THE NMR NEWSLETTER

No. 468

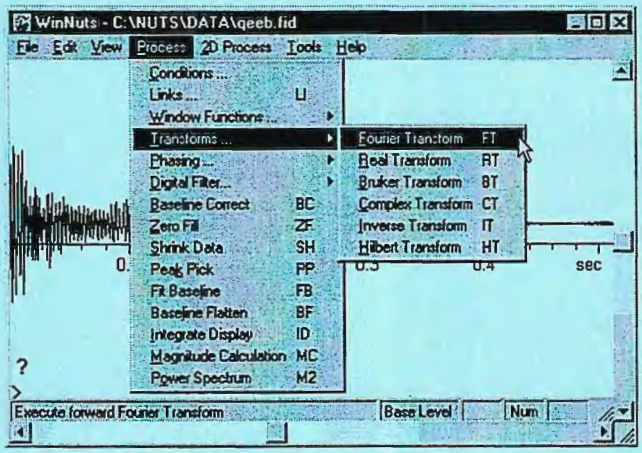
September 1997

Now NUTS is easier to learn than ever!

NUTS Help files are now on the web, including:

* ability to search Help files
* description of every NUTS command, organized by topic
* step-by-step instructions for NUTS operations, illustrated with screen captures
* sample macros with explanation of each line

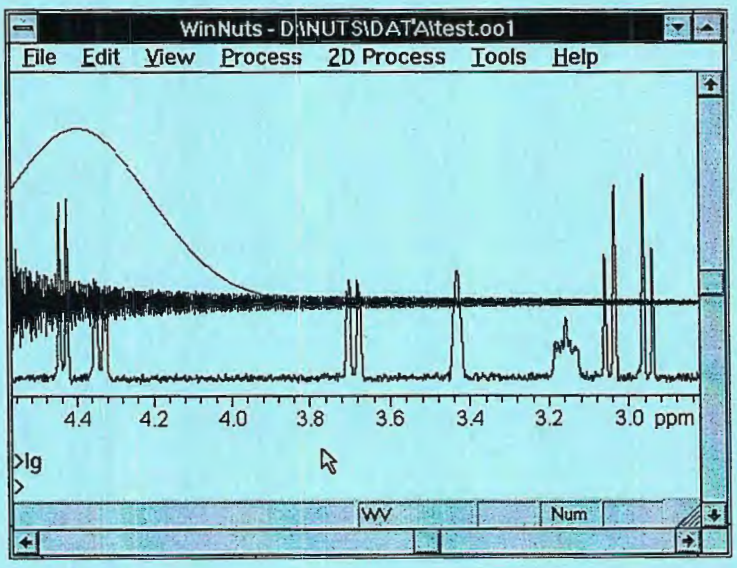
http://www.acornnmr.com/NutsHelp



Screen captures and accompanying explanations teach users how to perform each operation.

New! Interactive adjustment of apodization functions

FID, window function and chosen region of the spectrum are displayed simultaneously. As the window function is changed, the spectrum is updated automatically.



Acorn NMR Inc.
46560 Fremont Blvd. #418
Fremont, CA 94538

(510) 683-8595
(510) 683-6784 FAX
info@acornnmr.com

ftp.acornnmr.com
http://www.acornnmr.com

SPONSORS OF *THE NMR NEWSLETTER*

Abbott Laboratories
Aldrich Chemical Company, Inc.
AMT
Amgen, Inc.
Anasazi Instruments, Inc.
Astra AB
Bruker Instruments, Inc.
Cambridge Isotope Laboratories
Cryomag Services, Inc.
The Dow Chemical Company
E. I. du Pont de Nemours & Company
Eastman Kodak Company
Hewlett-Packard Company
Isotec, Inc.
JEOL (U.S.A.) Inc., Analytical Instruments Division
Kontes Glass Company
The Lilly Research Laboratories, Eli Lilly & Company
Merck Research Laboratories
Nalorac Cryogenics Corporation
Otsuka Electronics USA Inc.
Oxford Instruments
Pharmacia and Upjohn, Inc.
Programmed Test Sources, Inc.
SINTEF Unimed MR Center, Trondheim, Norway
Tecmag
Unilever Research
Union Carbide Corporation
Varian NMR Instruments

FORTHCOMING NMR MEETINGS

New Directions in NMR: A Symposium Honoring Aksel A. Bothner-By, Irving J. Lowe, Joseph Dadok, and Robert T. Schumacher, Pittsburgh, PA, **Sept. 20, 1997**. See Newsletter 467, 43.

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.

Missouri Magnetic Resonance Symposium (MMRS-VII), Tan-Tar-A Lodge, Lake of the Ozarks, Osage Beach, MO, **October 31, 1997**. Contact: Frank D. Blum, Department of Chemistry, University of Missouri-Rolla, Rolla, MO 65409-0010; 573-341-4451, fblum@umr.edu, http://www.chem.umr.edu/midwest32.html. See Newsletter 467, 39.

Symposium "Magnetic Fields: Recent Advances in Diagnosis and Therapy", London, Ont., Canada, **November 14 - 16, 1997**. See Newsletter 468, 8.

39th ENC (Experimental NMR Conference), Asilomar Conference Center, Pacific Grove, CA, **March 22 - 27, 1998**; Contact: ENC, 1201 Don Diego Avenue, Santa Fe, NM 87505; (505) 989-4573; Fax: (505) 989-1073. See Newsletter 460, 41.

Sixth Scientific Meeting and Exhibition, International Society for Magnetic Resonance in Medicine, Sydney, Australia, **April 18 - 24, 1998**. Contact: International Society for Magnetic Resonance in Medicine, 2118 Milvia St., Suite 201, Berkeley, CA 94704; 510-841-1899.

Fifth International Conference on Heteroatom Chemistry, London, Ont., Canada, **July 5 - 10, 1998**. For details, see Newsletter 468, 40.

XIVth International Conference on Phosphorus Chemistry, Cincinnati. OH, **July 12 - 17, 1998**. For details, see Newsletter 468, 40.

Additional listings of meetings, etc., are invited.

Duke University
Duke Nuclear Magnetic Resonance Spectroscopy Center

Leonard D. Spicer, Director
Anthony A. Ribeiro, Manager

919 684 4327
919 613 8887

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

August 17, 1997
(received 8/22/97)

Re: Diagnostic Entry Points for NMR Analyses of Spongian Compounds

Dear Barry,

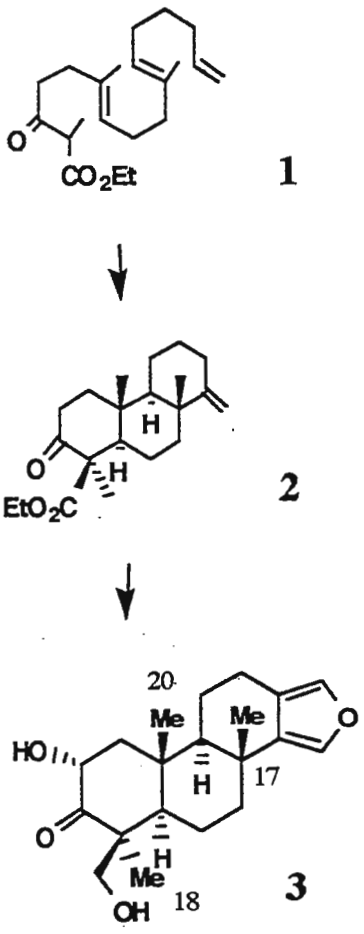
In addition to bio- and perfluorinated molecule interests, we are working with Prof. Phil Zoretic (Chemistry, East Carolina Univ.) to study biologically-interesting, multicarbocyclic rings derived from novel, free radical cyclizations of polyenes (Tetrahedron Lett. 36 2925 1995; 36 2929 1995; 37 1751 1996; J. Org. Chem. 61 1806 1996). Stereo-selective tandem cyclization of **1** e.g. introduces in one step five chiral centers and a C-4 pro-β-hydroxymethylene in spongian intermediate **2**. **2** is a synthon towards the rare marine sponge furanoditerpene, isospongiadiol **3**, which shows activity against Herpes virus, type 1.

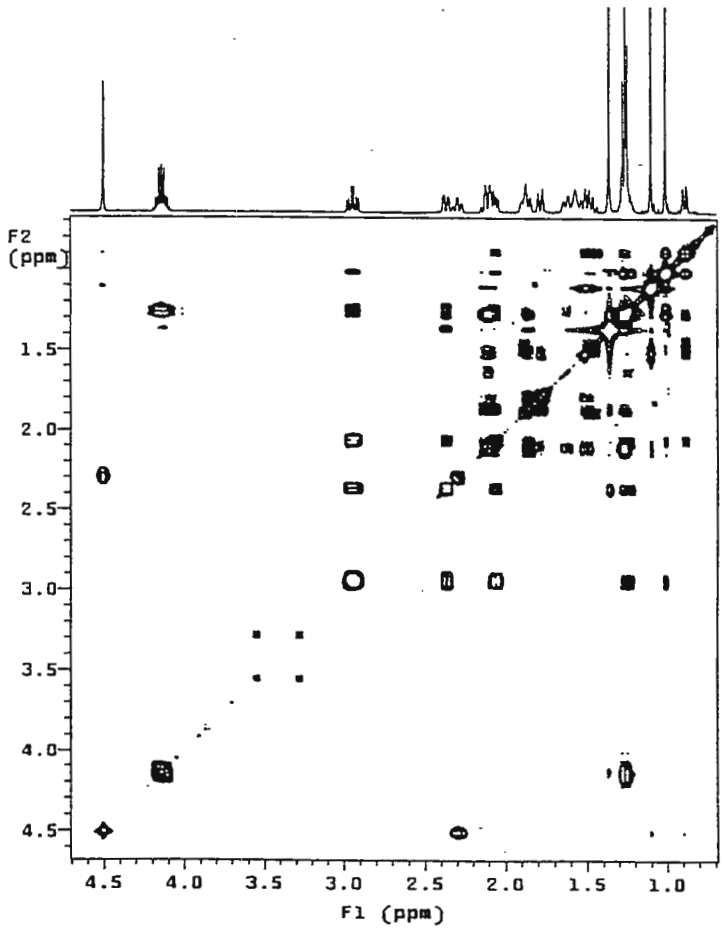
We have found four- and five-bond connectivities detected in a COSY variant to be diagnostic in differentiating the 17-, 18- and 20-CH_3 resonances and to serve as useful entry points for the NMR analyses. The 1.02 ppm methyl singlet in **2** (Fig. 1) shows four or five bond cross peaks to H-2β, H-1β and H-1α (2.95, 2.06 and ~1.27 ppm) which establishes its assignment to the angular 20-CH_3 group. Similar long range connectivites establish the 1.11 ppm and 1.37 methyl signals as the angular 17-CH_3 and equatorial 18-CH_3 groups. The ability to detect these weak couplings is greatly aided by the excellent stability of our Varian spectrometers. The differentiation of the methyls is crucial as the 20- and 17-CH_3 resonances reverse their relative chemical shift positions in various spongian compounds.

Regards,

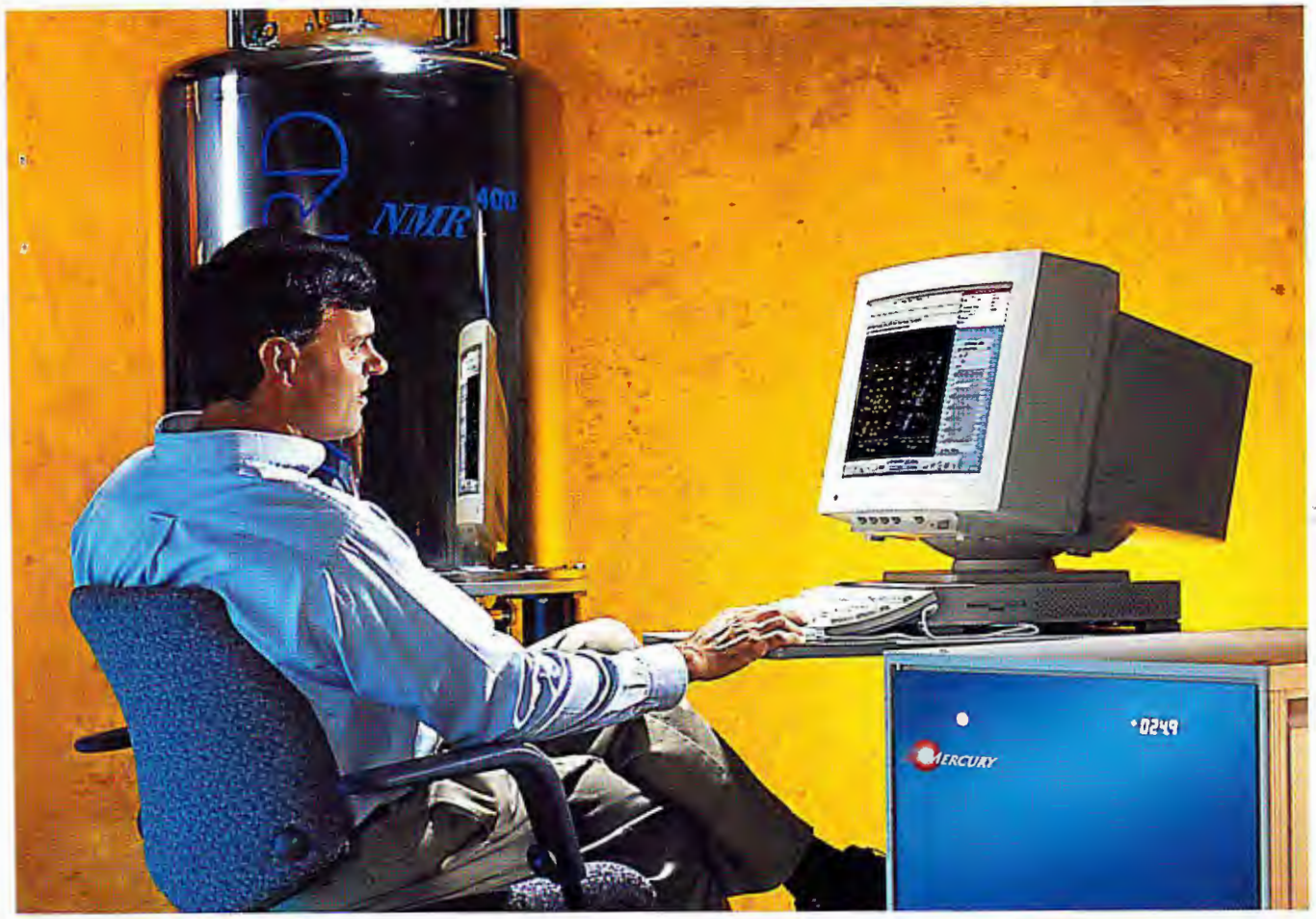
Tony

Anthony A. Ribeiro (A^2R)





The Wait is Over.



MERCURY: A New Dimension in NMR™.

Varian, the recognized leader in NMR, proudly introduces *MERCURY*™, the smallest, most advanced two-channel, high-resolution superconducting spectrometer. Based on customer feedback and state-of-the-art technology, *MERCURY* answers your need for a simple yet sophisticated instrument.

Available in either 4-Frequency ($^{1}H/^{19}F/^{31}P/^{13}C$) or Broadband configurations, *MERCURY* delivers real problem-solving power and big performance at a compact price.

Featuring 1D, 2D, Indirect Detection, and highest-quality spinlock analyses, as well as optional Pulsed Field Gradient (PFG) and Variable Temperature capabilities, *MERCURY* provides a level of sensitivity that rivals that of research-grade spectrometers.

Experience a new dimension in NMR. Contact the Varian office nearest you for more information on Varian's newest NMR spectrometer system.

MERCURY's advantages are clear:

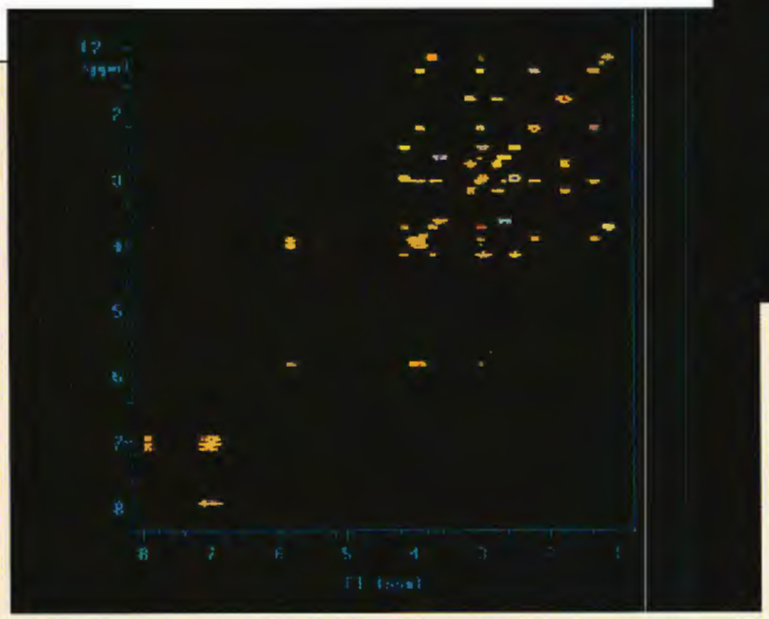
- Compact size
- State-of-the-art surface mount technology
- Fully automated applications
- VNMR™ software featuring enhanced *GLIDE*™ user interface and CDE
- Powerful and versatile configurations

ISO 9001 REGISTERED

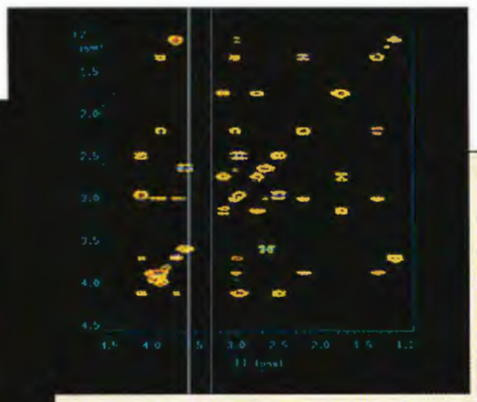
varian

MERCURY: Big Performance in a Compact Design

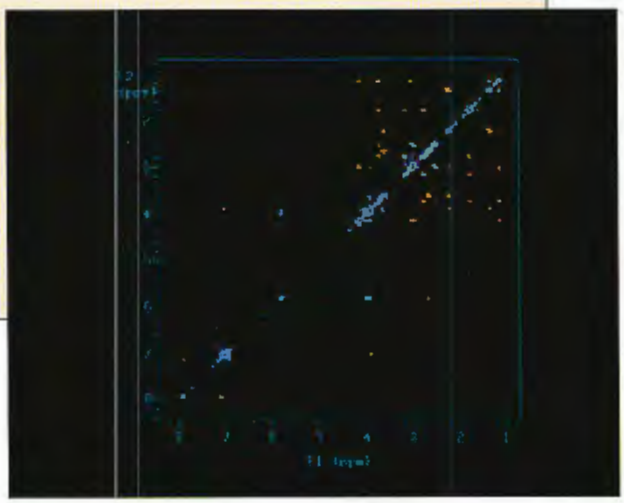
Achieve the highest levels of performance with ***MERCURY's*** complete range of experiments. High definition TOCSY and other spinlock spectra found with ***MERCURY*** demonstrate the tremendous stability of the entire system, as well as the speed and accuracy (phase accuracy and timing accuracy) of the phase shift.



