. Bayesian analysis of the FID of polystyrene, showing individual modeled resonances in the frequency domain, c. Difference spectrum from subtraction of FT spectrum from FT of Bayesian-modeled spectrum.



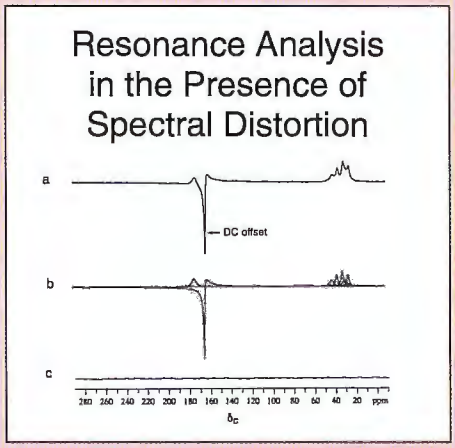
I

II.
a. ^{13}C FT spectrum of cholesterol, 8192 transients, total experiment time 960 minutes (bottom) and Bayesian-modeled spectrum (top), b. ^{13}C FT spectrum of cholesterol, 80 transients, total experiment time 9.4 minutes (bottom) and Bayesian-modeled spectrum (top), demonstrating the accuracy of Bayesian analysis even under low signal-to-noise conditions.



II

III.
a. Overlay of Bayesian-modeled and experimental 32 MHz ^{13}C CP/MAS spectra of Nylon 6,6, b. Bayesian analysis of the FID of Nylon 6,6, showing individual modeled resonances in the frequency domain, and demonstrating accurate modeling of resonances in the presence of a strong DC offset, c. Difference spectrum from subtraction of FT spectrum from FT of Bayesian-modeled spectrum.



III

Spectra provided courtesy of Dr. W. C. Hutton, Monsanto, Co.

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December 13, 1996
(received 12/18/96)

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

RE: SAR by NMR: All ligands are not created equal

Dear Barry:

Recently, we described a method for discovering high affinity ligands that bind to proteins (*Science*, **274**, 1531-1534, 1996). Using this method dubbed "SAR by NMR", small molecules that bind to proximal subsites of a protein are identified by observing changes in the amide chemical shifts of an ^{15}N-labeled protein upon the addition of a potential ligand. The two molecules are then linked together guided by NMR-derived structural information on how the untethered ligands bind to the protein. The method reduces the amount of chemical synthesis and time required for the discovery of high affinity ligands and when applied to protein drug targets can be a useful tool in drug research.

An important part of the SAR by NMR method is the structure determination of the untethered ligands when bound to the protein. This information is critical for designing the linker length and sites of attachment. Unfortunately, not all ligands are created equal. Some give broad lines due to exchange broadening and some exhibit time-dependent changes in the NMR spectra. Figure 1A depicts a proton NMR spectrum of a well-behaved ligand in D_2O. In contrast, the signals corresponding to a different ligand (Figure 1B) disappear with time, but the solution remains clear. Thus, the choice of ligands for detailed NMR studies is based on many criteria besides the binding affinity for the protein.

Sincerely,

Andrew Petros Phil Hajduk Stephen Fesik

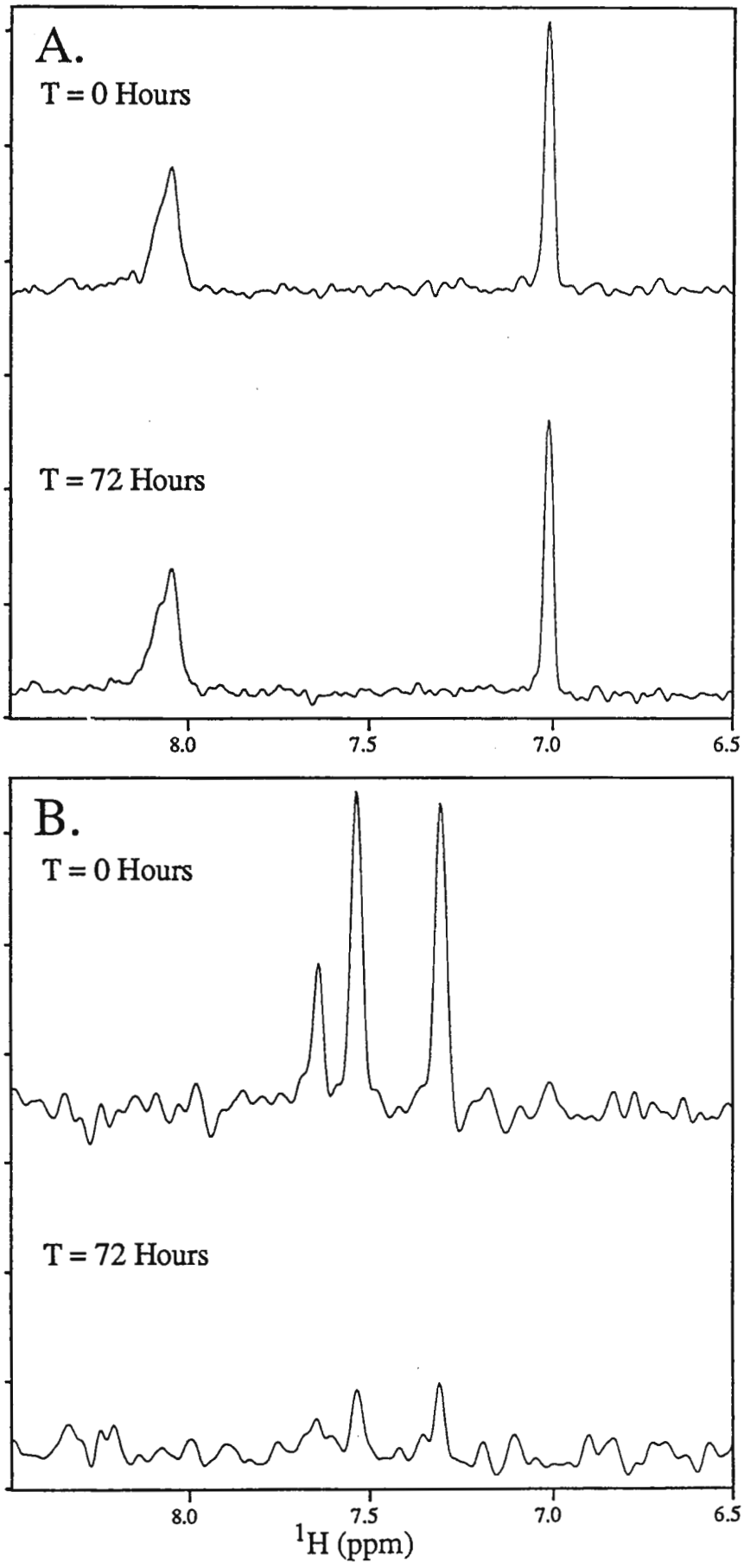

Fig. 1 Time dependence of the ^{1}H NMR signals for two ligands

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MAS ^{1}H-^{13}C-HMQC experiment on lipids

December 11, 1996
(received 12/15/96)

Dear Dr. Shapiro,

We are investigating biophysical properties of phospholipid membranes using a variety of solid-state NMR methods. Recently we encountered difficulty assigning the lipid headgroup signals in ^{1}H MAS NMR spectra of ternary lipid mixtures. Our 500 MHz MAS ^{1}H NMR spectra have a typical linewidth of 20 Hz but signal assignment is still difficult because of resonance superposition. In contrast, the proton decoupled ^{13}C NMR spectrum has no superimposed resonances and assignment was easy.

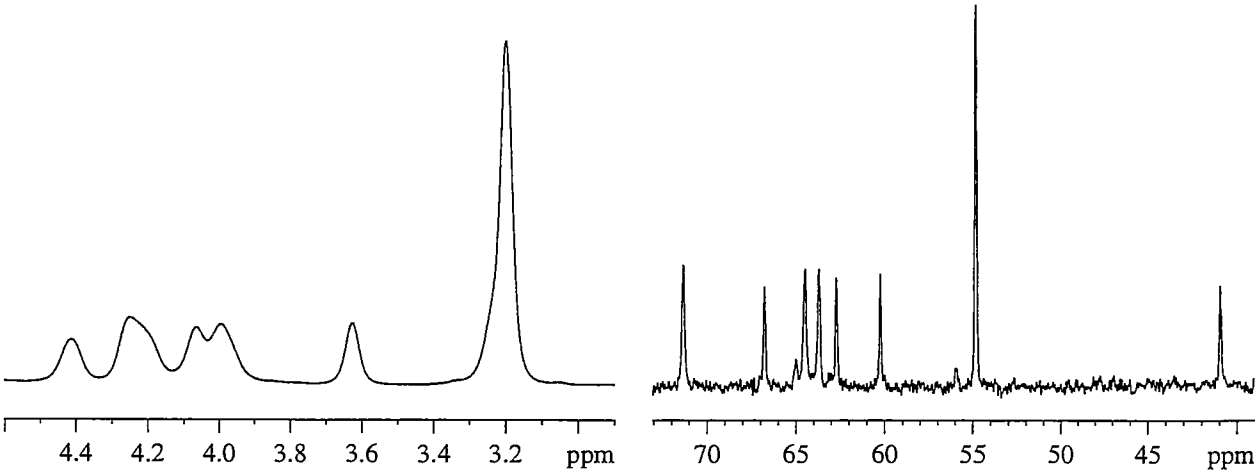


Figure 1. ^{1}H and ^{13}C MAS NMR spectra of a PC/PE/PS (4/4/1, mol/mol/mol) dispersion in D_2O. Approximately 4 mg of lipid were investigated, using a 4 mm rotor with sample insert spinning at 5 kHz.

In high resolution NMR, ^{1}H signal assignment of the lipid headgroup protons could be achieved with an inverse heteronuclear quantum coherence experiment [1]. The question arises, do these experiments also work with phospholipids in the liquid-crystalline phase under conditions of MAS? Griffin's laboratory demonstrated recently that solution-style transfer schemes such a INEPT and TOCSY may be employed without modification provided that the sample rotation frequency is greater than the motionally averaged CSA and dipolar couplings [2].

Here we report results of a ^{1}H -^{13}C HMQC experiment for a lipid mixture of phosphatidylcholine, -ethanolamine, and -serine (4/4/1, mol/mol/mol) at natural abundance ^{13}C. Strong proton resonances of nuclei not coupled to ^{13}C were suppressed with a BIRD sequence. Fig. 2 shows the proton - carbon chemical shift correlation of the lipid headgroup signals. All cross-peaks are well resolved and t_1-noise artifacts are small. Although it is recommended for

high resolution NMR to keep the delay time between acquisitions short (approximately 1.3 * T_1) we observed a significant reduction of spectral artifacts for longer repetition times near 4 * T_1. Cross-peak signal intensity is heavily weighted by differences in T_2 relaxation. Signals from the lipid glycerol and upper hydrocarbon chain regions have shorter T_2 relaxation times and are much lower in intensity.

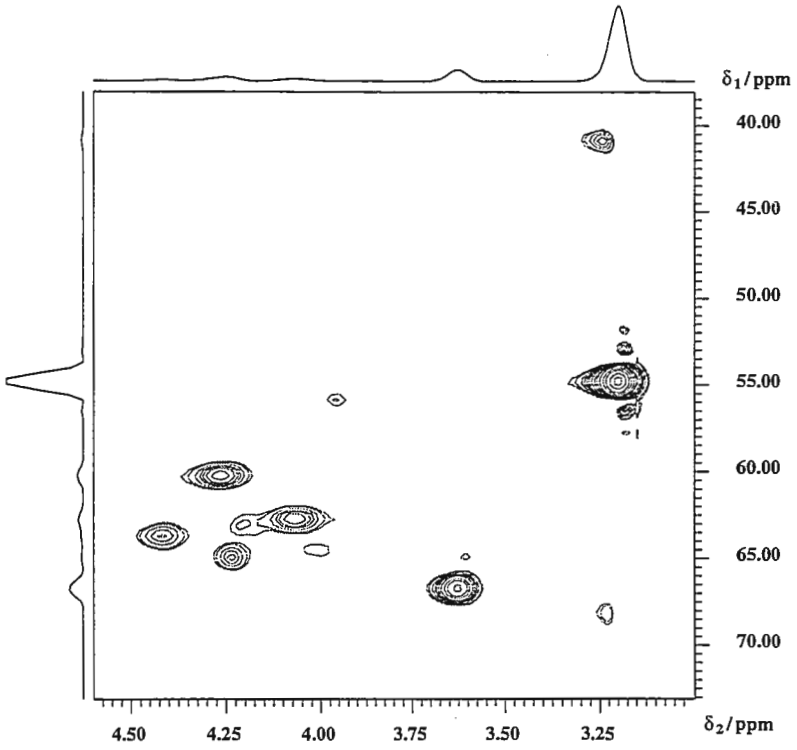


Figure 2. Headgroup signal region of the ^1H -^{13}C- MAS-HMQC spectrum. Bruker DMX500 spectrometer, widebore magnet, triple resonance 4 mm MAS probe, D_2O deuterium lock, 4 mg of PC/PE/PS mixture, temperature 40 °C, resolution: 512 acquired data points in ^1H and 256 points in ^{13}C dimensions, 64 scans per t_1-increment with a relaxation delay of 4 s, total acquisition time approximately 21 hours.

