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No. 500
May 2000

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FORTHCOMING NMR MEETINGS

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.

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

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

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

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

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

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

continued on inside back cover

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April 26 2000 (received 4/28/2000)

Time-Dependent Gas Diffusion in Model Porous Media under Non-Ideal Lab Conditions

Dear Barry,

Thank you once again for your colorful reminder. I apologize for the delays in bringing this letter to you, and I trust this reaches you in time to avoid the "dreaded ultimatum". Besides, I don't want miss issue #500! Since receiving your reminder, things have been even more chaotic than usual. With the decision to hold the ISMRM and ENC conferences on back to back weeks, I was away for more than 2 weeks, and despite good intentions, the temptations of Asilomar kept me away from my computer and prevented me writing to you!

We are currently closing out the porous media gas-diffusion work at Brigham and Women's Hospital, and attempting to move that work to more pleasant environments. The situation there has always been fraught with political complications as they barely tolerated someone who's prime focus was geo-physical research, despite the fact the magnet was often under-used and I maintained the old GE Omega console in working order. In the next couple of weeks, BWH will move the 4.7 T animal magnet out of its current location and into storage while they figure out to where re-locate it, and so leave its current lab space empty (for possibly 4-6 months!) while awaiting delivery of 3 T human imager. We are set to relocate the work to David Cory's lab at MIT, where I look forward to once again working with physical scientists who appreciate the work I'm doing! However, my salary will continue to come from its current source, so the letterhead wont change!

The reason I am particularly disillusioned about this change over is the fact that the 4.7 T system at BWH had become a work-horse spectrometer. Despite its many faults, which I had learnt to work around, it has been churning out xenon gas diffusion data almost faster than I could process it. Given that most of this work has been done with regular (thermally-polarized) xenon rather than laser-polarized xenon, extensive signal averaging has been required. However, using 90% enriched ^{129}Xe gas at high pressures, it has been possible to reduce the required time down to 1 or maybe 2 weekends to study a given sample of glass beads infused with xenon, at up to 25 different diffusion times, with 3-5 repetitions of each diffusion time.

You may recall that the prime focus of this work has been to look at long-distance diffusion (using modified pulse-gradient spin echo techniques) through multiple pore spaces in model and real-life porous media (ie. glass beads and rocks). Xenon is particularly ideal for this, given its diffusion coefficient ~ 3 orders of magnitude higher than water, and its low surface interactions with the glass beads or grains, due to it being a noble gas. In previous work (1), we had shown that the basic characteristics of time-dependent diffusion, as predicted theoretically (2) and seen in water diffusion experiments (3) are indeed observed for gas diffusion as well. These include, principally, that the early time decrease of the time dependent diffusion coefficient ($D(t)$) is related to the surface-area/volume ratio of the medium, while the long-time $D(t)$ limit is equivalent to the inverse tortuosity (~ pore connectivity) of the sample. This was quite an achievement in rocks such as limestones with their very heterogeneous pore structures, and as a result, we are attempting to set up a parallel research effort at our collaborator's facility at Schlumberger Doll-Research in Connecticut, in order to measure tortuosity in more rocks more quickly.

A couple of anomalies presented themselves in our first work, however. The most obvious was that as we studied smaller and smaller glass beads, the short-time *D(t)* data deviated further and further from the expected *S/V* limit. This, I suspected, was due to significant diffusion of the gas spins during the application of the diffusion gradient pulse. In other words, a violation of the narrow-pulse approximation of the PGSE technique - an assumption usually valid when studying water or other liquid diffusion, except perhaps in the very smallest of restricted areas. Fukushima and colleagues have shown that the narrow-pulse approximation can be violated if the spins diffuse across more than about 14% of the pore space during the application of the gradient pulse (4). For water, with a typical gradient pulse time (δ) of 2 ms, this implies a distance of 2 μm, or a limiting pore size of 14 μm. For xenon gas, however, we use $\delta = 750$ μs, and yet the diffusion distance during this time is ~ 25 - 35 μm, for xenon at 6 - 3 atm pressure. Therefore, for pore sizes less than ~ 200 μm (or bead sizes less than ~ 1 mm), such effects should be observed. Due to T_2* and gradient coil hardware limitations, the only feasible way of testing this behavior was to increase the gas pressure in the sample cell, and so reduce the gas diffusion coefficient (inversely related to pressure). Therefore, I have been attempting to work through all the packed bead samples, with beads ranging from 4 mm down to 0.1 mm, now infused with xenon gas at ~ 6 atm pressure, rather than the 3 atm used in the earlier publication.

The results from a couple of the bead packs are shown in Figs 1 - 3. In addition, we are now using the Pade approximation to interpolate between the long and short-time *D(t)* limits, and so give a theoretical prediction for medium *t* data - a method that has been used successfully in the past for the water *D(t)* experiments (3). In all plots, the points in white are Xe *D(t)* when the samples were pressurized to 3 atm Xe, and was the data reported previously (1). The new data is in black, taken at 6 atm Xe pressure. The deviation at short diffusion times from the predicted *S/V* limit is very obvious in the 3 atm data. By increasing the Xe gas pressure, and so reducing *D*(0) (the free gas diffusion coefficient), the deviation from the predicted *S/V* limit is much reduced in the smaller beads. In fact, the pressure increase restores the data to the *S/V* limit in the 2 mm beads, and almost achieves the same result in 1 mm beads. Limitations to gas pressures in standard lab glassware are the only reason the deviation cannot fully eliminated. The reduction in the deviation is even starker in 0.5 mm beads, however the 6 atm D(t) from that sample is still being processed.

At the new higher gas pressures, it should be noted, multi-pore diffusion is still observable in the smaller beads. This distance is also a function of the maximum diffusion time allowable, which is related to T_1. At the expense of longer signal averaging times, T_1 can be increased in these samples, by reducing the amount of O_2 present in the gas mixture. The current research plan is now to look at the deviations from the Pade fit in the large beads - a result of ordered packing, rather than random packing in this sample?? The second area

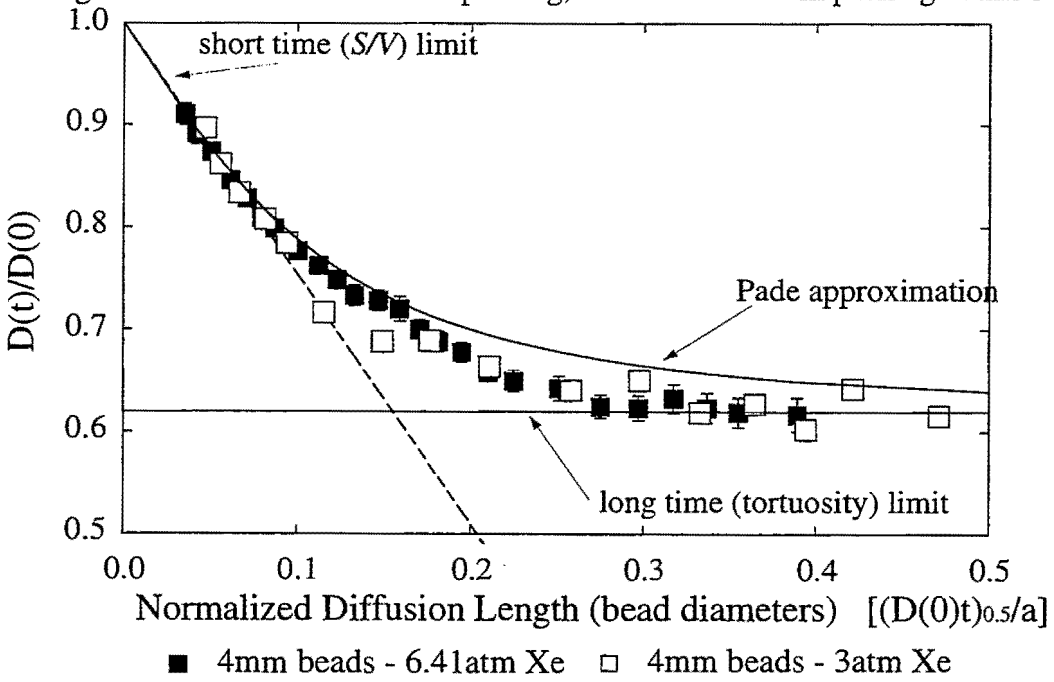

Figure 1: ^{129}Xe time-dependent diffusion coefficients *D(t)* measured in a 4 mm glass bead pack infused with Xe gas at 6 and 3 atm pressure. *D*(0) is the free gas diffusion coefficient, ~ 8.1×10^{-7} m^2s^{-1} for the 6 atm gas sample, and ~ 1.4×10^{-6} m^2s^{-1} for the 3 atm gas sample. Normalized diffusion length is a function of the diffusion time (Δ), which ranges from 20 to 3000 msec.

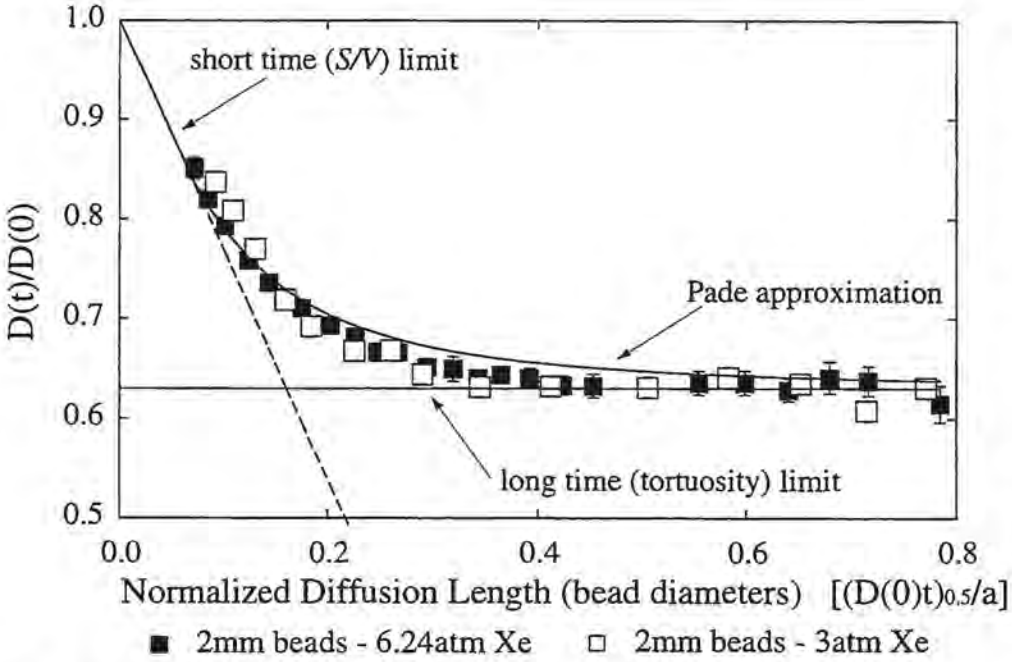

Figure 2: ^{129}Xe time-dependent diffusion coefficients $D(t)$ measured in a 2 mm glass bead pack infused with Xe gas at 6 and 3 atm pressure. $D(0)$ is ~ 8.1×10^{-7} m^2s^{-1} for the 6 atm gas sample, and ~ 1.4×10^{-6} m^2s^{-1} for the 3 atm gas sample. Note the 6 atm data lies on the theoretical S/V line and the Pade approximation at short times, and matches the Pade line well at most diffusion times.

we will continue to pursue is the study of mixed bead packs, with the aim of elucidating pore structural information from any other non-monotonic decrease in $D(t)$, as has already been seen in the Indiana Limestone rock sample (1). With the move to MIT, and the addition of a dedicated, computer controlled xenon gas polarizer, these studies should advance at an even greater rate in coming months.