Spinlock experiments, such as this TOCSY spectrum of strychnine, are routinely performed with ***MERCURY***.



Expanded region of the TOCSY spectrum.



MERCURY features advanced experiment capability, as is demonstrated in this high-quality ROESY spectrum.

Manufacturing Facilities Varian NMR Instruments, Building 4, 3120 Hansen Way, Palo Alto, California 94304-1030, Tel 415.493.4000 • **Australia** Mulgrave, Victoria, Tel 3.9.566.1133 • **Austria** Vösendorf, Tel 1.695.5450 • **Belgium** Brussels, Tel 2.721.4850 • **Brazil** São Paulo, Tel 11.820.0444 • **Canada** Mississauga, Ontario, Tel 1.800.387.2216 • **China** Beijing, Tel 1.256.4360 • **France** Les Ulis, Tel 1.6986.3838 • **Germany** Darmstadt, Tel 06151.7030 • **Italy** Milan, Tel 2.921351 • **Japan** Tokyo, Tel 3.5232.1211 • **Korea** Seoul, Tel 2.3452.2452 • **Mexico** Mexico City, Tel 5.514.9882 • **Netherlands** Houten, Tel 3063.50909 • **Russian Federation** Moscow, Tel 095.290.7905 • **Spain** Madrid, Tel 91.472.7612 • **Sweden** Solna, Tel 8.445.16.20 • **Switzerland** Basel, Tel 295.8000 • **Taiwan** Taipei, Tel 2.705.3300 • **United Kingdom** Walton-on-Thames, Tel 01932.898.000 • **United States** California, Tel 800.356.4437 • **Other sales offices and dealers throughout the world**

MAG-8618/735

varian

UNIVERSITÀ DEGLI STUDI DI MILANO

Dipartimento di Scienze Molecolari Agroalimentari

DISMA Via Celoria, 2 I-20133 Milano Tel. 02-2663662 /2365029 /2362721

Milano, 08/7/1997 (received 8/14/97)

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

Deuterium isotope effects in 4,9-dihydroxyperylene-3,10-quinones

Dear Barry

We have continued our study on the deuterium isotope effects on perylenequinones and other intramolecularly hydrogen bonded compounds, which present a phenol-quinone tautomerism (Scheme 1), and a paper will appear in J. Chem. Soc. Perkin 2.

We concluded that the primary isotope effects on OH protons of hydroxyperylenequinones and hydroxyanthraquinones (large and positive) fit well with the correlation between such effect and the OH chemical shift, found[1] for β-Ketoesters and o-hydroxyacyl aromatics.

This effect can be successfully used to estimate the strength of the hydrogen bonds in solution, for all intramolecularly hydrogen bonded enol compounds, indipendently from the tautomeric process. On the contrary the secondary $^2\Delta$ effect on the carbonyl carbon shift depends on the tautomeric process and must be used with caution.

The results for perylenequinones gave a substantial parallelism between solid and liquid phase. and showed that the strength of the hydrogen bond in perylenequinones depends on the planarity of the naphthalene units, rather than on the distorsion of the polycyclic ring.

The secondary isotope effects on carbon nuclei in perylenequinones are transmitted along the whole extended π-conjugated system (Scheme 2), with positive and negative, but not alternating signs. The negative sign appears characteristic of the quinonoid and the positive one of the benzenoid ring; thus we could exclude a significant presence at the equilibrium of cross-quinone tautomers, like 3,10-dihydroxyperylene-4,9-dione.

In such equilibrating systems it is difficult to ascertain whether isotopic effects on chemical shifts are caused by intrinsic (involving a single species) or equilibrium effects. As the chemical shift difference between the two tautomeric species is large for the oxygen-bound carbon atoms (*ca* 17 ppm), the long range effects on C-3,10 and C-4,9 might be due to a variation of the tautomeric equilibrium, caused by the presence of deuterium. In order to check whether the equilibrium perturbation is responsible of the experimental long-range isotope effects, we calculated the equilibrium constant modified for the isotopic substitution at one of the hydroxyls, by using the following equation[2]:

$$\Delta\delta = \frac{K(k-1)\,(\delta_B - \delta_A)}{(1+K)\,(1+kK)} \qquad (1)$$

where $\Delta\delta$ is the observed isotope effect, k is the factor modifying the equilibrium constant K = [A]/[B] and δ_B, δ_A are the chemical shifts for the pure tautomers A and B. These were estimated from values for the hexamethylethers of tautomer A and B respectively. In the case of perylenequinones of type I,for instance cercosporin with δ_B-δ_A = 17 ppm and isotope effects

of +0.075 and -0.037 ppm, we obtained for C-4,9 and C-3,10 respectively k = 1.1095 and 1.048, corresponding to an increment of tautomer A of the order of 0.4-0.2%. This might be reasonable, as we found[3] that the population of tautomer A in the non-deuterated equilibrium mixture is 95-100%. However the same calculation for C-9b,12c performed with the k values thus obtained, gave δ_B-δ_A = -14.73 and -27.97 ppm, which are not compatible with the chemical shift difference measured for the same carbon atoms of the hexamethylethers, i.e. -4.43 ppm. Similar procedure performed for C-12b,12a gave δ_B-δ_A = +4.30 ppm, also not compatible with data from the models, *i.e.* -5.74 ppm. Consequently we must conclude that the contribution of the equilibrium shift to the isotope effects for cercosporin , if any, is very low, and therefore the long-range effects on ^{13}C must be largely intrinsic. This conclusion is in agreement with the results obtained for 9-hydroxyphenalen-1-one[4], which has a symmetric energy profile. It is further strengthened by the fact that both ^{13}C and ^{1}H chemical shifts and the primary and secondary isotope effects for cercosporin do not change with solvent and temperature.

On the contrary, for perylenequinones of type II, for instance phleichrome and it atropoisomer isophleichrome the tautomeric equilibrium depends on these factors[3] (All these compounds assumes a helical shape, thus the helicity generates axial chirality). In the case of isophleichrome the tautomeric equilibrium is depending on solvent and temperature, as shown by $J_{C,OH}$ values[3] and is also reflected on the isotope effects reported in Scheme 2. In diluted $CDCl_3$ solution, tautomer A is predominant, as well as in acetone (55-70%)[2], and the isotope effects are strong and positive for C-4 and negative for C-3 respectively. On the contrary in more concentrated $CDCl_3$ solution, tautomer B is predominant (*ca* 70%)[2], and a strong positive value, mainly due to $^{2}\Delta$ effect, was observed for C-3, in agreement with the structure of tautomer B; the value for C-4 is also positive, but smaller. In the case of phleichrome, the calculation performed through equation (1) gave a 1.9% increment of tautomer A. With the modified equilibrium constant, the calculated chemical shift difference δ_B-δ_A for C-9b,12c in $CDCl_3$ results -6.21 ppm, which could be considered compatible with the value of -4.43 ppm, measured for the two hexamethylethers. But in acetone the discrepancy becomes larger (-8.26 ppm). However, a contribution of the equilibrium shift to the isotope effect cannot be excluded in this case. For phleichrome, we measured the coupling constants between the OH proton and the oxygen-bound carbon atoms in the monodeuterated species. They resulted substantially the same, within experimental errors, of those obtained both from the signal of the non-deuterated species present in the same solution, and from the spectrum without D_2O. As the error in this measurement (±0.2 Hz) is reflected in the population of tautomers for an amount of *ca* 8% a small increment of tautomer A by deuteration is thus possible. Phleichromes also showed significant changes with temperatures in the secondary effects at C-3,10 and C-4,9 sites: the opposite trend found for C-4 and C-9 with respect of C-3 and C-10 clearly indicates an increase of the most stable tautomer A at low temperature (see Figure).

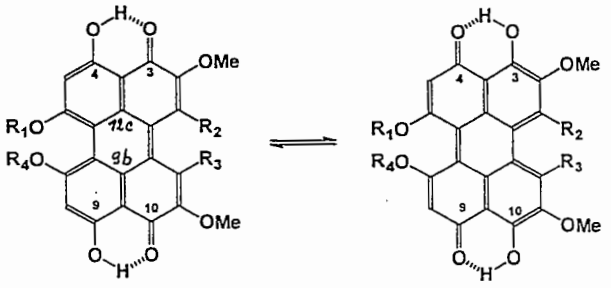
References

1) L. J. Altman, D. Laungani, G. Gunnarsson, H. Wennerström and S. Forsén, *J. Am. Chem.* Soc., 1978, **100**, 8264.

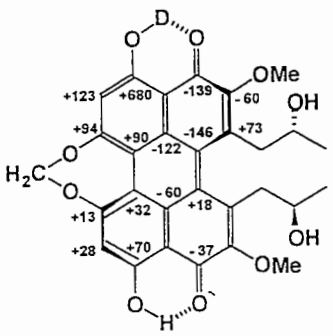
2) J. Reuben, *J. Am. Chem. Soc.*, 1984, **106**, 6180.

3) A. Arnone, L. Merlini, R. Mondelli, G. Nasini, E. Ragg, L. Scaglioni and U. Weiss, *J. Chem. Soc. Perkin Trans. 2*, 1993, 1447.

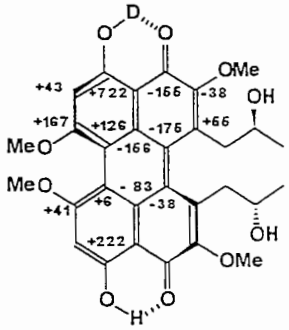
4) C. Engdahl, A. Gogoll and U. Edlund, *Magn. Reson. Chem.*, 1991, **29**, 54.



Scheme 1

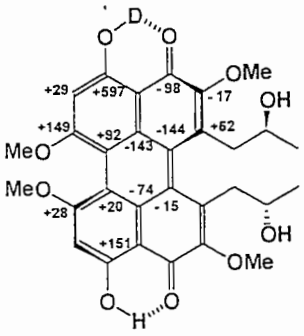


Cercosporin



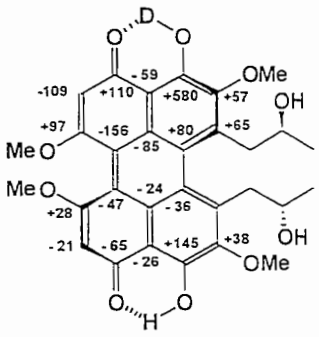
Phleichrome

Scheme 2

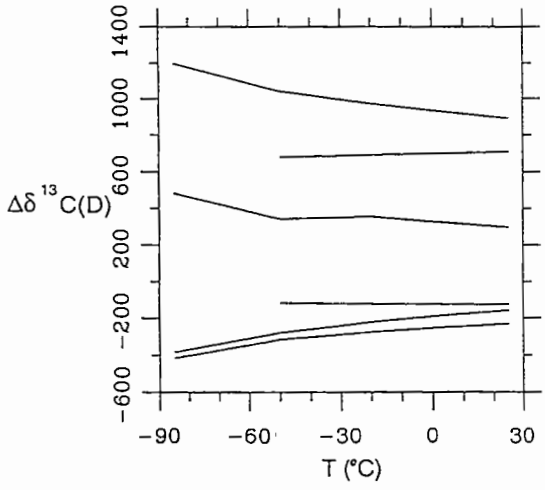


Diluted (1.5×10^{-2} M) solution
Tautomer A prevailing

Isophleichrome



Concentrated (2.8×10^{-1} M) solution
Tautomer B prevailing



with best greetings
sincerely yours

R.Mondelli L.Scaglioni

468-8

St Joseph's Health Centre of London

The University of Western Ontario

The Lawson Research Institute 268 Grosvenor Street, London, Ontario N6A 4V2 (519) 646-6000
Fax # (519) 646-6110

August 21, 1997

Dear Barry,

I've become involved, fairly late in the game, in the organization and coordination of another Symposium, **Magnetic Fields: Recent Advances in Diagnosis and Therapy**, which will be held here in London, November 14-16, 1997. The program for Friday, November 14 , *High Field Applications in Neonatal/Musculoskeletal MRI/MRS and NIRS*, will probably be of most interest to your readers.

What are the Technical Obstacles to Overcome at High Fields?
J. Helpern, The Nathan Kline Institute
Practical Implementation of High Field Technology
J. Allis, Siemens Medical Engineering Dedicated Systems Unit
Neonates and Magnetic Resonance Imaging
F. Cowan, Hammersmith Hospital
Neonate/Pediatric Functional Imaging
R. Menon, Robarts Research Institute
Neonatal/Pediatric Magnetic Resonance Spectroscopy
R. Ordidge, University College London
Complementary Nature of IRS and MRI/MRS
I. C. P. Smith, National Research Council of Canada
What are the Needs in Musculoskeletal Clinical/Research High Resolution Imaging?
D. Vellet, University College London
Hand and Wrist MRI on a Small Bore Magnet
G. Garvin and W. Romano, University of Western Ontario
MRI of the Wrist — Current Indications and Future Directions
M. Dalinka, University of Pennsylvania

The program for Saturday is on *The Therapeutic Uses of Magnetic Fields*. The program on Sunday is *The Technology of MRI/MRS*. Talks will presented Sunday morning on MRI of the elbow, wrist, shoulder, neonates and pediatric abdominal tumors as well as on new contrast agents for MRI and *in vivo* MR spectroscopy; workshops on Neonatal/Pediatric Imaging, Musculoskeletal Imaging and MRS are scheduled for the afternoon in the Dr. Reese Memorial MRI Suite, St. Joseph's Health Centre.

Further information may be obtained by contacting me by mail at the above address, by fax at the above number or by e-mail, mgordon@lri.stjosephs.london.on.ca
Information and a registration form may also be obtained from our web site, http://www.stjosephs.london.on.ca/LRI/bemw/

Sincerely,

Myra

Myra Gordon

UltraShield™ Magnets Win the Space War

Introducing the BRUKER SPECTROSPIN 500 MHz/52 mm UltraShield™ High Resolution NMR Magnet

Siting is now much easier than ever before because the space required for NMR systems has just become considerably smaller. The magnetic stray field has been significantly reduced by redesigning the coil of a standard magnet and adding a superconducting active shield. The volume enclosed by the 5 Gauss surface for an UltraShield™ magnet is ten times smaller than for a comparable standard magnet, without sacrificing any specifications.

BRUKER SPECTROSPIN

SPECTROSPIN is a member of the BRUKER family of companies and is located near Zurich, Switzerland. SPECTROSPIN is the world's largest manufacturer of superconducting NMR magnets. Our more than 30 years of experience in development and production of superconducting magnets enables us to deliver NMR magnets with exceptional performance and reliability. Many SPECTROSPIN superconducting magnets built in the late 70's and early 80's are still on field, providing quality data and dependable service.

CE
Health and Safety

ISO 9001 CERTIFIED
Development and Production

Main Features

- Active shielding technology strongly reduces stray fields and decreases the volume enclosed by the 5 Gauss surface by a factor of ten.
- Advanced magnet design and a new z^3 cryoshim provides outstanding field homogeneity with excellent resolution and non-spinning lineshape.
- Exceptionally low ceiling height requirements for installation and operation.
- Optimized cryostat design provides an extremely low helium evaporation rate.
- Lowest drift rates.
- Special sensors connected to the Automatic Cooling Device (ACD) prevent stresses during magnet cooling.
- Advanced vibration isolation system integrated in the cryostat stand provides optimal dampening of ground vibrations.
- Electronic atmospheric pressure device stabilizes the field drift and helium boil-off when changes in atmospheric pressure occur (optional).

