THE
NMR
NEWSLETTER

No. 501

June 2000

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PTS 040	.1-40 MHz	optional .1 Hz to 100 KHz	1-20μs	optional	5¼"H×19"W	BCD (std) or GPIB (opt)	$5,330.00 (1 Hz resol., OCXO freq. std.)
PTS 120	90-120 MHz	optional .1 Hz to 100 KHz	1-20μs	optional	5¼"H×19"W	BCD (std) or GPIB (opt)	$5,330.00 (1 Hz resol., OCXO freq. std.)
PTS 160	.1-160 MHz	optional .1 Hz to 100 KHz	1-20μs	optional	5¼"H×19"W	BCD (std) or GPIB (opt)	$6,495.00 (1 Hz resol., OCXO freq. std.)
PTS 250	1-250 MHz	optional .1 Hz to 100 KHz	1-20μs	optional	5¼"H×19"W	BCD (std) or GPIB (opt)	$7,440.00 (1 Hz resol., OCXO freq. std.)
Type 1 **PTS 310** Type 2	.1-310 MHz	1 Hz	1-20μs	standard	3½"H×19"W	BCD (std) or GPIB (opt)	1 Hz resol., OCXO: $6,425.00 1 Hz resol., OCXO: $5,850.00
PTS 500	1-500 MHz	optional .1 Hz to 100 KHz	1-20μs	optional	5¼"H×19"W	BCD (std) or GPIB (opt)	$8,720.00 (1 Hz resol., OCXO freq. std.)
PTS 620	1-620 MHz	optional .1 Hz to 100 KHz	1-20μs	optional	5¼"H×19"W	BCD (std) or GPIB (opt)	$9,625.00 (1 Hz resol., OCXO freq. std.)
PTS 1000	0.1-1000 MHz	optional .1 Hz to 100 KHz	5-10μs	optional	5¼"H×19"W	BCD (std) or GPIB (opt)	$11,830.00 (1 Hz resol., OCXO freq. std.)
PTS 3200	1-3200 MHz	1 Hz	1-20μs	optional	5¼"H×19"W	BCD (std) or GPIB (opt)	$14,850.00 (1 Hz resol., OCXO freq. std.)
PTS x10	user specified 10 MHz decade	1 Hz	1-5μs	standard	3½"H×19"W	BCD (std) or GPIB (opt)	$3,000.00 (1 Hz resol., OCXO freq. std.)
PTS D310	two channels .1-310 MHz	.1 Hz	1-20μs	standard	5¼"H×19"W	BCD (std) or GPIB (opt)	$8,560.00 (.1 Hz resol., OCXO freq. std.)
PTS D620	two channels 1-620 MHz	.1 Hz/.2 Hz	1-20 μs	standard	5¼"H×19"W	BCD (std) or GPIB (opt)	$13,240.00 (.1 Hz/.2 Hz resol., OCXO freq. std.)

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FORTHCOMING NMR MEETINGS

15th European Experimental NMR Conference, Leipzig, Germany, **June 12-17, 2000**. For information, see http://eenc.uni-leipzig.de.

XEMAT 2000, a Conference on "Optical Polarization and Xenon NMR of Materials", Sestri Levante, Italy, **June 28-30, 2000**. For information, see http://www.mater.unimib.it/xemat2000/

NMR Course: Part 1 - NMR-based Metabonomics; Part 2 - Hyphenated Spectroscopic Techniques, Imperial College, London, England, **July 10-14, 2000**; Contact: Hersha Mistry,Centre for Continuing Education, Imperial College, 526 Sherfield Building, Exhibition Road. London, SW7 2AZ, UK. Tel: +44 (0)20 7594 6884; Fax: +44 (0)20 7594 6883; Email: h.mistry@ic.ac.uk; Website: http://www.ad.ic.ac.uk/cpd/nmr.htm

Royal Society of Chemistry: 15th International Meeting on NMR Spectroscpy, Durham, England, **week of July 8-13, 2001**; Contact: Mrs. Paula Whelan, The Royal Society of Chemistry, Burlington House, London W1V 0BN, England; +44 0171 440 3316; Email: conferences@rsc.org\

SMASH-2000, Argonne, IL, **July 16-19, 2000**. Contact: G. E. Martin (gary.e.martin@amu.pnu.com). See Newsletter 493, 21.

42nd Rocky Mountain Conference on Analytical Chemistry, Omni Interlocken Resort, Broomfield, CO, **July 31 – August 3, 2000**. NMR Symposium Chair: Lucio Frydman, Univ. of Illinois at Chicago, Dept. of Chemistry (M/C 111) 845 West Taylor St., Room 4500, Chicago, IL 60607-7061; 312-413-1053; Fax: 312-996-0431; lucio@samson.chem.uic.edu

XIX International Conference on Mag. Res. in Biological Systems, Florence, Italy, **August 20-25, 2000**. Contact: Profs. Ivano Bentini or Lucia Banci, Chem. Dept., Univ. of Florence, Via G. Capponi 7, I-50121, Florence, Italy; Phone: +39-055-2757600; Email: icmrbs@lrm.fi.cnr.it; Fax: +39-055-2757555; http://www.lrm.fi.cnr.it//icmrbs.html.

NMR: Drug Discovery and Design Conference – Post-Genomic Analysis, McLean, Virginia, **October 24-26, 2000**. Contact: Mary Chitty, Cambridge Healthtech Institute, mchitty@healthtech.com; Fax 617-630-1325.

NMR Spectroscopy of Biofluids and Tissues, Imperial College, London, England, **November 13-17, 2000**. Contact: Hersha Mistry, Centre for Continuing Education, Imperial College, 526 Sherfield Building, Exhibition Road. London, SW7 2AZ, UK. Tel: +44 (0)20 7594 6884; Fax: +44 (0)20 7594 6883; Email: h.mistry@ic.ac.uk; http://www.ad.ic.ac.uk/cpd/nmr.htm

continued on inside back cover

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NOVARTIS

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

April 30, 2000
(received 5/8/2000)

"Screening Buffer Conditions for Protein NMR Samples"

Dear Dr. Shapiro,

In our attempts to identify appropriate buffer conditions for a 24 kDa Serine protease which has been shown by light scattering to exist as a trimer in solution, we employed the micro-drop screening method described by Lepre and Moore (*J. Biomolecular NMR. 12*, 493-499 **1998**). We screened a number of buffers (listed below) at temperatures of 25 °C and 40 °C. This method allows a large number of sample conditions to be tested using only sub-milligram quantities protein and was promoted as a means of increasing the efficiency of the search for NMR solvent conditions.

Protein solubility was observed in the following buffers:
100 mM NaAc pH 5.0; 100 mM NaAc pH 6.0; 100 mM NaAc pH 7.0.
100 mM MES pH 5.0; 100 mM MES pH 5.5; 100 mM MES pH 6.0; 100 mM MES pH 6.6.
100 mM Tris pH 6.5; 100 mM Tris pH 7.0; 100 mM Tris pH 7.5; 100 mM Tris pH 8.0.
100 mM HEPES pH 6.0; 100 mM HEPES pH 7.0; 100 mM HEPES pH 7.5;
100 mM HEPES pH 8.0.
100 mM KH_2PO_4 pH 5.0; 100 mM KH_2PO_4 pH 6.0; 100 mM KH_2PO_4 pH 7.0;
100 mM KH_2PO_4 pH 7.5; 100 mM KH_2PO_4 pH 8.0.
100 mM NaH_2PO_4 pH 5.0; 100 mM NaH_2PO_4 pH 6.0; 100 mM NaH_2PO_4 pH 7.0;
100 mM NaH_2PO_4 pH 8.0.
100 mM NaH_2PO_4, 2 mM CHAPS pH 5; 100 mM NaH_2PO_4, 2 mM CHAPS pH 6;
100 mM NaH_2PO_4, 2 mM CHAPS pH 7.
100 mM NaH_2PO_4, 2 mM n-octyl-β-D-glucoside pH 5;
100 mM NaH_2PO_4, 2 mM n-octyl-β-D-glucoside pH 6;
100 mM NaH_2PO_4, 2 mM n-octyl-β-D-glucoside pH 7.

Protein Precipitation was observed in the following buffer conditions:
100 mM NaH_2PO_4, 2 mM n-Dodecyl-β-D maltose pH 5;
100 mM NaH_2PO_4, 2 mM n-Dodecyl-β-D maltose pH 6 ;
100 mM NaH_2PO_4, 2 mM n-Dodecyl-β-D maltose pH 7.