Best Regards,

RW Mair

Ross Mair WITH SAM PATZ, PABITRA SEN + RON WALSWORTH

1. R. Mair, G. Wong, D. Hoffmann, M. Hürlimann, S. Patz, L. Schwartz and R.Walsworth *Phys. Rev. Lett.* **83**, 3324 (1999).
2. P. Mitra, P. Sen et al., Phys. Rev. B 47, 8565 (1993); Phys. Rev. B 49, 215 (1994).
3. M. Hürlimann, K. Helmer, L. Latour and C. Sotak, *J. Mag. Res.* **A111**, 169 (1994).
4. L. Wang, A. Caprihan and E. Fukushima, *J. Magn. Reson.* **117**, 209 (1995).

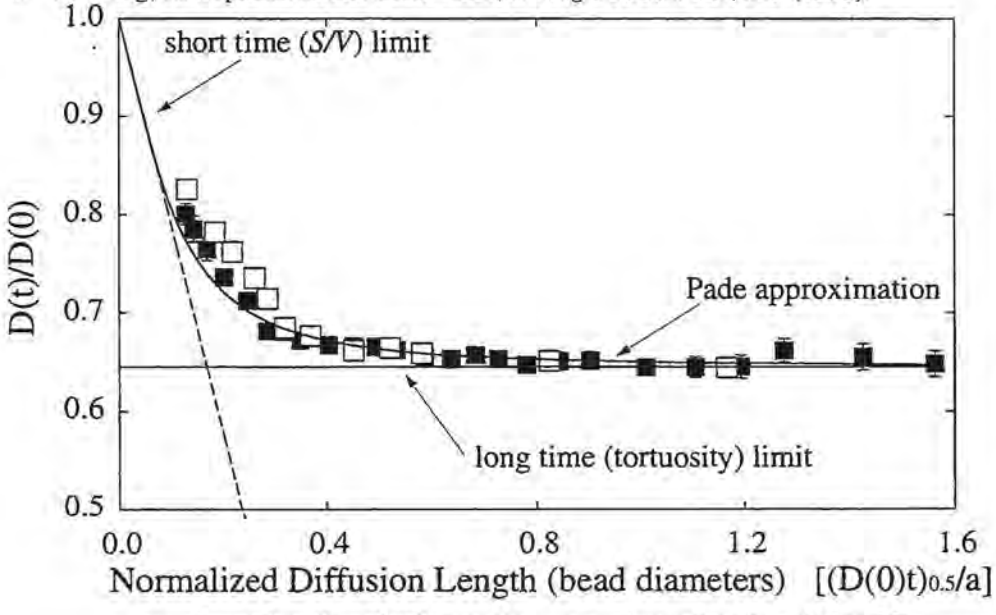

Figure 3: ^{129}Xe time-dependent diffusion coefficients $D(t)$ measured in a 1 mm glass bead pack infused with Xe gas at 6 and 3 atm pressure. $D(0)$ is ~ 8.1×10^{-7} m^2s^{-1} for the 6 atm gas sample, and ~ 1.4×10^{-6} m^2s^{-1} for the 3 atm gas sample. Note the large deviation from the S/V and Pade lines in the 3 atm data at short times, while the use of higher pressure Xe (6 atm) almost returns the data to the theoretical predictions.

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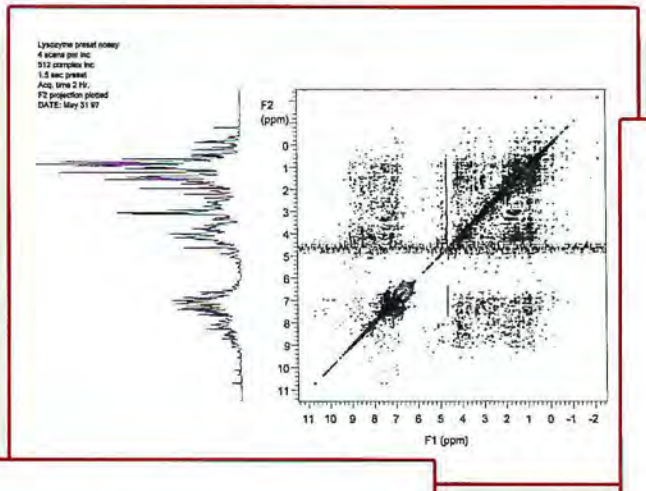

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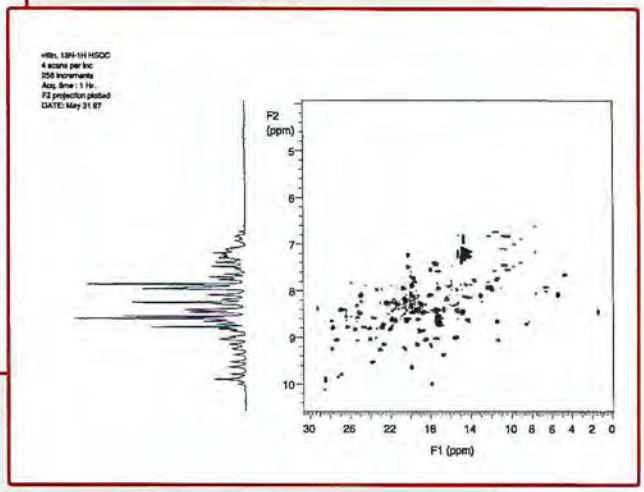

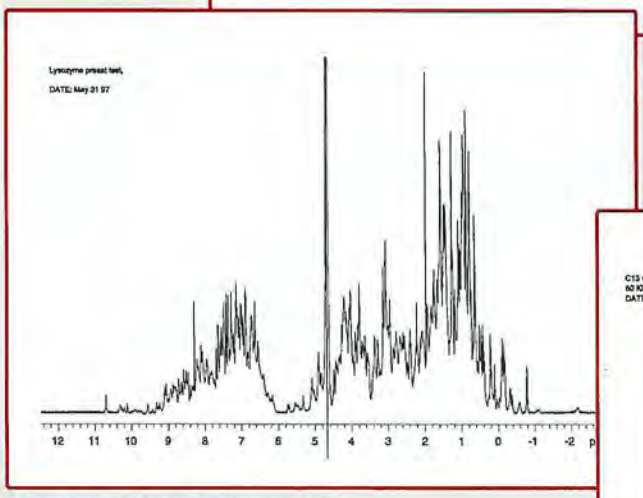

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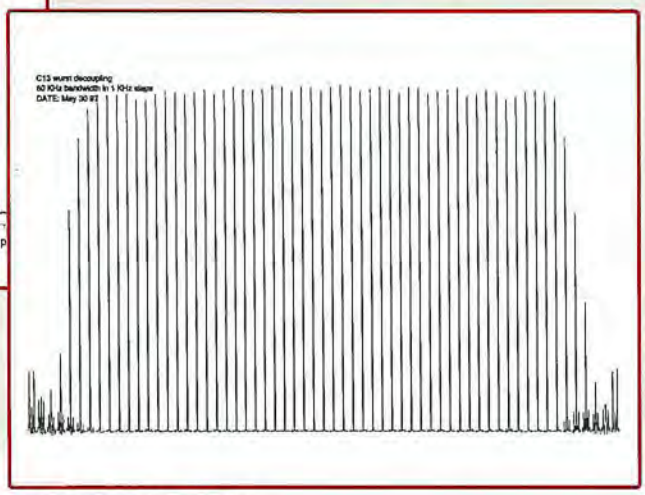

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April 24, 2000
(received 4/25/2000)

Vnmr , UNIX and NIS+

Dear Barry:

The computer front end for NMR spectrometer systems has become more elaborate and involved over the past two decades, progressing from a single user system with a proprietary operating system to general purpose UNIX systems supporting many users. This evolution has resulted in an increasingly large administrative load, especially on UNIX-based systems that have several inter-related files for setting up and managing user accounts.

For the past several years, we have been using NIS+ to maintain the files necessary to support our user base. This has had the benefit of both simplifying administration and reducing redundancy. All of the information that is normally stored in the /etc/passwd and /etc/group files on a workstation are now maintained in NIS+ data base tables (passwd.org_dir and group.org_dir). This data base is available to all of the systems, and only the master server needs to be updated with changes. Unlike the older NIS (and its predecessor yp), data added to NIS+ tables is available immediately, without the need to regenerate tables from flat files. This arrangement also simplifies things for users: they have a single login account, with a single password, and all of their NMR data is in their one home directory. They don't have to go to multiple instruments to either archive data or look for a particular data set. This also provides for more efficient utilization of disk space.

User access to the various spectrometers and workstations is controlled through one more NIS+ table, netgroup.org_dir. Netgroups allow you to group a set of users together and pass them to the login system as a single group. Any users I have who are checked out on the Gemini 200 spectrometer are placed in netgroup gem200. When the Chemistry 234 students get passwords on the system, they are placed in netgroup chem234. The Gemini 200 spectrometer system file /etc/passwd has the usual default accounts set up (root, lp, smtp, adm, nobody, etc.) plus one more line: +@gem200. This line tells the system to add the passwords for users in the gem200 netgroup to the allowed login password file. By adding a line +@chem234, I can also add the chemistry 234 users. At the end of the semester, removing that one line from /etc/passwd removes their login privileges on the system. Their student accounts can be removed at leisure, after the next billing cycle is complete. The netgroup table is also an easy, convenient way to get a list of all the users on a system if you need to put a user list into a reservation system. We have a crontab entry that goes out once a day and refreshes the user data base in the reservation system based on the contents of the netgroup table. Once a user is added to (or removed from) a spectrometer system, she is automatically added to (or removed from) the reservation system as well.

In order to make this work, there are several things that must be in place. One is that NFS sharing of home directory space, and the use of the auto_home table for the automounter, must be used to provide a single home directory for each user (with the same absolute path) on each system. In fact, NIS+ provides for an auto_home.org_dir table, making it easy to get the same automounter configuration on all of the systems.