BRUKER

Innovation for customers delivered with Integrity

SPECIFICATIONS

MAGNET

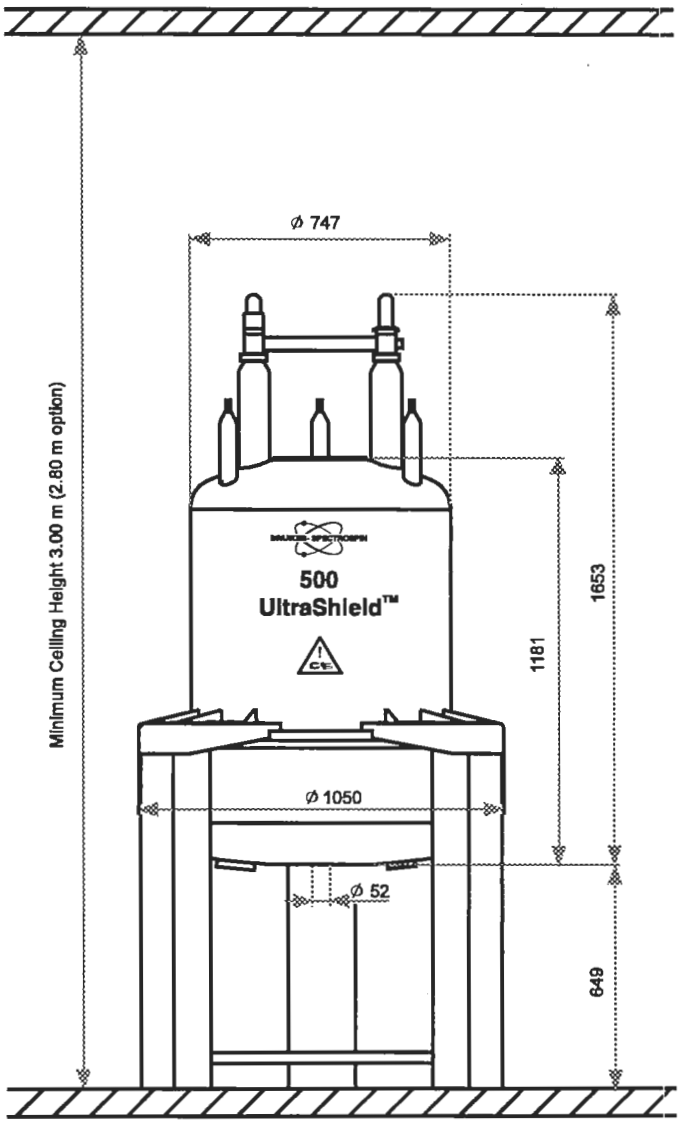
Central Field	11.7 Tesla
NMR Frequency	500 MHz
Field Drift	< 7.5 Hz/hr
Superconducting Shims	z, z^2, z^3, x, y, xz, yz, xy, x^2-y^2
Axial Range with Field Homogeneity better than 10 ppm (w/o RT Shimming)	~ 60 mm
5 G Line from the Magnetic Center	
-radially	< 1.3 m
-axially	< 1.9 m
Resolution at 50% 1% $CHCl_3$ 5 mm spinning	< 0.45 Hz
Lineshape 1% $CHCl_3$ 5 mm non-spinning	
at 0.55%	< 6 Hz *
at 0.11%	< 12 Hz *
Spinning Sidebands	< 1%

* Typical values obtained with the BOSS II™ shim system.

CRYOSTAT

Helium Evaporation Rate	~ 19 ml/hr
Helium Refill Volume	~ 64 liters
Helium Hold Time	> 140 days
Nitrogen Evaporation Rate	~ 250 ml/hr
Nitrogen Refill Volume	~ 108 l
Nitrogen Hold Time	> 18 days
Magnet Stand	included
Anti-Vibration Columns	included
Weight Without Cryogens	648 kg
Weight Including Cryogens	749 kg
Minimum Ceiling Height	3.00 m
Reduced Minimum Ceiling Height	2.80 m

500 MHz / 52 mm UltraShield™ Magnet



Dimensions in millimeters unless stated otherwise

USA

BRUKER SPECTROSPIN, INC.
19 Fortune Dr., Manning Park
Billerica, Mass. 01821
Tel. (508) 667 - 9580
Fax. (508) 667 - 3954
E-mail: magnets@bruker.com

http://www.bruker.com

Switzerland

SPECTROSPIN AG
Industriestrasse 26
CH-8117 Fällanden
Tel. (41) 1 825 91 11
Fax. (41) 1 825 96 96
E-mail: magnets@spectrospin.ch

M500US.1/BSI/DMI/3/97

Brock University

Department of Chemistry
e-mail jmiller@spartan.ac.brocku.ca

St. Catharines, Ontario
Canada
L2S 3A1

Telephone: (905) 688-5550 Extension 3402
Facsimile: (905) 688-2789 or 682-9020

Wed, Aug 6, 1997
(received 8/12/97)

Dear Barry,

^{19}F nmr using the DEPTH Sequence

David Wails in my group continues to investigate sol-gel routes to supported Friedel-Crafts alkylation catalysts, and have now begun to investigate the ^{19}F MAS NMR spectra of some of our materials, for which our Bruker standard bore 4 mm MAS probe has been retrofitted with a Bruker HFX unit. The unit allows the single proton channel to be split into two channels capable of observing proton and fluorine frequencies simultaneously, which allows 1H{19F} decoupling and vice versa. However due to our current amplifier configuration we are only capable of observing one frequency at a time.

The biggest drawback with our probe is the background fluorine signal, arising, for example, from the Kel-F screws used to hold the stator together. We have investigated DEPTH background suppression, which uses a 90^{o} pulse followed by 2 180^{o} phase dependent pulses, which either invert the sample signal phase or leave it unchanged. Nuclei outside the coil receive less than a 180^{o} pulse. Over 16 phase cycles, the background signal subtracts out [1]. Figure 1 shows the MAS spectra of a sample of powdered Teflon, spun at 10KHz in air recorded over a 14KHz spectral window. The spectrum in figure (1a) was recorded with DEPTH sequencing and (1b) with a single 90^{o} pulse. 16 scans with a 5 s delay between pulses were recorded for each. Spinning side bands are denoted by asterisks. The DEPTH sequence appears less effective and gives rise to some baseline distortion with this sample having a high fluorine content.

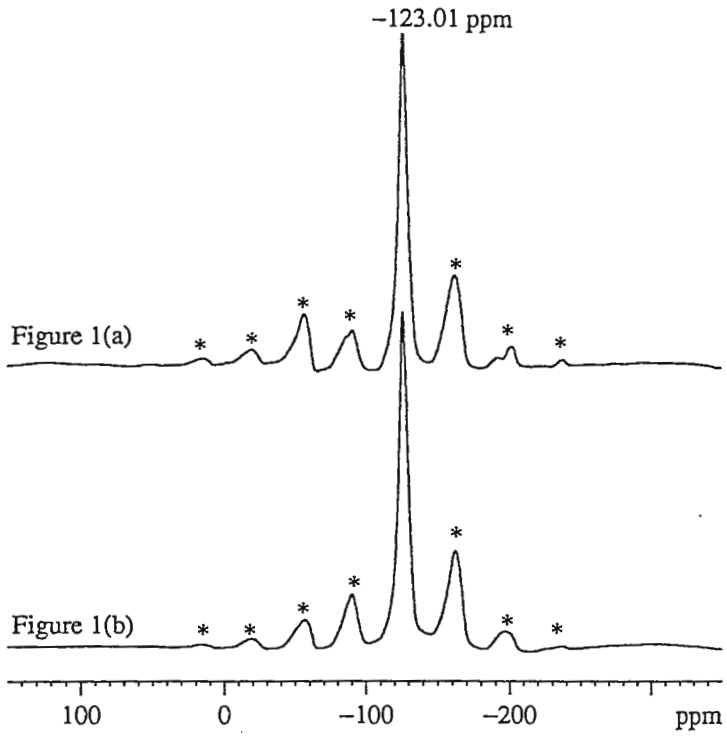
Figure 2 shows the spectra of one of our catalysts with low (< 5 wt%) fluoride levels with (2a) and without (2b) DEPTH sequencing. In this instance, the usefulness of the DEPTH sequence is obvious. The spectra show peaks at -142.7 and -156.9 ppm which are attributed to SiF_6^{2-} units and a broad band at around -107 ppm which is attributed to F^-. Without background suppression, only the sharpest peak at -142.7 ppm is discernible over the broad background signal.

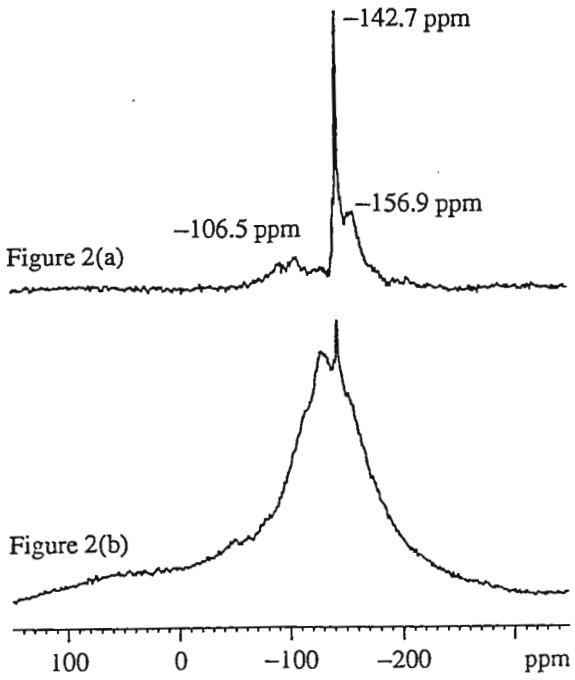
We would like to thank Dr. Tony Bielecki (Bruker Instruments Inc., Billerica, MA) for experimental assistance with the DEPTH setup.

Yours Sincerely,

Jack M. Miller
Professor of Chemistry.

[1]. J.M. Miller, Progress in Nuclear Magnetic Resonance Spectroscopy, 28 (1996) 255-281.


−123.01 ppm
Figure 1(a)
Figure 1(b)
100
0
−100
−200
ppm


−142.7 ppm
−156.9 ppm
−106.5 ppm
Figure 2(a)
Figure 2(b)
100
0
−100
−200
ppm

THE COLLEGE OF STATEN ISLAND
THE CITY UNIVERSITY OF NEW YORK

July 25, 1997
(received 7/29/97)
Dr. Bernard Shapiro
The NMR Newsletter
966 Elsinore Court
Palo Alto, CA 94303

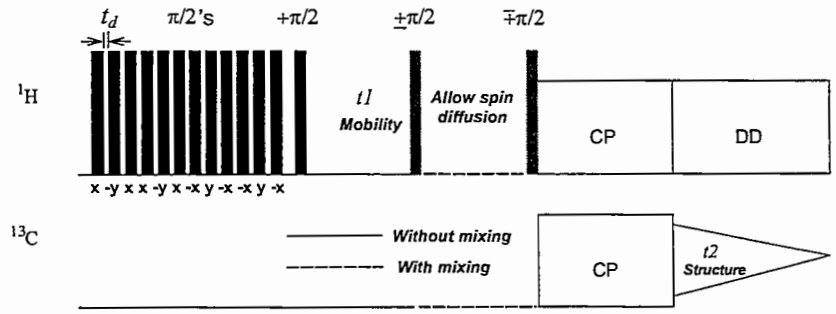
Dipolar-Filtered WISE Spectroscopy

Dear Barry,

The 2D wideline separation experiment (WISE) [1] typically correlates the ^{1}H wide lines on one dimension (*t1*) for mobility, and the ^{13}C chemical shifts on the other (*t2*) for structural information. To get this correlation, we have used WISE spectroscopy for suberin, which is a solid biopolymer blend isolated from wound-healing potatoes.[2,3]

When the suberin sample is hydrated with H_2O and then studied by the WISE experiment, however, there are both narrow lines from the H_2O protons and wide lines from the rigid suberin. This is not surprising since H_2O molecules are highly mobile and the polymers in suberin are generally very rigid. Therefore, when H_2O is in close proximity to the cell-wall polysaccharides, the spectrum will show overlapped ^{1}H lines at the ^{13}C chemical shift of the cell wall. This type of phenomenon was observed previously in a PS-*b*-PDMS co-polymer by Schmidt-Rohr *et al.* [1] Usually, it does not interfere with the spectral analysis. Nevertheless, when the intensity of the narrow line is of interest, or its intensity is quite low compared to the wide line, it can be advantageous to separate them spectroscopically.

A WISE sequence augmented with a dipolar filter [4] can serve this purpose. We propose to add the commonly-used 12-90° pulse dipolar filter before the WISE sequence as shown below.



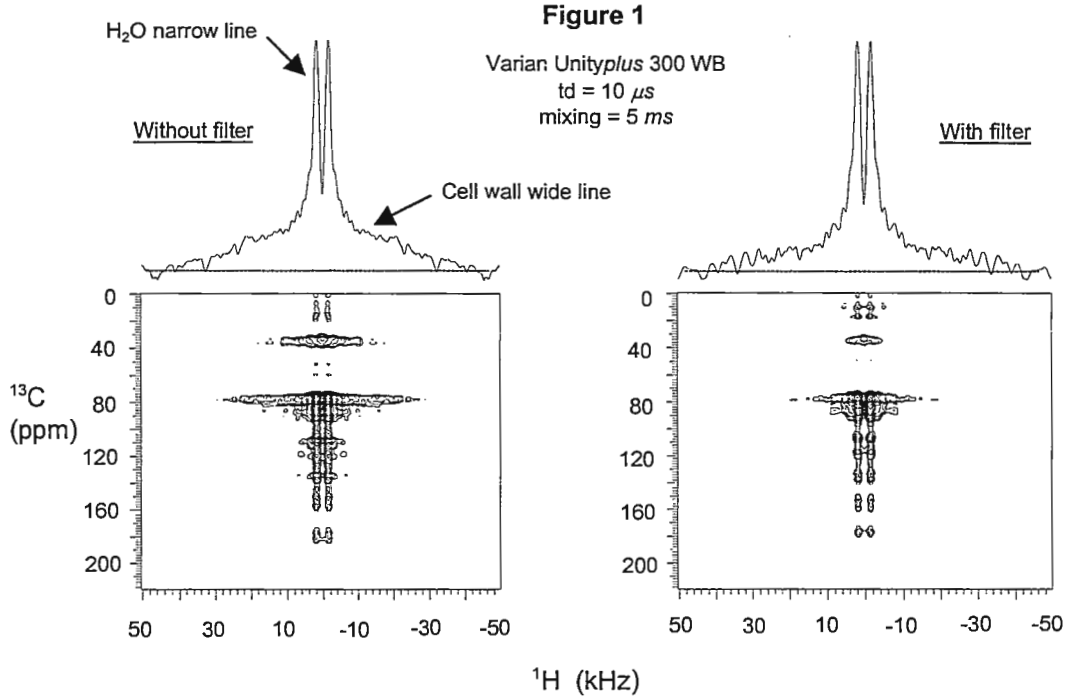
The strength of the dipolar filter can be adjusted by changing the delay interval t_d between the pulses, in order to adequately filter out the wide lines.

---

[1] K. Schmidt-Rohr, J. Clauss and H. W. Spiess, *Macromolecules*, 25 (1992) 3237.
[2] R. E. Stark and B. Yan, *The NMR Newsletter,* 460-25, January, 1997.
[3] B. Yan and R. E. Stark, The 38th Experimental Nuclear Magnetic Resonance Conference, 1997.
[4] J. Clauss, K. Schmidt-Rohr, A. Adam, C. Boeffel, and H. W. Spiess, *Macromolecules*, 25 (1992) 5208.

The dipolar-filtered-WISE spectrum of hydrated suberin is shown in **Figure 1** (right) in comparison with its normal WISE spectrum (left). The contour plot demonstrates that ^{1}H lines with half-height linewidth greater than 30 kHz can be substantially removed with a

Figure 1



t_d of 10 μs. The 74-ppm cell-wall signal slices for both spectra are displayed at the top of the contour plots, confirming the editing effect on the wide-line components.

We believe this dipolar-filtered experiment can be applied to other polymer mixtures or co-polymers in which the constituents have contrasting mobilities (e.g. the PS-*b*-PDMS polymer). In other cases, it can be used to characterize the narrow lines from an external mobile source (e.g. H_2O) which resides near or inside a rigid polymer.

Sincerely,

Bin Yan
Postdoctoral Research Associate

Ruth E. Stark
Professor of Chemistry

Model 3445/3446
Amplifiers from AMT

10-130 MHz Bandwidth

1000 and 2000 watt Models available

For High Performance NMR/NMRI Applications

Your NMR/NMRI requirements are pushing the leading edge of science and you need AMT RF power technology! The 3446 and 3445 operate from 10-130 MHz and are rated at 1000 watts for low field NMR and up to 2000 watts for NMRI applications up to 3 Tesla. AMT has brought together the highest possible RF performance at a most cost effective price. Nobody builds a better NMR/NMRI amplifier than AMT...

