THE NMR NEWSLETTER

No. 505

October 2000

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## FORTHCOMING NMR MEETINGS

Contributions of NMR to Structural Biology, Biozentrum Basel, Switzerland, **October 18, 2000**. Contact: S. Frauenknecht, Tel: +-41-61-267-21-01; E-mail: sabine.frauenknecht@unibas.ch.

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

36th Western Regional Meeting of the American Chemical Society, Cathedral Hill Hotel, San Francisco, CA, **October 25-28, 2000**. Abstract deadline: September 29. Contact: Uschi Simonis, Assoc. Prof., Dept. of Chemistry & Biochemistry, San Francisco State University, 1600 Holloway Ave., San Francisco, CA 91432; Tel: 415-338-1656; Fax: 415 338-2384; E-mail: uschi@sfsu.edu; or Dr. Silvio Rodriguez, Tel:209-946-2598; Fax: 209-946-2600; E-mail: srodriguez@uop.edu.

Protein Structure: Models, Fold, and Function Analysis Applications for Target Validation, McLean, Virginia, **October 26-27, 2000**. Contact: Mary Chitty, Cambridge Healthtech Institute, mchitty@healthtech.com; Fax 617-630-1325.

International Conference on Structural Genomics 2000, Yokohama, Japan, **November 2-5, 2000**. Contact: ICSG2000 Secretariat Protein Research Group, Genomic Sciences Center, RIKEN, 2-1 Hirosawa, Wako, Saitama, Japan. Tel: +81-(0)48-467-9427; Fax: +81-(0)48-462-4675; E-mail: icsg@icsg2000.riken.go.jp.

37th New Mexico Regional NMR Meeting, Albuquerque, New Mexico, **November 11, 2000**. Contact Eiichi Fukushima, New Mexico Resonance, 2301 Yale Blvd, SE; Suite C-1, Albuquerque, NM 87106-4237; E-mail: http://www.nmr.org. See Newsletter 505, 22. For more information: http://www.unm.edu/~karenann/nmr2.html

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

Frontiers of NMR in Molecular Biology VII, Big Sky, Montana, **January 20-26, 2001**. Contact: Keystone Symposia, Drawer 1630, 221 Summit Place, Suite 272, Silverthorne, CO 80498. Tel: 800-253-0685 or 970-262-1230; Fax: 970-262-1525; E-mail: keystone@symposia.com. http://www.symposia.com.

*continued on page 32*

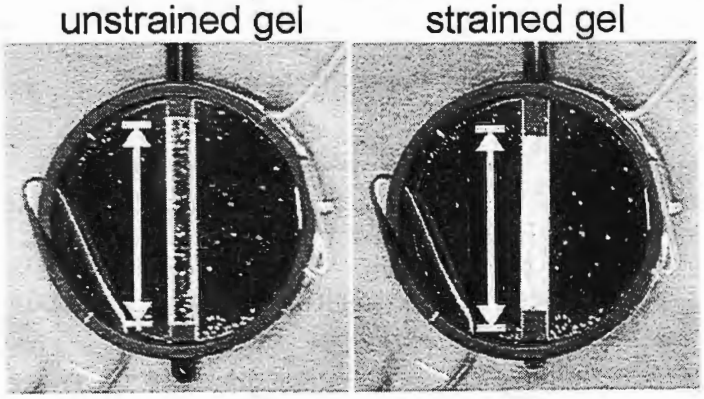
DEPARTMENT OF HEALTH & HUMAN SERVICES Public Health Service

National Institutes of Health
Laboratory of Chemical Physics, NIDDK
Building 5, Room 112
Bethesda, Maryland 20892-0520
September 18, 2000 (received 9/18/2000)

**Orientation of biopolymers in solution by "strain-induced alignment in a gel (SAG)"**

Dear Barry:

Yoshitaka Ishii, Francisco J. Blanco, and I have recently demonstrated that a protein can be weakly aligned by dissolving it in an aqueous gel rod (*e.g.*, crosslinked polyacrylamide) with a diameter less than the inner diameter of an NMR tube and then compressing the gel so that it shrinks in length and expands in diameter. The figure below shows such a gel positioned between crossed polarizers and illuminated from the back. Upon compression, the gel lights up, indicating the creation of an anisotropic medium and the concomitant development of optical birefringence. We were able to measure $^{1}H$-$^{15}N$ dipole-dipole couplings in HSQC spectra of $^{15}N$-labeled protein G/B1 in a compressed gel and to show that these dipole-dipole couplings correlate well with the known protein structure (*J. Am. Chem. Soc.*, in press). Strain-induced alignment in a gel (SAG) has the advantageous features that it works over wide ranges of temperature, pH, and ionic strength, that valuable samples can be recovered by placing the gel in an excess volume of buffer and allowing the protein or other molecules to diffuse out, and that the degree of alignment can be varied by varying the extent of compression, the gel density, and possibly other parameters.




We offer the following tips to anyone who may want to try SAG: (1) We have found that casting the gel rods in glass tubes, rather than in teflon molds as originally reported, leads to smoother gel surfaces and somewhat better spectra; (2) We normally wash the gel rods in water for several days after polymerization, to remove unreacted reagents; (3) Uniform strain (*i.e.*, compression or extension) is essential. If the initial gel diameter is too small, the gel may buckle when compressed. The strain in the gel is then not uniform and inhomogeneous broadening results; (4) Some degree of inhomogeneous broadening of the NMR lines may be unavoidable, because the gels are made by a random polymerization process that inevitably involves structural disorder, but this broadening should not be so severe as to preclude acquisition of useful data; (5) Shigemi-type NMR tubes are ideal for SAG experiments, but take care not to twist the glass plunger when the gel is compressed, otherwise the gel may tear; (6) It will probably prove necessary to tailor the gel composition (%T and %C, in gel nomenclature) to match the size of the protein or other biopolymer. If the gel "pore size" is too small, the protein will not diffuse uniformly into the gel. If the pore size is too large, the degree of alignment may be too small.

Sincerely yours,

Robert Tycko

Dr. Robert Tycko
phone: 301-402-8272
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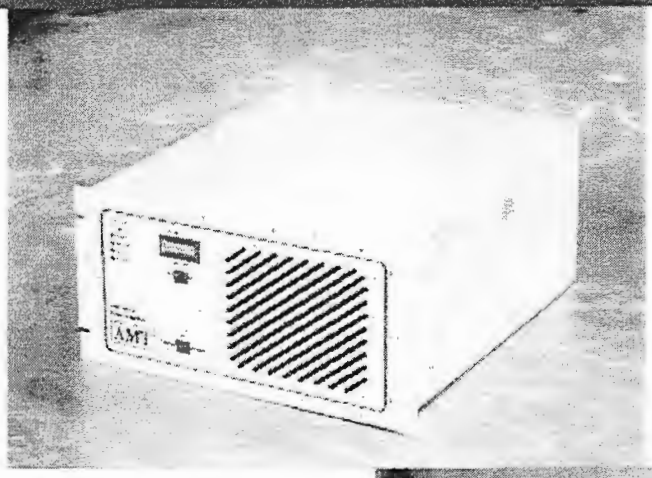

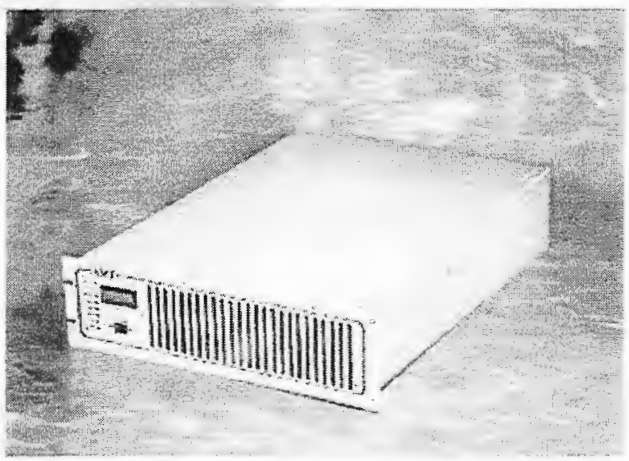

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Halle, August 22, 2000
(received 8/29/2000)

Prof. B.L. Shapiro
The NMR Newsletter,
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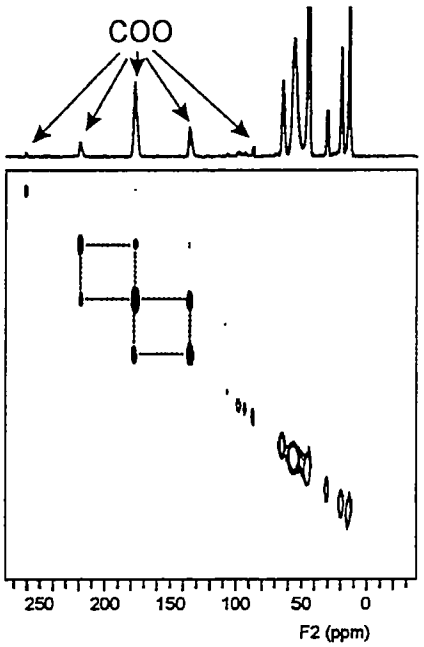
# *2D or NOT 2D ?!?!?* ☺

Dear Barry,

some time ago, we became interested in the investigation of slow segmental processes in organic solids like polymeric systems, proteins etc. To avoid the necessity of using isotopically enriched substances, we focused on MAS techniques. The well known 2D-MAS exchange experiment of Kentgens & Veeman [1], later improved by Hagemeyer & Spiess [2], seems to fit our purpose well: it detects changes of the anisotropic chemical shift due to reorientations of the chemical shift tensors on a time scale of milliseconds up to some seconds and provides reasonable spectral resolution and signal-to-noise. As an example, the 1st figure displays the 2D-MAS exchange spectrum of the amorphous polymer PnBMA; the cross peaks linking the spinning-side bands (ssb) of the COO carbon at 178ppm (see dashed line in spectrum) indicate the presence of molecular reorientations. Please note that this method requires the presence of ssb, i.e. slow MAS rates.

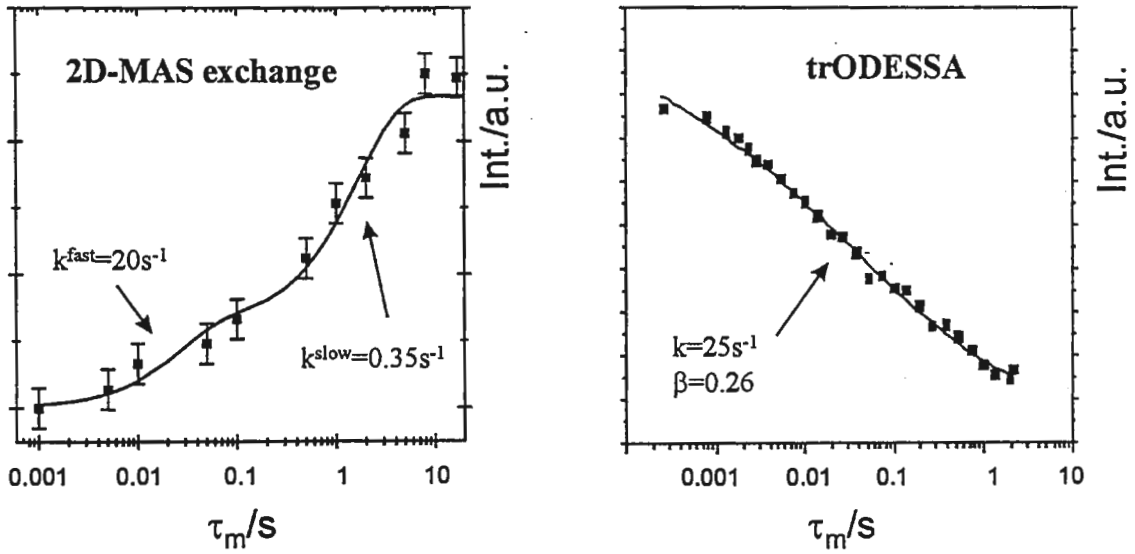



To obtain information about the time constant of this process, we need to run such experiments for a number of mixing times and plot the growing intensity of these cross peaks (or, alternatively, the decay of the main-diagonal ssb) vs. the mixing time and fit this dependence with an appropriate function. For low-molecular weight organic solids, a purely exponential behaviour is expected, while for polymers, a more complicated behaviour (double-exponential or stretched exponential) is more common. The 2nd figure shows the intensity of the most intense COO cross peak (linking the N=0 with the N=-1 ssb). In a reasonable experimental time (about four days), it was possible to acquire 2D spectra for 11 logarithmically spaced mixing times only; the signal-to-noise, as obtained from the spectra, is indicated by error bars. We added a theoretical function, assuming two dynamic processes with different time constants. However, it is very clear that by the quality of data (number of mixing times and signal-to-noise), the experimental data are compatible with a wide variation of the dynamic parameters (these given in the figure are far from being unambiguous). We thus needed to conclude that is fairly impossible to obtain reliable information about the dynamic behaviour of such systems with the existing zoo of NMR techniques.