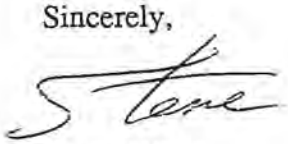
There is one other change necessary in order to run Varian's Vnmr program on several different systems, with a single login account. Normally, a user has a file ~/vnmrsys/global which has his personal configuration parameters (printer, plotter, lock power, etc.) that will change from system to system. In order to accommodate this file, we have added a wrapper script around the vn executable file. This wrapper looks for a file global.*<node>* and copies it to global, runs the vn executable, and then copies global back to global.*<node>* when it is done. The *<node>* extension comes from /etc/nodename so that nothing special need be done to implement the change or add new spectrometers. If the wrapper doesn't find a global.*<node>*, it simply gets a fresh copy from /vnmr/user_templates. This is especially useful when a global file becomes corrupted - simply delete it and re-run vnmr to recreate it.

The final (or perhaps the first!) caveat is that this requires a reasonably stable network environment. If all of your login password information is on a NIS+ server that is only up or available 80% of the time, then users will have problems logging in 20% of the time. If NFS mounts are slow and unreliable, then users may login but have trouble accessing or storing data. We have run this arrangement for several years now across our department-wide subnet, and have experienced little down time due to network related problems. (The day the department's central hub failed, however, everyone, including all of the spectrometers, was down!) In a spectrometer lab environment where all of the spectromters and workstations could be placed on a single hub, with its own UPS, speed and reliability should be very good.

I should mention that while NIS+ is most useful in a moderately sized environment (more than 3 or 4 workstations), it does have a fairly steep learning curve. When I had problems with migrating from Solaris 2.4 to Solaris 2.6 however, the Sun technical support was most helpful in resolving some relatively minor issues, as well as the big problem of how to actually move the existing tables. On balance, this has been a tool well worth the time and effort it took to learn and implement. Since it is also possible to add your own tables to the NIS+ structure, you can add local information for all of your workstations the same way. We have added another table for the automounter that lists directories on remote hosts that may be mounted on the spectrometer systems for archiving data. All of these directories can be mounted under the /nmrdata mount point as necessary. Thus, a student in a research group with shared disk space can simply save his data to /nmrdata/group_name/filename and have it stored on his workstation.

The advantage of all of this is that you need make these changes only once, and they are available on all the NIS+ client machines. If you add a new system, simply add it as a NIS+ client and it gets all of the NIS+ tables immediately. The most you may have to do is to create a new netgroup for the new system and populate it with the authorized users. I encourage you to try NIS+ - it may give you more time for doing NMR!

Sincerely,



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(received 4/28/2000)

April 26, 2000

Collisional correlation time measurement by use of repeated and compensating pulsed magnetic field gradients.

Dear Barry,

We describe a method for estimating parameters $\langle u^2 \rangle$ and τ_c of the velocity autocorrelation function $R_u(\tau) = \langle u^2 \rangle e^{-|\tau|/\tau_c}$, which describes the velocity of a Brownian particle. The diffusion coefficient for such a process is given by $D = \langle u^2 \rangle \tau_c$. Two pulse sequences are used: a) a repeated version of the standard bipolar pulsed gradient spin-echo sequence (PGSE) with two cycles (RSPG), and b) a flow compensated pulsed gradient spin-echo sequence with two cycles (CPG) (Fig. 1). The flow compensated pulsed gradient sequence has its first moment zero, $\int tg(t)dt = 0$, where $g(t)$ is the effective gradient, and is akin to the even spin echo. It has the property that the magnetization of a spin moving with constant velocity accumulates no net phase during the sequence. Even for stochastic motion with correlations, such as turbulent flow, even echoes refocus spins partially and reduce signal loss. We use this sensitivity towards correlated motion to improve correlation time measurements.

For a train of n (n = 2 in Fig. 1) bipolar gradient pulse pairs whose polarity is chosen to probe spin dynamics we define the apparent diffusion coefficient $D_a(\Delta)$ measured by

$$E(q,\Delta,\delta) = e^{-4n\pi^2 q^2 (\Delta - \frac{\delta}{3}) D_a(\Delta)} . \qquad [1]$$

The factor n in the exponent (Eq. [1]) ensures that we have $D_a = D$ for any number n of bipolar pulses for uncorrelated Brownian motion ($R_u(\tau) = 2D\delta(\tau)$).

The functional form of the two apparent diffusion coefficients can be calculated in terms of velocity autocorrelation parameters.

$$D_R(\Delta) = D(1 + \frac{a + e^{-\frac{\tau_m}{\tau_c}} b/2}{\delta^2(\Delta - \delta/3)}) , \quad \text{RSPG sequence} \qquad [2]$$

$$D_C(\Delta) = D(1 + \frac{a - e^{-\frac{\tau_m}{\tau_c}} b/2}{\delta^2(\Delta - \delta/3)}) , \quad \text{CPG sequence} \qquad [3]$$

where

$a = -2\tau_c^2\delta + \tau_c^3(1 - e^{-\frac{\delta}{\tau_c}})(2 - e^{-\frac{\Delta}{\tau_c}} + e^{-\frac{\Delta-\delta}{\tau_c}})$, $b = \tau_c^3(1 - e^{-\frac{\delta}{\tau_c}})^2(1 - e^{-\frac{\Delta}{\tau_c}})^2$ and δ, Δ, and τ_m are defined in Fig. 1.

In Fig. 2 we compare the two apparent diffusion coefficients for a small δ and $\tau_m = 0$. Both the apparent diffusion coefficients tend to the asymptotic diffusion

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coefficient D for $\tau_c << \Delta$. In other words if the spin motion is uncorrelated in the time scale of our pulsed gradient sequence, then both the apparent diffusion coefficients measure the same quantity D. For highly correlated motion within the time Δ ($\tau_c >> \Delta$) Eq. [3] implies that $D_C(\Delta)$ approaches zero for any Δ as τ_c tends to infinity. In other words, there is no attenuation in image intensity because of motion. This is the distinguishing feature of the compensated CPG sequence. It compensates for correlations in motion and increases sensitivity for correlation time measurements. In addition, for $\tau_c >> \Delta$, we have $D_R(\Delta) = \langle u^2 \rangle \Delta$, the ballistic ($\sigma_X^2 \propto \Delta^2$) short time motion.

We have also plotted the normalized difference $\frac{D_R(\Delta) - D_C(\Delta)}{D}$ of the apparent diffusion coefficients for the repeated and the compensated pulse sequences for $\delta = \tau_m = 0$ in Fig. 2. It is a measure of the spin phase refocusing by the CPG sequence and depends on the relative values of Δ and τ_c. The difference is a linear function of Δ for $\tau_c >> \Delta$, because $D_C(\Delta) \to 0$ and $D_R(\Delta)$ is linear. It has the interesting property of having a maximum at $\frac{\Delta}{\tau_c} = 1.24$. It approaches zero for $\tau_c << \Delta$ because the particle motion appears random. Thus we immediately know the range of τ_c in our experiment from the difference of $D_R(\Delta)$ and $D_C(\Delta)$ as a function of Δ.

Some of the results of correlation time measurement for granular materials (2 mm hard plastic spherical beads) rotated in a half-filled horizontal cylinder are contained in a paper published in Phys. Rev.Lett. Vol, 84, 266-269 and other details will be published in J. Mag. Res. later this year.

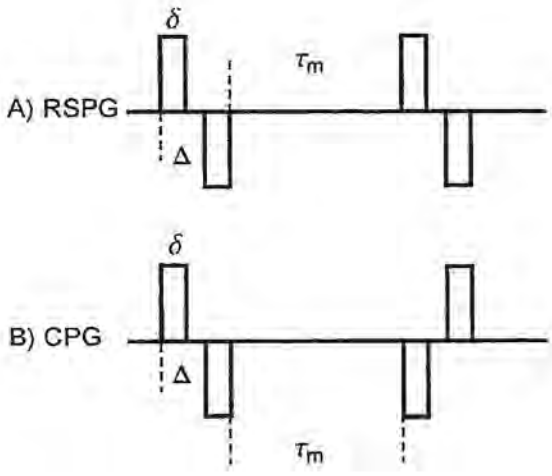

Fig. 1

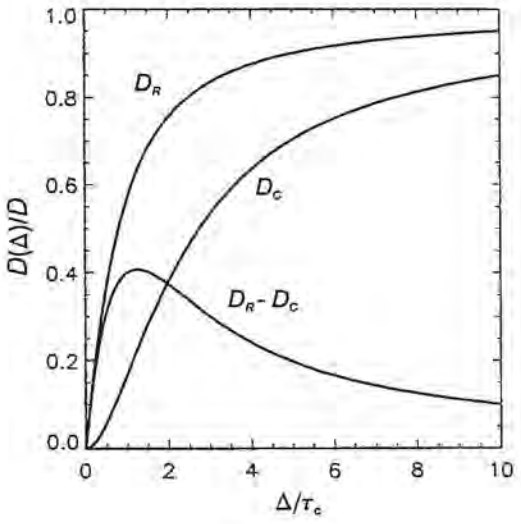

Fig. 2

Sincerely,

S.A. Altobelli, A. Caprihan, J.D. Seymour, and E. Fukushima

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

March 31, 2000 (received 4/4/2000)

VARIAN

RnaPack- A Package for NMR Studies of RNA and DNA

Dear Barry,

During a short sabbatical at Varian, Robin Bendall (currently based at the University of Melbourne in Australia) developed a useful and convenient method by which triple-resonance experiments on proteins can be done without extensive calibration or parameter entry. This approach eventually resulted in ProteinPack, a set of pulse sequences, parameter sets, manuals and macros that has now been placed in the USERLIB (available on-line via our Web site (nmr.varianinc.com).

Peter Lukavsky at Stanford University (lukavsky@stanford.edu) has used the same philosophy in developing RnaPack. He was motivated to do so by the speed and accuracy with which complex, high performance experiments could be carried out in H2O solutions at a variety of magnetic field strengths. The unique requirements of RNA and DNA with respect to proton exchange and the desirability of using H2O solutions required the addition of a large number of pulse sequences. Further enhancements such as the use of WET water suppression and region-selective adiabatic pulses have been added, as well as TROSY versions of some experiments. Pulse sequences have been coded based upon primarily those published by the Bax, Kay and Pardi labs, as well as some developed by Peter.

Peter and I have been working together on distributing RnaPack to the Varian NMR community. The result is a package which is described below. Users can rapidly and accurately automatically calibrate and update their parameter sets. This permits experiment selection by using a menu button, producing a calibrated parameter set only needing input of the numbers of increments and transients.

Pulse Sequences in RnaPack:

13C gradient HSQC	15N gradient HSQC
13C CT-gradient HSQC	15N watergate HSQC
13C HMQC	15N HMQC
13C CPMG HSQC	15N CPMG HSQC
13C NOESYHSQC	15N NOESYHSQC

1H WATER (incl. presat,jr,wet,watergates,shaped pulse)
1H WGNOESY (watergate)
1H SSNOESY
1H WET/WATERGATE ROESY
HCCHCOSY HCCHRELAY
HCCHTOCSY DE-HCCHTOCSY
CPMG-NOESY

HNN-COSY
C-HNCCCH U-HNCCCH
A-HNC-TOCSY-CH
G-HNC-TOCSY-CH
A-HCCH-TOCSY

Installation

Users can obtain the compressed file (see below), place it in their /vnmr/userlib/psglib, and use the normal userlib extract procedure, e.g. "extract psglib RnaPack". Once extracted, the various pulse sequences need to be compiled. Appropriate parameter sets are now in your vnmrsys/parlib. The parameter sets are organized in a general manner and the "dg" screen shows just the relevant parameters for the detection channel and normal parameters used for broadband X-nucleus decoupling (much as in hmqc.par). In fact, the "dg" is fairly close in appearance to that for s2pul.par. Parameters that are specific to a pulse sequence are shown in "dg2", and these are relatively few. The tcl/tk graphical interface is also included, making parameter entry and option selection very convenient and more robust than with traditional parameter entry.