Additional Features Include:

- 10-130 MHz bandwidth for use in systems up to 3T
- Up to 2000 watts of power for imaging
- CW power capability for decoupling
- Blanking delay time >1 µs for multi-pulse

AMT

3080 Enterprise Street ■ Brea, CA 92821 ■ Phone: (714) 993-0802 ■ Fax (714) 993-1619

Models 3445/3446

10-130 MHz, pulsed, solid-state, RF power amplifier systems

Key Specifications:

Models:	3445	3446
Frequency range	10-130 MHz	10-130 MHz
Pulse power (min.) into 50 ohms	2000 W	1000 W
CW power (max.) into 50 ohms	200 W	100 W
Linearity (±1 dB to 30 dB down from rated power)	1500 W	800 W
Pulse width	20 ms	20 ms
Duty cycle	Up to 10%	Up to 10%
Amplitude droop	5% to 20 ms typ.	5% to 20 ms typ.
Harmonics	Second: -25 dBc max. Third: -24 dBc max.	
Phase change/output power	10° to rated power, typ.	
Phase error overpulse	4° to 20 ms duration, typ.	
Output noise (blanked)	< 10 dB over thermal	
Blanking delay	< 1 µ s on/off, TTL signal	
Blanking duty cycle	Up to 100%	
Protection	1. Infinite VSWR at rated power 2. Input overdrive 3. Over duty cycle/pulse width 4. Over temperature	

Other members of AMT's NMR/NMRI Family:

3205/3200
6-220 MHz, 300/1000 W

3304/3303
30-310 MHz, 400/700 W

PowerMaxx™ series
25-175 MHz, 4kW/7 kW

3137/3135/3134
200-500 MHz, 50/150/300 W

Supplemental Characteristics:

Indicators, front panel	1. AC power on 2. CW mode	4. Overdrive 5. Over pulse width	6. Over duty cycle 7. LCD peak power meter
System monitors	1. Forward/Reflected RF power 2. Over pulse width/duty cycle	3. DC power supply fault	4. Thermal fault
Front panel controls	1. AC power	2. Forward/Reflected power	
AC line voltage	208/230 VAC, 10%, 1Ø, 47-63 Hz		

	3445	3446
AC power requirements	1400 VA	700 VA
Size (HWL, inches)	8.75 x 19 x 24	8.75 x 19 x 24
Net weight	110 lbs.	75 lbs.

AMT

FOR ADDITIONAL INFORMATION, PLEASE CALL:

AMT United States	Goss Scientific Europe	ISATEL Emitec AG SWITZERLAND	JEOL Trading Co. Japan	Qualls Scientific New Zealand, Australia
Ph: (714) 993-0802 Fx: (714) 993-1619	Ph: 44 1245 478441 Fx: 44 1245 473272	Ph: 44 4174 86010 Fx: 44 4174 85055	Ph: 813 3342 1921 Fx: 813 3342 1944	Ph: 64 3385 6973 Fx: 64 3385 6949

3080 Enterprise Street ■ Brea, CA 92821 ■ Phone: (714) 993-0802 ■ Fax (714) 993-1619

SOPHISTICATED INSTRUMENTS FACILITY
(Sponsored by the Department of Science & Technology, Government of India)
INDIAN INSTITUTE OF SCIENCE
BANGALORE-560 012 INDIA

Prof. C. L. KHETRAPAL
Convener

31 July 1997
(received 8/4/97)

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

Dear Barry,

Sub: NMR of Oriented Systems--Future Hopes!

It may be of interest to the readers of the NMR Newsletter to know about some of the recent developments and future prospects in NMR spectroscopy of oriented molecules; hence, we consider it worthwhile to communicate this information through a letter.

Since proton NMR spectra of molecules oriented in thermotropic liquid crystalline solvents are usually "strongly coupled" and, consequently, their complexity rises rapidly with the increasing number of interacting nuclei, the scope of the technique for the study of molecular structure and function has been somewhat limited. In order to investigate the structures of larger molecules, other techniques employing multiple quantum spectroscopy, partial deuteration of samples with proton decoupling, Near Magic Angle Sample Spinning, and mixed liquid crystals of opposite diamagnetic anisotropies have been suggested and developed. The future of the field seems bright due to additional recent advances that include:

(1) The discovery of new thermotropic liquid crystals with low order parameters.
(2) The more routine availability of high magnetic fields.
(3) The use of natural abundance ^{2}H-NMR spectroscopy.

We have recently observed that neat phases of quarternary ammonium halides based on trioctadecylamine exhibit nematic liquid crystalline behavior within a certain temperature range.[1] We have also observed that solutes such as dimethyl sulfoxide and methyl iodide induce another phase (identified as smectic) of methyltrioctadecylammonium iodide (MTAI) within certain temperature and concentration ranges of the solutes (^{13}C spectra in Figure 1; [$^{13}CH_3I$] $\approx$ 7 mass %). An extremely important feature of the neat MTAI liquid crystalline phase is that its order parameters, as well as those of the dissolved molecules, are very low. This results in 'first order' proton NMR spectra of the molecules dissolved in these phases, and demonstrates the potential of such phases when spectra with low dipolar couplings are needed. An example with 5.4 mass % *cis*, *cis*-mucononitrile is given by the ^{1}H spectra in Figure 2.

As reported by Ad Bax of the National Institutes of Health (USA) in the December 1996 issue of the NMR-Newsletter, the proton spectra of bio-molecules recorded at higher magnetic fields (750 MHZ) show orientational effects which are likely to prove valuable in structure determinations. Recently, in a talk entitled 'NMR Parameters Accessbile at High Fields' and presented at the Tsukuba NMR-97 meeting organized by Yoji Arata in Tsukuba, Japan during 5-6 June 1997, Bax demonstrated the utility of such effects in the determination of biomolecular

structures.

Lastly, Kasu Akasaka at Kobe, Japan has recently observed natural abundance ^{2}H-NMR spectra in liquid crystals. This has been possible in a reasonable amount of spectrometer time (about 2 hours at 750 MHZ spectrometer frequency) due to the high sensitivity of present day instruments.

All of these developments should lead to widespread applications of NMR spectroscopy of oriented molecules in the years ahead.

Yours sincerely,

C. L. Khetrapal

(C.L. Khetrapal)
Sophisticated Instruments Facility
Indian Institute of Science
Bangalore 560 012
India

Richard Weiss

(R.G. Weiss)
Department of Chemistry
Georgetown University
Washington, DC 20057-1227
USA

Figure 1.

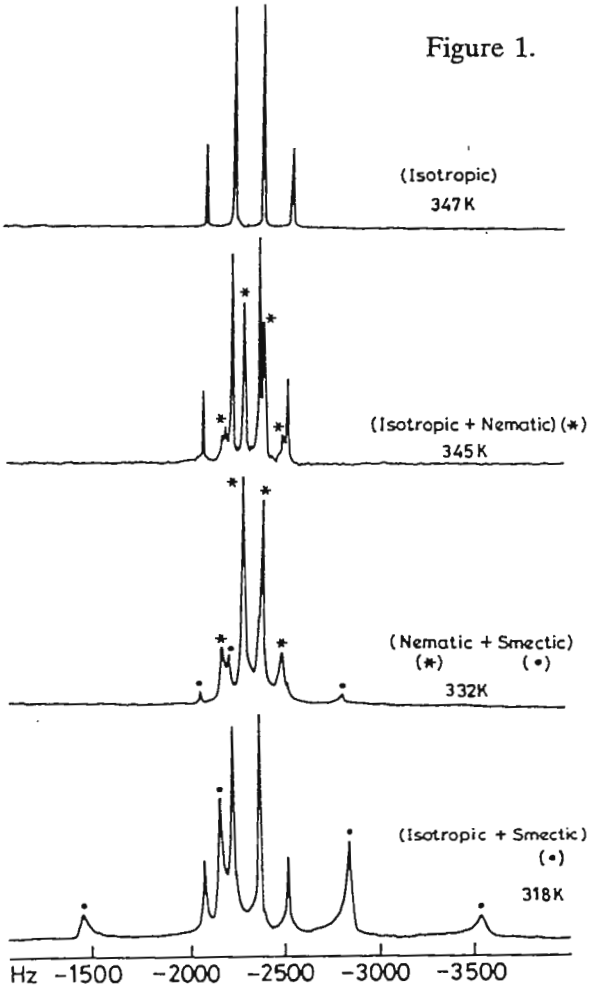
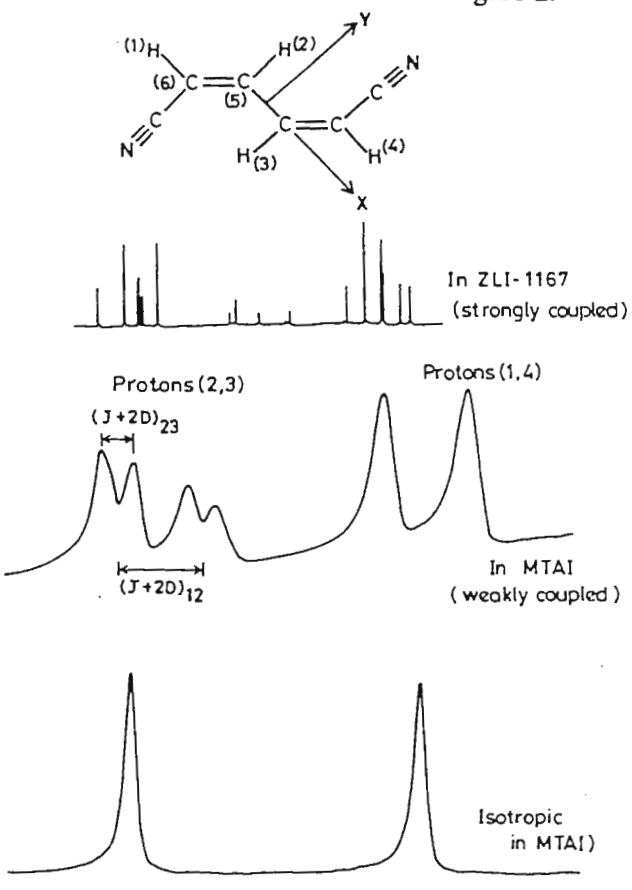


Figure 2.



JOHNS HOPKINS
MEDICINE

Radiology and Radiological Science
600 North Wolfe Street
Baltimore, MD 21287

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

July 25, 1997
(received 7/30/97)

HIGH FIELD - IS IT BETTER?

Dear Professor Shapiro,

It is almost axiomatic in high resolution NMR spectroscopy that resolution and sensitivity improve with increasing field strength, and, in fact, one can feel quite inadequate if someone else has a larger magnet than your own. In MR imaging, the choice of optimum field strength has been more contentious, and few systematic comparisons of *in vivo* spectroscopy at different field strengths have been reported [1].

At Henry Ford Hospital, in collaboration with Mike Boska and David Hearshen, I recently had the opportunity to compare single-voxel proton spectra of the human brain from the same subjects at 1.5 Tesla and 3.0 Tesla [2]. The figure shows short echo time (TE = 20 ms) from the same subject at both field strengths, recorded using the STEAM pulse sequence from an 8 cc voxel of white matter. In general, note the similar resolution and signal-to-noise at both field strengths; the glutamate/glutamine region of the spectrum is better resolved at 3T, and the amide resonance of NAA (downfield from water) is also better visualized. In 5 subjects, the S/N ratio for NAA was 28% greater at 3T compared to 1.5T, significantly lower than the theoretical 100% improvement expected for biological samples [3]. At long echo times (272 msec), S/N was almost identical, reflecting the shorter metabolite T_2 relaxation times at the higher field (210 vs. 480 msec, for NAA). While S/N comparisons between different machines at different field strengths depend on numerous factors (T_1 and T_2 relaxation times, field homogeneity, RF coil efficiency, preamplifier/receiver noise figure, slice profiles and pulse sequence efficiencies) it appears that the less than expected improvement in the current study can largely be explained by the decreased T_2's and increased linewidth at 3T. Other factors appear to be quite comparable between the two systems. So far we have not attempted to measure metabolite T_1's at 3T.

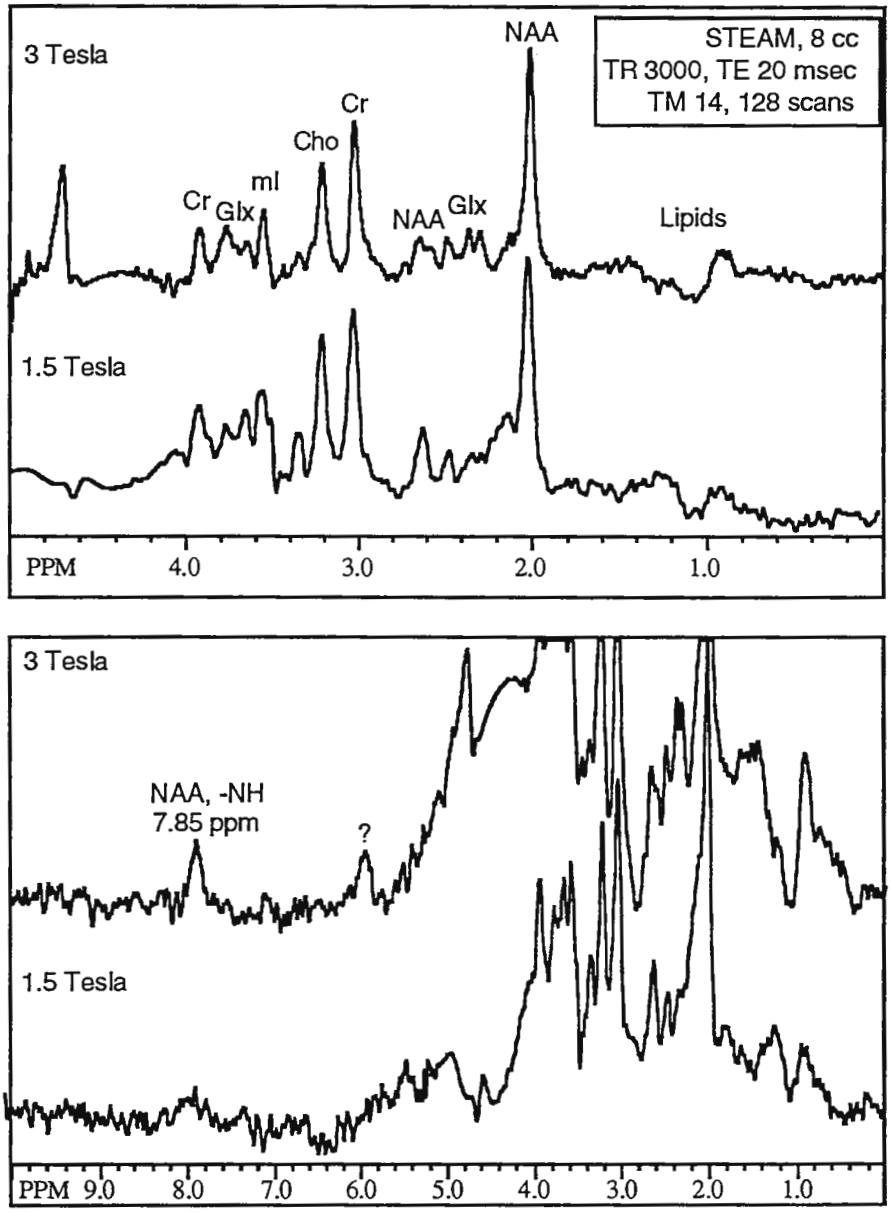
The current study does not rule out the possibility of improved resolution with further increases in field strength, particularly with the use of advanced shimming routines. Very well-resolved single-voxel brain spectra have recently been demonstrated in children at 4.0 Tesla and in dogs at 9.4 Tesla [4]. Please note my change of address,

Sincerely Yours,

Peter Barker

Peter B. Barker.

1. MD Boska et al., *J. Magn. Reson.* **13,** 228-238 (1990).
2. PB Barker et al., *in* "ISMRM, 5th Scientific Meeting, Vancouver, BC, 1997", p. 484.
3. DI Hoult et al., *J. Magn. Reson.* **34,** 425 (1979).
4. R Gruetter et al., *in* "ISMRM, 5th Scientific Meeting, Vancouver, B.C., 1997", p. 239.


3 Tesla
STEAM, 8 cc
TR 3000, TE 20 msec
TM 14, 128 scans
NAA
Cr
Cho
mI
Cr
Glx
NAA
Glx
Lipids
1.5 Tesla
PPM
4.0
3.0
2.0
1.0
3 Tesla
NAA, -NH
7.85 ppm
?
1.5 Tesla
PPM 9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0

OXFORD

Technical Specifications

200 - 600MHz Wide Bore NMR Magnet Systems

Oxford Instruments, NMR Instruments are the pioneers of superconducting magnet technology for NMR spectroscopy and are widely regarded by the worlds research community as the foremost manufacturer and supplier. A worldwide base of over 4500 successfully installed magnet systems serves as testament to an innovative magnet design approach and quality manufacturing processes. As an integral part of a high resolution NMR spectrometer, Oxford Instruments magnets are recognized the world over for their superior performance in chemical, pharmaceutical, biological, and materials research applications.

