THE NMR NEWSLETTER

No. 506

November 2000

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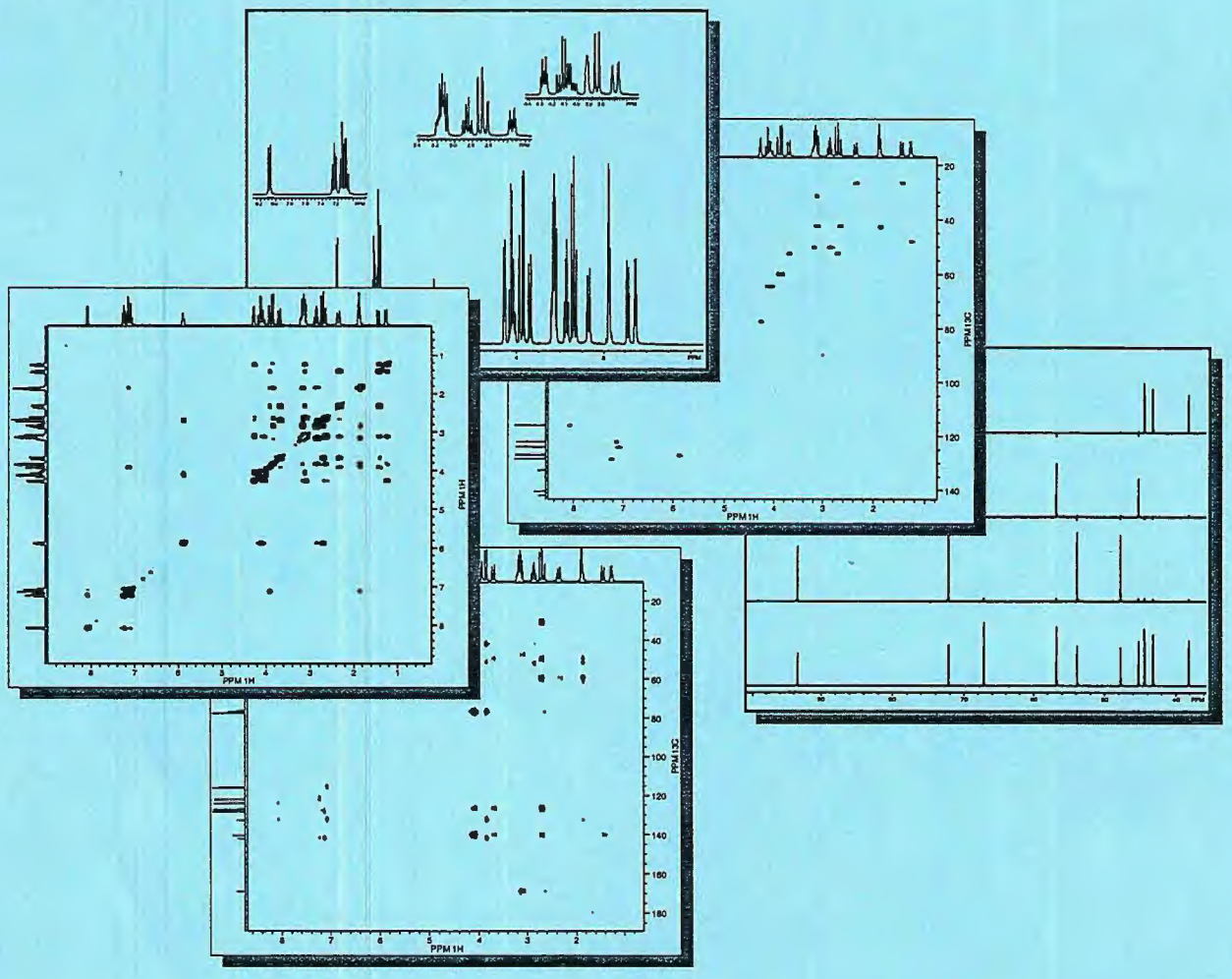

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FORTHCOMING NMR MEETINGS

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.

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, 29.

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.