The micro screening method was found to be an effective tool for identifying those conditions that cause precipitation of the protein, however this method was not effective in identifying those conditions that provide the best NMR spectral quality. In our case the protein exhibited high solubility in a number of buffers, however because of soluble aggregation and associated relaxation properties, this micro-drop screening method was not effective in identifying appropriate conditions needed to produce quality of ^{1}H, ^{15}N HSQC spectra.

Sincerely,

Xiaolu Zhang, Ph.D.

Nina C. Gonnella, Ph.D.

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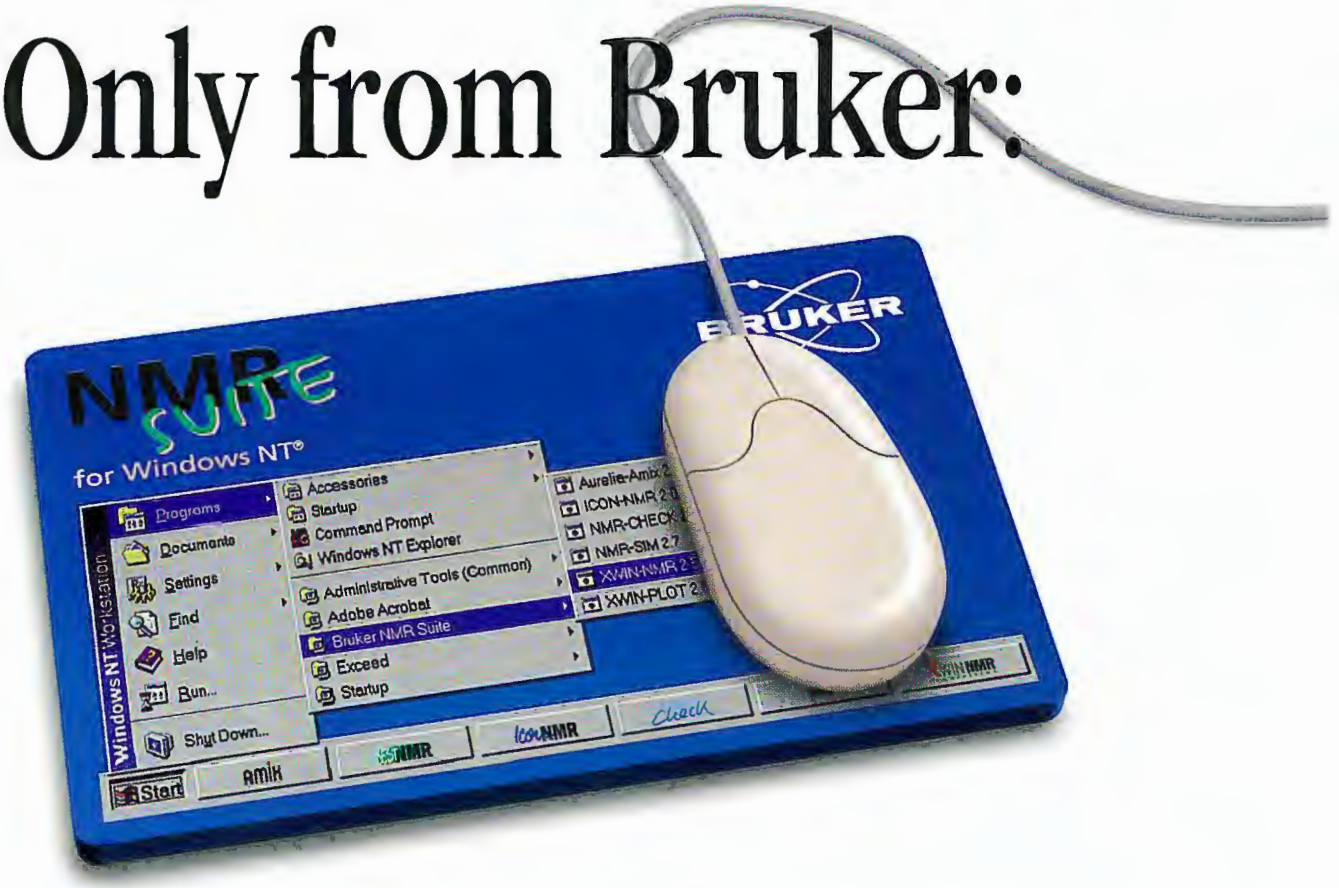

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Answers to some frequently asked questions about NMR Suite™ for Windows NT®:

Q: What exactly is NMR Suite for Windows NT?

A: NMR Suite consists of the following programs:

XWIN-NMR (spectrometer control and data processing)

XWIN-Plot (object oriented plot editor)

ICON-NMR (automation workhorse with icon driven desktop for laboratory management)

NMR-Sim (numerical simulation of NMR spectra)

NMR-Check (diagnostic software).

These programs run on a PC with Microsoft's Windows NT operating system, the same as they also run on a Silicon Graphics Inc (SGI) workstation under the IRIX operating system.

Q: Can NMR Suite for Windows NT control the spectrometer?

A: Yes! NMR Suite for Windows NT running on a PC has full control of the spectrometer and also does the data processing and data manipulation.

Q: Can I import spectra generated in NMR Suite for Windows NT into Windows programs such as MS Word or MS PowerPoint?

A: Yes! Our programs can write the plots into the Windows Clipboard or into a Windows Enhanced Metafile. From there the files can be imported into Word, PowerPoint and other Windows programs.

Q: Does NMR Suite for Windows NT replace NMR Suite running on the Silicon Graphics computers?

A: No! Bruker continues to support the SGI/IRIX platform. NMR Suite for Windows NT is an option, and the choice is yours!

Q: Is special hardware required for the PC to control the spectrometer?

A: No! The PC is connected to the spectrometer by a standard ETHERNET card. We require a second ETHERNET card to connect the PC to the INTERNET/INTRANET.

Q: Is NMR Suite for Windows NT "Year 2000 compliant"?

A: Yes! To learn more, please check our Year 2000 homepage at www.bruker.com/y2000.

Q: What minimum configuration do I need for the PC to run NMR Suite for Windows NT?

A: The following hardware is recommended:

- Pentium II/266 MHz
- 64 MB ram (128 MB recommended)
- 4 GB disk (SCSI recommended)
- PCI graphics card, 2MB, minimum 256 colors and 1024 x 768 pixel resolution
- 3.5" floppy, 1.44 MB
- CD ROM
- 3 button mouse for PS/2 port
- Keyboard
- 2 ETHERNET cards, 10/100 Mbit 3COM 3C905 PCI bulk
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Professor B. L. Shapiro
The NMR Newsletter
966 Elsinore Court
Palo Alto CA 94303, USA

May 15, 2000
(received 5/22/2000)

Title: Following enzymatic activity using NMR nano-probe technology.

Dear Barry,

We have previously described how using the 4 mm Varian nano-probe has allowed us to assign the structure of peptides attached to single polymer beads[1]. This prompted us to investigate the limits of detection using the nano-probe for solution studies of carbohydrates, which is another main interest of the group. The initial results of this effort demonstrated that using a carbohydrate separation system normally used for analytical purposes, High Performance Anion Exchange Chromatography (HPAEC, Dionex DX500 with CarboPac PA-100 analytical column), enough material could be isolated from single runs for ^{1}H NMR. This was demonstrated using arabinoxylan oligosaccharides from barley malt and typically between 3 and 30 nmol of material was isolated, which is sufficient to obtain good 1- and 2-D NMR spectra using a 500 MHz, 4 mm nano-probe[2].

These results lead us to investigate if the high sensitivity of the probe could be utilized also in studies of the specificity of carbohydrate hydrolyzing enzymes. The enzyme studied hydrolyses the linkage between arabinofuranose and the xylose backbone of arabinoxylan from barley. The enzyme is isolated from barley malt and the corresponding gene is not cloned, so only the naturally occurring isolated material is available. The oligosaccharides from the previous study represent substructures occurring in the intact barley polysaccharide and are therefore ideal substrates for the enzyme. The study has shown that it is possible to carry out enzymatic studies in the nano-probe and follow the hydrolysis of the substrates in the NMR spectrometer on appropriate time scale, typically over night. This is demonstrated in figure 1, where the enzymatic hydrolysis of two substrates is shown, and using several of this type of measurements, the specificity of the enzyme toward natural substrates has been determined.