Advanced Design and Manufacture

State of the art superconducting magnet and cryostat designs provide superior NMR performance, as demonstrated in lineshape, resolution, magnetic field stability, and system siting:

- Advanced electromechanical modelling and system design tools optimise magnetic field homogeneity (to at least eighth order, on-axis) while at the same time minimising residual transverse gradients
- Designed for optimal system siting, minimising ceiling height, weight, service access, stray field and system footprints
- High efficiency room temperature shims provide optimal field homogeneity with low heat dissipation
- Rigorous magnet system testing in Oxford Instruments' extensive cryogenic and NMR test facilities ensure that optimal magnet performance is achieved on site
- All systems supplied with a wide range of safety features, providing worry-free operation at all field strengths
- All manufacturing processes certified to the internationally recognised ISO 9001 quality standards

Performance Excellence

Oxford Instruments magnet and room temperature shim systems provide outstanding performance in a complete range of NMR applications. Sample sizes from 37 µl magic-angle-spinning (MAS), 10 mm high-resolution biomolecular to 60 mm microimaging, as well as all other commonly used NMR sample types, are supported:

- Exceptionally low magnetic-field drift rates provide superior stability
- Full range of room temperature shim options for all magnet systems deliver unparalleled lineshape and resolution for both large and small samples
- Minimal cryogen usage provides the lowest possible cost of operation with a range of standard or year hold helium cryostat options available
- All Oxford Instruments magnets feature a virtually unlimited lifetime (with proper maintenance and cryogen service)

See reverse for full technical specifications

Specifications

Specification	System Type					
Magnet	**200/89**	**300/89**	**300/150**	**400/89**	**500/89**	**600/89**
Operating Field (Tesla)	4.7	7	7	9.4	11.7	14
NMR Operating Frequency (MHz ^{1}H)	200	300	300	400	500	600
Field Stability (Hz/hour ^{1}H)	<2	<3	<15	<10	<12	<12
Axial 5 Gauss Stray Field Contour (Metres)	2.6	2.7	4.2	3.3	4.5	5.0
Radial 5 Gauss Stray Field Contour (Metres)	2.0	2.2	3.3	2.6	3.5	3.9
Cryostat						
Standard Cryostat Minimum Helium Refill Interval (Days)	203	203	120	180	150	90
Standard Cryostat Helium Refill Volume (Litres)	68	68	101	60	80	135
Year Hold Cryostat Option Available	✓	✓	X	X	X	X
Nitrogen Refill Interval (Days)	14	14	22	14	18	18
Nitrogen Refill Volume (Litres)	61	61	135	67	131	121
Nominal Room Temperature Bore Diameter (mm)	89	89	150	89	89	89
Minimum Operational Ceiling Height (Metres)	2.9	2.9	4.1	2.9	3.4	3.4
System Weight (kg) Including Cryogen's	391	399	1050	410	1075	1200

Customer Support

Oxford NMR Instruments provides a worldwide network of subsidiary companies and representatives available to provide technical siting, service and sales support.

Certificate No. FM30231

ISO 9001 CERTIFIED

UK
Oxford Instruments
NMR Instruments,
Osney Mead,
Oxford OX2 0DX,
England
Tel: +44 (0)1865 269500
Fax: +44 (0)1865 269501
e-mail: info.nmr@oxinst.co.uk

France
Oxford Instruments SA
Parc Club-Orsay
Universite,
27, rue Jean Rostand,
91893 - Orsay Cedex,
France
Tel: +33 1 6941 8990
Fax: +33 1 6941 8680

Germany
Oxford Instruments
GmbH
Kreuzberger Ring 38,
Postfach 4509,
D-6200 Wiesbaden,
Germany
Tel: +49 611 76471
Fax: +49 611 764100

Japan
Oxford Instruments K.K.
Haseman Building,
2-11-6 Kotoku,
Tokyo,
Japan 135
Tel: +8 3 5245 2361
Fax: +8 3 5245 4472

USA
Oxford Instruments Inc.
45950 Hotchkiss Street,
Fremont CA94539
USA
Tel: +1 415 813 9068
Fax: +1 415 813 9069
e-mail: oinmrwest@aol.com

Visit the Oxford Instruments Web site at http://www.oxinst.com/

OXFORD

Version: 003 Issue: B May 1997

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

August 12, 1997
(received 8/16/97)

ProteinPack: A Package of Triple-Resonance Experiments

Dear Barry,

During a short sabbatical at Varian, Robin Bendall of James Cook University in Australia developed a useful and convenient method by which triple-resonance experiments on proteins can be done without extensive calibration or parameter entry. Robin and I have extended this approach in ProteinPack, a set of pulse sequences, parameter sets, manuals and macros that has now been placed in the USERLIB (available via our Web site (nmr.varian.com) or via email request at userlib.request@nmr.varian.com).

The pulse sequences provided in ProteinPack are all gradient-based and, if NH-detected, sensitivity-enhanced. They include HNCA , HNCO, HN(CO)CA, HNCACB, CBCANH, CBCA(CO)NH, HCCH-TOCSY-NNH, CCH-TOCSY-NNH, HCCHTOCSY and DE H-(CC)CH-TOCSY. All except HNCO come with the option of 2H-decoupling using a fourth channel.. The bulk of the sequences are based on those resulting from the Kay and Bax labs.

Installation

Users can obtain the compressed file, place it in their /vnmr/userlib/psglib, and use the normal userlib extract procedure, e.g. "extract psglib ProteinPack". Once the "extract" is done the user can use the mouse to select the "setup" button, followed by the "ProteinPack" and "Install" buttons. This performs pulse sequence and shaped-pulse creation. Another button, "AutoCal" will update all the ProteinPack parameter sets with spectrometer-specific values for tof, sw, temp, tpwr, pwC, pwClvl, pwN, pwNlvl, etc.- the normal calibrations used in double-resonance experiments such as hmqc or hsqc. The power levels are those used typically for "hard" (full-bandwidth) pulses.

The user can automatically update all parameter sets for a particular sample by using a "AutoTripRes" button that determines the proton, carbon, and nitrogen 90's, optimizes the coherence transfer gradient level and runs 16-scan 1D spectra for all the ProteinPack pulse sequences. When each is finished the result is plotted, the fid is stored, and each parameter set, now updated, is stored back in vnmrsys/parlib. The total process takes a little over 10 minutes.

From now on, the user only needs to enter the name of the pulse sequence (this runs a macro by the same name). The parameters are, for the most part, ready to go, with only, perhaps, the 1H pw90 needing adjustment to reflect differences in sample salt content. The time of the experiment is variable, of course, depending on the resolution desired in F1 and F2 (determined by ni and ni2) and the desired signal-to-noise ratio (determined by nt). Once these parameters are set the user can enter "go". A series of all possible 2D experiments (150 minutes or overnight) can be done by button also.

If 2H decoupling is desired, the user enters dm3='nyn' (assuming a 4th channel is available). This enables 2H decoupling using the normal channel 4 parameters such as dn3, dmm3, dmf3, etc. .These only have to be determined once and they will be the identical for all ProteinPack experiments (except for HNCO where there is no deuterium attached to any of the relevant nuclei).

Once data are acquired, they may be processed conveniently using VNMR, both for 2D and 3D. Sensitivity-enhanced experiments do not need any "fid sorting" to be processed. Using VNMR versions 5.2 and above, wft2da and ft3d recognize the preset f1coef and f2coef parameters.

The Basis for ProteinPack

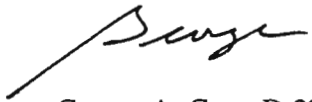
The underlying basis for the success of using ProteinPack is the predictability of spectrometer output. This means that calibrations of pulse widths at single power levels are sufficient for prediction of power levels for region-selective pulses. This is highly relevant for 13C in all of these experiments, but also for protons, where soft selective pulses are done on the water and spinlock periods are employed. No small-angle phase corrections are needed for different power levels and no amplitude/phase correction tables are employed.

Since all of these sequences involve region-selective 90's and 180's on the carbonyl, alpha, or alpha/beta carbon regions, the lengths of the pulses are easily and automatically determined so as to provide the required excitation nulls (e.g. at 600 Mhz a 55 usec 90 degree pulse centered at 56 ppm has its first excitation null at 174 ppm). ProteinPack provides a single macro, "proteincal", that based on the value of "sfrq", automatically creates all shapes needed by all of the ProteinPack pulse sequences. This is done once at installation and not again, since these pulses are sample and probe independent.

However, the power levels necessary for these pulses do need to be determined so that the proper flip angles are generated. This operation is done as a part of "go" within the pulse sequence, based on the "hard-pulse" power levels and pw90's. Power levels are adjusted via the linear modulators present on each channel (Unity*plus* and UnityINOVA). Thus, as long as the "hardpulse" pulse widths are calibrated, nothing else is required. The flexibility necessary to do this is, of course, a property of the "c" language used in the pulse program. This same approach could be used in many pulse sequences in all areas of NMR.

Figure 1 shows a comparison of the automatic output generated by the use of the "AutoCal" button. This is a valuable comparison showing the effect of different pulse sequences on the same sample and spectrometer. The gradient hsqc spectrum provides a useful benchmark for triple-resonance pulse sequence and probe efficiencies.

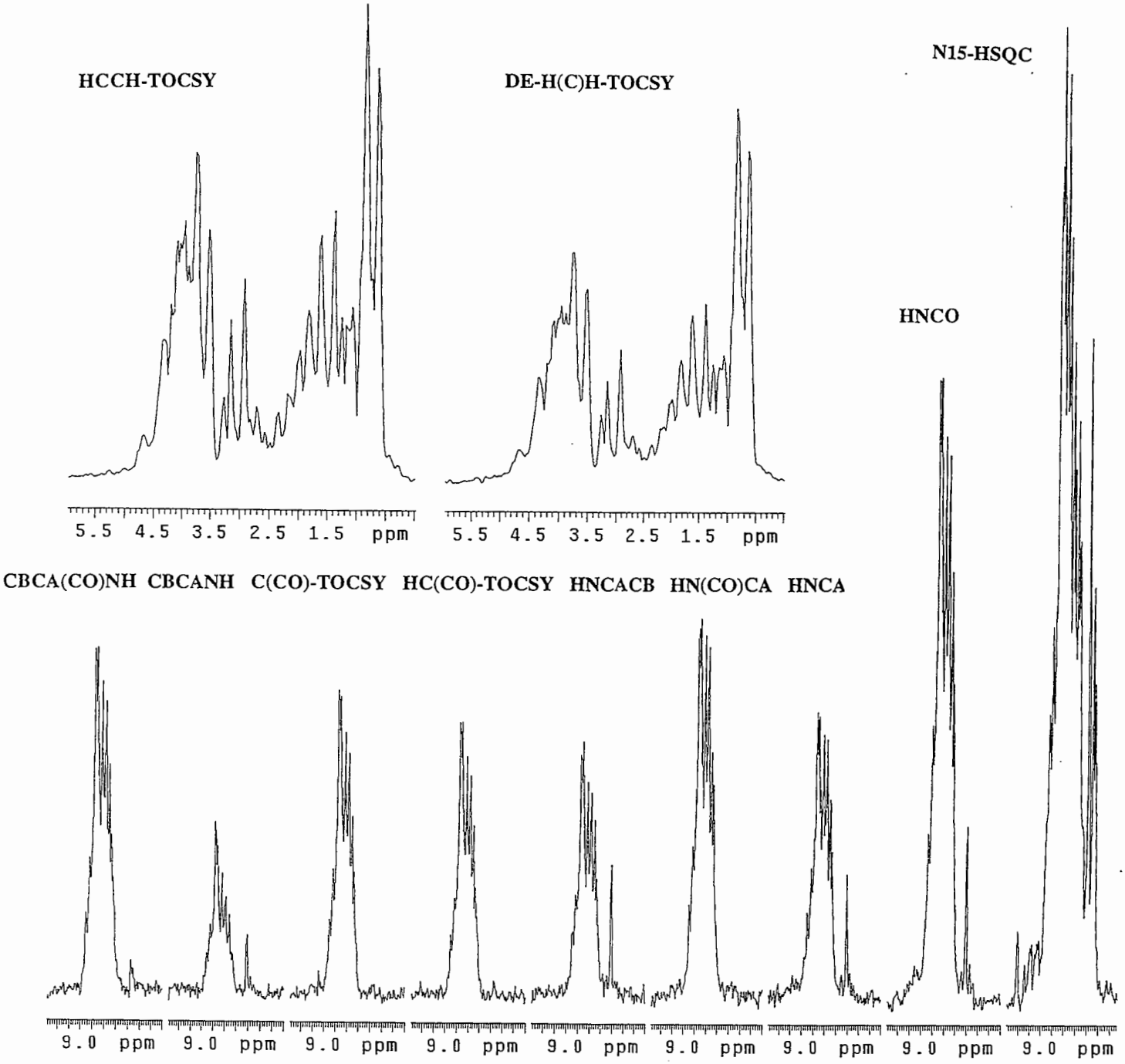
Sincerely,



George A. Gray, D-298
NMR Applications Laboratory
Varian Associates
Palo Alto, CA 94304-1030

Figure 1. ProteinPack 1D (16 scan) experiments at same gain. All of the lower spectra are at the same vertical scale and only the amide region of the proton spectra is shown. The upper spectra contain only aliphatic protons (no 13C broadband decoupling was used).

These illustrate, for the same protein, the "receptivity" of each pulse sequence to the particulars of molecular motion, aggregation and relaxation. All spectra were collected automatically as part of the autocalibration process using the "AutoTripRes" macro (initiated by the "AutoCal" menu button). The protein was alphalytic protease (<0.5 mM in 90% H2O, 10 % D2O at 600 Mhz using a HCN triax PFG probe in an unity*INOVA* 600).



NMR Specialist

The Department of Chemistry at the University of Iowa invites applications for an NMR Specialist position. This individual will assume technical operation, management, service, training, and maintenance of at least five superconducting NMR spectrometers (300-600 MHz). Applicants should hold a Ph.D. in chemistry, biochemistry, biophysics, or a related science or have extensive experience in the area of NMR spectroscopy. A minimum of three years of experience as an NMR spectroscopist is expected. The individual must have experience with application of modern NMR techniques to solve chemical problems. Familiarity with NMR hardware and software is essential. Excellent interpersonal, communication, and instructional skills are expected. The individual must have an ability to function in a service role, but should also have an interest in professional growth through technique development and collaborative activities. Other desirable qualifications include experience as an NMR facility manager, and familiarity with Bruker instruments, solid state NMR spectroscopy, solution gradient spectroscopy, and solution spectroscopy of biomolecules. Applicants should submit a letter of application, a resume, and have three letters of recommendation sent to: NMR Staff Search Committee, Department of Chemistry, University of Iowa, Iowa City, IA 52242 (FAX: 319 335-1270. e-mail: harold-goff@uiowa.edu) *The University of Iowa is an Equal Employment/Affirmative Action employer. Women and minorities are encouraged to apply.*

* *

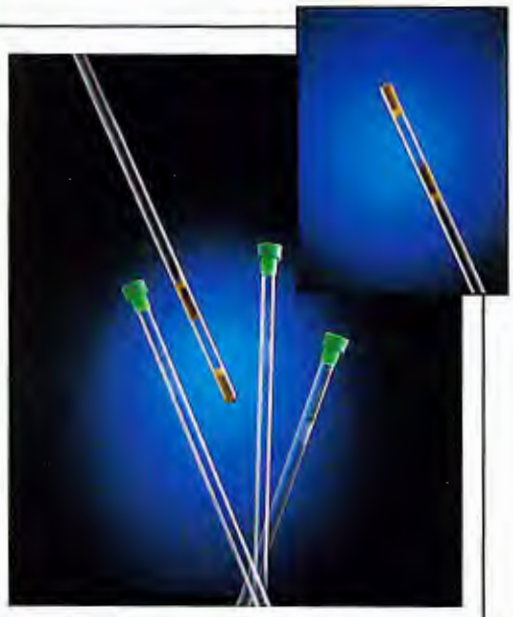
POSTDOCTORAL POSITION

A post-doctoral position is open at the Weizmann Institute of Science, for a project involving the application of NMR to study chemical and pharmacokinetic properties of molecules used in Boron Neutron Capture Therapy (BNCT), and to their quantitative *in-vivo* detection. The facilities at the WIS MR center include a recently upgraded 30-cm horizontal bore 4.7T imaging spectrometer (with shielded gradients), a 9.4T wide-bore spectrometer with microscopy and mini-imaging inserts, and a 500 MHz high-resolution spectrometer.

Please direct inquiries to: Dr. Peter Bendel
Dept. of Chemical Services-MR Center
Weizmann Institute of Science
Rehovot 76100
ISRAEL
e-mail: cvbendel@wis.weizmann.ac.il

Nothing But Accurate!

DOTY SUSCEPTIBILITY PLUGS EXCLUSIVELY FROM WILMAD



Over the workstation, off the console, through the magnet stacks, down the bore, into the Wilmad sample tube, between the Doty Susceptibility Plugs, long term acquisition,
Nothing but Accurate!

Huh!
Under the magnet, off the cabinet, around the workstation,off the New Wilmad NMR Catalog, down the bore, into the Wilmad sample tube, between the Doty Susceptibility Plugs, long-term acquisition,
Nothing but Accurate!

Here's why you'll find Doty Susceptibility Plugs better than those other Glass Microcells

SAVE RESEARCH DOLLARS
- Less costly than the susceptibility altered glass option
- Use them with standard Wilmad NMR tubes

EASE OF USE
- Simple bubble removal
- Store samples in screw cap tubes

BETTER MATCHING
- More plug materials match more solvents
- Doty plug susceptibility more consistent than glass alternative

Critical Applications Need All-Star Accuracy!

*"Structure and coordinates of sex determining factor (SRY)-DNA complex kindly provided by Drs. G. M. Clore and A.M. Gronenborn"

WILMAD®

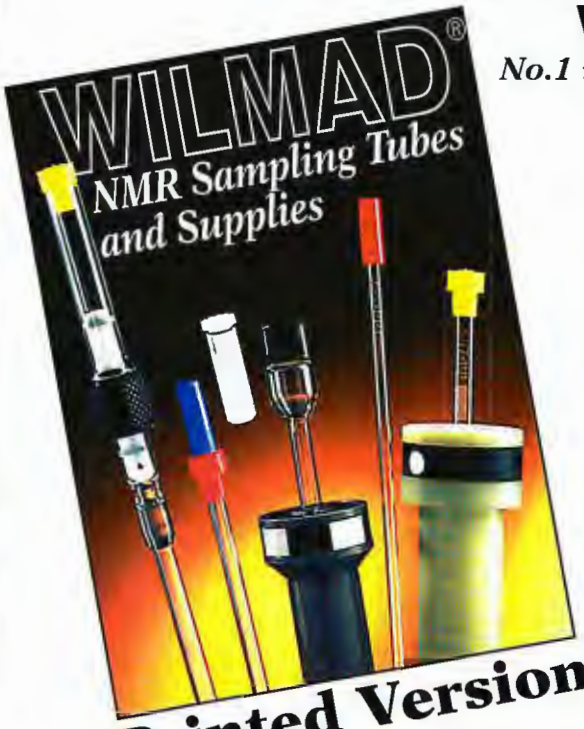
No. 1 In NMR Worldwide!