In combination with magic angle spinning the heteronuclear single and multiple quantum coherence experiments may become an important building block for multidimensional NMR experiments on biomembranes.

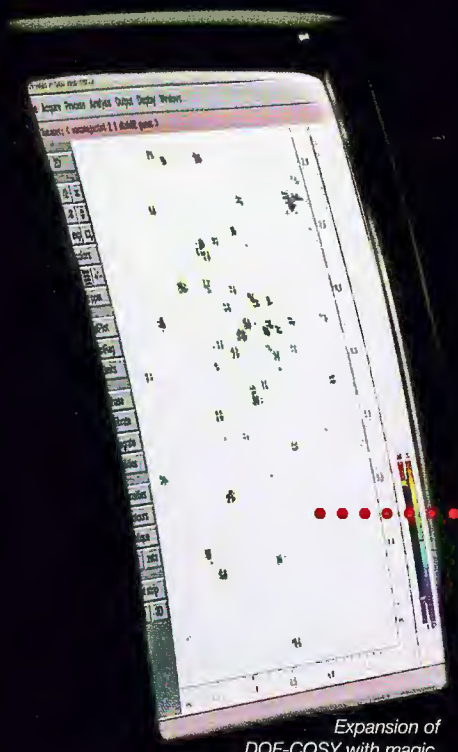
Very sincerely,

Daniel Huster

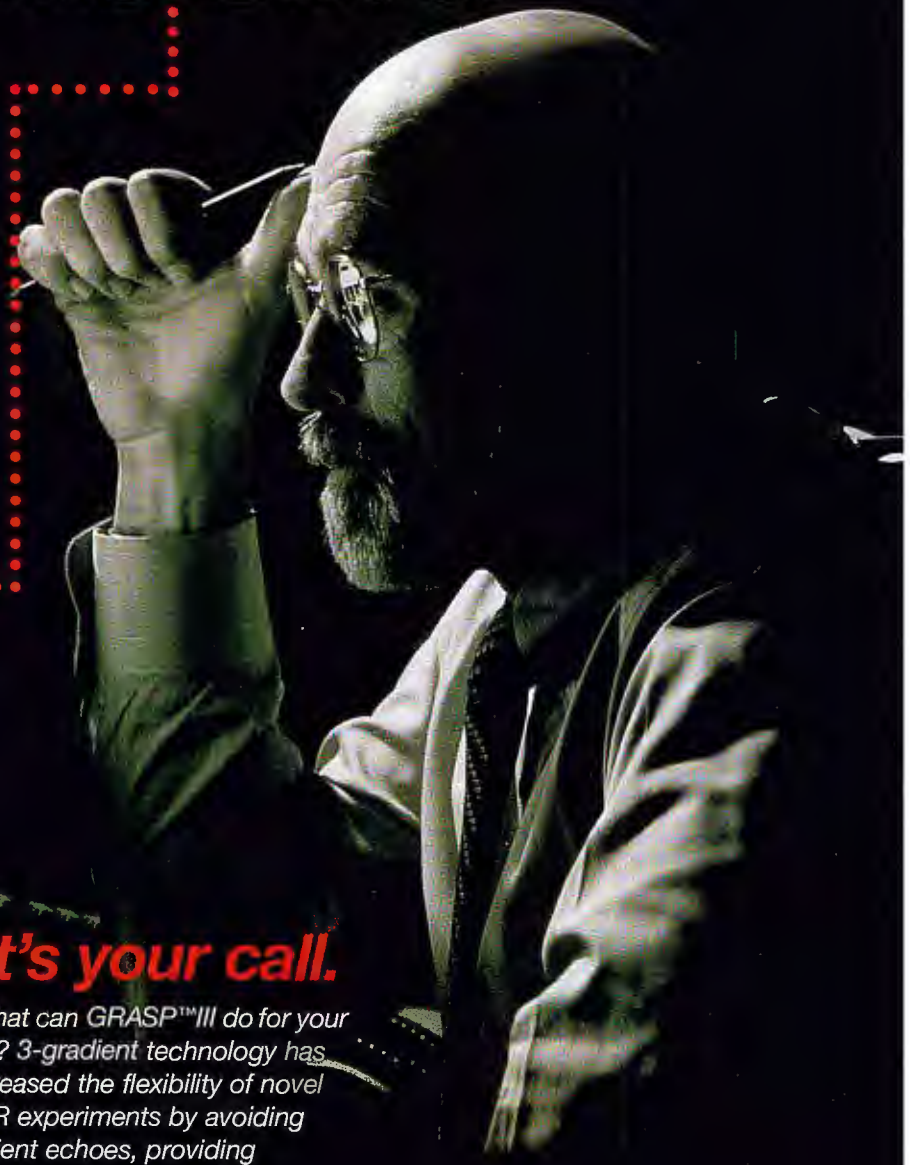
Klaus Gawrisch

[1] A. Bax & S. Subramanian, *J. Magn. Reson.*, **67**, 565-569 (1986)
[2] J.D. Gross, P.R. Costa, J.-P. Dubacq, D.E. Warschawski, P.-N. Lirsac, P.F. Devaux & R.G. Griffin, *J. Magn. Reson.*, **106**, 187-190 (1995)

Take a hard look at the difference.



Expansion of DQF-COSY with magic angle gradient, 1.5 mM BPTI in 90% H_2O/10% D_2O.



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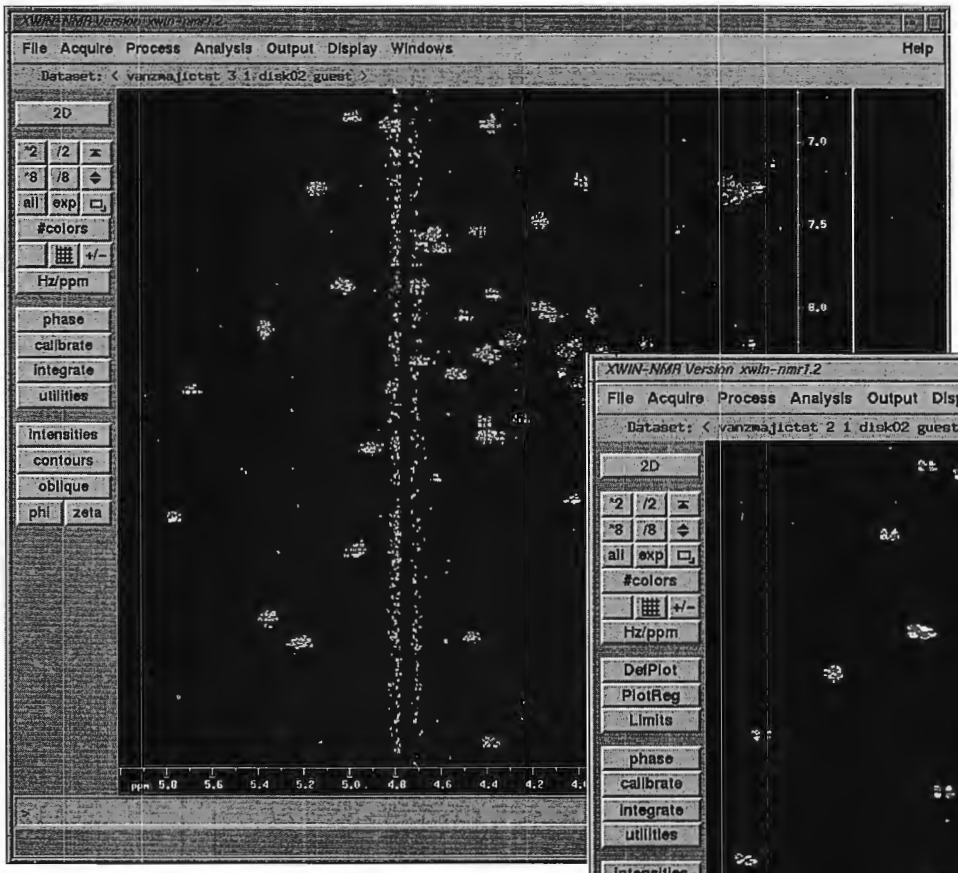
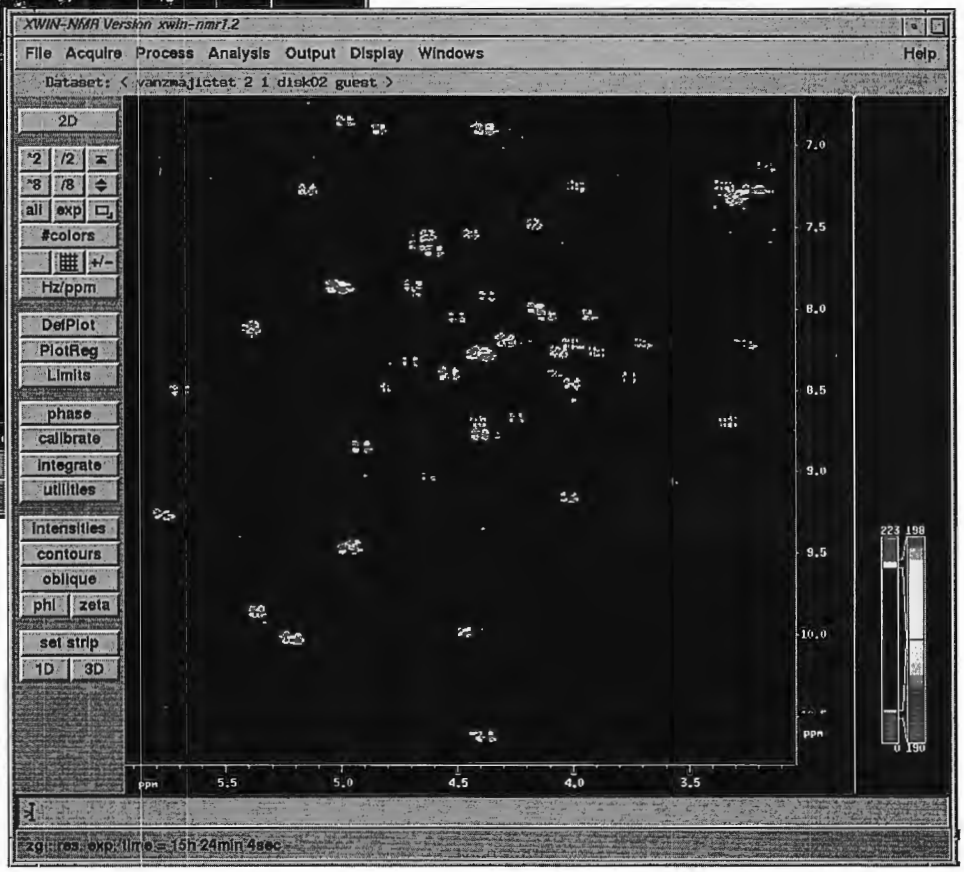


Results with the z-gradient only... residual water ridge is clearly visible and overlaps crosspeaks of interest.

Using the Bruker GRAdient SPectroscopy III (GRASPTM-III) accessory with x,y,z-gradients, an effective gradient at the magic angle can be easily produced by applying three gradients simultaneously. This greatly improves the elimination of residual water by coherence selection in multiple-quantum filtered COSY experiments.

Both experiments were acquired on the Bruker AVANCE 500 equipped with a 5 mm inverse triple resonance (TXI) probe with GRASPTM-III. The sample is 1.5 mM BPTI in 90% H_2O/10% D_2O. The experiment is DQF-COSY.



Results with magic angle gradient... residual water ridge is eliminated! Crosspeaks previously overlapped by the water can be observed and used for correlation assignment. The elimination of the water signal is achieved by coherence selection. No presaturation is used in either experiment!