Yours Sincerely

Anders Broberg Karl Kristian Thomsen Jens Ø. Duus

[1] C.H. Gotfredsen *et al.*, *J.Chem.Soc., Perkin Trans 1*, 2000, 1167-1171
[2] A.Broberg *et al.*, *Carbohydr. Res.*, 2000, in press

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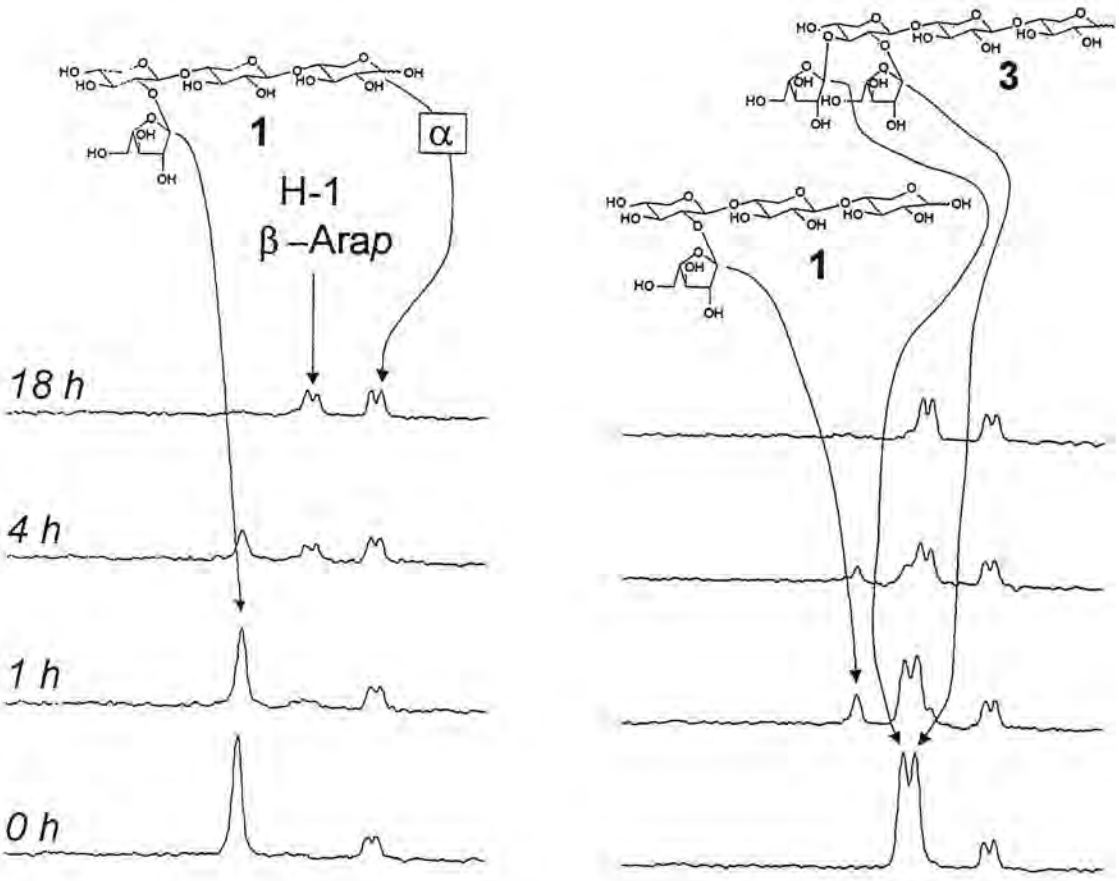

Figure 1

Enzymatic hydrolysis of oligosaccharides by an arabinoxylan arabinofuranohydrolase from barley malt. Approximately 30 nmol of substrate in 40 µl 50 mM $NaOAc\text{-}d_3$ buffer, pH 4.2 in D_2O, incubated with 1.3 mU enzyme at 27°C. Data obtained on a Varian UNITY INOVA 500 equipped with a ^1H only 4 mm nano-probe (spinning rate 2000 Hz).

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- Simultaneously view and manipulate the spectrum in 3D.
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- Attach the chemical structure and additional data to the spectrum.
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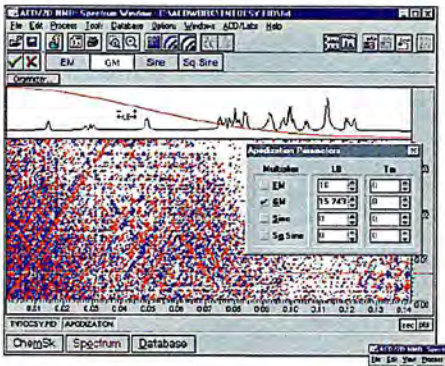
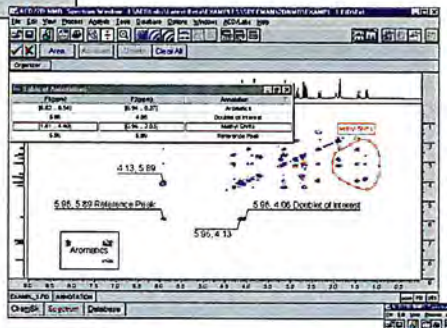
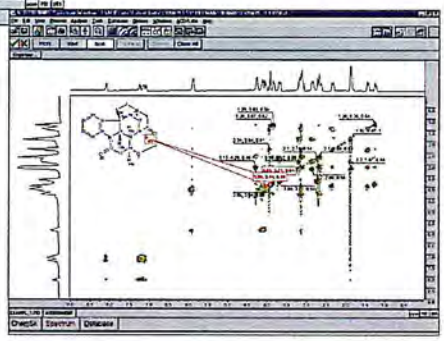


Interactive apodization mode

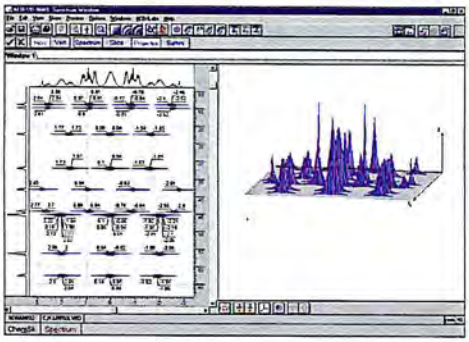


Annotate peaks & regions

Assign 2D spectra to structures



View in 3D from any perspective



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- Solvent suppression: filtering in frequency or time domain.
- Improved baseline correction: an additional automated method (Spectrum Averaging), a manual correction method (by multiple points and spline), as well as correction over the entire spectrum or region of interest.
- Editing of spectrum: an interactive spectral point adjustment, set a selected region to constant value, or cut off a selected region of interest.
- Improved reference capabilities. Autodetect TMS and solvent peak(s) in ^{1}H and ^{13}C NMR spectra.
- Adjustment of manual phasing speed option.
- Improved AutoSimple phasing routine.
- Pop-up window for chemical structure.
- Flexible zoom for calculated and experimental spectra.
- Option for displaying any spectrum parameter or user data value on the status bar.
- Enhanced support for VNMR and XWINNMR data formats. Import the table of integral ranges for processed spectra. Import the phase parameters for VNMR and XWINNMR acquisition data.
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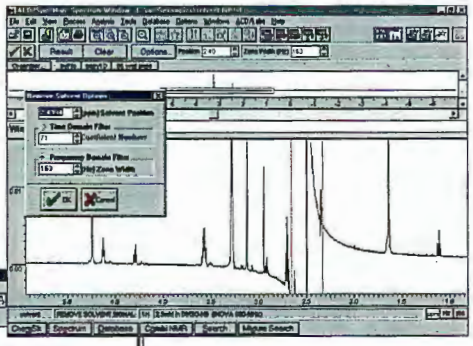