ISMRM 9th Scientific Meeting and Exhibition, and ESMRMB 18th Annual Meeting and Exhibition, Joint Annual Meeting, **April 21-27, 2001**, 10th Annual Meeting of the Section for Magnetic Resonance Technologists, and 17th Annual Meeting of the British Association of MR Radiographers, **April 20-22, 2001** Glasgow, Scotland, UK; Contact: ISMRM, P.O. Box 45690, San Francisco, CA 94145-0690; http://www.ismrm.org

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THE NMR NEWSLETTER

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Help Needed Urgently.

The low number of technical contributions in several of the last issues of the Newsletter continues to be very worrisome. This issue (November) is an all-time low. The question is whether this is a temporary problem or whether this is a signal concerning the future of the Newsletter. We must think about the latter, very real possibility.

If you share my view that even in this day of the internet and e-mail, the Newsletter still has a useful role to play, please respond with a timely technical letter, even in advance of the Reminder or Ultimatum notices. Please consider this a most serious plea for technical contributions from those of you who "owe" The Newsletter a contribution. For those of you who are "paid up", or who, because of Sponsorship status are not required to make contributions, a voluntary contribution would be doubly welcome.

Without enough technical contributions there is no Newsletter. The loyalty of its advertisers and sponsors cannot make up for lack of content. Only a continuous, substantial increase in the number of contributions submitted for inclusion in the Newsletter can make it a viable publication worthy of subscription, advertising and sponsor support.

I would be very happy to receive any ideas you might have about how to revitalize the Newsletter, or whether the time is coming soon for the curtain to be rung down on this informal medium of communication among those doing NMR spectroscopy.

Barry Shapiro
001109

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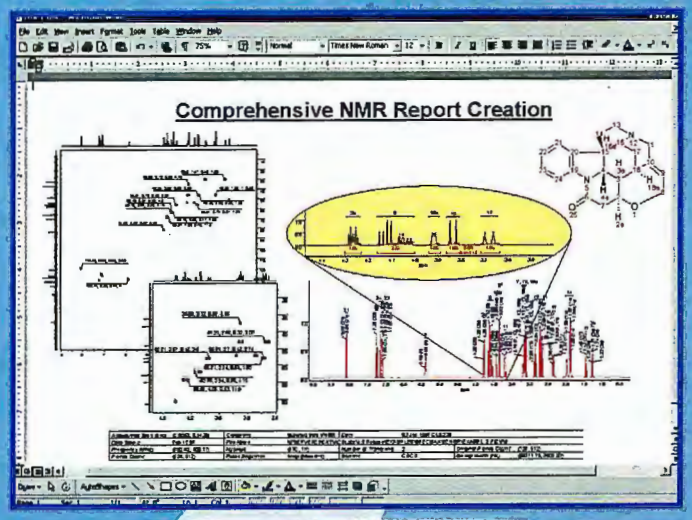