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November 29, 1996
(received 12/9/96)

Re: **Appllication of Double-Filtered NMR Spectral Analysis Including Exchange for Studying Benzene in USY Zeolite**

Dear Dr. Shapiro,

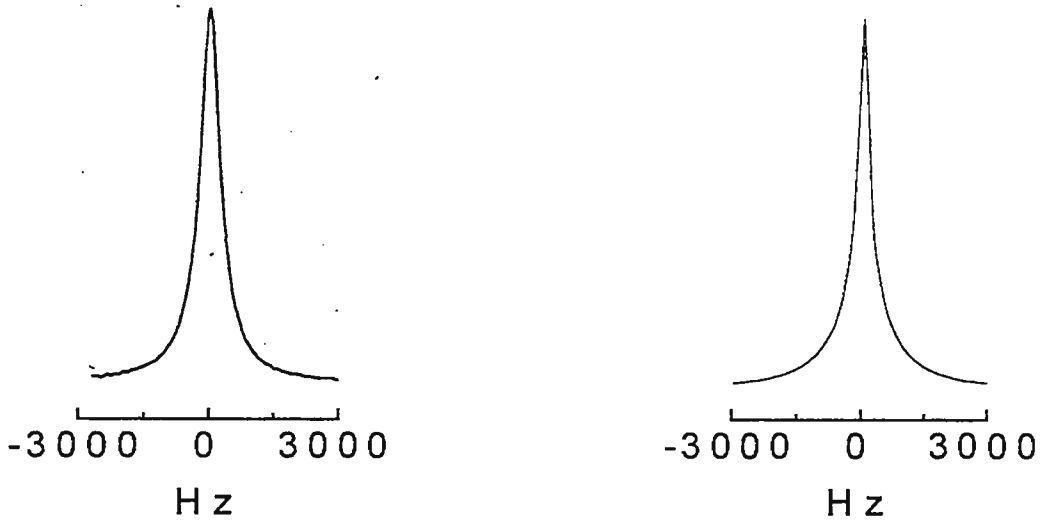
There has been great interest in the study of adsorption process in zeolites by using NMR relaxation techniques. However many difficulties remain in these studies. It seems completely unphysical to assume isotropic motion of the sorbate molecule while remaining attached to the adsorption site, but it is observed that the collapse of the solid state pattern for quadurpolar system into singlet at elevate temperature. Based on this observation, the residual interaction on adsorption is considered to be averaged out with the isotropic motion during exchange process about the adsorption sites.

Recently the development of double quantum filtered (DQF) NMR spectroscopy is a diagnostic tool for the detection of anisotropy in macroscopically disordered system [1,2] . It is a very sensitive method for the determination of the residual quadurpolar interaction resulting from the local order. For I=1 spin system no DQ coherence can be detected in isotropic medium and, hence, the observation of the DQF spectra indicates the presence of anisotropic motion of spin-bearing molecules. The relaxation of DQF spectra with respect to the creation period may be applied the study of dynamic and exchange process in zeolite system. We employ the DQF technique to the study of absorption of benzene-d_6 in the USY zeolite. ^2D single quantum and DQF spectra are shown in the figure 1. The corresponding simulated spectra are also shown for comparison.

Sincerely yours,

Wen-Tsung Chang Yu-Huei Chen Bang-Chih Jiang Lian-Pin Hwang

Wen-Tsung Chang Yu-Huei Chen Bang-Chih Jiang Lian-Pin Hwang



(a)

(b)

Figure 1. (a) Single quantum ^2D NMR spectrum of benzene-d_6 adsorbed in the USY zeolite at 250 K ; (b) ^2D DQF spectral corresponding to the creation period 40, 80 , 160, 240 , and 560 μs , respectively. The spectral width is ± 41667 Hz.

1. U. Eliav, H. Shinar, and G. Navon, *J. Magn. Reson.* **98** , 233(1992)
2.Y. Sharf, U. Eliav, H. Shinar, and G. Navon, *J. Magn. Reson.* **B107**, 60(1995)

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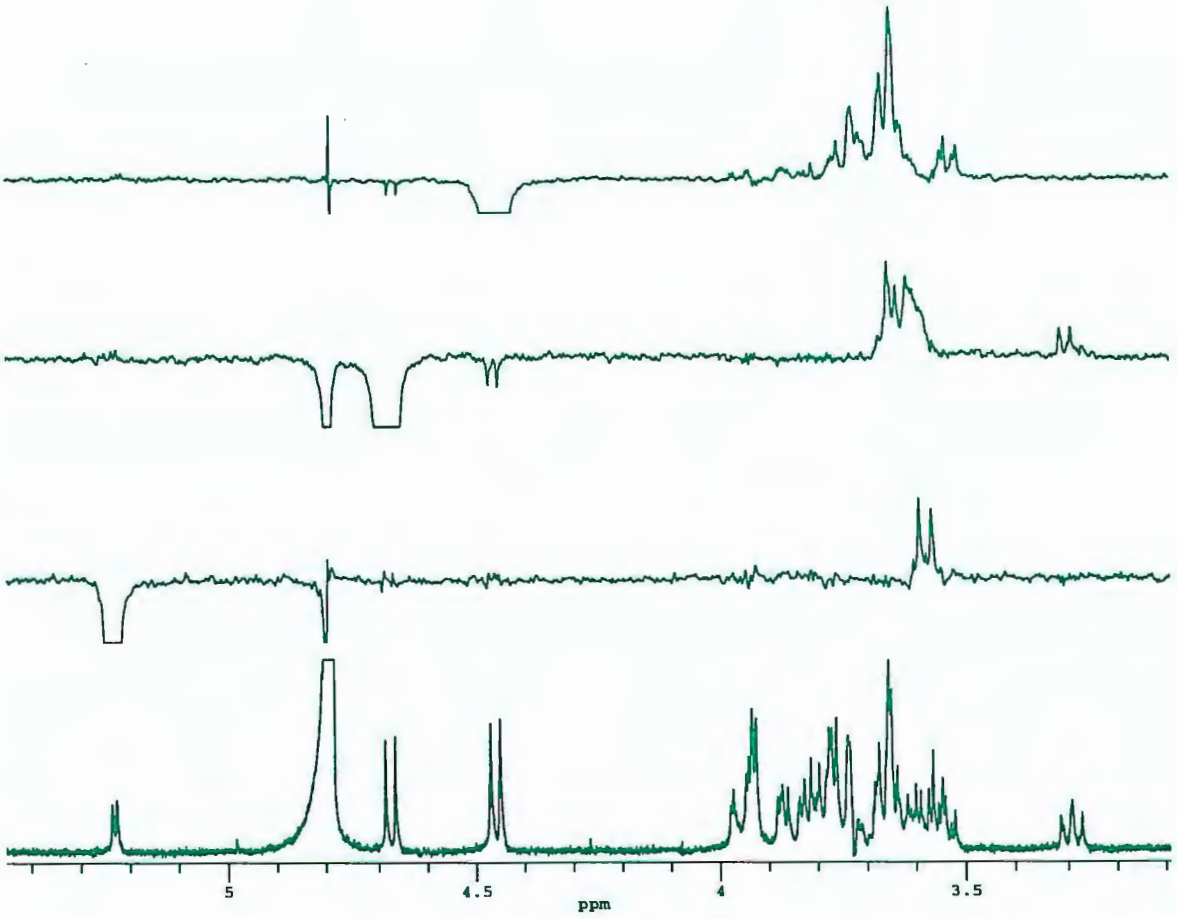

This spectrum was collected in 128 acquisitions

NOE DIFFERENCE SPECTRA OF LACTOSE

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NOe difference spectra of 1.0 mM lactose in D_2O are shown here. The bottom trace is the normal ^1H spectrum acquired in a single acquisition. Upper traces are the nOe spectra acquired with presaturation of different resonances. Note the excellent cancellation of unperturbed peaks.

Data were acquired on a CMX Infinity 400 MHz spectrometer equipped with a Nalorac™ 5 mm indirect detection triple resonance gradient probe. The nOe spectra were collected in 128 acquisitions.

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

Thu, Nov 28, 1996
(received 12/6/96)

Effect of 2,4-pentanedione on ^{29}Si and ^{27}Al MAS-NMR spectra of sol-gel derived aluminosilcates

Dear Barry,

Our MAS - NMR investigations continue using our Bruker DPX 300 spectrometer, which we have had for almost one year. In particular, we are investigating the physical properties of sol-gel derived aluminosilicates which we use as suportes for catalysts with applications in Friedel-crafts catalysis.

A recent report in the NMR newsletter (Gordon J. Kennedy, NMR Newsletter, 455-31.) discussed the effects of 2,4-pentanedione on the ^{29}Si MAS NMR spectra of highly silicious MCM-22. An observable effect on the silicon sites was reported. Differences in the NMR spectra upon sorbate addition have generally been attributed to some rearrangement of sites within the silicate framework. This was of interest to us since we are studying sol-gel derived aluminosilicate supports for Friedel-Crafts catalysts.

We therefore investigated the NMR spectra of our aluminosilicates, with and without adsorbed 2,4-pentanedione, with even more dramatic results than those previously reported. Figure 1 shows the effect of addition on the ^{27}Al MAS NMR spectra, obtained with a 30^{o} pulse, 250ms recycling delay and 10kHz spinning speed. The previously, observed framework (4 and 5 co-ordinate) and non-framework (6 co-ordinate) sites are replaced by a particularly sharp 6 co-ordinate species, suggesting complexation to the framework by 2,4-pentanedione.

Even more noticeable effects are observed in the ^{29}Si Cross-Polarized MAS NMR spectra (figure 2), obtained with 5ms contact time and a 5s recycling delay at 4kHz spinning speed. Previously, the amorphous nature of the materials gave rise to a broad, featureless peak, but on complexation, the band is resolved into three distinct peaks. We are still investigating the exact physical interactions responsible for these dramatic effects, and hope that T_1 relaxation studies to be carried out in the near future will clarify these issues.

Any suggested alternate interpretations would be appreciated.

Yours Sincerely,

Jack M. Miller
Professor of Chemistry.

David Wails

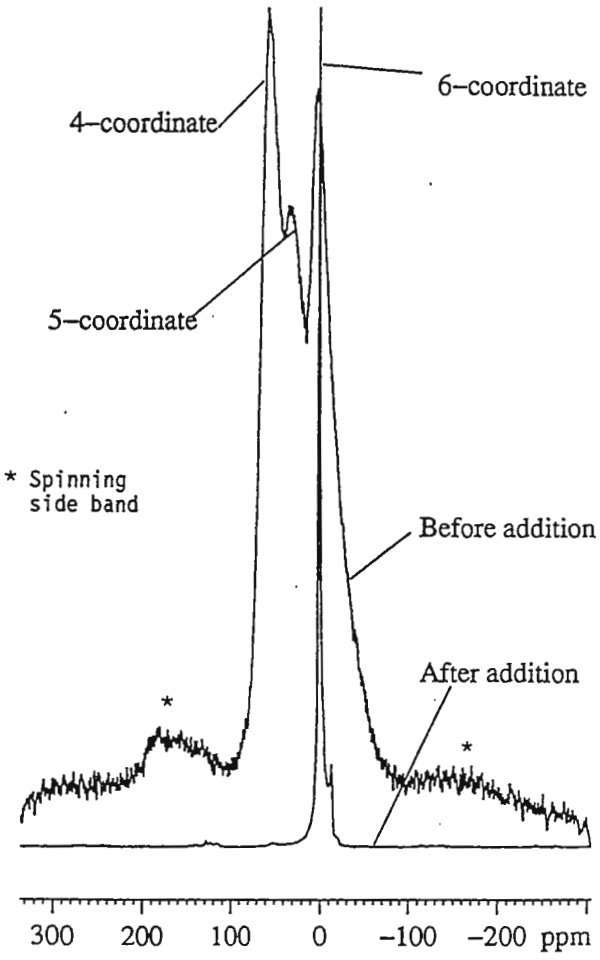


Figure 1 : ^{27}Al NMR spectra
before and after 2,4-pentanedione addition

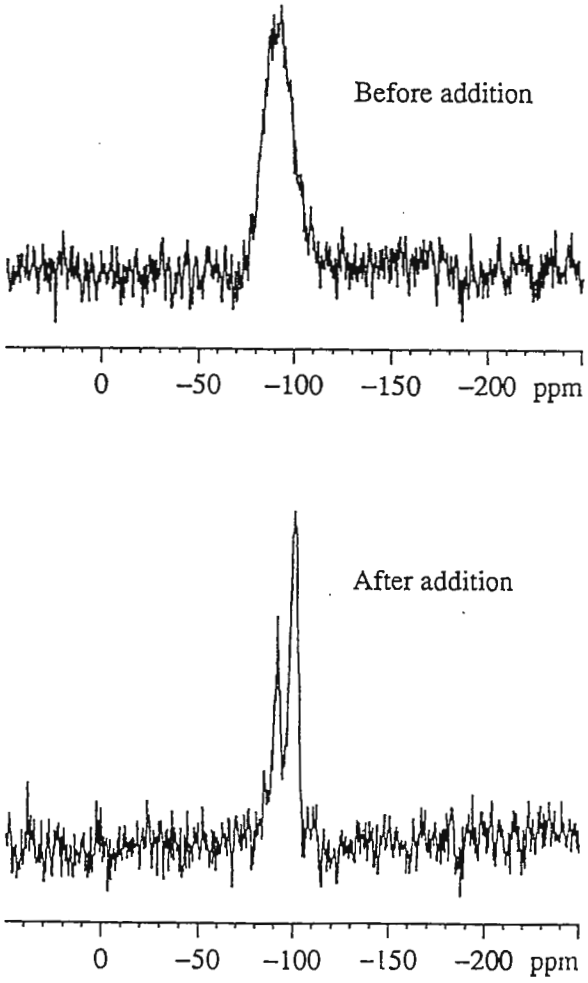


Figure 2 : ^{29}Si NMR spectra
before and after 2,4-pentanedione addition

Forthcoming NMR Meetings, continued from page 1:

39th Rocky Mountain Conference on Analytical Chemistry, Denver, Colorado; NMR Symposium, **August 4-7, 1997**: Contact: J. P. Yesinowski, Code 6120, Naval Research Laboratory, Washington, DC 20375-5342; 202-767-0415; fax 202-767-0594; email yesinowski@nrl.navy.mil. See Newsletter 458, 8.