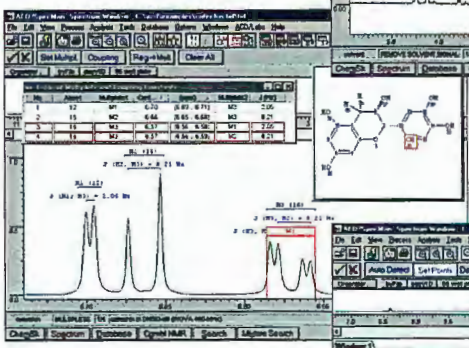

Advanced processing includes multiplet analysis

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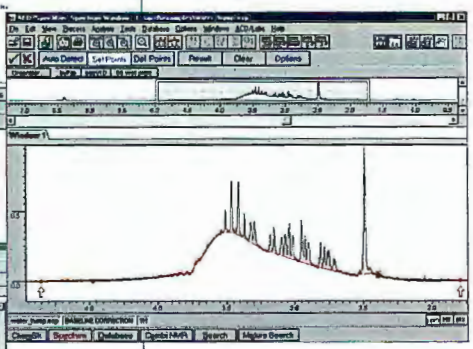

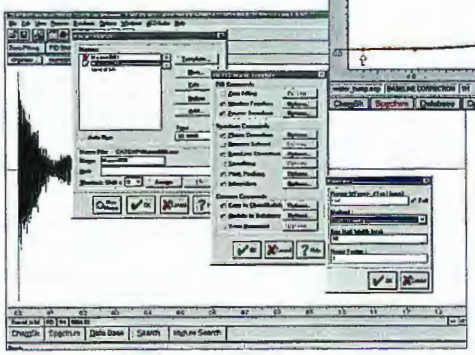

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Laboratory of Biomolecular Structure and Dynamics

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

(received 4/28/2000)
April 28, 2000

^{15}N and ^{1}H CSA in Hydrogen-bonded GC base pair

Dear Barry:

As you might remember, in our previous report we discussed the TROSY merits in triple-resonance HCN experiments designed to correlate the sugar H1' with the base H8/H6 protons in ^{13}C/^{15}N labeled nucleic acids. In order to gain insight into the efficiency of various relaxation processes, we calculated the ^{1}H and ^{13}C chemical shielding tensors of purine and pyrimidine bases using *ab initio* approach based on the sum-over-states density functional perturbation theory. The results and comparison of TROSY and multiple-quantum suppression of the proton-carbon dipol-dipol relaxation have been presented in detail in our recent paper[1] in Journal of Biomolecular NMR. Since we had positive experience also with the *ab initio* calculation of chemical shifts and spin-spin coupling constants in anhydrodeoxythimidines[2,3] we have decided to look at the influence of hydrogen bonding on the ^{1}H and ^{15}N chemical shift anisotropy in nucleic acid base pairs. Motivation for such a study was quite straightforward.

As you know, successful interpretation of ^{15}N NMR relaxation data requires an accurate knowledge of the chemical shift anisotropy (CSA). So far, the ^{15}N relaxation data has been interpreted based on assumptions that the ^{15}N shielding tensor is axially symmetric, its axis of symmetry is collinear with the ^{15}N-^{1}H dipolar tensor and that the CSA values are uniform throughout the molecule. These assumptions, however, are not generally valid. For non-collinear tensors, the highly anisotropic molecular rotation results in differences in site-specific correlation functions and spectral densities. In addition, the non-collinearity significantly affects the efficiency of dipolar and CSA compensation in TROSY experiments.

The hydrogen bonding effects on the ^{1}H isotropic chemical shift have been known, and understood in a qualitative fashion, for a long time. A detailed examination[4] of 77 A, B and Z-DNA crystal structures revealed that imino hydrogen bond length (N...N) varied between 2.7 and 3.1 Å with a small number of very short values (2.2 – 2.6Å). The observation of variations in ^{15}N – ^{15}N scalar couplings ($^{2}J_{NN}$) across the Watson-Crick base pairs[5,6], and *ab initio* calculations of the $^{2}J_{NN}$ dependence on the N...N distance indicates that the length of a hydrogen bond also changes in solution. The recently published methods[7,8] determining ^{15}N CSA and θ indirectly from the measured relaxation data open up a possibility to assess the length of the NH ...H hydrogen bond providing the distance dependence of the ^{15}N chemical shielding parameters can be established independently. Since the influence of hydrogen bond length on ^{1}H and ^{15}N shielding anisotropy cannot be studied experimentally in a systematic way, the theoretical approach remains the only possibility.

Here we summarize the basic conclusions of our study:

- The calculated isotropic chemical shifts of both the imino nitrogen and the proton decrease with the increasing distance between G and C. This is in agreement with experimental values. The tendency is easily understood in terms of the weakening of the hydrogen bond with distance. The longer the hydrogen bond, the higher the shielding of the imino group (as a result of decreasing charge transfer between G and C) which manifests itself in the lowering of the chemical shifts.
- Significantly more pronounced is the effect of the hydrogen bond length on the CSA values of the hydrogen and nitrogen nuclei. As the length changes from 2.57 Å to ∞, the absolute value of $|CSA|$ decreases by about 35 and 27 ppm for ^{15}N and ^{1}H, respectively. The dependence of both CSA(N) and CSA(H) values on the

hydrogen bond length can be approximated by an exponential. However, the ^1H shielding depends strongly on stacking interactions.

- Not only the absolute value of CSA, but also the orientation of the ^{15}N chemical shift tensor depends on the length of the hydrogen bond. For a nitrogen nucleus not involved in hydrogen bonding, the shielding tensor is nearly axially symmetric with asymmetry factor $\Delta\eta$=0.15, $\sigma_{\parallel}\equiv\sigma_{11}$ is almost collinear with the bond vector (θ_1=9.0°) and the most shielded component is almost perpendicular to the plane of the G base. As the length of the hydrogen bond decreases the least shielding component σ_{11} deflects from the N-H vector and the shielding tensor becomes increasingly asymmetric. For a hydrogen bond typical length of 2.87 Å, the angle θ_1 reaches the value of 18.9°. At shorter hydrogen bond lengths, the larger value of θ_1 more than compensates for the greater absolute value of CSA.
- The orientation of the ^1H shielding tensor is rather different from that of ^{15}N. Here, the tensor is not even approximately axially symmetric for any length of the hydrogen bond and the shielding component closest to the N-H bond is σ_{33}. The angle, θ_3, between σ_{33} and the N-H bond is very small and neither the orientation of the shielding tensor nor its asymmetry factor depend significantly on the hydrogen bond length.
- The non-collinearity of the ^{15}N chemical shift and $^1H - ^{15}N$ dipolar tensors in combination with the anisotropy of the overall molecular motion has an effect on the TROSY linewidth, especially at very high magnetic fields. The effect is not dramatic, however, and will probably be noticeable only at very high (800 MHz) fields. The significantly lower value of $\Delta\sigma^*$ for G imino (~90 ppm) compared to that for the amide proton in proteins (~140 ppm) suggests that the efficiency of TROSY for ^{15}N in G is lower and requires higher fields. Our calculations show that for $r_{N\ldots N} \rightarrow \infty$ the optimum requires as high a field as 1.7 GHz. Only for very short hydrogen bond lengths (2.57 Å), does the optimal frequency approach 800 MHz.

More detailed account of our study will be published elsewhere[9].

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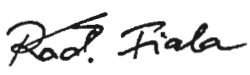
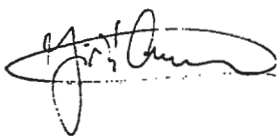
Best regards,

Radovan Fiala



Jiří Czernek



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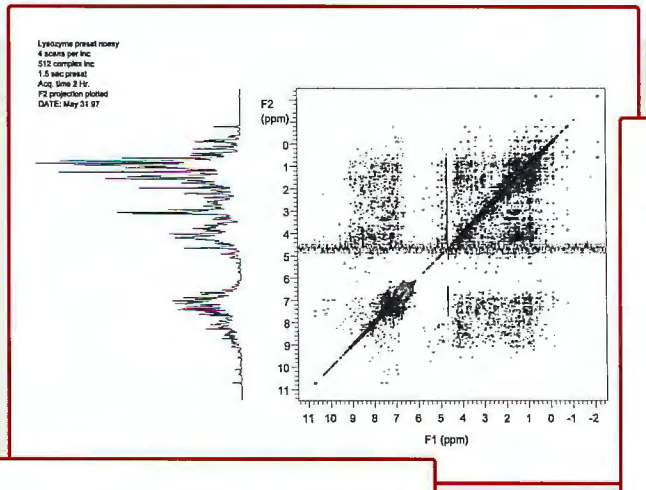