1-800-220-5171 • www.wilmad.com

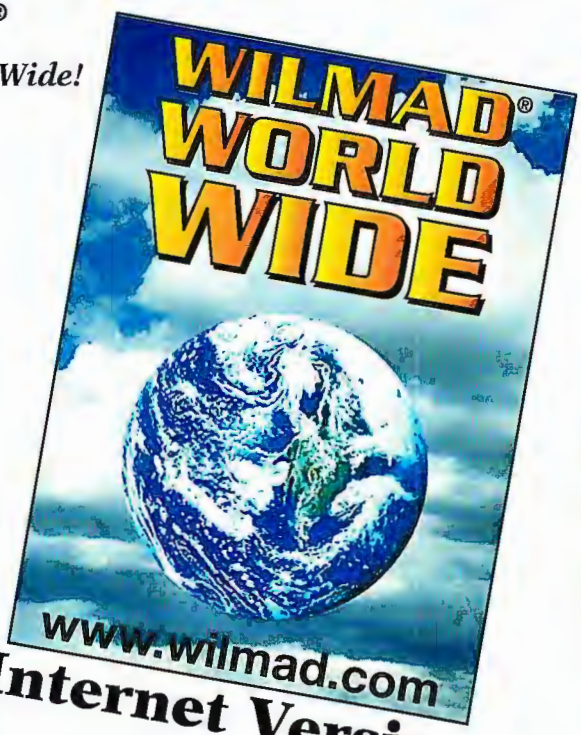
IT'S YOUR CHOICE

Wilmad leads the way with NMR Catalogs.
Now available on the world wide web and in full color print format!
[Look for updated product and technical information on our internet version]

WILMAD®
No.1 in NMR World Wide!



Printed Version



Internet Version

A Sample of the New Products in the all New Wilmad NMR Catalog!





Susceptibility Plugs for 3 & 5mm NMR tubes
RotoTec Zirconia MASS Rotors
RotoTec Spinner Turbines for Varian & Bruker Sample Changers
High Quality Aldrich Deuterated NMR Solvents
Universal NMR Tube Washers

☐ ***YES!*** Send me your all new printed NMR Catalog

Co. Name: _____ **Name:** _____
Address: _____
City: _____ **State:** _____ **Zip:** _____
Phone: _____ **Fax:** _____ **E-Mail:** _____

Call 1-800-220-5171 ask for catalog fulfillment or
Fax Back to... 1-800-220-1081 • International Fax... 1-609-697-0536

מחלקה לפיסיקה כימית
Chemical Physics Department

מכון ויצמן למדע
Weizmann Institute of Science

רחובות 76100
טלפון 08 934
פקס המכון 08 9466966
פקס המחלקה 934 4123
טלקס 381300
דואר אלקטרוני

76100 Rehovot, Israel
Phone 972 8 934
Institute Fax 972 8 9466966
Direct to Dept. Fax 972 8 934 4123
Telex 381300
e-mail

July 14, 1997
(received 8/5/97)

Dr. Barry L. Shapiro
The NMR Newsletter
966 Elsinor Court
Palo Alto, CA 94303
U.S.A.

The effect of ^{79,81}Br on the ^{13}C MAS spectrum. Another example.

Dear Barry

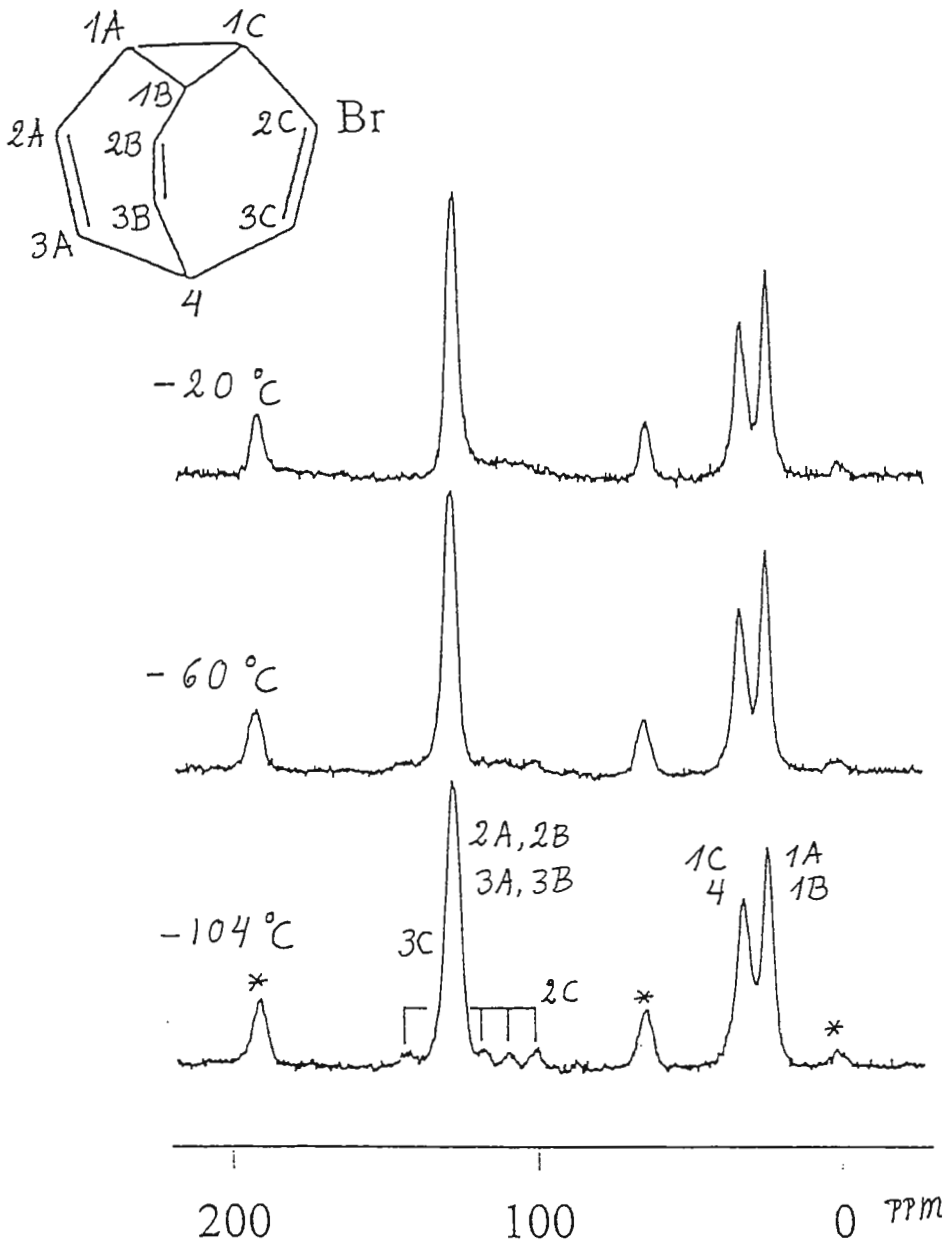
The effect of quadrupole nuclei on the MAS spectrum of neighboring carbon-13 is well understood and documented for a number of cases, including C-N, C-D, C-Cl and C-Br pairs.[1] The structure of such spectra depend on a number of magnetic parameters, including the direct (D) and indirect (J, ΔJ) coupling between the nuclei, and the quadrupole interaction constant (χ) [and Larmor frequency (ν)] of the quadrupole nucleus. It thus provides a tool to study the interrelation between the various interactions. Since there are not so many cases where such spectra were reported for bromine bonded carbons[2] we present an example below with its tentative interpretation.

In the figure are shown carbon-13 MAS spectra of bromobullvalene at several temperatures, recorded under the indicated conditions. The spectrum of the bromine bonded carbon (2C) at low temperatures (< -80°C) consists of an asymmetric quartet reflecting the incomplete averaging of the bromine-carbon interactions by the magic angle spinning. Since there are two bromine isotopes with different, but similar, nuclear magnetic moments, the spectrum is poorly resolved. Nevertheless it lends itself to interpretation using the theory described in references 3 (for $\chi >> \nu$ and assuming that all anisotropic tensors are axially symmetric and collinear with the C-Br bond). A tentative estimate of the relevant magnetic parameters (averaged over the two bromine isotopes) yields: D=1130Hz, ΔJ=+490Hz, J=-200 and $|\chi|$=400MHz.

The temperature dependence of the spectrum (see figure) shows the effect of self decoupling by the T_1 relaxation of the bromine nuclei. At higher temperatures (not shown) other dynamic processes (bond shift rearrangement) masks the effect of the fast relaxation limit on the spectrum.

With best wishes

R. Poupko Z. Luz K. Müller H. Zimmermann



Carbon-13 MAS NMR spectra of bromobullvalene at different temperatures as indicated. The spectra were recorded at 75.46 MHz with a spinning frequency of 4.8 kHz. Note the asymmetric quartet due to the bromine bonded carbon (2C).

---

1) R.K. Harris and A.C. Olivieri, *Progress. NMR spectroscopy*, **24**, 435 (1992); B. Nagasaka, S. Takeda and N. Nakamura, *Chem.Phys.Letters*, **222**, 486 (1994).

2) A.E. Aliev, K.D.M. Harris, R.K. Harris, S.A. Carss and A.C. Olivieri, *J.Chem.Soc. Faraday Trans.* **91**, 3167 (1995).

3) A.C. Olivieri, *J.Magn.Reson.*, **A101**, 313 (1993); S.H. Alarcon, A.C. Olivieri, S.A. Carss and R.K. Harris, *Magn.Reson.Chem.* **33**, 603 (1995);

Carnegie Mellon University
Department of Chemistry
4400 Fifth Avenue
Pittsburgh, PA 15213

15 August 1997 (received 8/23/97)

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

Ring Currents and Rings

Dear Barry,

The lore of "ring currents" is venerable. Probably the first time relevance to NMR was mentioned was John Pople's calculation of the extra downfield shift of aromatic protons from the "ring current" in benzene rings. In my view, the most penetrating analysis of "ring currents" was made by W.H.Flygare, in his review article in Chem. Rev. 1974 - he used the term nonlocal circulation of electrons, and was able to extract the magnitude of the ring current in benzene, and a number of other compounds, from measurements of the magnetic susceptibility anisotropy using Zeeman microwave spectroscopy. In his analysis, Flygare derived a table of bond susceptibility tensors, which could simply be added to give the molecular susceptibility tensors of a large number of acyclic compounds. The summation followed the simple rule

$$\chi_{mol} = \Sigma\chi_b$$

as in the Pascal additivity scheme. Cyclic compounds required the addition of a term accounting for nonlocal circulation:

$$\chi_{mol} = \Sigma\chi_b + \chi_{nonlocal}$$

For benzene, $\chi_{zz,nonlocal}$ amounted to $-0.58x\,10^{-28}$ cc/molecule.

Early estimates of the ring currents were made by Hyp Dauben, Pacault, and others from the measured isotropic susceptibilities, χ_{mol} = trace(χ_{mol}/3), assuming the "exaltation", χ_e, to arise solely from a ring current, and using the Pascal relation

$$\chi_{mol} = \Sigma\chi_b + \chi_e$$

with $\chi_{zz,nonlocal} = 3\chi_e$. Actual direct measurements of the anisotropy and asymmetry of the susceptibility tensors have been made by the crystal flip angle method, by the temperature dependent Cotton-Mouton effect, the Zeeman microwave effect, and, more recently, by the observation of direct dipolar and quadrupole splittings in high-field high resolution NMR. We have been active in making measurements of the anisotropies and asymmetries in the tensors of small molecules, including cyclic organic molecules by this method; in particular, Dr. Mark Lisicki in my lab has made a substantial body of

measurements, which I am still trying to systematize.

One of Flygare's very interesting findings, was that the ring current in cyclopentadiene is sufficiently large to contribute an exaltation of the diamagnetic susceptibilty perpendicular to the ring 1/2 as large as that in benzene. Since the area of the cyclopentadiene ring is close to 2/3 that of the benzene ring, the current must be close to 3/4 that in the benzene ring. What this seems to imply is that the HCH antisymmetric bonding orbital overlaps with the adjacent π orbitals on the neighboring carbons, and that the two electrons added to the four in the π orbitals make a nice Hueckel aromatic sextet. The six electrons whiz around the ring, not feeling much hindrance from the protons in the CH_2 group. Our meaurements on cyclopentadiene agree closely with Flygare's. Not surprisingly thiophene, furan, and pyrrole exhibit the same kind and magnitude of ring current.

If this holds in general, then both cyclopropane and cyclobutene should by analogy show aromatic ring currents, and indeed the measurements of diamagnetic susceptibility made by us and others say this is true. For example, for cyclopropane we measure $\chi_{zz} - 1/2(\chi_{xx}+\chi_{yy})$ to be $-0.19x\,10^{-28}$ cc/mol. Adding the local bond susceptibilities gives $+0.05x\,10^{-28}$ cc/mol., so $\chi_{zz,nonlocal}$ comes out to be $-0.24x\,10^{-28}$ cc/mol., almost as large as in cyclopentadiene. Cyclobutene, measured by Flygare and by us, gives $\chi_{zz,nonlocal}$ to be $-0.14x\,10^{-28}$ cc/mol., smaller. Ethylene oxide and ethyleneimine (Flygare) give very nearly the same numbers and $\chi_{zz,nonlocal}$ as cyclopropane.

8- electron cyclic arrays should give "antiaromatic" paramagnetic ring currents. Consistent with this, cyclobutane (us) and trimethylene oxide (Flygare) show susceptibility anisotropies with paramagnetic ring current contributions perpendicular to the ring, and downfield proton shifts. Cyclopentene also shows a small positive ring current.

This all seems to hang together pretty well, but I can't claim anything has been proved. I'll feel much better if I can get a more quantitative relation. In the meantime, it provides a handy empirical tool for predicting the sign of the magnetic susceptibility anisotropies in small carbon and heterocyclic rings.

Sincerely

Aksel

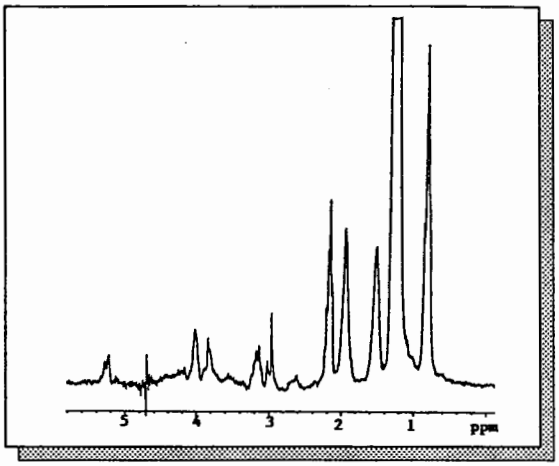
Aksel A. Bothner-By
University Professor Emeritus

The Doty XC5 Takes Decoupler Heating of Biological Samples to a New Low

in Solids, Semi-solids, and Liquids
Double-Tuned, H-F, Triple-tuned, Quad-tuned, Even Quad-tuned with Lock

Results from a Quad-tuned XC5 VAS Probe - H-F-X-N XC5 for a 500 MHz Wide Bore

- 2.5 μs π/2 pulses were obtained on all channels except ^{15}N on a 1.0 molar salt solution.
- Proton linewidth was 2.5 Hz, with excellent line shape.
- 80 kHz decoupling was obtained with ^{1}H and is expected with ^{19}F.
- Max. spinning speed: 18 kHz.
- Active sample volume: 110μL.
- B_1 inhomogeneity ~5%, all channels, for 70 μL.
- No fluorine backgrounds were detectable.
- Temperature range: -160°C to 200°C.
- Angle adjustment range: 0 to 90 degrees.
- **Thermal gradients were determined to be less than 0.7°C for a 70 μL sample of lead nitrate spinning at 13 kHz at 65°C and for 120 W dec. at 10% duty cycle.**

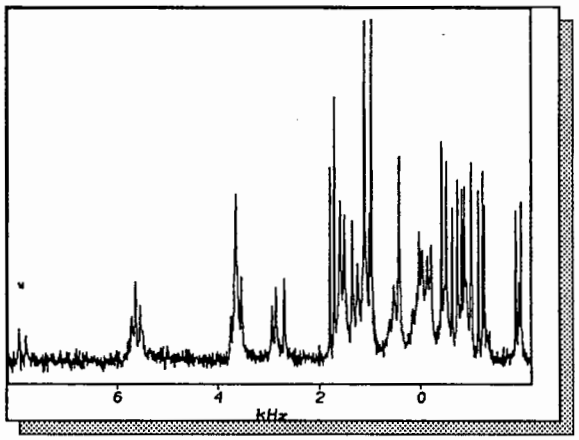


^{1}H HR Fast MAS, Ground Beef at 14 kHz. 300 MHz, nt=16, 70 μL, min. LW~5 Hz. Low thermal gradients and centrifugation.

'The next time someone tries to convince you that active sample volume, filling factor, thermal gradients, and rf efficiency aren't all that important, ask for a S/N spec for high-field REDOR, HR Fast MAS, and CPMAS.'