4th International Conference on Magnetic Resonance Microscopy "Heidelberg Conference in Albuquerque", **Sept. 21-25, 1997**: Contact: E. Fukushima, The Lovelace Institutes, 2425 Ridgecrest Drive SE, Albuquerque, NM 87108-5127; (505) 262-7155; Fax: (505) 262-7043. See Newsletter 449, 37.

39th ENC (Experimental NMR Conference), Asilomar *[sic]* Conference Center, Pacific Grove, CA, **March 22 - 27, 1997**; Contact: ENC, 1201 Don Diego Avenue, Santa Fe, NM 87505; (505) 989-4573; Fax: (505) 989-1073. See Newsletter 460, 41.

Additional listings of meetings, etc., are invited.

UNIVERSITY OF THE PACIFIC

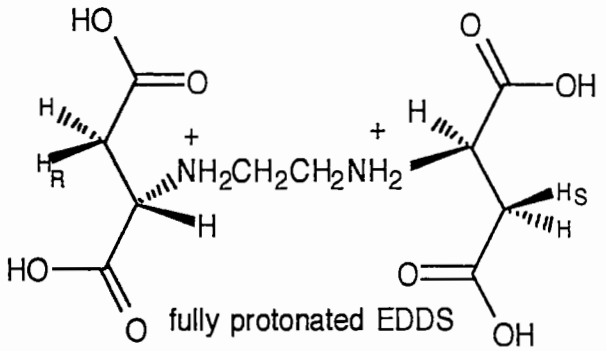
College of the Pacific

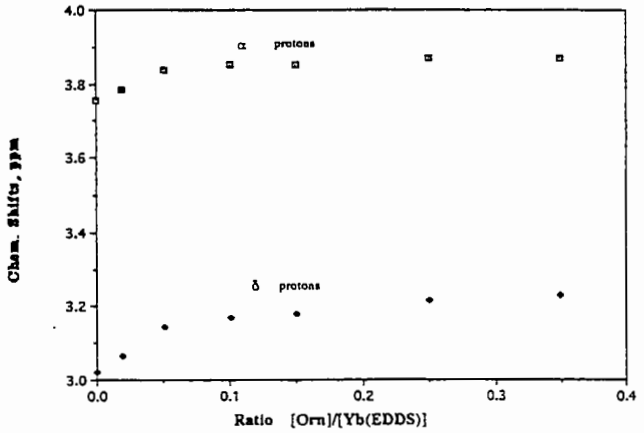
Department of Chemistry

$Yb(EDDS)(D_2O)_n^-$, a Water Soluble Chiral Chemical Shift Reagent.

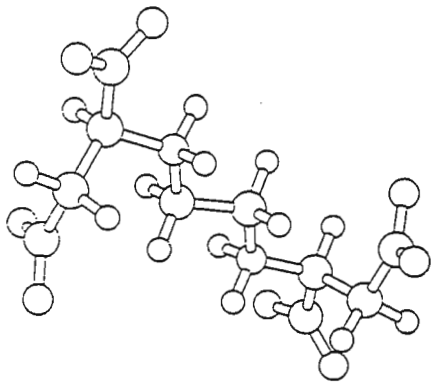
(received 12/20/96)

The europium complex of (S,S)-1, 2-ethylenediamine-N, N'-disuccinate (EDDS) is a chiral lanthanide shift reagent for aqueous solutions between pH 9 and 10.[1,2] The ligand is relatively easy to prepare[3] and we set out to investigate other lanthanide derivatives which might serve as chiral shift reagents (CSRs) at more convenient pH-levels. We found that the Yb^{+3} complex of EDDS gives large changes in chemical shift for amino acids even in neutral solutions (see graph of shifts induced for ornithine).

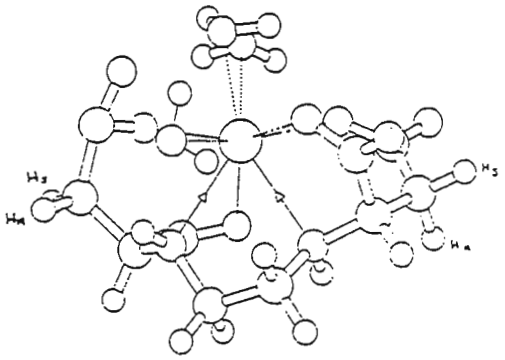




As part of this study we observed the ^1H NMR spectrum of the uncomplexed EDDS ligand as a function of pH from 3.08 to 12.61 in order to demonstrate that its titration curve shares properties with that of EDTA.[4] It does follow the same trends implying that all four carboxylates are ionized at low pH and that the nitrogens are deprotonated in the complex. The alpha carboxyls deprotonate below pH 3, the beta carboxyls between 4 and 5, and the protonated amines deprotonate at 6-7 and 11. The neutral ligand comes out of solution below pH 3. The alpha protons and the two N-CH_2 proton pairs become nonequivalent between pH 4 and 10. Most interesting is the observation that the diastereotopic beta methylene protons of the free ligand are not shifted by the same amount ($\Delta\delta_{\beta R}$ = -0.52, $\Delta\delta_{\beta S}$ = -0.59 ppm) upon going from 3.0 to 12.6 and that they show unequal coupling constants with the alpha proton ($J_{\alpha\beta R}$ = 4.2, $J_{\alpha\beta S}$ = 8.7 Hz). This suggests that there are unequal rotamer populations about the C_α-C_β bond in the free ligand and that the average environments of the two types of β-methylene protons differ. Molecular modeling MM2 of the free ligand $EDDS^{-2}$ confirms this difference in average orientation. Similar modeling of the 1:1 complex between Yb^{+3} and $EDDS^{-4}$ ligand puts both methylene protons in similar environments. NMR titration of EDDS with Yb^{+3} leads to the broadening and coalescence of the β-methylene proton signals. The model of the complex also suggests a high level stereochemical rigidity for the CSR so that the further association complex between the CSR and a chiral biomolecule could hold diastereotopic protons at different average distances from the metal, resulting in different chemical shift changes ΔΔδ for these protons. We are especially interested in using such reagents to resolve and make possible assignments for overlapping multiplets due to diastereotopic methylene protons in water soluble amino acids, nucleosides, and antibiotics.



MM2 Model of free EDDS ligand



Yb(EDDS) complex

Mike Minch

Mike Minch and Jason Zhao
Department of Chemistry
University of the Pacific

1. J. Kido *et al* (1991), *J. Org. Chem.*, **56**, 1412-1415.
2. R. Hulst *et al* (1994) *J. Org. Chem.*, **59**, 7453-7458.
3. J. A. Neal and N. Rose (1968) *Inorg. Chem.*, **7**, 2405.
4. K. Nakamoto (1962) *J. Am. Chem. Soc.*, **84**, 2081; **85**, 309.

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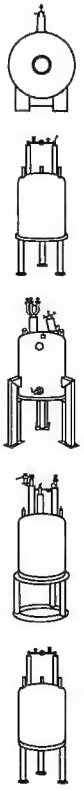

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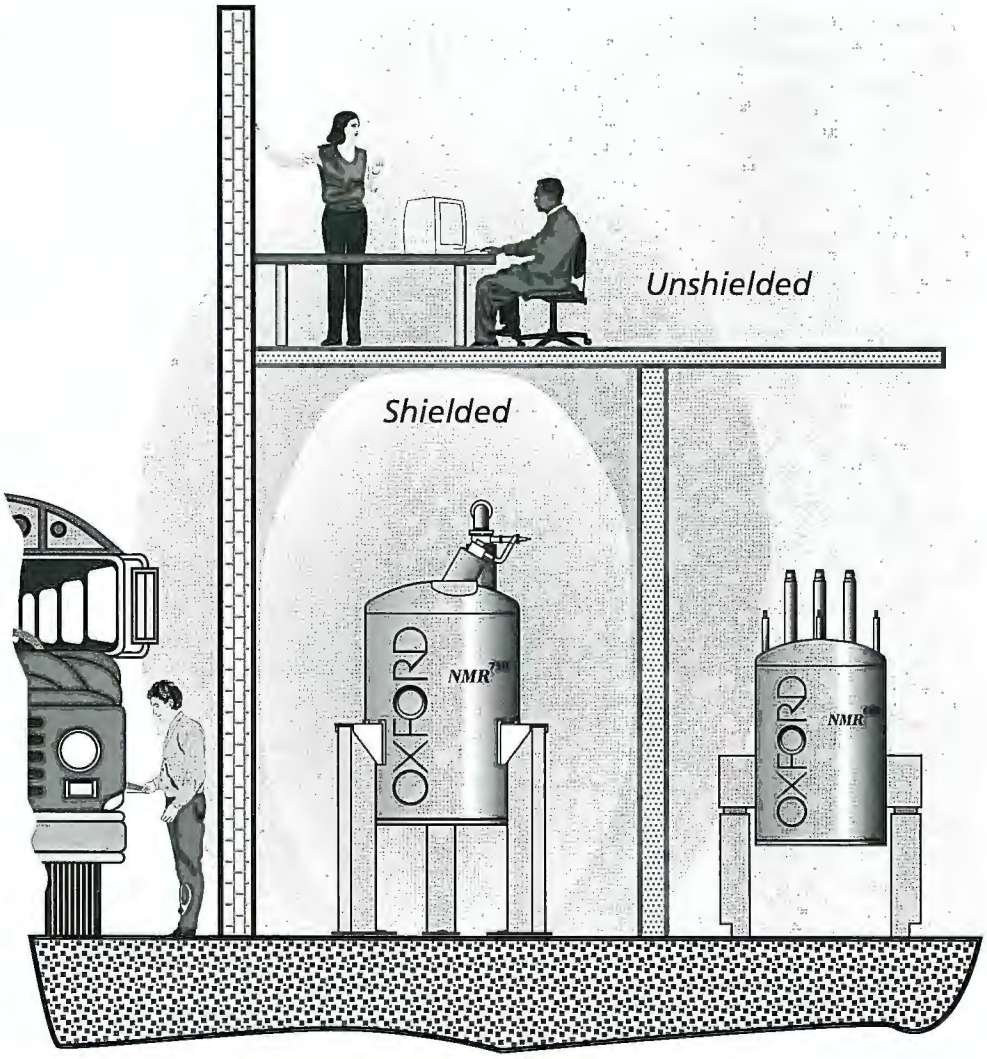

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500	51	10	150	3.2
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360	54	8	365	2.8
300	54	3	365	2.8
270	54	2.7	365	2.8
200	54	2	365	2.8
100	54	1	365	2.8
600	89	12	90	3.4
500	89	15	120	3.4
400	89	10	180	2.8
360	89	10	365	2.8
300	89	3	365	2.8
270	89	2.7	365	2.8
200	89	2	365	2.8
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The NMR Newsletter - *Software* Reviews

Software Review Editor: **William B. Smith**, Texas Christian Univ., Fort Worth, TX 76129

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There are times when most of us wish to have the capability of processing our NMR data at our desks and incorporating the output into our own reports and publications. The last time I investigated buying a duplicate software set from the manufacturer of our instrument the price was prohibitively high. Happily one doesn't have to stretch so far to get the job done. Several of the NUTS programs are directed towards achieving that goal using currently available networking facilities and your desktop PC or Mac. This review is devoted primarily to NUTS 1D, NUTS 2D and Virtual Spectrometer. NUTS Lite is a 1D processing program allowing one to FT, apply window functions, do peak picking and spectral plotting. It doesn't offer the flexibility of NUTS1D, which is reflected in the relative pricing. I also didn't concern myself with SAM which allows one to practice magnet shimming without having an instrument to practice on.

NUTS1D is a general purpose spectrum-processing program (all nuclei) having all or virtually all the capabilities of the software delivered with the major brands of instruments. FIDs are ported from the spectrometer by networking into the PC (I used a 66 MHz, 486DX machine with 32 MB of RAM) or Macintosh. The program offers several window options for pre-transform data massaging. 1D transforms, even with a 66 MHz computer, were very rapid. A large number of sizing, peak picking and printing options are available, allowing one great flexibility equal to that of the manufacturer's software.