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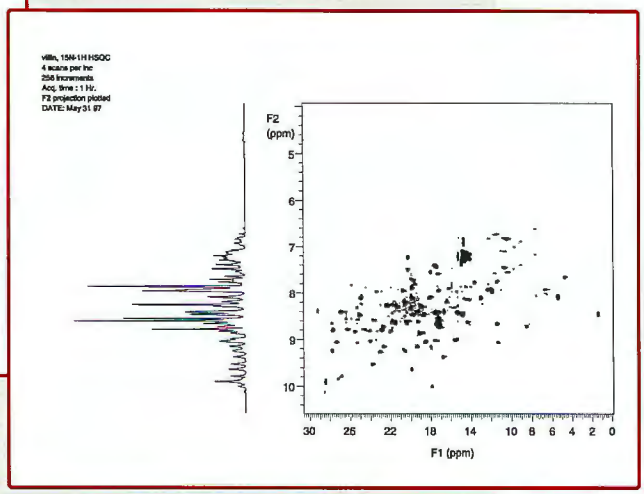

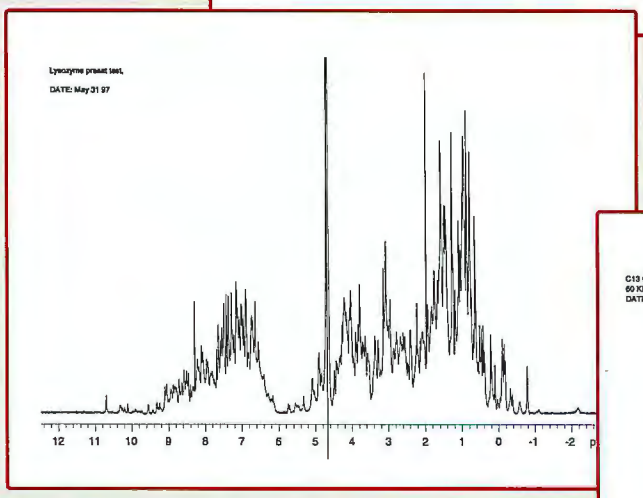

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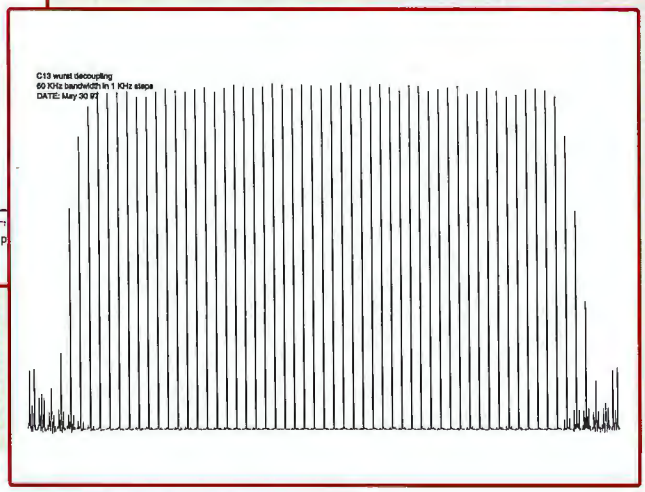

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(received 5/16/2000)

Troubleshooting Deuterium Gradient Shimming

Dear Barry,

The possibility of using the deuterium signal to adjust all the z shims has really been one of the most significant improvements in the last two years.
Nevertheless, you may encounter a few simple problems, if you use this technique. Here is a short summary of our experience using deuterium gradient shimming with Bruker Advance spectrometers.

1. In the start window of the gradshim procedure there is one field called "user". The default value for this field is "gradshim". If you use this default value, you run into a couple of problems concerning file system access rights. Most of the error messages sound very strange. Change this entry to an existing user (we use "guest" at all of our spectrometers).
2. The parameters for the gradient shim procedure are sometimes not optimal under all circumstances. To change them read in the parameter set (rpar gradshim1d2h), modify the appropriate parameters (i.e. increase NS to 16, increase D1 to 7s or higher, modify P1) and write the parameter set back (wpar gradshim1d2h) to disk.
3. If you don't observe any signal during the field map acquisition or the deuterium shimming itself, there may be a routing error. To check this, once again read in the shimming parameter set (see above) and check the routing (edasp). Finally, the output of the X transmitter must be connected with the 2H preamplifier. If there is a connection to the BB preamplifier, change the routing manually and write the modified parameter set back to disk.
4. If you don't have either a deuterium switch or the specialized 20W deuterium transmitter within the BSMS housing, don't worry. Of course you need at least the equipment to work with z-gradients. With a little bit extra cable work you can use deuterium shimming too. How is this done? Disconnect the X transmitter cable from the backside of the BB preamplifier module. Then disconnect the transmitter cable from the backside of the 2H module and attach the X transmitter cable to the backside connector of the 2H module. Now you are ready to perform deuterium shimming. Don't forget to return to standard wiring prior to any serious measurements

Sincerely

Rainer Haeßner Gerd Gemmecker Tom Malia

(Please contribute the article to Frank Koehler's account)

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The NMR Newsletter
966 Elsinore Court
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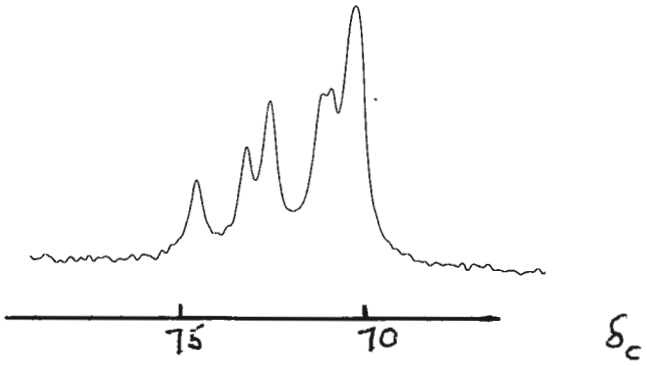
USA

May 12,2000
(received 5/22/2000)

Title: Solid State ^{13}C NMR Spectrum of 30-Crown-10 Ether

After 18-crown-6, 30-crown -10 is the next largest unsubstituted crown system for which a single crystal X-ray structure has been reported[1]. The asymmetric unit in the crystal is one half the molecule-ie. ten carbons. In view of our continuing interest[2] in the dynamic stereochemistry of macrocyclic polyethers in the solid state and in solution, Research Associate Majid Rastegar has prepared 30-crown-10 and looked at its ^{13}C CPMAS spectrum at 50.3 MHz. In contrast to the solution ^{13}C spectrum where only one line is seen, the solid phase spectrum is shown below.



There are six distinct resonances in the ratio of 4:1:1:2:1:1 with an overall shift range of ca. 5 ppm. This is consistent with the constitution of the asymmetric unit in the crystal. Notably, the dipolar dephased spectrum shows no residual intensity, indicative of an effectively rigid system on this timescale. This is in contrast to our early findings for 18-crown-6 where large amplitude motion occurs in the solid. A detailed analysis of the spectrum is being carried out using the individual torsional environments of each unique site in the asymmetric unit as a starting point and looking at gauche vs. trans effects etc.

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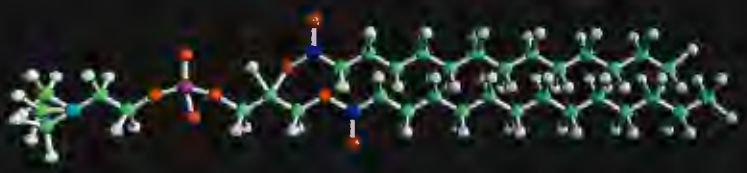

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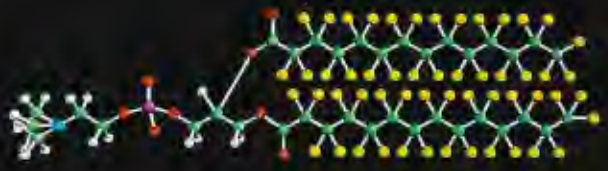

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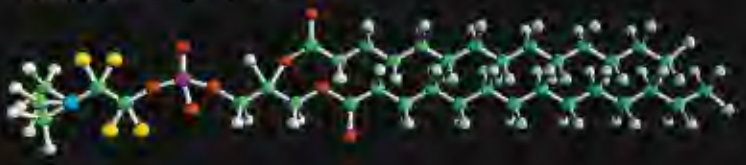