This prompted us to search for a more time-efficient method; i.e. one that delivers the desired dynamic information but provides a better signal-to-noise in shorter experimental time. Since in a 2D experiment, one needs to acquire a number of $t_1$ increments and those do not contribute to the overall signal-to-noise as additional data accumulations would do, we asked ourselves: Is there a 1D-experiment that does the job? Well, most of us know

the 1D-Magnetizations transfer experiment [3], that detects dynamic processes which change the *isotropic* chemical shifts (like H-shifts etc.). However, it is not justified to apply its idea to ssb belonging to the same isotropic chemical shift (i.e. to processes that change the *anisotropic* chemical shift by the reorientation of the tensors only): ssb are not NMR lines in the common sense but representations of the periodicity of the MAS-time domain signal! Thus, other ideas must be developed, based on the properties of MAS-ssb.

The 1st approach was the TOSS-exchange method of Yang & Blümich [4], however, due to the delicateness of the TOSS building block and the signal loss by the additional pulses, the overall gain in signal-to-noise is not satisfying. We find that ODESSA-type experiments [5], in particular the time-reverse ODESSA [6] are much better suited: requiring a minimum number of pulses and length of $T_2$–sensitive delays, it maintains a very good signal-to-noise and provided exactly the information we are interested in, namely the decay of the intensity of the desired NMR line vs. the mixing time which tells us about the molecular reorientation. The 3rd figure shows the time-reverse ODESSA decay, recorded under the same condition and within the same overall experimental time as for the 2nd figure. The number of mixing times is about three times as big as before and the signal-to-noise is now in the order of the size of the symbols. It is obvious that the decay must be described by a stretched exponential function; the dynamic parameters can be determined with good accuracy.




We applied the time-reverse ODESSA method to a couple of problems, including molecular dynamics in organic solids and proteins as well as to spin dynamics (spin diffusion). We currently focus on polymers applications as described above. We like to point out that the power of ODESSA-type experiments is the ability to determine dynamic time constants with high accuracy while they are less well suited for the investigation geometrical parameters (jump angles etc.). For that, either static method (requiring isotopical enrichment) or alternative MAS methods (like CODEX [7]) must be used, however, the latter provides only inferior signal-to-noise, as compared to the ODESSA. If one is aimed to the latter problems, the combination of the methods might be the way to go.

Best Regards

Detlef Reichert Horst Schneider Ovidiu Pascui


[1] A.P.M. Kentgens, A.F. de Jong and W.S. Veeman, *Macromolecules* **18**, 1045-1048 (1985).
[2] A. Hagemeyer, K. Schmidt-Rohr and H.W. Spiess, *Adv. Magn. Reson.* **13**, 85-130 (1989).
[3] N.M. Szeverenyi, A. Bax and G.E. Maciel, *J. Amer. Chem. Soc.* **105**, 2579-2582 (1983).
[4] Y. Yang, M. Schuster, B. Blümich and H.W. Spiess, *J. Magn. Reson.* **139**, 239-243 (1987).
[5] V. Gerardy-Mountouillout, C. Malveau, P. Tekely, Z. Olender and Z. Luz, *J. Magn. Reson.* **A123**, 7-15 (1996).
[6] D. Reichert, H. Zimmermann, P. Tekely, R. Poupko and Z. Luz, *J. Magn. Reson.* **125**, 245-258 (1997).
[7] E.R. DeAzevedo, W.-G. Hu, T.J. Bonagamba and K. Schmidt-Rohr, *J. Am. Chem. Soc.*, **121**, 8411-8412 (1999)

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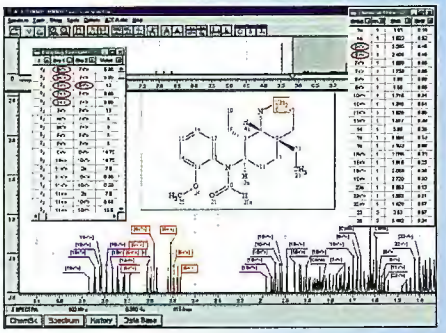
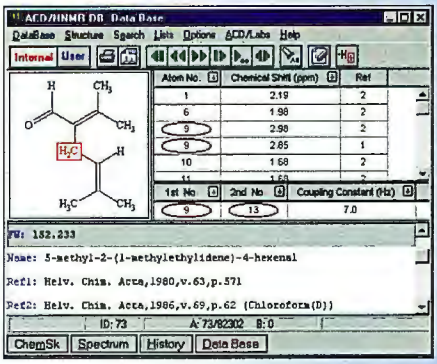


View coupling constants with highlighted atoms

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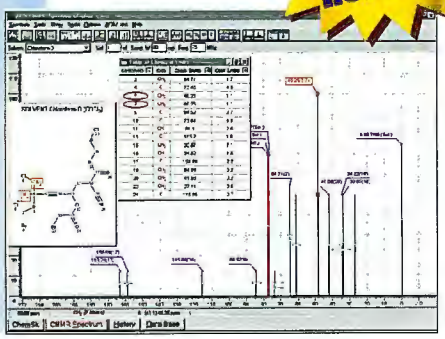
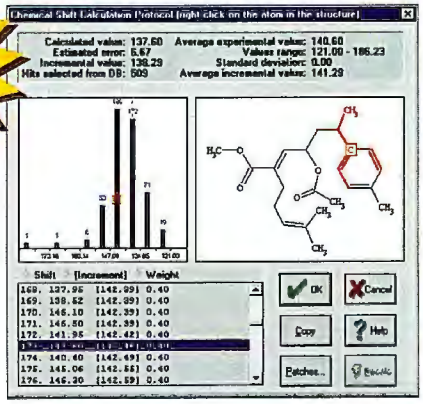


Verify shifts & coupling constants with on-screen structural relation

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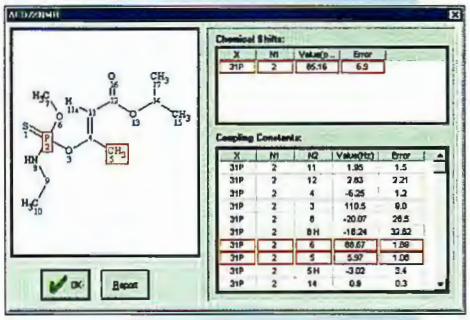
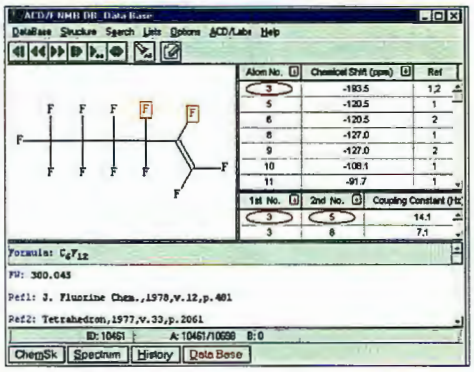
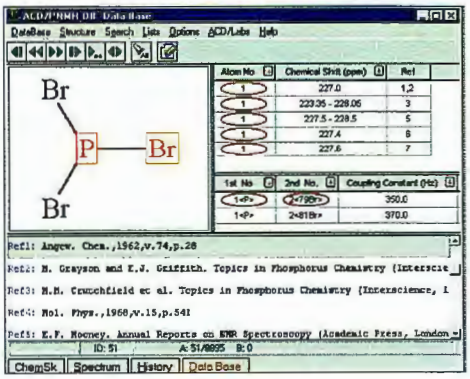


Display the predicted structure, interactively related to assigned shifts & coupling constants



Phosphorus database window showing chemical shifts, coupling constants, references, formula and IUPAC name

Fluorine Database window showing chemical shifts, coupling constants & references



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The NMR Newsletter
966 Elsinore Court
Palo Alto, CA 94303

September 8, 2000
(received 9/13/2000)

Re: NMR of Membrane Glycolipids (Lipid A) in *Rhizobia* (Symbiotic Bacteria)

Dear Barry,

We are continuing collaborative efforts with Prof. Chris Raetz (Duke, Biochemistry) to study bacterial glycolipids like Lipid A from the outer membrane of Gram-negative bacteria. A single Lipid A species lacking an acyloxyacyl moiety was previously described in *Rhizobia*. Dr. Nan Que has recently isolated six related Lipid As from *Rhizobia* at quantities sufficient for NMR analyses. Compounds **B**, **C**, **D-1** and **E** are physiologically relevant species. Two glucosamine units and one galacturonic acid (designated I, II and III) are observed in the COSY (Fig. 1) and TOCSY (Fig. 2) spectra of species **B**. Because the proximal glucosamine is not phosphorylated, both α and β forms of the proximal glucosamine are detected (designated I and $I_\beta$).

COSY reveals that H-3 and H-3' (~5.2 and 5.3 ppm, respectively) of the proximal and distal glucosamines of **B** are considerably downfield shifted relative to other sugar oxymethine resonances. Thus, COSY provides unequivocal evidence for the O-acylation sites of the β-hydroxy acid chains at the 3 and 3' positions of the glucosamine rings.

The β-oxymethine protons of 3-hydroxyacyl chains resonate near 3.7-4.1 ppm if the 3-OH is unsubstituted, but near 5.2 ppm if the 3-OH is further acylated. The number and substitution state of the 3-hydroxyacyl chains can thus be estimated from the number of cross peak pairs between α-methylene (2.2-2.6 ppm) and β-oxymethine protons, as well as γ-methylene (1.2-1.6 ppm) and β-oxymethine protons. The COSY of **B** displays five α/β and γ/β cross peak pairs. Three of these correspond to what is expected for α- and γ-methylene protons adjacent to β-oxymethines of unsubstituted 3-hydroxyacyl chains. One set of α/β and γ/β cross peaks is considerably farther downfield (near 5.2 ppm), indicating that this particular β-oxymethine group is acylated. A peculiar feature of the Lipid A in *Rhizobia* is that this acylation occurs with 27-hydroxyoctacosanoic acid, a C28 fatty acid that is not found in Lipid A of other Gram-negative bacteria like *E. coli*. The fifth set of α/β and γ/β cross peak pairs in the COSY spectra provides evidence for the further attachment of a β-hydroxybutyrate chain to the 27-OH group of the C28 acyl chain. This pattern of cross peaks is also seen for the other Lipid A molecules isolated from *Rhizobia* and is in fact diagnostic for the presence of an acyloxyacyl unit in each of these Lipid A species.

Species **D-1** contains an acylated aminogluconate unit in place of the proximal glucosamine residue of **B**. Species **C** and **E** lack the ester-linked β-hydroxymyristic acid chain at position 3.

The NMR results have aided the discovery of unusual enzymes in the pathways of *Rhizobia*. The presence of the unusual long chain fatty acid in the NMR spectra of the isolated species directly indicated the presence of a novel long chain acyltransferase in *Rhizobia* which has now been identified along with a novel acyl carrier protein. The NMR results have also stimulated the discovery of a membrane-bound deacylase in *Rhizobia* that removes a single O-linked hydroxymyristate from certain lipid A precursors. Furthermore, extracts of *Rhizobia* appear to possess enyzme(s) that catalyze the oxidation of the proximal glucosamine in **B** to the aminogluconate unit of **D-1**.