Macros are provided for easy installation. Once the "extract" is done the user can select the "Install" menu button which performs pulse sequence compilation and shape creation, then recalls the rna_gChsqc parameter set. If the stored values are not appropriate for the spectrometer a table of important parameters is displayed and the user can update the parameter set. Once these have been updated (or the updating skipped by supplying "n" as an answer to a question), the macro will update all the RnaPack parameter sets with appropriate values.

In the update process the user changes 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.

After the rna_gChsqc.par parameter set is updated and stored, the user can automatically recalibrate by using a macro that fully calibrates all pulse widths, including WET, and all gradients. When each is finished the result is plotted, the fid is stored, and each parameter set, now updated, is stored back in vnmrsys/parlib.

Operation

From now on, the user only needs to enter the name of the pulse sequence or use a menu button (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".

If 2H decoupling is desired, the user enters dm3='nyn' (assuming a 4th channel or Lock/Decoupler 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 RnaPack experiments.

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. Since these contain text strings corresponding to the normal coefficients used by wft2d, the macros wft2da and ft3d automatically use them as arguments for wft2d and for a coefficient table for ft3d. Hence, wft2da(<#>) and wft2da('ni2'<,#>) are all that are necessary for 2D processing from a 2D <or 3D>

data set. The 3D ft command ft3d operates normally, e.g. ft3d, ft3d('f1f3'), ft3d('f1f3,'f2f3'), etc

The Basis for RnaPack

The underlying basis for the success of RnaPack 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.

The other factor allowing parameter prediction is that natural resonance frequencies for carbon and proton nuclei in RNA and DNA are observed to fall in specific regions with few but predictable exceptions. Each of these experiments is designed so that the 13C and 15N frequencies are not changed, eliminating operator error. Any required frequency shifts are done within the pulse sequence itself. The proton frequency is always placed at the water position .

Many sequences involve region-selective pulses (normal and adiabatic). 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). RnaPack provides a single macro, "rnacal", that based on the value of "sfrq", automatically creates all shapes needed by all of the RnaPack 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 tpwr, pwClvl, pwNlvl and amplifier compression values. The coarse attenuators are usually not changed and power levels are adjusted via the linear modulators present on each channel (Unityplus and UnityINOVA). Thus, as long as the "hardpulse" pulse widths are calibrated, nothing else is required.

Obtaining RnaPack:

1. Use browser to connect to www.varianinc.com. Go to NMR userlib pages and download RnaPack.
2. Place the files in /vnmr/userlib/psglib (as vnmr1). Logout and login as user. cd/vnmr/userlib.
3. extract psglib RnaPack
4. Printout and read RnaPack.README from /vnmr/userlib/psglib
5. (in VNMR) use menus:
   Setup...RnaPack...Install
   answer questions

See you at Asilomar,

George

George A. Gray
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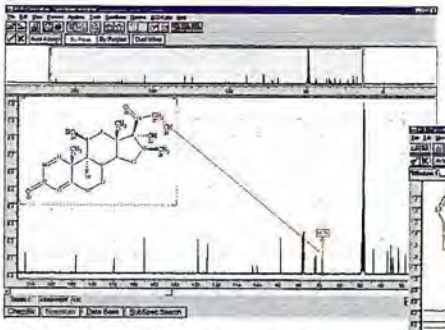

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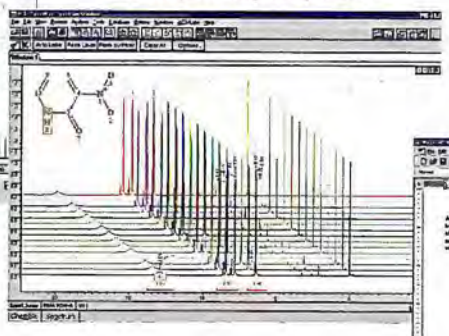

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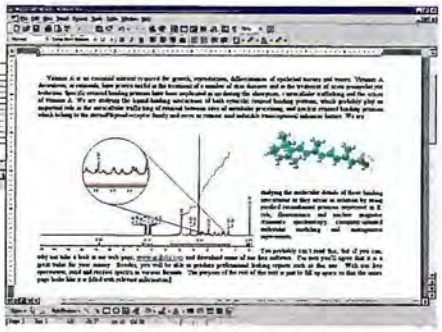

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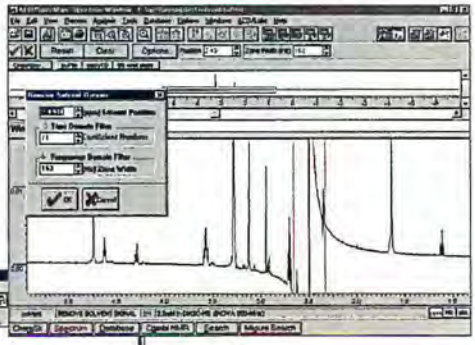

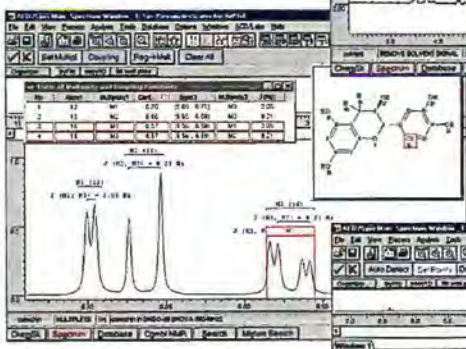

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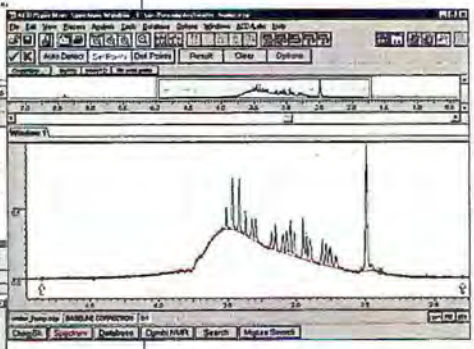

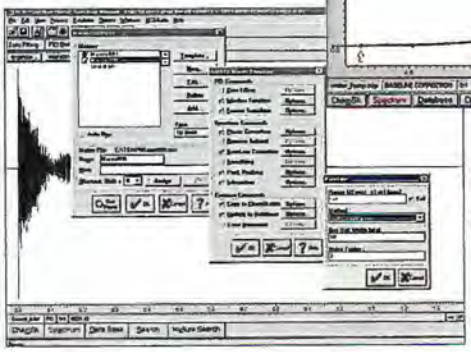

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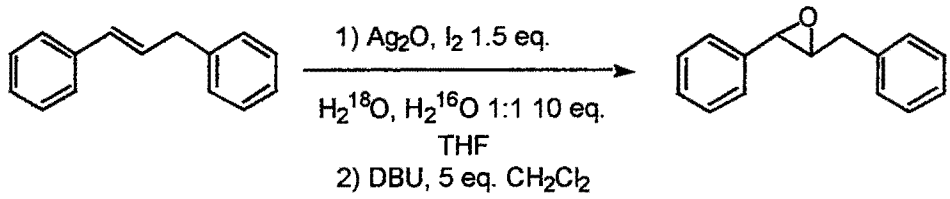
(received 4/21/2000)
April 20, 2000

TITLE: Oxygen-18 Isotope Effect on ^{13}C Chemical Shifts

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

Dear Dr. Shapiro:

We were interested in using the heavy atom oxygen-18 isotope effect on the ^{13}C chemical shifts of epoxides to evaluate the outcome of double label experiments involving ^{18}O. To determine the magnitude of the isotope effect, 1, 3-diphenyl propene oxide was prepared from a 1:1 mixture of $H_2{}^{18}O$ and $H_2{}^{16}O$.



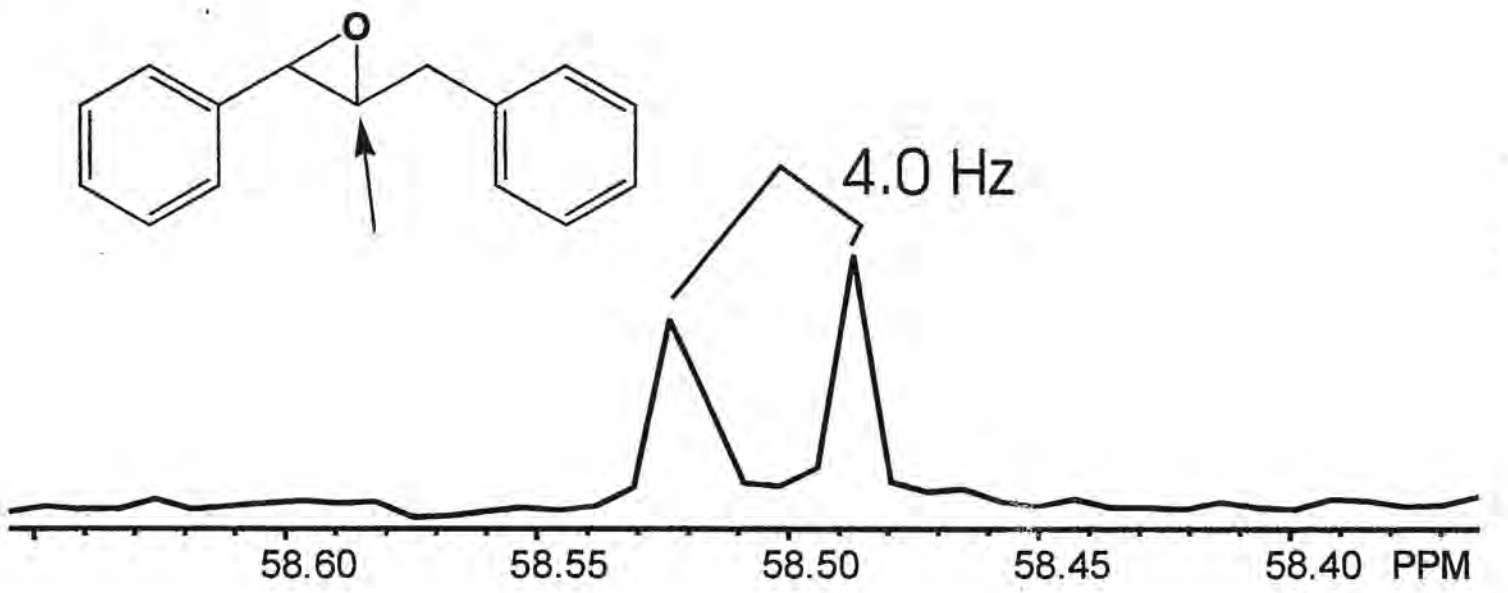
The ^{13}C spectrum was obtained on a Varian Unity 500 spectrometer at 125.6 MHz and the isotope effect was found to be substantial. A difference of 3.7 and 4.0 Hz was observed for the alpha and beta carbons, and the signals were well resolved. Large isotope effects have been observed for carbonyl groups and ethers, but to our knowledge this is the first measurement of an ^{18}O heavy atom isotope effect in epoxides.