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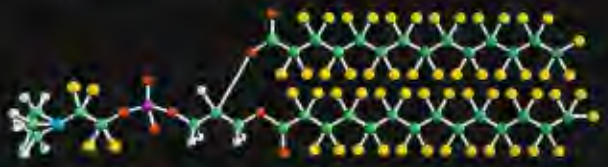

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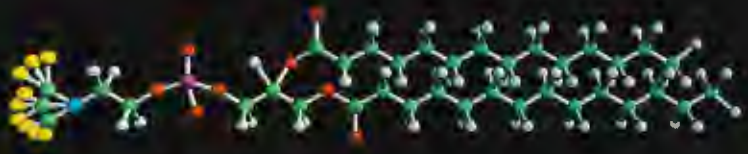

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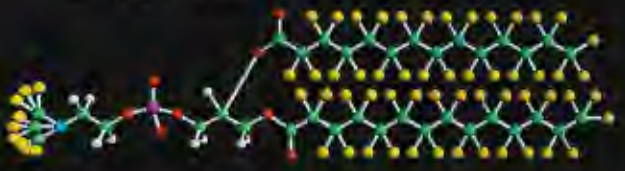

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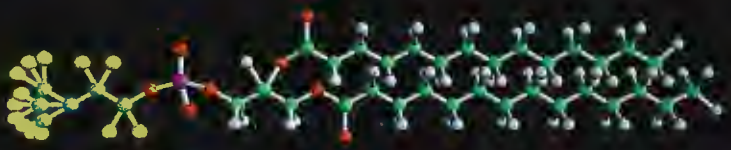

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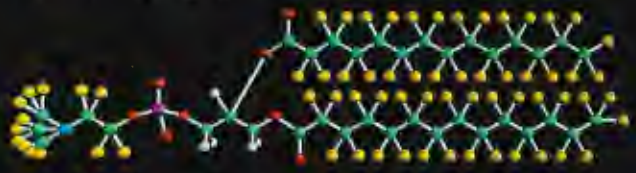

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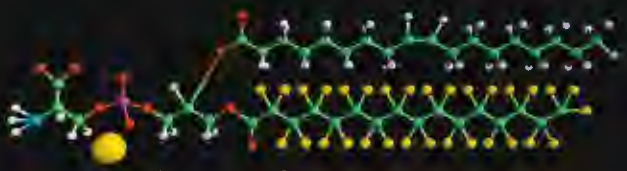
Asymmetric Fatty Acid



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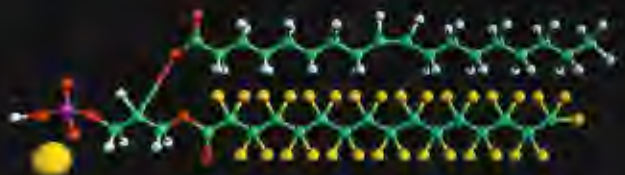

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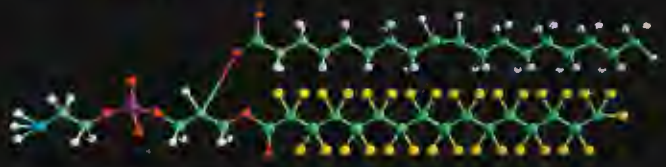

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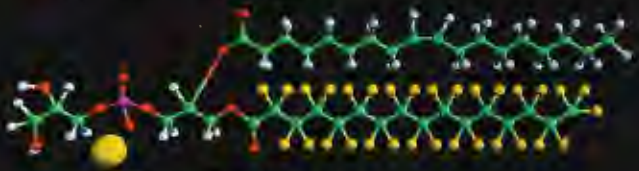

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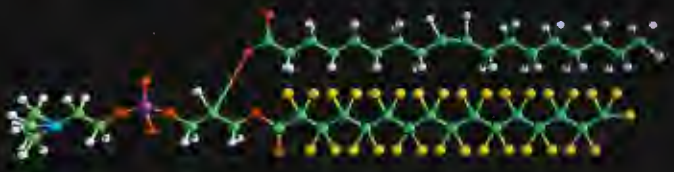

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Dr. B.L. Shapiro
The NMR Newsletter
966 Elsinore Court
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(received 5/4/2000)
March 24, 2000

High Resolution NMR from Paramagnetic Solids

Dear Barry,

Solution-state NMR of paramagnetic proteins and in the presence of lanthanide shift reagents are well known, under the right conditions, to give high resolution spectra with large isotropic paramagnetic shifts (contact and pseudo-contact shifts).[1,2] Solid-state ^{13}C NMR of lanthanide acetates undergoing magic angle spinning (MAS) have also been shown to give surprisingly high-resolution spectra.[3] Ann McDermott and coworkers[4] noticed that the measured carbon linewidth was invariant to the presence or absence high-power ^{1}H decoupling while Grey, Dobson and coworkers[5] noticed that deuterated samples gave rise to much narrower spectra. These seemingly contradictory results arise from the large distance and orientation dependent paramagnetic shifts of the ^{1}H spectra. These shifts are due to the classical magnetic field produced by the paramagnetic lanthanide atom and are strongly position dependent. Neighboring ^{1}H's can experience significantly different paramagnetic shifts effectively decoupling the flip-flop interaction between them and allowing MAS to remove a large portion of the CH dipolar coupling. When high power constant wave (CW) decoupling is applied to these systems, residual broadening occurs due to the interference of off-resonance effects and the residual H-H couplings. CW decoupling does not effectively remove this type of interaction.[6]

Two-pulse phase modulated (TPPM) decoupling is a recent improvement in high power decoupling for solid-state NMR.[7] This decoupling sequence applies a series of approximately 180° pulses phase switched by 14° to the ^{1}H channel resulting in improvement of the off-resonance effects of decoupling.

I have been performing a series of solid-state NMR experiments on the microporous, MIL-8 lanthanide glutarates, $[Ln(H_2O)_2][O_2C(CH_2)_3CO_2]_3 \cdot 4H_2O$ with Ln = Y, Nd, Sm, Eu, Gd, Tb, Dy, Ho, and Er.[8,9] The ^{13}C CPMAS spectra of the Y-glutarate diamagnetic analog of the MIL-8 series shows three resolved aliphatic resonances and three carbonyl resonances (figure 1a, full spectrum, and 1d, carbonyl region expansion). This CPMAS (ν_{rot} = 15kHz, contact time = 4ms) spectrum was taken on a Bruker

DMX400 system with 71kHz TPPM decoupling. Figure 1b shows the ^{13}C CPMAS spectrum of Sm glutarate taken with 89kHz CW decoupling, a 1 ms contact time, and a rotation rate of 15kHz. Figure 1e is the carbonyl region expansion of this spectrum. Figure 1c and 1f show the Sm glutarate spectrum taken under the same conditions as before but with TPPM decoupling. These spectra show that TPPM decoupling significantly improves the resolution over the same power CW decoupling. The TPPM decoupled spectrum begins to approach the resolution seen in the diamagnetic sample. The expansion in the carbonyl region of the three spectra (Figure 1d-f) dramatically demonstrates this improvement. It shows that three resonances can be resolved in Sm glutarate when TPPM decoupling is used and that they are significantly shifted from the carbonyl resonances in Y-glutarate.

Utilization of off-resonance compensated, high-power decoupling sequences, like TPPM, in paramagnetic samples is necessary if high-resolution solid-state NMR are going to be produced. High decoupling fields will still be required and for paramagnetic samples with large electronic magnetic moments 100's of kHz decoupling fields will be necessary. Under these conditions and if the paramagnet acts like a true classical magnet ($T_{1e} < 10^{-10}$ sec), high resolution solid-state NMR spectra can be produced. Analysis of these spectra gives the isotropic paramagnetic shift and paramagnetic shift anisotropy, which can provide detailed structural information about the environment around the paramagnet. [10]

Sincerely,

Joe Sachleben

Joseph R. Sachleben

Acknowledgements

I would like to thank A. Cheetham, G. Férey, F. Serpaggi, and T. Luxbacher for graciously providing the MIL-8, Lanthanide glutarate samples to me.