NUTS includes a ten-spin simulation program, which places it among the largest readily available as far as I know. The operation is very straightforward. Though a ten-spin simulation at 66 MHz might take several hours to complete, six- and seven-spin simulations seemed to go very rapidly, ca. 10 seconds or less.

The NUTS 2D program is equipped to translate Varian VXR and UNITY files. However, I initially found that the available macros failed on a 2D data set acquired with our new INOVA. An email inquiry to Acorn resulted promptly in a revised macro customized for this specific

continued

experiment. The software will also translate a wide variety of other data sources. To name a few: Bruker (ASPECT 3000 and UNIX), Nicolet, GE, and JEOL files. This list is not complete. A standard 2D data set for trial is included in NUTS 2D, and while slower at 66 MHz than on a UNIX workstation, the transform time was only three or four minutes, much faster than on our XL-300 for a comparable transform. Again a wide range of data processing options are included which compared very favorably with those available on our two Varian instruments.

The Virtual Spectrometer is an add-on program to the NUTS data processing software. A set of commands are given which allows one to simulate the operation of an FT NMR spectrometer. A sample file is included with a number of stored FIDs which the student can process. Additional files can be constructed from the spin simulation routine. The student sets a series of acquisition parameters, then "acquisition" is initiated and a FID displayed. Processing and display are done as with real data. The spectrum can be sized, peak peaking initiated, and the result printed, etc.

I did not have an opportunity to test the SAM (Shimming Ain't Magic) program on magnet shimming. I would point out that Woody Conover of Acorn has written the section on shimming in the new Wiley Encyclopedia of NMR. For those of us who find magnet shimming a hit-or-miss operation, I predict this program will be a real help. For those needing to process NMR data on a desktop computer, this software has the capabilities of more expensive programs, but at an affordable price. Each program comes with an accompanying manual which appeared quite complete and certainly less complex that those put out by many manufacturers. I found the NUTS programs to be user-friendly, and Acorn NMR to be very responsive.

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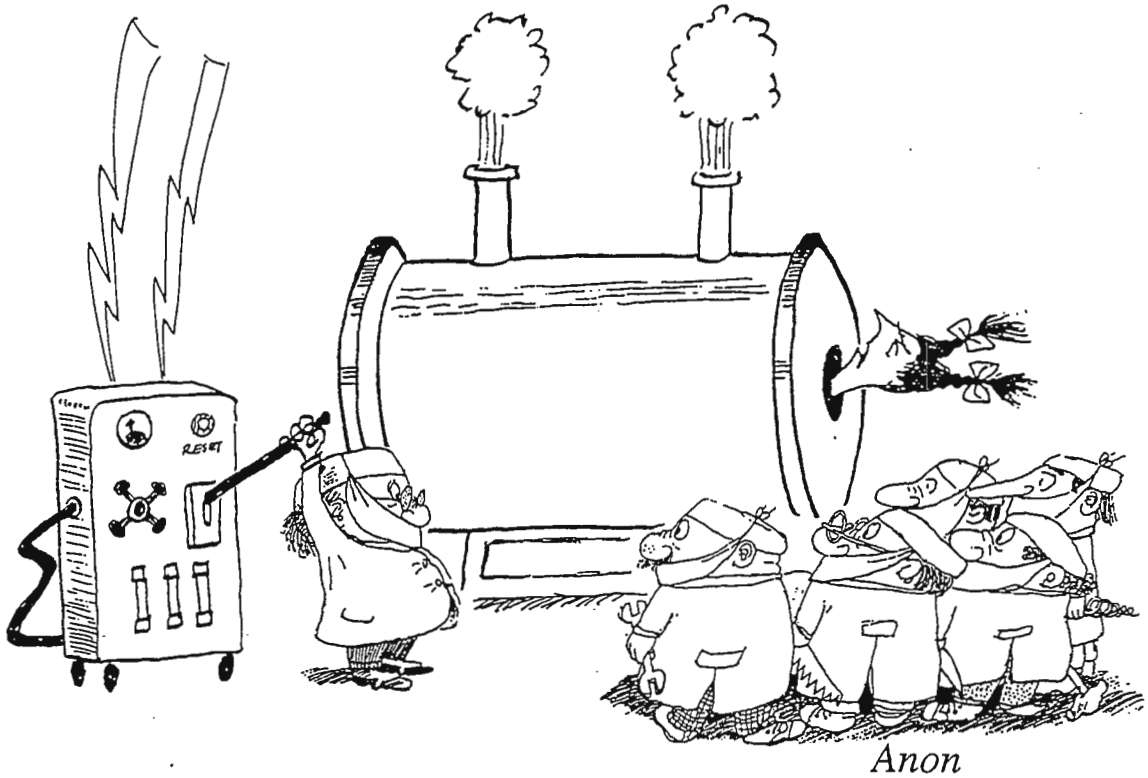

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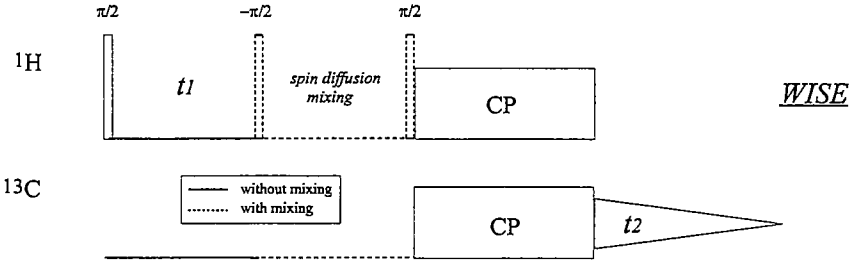
THE COLLEGE OF STATEN ISLAND
THE CITY UNIVERSITY OF NEW YORK

December 18, 1996
(received 12/19/96)
Dr. Bernard Shapiro
The NMR Newsletter
966 Elsinore Court
Palo Alto, CA 94303

A WISE Approach to Heterogeneous Biopolymer Mixtures: Dynamics & Domains in Wounded Potato Tissues

Dear Barry,

Since its development, the 2D ^{1}H-^{13}C wideline separation experiment (WISE)[1] has been utilized to investigate the mobility of domains and proximity of molecular moieties in synthetic co-polymers. Recently, we have obtained some interesting results when this method is applied to a suberin bio-polymer mixture. The lamellar structure of suberin prepared from wound-healing potato tubers consists of cell-wall polysaccharides and a crosslinked aromatic-aliphatic polyester that regulates water transport and protects the tissue from pathogenic attack.



The WISE spectrum of *dry suberin* in Figure 1a shows clearly that without a period for ^{1}H spin diffusion, only a portion of the aliphatic methylene carbon chains exhibit particular flexibility (narrow lines) on the 50-kHz timescale, while other carbons in the polyester or the cell-wall polysaccharides are very rigid (wide lines). With some mixing time to allow spin diffusion, this line narrowing is propagated rapidly (within 1 ms) to other residues within the suberin domain (Fig. 1b) but very slowly to the cell wall (>100 ms required for completion). These WISE results reinforce the finding of spatially separated suberin and cell-wall domains made previously from rotating-frame ^{1}H spin relaxation experiments.[2] In addition, quantitative ^{1}H spin-diffusion measurements using a dipolar-filtered sequence with ^{13}C detection[3] (data not shown) yield a spin diffusion coefficient of suberin >10 times larger than that of previously studied synthetic polymers, suggesting that suberin is a resilient material with significant spectral density at 50 kHz.

The WISE spectrum of *hydrated suberin* shows split peaks along the ^{1}H axis for cell-wall carbons (Figure 2). This is due to the ^{1}H frequency offset from the H_2O signal, because WISE intrinsically detects only the cosine dataset.[4] Moving the ^{1}H frequency to the H_2O peak makes the splitting disappear. Results obtained at various mixing times demonstrate that spin diffusion

[1] K. Schmidt-Rohr, J. Clauss and H.W. Spiess, *Macromolecules*, 25 (1992) 3237.
[2] R. E. Stark and J. R. Garbow, *Macromolecules*, 25 (1992) 149.
[3] W. Z. Cai, K. Schmidt-Rohr, N. Egger, B. Gerharz and H. W. Spiess, *Polymer*, 34 (1993) 267.
[4] K. Schmidt-Rohr and H. W. Spiess, *Multidimensional Solid-State NMR and Polymers*, Academic Press, 1994.

proceeds from the H_2O to the cell wall, and subsequently to the suberin domain, indicating that water is localized near the hydrophilic cell-wall carbohydrates. This supports the hypothesis that suberin's protective role relies on the ability of the crosslinked aromatic-aliphatic polyester to resist water permeation and possibly bacterial attack. Finally, our results demonstrate that WISE spectroscopy can be applied easily to bio-polymer systems in addition to relatively "well defined" synthetic co-polymers.

Sincerely,

Ruth E. Stark

Ruth E. Stark
Professor of Chemistry

Bin Yan
Postdoctoral Research Associate

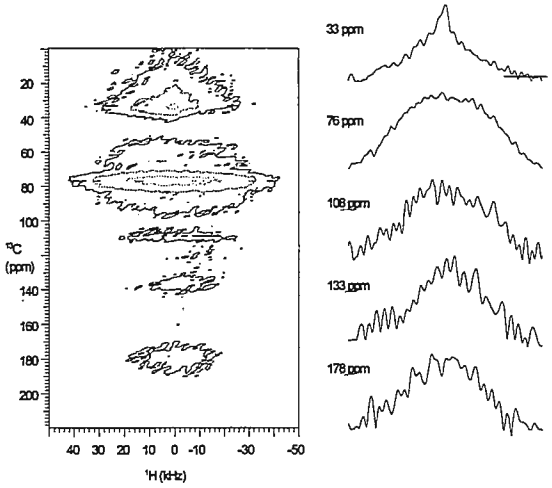
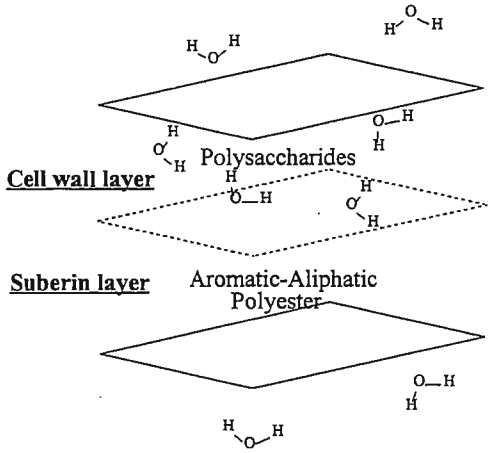


Fig.1a WISE of *dry suberin* without mixing.



Lamellar structure of *potato suberin*

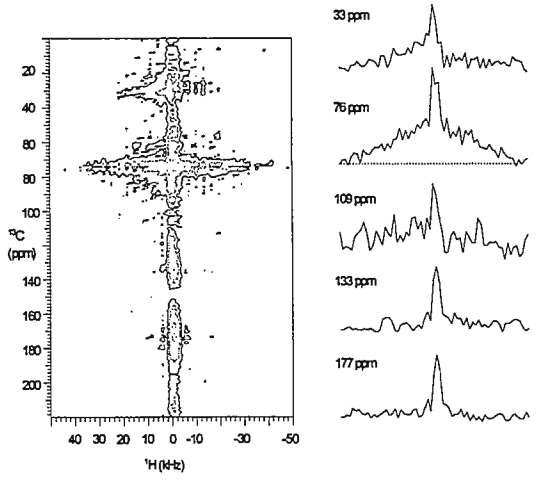


Fig.1b WISE of *dry suberin* with 1 ms mixing.

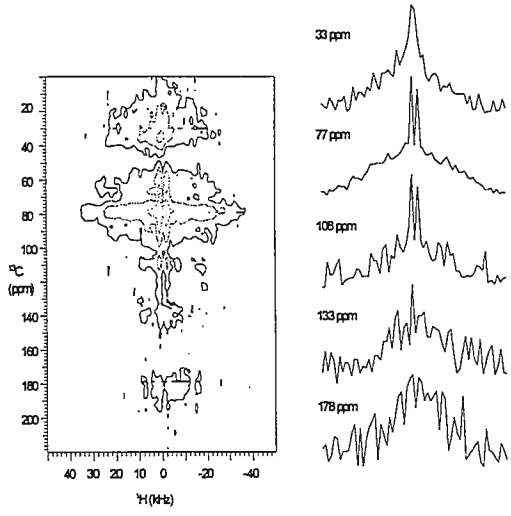


Fig. 2 WISE of *hydrated suberin* w/o mixing.