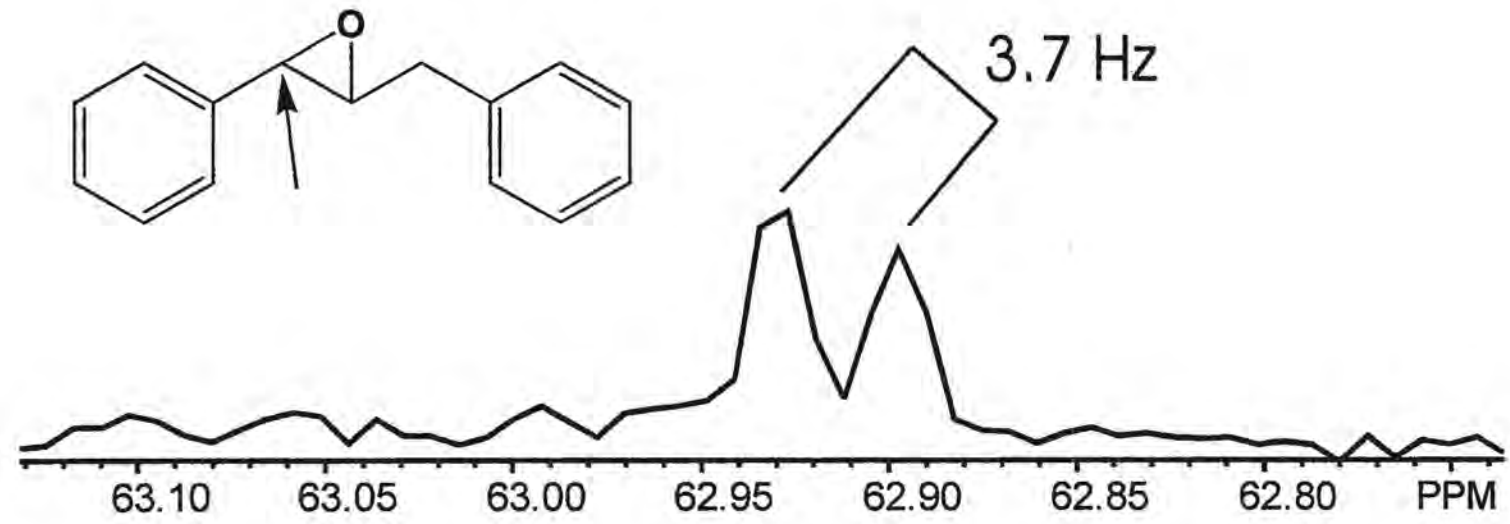
Sincerely,

Peter Beak

Prof. Peter Beak and David Anderson

Please credit Vera V. Mainz's account at UIUC.


O
4.0 Hz
58.60
58.55
58.50
58.45
58.40
PPM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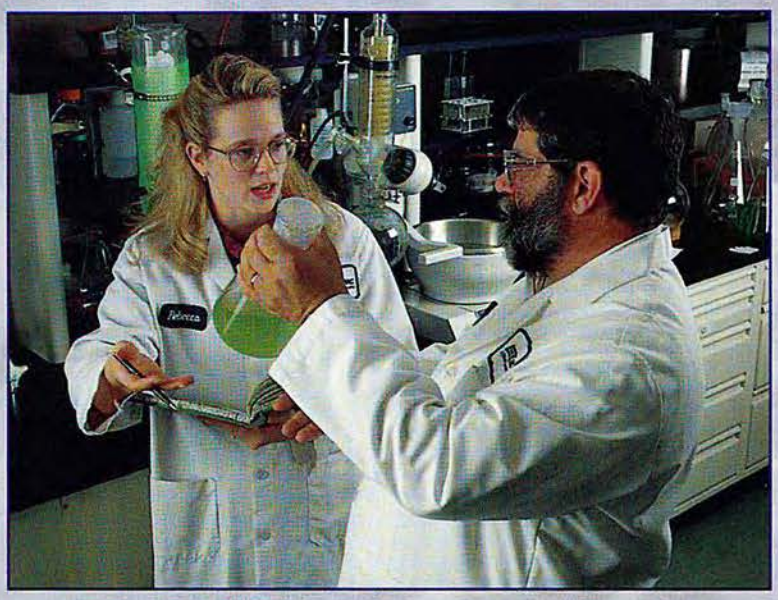

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L-Aspartic Acid-$^{13}C_4$,^{15}N
L-Cysteine-$^{13}C_3$,^{15}N*
L-Glutamic Acid-$^{13}C_5$,^{15}N
L-Glutamine-$^{13}C_5$,$^{15}N_2$*
Glycine-$^{13}C_2$,^{15}N
L-Histidine-$^{13}C_6$,$^{15}N_3$*
L-4-Hydroxyphenylalanine-$^{13}C_9$,^{15}N
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L-Lysine-$^{13}C_6$,$^{15}N_2$
L-Methionine-$^{13}C_5$,^{15}N
L-Phenylalanine-$^{13}C_9$,^{15}N
L-Proline-$^{13}C_5$,^{15}N
L-Serine-$^{13}C_3$,^{15}N
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April 13, 2000
(received 4/21/2000)

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

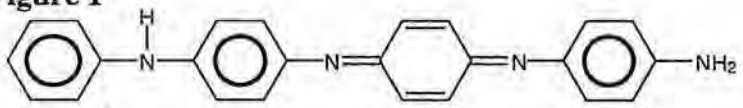
Please credit to the subscription of Peter Rinaldi

Monitoring Isomer Distribution in the Solid-State

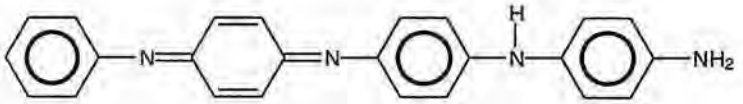
Dear Dr. Shapiro,

We have been applying solid-state NMR to understand better the solid-state structure of polyaniline in an effort to relate the structure to the observed conductivity; polyaniline is a conducting organic polymer. Recently, Alan MacDiarmid and his group have suggested that the presence of positional as well as cis/trans isomers may have a significant impact on the conductivity.[1] In order to simplify their isomer characterization they synthesized and investigated by solution NMR the aniline tetramer. As the repeat unit of aniline consists of four aniline groups (Figure 1), the tetramer is the smallest relevant oligomer. The ^{13}C solution NMR data show that the positional isomer present in solution is dependent on the solvent; the isomers present in $CHCl_3$ and DMSO are shown in figure 1.[2]

Figure 1



A $CHCl_3$



B DMSO

As the conductivity is a solid-state property we were motivated to see if the solvent dependent isomer composition was retained when the solvent was removed. We have synthesized the tetramer and cast films of the tetramer from $CHCl_3$ and DMSO with slow drying. Figure 2 shows the ^{13}C CP/MAS data for the two films as well as the as-synthesized tetramer. The tetramer is synthesized from an aqueous suspension and is never in solution. Comparison of the data show that there are significant differences in the carbon chemical shifts for the two films, we currently assign these differences to the fact that only one of the two positional isomers is present in each sample. While the resolution is lower for the as-synthesized material, the data indicate that it contains a mixture of the two isomers, with a larger portion of isomer A. Also, the peak at 160 ppm in the film cast from $CHCl_3$ indicates that it does contain a sub-population of isomer B, consistent with the solution NMR results.[2]

The results show that at least for the lower molecular weight species, solid-state NMR is an ideal technique to investigate the presence of various isomers and their distribution.

Sincerely,

Matthew Espe Michael Schmeida Bryan Gates

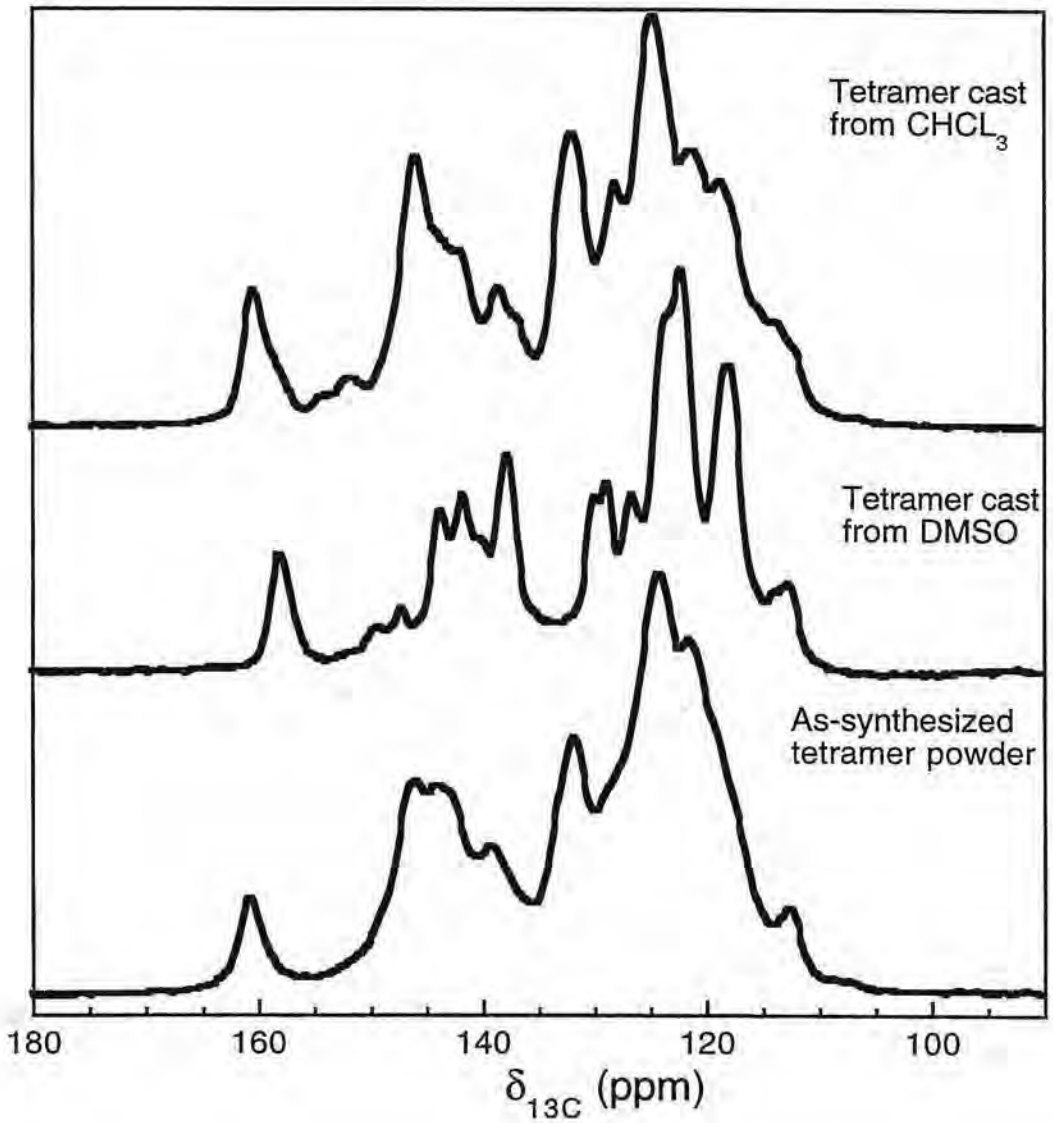


Figure 2: ^{13}C CP/MAS Hahn-echo NMR spectra.
Spinning speed is 5 kHz and field strength is 200 MHz (proton).