Regards,

Tony

Anthony A. Ribeiro ($A^2R$)

Fig. 1. COSY of aliphatic resonances of Species B

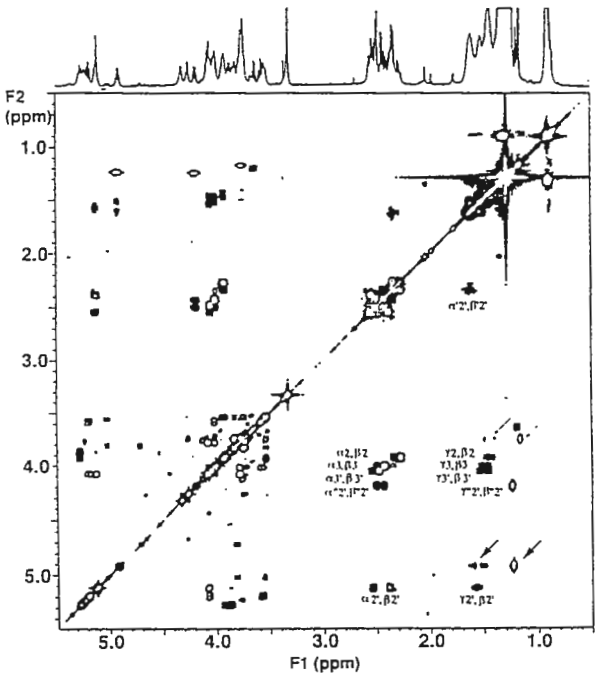
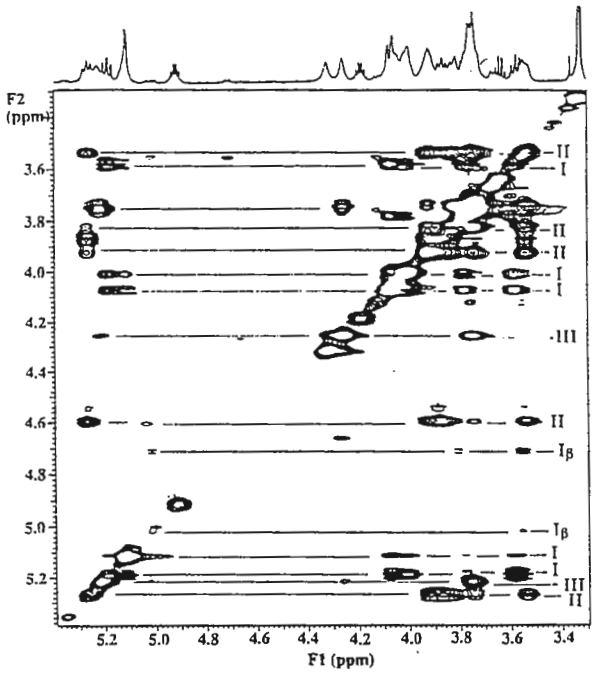
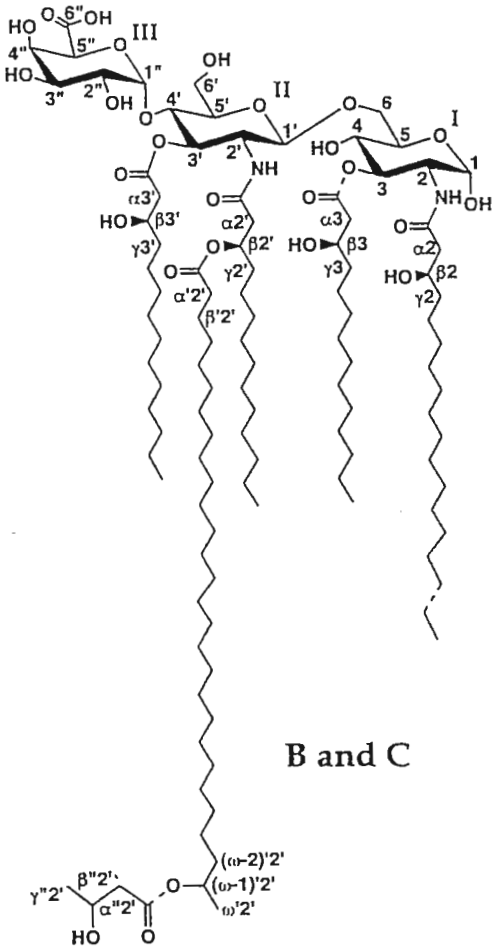
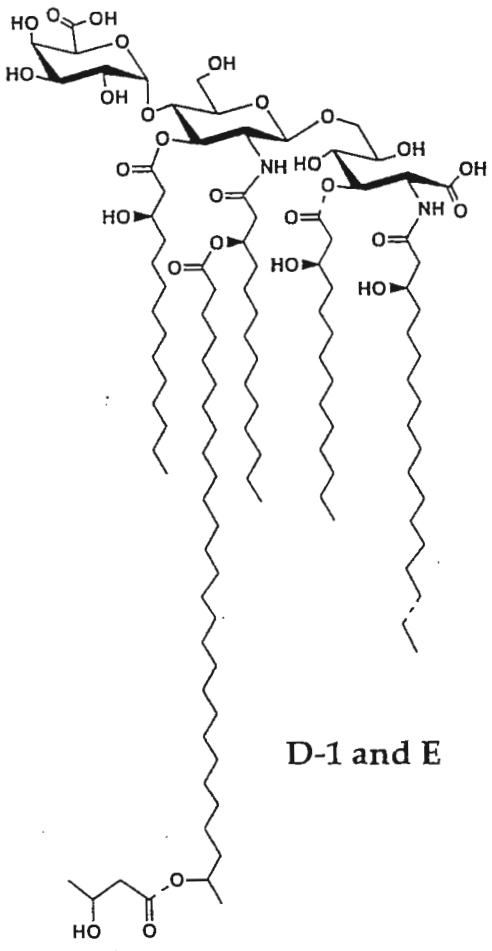



Fig. 2. TOCSY of sugar resonances of Species B



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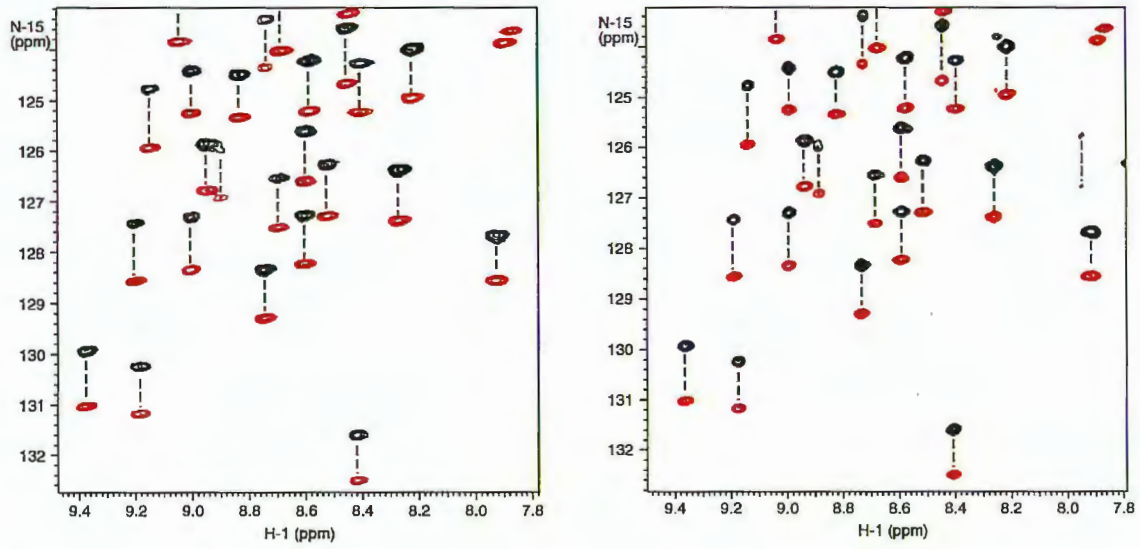
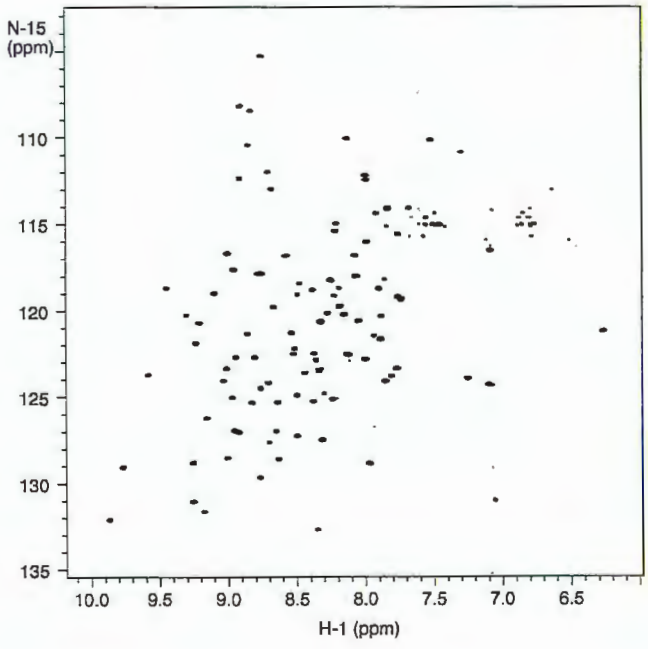



*Measurement of residual dipolar N-H couplings at 900 MHz in partially oriented proteins with (right) and without the use of band-selective homonuclear decoupling.*

## TROSY at 900 MHz




*The $^{15}N$-$^{1}H$ TROSY correlation spectrum of 6F1 1F2 module pair from the gelatin-binding domain of fibronectin. Sample courtesy of Prof. .I.D. Campbell of Oxford University.*

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UNIVERSITY OF VIRGINIA
DEPARTMENT OF CHEMISTRY
McCORMICK ROAD
CHARLOTTESVILLE, VIRGINIA 22901

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

September 11, 2000
(received 9/12/2000)

**Use of $^2$H NMR to Observe the Effect of Alamethicin on Biomembrane Thickness**

Dear Dr. Shapiro:

Alamethicin is a peptide that contains 20 amino acids and forms voltage-gated ion channels in biomembranes. The conductance state of alamethicin channels and the free energy of alamethicin binding to model biomembranes has been found to be dependent on bilayer composition (1,2). Bilayers containing different amounts of dioleoylphosphatidylcholine (DOPC) and dioleoylphosphatidylethanolamine (DOPE) have been studied. Single channel conductances increase as does alamethicin binding free energy when the relative amount of bilayer DOPE is increased. DOPE undergoes a bilayer to hexagonal II phase transition at about 10°C and imparts a negative spontaneous curvature to DOPC bilayers. A linear correlation between bilayer spontaneous curvature and alamethicin binding free energy was observed (2). It has also been observed that acyl chain order in bilayers containing DOPC and DOPE increases as the relative amount of DOPE increases (3). In order to further understand the DOPC/DOPE/alamethicin system we have examined the effect of alamethicin on bilayer thickness. The average deuterium order parameter of a saturated acyl chain attached to a bilayer phospholipid can be readily converted into a bilayer hydrophobic thickness (4,5). Figure 1 shows spectra of 10 mol% 1-palmitoyl(d31)-2-oleoyl-sn-glycero-3-phosphatidylcholine in DOPC and DOPC/DOPE bilayers with and without alamethicin. A vertical line is drawn through the spectra near the maxima of one of the quadrupolar doublet intensities arising from the acyl chain methylene (15 position) located next to the acyl chain methyl group. Note that the center of the quadrupolar doublets are at 0 on the frequency (kHz) scale and the doublets are symmetric about 0 kHz. Quick inspection of the spectra shows that the 15-methylene doublet splittings in Figs. 1A (DOPC), 1B (DOPC with alamethicin at 1:15 peptide:lipid mol/mol), and 1D (45mol% DOPE in DOPC with alamethicin at 1:15 peptide:lipid mol/mol) are similar and clearly smaller than the analogous splitting in Fig. 1C (45mol% DOPE in DOPC). We have used software created by Klaus Gawrisch (6) to assist us in converting the quadrupolar splittings into bilayer hydrophobic thicknesses; thanks Klaus.