REDOR Probes	Rotor dia./speed	Lineshape ^{1}H Hz	Thermal Grad.	VAS	S/N,*nt=4 (500 WB)
Doty	5 mm, 18 kHz	3/30/60	1.2°C	Yes	>400
Vendor 2	4 mm, 15 kHz	~8/300/600†	~30°C	No	~150†
Vendor 3	2.5 mm, 35 kHz	?	~30°C	No	~30†

*HMB, CP. †Estimates based on available, related data.

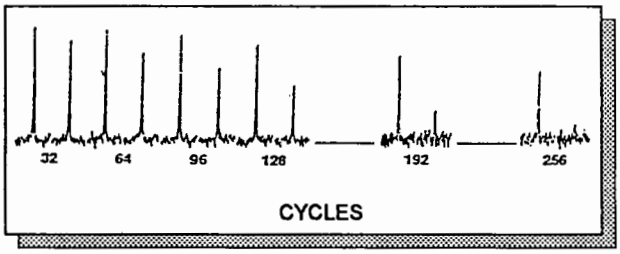


Above: Cholesterol Acetate, ^{1}H-^{13}C CPMAS, WB 500 Mhz , 64 scans in 5-mm REDOR probe. 90 kHz ^{1}H decoupling for 51 ms, 50 kHz CP for 4ms, LB=0. 40+ resonances appear resolved. Spectra courtesy of K. Zilm and B. Tounge, Yale.

Why silicon nitride stators? Compared to zirconia, silicon nitride has an order-of-magnitude higher thermal conductivity (allowing much lower thermal gradients), is an order-of-magnitude more wear resistant, and has an order-of-magnitude lower dielectric loss factor for higher Q.

Why is HF balancing necessary? The only satisfactory approach to stable HF tuning, low decoupler noise, and high salt tolerance is balanced HF.

Why is S/N so much higher than in competitors' designs? Larger sample volume helps most. Also, there are severe manufacturing limits on minimum wall thickness in hard ceramics and physical limits on coil leads. Filling factor and Q both drop rather sharply as rotor diameter drops below 5 mm. External (transmission line) tuning usually reduces LF and/or MF filling factor (as rigorously defined) by a factor of 2 to 4 in high-field REDOR probes. **Note that some competitors quote S/N for 12 scans instead of 4 scans.**



Above: WHWLQLKPGOPNLeY REDOR, 300 MHz WB, 95 kHz 1H during evolution. ^{13}C π pulses on alternate cycles. Spectra courtesy of Ruth Stark, CUNY, College of Staten Island

Making a great NMR probe involves thousands of details. Some make the difference between a fair probe and a great probe. Here are a few secrets.

Doty Zero-Susceptibility NMR RF Coil Wire and Adhesives

High-purity copper is far too diamagnetic (χ=-9.6 ppm, SI) for use in sample coils in high resolution NMR probes and in many MRI microscopy applications, and coil adhesives are an equally critical probe component in most cases. For many years, the NMR system manufacturers have carefully guarded their wire and adhesive technologies. We are the first to offer high-quality, zero-susceptibility wire, adhesives, and solder for the do-it-yourself probe builder.

This wire is manufactured by feeding hardened, high-purity aluminum wire through precision, high-purity copper tubing. The composite wire is then annealed in vacuum. The wire is currently available in six round sizes and later will also be available in several flattened sizes. When free of surface oil films, the composite wire has typical magnetization less than ±2% that of pure copper of the same outside diameter. The wire is gold plated to help protect against the formation of highly paramagnetic copper chlorides and oxides. The gold plate thickness is about 0.08 μm, or about 1% of the rf skin depth at 100 MHz. The wire is sold in individually packaged lengths of approximately 500 mm.

While the need for zero-susceptibility wire is well recognized, in most cases compensated wire by itself provides relatively little improvement in B_0 homogeneity, as numerous other factors may be comparable or even more significant. For instance, the least magnetic capacitors commercially available typically have magnetism greater in magnitude (but of opposite sign) than that of copper, so they need to be positioned at least twice their mean diameter from the sample and magnetically compensated with small pieces of diamagnetic materials such as silver or quartz.

Zero-susceptibility NMR Coil Wire		
Part #	O.D. (mm)	$/pc
90185	0.53	90
90180	0.64	80
90186	0.81	70
90179	0.97	60
90178	1.09	50
90177	1.62	40

Coil adhesives often must be magnetically compensated and should have low dielectric constant, high thermal conductivity, and very low dielectric loss (tan δ). Low proton background signal and wide (background) linewidth are also usually important. We offer several magnetically compensated, highest-quality NMR coil adhesives for probe builders. The silicones and epoxies require a cure at a minimum of 140°C.

Non-magnetic solder (96Sn-3.5Ag, m.p. ~220°C) is available in 220 g spools of rosin-core 0.7 mm wire. Part # 89875, $200/spool.

Compensated (Zero-Susceptibility) Coil Adhesives									
Part #	Adhesive Description	Viscosity	Max. Temp. °C	Therm. Cond.	^{1}H linewidth	^{1}H signal	Diel. Const.	tanδ 300 MHz	$ / 50 mL
99779	Polystyrene in toluene	med.	100	low	16 kHz	m. low	2.7	0.001	40
99778	2-part (10:1) silicone	low	240	med.	6 ppm	high	2.8	0.005	60
99776	2-part (10:1) silicone	high	250	high	15 ppm	med.	3.8	0.008	80
99775	2-part (10:1) epoxy	med.	320	low	18 kHz	m. low	3.6	0.02	50
99774	2-part (10:1) epoxy	high	350	high	25 kHz	low	4	0.012	120

Doty Scientific, Inc. 700 Clemson Rd. Columbia, SC 29229 USA
Phone: (803) 788 - 6497 Fax: (803) 736-5495 E-Mail: Sales @doty.usa.com
Website: HTTP: // www.dotynmr.com

Department of Chemistry
Gordon House
29 Gordon Square
London WC1H 0PP
Tel: 0171-380-7527 (direct)
Fax: 0171-380-7464

BIRKBECK COLLEGE
University of London

Professor J. C. Lindon

28 July 1997
(received 8/4/97)

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

Dear Barry,

NMR OF CATERPILLAR JUICE (FRESHLY SQUEEZED)

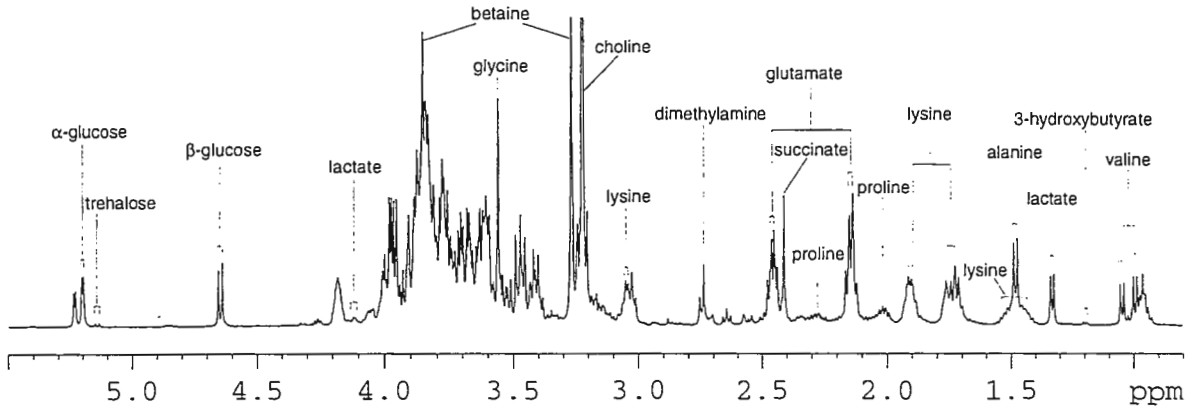
For some time we have been interested in how soil pollutant chemicals affect invertebrate species' biochemistry and this has led us into a search for novel biomarkers of environmental toxicity.

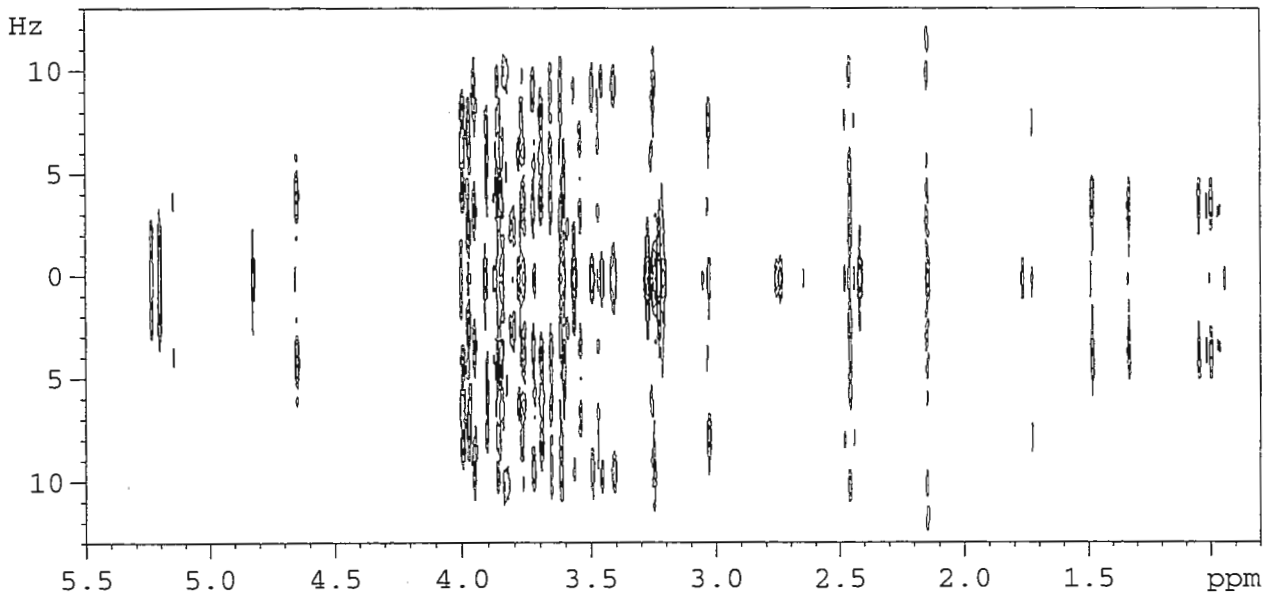
One way of doing this is to examine the biochemical composition of the substance known as haemolymph found in insects, their larvae and pupae and which is used by them in much the same way we use blood plasma. We have been analysing haemolymph using high field high resolution NMR spectroscopy and the spectra show a wealth of well resolved resonances with high sensitivity.

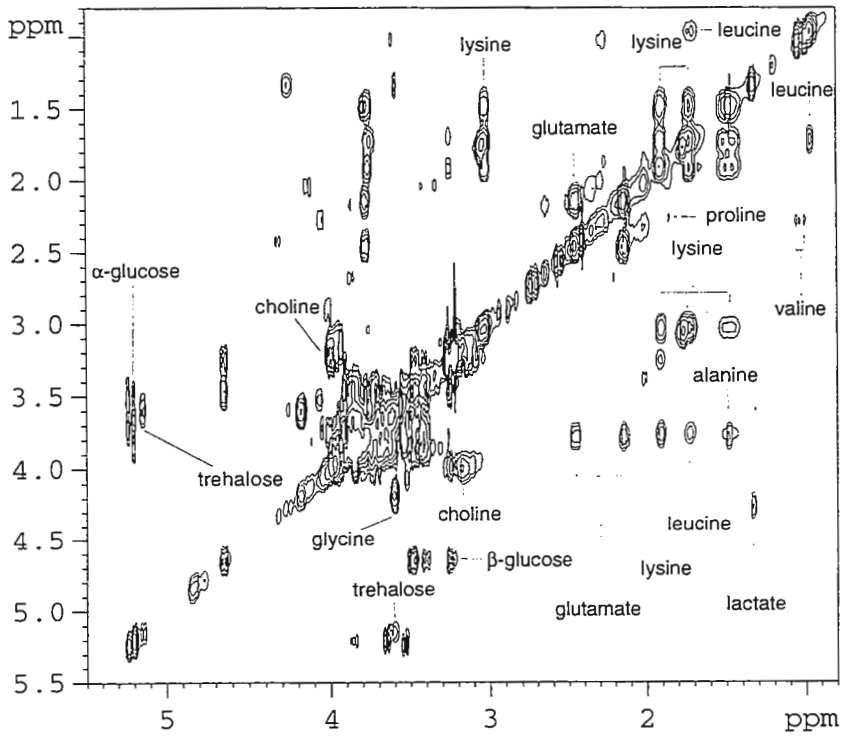
Initialy, we have been looking at the haemolymph from the Tobacco Hornworm Moth *Manduca sexta* caterpillar and typical results are shown in the figure. This work has been done in collaboration with Dr. Dan Osborn and his colleagues at the Institute for Terrestrial Ecology in the UK, Prof. Stuart Reynolds from the University of Bath, UK and Prof. Ian Wilson of Zeneca, Macclesfield, UK and funded by the UK National Environmental Research Council and the Royal Thai Government. The upper trace is the conventional 1-dimensional ^{1}H spectrum with NOESYPRESAT water suppression, the middle trace is a ^{1}H homonuclear J-resolved spectrum whilst the bottom trace is the ^{1}H-^{1}H TOCSY spectrum. Assignments of many of the endogenous metabolites can be made based upon our earlier work on mammalian biofluids and some are marked on the spectra. We are now moving on to look at other species of caterpilar and pupa and to investigate the effects of pesticides and other environmentally important substances. It is likely that NMR spectroscopy of insect biofluids will become an important tool in deriving biomarkers of environmental effects and for investigating the biochemical mechanisms of environmental toxicity.

Yours sincerely,

CHITCHOL PHALARAKSH JEREMY NICHOLSON JOHN LINDON


betaine
choline
glycine
glutamate
dimethylamine
lysine
3-hydroxybutyrate
α-glucose
β-glucose
lactate
succinate
alanine
valine
proline
trehalose
lysine
lactate
proline
lysine
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
ppm


Hz
10
5
0
5
10
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
ppm


ppm
lysine
lysine
leucine
leucine
glutamate
proline
lysine
α-glucose
choline
valine
alanine
trehalose
choline
leucine
glycine
β-glucose
lysine
trehalose
glutamate
lactate
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
5
4
3
2
ppm

THE UNIVERSITY OF TEXAS
SOUTHWESTERN MEDICAL CENTER
AT DALLAS

Department of Radiology
The Mary Nell and Ralph B. Rogers
Magnetic Resonance Center

Southwestern Medical School
Southwestern Graduate School
of Biomedical Sciences
Southwestern Allied Health Sciences School

August 6, 1997
(received 8/11/97)

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

Title: CORVUS in Depth

Dear Barry,

In the December 1996 edition of the NMR Newsletter (No. 459), I tantalized the readers with a brief introduction to my program (CORVUS) that simulates the real channel in-phase, on-resonance NMR signals of spin-3/2 nuclei with a residual quadrupole splitting. CORVUS simulates the effects of motion (through various distributions and time dependencies of the quadrupole frequency ω_Q) on the NMR signals from several different "two-pulse" pulse sequences.

In the following discussion, τ is the time between the two pulses and t is the total time after the initial pulse. In various multiple-quantum filters with preparation time τ and a very small evolution time, the final pulses can be considered as a composite second pulse in a two-pulse sequence. A single pulse can be considered as a two-pulse sequence with $\tau = 0$.

I have written the NMR signals from various pulse sequences as a general equation that is a linear combination of five phase coherences with numerical coefficients that depend on the particular pulse sequence:

$$S(t > \tau) = \sum_{i=1}^{5} c_i \, PC_i$$

where

$$PC_1 = \exp[-s_2 t\,]$$

$$PC_2 = \exp[-s_1 t\,] \cos[\omega_Q t]$$

$$PC_3 = \exp[-s_1 \tau\,] \exp[-s_2 (t - \tau)] \cos[\omega_Q \tau]$$

$$PC_4 = \exp[-s_2 \tau\,] \exp[-s_1 (t - \tau)] \cos[\omega_Q (t - \tau)]$$

$$PC_5 = \exp[-s_1 t\,] \cos[\omega_Q (t - 2\tau)].$$

Note that PC_5 is a spin echo that comes from refocussing of accumulated phase angle.

TABLE 1. Numerical coefficients c_i of the various phase coherences in NMR signals from one-pulse, quadrupole echo, and several multiple quantum filter sequences.

Phase Coherence	One-Pulse	QE	TQ	DQ	DQ-MA	Composite
PC_1	0.400	0.100	-0.159	0.106	0	0
PC_2	0.600	-0.150	-0.080	0.265	0.141	0.127
PC_3	0	0.300	0.159	-0.106	0	0
PC_4	0	0.300	0.159	-0.106	0	0
PC_5	0	0.450	-0.080	-0.159	-0.141	-0.127

The DQ-MA filter is the double quantum filter in which the two pulses after the preparation time are "magic angle" flip pulses. Like the other multiple-quantum filters, it is zero right after the pulse sequence. It remains zero at all times t that are greater than τ unless $\omega_Q \neq 0$, unlike the other filters. Therefore, it is a sensitive detector of residual quadrupole splitting. The "Composite" filter is formed, after data collection, from a linear combination of the TQ and DQ filters that have all 90° pulses. Because the TQ and DQ filters are less sensitive to errors in flip angle than the DQ-MA filter, the Composite filter may have some advantages in spite of the somewhat smaller signal amplitude. In the above equations, s_1 is the transverse relaxation rate of the satellite transitions and s_2 is that of the central transition.