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Rotor Volume	20 uL	With spacers
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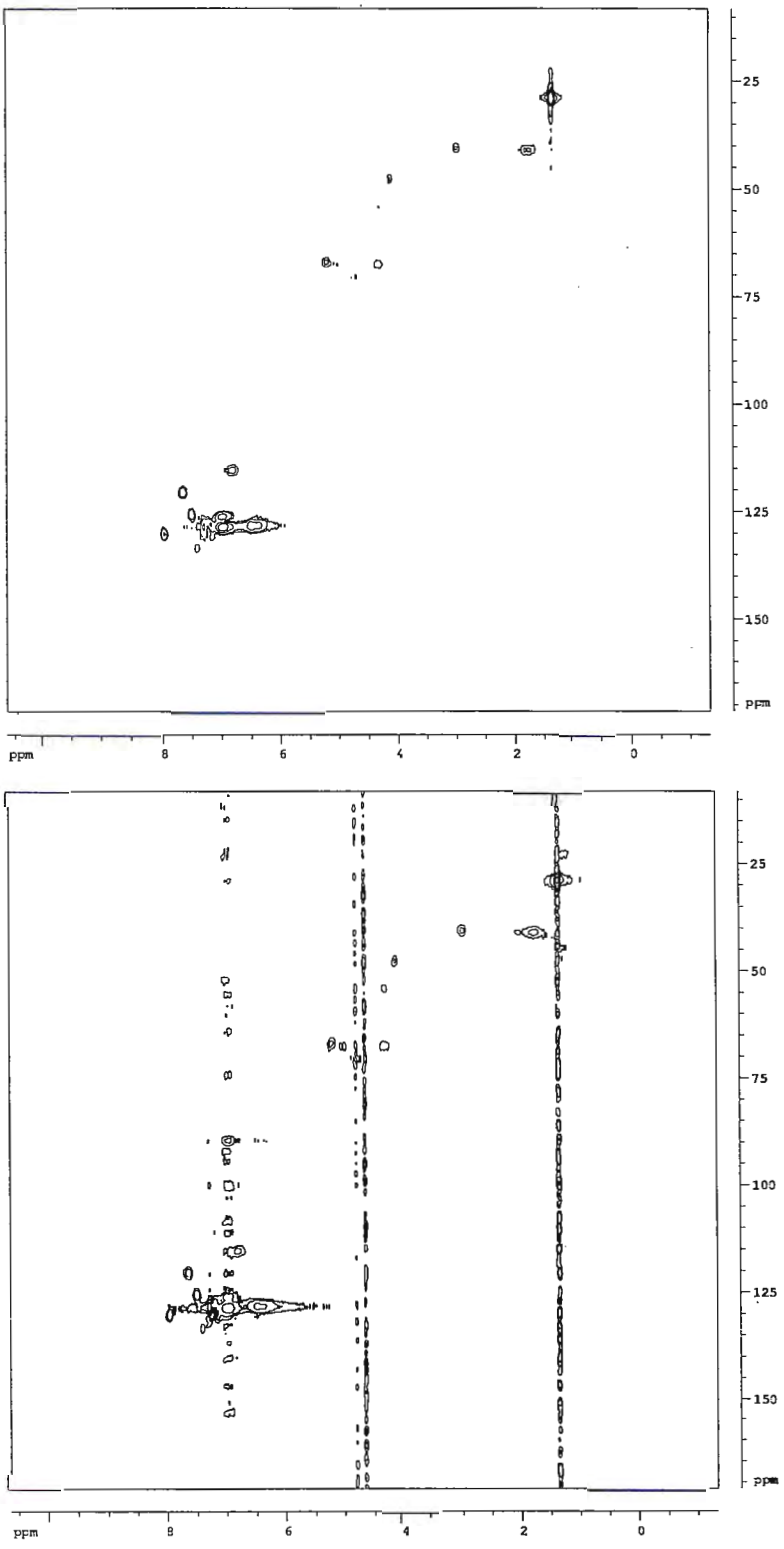
^{1}H 90° pulse	5 us	100 W
^{13}C 90° pulse	5.5 us	300 W
Gradient Strength	30 G/cm	at 10 A
VT range	-20 to +70 °C	with ceramic rotor cap
Max. Spin Rate	10 kHz	With ZrO rotors

Gradient MAS Heteronuclear Correlation Experiment



A. ^{1}H-^{13}C HMQC spectrum of an N-FMOC-N-Boc-L-Lysine derivatized Wang resin swollen with $CDCl_3$, obtained at a proton frequency of 400 MHz and at a spinner frequency of 5 kHz. 1 ms pulsed field gradients were used (with strengths of 10, 10 and 5 G/cm) to select magnetization only from those protons coupled to a ^{13}C. The lower spectrum (B) is a phase cycled version, acquired under identical conditions as the spectrum of figure A. Note the excellent suppression of t_1-noise in the gradient spectrum versus the phase cycled version.

Lilly

Lilly Research Laboratories
A Division of Eli Lilly and Company

Lilly Corporate Center
Indianapolis, Indiana 46285
(317) 276-2000

December 5, 1996
(received 12/19/96)

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

Automated Sample Preparation with the Bruker BASP

Dear Barry,

We have recently installed in our lab a DPX-300 replacing an aging QE-300. This instrument is used exclusively for routine ^{1}H, ^{13}C, ^{19}F, and ^{31}P. The data (paper copies) are returned to the chemists and also stored electronically in our corporate LIMS (called CASSPER) for future referencing.

Since we have responsibilities for routine data, we are concerned with efficient sample preparation and data acquisition. To this end, we have also purchased the Bruker BASP robot for preparing samples along with the SamTrack software. The information on each sample is imported into SamTrack from CASSPER and Oked. The sample is prepared by the robot, taken (by human intervention) to the DPX-300 and placed on the sample changer. The information on each sample is imported into the DPX-300 through a bar code collar. When data acquisition, processing , etc. is complete, a JCAMP file is generated and placed into a file for archival into CASSPER.

While there are still some software changes that we want to make, such as automating the upload of the JCAMP files into CASSPER, the system seems to be settling into the lab. The time for moving data through the lab has decreased 16% (assuming the same sample load). In addition, we look forward to an increase in samples because of combinatorial methods being instituted by our chemists.

It is reasonable to recognize the international effort in the installation process of this equipment. At Lilly, the computer and engineering groups with Doug Fegenbush (computer) and Mike Holdmann (engineering) have provided excellent expertise. Along with these, Hans Kleeburg and Martin Beck at Bruker Germany have been very helpful even though mostly they were 7 hours ahead of us in time. Our Bruker collaborators continue to offer input into issues as they occur.

Sincerely,

Jon

Jonathan W. Paschal

Larry

Larry A. Spangle

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Jeffrey C. Hoch, Rowland Institute, USA
Oleg Jardetzky, Stanford University, USA
Martin Karplus, Harvard University, USA
Anthony A. Kossiakoff, Genentech Inc., USA
Jean-François Lefèvre, Université Louis Pasteur, France
Michael Levitt, Stanford University, USA
William N. Lipscomb, Harvard University, USA
John L. Markley, University of Wisconsin , USA
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GENERAL INFORMATION

Prospective participants should apply to either:

Prof. Oleg Jardetzky
Stanford Magnetic Resonance Laboratory
Stanford University
Stanford, CA 94305-5055
USA
fax: +415/723-2253
phone: +415/723-6270
jardetzky@camis.stanford.edu

or

Prof. Jean-François Lefèvre
ESBS, CNRS-UPR9003
Université Louis Pasteur
Blvd. Sébastien Brant
F67400 Illkirch Graffenstaden
France
fax: +33/88 65 52 62
phone: +33/88 65 52 69
lefevre@bali.u-strasbg.fr

stating: (1) date and place of birth, nationality, qualifications and present position; (2) address, fax and phone numbers and email address; and (3) list of publications.
Applicants interested in submitting unpublished results should send the title and an abstract of about 200 words. Selected papers will be presented and discussed in special sessions.

The total fee, including full board and lodging (arranged by the School) will be US $1,200. Limited financial aid available. Participants should arrive by 5 p.m. on the 16th.

THE CLOSING DATE FOR RECEIPT OF APPLICATIONS IS MARCH 15, 1997. NO APPLICATION FORM IS REQUIRED.

Information on the Course is available on the world wide web at http://cmgm.stanford.edu/SMRL/Erice97.html

VENUE

The Ettore Majorana Centre for Scientific Culture was founded in 1963 in the pre-medieval mountain town of Erice near Palermo as a Conference Centre, taking its inspiration from the Italian Physicist, Ettore Majorana. The Centre's lecture halls are located in two restored monasteries and the ancient Palazzo Ventimiglia. School participants are housed in the Centre Institutes or local hotels and meals are taken at local restaurants.

Attendance will be limited to ~75 students, to be selected by the Co-Directors. Further details will be mailed with the acceptance letter.

Jean-François Lefèvre
Co-Director of the School

A. Zichichi
Director of the Centre

Oleg Jardetzky
Co-Director of the School

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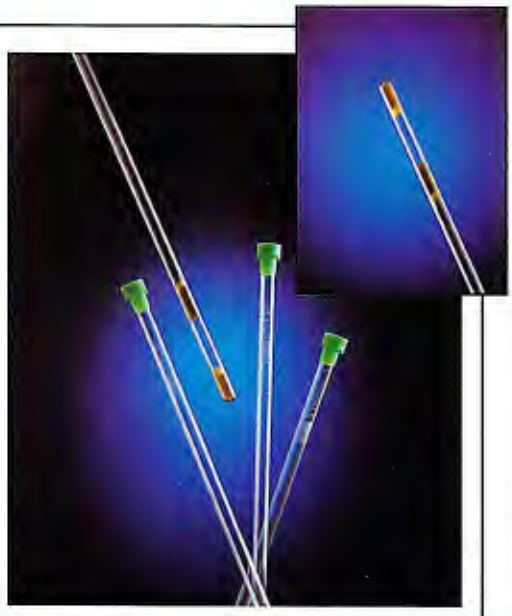

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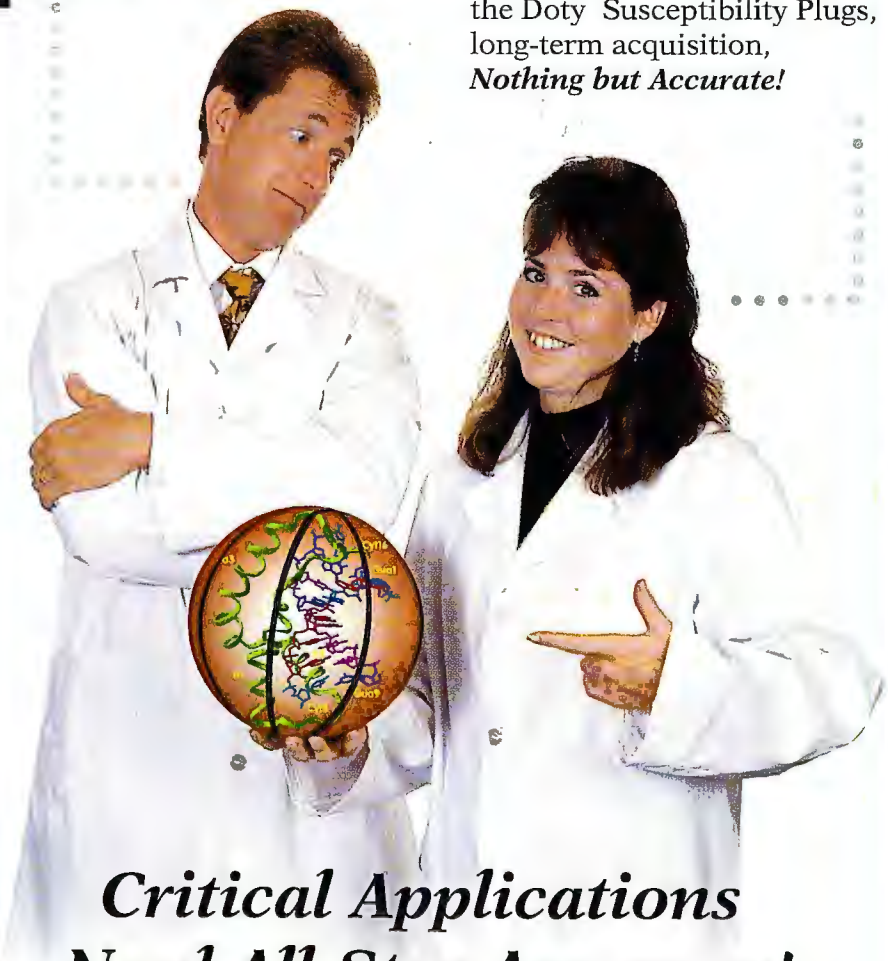