The dependence of bilayer hydrophobic thickness on phospholipid composition and alamethicin content is shown in Figure 2. The hydrophobic thickness increases as the mol% DOPE increases. Addition of alamethicin to DOPC bilayers causes little or no change in bilayer thickness. Addition of alamethicin to bilayers containing DOPE and DOPC causes a decrease in bilayer thickness. A larger thickness decrease induced by alamethicin is observed for thicker bilayers. The fact that alamethicin has little or no effect on DOPC bilayer hydrophobic thickness is consistent with a match between the bilayer hydrophobic thickness and the effective hydrophobic length of alamethicin. Alamethicin appears to thin bilayers containing DOPE and DOPC to a thickness that is similar to that of DOPC bilayers (Fig. 2). Thus it appears that in DOPE/DOPC systems the bilayer hydrophobic thickness may adjust to match the hydrophobic length of alamethicin. Further studies on a variety of phospholipid bilayer systems will likely elucidate structure-function relationships in bilayers containing alamethicin.

Regards,

Jennifer R. Lewis  Jeff Ellena  David S. Cafiso

Figure 1

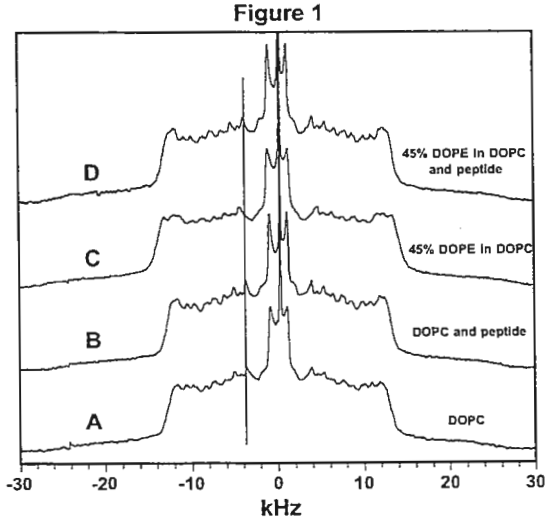
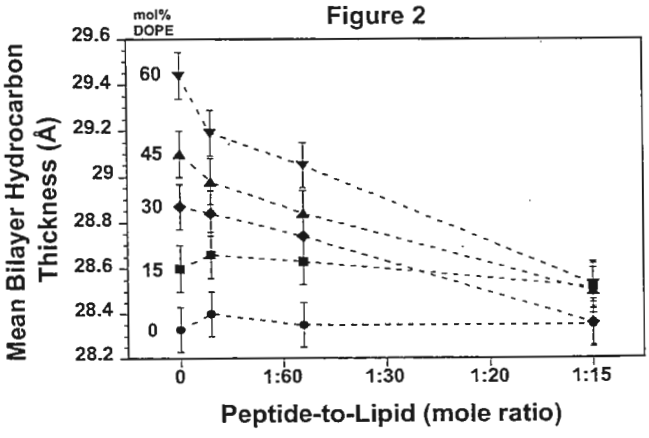



Figure 2

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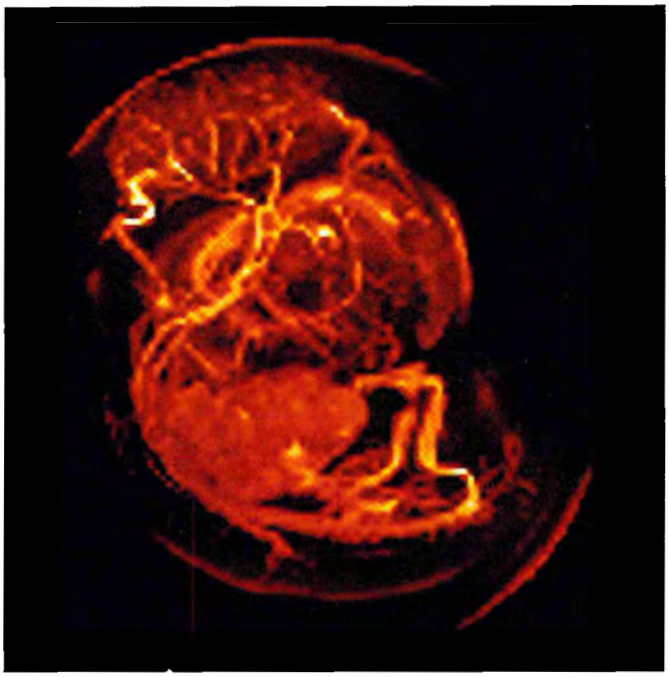

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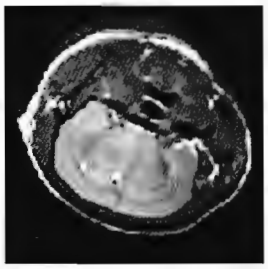
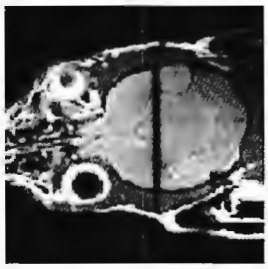
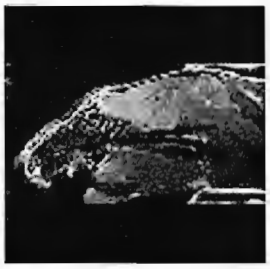
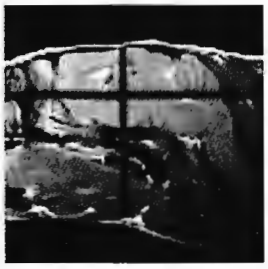


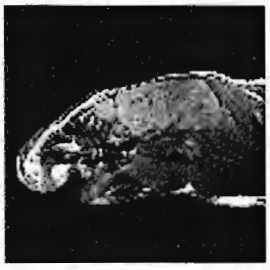




RARE mouse head image
- 256x128,
- 117 um pixels
- 500 um slice
- TR 3s,
- TE 5.5 ms
- Rare factor 8
- 2 averages



Spin echo images through a mouse head. The dark stripes indicate the position of the three intersecting orthogonal slices.



MSME, Sum of 8 echoes,
- 512 x 256,
- 59 um pixels,
- 500 um slice,
- TR 2s,
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- 4 averages

We gratefully acknowledge the valuable collaboration with "Spin System (Qld)" for support in developing the imaging probes and in-vivo accessories.

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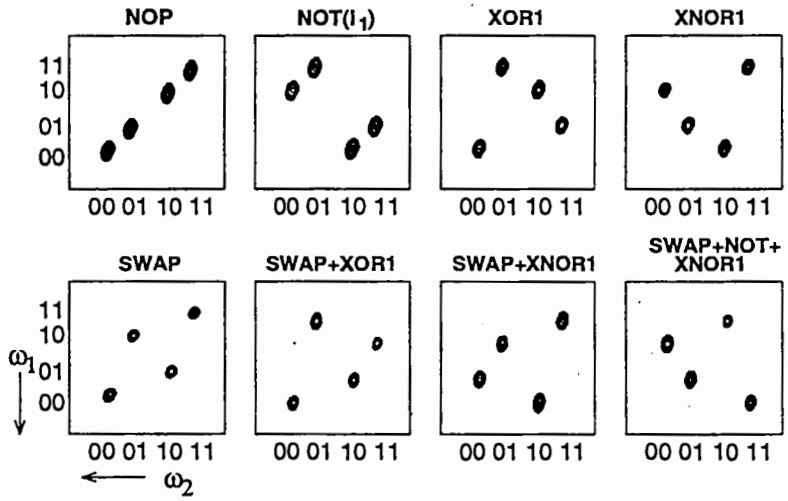
Prof. ANIL KUMAR
Convener, SIF

# Quantum Gates

24 AUG. 2000
(received 8/31/2000)

Dear Barry,

Following Alice into her Wonderland, we also found 'portmanteau' gates in NMR [1], some of which are reproduced below. These are experimental two-dimensional NMR spectra obtained using cascades of spin- and transition-selective pulses on two spins of the three spin system (protons) of 2,3-dibromo propionic acid.The third spin is the observe spin whose transitions are labelled as 00, 01, 10 and 11 depending on the states $\alpha$=0 or $\beta$=1 of the other two spins. The vertical dimension is the input and the horizontal dimension is the output of the gate [2]. Thus an XOR1 is a gate which follows the Boolean algebra $|\epsilon_1, \epsilon_2\rangle \stackrel{XOR1}{\longrightarrow} |\epsilon_1 \oplus \epsilon_2, \epsilon_2\rangle$ ($\equiv 00 \rightarrow 00; 01 \rightarrow 11; 10 \rightarrow 10; 11 \rightarrow 01$). The second qubit is unchanged and the first undergoes addition modulo 2 operation [$0 \oplus 0 = 0, 0 \oplus 1 = 1, 1 \oplus 0 = 1, 1 \oplus 1 = 0$]. The SWAP gate is $|\epsilon_1, \epsilon_2\rangle \stackrel{SWAP}{\longrightarrow} |\epsilon_2, \epsilon_1\rangle$. The SWAP gate is particularly interesting, since when we performed the SWAP gate using 1D NMR, we got the equilibrium spectrum [1]. In 2D, since we correlate the input and output (with the sacrifice of one qubit), the result is directly readable. The other gates can be interpreted similarly. The details of this work have been submitted to Journal of Magnetic Resonace [3] (the manuscript has a complete set (24) of one-to-one 2-qubit gates and the details of the selective pulses employed).

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T. S. Mahesh

Anil Kumar

P.S: Kindly credit this contribution to the account of Prof. C. L. Khetrapal.

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UNIVERSITY OF FLORIDA

Department of Chemistry

PO Box 117200
Gainesville, FL 32611-7200

September 18, 2000

(received 9/25/2000)

Dr. Bernard L. Shapiro
*The NMR Newsletter*
966 Elsinore Court
Palo Alto, CA 94303-3410

**Fluorinated Ethers**

Dear Barry:

Continuing our work on seeking out interesting effects and correlations in the NMR spectra of fluorinated materials, we have been examining spectra obtained on various spectrometers and have been running additional fluorine and carbon-13 spectra on our Inova-500 for a group of almost 100 ethers containing one or more fluorine atoms and up to seven carbons.

Many years ago, we reported data for three trifluorovinyl ethers with fluoromethyl, fluoroethyl and trifluoroethyl chains on the other side of the oxygen from the trifluorovinyl group. Within the trifluorovinyl group, the effect of the attached oxygen is to give relatively large cis F-F couplings of $62 \pm 3$ Hz and relatively large geminal couplings of 88 Hz or greater, although the trans value falls in the normal trifluorovinyl range of about 110 Hz.

Couplings across the oxygen are of special interest. In methyl trifluorovinyl ether, the methyl fluorines are coupled to those vinyl fluorines cis and geminal to the oxygen by about 4 Hz, but show no resolvable coupling to the trans fluorine. Incidentally, this is in contrast to the situation for trifluoromethyl trifluorovinyl *thioether*, where the methyl fluorines are coupled to all three trifluorovinyl fluorines. In perfluoro*propyl* trifluorovinyl ether, the fluorines in the $CF_2$ group attached to oxygen are coupled to the same two vinyl fluorines but the $CF_3$ fluorines are coupled by 0.7 Hz to only the F cis to the oxygen.

In dimethyl ether derivatives, the four-bond F-F coupling across the oxygen is 4-6 Hz, but when the methyl group is paired with an ethyl, propyl, or longer group, the value is typically 10-12 Hz. Methyl-ethyl thiothers show a similar value of about 10 Hz. Indeed, the only major difference in F-F coupling caused by introduction of a sulfur in place of an oxygen is that the five bond coupling from methyl fluorines to beta fluorines in the ethyl group is increased from less than 1 to 2-5 Hz. There is, however, one oxygen-containing molecule that we have found, $(CF_3O)_2CHCF_2Cl$, with $^5J$ of 2 Hz, probably a result of steric crowding.