1) MacDiarmid, A,G., Zhou, Y., and Feng, J., *Synth. Met.*, 1999, 100, 131
2) MacDiarmid, A.G., Zhou, Y., Feng, J., Furst, G.T., and Shedlow, A.M., *Poly. Prep.* 1999, 40, 246.

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

April 21, 2000
(received 4/23/2000)

^{129}Xe NMR Investigation of Gas Mixtures Inside Nanochannels

Dear Barry,

Recently, we have been using optically polarized (OP) ^{129}Xe as a probe to investigate gas dynamics in a one-dimensional channels system. The xenon inside the channels can be regarded as a one-dimensional gas phase. The gas can only move along the channel axis, and two xenon atoms can not pass each other while inside the channels.

The material used for the channels system is tris(*o*-phenylenedioxi)cyclophosphazene (TPP) obtained by evacuation of the TPP inclusion compound with benzene. Its channels are about 5 Å diameter, just slightly larger than the diameter of a xenon atom, 4.4 Å. The ^{129}Xe was optically pumped using a home-built optical pumping apparatus. The ^{129}Xe spectra were obtained at 138.33 MHz using a Chemagnetics 500 MHz spectrometer (11.7 T).

The xenon resonance from inside the channels exhibits a chemical shift anisotropy (CSA). The orientational dependence of the resonance stems from xenon–channel wall and xenon–xenon interactions within the channel. The gas mixtures used in the present study are 2.5% mixtures of xenon in He, Ne, or Ar. The principle values of the CSA tensor (diagonal elements in the principle axis system (PAS)) were calculated using a nonlinear least squares fitting routine. The following definitions were used for the CSA tensor values (isotropic chemical shift, σ_{iso}; anisotropy, $\Delta\sigma$; and asymmetry parameter, η)

$$\sigma_{iso} = (\sigma_{11} + \sigma_{22} + \sigma_{33})/3$$

$$\Delta\sigma = \sigma_{33} - \sigma_{iso}$$

$$\eta = (\sigma_{22} - \sigma_{11})/\Delta\sigma$$

with $|\sigma_{33} - \sigma_{iso}| \geq |\sigma_{11} - \sigma_{iso}| \geq |\sigma_{22} - \sigma_{iso}|$ such that $\eta \in [0,1]$.

The asymmetry parameter was close to zero in all cases, indicating a nearly cylindrically symmetric environment. The resulting values of σ_{iso} and $\Delta\sigma$ are plotted vs temperature for each gas mixture in Figure 1. Each mixture behaves similarly. The isotropic chemical shift decreases with increasing temperature. The experiments were all done at ambient pressure so the xenon density decreases as the temperature increases; and the chemical shift of xenon is proportional to its density.

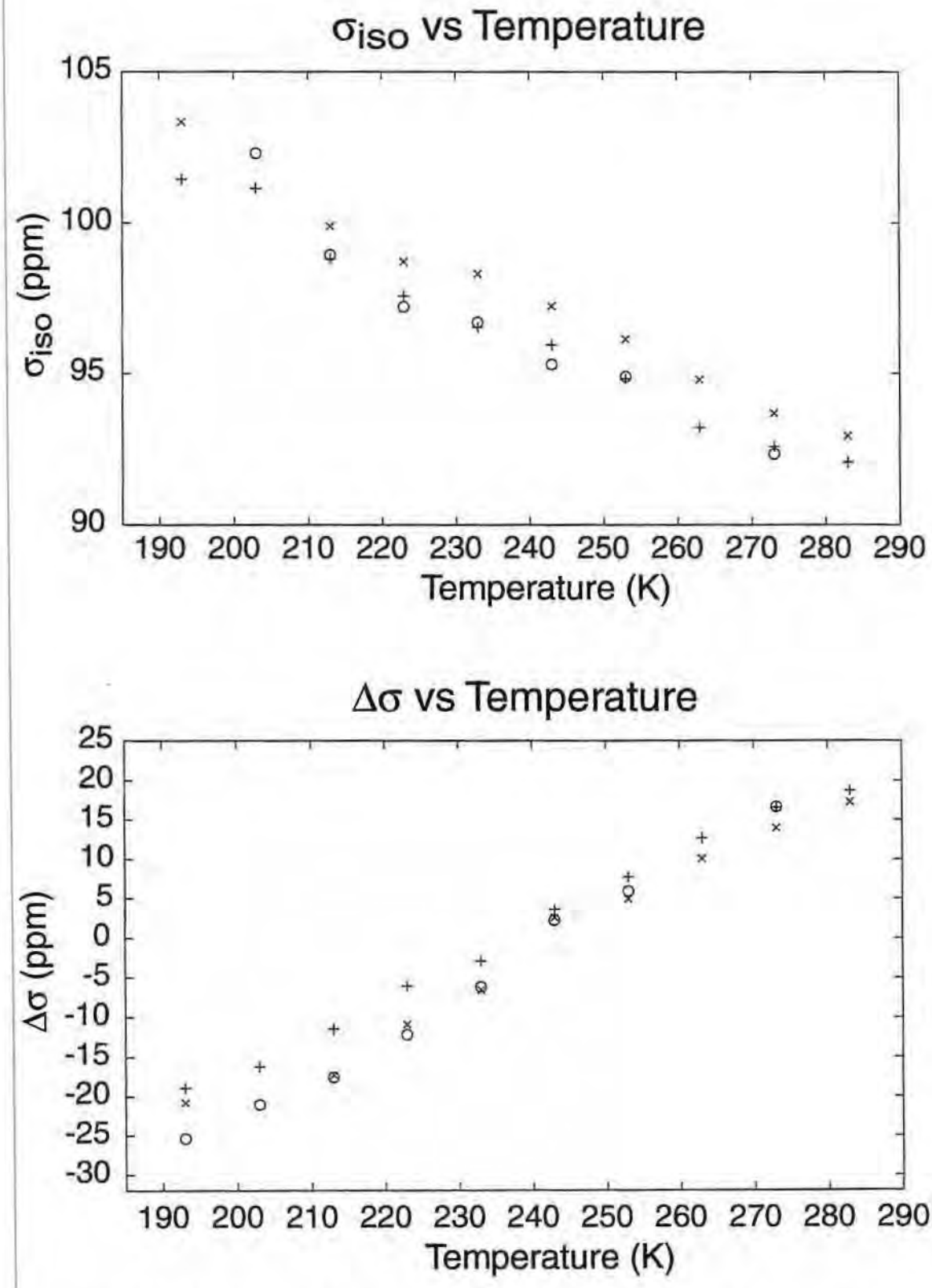
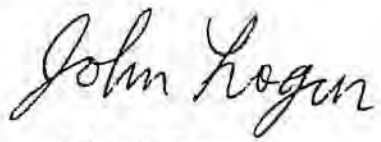


Figure 1. **O** = Xe/He micture, **X** = Xe/Ne mixture, and **+** = Xe/Ar mixture. *top*: isotropic chemical shift vs temperature; *bottom*: anisotropy vs temerature for the three gas mixtures.

To explain the anisotropy temperature trend, consider the strength of the Xe–Xe interactions. At high temperatures, the gas is not very dense, so the average distance between two xenon atoms is large. In addition, the atoms move at higher speeds, making the Xe–Xe interaction small compared the Xe-wall interaction. The Xe–wall interaction distorts the electron cloud around the xenon nucleus (and hence the CSA tensor) by stretching it perpendicular to the channel axis. On the other hand, at low temperatures, the gas mixture is more dense and the atoms move slower, making the effect of the Xe–Xe interaction more pronounced. The Xe–Xe interaction distorts the electron cloud parallel to the channel axis. Therefore, as a result of the two orthogonal interactions (Xe–Xe and Xe–wall) each being dominating the line shape at different temperatures, an inversion in the sign of the anisotropy is observed. For each gas mixture, there was a temperature that had an essentially isotropic line shape. At this temperature, the two interactions have equal strengths yielding a nearly spherical electron distribution around the nucleus, and thus an isotropic line shape. We did not observe any large, mixture-dependant characteristics in the ^{129}Xe NMR spectra of xenon in the TPP channels.

In closing, I would like to acknowledge my coworkers: Thomas Meersmann, Roberto Simonutti, Stefano Caldarelli, Angiolina Comotti, Piero Sozzani, and Alexander Pines for their contribution to this work.

Sincerely,



John W. Logan

Postdoctoral Position: Characterization of Conducting Polymers

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966 Elsinore Court
Palo Alto, CA 94303

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Rockville, MD 20852

April 21, 2000
(received 4/25/2000)

How oriented "oriented" systems really are?

Dear Barry:

Recently, there has been an increased interest in oriented lipid systems, both for high resolution and solid state NMR studies of protein structure. Best spectroscopic results are achieved when membranes are oriented with very little mosaic spread. Precise knowledge of orientational distribution functions of lipid particles in the magnetic field is desirable for data analysis.