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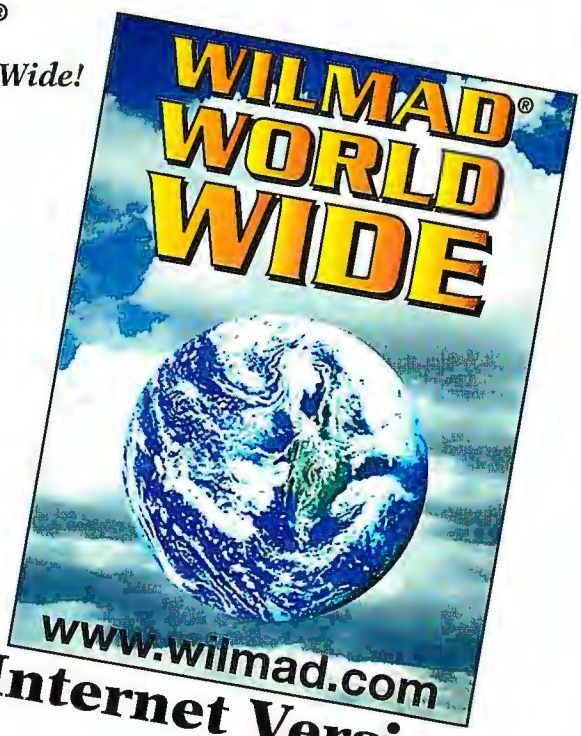

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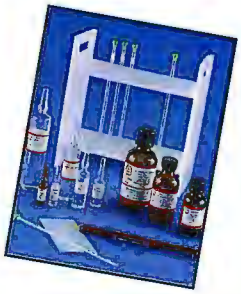


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Position Available

Three positions exist at the SINTEF Unimed MR-Center in Norway. Two of the positions are for experienced NMR scientists in the fields of biomedical and petroleum related NMR research. The third is for a specialist in image / data analysis. The MR-Center is one of the best equiped centers for applied magnetic resonance research in the world. Ca. 25MNOK was invested in 1995/6 in state-of-the-art NMR equipment, ranging from 600MHz down to 2MHz. The MR-Center presently employs 10 full-time members of staff and is looking to expand signifcantly in 1996/7. Research application areas include; petroleum, food, materials, biomedical and biotechnical research.

Candidates for the Biomedical NMR position are expected to have experience in operating the latest Bruker DBX100 instrument in both imaging and in-vivo spectroscopy modes. Experience in implementing EPI would be an advantage. The main applications will be in the field of pre-clinical brain research: evaluation of cerebro-protective drugs, brain metabolism studies and new applications for MR contrast agents. There is also the opportunity to carry out patient studies jointly with the clinical section of the MR-Center. This position is available immediately.

Candidates for the position in data / image analysis are expected to have proven experience in the latest methodologies, including: multi-variate image analysis, principal component analysis and Neural Networks. Experience in using and developing image display and manipulation software would be an advantage. The successful candidate would also be responsible for running and building-up our existing computer center, and experience operating in a UNIX environment would be useful. This position is available immediately.

Candidates for the Petroleum NMR position are expected to have several years experience in carrying out NMR research within the fields of NMR core analysis and petrophysics, and also to have an interest in NMR Core-to-Log integration. Experience with NMR Logs would be considered an advantage. The candidate would also be expected to be involved in supervising our successful NMR core analysis technical services. This position is available in February 1997.

Further information about the positions can be obtained from research manager John Attard, tel:+47 73 59 89 25, fax: +47 73 99 77 08, E-mail: john.attard@unimed.sintef.no.
Applications, including CV and relevent publications, should be submitted to personnel manager Hjørdis Bjørseth, SINTEF Unimed, N-7034, Trondheim, Norway. Please mark the envelopes Research Scientist MR-Center.

It would be useful if you would include your e-mail address in any written communications to The NMR Newsletter, or send this address to me separately by e-mail. Thanks.

BLS

DEPARTMENT OF CHEMISTRY
AARHUS UNIVERSITY

Langelandsgade 140 · DK-8000 Aarhus C · Denmark
Tel. +45 8942 3333 · Fax +45 8619 6199

HANS JØRGEN JAKOBSEN (☎ direct +45 8942 3842 · E-mail: hja@kemi.aau.dk)
Director, Instrument Centre for Solid-State NMR Spectroscopy

November 27, 1996
HJJ/ATL

Dr. B. L. Shapiro, Publisher
The NMR Newsletter
966 Elsinore Court
PALO ALTO, California 94303
U.S.A.

Dear Barry,

Staff Scientist: Research Associate in Solid-State NMR Spectroscopy

A "National Instrument Centre for Solid-State NMR Spectroscopy" will be opened in 1997 at the Department of Chemistry, The Faculty of Sciences, University of Aarhus, Aarhus, Denmark. A position as a research associate at the centre for a 5-year period will be available from March 1, 1997 or later. The facility has been established by an agreement between the Danish Natural Science Research Council and the University of Aarhus and involves the installation of a 600 MHz (14.1 Tesla) widebore (89 mm) Oxford magnet and Varian UNITY INOVA solid state NMR spectrometer in early 1997. In addition, the instrument centre also has a Varian UNITY-400 widebore and a UNITY INOVA-300 standard-bore magnet solid state NMR spectrometer available for its research and services for external users. State-of-the-art probes (homemade and Doty Scientific) for high-speed CP/MAS (22 kHz), DOR, $^{1}H/^{19}F$ CRAMPS, triple resonance (^{1}H, X, Y), and single-crystal NMR are available. The research projects involve studies of all kinds/combinations of NMR interactions in the solid state by experimental and theoretical methods and applications of these methods to chemistry, biochemistry, mineralogy, and materials science.

The research associate shall serve as a facility manager for the three solid state NMR spectrometers. He/she will be responsible for maintenance, troubleshooting, upgrades, development and implementation of new instrumental hardware for these spectrometers. The position also involves the opportunity for doing solid state NMR research/applications and collaboration with the external users. Priority will be given to applicants with research areas within inorganic chemistry. No teaching obligations (e.g. in the Danish or English language) are required. The successful candidate shall report to the director of the instrument centre.

The candidate for the position must have a PhD, post-doctoral training and research acccomplishments, and demonstrated expertise in NMR instrumentation related to the requirements described above. Initially, a 2- or 3-year appointment will be offered to the successful candidate.

Futher information on the position, which will be announced in *NATURE* (December 1996), may be obtained from professor Hans J. Jakobsen. The deadline for the application is February 3, 1997 and it is expected that the successful candidate can take up the position within the first six months of 1997.

Sincerely

Hans J. Jakobsen

Hans J. Jakobsen
Professor, Director of the Instrument Centre

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- CW mode
- Power supply
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- Energy (F)
- Power limit select Po or Po/2 (C/F)
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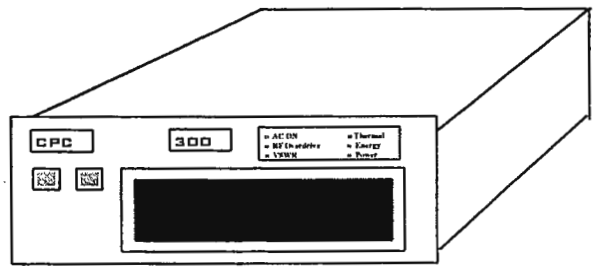

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Prof. Dr. Paul S. Pregosin

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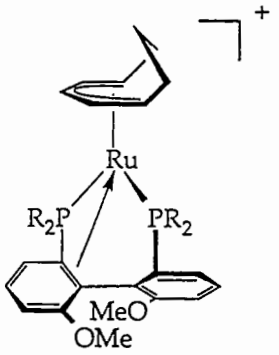
Prof. Barry Shapiro
Editor/Publisher
NMR Newsletter
966 Elsinore Court
Palo Alto Ca. 94303
USA

11/26/96
(received 12/7/96)

Dear Barry,

Enantioselective homogeneous catalysis, using atropisomeric chelating di-phosphines as chiral auxiliaries, remains an exciting field of research. The class of ligands called BIPHEP has proven to be particularly successful in Ru(II)-based homogeneous hydrogenation. We have recently shown, via a long-range $^{13}C,^{1}H$-correlation, that the cyclo-octapentadienyl-complex **1** reveals two *non-protonated* ^{13}C resonances at 74.5 ppm, as a doublet, and 95.1 ppm, as a very weak triplet. These can be assigned to a coordinated biaryl double bond, as indicated by the arrow. This is a new type of coordination mode for this ligand and is suggestive of heretofore unrealized potential for the stabilization of reactive intermediates.



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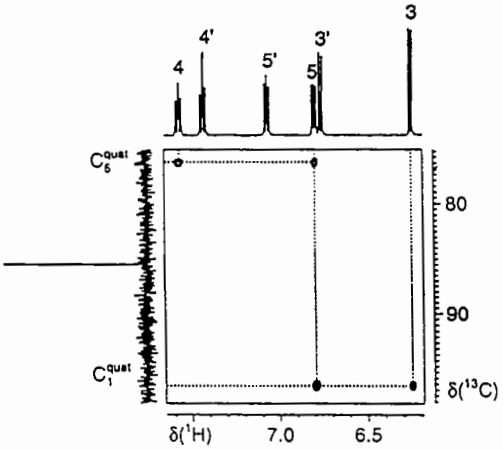
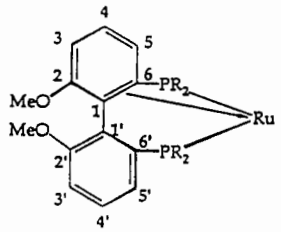




Sincerely,

Prof. P. S. Pregosin

Suggested Title:Unusual Bonding Mode for Chiral BIPHEP ligands.

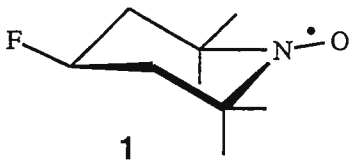
Service de Chimie Inorganique et Biologique

DIRECTION DES SCIENCES DE LA MATIÈRE
DÉPARTEMENT DE RECHERCHE FONDAMENTALE
SUR LA MATIÈRE CONDENSÉE

(received 12/9/96)

High field NMR spectroscopy is it always the best choice ?

Some time ago we were looking to measure T_1 for ^{19}F in a paramagnetic compound, namely fluorinated Tempol **1** at room temperature. We were very surprised in getting no fluorine NMR signal at 400 MHz. We therefore questionned the paramagnetic influence of the unpaired electron and we reduced the compound to observe its diamagnetic form. Unfortunately, we were still unable to observe any well defined signal. This was very intriguing. The set up of the machine was not in question. However we decided to test it with a commercial sample of fluorocyclohexane. No signals could be recorded in the same conditions on the same apparatus. We decided then to return to litterature and found that it was necessary to lower the temperature to get a well resolved spectrum.



1

At 193K we obtained nice doublet at -3ppm and quartet at -23.5ppm (ref. C_6F_6) looking like those recorded by Bovey and collaborators in 1964 on a DP60 Varian spectrometer at 210.4K.
In the same conditions we were able to record the spectrum of the 4-fluoro-Tempol in the diamagnetic form (δ= -21 ppm ; doublet, J_{HF} = 49.5 Hz) and moreover we could measure T_1=0.32 s for ^{19}F at that temperature while no valuable result could be obtained at room temperature owing to the peak's width. In the case of the paramagnetic form no reproducible spectrum could be recorded.
We also tried to observe the spectra of the three compounds on a 200 MHz spectrometer and we were successful in getting quite large but nevertheless good signals for fluorocyclohexane, even at room temperature while fluoro-Tempol gave rather sharp peaks in the same conditions (doublet, J_{HF} = 49 Hz). Of course they arose from rapid interconversion between axial and equatorial forms for fluorocyclohexane and this was ascertained by the spectra at 193K. At that temperature the two axial and equatorial forms were clearly apparent. But only one conformation was detectable at 193K for fluoro-Tempol corresponding to the equatorial fluorine with reference to fluorocyclohexane. It is noteworthy that the equatorial conformation is also the conformation observed in the solid state.
Unfortunately those experimental conditions did not give us the opportunity to test the fluorine nucleus as a probe in evaluating intramolecular distances in paramagnetic species. The reason for this is probably that fluorine is too close to the unpaired electron and consequently T_1 is too small to be measured with accuracy. Nevertheless it remains that in this instance the spectra were more easily recorded at 200 MHz than at 400 MHz !

References :

F.A. Bovey, E.W. Anderson, F.P. Hood, and R.L. Korngay, *J. Chem. Phys.*, **40**, 3099-3109 (1964).

F. Cinget, P.H. Fries, U. Greilich, and Ph.J. A. Vottéro, *Magn. Reson Chem.*, **33**, 260-272 (1995).

Cécile Vigouroux

Philippe Vottéro

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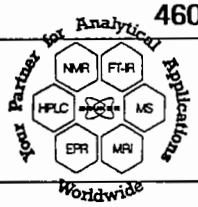


Two different ways to cancel relaxation artefacts in 2D INADEQUATE by using field gradients.