In the methyl-ethyl thioethers as compared to the oxygen ethers, the shifts of fluorines in the ethyl group differ by only 3-4 ppm, while those in the $SCF_3$ group are 20-25 ppm to lower frequency than those of the $OCF_3$ group.

There are some interesting long-range carbon-fluorine couplings in a somewhat larger molecule:

$(CF_3)_2CF\text{-}O\text{-}CH_2CH_2CH_2Si(CH_3)_2Cl$

The carbon in the $CH_2$ group attached to the oxygen is coupled to the CF fluorine by 4.9 Hz, not too unusual, but seems also to be coupled to the six fluorines in the two methyl groups by 1.0 Hz. This is shown by the proton-decoupled spectrum. Perhaps more surprising, the CF carbon is coupled to the alpha $CH_2$ hydrogens by 1.8 Hz.

We are in process of analyzing carbon spectra of many other of these ethers, and we hope that these spectra will yield information about bonding and geometry in these molecules.

Wallace S. Brey

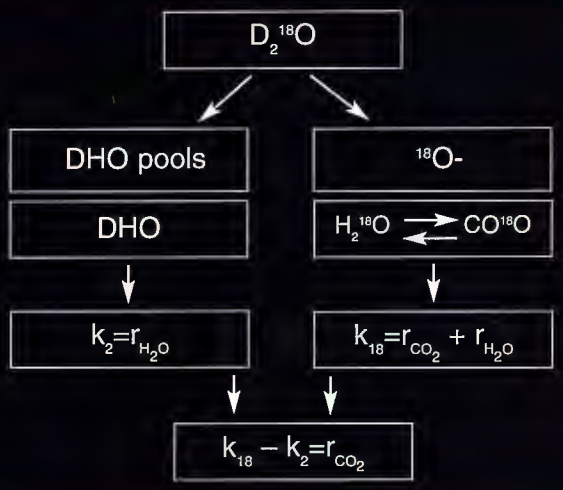
wsbrey@ufl.edu

# $^{18}$O-Labeled Water

**Current demand for $^{18}$O-labeled water is greater than the supply of this versatile and important isotopic reagent. Labeled water is primarily used as a source of $^{18}$O, which has many applications including medical imaging,[1] energy expenditure studies,[2] and in geochemical measurements.[3] $^{18}$O is used as a precursor for the production of the radionuclide, $^{18}$F. Labeled water, along with deuterium, is used to determine the energy expenditure of organisms via the doubly labeled water method. This method utilizes the difference in levels of labeled by-products from energy consumption to calculate the rate of $CO_2$ production. Finally, labeled water has been used as a tracer in oceanic studies and other geochemical applications. The variety of uses in many scientific areas makes this an invaluable reagent for chemical synthesis and analytical studies.**

## The doubly labeled water method[4]

k = experimentally determined rate constant
r = production rate

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| **45,336-6** | **Deuterium oxide,** 100.00 atom % D | **1pak=10 x 0.5mL - $51.40** |
| **45,333-1** | **Deuterium oxide,** 100.0 atom % D | **1pak=10 x 0.25mL - $17.10** |
| **26,978-6** | **Deuterium oxide,** 100.0 atom % D | **1pak=10 x 0.5mL - $19.80** |
| **44,136-8** | **Deuterium oxide,** 100.0 atom % D | **1pak=10 x 0.75mL - $29.00** |
| **42,345-9** | **Deuterium oxide,** 100.0 atom % D | **1pak=10 x 1.0mL - $36.60** |
| **30,875-7** | **Deuterium oxide,** 99.9 atom % D | **1pak=5 x 0.5mL - $7.00** |
| **52,196-5** | **Deuterium oxide,** 99.9 atom % D | **1pak=10 x 0.6mL - $12.50** |
| **44,137-6** | **Deuterium oxide,** 99.9 atom % D | **1pak=10 x 0.75mL - $13.40** |
| **26,979-4** | **Deuterium oxide,** 99.9 atom % D | **1pak=10 x 1.0mL - $17.80** |
| **30,876-5** | **Deuterium oxide,** 99.9 atom % D (contains 0.75 wt. % TSP-$d_4$) | **1pak=5 x 0.5mL - $9.10** |
| **53,052-2** | **Deuterium oxide,** 99.9 atom % D (contains 0.05 wt. % TSP-$d_4$) | **1pak=10 x 0.75mL - $14.40** |
| **29,838-7** | **Deuterium oxide,** 99.9 atom % D (contains 0.75 wt. % TSP-$d_4$) | **1pak=10 x 1.0mL - $19.40** |

***Listings with an isotopic purity of 100.00 atom % D and 100.0 atom % D have a minimum isotopic purity of 99.990% and 99.96%, respectively.***

## Deuterium oxide standards

| | | |
|---|---|---|
| **36,431-2** | **Deuterium oxide,** standard, 99.98 ± 0.01 atom % D | **10g $22.60** |
| **36,432-0** | **Deuterium oxide,** standard, 99.92 ± 0.01 atom % D | **10g $20.30** |

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Larger quantities of deuterium oxide are available through Sigma-Aldrich Fine Chemicals. Please call **1-800-336-9719 (USA)** or **1-314-534-4900**, or your local office for availability.

**References:** (1) North American Society for the Study of Obesity, J *21*, **1999.** (2) Schoeller D.; Santen van, E. *J. Appl. Physiol.* **1982**, *53*, 955. (3) Urey, H *Science* **1948**, *108*, 489. (4) Cole, T. J. et al. *The Doubly Labeled Water Method for Measuring Energy Expenditure: A Consesus Report by the IDECG Working Group*, NAHRES-*4*, **1990**, International Atomic Energy Agency


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Naval Research Laboratory
4555 Overlook Avenue, S.W.
Washington, DC 20375-5342

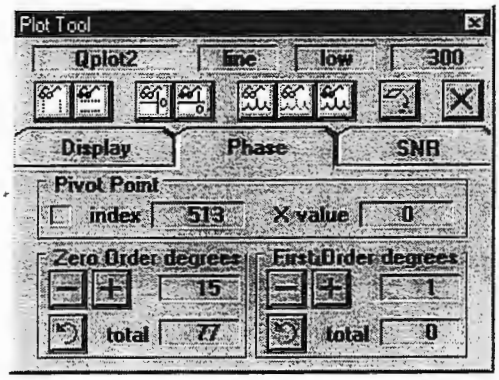
31 August, 2000
(received 8/31/2000)

Dr. B. L. Shapiro
The NMR Newsletter
966 Elsinore Court
Palo Alto, CA 94303-3410

## NMR Tools for Origin

Those of you who use the Origin data analysis and graphing software by OriginLab Corporation (formerly Microcal Software, Inc.) may already know about this free add-on for importing and analyzing NMR data. I began developing these tools over five years ago to work up and plot my own data in Origin 3.0 and by April of last year they seemed mature enough (more benefits than bugs) to share with others. NMR Newsletter readers who are not aware of this Origin add-on might be interested in learning about it.

The data importing routines supplied with the tools support MacNMR, CMXW, Spinsight and XWINNMR data files and can be customized to handle other file formats. The basic NMR routines are provided of course, such as baseline correction, exponential multiplication, Fourier and Hadamard transformation and phase correction. Much of my research involves multiple pulse sequences and I find it convenient to handle the resulting data as 2D data in which the signal following each pulse is treated as an individual record. Therefore, 2D operations such as record coaddition and 2D FT are also provided and can be used with more conventional 2D data as well. The NMR commands are conveniently grouped under a separate Origin menu or on floating toolbars such as the Plot Tool shown below.




Details are in the HTML manual at <http://users.erols.com/mlbuess> and the tools themselves can be downloaded from the same page. They can be used with Origin 5.0 and 6.0 (Win9x/NT) and come in a self-extracting ZIP file that is smaller than 200KB. The tools and manual are also available from OriginLab's Custom Tools Directory: <http://www.OriginLab.com/applications/index.asp>

Please credit this contribution to Al Garroway's account.

Sincerely,

M. L. Buess (SFA, Inc.)
buess@ccs.nrl.navy.mil
(202) 767-3549

505–22

*NEW MEXICO RESONANCE*
A nonprofit research corporation

September 24, 2000

The 37th New Mexico Regional NMR Meeting

Dear Barry,
The 37th New Mexico Regional NMR Meeting will be held in Albuquerque, New Mexico, on November 11, 2000. The speakers and their subjects are 1) Brian Saam, University of Utah, on Hyperpolarized Gas NMR: Application to lungs; 2) Catherine Clewett, New Mexico Resonance, on Characterization of Partially Sintered Ceramic Powder Compacts Using Thermally-polarized Fluorinated Gas NMR Imaging; 3) Doug Harris, Sandia National Laboratories, on Solid-State NMR of Polyethylene; and 4) Atholl Gibson on Cryogenic High Resolution NMR Probes. This small and informal one-day meeting will end with the traditional dinner/drinks/reception in the evening. There is no registration fee but please let us know if you are attending so we can find enough chairs. For more information on this meeting, past and present, please consult http://www.unm.edu/~karenann/nmr2.html or write/call/email New Mexico Resonance at our new contact coordinates given in the letterhead or http://www.nmr.org.

Best wishes,

Eiichi Fukushima

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---

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# Stable Isotope Derivatives

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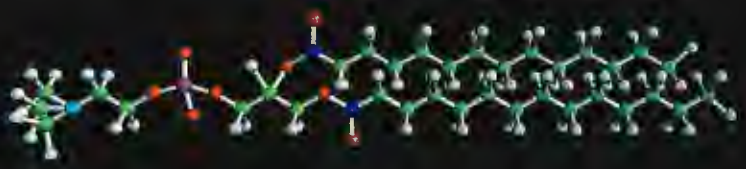
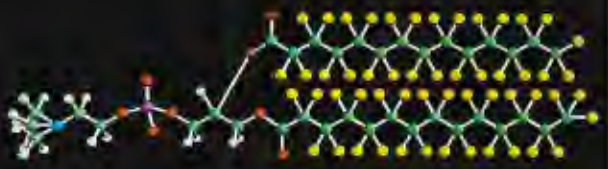


Carbon 13 14:0 PC



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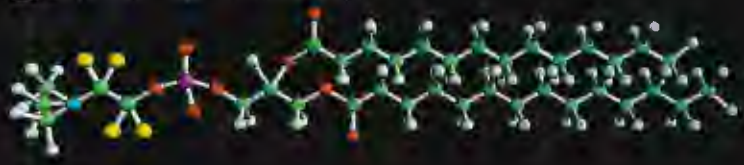

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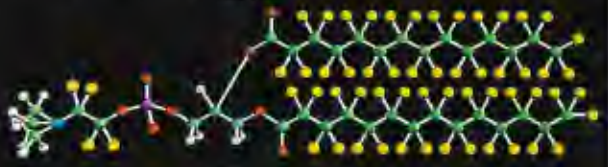

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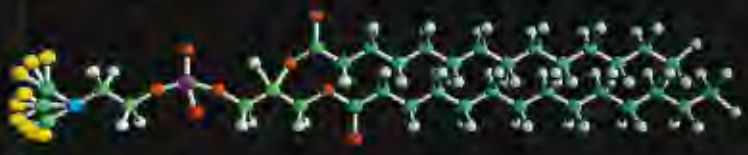

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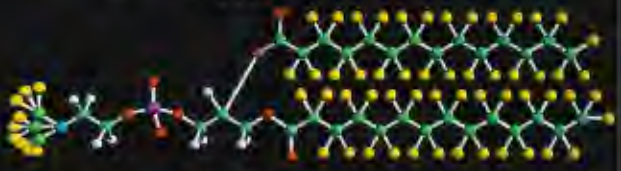

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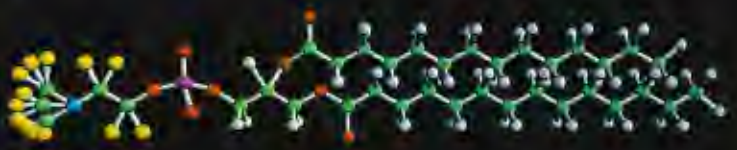