CORVUS takes motion into account by causing the ω_Q value of a given nucleus to be time dependent. The sample is comprised of spatial regions (domains) that have different ω_Q values. The diffusion of nuclei among the domains causes this time dependency. The basic task in using the phase coherences to describe transverse magnetization when the ω_Q value of a given nucleus is time dependent is to evaluate averages of terms that have the form $\cos[\Phi(t)]$ where

$$\Phi(t) = \int_0^t \omega_Q(t)\, dt,$$

in which the distribution of $\omega_Q(t)$ values is stationary and the time t is appropriately defined. To evaluate $\Phi(t)$, we assume that a spin experiences a constant ω_Q for periods of various durations, DUR. The value of DUR is chosen randomly:

$$DUR = -\tau_e \log_e(RND),$$

where RND is a new random number and τ_e is the exchange or correlation time. At the initial time, a value of ω_Q is chosen randomly from the distribution. At the end of the period DUR, a new value of ω_Q and the next value of DUR are chosen randomly. The value of Φ for a given nucleus after n durations is given by

$$\Phi_n = \sum_{i=1}^{n} \omega_{Qi}\, DUR_i.$$

For the spin echo, the value of Φ accumulated from time $t = 0$ to $t = \tau$ is multiplied by -1 and further phase accumulation also obeys this equation. To simulate a motionless system, a duration greater than the longest time in the computer experiment can be added to *DUR*. The net phase coherence is calculated from the summation over the normalized distribution of ω_Q values.

CORVUS works as follows. The five phase coherences are calculated for a given set of input parameters, except for the transverse relaxation rates s_1 and s_2. Then, using these phase coherences, all of the various NMR signals can be rapidly calculated for chosen values of s_1 and s_2.

Simulations based on CORVUS are useful in interpreting the ^{23}Na NMR signals in incompletely disordered aqueous heterogeneous systems. They can aid in interpreting 1-pulse and QE signals and also provide a powerful tool in quantifying multiple-quantum filter signals. This is done most straightforwardly with time-domain signals, compared to the frequency domain spectra. For example, all of these signals have been quantified for a sample made from xanthan gum (a constituent of many common gourmet food items such as catsup and jalapeño sauce).

An extensive description of CORVUS together with deep insights gained from contemplating signals from a wide range of physical situations is about to be submitted to the *Journal of Magnetic Resonance*.

Sincerely,

Don

Donald E. Woessner

FIFTH INTERNATIONAL CONFERENCE ON HETEROATOM CHEMISTRY

UNIVERSITY OF WESTERN ONTARIO
LONDON, ONTARIO CANADA
JULY 5-10, 1998

ICHAC-5

August 21, 1997

Dear Barry,

I've just mailed First Announcements for *ICHAC-5* to people whose names are included in a variety of mailing lists. We requested that potential attendees indicate that they would like to receive further information; we also requested tentative titles for talks and posters. Since about 10% of the titles I've received so far include NMR studies on various nuclei, I thought some of your readers might be interested in obtaining more information about this Symposium.

The Plenary Speakers include

Derek H. R. Barton, USA
Valery K. Brel, Russia
Robert Corriu, France
Li-Xin Dai, P. R. China
Yong Hae Kim, Korea
Anthony J. Kirby, UK
Gerald Pattenden, UK
Edward Piers, Canada
Peter J. Stang, USA
Kohei Tamao, Japan
Michael Veith, Germany
Peter Wipf, USA
Binne Zwanenburg, The Netherlands

The Invited Speakers include

Jose Barluenga, Spain
Matthias Drieß, Germany
William J. Leigh, Canada
Marian Mikołajczyk, Poland
Shlomo Rosen, Israel
Ryo Sato, Japan
Norihiro Tokitoh, Japan
Masaaki Yoshifuji, Japan

Information on this Symposium (and on the 31st Organosilicon Symposium, May 29-30, 1998 in New Orleans) can be obtained by writing me at University of Western Ontario, Department of Chemistry, London, Ontario, Canada N6A 5B7, faxing me at (519) 661-3022 or sending me an e-mail message, mgordon@julian.uwo.ca

The XIVth International Conference on Phosphorus Chemistry - ICPC will be held in Cincinnati, July 12-17, 1998, the week following *ICHAC-5*. One of their topics is ^{31}P NMR in Biological Systems. The Conference Chair is F. H. Ebetino, (513) 622-3630, phone and (513) 622-1195, fax. Additional information can be found on their web site, http://www.usc.edu/dept/chemistry/ICPC14/index.html

Sincerely,

Myra

Myra Gordon

DEPARTMENT OF CHEMISTRY, UNIVERSITY OF WESTERN ONTARIO, LONDON, ONTARIO CANADA N6A 5B7
(519) 661-2166 PHONE (519) 661-3022 FAX
ORGANIZERS: KIM M. BAINES AND JAMES F. KING
KBAINES2@JULIAN.UWO.CA SCIJFK@UWOADMIN.UWO.CA

NEW AVANCE DEVELOPMENTS

BRUKER

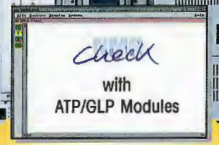
CCU/9
Highest Speed through Fast Ethernet

CE ISO 9001
Enhanced Safety and Quality

MULTIPLE RECEIVER

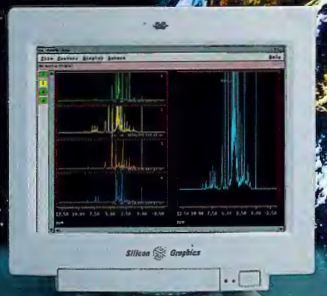
ADC
16bit, 2 MSample/s

The most comprehensive Software available in industry



SERVICE Software

AUTOMATION and ANALYSIS Software



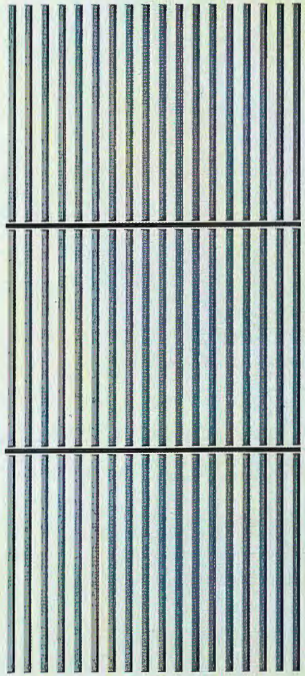
SHIELDED MAGNETS

http://www.bruker.de
http://www.bruker.com

1997

DIGITAL NMR

AVANCE 600



BRUKER

EUROPE

Germany
BRUKER ANALYTIK GMBH
Software Department
Rudolf-Plank-Str. 23
D-76275 Ettlingen
Tel. (0 72 43) 50 44 21
Fax (0 72 43) 50 44 80

BRUKER MEDIZINTECHNIK GMBH
Rudolf-Plank-Str. 23
D-76275 Ettlingen
Tel. (0 72 43) 50 45 33
Fax (0 72 43) 50 45 39

BRUKER-FRANZEN ANALYTIK GMBH
Fahrenheitstr. 4
D-28359 Bremen
Tel. (04 21) 22 05-0
Tx. 246404
Fax (04 21) 22 05-1 00

BRUKER SAXONIA ANALYTIK GMBH
Permoserstr. 15
D-04318 Leipzig
Tel. (03 41) 24 31 30
Fax (03 41) 2 43 14 00

France
ODAM, Groupe Bruker
19, avenue de la gare
F-67162 Wissembourg/Cedex
Tel. (03) 88 63 36 00
Tx. (042) 870261
Fax (03) 88 54 36 32

SADIS BRUKER SPECTROSPIN
34, rue de l'industrie
F-67166 Wissembourg/Cedex
Tel. (03) 88 73 68 00
Fax (03) 88 73 68 79

SADIS BRUKER SPECTROSPIN PARIS
Bureau Parc No. 1
3, avenue du Général de Gaulle
F-91090 Lisses
Tel. (01) 69 89 85 20
Fax (01) 60 86 34 90

Switzerland
SPECTROSPIN AG
Industriestrasse 26
CH-8117 Fällanden
Tel. (01) 8 25 91 11
Tx. 828416 spec ch
Fax (01) 8 25 96 96

Netherlands
BRUKER SPECTROSPIN NV
Bruynvisweg 18
NL-1530 AB Wormer
Tel. (075) 6 28 52 51
Fax (075) 6 28 97 71

Scandinavia
BRUKER SPECTROSPIN AB
Polygonvägen 79
S-18766 Täby
Tel. (08) 4 46 36 30
Fax (08) 6 30 12 81

Belgium
BRUKER SPECTROSPIN S.A.
4, avenue du Four à Briques
4 Kareelovenlaan
B-1140 Bruxelles - Brussel
Tel. (02) 7 26 76 26
Fax (02) 7 26 82 82

Israel
BRUKER SCIENTIFIC ISRAEL LTD.
Kiryat Weizmann-Science Park
P.O. Box 2445
Rehovot 76 123
Tel. (08) 9 40 96 60
Fax (08) 9 40 96 61

Italy
BRUKER SPECTROSPIN SRL
Via Giovanni Pascoli 70/3
I-20133 Milano
Tel. (02) 70 63 63 70
Fax (02) 23 61 294

United Kingdom
BRUKER SPECTROSPIN LTD.
Banner Lane
Coventry CV4 9GH
Tel. (01203) 85 52 00
Fax (01203) 46 53 17

Spain
BRUKER ESPAÑOLA, S.A.
Parque Empresarial San Fernando
Avda. de Castilla, 2
Edificio Berlin pta. baja
E-28831 San Fernando de Henares (Madrid)
Tel. (01) 504 62 54
Fax (01) 656 62 37

North America

USA
BRUKER INSTRUMENTS, INC.
Manning Park
19 Fortune Drive
Billerica, Ma. 01821-3991
Tel. (508) 667 9580
Tx. (023) 4977305
Fax (508) 667 3954

BRUKER INSTRUMENTS, INC.
3411 Silverside Road
Webster Building, Suite 107
Concord Plaza
Wilmington, Delaware 19810
Tel. (302) 478 8110
Fax (302) 478 1431

BRUKER INSTRUMENTS, INC.
47697 Westinghouse Drive
Fremont, CA 94539
Tel. (510) 683 4300
Fax (510) 490 6586

BRUKER INSTRUMENTS, INC.
5111 Academy Drive
Lisle, Illinois 60532
Tel. (708) 971 4300
Fax (708) 971 4303

BRUKER INSTRUMENTS, INC.
2635 North Crescent Ridge Drive
The Woodlands, TX 77381
Tel. (281) 292 2447
Fax (281) 292 2474

Canada

BRUKER SPECTROSPIN LTD.
555 Steeles Avenue East
Milton, Ontario L9T 1Y6
Tel. (905) 876 4641
Fax (905) 876 4421

BRUKER SPECTROSPIN LTD.
9865 West Saanich Road
Sidney, B.C. V8L 3S1
Tel. (604) 656 1622
Fax (604) 656 7289

AUSTRALIA

BRUKER (AUSTRALIA) PTY. LTD.
Unit 7 / 163, McEvoy Street
P.O. Box 202
Alexandria, New South Wales 2015
Tel. (02) 9550 64 22
Fax (02) 9550 36 87

ASIA

India
BRUKER INDIA SCIENTIFIC Pvt. Ltd., 48, B Wing, Abhishek Lokandwala Complex
Char Bungalows, Azad Nagar
Bombay 400053
Tel. (022) 62 62 232
Tx. 1179340 deep
Fax (022) 62 68 844

Japan
BRUKER JAPAN CO. LTD
21-5, Ninomiya 3-Chome
Tsukuba-shi, Ibaraki-ken 305
Tel. (0298) 52 12 34
Fax (0298) 58 03 22

P. R. China
BRUKER INSTRUMENTS, LTD.
Science and Technology Tower
12th Floor, Suite 1203
11 Baishiqiao Road
Beijing 100 081
Tel. (010) 6847 7036
Fax (010) 6847 7035

NMR 8961/02/97 3.000 S&P

BRUKER

BRUKER

Address all Newsletter correspondence to:

Dr. B. L. Shapiro
The NMR Newsletter
966 Elsinore Court
Palo Alto, CA 94303.
☎650-493-5971* - *Please call only between 8:00 am and 10:00 pm, Pacific Coast time.*

Deadline Dates

No. 469 (Oct.)	26 Sept. 1997
No. 470 (Nov.)	24 Oct. 1997
No. 471 (Dec.)	21 Nov. 1997
No. 472 (Jan.)	19 Dec. 1997
No. 473 (Feb.)	23 Jan. 1998

* Fax: ☎650-493-1348, 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

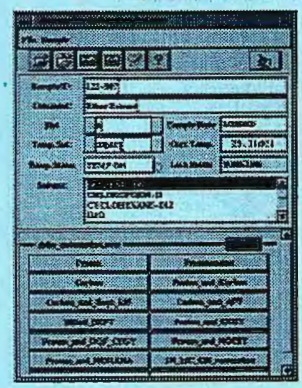
☎ Effective August 1, 1997, our area code is *650*.

☆ **The Newsletter's fiscal viability depends very heavily on the funds provided by our Advertisers and Sponsors. Please do whatever you can to let them know that their support is noted and appreciated.**

Mailing Label Adornment: Is Your Dot Red ?

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

How To Run JEOL's Eclipse+ Spectrometer



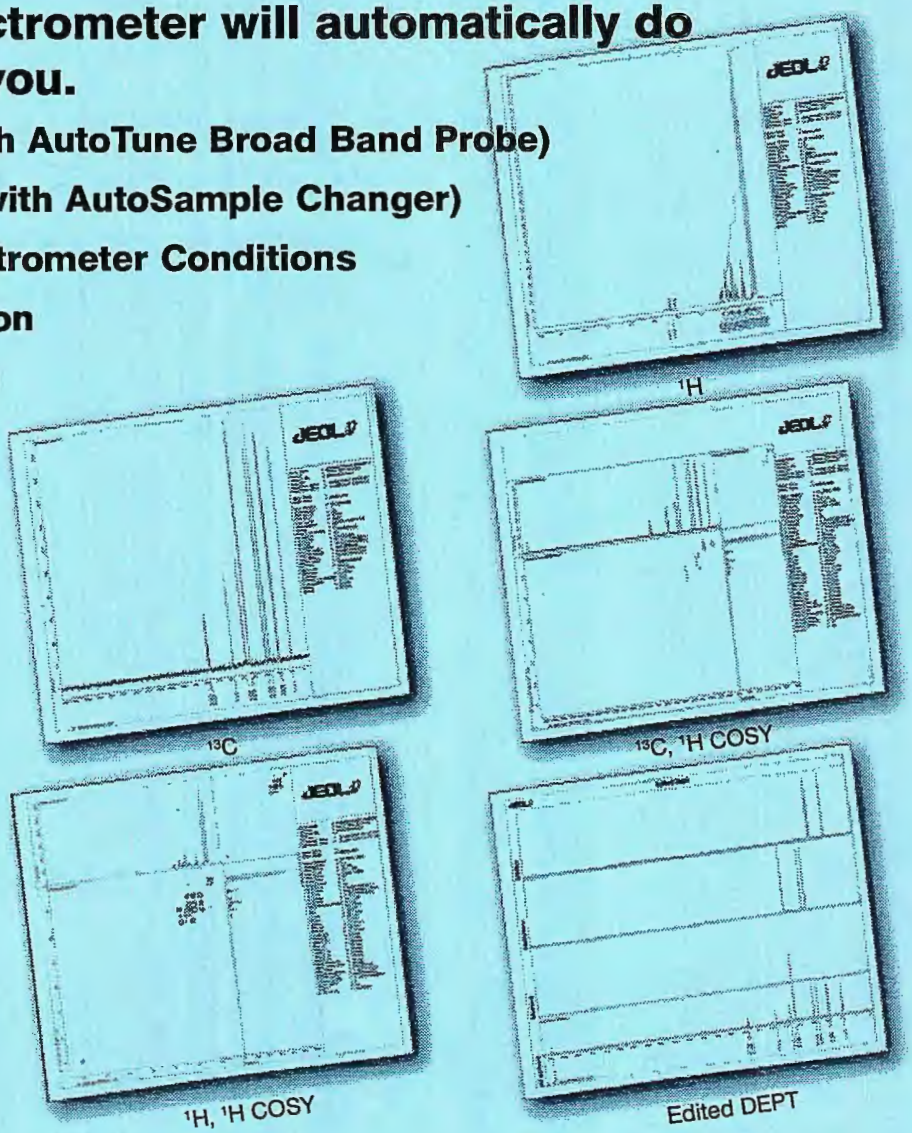
Step 1: Enter your sample name and the solvent.

Step 2: Click the mouse button on the data you want.

Step 3: Walk away with your data.

JEOL's Eclipse Spectrometer will automatically do everything else for you.

- ✔ Auto Probe Tuning (with AutoTune Broad Band Probe)
- ✔ Auto-sample Control (with AutoSample Changer)
- ✔ Auto Selection of Spectrometer Conditions
- ✔ Auto Baseline Correction
- ✔ Auto Data Presentation
- ✔ Auto Phase Correction
- ✔ Auto Digital Filtering
- ✔ Auto S/N Monitoring
- ✔ Auto Queue Control
- ✔ Auto Receiver Gain
- ✔ Auto Data Storage
- ✔ Auto Referencing
- ✔ Auto Processing
- ✔ Auto Peak Picks
- ✔ Auto Integration
- ✔ Auto Plotting
- ✔ Auto Shim
- ✔ Auto Lock



JEOL USA, Inc., 11 Dearborn Road, Peabody, MA 01960
Tel: (508)535-5900 Fax: (508)536-2205
Email: nmr@jeol.com WWW: http://www.jeol.com

JEOL