---

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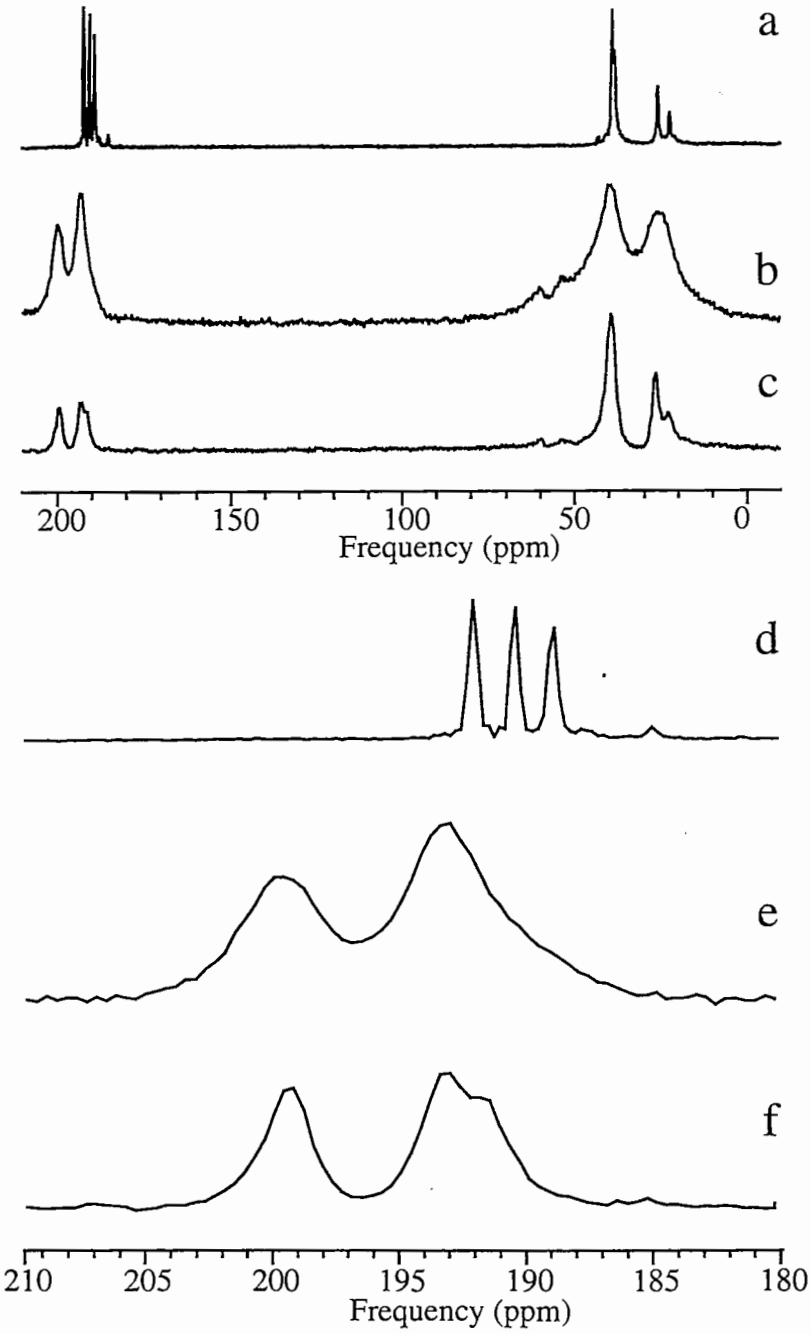


Figure 1: ^{13}C NMR of Y and Sm Glutarate. All spectra taken on a Bruker DMX400 NMR Spectrometer. a) Full spectrum of Y-Glutarate with 71kHz tppm decoupling. b) Full spectrum of Sm-Glutarate with 89kHz CW decoupling. c) Full spectrum of Sm-Glutarate with 89kHz tppm decoupling. Resolution is much improved over 1b. d) Carbonyl region expansion of Y-glutarate spectrum. e) Carbonyl region expansion of Sm-glutarate spectrum with 89kHz CW decoupling. Only two carbonyl resonances are resolved. f) Carbonyl region expansion of Sm-glutarate spectrum with 89kHz tppm decoupling. All three carbonyl resonances are resolved.

May 20, 2000 — FOR IMMEDIATE RELEASE
Contact: Luis Moreno, Marketing Manager @ 800-448-9760 x 266

Isotec Doubles Production Capacity for ul-Labelled D-Glucose

ISOTEC. Inc. announced today the expansion of their production capacity for stable isotope ul-labelled D-Glucose and Algal Products. The expansion is expected to double Isotec's current production capacity of these important intermediates used in the pharmaceutical industry for the Genome Project, Drug Design, and Metabolic Research. "Feedback about increased ul-labelled D-Glucose demand from researchers at the ENC Conference in Asilomar, California was a major factor in the decision to ramp-up production immediately", said Rick Baeder, Isotec's General Manager. "ISOTEC is committed to producing an ample supply of ul-labelled D-Glucose and related products for the continued support of Biomolecular NMR, Metabolism, and Drug Discovery researchers."

A significant scale-up in D-Glucose-$^{13}C_6$ production capacity has already been completed and, by year-end, an additional capital expansion project, already underway, will double production of Isotec's D-Glucose-$^{13}C_6$; -$^{13}C_6$,C-d_7; -C-d_7 and ul-Amino Acids output.

ISOTEC, Inc. is the world's leading commercial producer of enriched stable isotopes and a leading manufacturer of stable isotope labelled compounds. Its products have been utilized in numerous research projects in the fields of nutrition, pharmaceutical drug development, medical imaging, and diagnostic research. "Demand for stable isotopes in cutting edge medical research continues to increase, and Isotec is committed to providing the labelled compounds required to help the research market reach its goals" said Dr. C. T. Tan, Senior Vice-President Chemical Operations at Isotec.

ISOTEC, Inc. has been providing labelled chemical synthesis technology expertise to the research community ever since it became the first commercial producer of stable isotopes in 1980. It continues to service the research community, not only by meeting customers demands for ul-labelled D-Glucose products, but also by supplying other important labelled products for clinical research such as D-Glucose-6,6-d_2 for HIV research and D-Glucose-1-^{13}C for metabolic studies along with many other uniquely labelled research products.

100- 24

Académie des Sciences de Tahiti
Service de la Physique Expérimentale
et Résonance Magnétique
Avenue Louis de Bougainville, 13
Papeete

le 4 janvier 1967

M. le Professeur B. L. SHAPLIERO
Department of Chemistry
Illinois Institute of Technology
Chicago, Illinois 60616 U.S.A.

Mon cher Barrie,

Lors de votre récent passage à notre laboratoire, vous avez sollicité une communication digne de la centième édition de l'I.I.T.N.M.R.

Depuis quelque temps nous étudions les signes relatifs des constantes d'intéraction spin-spin à longue distance dans certains extraits de l'huile de coprah. Au cours de ce travail nous avons éprouvé quelques difficultés à établir le réseau des niveaux d'énergie pour certains systèmes de deux groupes de spins nucléaires, par exemple A_2X_3 (selon la nomenclature de POPLE). Le problème a été résolu par une nouvelle méthode de construction graphique qui s'explique par l'exemple suivant.

On trace d'abord sur le graphique (Fig. 1) le profil du spectre A_2X_3, se rappelant qu'un quadruplet 1:3:3:1 doit être décomposé en quadruplet (1:1:1:1) auquel on superpose un doublet (2:2) par dessus les deux lignes centrales, tandis qu'un triplet (1:2:1) est décomposé en triplet (1:1:1) avec un singulet central. Ainsi chaque résonance est en réalité deux sous-spectres[1] qui doivent être traités séparément.

Avec un compas centré sur chaque ligne d'un sous-spectre A quelconque, on trace des arcs qui se coupent avec d'autres arcs (à rayon r égal) centrés sur les lignes d'un sous-spectre X. Les intersections de ces arcs définissent les niveaux d'énergie, et, dans ce cas particulier de deux sous-spectres A et deux sous-spectres X, établissent quatre systèmes indépendants de niveaux (voir la figure ci-contre). Les transitions entre niveaux d'énergie peuvent être identifiées par l'origine de l'arc générateur.

Par dessus le marché, la méthode se prête facilement à la prédiction des spectres à deux quanta. Chaque parallélogramme sur le graphique engendre une transition à deux quanta dont la fréquence correspond à la mi-hauteur du parallélogramme (on doit tourner la figure pour que la direction énergie soit verticale). Le cas A_2X_3 donne naissance à un quadruplet de transitions à deux quanta, avec les intensités relatives 1:4:4:1, en accord avec l'expérience de KAPLAN et MEIBOOM[2] sur l'alcool éthylique.