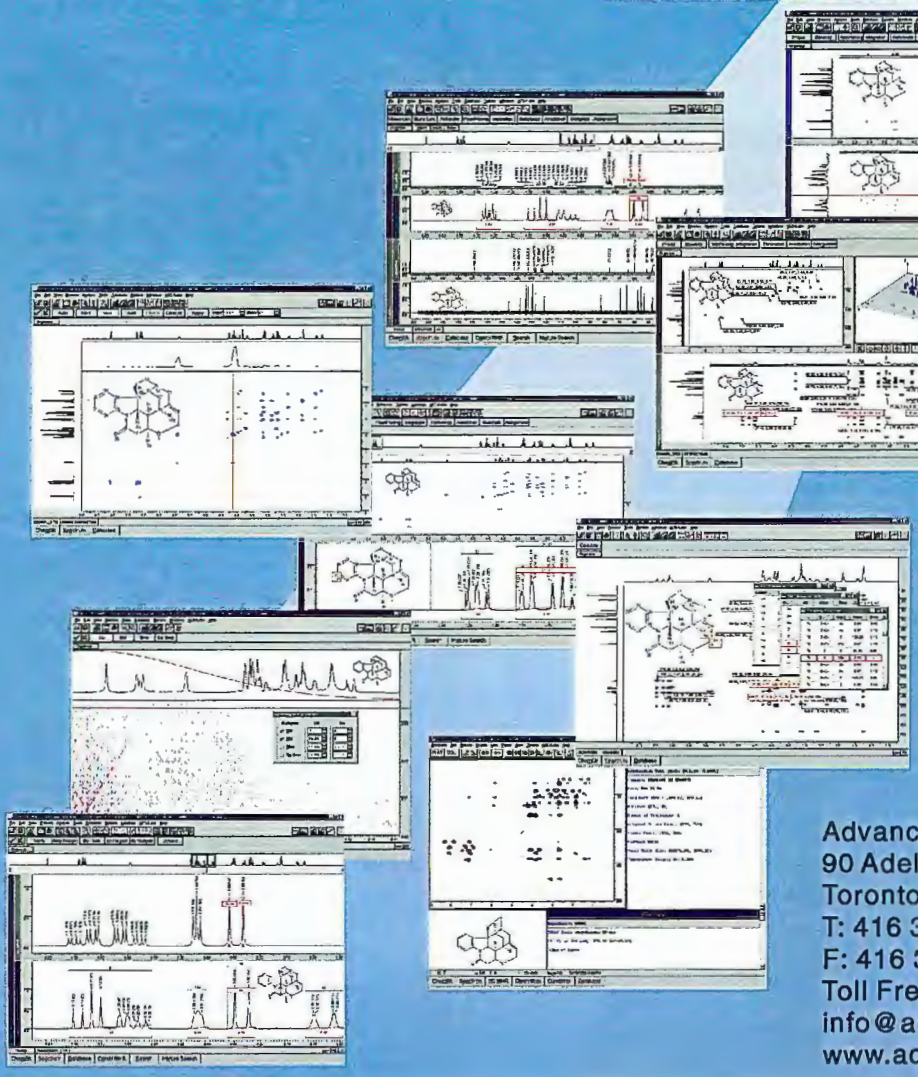

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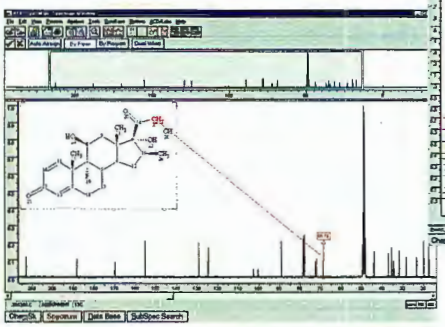

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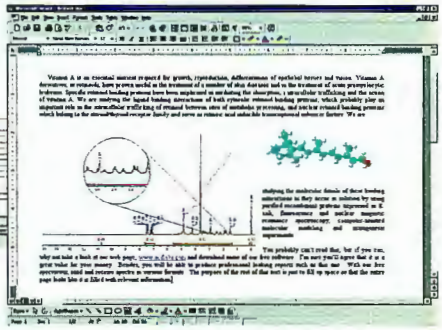

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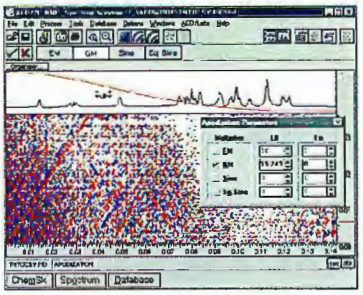

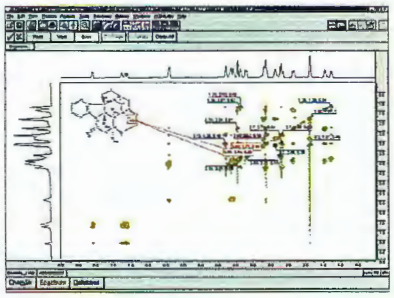

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September 30, 2000
(received 10/4/2000)

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

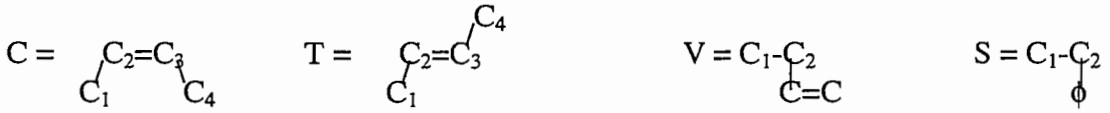
Dear Dr. Shapiro:

^{13}C NMR Analysis of Styrene/Butadiene Rubber

Styrene/butadiene rubber (SBR) is a commercially important polymer. Previously, this polymer has been studied by ^{13}C NMR, and assignments made by a number of workers[1-4]. Recently I have revisited the assignments and devised an analytical scheme for this polymer[5].