Two elements are essential to obtain this information: (i) spectra of the lipid matrix that are acquired with sufficient bandwith and features that manipulate intensities of the first data points in the FID, like digital filtering, turned off, and (ii) echo sequences like the qudrupolar echo [1] and or the Hahn echo [2] that result in spectra without baseline distortions and free of first order phase errors. To obtain orientational distribution functions, one typically measures chemical shift, dipolar, or quadrupolar interactions that depend on orientation of lipid patches to the external magnetic field. In cases of second-rank tensorial interactions, this involves an angular dependence that is described by the second order Legendre polynomial $P_2(\cos\theta) = \frac{1}{2}(3\cos^2\theta - 1)$. This scales interactions by a factor between +1 and –0.5 (including zero at the magic angle). The resulting spectra are broad powder averages representing superposition of spectra from lipid patches with all possible orientations. The measured spectrum, $S(\omega)$, contains not only information about the effective strength of anisotropic interactions, determined by conformation and motions of lipid molecules in these patches and represented by an anisotropy distribution function, $g(x)$, but also the angular distribution function $p(\theta)$ that describes orientation of lipid aggregates with respect to the magnetic field

$$S(\omega) = \int p(\theta)\left[g(x)\frac{\partial x}{\partial \omega}\right] d\theta, \quad x = x(\theta,\omega).$$

The extraction of $g(x)$ from spectra with a random distribution of orientations, $p(\theta) = \sin\theta$ by a procedure called dePakeing has been widely used in lipid research for the past 15 years [3,4]. Better numerical approaches have made it possible to extract not only $g(x)$ but also $p(\theta)$ albeit with some limitations [5]. We have recently been able to adapt these methods to systems such as bicelles or lipid bilayers oriented on glass plates, where the orientational distribution function appears to consist of two components, only one of which has a certain preferred orientation, whether magnetically (bicelles) or mechanically (glass plates) induced [6]. The capabilities of our numerical methods can be illustrated by the following figure:

– continued on page 35

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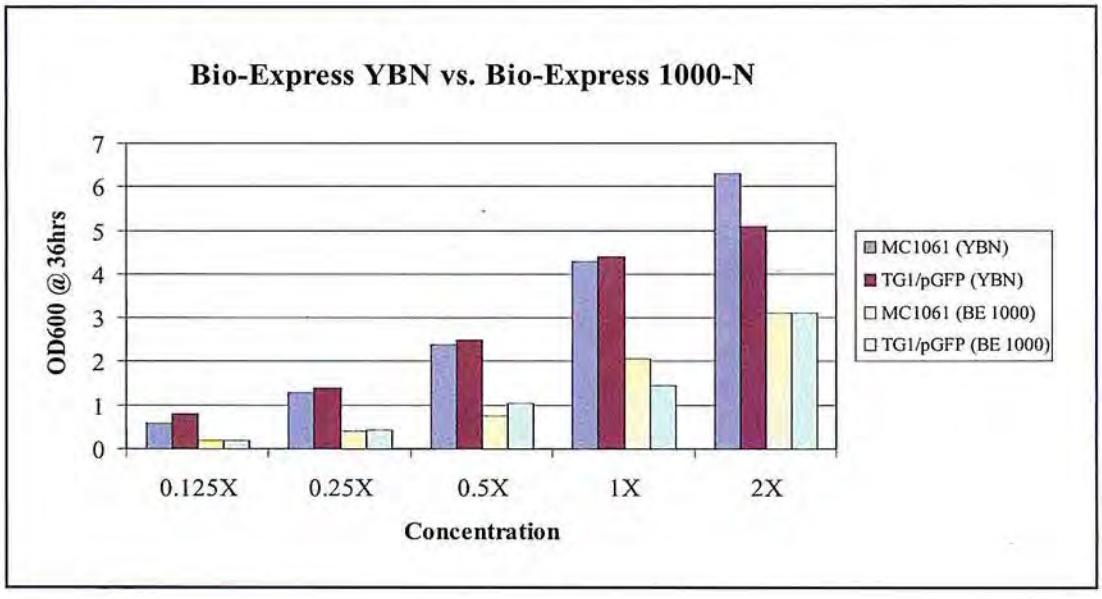

Figure 1

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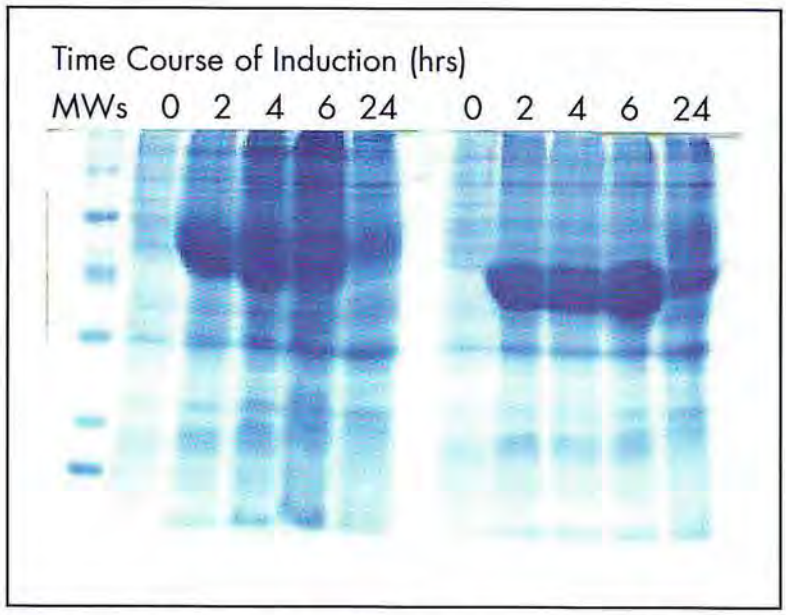

Figure 2*

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continued from page 32

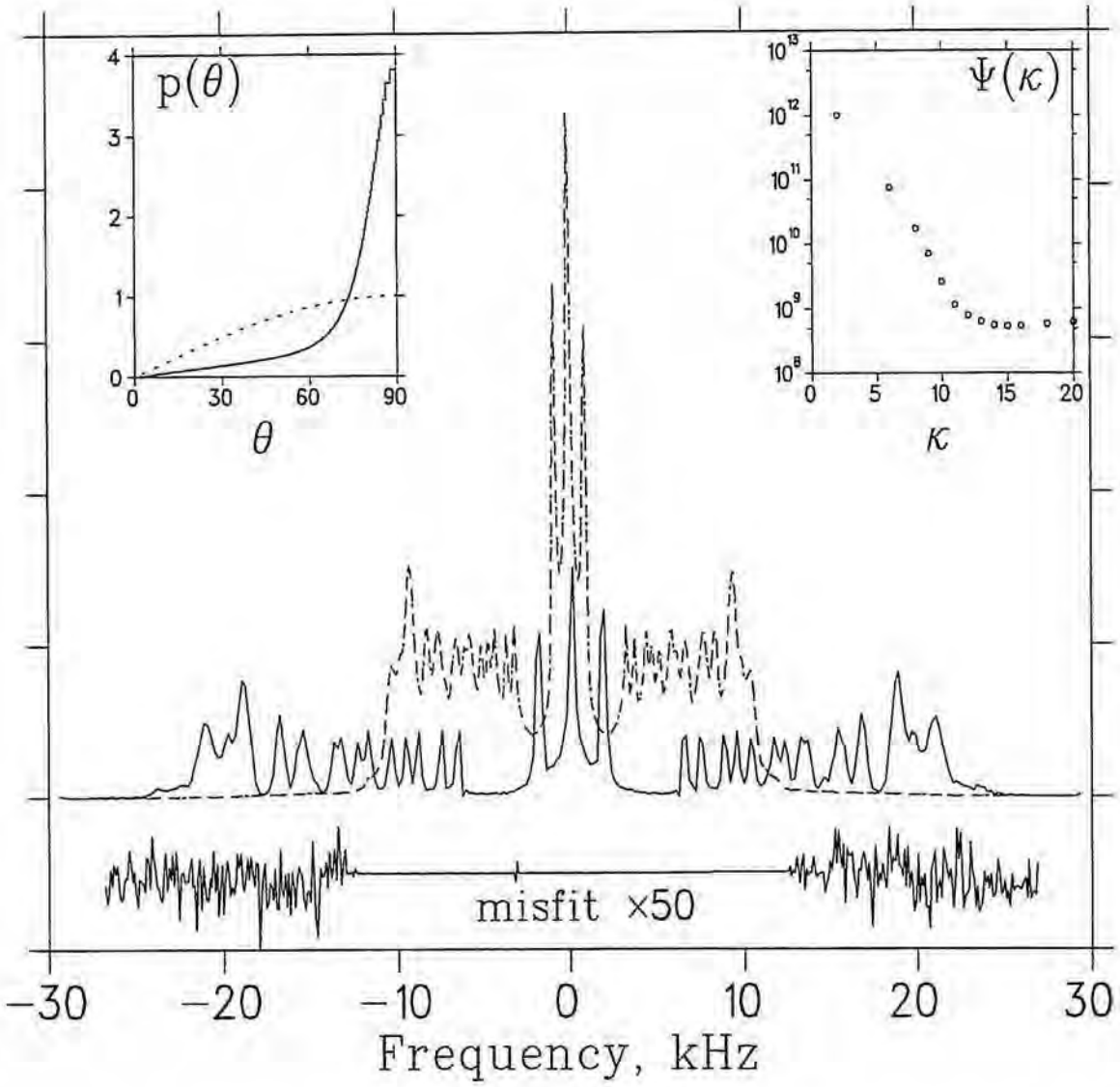
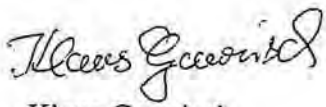


Figure 1. The ^2H NMR spectrum of a d_{54}-DMPC/DHPC (4.4:1, mol/mol) bicelle dispersion was recorded on a Bruker DMX300 widebore spectrometer, using a quadrupolar echo sequence [1]. Mixed-model dePakeing using the Tikhonov regularization technique [5] of the spectra yields virtual spectra of perfectly oriented bilayers with their bilayer normal parallel to the magnetic field (solid line). The dashed line above this spectrum represents the original experimental spectrum. Below the spectrum the 50-fold magnified deviation between experimental spectrum and theoretical fit is shown. The upper-left insert shows the angular distribution function of normals to bicelle bilayer patches with respect to the magnetic field, $p(\theta)$ (solid line). For comparison the distribution function of a powder (dotted line) is shown. Lipids in bilcelles oriented with their bilayer normal preferentially perpendicular (90°) to the magnetic field, but the distribution function is rather broad. The upper-right insert reports the quadratic deviation between experiment and fit as a function of elongation parameter, κ, of oriented particles assumed to be ellipsoids.

Data analysis of oriented samples, in particular those that have been oriented on glass slides, shows that quite often orientation is less than perfect. When lipid bilayers are oriented with the bilayer normal perpendicular to the magnetic field it takes surprisingly little preference in orientation to create the false impression that these systems are very well oriented. With a little bit of phase adjustment and baseline manipulation, one is easily fooled into seeing narrow peaks of a perfectly oriented domain. The use of broad band solid state NMR instruments and appropriate numerical methods reveals the true $p(\theta)$ distribution.

[1] Davis, J. H., Jeffrey, K. R, Bloom, M, Valic, M. I, Higgs, T. P. (1976) *Chem. Phys. Lett.* **42**, 390-394.
[2] Rance, M., Byrd, R. A., (1983) *J. Magn. Reson.* **52**, 221-240.
[3] Bloom, M., Davis, J. H., MacKay, A. L. (1981) *Chem. Phys. Lett.* **80**, 198-202.
[4] Sternin, E., Bloom, M., MacKay, A. L., (1983) *J. Magn. Reson* **55**, 274-282.
[5] Schäfer, H., Mädler, B., Sternin, E. (1998) *Biophys. J.* **74**, 1007-1014.
[6] Sternin, E., Nizza, D. T., Gawrisch, K., *submitted for publication.*

Sincerely yours,



Edward Sternin*

Klaus Gawrisch

Section of NMR
Laboratory of Membrane Biochemistry and Biophysics
Phone; (301) 594-3750; Fax: (301) 595-0035

*on sabbatical leave from Dept. of Physics, Brock University, St. Catharines, Canada

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References: (1) Roberts, J.K.; Smith, R.J. *J. Chromatogr., A* **1994**, *677*, 385. (2) Johnson, S. et al. *J. Chem. Soc., Perkin Trans. 1* **1994**, 1499. (3) Maple, S.R. et al. *Am. Pharm. Rev.* **1998**, *72*. (4) Smallcombe, S. H. et al. *J. Magn. Reson.* **1995**, *117*, 295.