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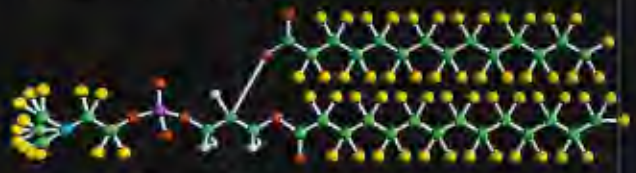
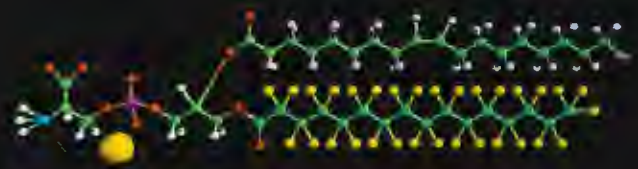


14:0 PC (D67)

## Asymmetric Fatty Acid



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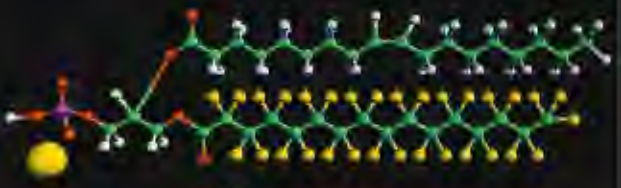

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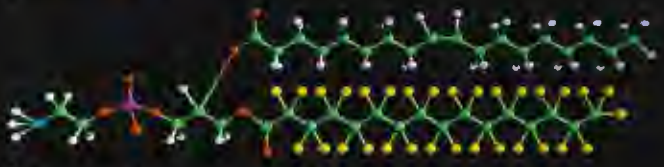

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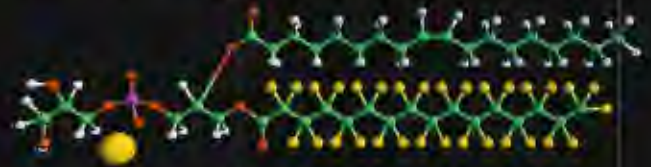

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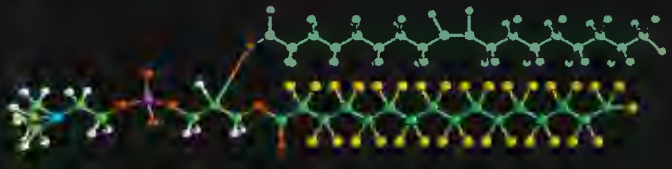

16:0-18:1 PC (D31)

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**The NMR Newsletter - Book Reviews**

Book Review Editor: István Pelczer, Dept. of Chemistry, Princeton University, Princeton, NJ 08544

# "Modeling NMR Chemical Shifts:

## Gaining Insights into Structure and Environment"

edited by

**Julio C. Facelli and Angel C. de Dios**

American Chemical Society, Washington DC, 1999, pp x + 373, ISBN 0-8412-3622-4, $97.50, http://www.oup-usa.org

This book is one of the ACS symposium series (#732), and arises from the second international symposium on NMR chemical shifts, organised by the editors at the ACS meeting in Boston, Mass. in August 1998. It exhibits the benefits and some of the problems that one might expect from multi-authored works. The editors have done an excellent job of getting a consistent presentation throughout the book, but inevitably it is to some extent a collection of research articles on a common theme. On the other hand, it consists of articles written by leaders in the field, and therefore presents a good cross-section of the field (as of August 1998).

A particularly good feature of the book is that it starts with four review articles: an overview of the state of the art in the calculation of NMR chemical shielding, across a wide range of nuclei (Jameson); the calculation of $^{13}C$ chemical shifts in vinyl polymers and polypeptides (Ando); correlations of $^{13}C$ and $^{15}N$ (and $^{57}Fe$) shift calculations with structural features in peptides, proteins and porphyrins (Oldfield); and applications of $^{13}C$ chemical shift calculations to the understanding of catalysis (Nicholas). Interestingly, both Ando and Oldfield present the conformation dependence of $^{13}C$ shifts in alanine-containing peptides, with rather different results. A frustration of the format of this kind of book is that it's not easy to know whether this represents a real contradiction or merely a difference in emphasis -- both results look convincing, but on the face of it at least, they can't both be right. The article by Jameson was for me particularly helpful, as it provides an introduction to many of the methods that are described in later articles in the book, and enabled me to understand at least some of the technical complexities of quantum mechanical chemical shift calculations. As a broad overview of the topic, it is hard to beat.

The remaining articles concentrate on first row elements ($^{13}C$, $^{15}N$, $^{17}O$, $^{19}F$), but include also reports on shifts for transition metals and for $^{29}Si$, $^{27}Al$, $^{31}P$, $^{23}Na$ and $^{129}Xe$. In addition there are two articles that concentrate on $^{1}H$ shifts, but more from an experimental or semi-empirical angle, reflecting the continuing difficulty of usefully calculating $^{1}H$ shifts using *ab initio* methods. There is a wide range of topics, ranging from detailed calculations of quantum mechanical electron current density to empirical correlations between hydrogen bond length and $^{1}H$ shift. Many of the calculations discuss shift tensors as well as the isotropic shifts.

In detail, the book covers shielding polarisabilities, bond polarisation theory, relativistic density functional calculations, electron correlation in $^{19}F$ shifts, correlation between transition metal shifts and reactivity, alkoxysilanes, oxides and polyoxometallates, main-group metal oxides and nitrides, phosphates, and zeolites, as well as shift/structure relationships in peptides, rhodopsin, nucleic acids and alkenes.

*continued*

This book is for those who know something about quantum mechanical calculations, and about chemical shifts, and want to know what are the current areas of interest. It's a mine of information, and impresses with the broad range of topics that can be studied, and the detailed information that can now be calculated. However, if you come to the book expecting to learn how chemical shifts can be applied to the refinement of protein structure, or the elucidation of polymer structure, you will be disappointed. Thus, this is a book more for physicists and physical chemists than for biochemists – a book packed with theory and techniques, but lots of applications as well.

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University of Sheffield
Sheffield, UK.

M.Williamson@sheffield.ac.uk

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Arthur J. Ragauskas
Professor of Wood Chemistry
Institute of Paper Science and Technology
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Volume 20, Issue 3

# Buffers for NMR Studies

In 1966, Dr. Norman Good introduced a series of nine buffers designed to have a minimum of side reactions in biological systems. These original "Good" buffers are widely used in biological reactions and tissue culture. CIL recognizes the requirements of the NMR community to have highly enriched solvents and buffers. We have made some of these original "Good" buffers as well as other potentially useful reagents.

No single buffer is best for every application. Consequently, it is useful to have a variety of buffers available in the $pK_a$ range that best suits your application. The following buffers meet our high standards of chemical purity and isotopic enrichment:

| Catalog # | | Compound (Isotope, Atom % Enrichment) | $pK_a$*(20°) | Range | Amount | $ Price |
|---|---|---|---|---|---|---|
| DLM-12 | | Acetic Acid-$d_4$ (D,99.5%) | 4.76 | 4.3-5.2 | 10 g<br>25 g<br>50 g | 27<br>63<br>112 |
| DLM-41 | | Acetic Acid-$d_4$ "100%" (D,99.96%) | 4.76 | 4.3-5.2 | 10x0.75 ml<br>5 ml | 69<br>39 |
| DLM-1099 | | Ammonium Bromide ($D_4$,98%) | 9.25 | 8.8-9.7 | 5 g | 85 |
| DLM-1273 | | Ammonium Chloride ($D_4$,98%) | 9.25 | 8.8-9.7 | 5 g<br>10 g | 70<br>90 |
| DLM-710 | | Ammonium Deuteroxide ($D_5$,99%) (26% solution in $D_2O$) | 9.25 | 8.8-9.7 | 50 g<br>100 g<br>(of solution) | 105<br>185 |
| DLM-407 | | Betaine ($D_{11}$,98%) (Trimethylglycine) | 1.83 | 1.4-2.3 | 1 g | 500 |
| DLM-4528 | | Bis-Tris ($D_{19}$,98%) | 6.54 | 5.8-7.2 | 1 g | 650 |
| DLM-4862 | *New* | Cacodylic Acid ($D_7$,98%) | 6.1 | 5.1-7.1 | 0.5 g | 500 |
| DLM-286 | | Formic Acid ($D_2$,98%) (contains ≤ 5% $D_2O$) | 3.77 | 3.3-4.2 | 5 g | 130 |
| DLM-280 | | Glycine ($D_5$,98%) | 2.34<br>9.60 | 1.9-2.8<br>9.1-10.1 | 5 g | 135 |
| DLM-3786 | | N-2-Hydroxyethylpiperazine -N'-2-ethanesulfonic Acid ($D_{18}$,98%) (HEPES) | 7.55 | 6.8-8.2 | 0.25g | 450 |
| DLM-3033 | | Imidazole ($D_4$,98%) | 7.05 | 6.6-7.5 | 1 g<br>5 g | 160<br>575 |
| DLM-4363 | | 2-(N-Morpholino)ethanesulfonic Acid ($D_{13}$, 98%) (MES) | 6.15 | 5.5-6.7 | 0.5 g | 750 |
| DLM-4781 | *New* | Piperazine-N,N'-bis(2-ethanesulfonic Acid) ($D_{18}$,98%) (PIPES) | 6.8 | 6.1-7.5 | 0.25 g | 375 |
| DLM-1361 | | Sodium Formate (D,99%) | 3.77 | 3.3-4.2 | 5 g | 155 |
| DLM-831 | | Succinic Acid ($D_6$,98%) | 4.19<br>5.48 | 3.8-4.6<br>5.0-5.9 | 5 g | 235 |
| DLM-1842 | | Tricine ($D_8$,98%) | 8.15 | 7.6-8.8 | 0.1 g | 290 |
| DLM-1814 | | Tris(hydroxymethyl)methylamine ($D_{11}$,98%) (TRIS) | 8.30 | 7.8-8.8 | 5 g | 490 |
| DLM-3593 | | Tris(hydroxymethyl)methylamine ($D_{11}$,98%) (1M solution in $D_2O$) | 8.30 | 7.8-8.8 | 10 ml<br>(of solution) | 200 |

* $pK_a$ values are for unlabeled compounds. Deuterated compounds may have slightly different $pK_a$ values. Ranges are approximately +/- 0.4-0.5 from unlabeled $pK_a$ value.

PH: 800.322.1174 (USA) PH: 978.749.8000 FAX: 978.749.2768 E-MAIL: cilsales@isotope.com URL: http://www.isotope.com

# Additional Reagents for Protein Chemistry

| Catalog # | | Compound (Isotope, Atom % Enrichment) | Amount | $ Price |
|---|---|---|---|---|
| **Detergents:** | | | | |
| DLM-2274 | | Dodecylphosphocholine ($D_{38}$,98%) (DPC) | 0.5 g | 700 |
| DLM-4342 | | n-Octyl β-D-Glucopyranoside ($D_{28}$,98%) (n-Octyl Glucoside) | | Request Price |
| DLM-3787 | | n-Octyl β-D-Glucopyranoside ($D_{28}$,98%) (n-Octyl Glucoside) (mixture of 85% β / 15% α) | 0.1 g<br>0.25 g | 425<br>750 |
| DLM-4341 | | DL-α-Phosphatidylcholine, Dihexanoyl ($D_{40}$,98%) (DHPC) | 0.1g | 1300 |
| DLM-197 | | Sodium Dodecyl Sulfate ($D_{25}$,98%) (SDS) | 1 g | 370 |
| **Protein Stabilizers:** | | | | |
| DLM-2622 | | DL-1,4-Dithiothreitol ($D_{10}$,98%) (DTT) | 0.5 g | 635 |
| DLM-2713 | | 2-Mercaptoethanol ($D_6$,98%) | 0.5 g | 630 |
| **Chelating Agents:** | | | | |
| DLM-414 | | Ethylenediaminetetraacetic Acid ($D_{12}$,98%) (EDTA) | 0.1 g<br>0.5 g<br>1 g | 115<br>450<br>690 |
| DLM-3908 | | Ethylenediaminetetraacetic Acid ($D_{16}$,98%) (EDTA) | 0.1 g<br>0.5 g<br>1 g | 115<br>450<br>690 |
| **Protein Folding Catalysts:** | | | | |
| DLM-4779 | *New* | Trimethylamine N-oxide ($D_9$,98%) | 1 g | 325 |

*If there is a buffer or protein chemistry reagent not listed above which is of interest to you, please contact us.*


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September 25, 2000
(received 9/25/2000)

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

Dear Dr. Shapiro:

Symposium on "High Resolution NMR Spectroscopy of Polymers"
ACS National Meeting, San Diego, April 1-5, 2001

At the ACS National Meeting in April 2001 at San Diego, we are organizing a symposium featuring the latest developments in the NMR studies of polymers and biopolymers, particularly in solution or in the melt. Equal emphasis will be placed on fundamental research and on industrially important problems. The symposium will consist of a tutorial session, 5 technical sessions, and a poster session. The symposium is sponsored by the ACS Division of Polymer Chemistry.