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

(received 12/16/96)

Dear Barry,

Recording 2D INADEQUATE with a short relaxation delay generates artefacts at the zero frequency in F1 and on the slopes 2 or 1 (fig.1) .

A first way to cancel these artefacts is to use a pair of field gradients separated by a 90° pulse before the relaxation delay. By applying this pair of gradients , the magnetisation of the artefacts is destroyed at the end of each acquisition and can not have an evolution during the following scan (fig.2).

A second way is to select , during the experiment , only the informations concerning the double quantum coherences with a good combination of field gradients . This can be done by adding a first gradient before the last 120° reading pulse and a second one before the acquisition in a ratio 1:2 for a N type selection (ref.1 and fig.3).

Using either one of these two methods , to destroy the magnetisation of the artefacts or to select the good magnetisation, is a good answer to the problem of relaxation artefacts in 2D INADEQUATE.

M.Bourdonneau

C.Brevard

1. H.Koshino and J.Uzawa, Bull.Magn.Reson. 17(260) , 1996

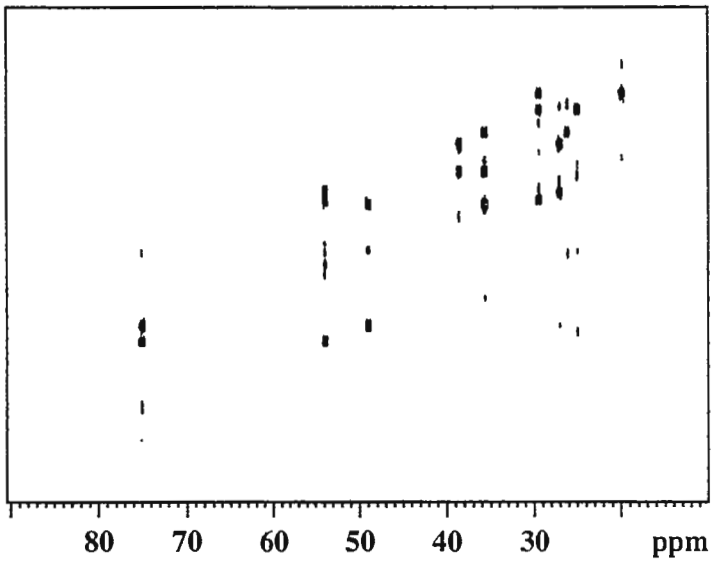


fig.1
INADEQUATE standard
d1+aq=0.9s

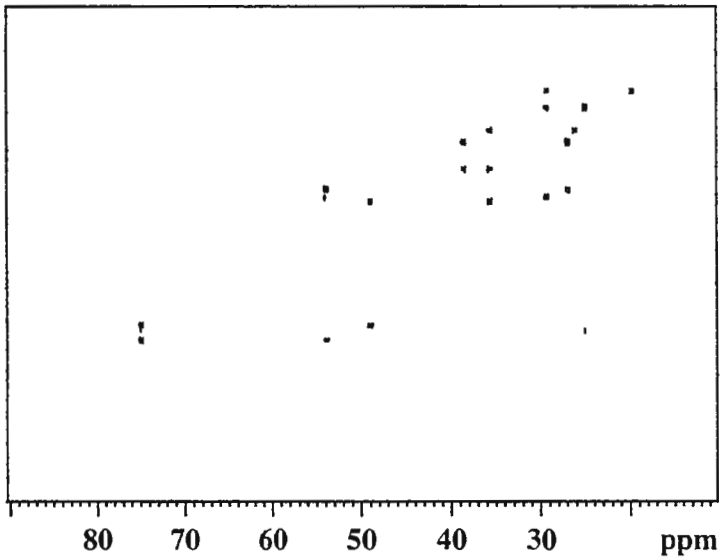


fig.2
INADEQUATE with two gradients separated by 90(13C) pulse before the relaxation time
d1+aq=0.9s

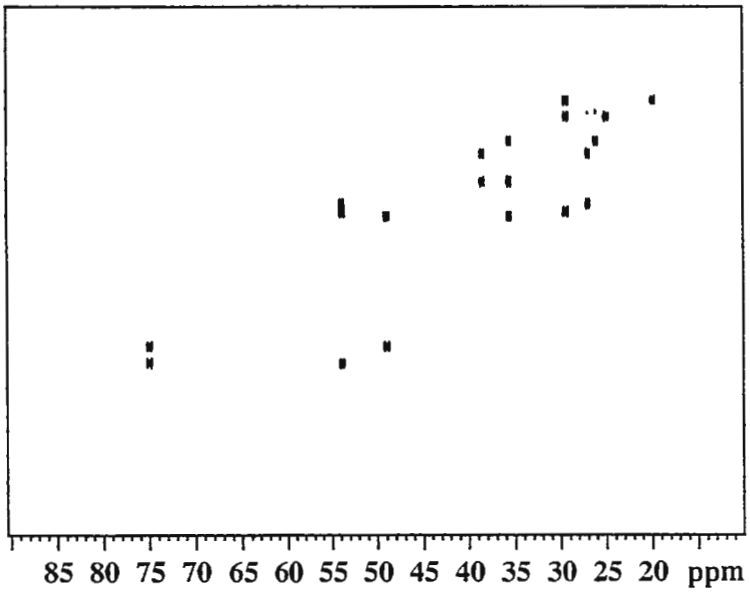


fig.3
INADEQUATE with two gradients for selection of coherence
d1+aq=0.9s

All the experiments were run with a sample of menthol in CDCl3 , without adding of relaxation agent, on a DRX 400 equipped with a 5mm qnp gradient probehead.

ENC 38th Experimental Nuclear Magnetic Resonance Conference

March 23 - 27, 1997
Clarion Plaza Hotel, Orlando, Florida (USA)

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

December 23, 1996

1997 AND 1998 ENC Meetings

Dear Barry,

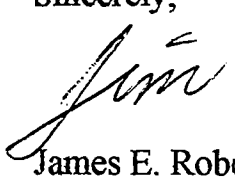
With greetings for the Holidays and the New Year, perhaps this letter can clear up some confusion regarding the next few ENC Meetings.

1997: The 38th ENC will be held March 23-27, 1997, at the Clarion Plaza Hotel on International Drive in Orlando, Florida. Although the abstract submission deadline has passed, preregistration is open until February 21. This year electronic abstract submission allows **ALL** abstracts to be posted on our active World Wide Web site at <http://www.enc-conference.org> in early February. The preliminary program (titles and authors only) will be sent to all preregistered participants; the customary book of full abstracts will be available at the meeting. The Clarion Plaza has a very nice arrangement, with **ALL THE VENDOR SUITES ON THE SAME FLOOR!!!** As Orlando is a popular destination in March, I urge you to make your travel arrangements early.

1998: The 39th ENC will be held March 22-27, 1998, **AT ASILOMAR CONFERENCE CENTER** in Pacific Grove, California. Please note the meeting runs until Friday at Noon; some rooms at Asilomar may be available for the weekend of March 20-22. Professor Regitze Vold will be the Chairperson.

As always, everyone should feel free to contact the ENC office (through the Web site, or see below) for information, or to contact anyone on the ENC Executive Committee (see left) to make suggestions. I look forward to seeing you again in Orlando.

Sincerely,



James E. Roberts
38th ENC Chairperson

You are invited to attend the

5th ANNUAL

ADVANCES IN NMR APPLICATIONS SYMPOSIUM

Featuring the Latest Developments in Experimental Techniques

To be held prior to ENC at the
Omni Rosen Hotel
Ballroom D & E
9840 International Drive
(located next to the Clarion Plaza Hotel, site of the 38th ENC)

Sunday, March 23, 2:30 to 6:00 p.m.

The agenda includes a presentation of recent results by leading NMR experimentalists concerning applications of pulsed field gradient and classical NMR techniques with both large and small molecular systems. The results obtained will be of interest to all liquid state NMR Spectroscopists.

Request a detailed program or RSVP by contacting Chris Tierney, Nalorac's ENC Coordinator.

NALORAC

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

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

Deadline Dates

No. 461 (Feb.)	24 Jan. 1997
No. 462(March)	21 Feb. 1997
No. 463 (Apr.)	21 Mar. 1997
No. 464 (May)	25 Apr. 1997
No. 465 (June)	23 May 1997

* Fax: (415) 493-1348, at any hour. Do not use fax for technical contributions to the Newsletter, for the received fax quality is very inadequate.

E-mail: shapiro@nmrnewsletter.com

http://www.nmrnewsletter.com

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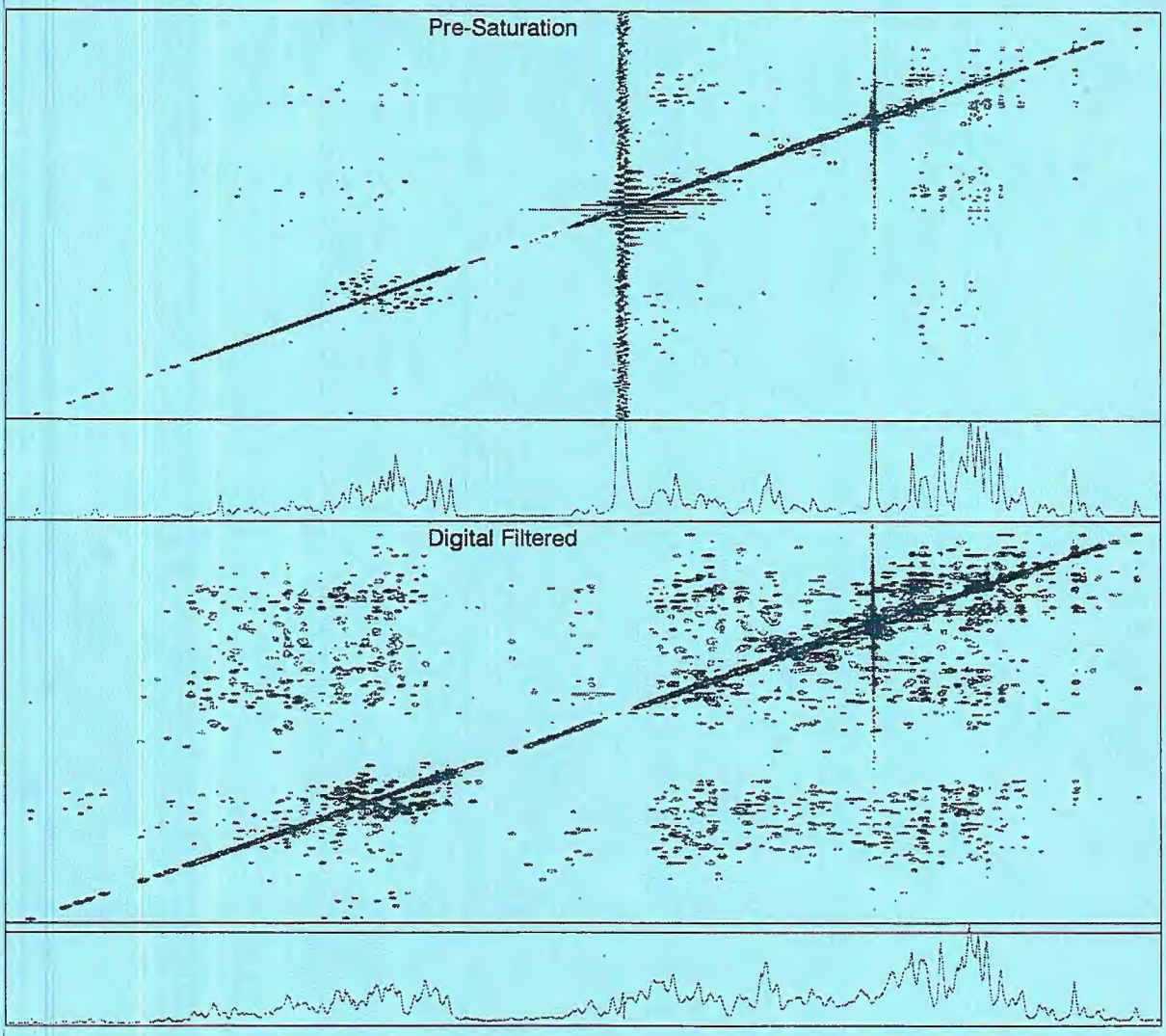
ECLIPSE NMR Advantage: Digital Filtering



Eclipse NMR *The Better Way!*

This data shows the digital filtering capability of JEOL USA's ECLIPSE NMR workstation. Eclipse does digital filtering via software after the data is acquired, not via hardware during acquisition. This offers a significant advantage because with software digital filtering the acquisition is completed before you filter the data.

JEOL feels spectrometer time is best spent acquiring new data rather than repeating experiments because conditions were not optimized. It takes more time to write this kind of software, but JEOL took the time. Now you can use the ECLIPSE NMR Advantage to your advantage.

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