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Molecular Dynamic Simulations, ^{6}Li Solid State NMR and Ultraphosphate Glasses

Our laboratory continues to use NMR to investigate the structure and dynamics in amorphous materials, including the local structure of ultraphosphate glasses. Changes in the alkali environment in these phosphate glasses as a function of modifier concentration has recently been probed using ^{6}Li and ^{23}Na solid state NMR.[1,2] Molecular dynamic (MD) simulations have also been performed in an attempt to gain additional insight into the variations of the local structure.[3] For example, Figure 1 shows a pictorial representation of the glass structure obtained from MD simulations.[3]

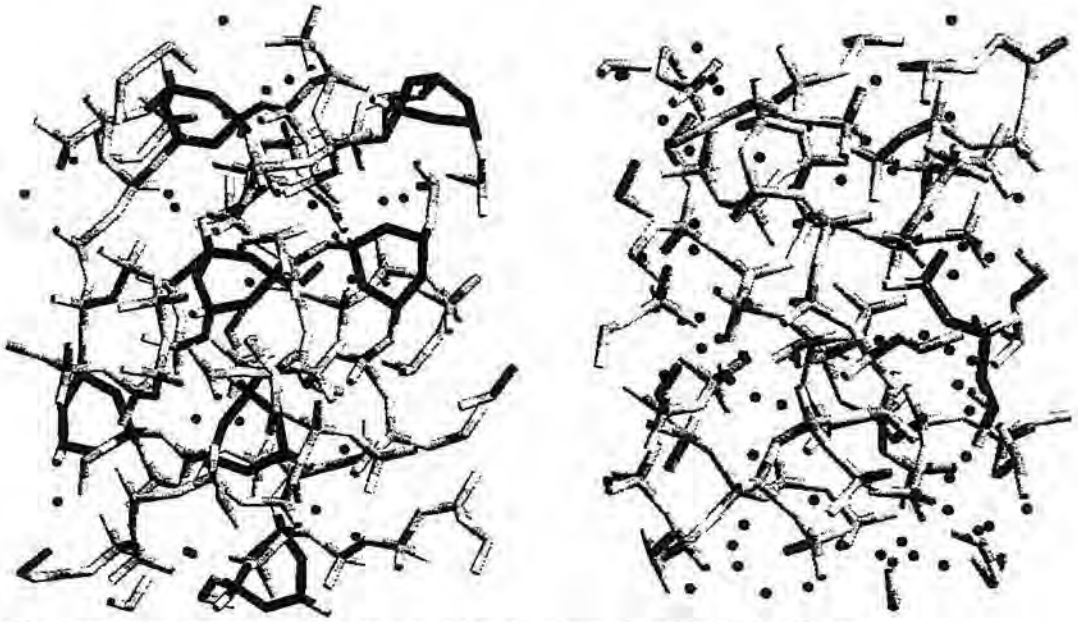

Figure 1: MD simulated structure for 20 and 50% Li_2O in $xLi_2O\bullet(100\text{-}x)P_2O_5$.

Interestingly, although there are distinct variations in the Li coordination number as well as the Li-O bond lengths in the MD simulations (with a minimum or maximum in these parameters near the 20% Li_2O concentration), a linear change in the ^{6}Li NMR chemical shift is observed between 5 and 50% Li_2O mole fraction. One would expect that such variations should be observable in the NMR chemical shift. In an attempt to understand this behavior we have performed empirical calculation of the ^{6}Li NMR chemical shift directly from the structures obtained in the MD simulations. It has been argued that the NMR chemical shift of alkali species can be related to a chemical shift parameter A, where A is defined as the summation of the shift contributions for all the oxygens located within the first (and possibly the second) coordination sphere around the cation.[1] For the present case of Li phosphate glasses, the chemical shift correlates directly to the bond valence of the coordinating oxygen. An empirical bond valence (s_{ij}) between oxygen i and the cation j can be calculated from the cation-oxygen bond length, r_{ij}, using

$$s_{ij} = \exp\left[\left(r_0 - r_{ij}\right)/B\right] \quad (1)$$

where r_0 is the empirically derived oxygen-cation bond length of unit valence, and B = 0.37 is a constant. The total valence of the *i*th oxygen (W_i) is simply the summation over all oxygen-cation bond valences s_{ij} for each of the *j* cations bonded to the oxygens, including both lithium and phosphorous cations:

$$W_i = \sum_j s_{ij} \quad (2)$$

The chemical shift parameter A is then be a summation of the oxygen shift contributions and assumes a $1/r_i^3$ dependence (where r_i is the Li-O bond distance):

$$A = \sum_i \frac{W_i}{r_i^3} \quad (3)$$

Recently we reported an empirical linear relation ship between the observed ^{6}Li chemical shift (δ_{CS})and the chemical shift parameter A for lithium phosphate system,[1]

$$\delta_{CS}(^6\mathrm{Li}) = +4.30A - 5.85 \quad (4)$$

Using Eqs. 1-4 the average ^{6}Li chemical shift was calculated from the MD structures as a function of Li_2O concentration, the results are shown in Figure 2. The observation of similar slope and similar chemical shifts is very promising. The offset can be controlled by variation of the cutoff distance used in the calculation of A. In this case, a 3.5 Å cutoff distance was utilized, the same as utilized in the development of Eqn. 4.[1] Even though there are minimum and maximum in the coordination number and the Li-O bond length, respectively; these effects are not observed in the resulting Li chemical shift. From Eqn. 3, if the average coordination number increases then the parameter A increases. From the MD simulations it appears that when this occurs, the Li-O bond distance also increases, thereby decreasing A. These two counteracting effects tend to cancel out any distinct minimum or maximum to produce a linear variation in chemical shift. Experimentally, this would suggest that subtle changes in the Li environment may be difficult to observe by Li NMR, since the chemical shift is a function of two different variables, and in some instances may produce changes that cancel out. A more detailed account of this work has been submitted and should appear soon.

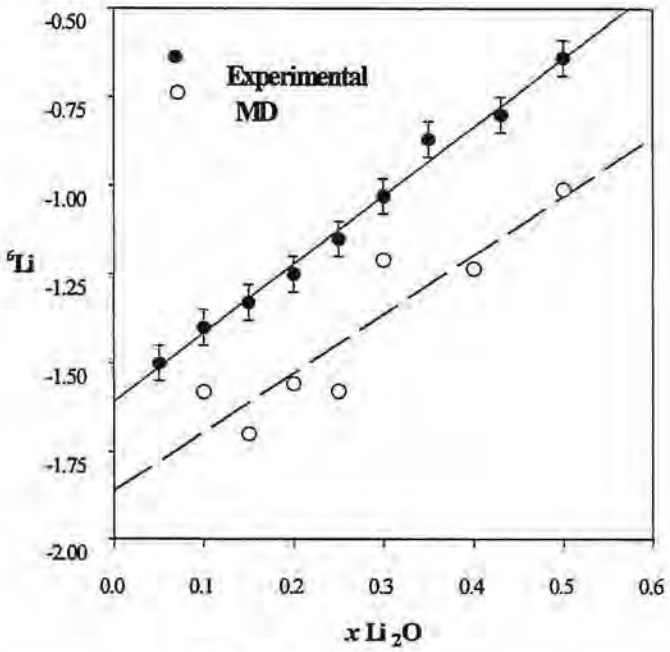

Figure 2: Variation of the ^{6}Li MAS NMR chemical shift, both experimental and predicted from the MD simulation, within a lithium ultraphosphate glass series as a function of Li_2O concentration.

Todd M. Alam

[1] T. M. Alam, S. Conzone, R. K. Brow, T. J. Boyle, J. Non-Cryst. Solids 258 (1999) 140-154.
[2] T. M. Alam, J. McLaughlin, C. C. Click, S. Conzone, R. K. Brow, T. J. Boyle, J. W. Zwanziger J. Phys. Chem. (2000) 104, 1464-1472.
[3] J. J. Liang, R. T. Cygan, T. M. Alam, J. Non-Cryst. Solids (2000) 263&264, 167-179.

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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

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

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

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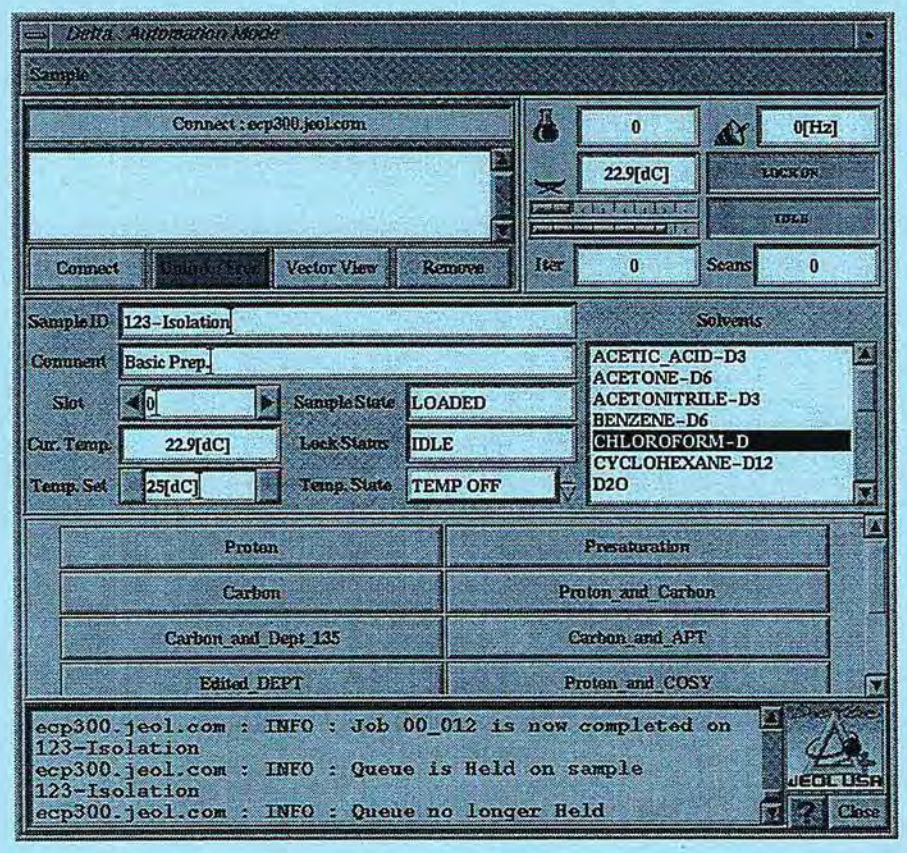

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