Cette méthode s'étend sans difficultés à d'autres systèmes à couplage faible, A_nX_m. On laisse au lecteur le soin de construire (en deux dimensions) les graphiques appropriés aux systèmes $A_nM_mX_p$ (trois groupes de spins inégaux).

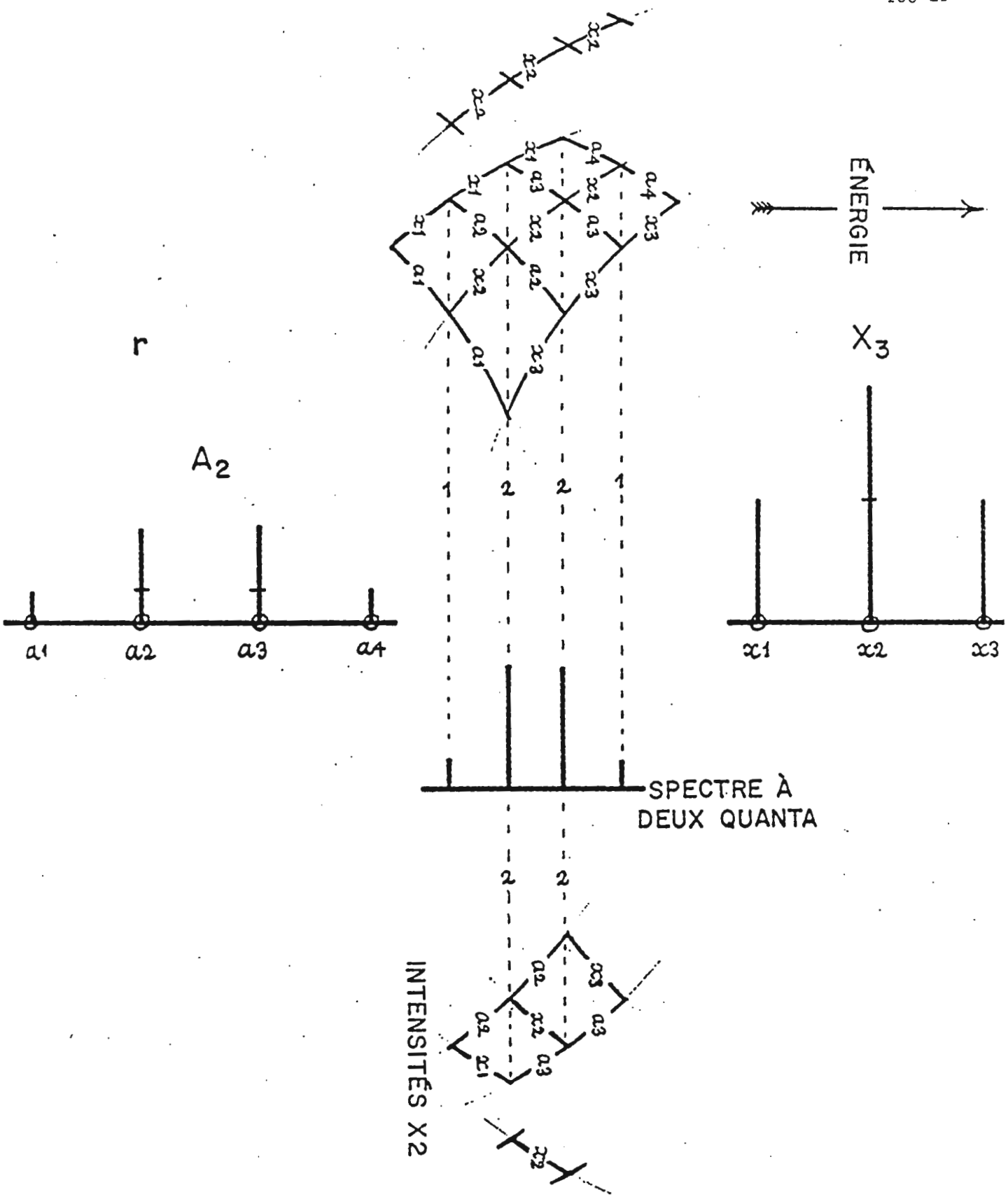
Sincères salutations,

Sue

(Mlle) Suzanne Perchery

1. Voir par exemple P. DIEHL, 6 ième Congrès de la R.M.N. Expérimentale (6th E.N.C.) Pittsburgh, 1965; Helv.Chim.Acta, 48, 567 (1965).
2. J. I. KAPLAN et S. MEIBOOM, Phys.Rev. 106, 499 (1957).

Titre: Niveaux d'énergie A_nX_m.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INTENSITÉS X2

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Deadline Dates

No. 502 (July)	21 June 2000
No. 503 (Aug.)	25 July 2000
No. 504 (Sept.)	24 Aug. 2000
No. 505 (Oct.)	27 Sept. 2000
No. 506 (Nov.)	27 Oct. 2000

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Forthcoming NMR Meetings, continued from page 1:

42nd ENC (Experimental NMR Conference), Clarion Plaza Hotel, Orlando, Florida, **March 11-16, 2001**; Arthur G. Palmer, Chair: Agp6@columbia.edu; Contact: ENC, 1201 Don Diego Avenue, Santa Fe, NM 87505; (505) 989-4573; Fax: (505) 989-1073; E-mail: enc@enc-conference.org. Web: enc-conference.org

Royal Society of Chemistry: 15th International Meeting on NMR Spectroscopy, Durham, England, **week of July 8-13, 2001**; Contact: Mrs. Paula Whelan, The Royal Society of Chemistry, Burlington House, London W1V 0BN, England; +44 0171 440 3316; Email: conferences@rsc.org\

ISMAR 2001, Jerusalem, Israel, **August 19-24, 2001**;
See http://www.tau.ac.il/chemistry/ISMAR.html.

Additional listings of meetings, etc., are invited.

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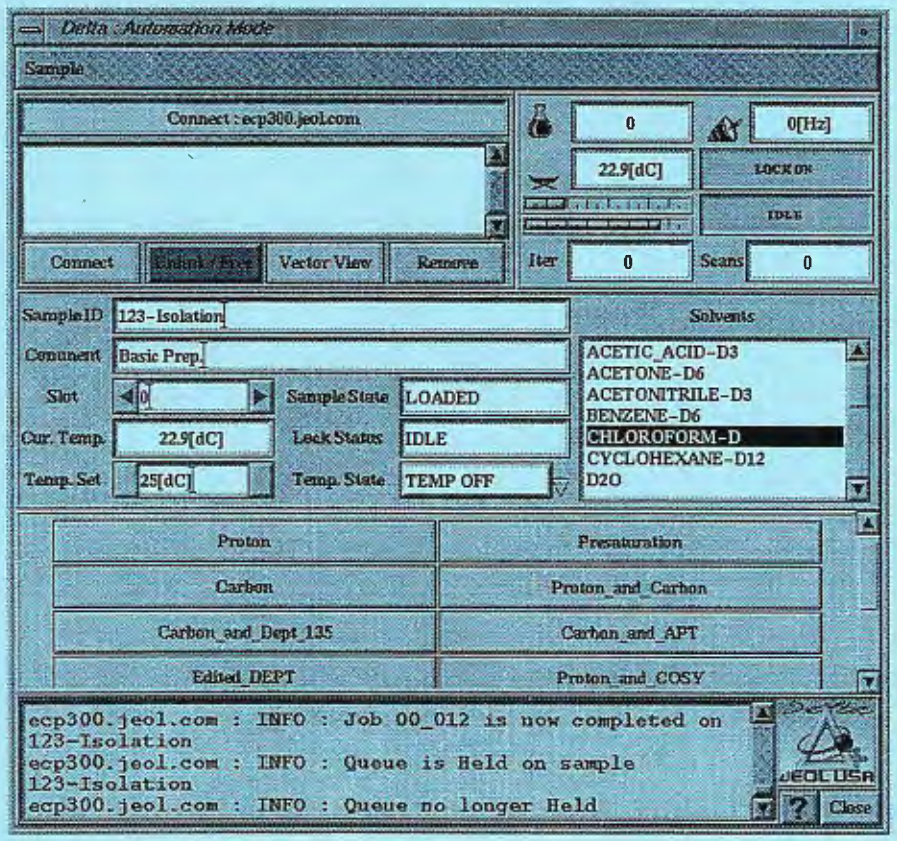

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