The following is a partial list of speakers:

T. Asakura (Tokyo Univ. of Agriculture)
I. C. Baianu (Univ. of Illinois, Urbana-Champaign)
A. S. Brar (Indian Inst. of Technology, New Delhi)
M. D. Brickhouse (Hercules)
C. A. Bush (Univ. of Maryland)
R. A. Byrd (NCI, Frederick)
H. N. Cheng (Hercules)
R. Chujo (Teikyo Univ. of Sci. & Technol., Japan)
A. D. English (DuPont)
A. Handley (LGC, Runcorn, UK)
H. J. Harwood (Univ. of Akron)
W. Hiller (Varian)
T. Huckerby (Lancaster Univ.)
W. Hutton (Monsanto)
P. T. Inglefield (Clark Univ.)
P. A. Mirau (Lucent – Bell Labs)
M. Montaudo (CNR, Catania, Italy)
T. G. Neiss (DuPont Pharmaceutical Co.)
A. Pardi (Univ. of Colorado)
J. H. Prestegard (Univ. of Georgia)
A. Martinez-Richa (Guanajuato, Mexico)
P. L. Rinaldi (Univ. of Akron)
N. Sachinvala (USDA, New Orleans)
A. L. Segre (CNR, Rome)
A. Serianni (Notre Dame)
K. Thakur (3M)
A. E. Tonelli (North Carolina State U.)
K. Ute (Osaka Univ.)
H. van Halbeek (UC San Diego)
J. F. G. Vliegenthart (Utrecht)
C. G. Wade (IBM)
N. L. Yang (CUNY, Staten Island)

If you are interested in presenting a poster, please respond to one of the symposium organizers prior to October 20, 2000. The deadline for abstracts and preprints is November 1, 2000.

Organizers

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From: Ad Bax
Ray Freeman
Gareth Morris

Dear Barry,

"INADEQUATE"

An experiment that offers an alternative to synthetic chemistry can't be all bad. We have been working on a technique for studying carbon-carbon spin couplings in natural abundance material, obviating the need for specific isotopic enrichment procedures. The idea is due to Ad Bax, who is working here in Oxford on leave from the University of Delft. It involves the momentary creation of double-quantum coherence, and in that sense is related to two-dimensional NMR.

If we wish to study the weak carbon-13 satellites which appear in conventional carbon-13 spectra, the basic problem is not so much one of sensitivity, but rather the difficulty of identifying these weak lines amid a jumble of spinning sidebands, impurity lines and spurious modulation effects in the flanks of the strong central resonance, often a result of incomplete proton decoupling. For natural abundance samples, we need consider only two coupled carbon-13 spins in the molecule, so the spectra are AX or AB, and the spin system possesses the key property of a double-quantum energy gap. We may therefore generate a double-quantum coherence, whereas the molecules with only a single carbon-13 spin can never exhibit this effect. The pulse sequence used to excite this double-quantum coherence is

$$90^{\circ}(X) - \tau - 180^{\circ}(\pm Y) - \tau - 90^{\circ}(X)$$

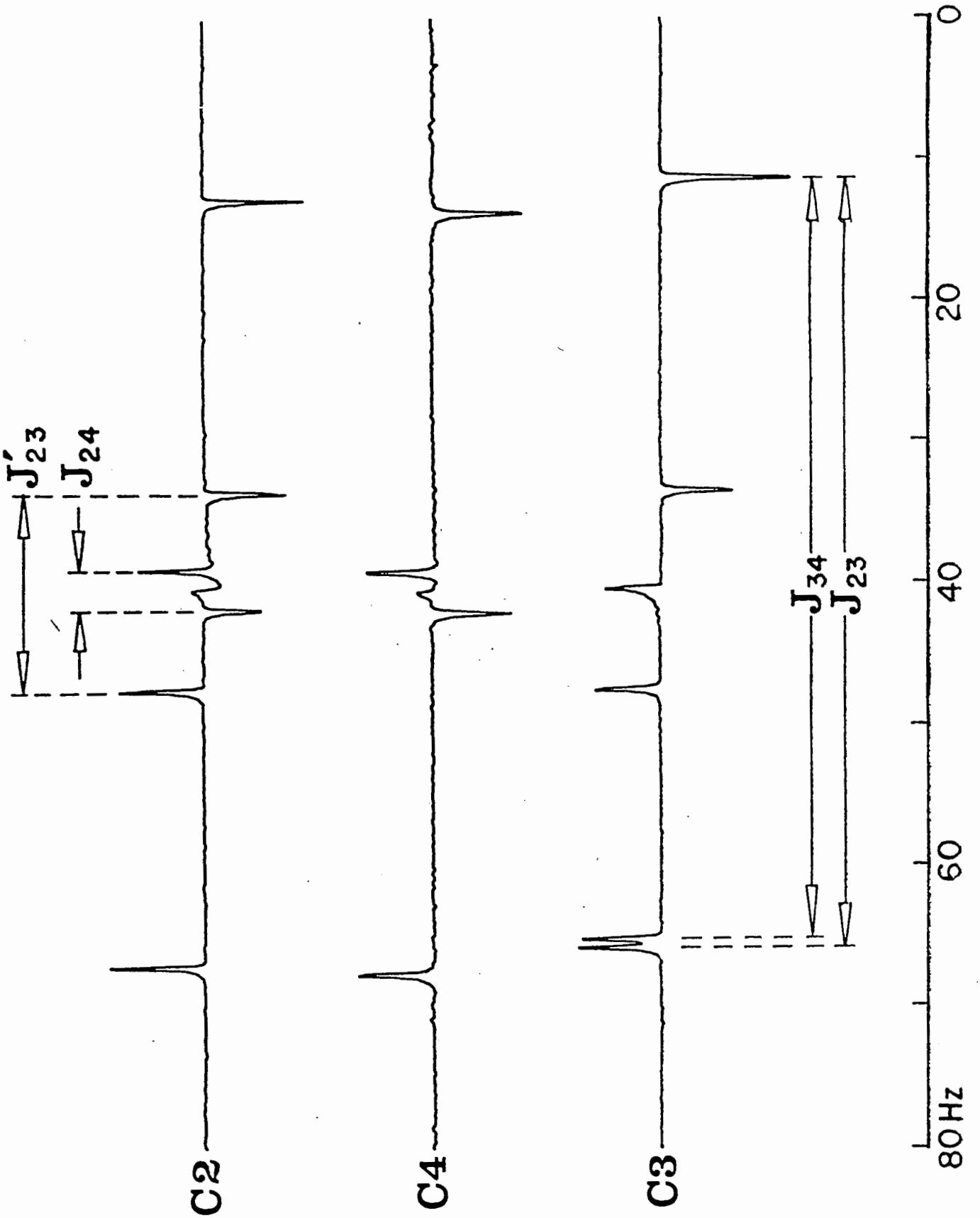
and the transfer is optimized for the cyclic condition $(2n+1)\tau = 1/(4J)$. This is similar to the condition for "INEPT" magnetization transfer. The protons are noise decoupled throughout the experiment.

Now double-quantum coherence is not directly observable, but once the information has been stored in this form, it can be retrieved by reconversion into transverse nuclear magnetization by a fourth pulse, $90^{\circ}(\Phi)$. We do this as soon as possible (after 10 microseconds) so there is no evolution of the double-quantum coherence. Discrimination against the strong central signal ($M_0$) hinges on the fact that double-quantum coherence is uniquely sensitive to the phase $\Phi$ of this last pulse. If $\Phi$ is changed by $90^{\circ}$, the signal derived from the double-quantum coherence ($M_2$) shifts phase by $270^{\circ}$. Thus if $\Phi$ is cycled counterclockwise in $90^{\circ}$ steps, $M_2$ apears to cycle clockwise in $90^{\circ}$ steps. The receiver phase is made to follow this clockwise rotation, and all other signals cancel, having a different dependence on $\Phi$. Phase alternation of the $180^{\circ}$ pulse helps suppress signals arising from pulse imperfections. The usual "CYCLOPS" is also used, giving a 32 step sequence altogether.

On our XL-200 it proved possible to achieve suppression ratios exceeding 1000 : 1, allowing a clear view of the inner satellites due to long-range C-C coupling. Longer $\tau$ values are used for small couplings, and the direct couplings can be included in the same spectrum by judicious choice of $\tau$ and n. The C-C couplings in pyridine are shown in the diagram. This was an early attempt; better suppression was obtained by careful attention to pulse length calibration and the establishment of a steady-state regime before starting the main sequence.

In fact the real reason for this note is to suggest a name for this technique before it becomes submerged in a mire of half-remembered pulse sequences. We wanted to get Ad's name into it, and at the same time continue the tradition of "INEPT" so we favour, INADEQUATE, the Incredible Natural Abundance DoublE QUAntum Transfer Experiment.

Kindest regards, Ad Ray Gareth




Bax, Freeman and Morris: Carbon-carbon splittings in pyridine observed by INADEQUATE pulse sequence. The two components of each doublet are always antiphase, up-down if n is odd, down-up if n is even.

**Forthcoming NMR Meetings**, continued from page 1:

PITTCON 2001, New Orleans, LA, **March 4-9, 2001**. Contact: The Pittsburgh Conference, Dept. CFP, 300 Penn Center Blvd., Suite 332, Pittsburgh, PA 15235-5503. Tel: 412-825-3220; Fax: 412-825-3224; E-mail: pittconinfo@pittcon.org.

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

ACS National Meeting, "Symposium on High Resolution NMR Spectroscopy of Polymers, " San Diego, CA, **April 1-5, 2001**; Contact: H. N. Cheng (hcheng@herc.com) or A. D. English (alan.d.English@usa.dupont.com); See Newsletter 505, xx.

Magnetic Resonance in Chemistry and Biology, XIth International Conference, Zvenigorod, Russia, **April 20-27,2001**. Contact: http://www.nmr.de/html/conf/zelino.shtml.

ISMRM 9th Scientific Meeting and Exhibition; ESMRMB 18th Annual Meeting and Exhibition, Joint Annual Meeting. Glasgow, Scotland, **April 21-27, 2001**.Contact: ISMRM Central Office, 2118 Melvia Street, Suite 201, Berkeley, CA 94704. Tel: 510-841-1899; Fax: 510-841-2340; E-mail: info@ismrm.org.

Computational Aspects of Biomolecular NMR, Gordon Conference, "Il Ciocco", Barga (Pisa) Italy, **May 6-11, 2001**. Contact: Michael Nilges nilges@embl-heidelberg.de, or Dave Cast case@scripps.edu.

Gordon Research Conference on Magnetic Resonance, **June 17-22, 2001**, Roger Williams University, Bristol, Rhode Island (note the new, improved location !!!). Contacts: Rob Tycko, Chair, 301-402-8272, tycko@helix.nih.gov, and Kurt Zilm, Vice-Chair, kurt.zilm@yale.edu. Site description and application information available at http://www.grc.uri.edu.

IXth International Symposium on Magnetic Resonance in Colloid and Interface Science, St. Petersburg, Russia, **June 26-30, 2001**. Contact: Mrs. L. Ya. Startseva, Secretariat of ISMRCIS, Boreskov Institute of Catalysis, 5, Prosp. Akad. Lavrentieva, Novosibirsk, 630090, Russia. Tel: +7 (3832) 34-12-97; Fax: +7 (3832) 34-30-56; E-mail: star@catalysis.nsk.su.

Royal Society of Chemistry: 15th International Meeting on NMR Spectroscopy, Durham, England, **July 8-12, 2001**; Contact: Mrs. Paula Whelan, The Royal Society of Chemistry, Burlington House, London W1J 0BA, England; tel: +44 (0) 207-437-8656; fax: +44 (0) 207-734-1227; Email: conferences@rsc.org\; Use the subject header '01NMR15'

ESR and Solid State NMR in High Magnetic Fields, Stuttgart, Germany, **July 22-26, 2001**. Contact: Prof. Hans Paus, 2 Physikalisches Institut, Universität Stuttgart, Pfaffenwaldring 57, D-70550 Stuttgart, Germany. Tel: ++49-711-685-5223 or -5217; Fax: ++40-711-685-5285; E-mail: ampere2001@physik.uni-stuttgart.de.

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

14th European Symposium on Polymer Spectroscopy, Dresden, Germany, **September 2-5, 2001**. Contact: Institut für Polymerforschung Dresden e. V., ESOPS 14, Postfach 12 04 11, 01005 Dresden, Germany. Tel: +49 351 4658-282; Fax: +49 351-4658-214; E-mail: espos@ipfdd.de.

Fourth International Conference on Molecular Structural Biology, Vienna, Austria, **September 5-9, 2001**. Contact: Andreas Kungl, Austrian Chemical Society (GÖCH), Biochemistry Subgroup, c/o Institute of Pharmaceutical Chemistry, Univeristy of Graz, Universitätsplatz 1, A-8010 Graz, Austria. Tel: +43 316 380 5373; Fax: +43 316 382541; E-mail: andreas.kungl@kfunigraz.ac.at.

2nd Alpine Conference on Solid-State NMR, Chamonix-Mont Blanc, France, **September 9-13, 2001**; Contact: Alpine Conference Secretariat, Laboratoire STIM, Ecole Normale Supérieure de Lyon, 46 allée d'Italie, 69364 Lyon Cedex 7, France; alpine.SSNMR@ens-lyon.fr; Tel. +33-(0)4 72-72-84-86/ 83 84; Fax. +33 (0)4 72 72 84 83; http://ens-lyon.fr/STIM/alpineweb,html

XXth International Conference on Magnetic Resonance in Biological Systems, Toronto, Ont., **August 25-30, 2002**. For further information check www.uwo.ca/chem/icmrbs/, or contact: mgordon@julian.uwo.ca

Additional listings of meetings, etc., are invited.

THE NMR NEWSLETTER

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## Policies and Practical Considerations

*(Revised 22 September 2000)*

***The NMR Newsletter*** (formerly the **TAMU NMR Newsletter**, the **IIT NMR Newsletter**, and originally, the **Mellon Institute NMR Newsletter**), now in its forty-third year of consecutive monthly publication, continues under the same general policies as in the past.

1. **Policy**:

***The NMR Newsletter*** is a means for the rapid exchange of information among active workers in the field of NMR spectroscopy, as defined broadly, including imaging. As such, the Newsletter serves its purpose best if the participants impart whatever they feel will interest their colleagues, and inquire about whatever matters concern them. Technical contributions should always contain a significant amount of information that has not already been published or that will appear in the formal literature within a few weeks of the appearance in the Newsletter.

Since the subscriber/participant clearly is the best judge of what he or she considers interesting, our first statement of policy is "We print anything." (This is followed by the reservation, "that won't land us in jail or bankruptcy court.") Virtually no editorial functions are performed, although on rare occasions there is the need to classify a contribution as 'not for credit'. The Newsletter is not, and will not become, a journal. We merely reproduce and disseminate exactly what is submitted.

2. **Public Quotation and Referencing:**

Reference to The NMR Newsletter by its present or previous names in the scientific literature is *never* permissible. Public quotation of Newsletter contents in print or in a formal talk at a meeting, etc., is expressly forbidden, except as follows. *In order to quote or use material from the Newsletter, it is necessary, in each individual case, to obtain the prior permission of the responsible author and then to refer to the material quoted as a "Private Communication".* If your copy of the Newsletter is shared with other readers, it is your obligation as the actual recipient of the Newsletter to see that these other readers of your copy are acquainted with, and abide by, these statements of policy.

3. **Participation is the prime requisite for receiving the Newsletter: In order to receive the Newsletter, you must make at least occasional technical contributions to its contents.**

We feel that we have to be quite rigorous in this regard, and the following schedule is in effect: Seven months after your last technical contribution, you will receive a "Reminder" notice. If no technical contribution is then forthcoming, nine months after your previous contribution you will receive an "Ultimatum" notice, and then the next issue will be your last, absent a technical contribution. Subscription fees are not refunded in such cases. If you are dropped from the mailing list, you can be reinstated by submitting a contribution, and you will receive back issues (as available) and forthcoming issues at the rate of nine per contribution.

Frequent contributions are encouraged, but no advance credit can be obtained for them. In cases of joint authorship, only one contributor may be credited. Meeting announcements, as well as "Position Available," "Equipment Wanted" (or "For Sale"), etc., notices are very welcome, but only on a not-for-credit basis, *i.e.*, such items do not substitute for a *bona fide* technical contribution.

4. **Finances**: The Newsletter is wholly self-supporting, and its funding depends on Advertising, Sponsorships, and individual Subscriptions. The **Subscription fee** for the October 2000 - September 2001 year is US$190, with a 50% academic or personal subscription discount. Subscriptions are available for a minimum of the twelve monthly issues which end with a September issue. However, a subscription can be initiated at any time, with the price for more than twelve issues being prorated.

*continued*

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Another major, indeed most essential, source of funds for the Newsletter is **Advertising**. We earnestly encourage present and potential participants of the Newsletter to seek advertising from their companies. Our rates are very modest. Please inquire for details.

5. **Practical Considerations**:

***NEW***: If possible, technical contributions should be submitted as attachments (in WORD7 format) to e-mail messages. Please see 'New' Way to Submit Technical Contributions – copy attached.

a) All technical contributions to the Newsletter will be included in the next issue if received on or before the published deadline dates.

b) Please provide short titles of all topics of your contributions, to ensure accuracy in the Table of Contents.

c) Contributions should be on 8.5 x 11" (21 x 27.5 cm) pages, *printed on one side only*. Contributions should be submitted in camera-ready condition. Contributions may not exceed three pages without prior approval. Each page must have margins of at least 0.5" (1.3cm) on all four edges. Black ink for typing, drawings, etc., is essential. All drawings, figures, etc., should be mounted *in place* on the 8.5 x 11" pages. We are not equipped to handle pieces of paper larger than 8.5 x 11" (21 x 27.5 cm).

d) Please include your e-mail address on your contribution.

Please do not fold, clip, or staple your pages. Protect the condition of your letters from the ravages of the mails by enclosing what you send in a cardboard or plastic folder, etc.

Foreign subscribers are reminded that regardless of the standard paper length you use, *all material* - letterhead, text, figures, addresses printed at the page bottom, *everything - must not exceed 10" (ca. 25.3 cm) from top to bottom.*

When formatting your contributions, please consider the following:

i) Try using a smaller type font: The body of this page is printed in 10 point type, which I believe is adequate for most purposes. Even 11 or 12 point type is acceptable if the particular font is not too large. Type smaller than 8 point should not be used.

ii) **PLEASE** avoid excessive margins. *Instruct your secretaries to avoid normal correspondence esthetics or practices, however time-honored or 'standard'!* This page has margins on both sides of 0.6" (*ca.* 1.55 cm), which is very adequate. Margins of the same size at the top and bottom are sufficient also, but don't worry if there is more space at the end of your document, for I can often use such spaces for notices, etc.

Also, please avoid large amounts of unused space at the top of letters. Give thought to the sizes of figures, drawings, etc., and please mount these so as to use the minimum space on the page.

iii) 'Position Available', 'Equipment Wanted', and Similar Notices. These are always welcome, but not for subscription credit. Such notices will appear, however, *only* if received with these necessarily rigid constraints: a) Single spaced; b) both side margins 0.6 - 0.7" (1.5 - 1.7 cm.)- NOT WIDER; c) the minimum total height, please, but definitely no more than 4.5" (11.5 cm.).

iv) AVOID DOUBLE SPACING LIKE THE BLACK PLAGUE ! ! ! This is extremely wasteful of space.

6. **Suggestions**: They are always welcome.

October 2000

*Telephone: 650-493-5971. Please confine telephone calls to 8:00AM-10:00PM, *Pacific Coast Time.*
*Fax: 650-493-1348 (Do not use for technical contributions which are to appear in the Newsletter, for Fax quality is not adequate.)
*Email: shapiro@nmrnewsletter.com
*http://www.nmrnewsletter.com

**Address all Newsletter correspondence to:**

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*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. 506 (Nov.) | 27 Oct. 2000 |
| No. 507 (Dec.) | 24 Nov. 2000 |
| No. 508 (Jan.) | 22 Dec. 2000 |
| No. 509 (Feb.) | 26 Jan. 2001 |
| No. 510 (Mar.) | 23 Feb. 2001 |

* 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

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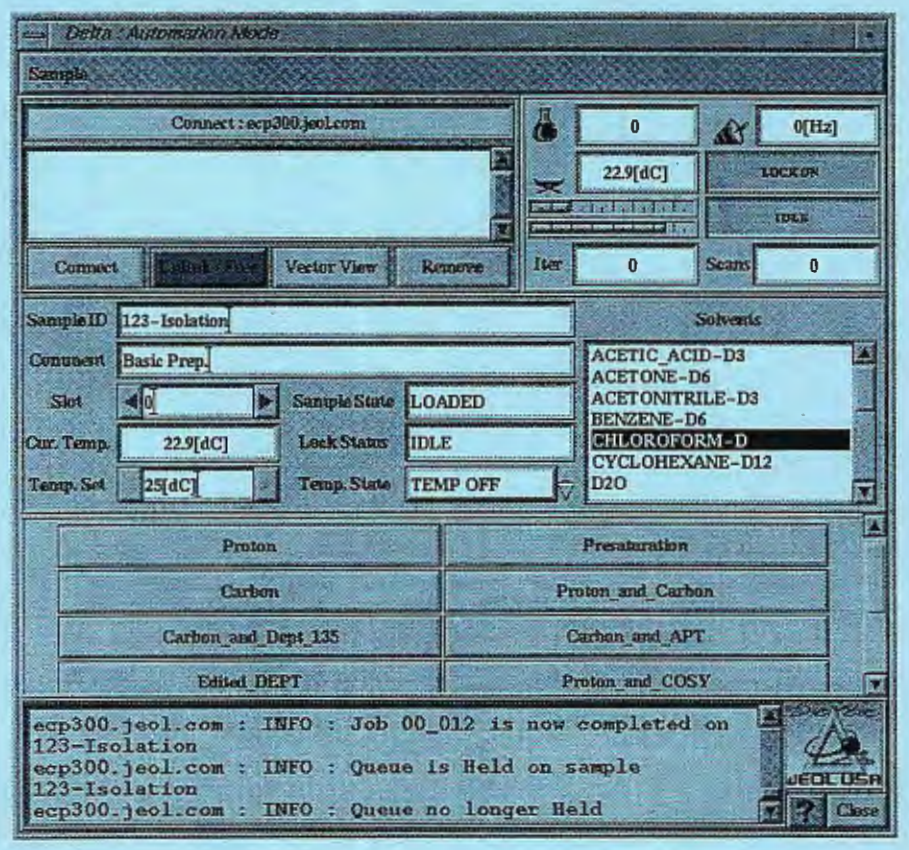


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