A typical ^{13}C NMR spectrum of SBR is shown in Figure 1. As a starting point, the assignments by Sato, et al[3] have been used, and adjustments in assignments[5] made to obtain the best fit between observed and calculated intensities. The assignment scheme is summarized in Table 1.

For computational purposes, SBR may be considered a tetrapolymer (i.e., 4-component copolymer), consisting of butadiene (which can polymerize in three ways: 1,4-cis, 1,4-trans, and 1,2 or vinyl) and styrene as the four components:



Earlier, the first-order Markovian model for tetrapolymerization has been devised and the probability expressions derived[6]. This model can be readily used for the SBR[5]. A detailed analysis of the ^{13}C NMR data can now be done via a computer-aided analytical approach. The results for the sample shown in Figure 1 are given in Table 1. The agreement between observed and calculated intensities is satisfactory (mean deviation < 1.0). From the reaction probabilities (P_{ij}), we can readily calculate copolymer composition, diad and triad sequences, as well as the products of the reactivity ratios. More detailed information can be found in ref. 5.

Yours very truly,

H. N. Cheng

References

1. A. R. Katritzky, D. E. Weiss, *JCS Perkin II*, 21 and 27 (1975).
2. F. Conti, M. Delfini, A. L. Segre, *Polymer*, **18**, 310 (1977), and references therein.
3. H. Sato, T. Ishikawa, K. Takebayashi, Y. Tanaka, *Macromolecules*, **22**, 1748 (1989).
4. S. Jiao, X. Chen, L. Hu, B. Yan, *Chin. J. Polym. Sci.*, **8**, 25 and 269 (1990).
5. H. N. Cheng, *Polymer News*, **25**, 114 (2000).
6. M. T. Roland, H. N. Cheng, *Macromolecules*, **24**, 2015 (1991).

Table 1. ^{13}C NMR spectral assignments of SBR and intensities (for the sample shown in Figure 1)

Peak No.	Shift	Assignment[a] C	T	V	S	Intensity[b] obsd	calc
1	43.6-45.8				$S_2¢$	14.6	16.2
				¢V_2¢	$S$$S_1$(0.6)		
2	42.4-43.5				¢S_2v	0.7	0.8
					$S$$S_1$(0.1)		
3	40.6-41.6		$s(r)$$T_1$	V_1(0.5)	$S$$S_1$(0.3)	4.2	4.2
				$V_2¢$	¢S_2s		
				¢$V_2$$	$S$$S_2$$		
4	39.8-40.6		¢sT_1	V_1(0.5)		5.1	6.1
5	38.9-39.5		$v(r)$$T_1$	$V_2$$		1.0	1.2
			$s(m)$$T_1$				
6	37.8-38.1		¢vT_1		¢$S_1$$(r)	5.6	5.8
7	37.2-37.7		$v(m)$$T_1$			1.0	0.4
8	35.4-37.0	$s(r)$$C_1$		¢$V_1$$(r)	¢S_1¢	7.9	7.9
					¢$S_1$$(m)		
9	34.4-34.8	¢sC_1		¢$V_1$$(m)		0.5	1.6
10	33.9-34.3	$s(m)$$C_1$		¢V_1¢		10.3	7.2
11	32.7-33.9	¢vC_1	¢T_1			104.2	104.2
		$v(r)$$C_1$	T_4¢				
12	30.4-32.3	$v(m)$$C_1$	T_4s			8.9	7.1
13	30.1		T_4v			6.0	6.0
14	27.4	C_4¢				28.4	28.4
		¢C_1					
15	25.2	C_4s				0.6	1.3
16	24.9	C_4v				0.7	1.7
					mean dev.	-	0.7

[a] For each sequence the unit being assigned is in the upper case, and the adjacent units in the lower case. The numerical subscripts correspond to the carbon numbers. ¢ = cis and trans 1,4; $ = styrene and vinyl; m and r = meso and racemic tacticity, respectively.

[b] Observed data have been fitted to the tetrapolymerization model[5]. Reaction probabilities are: $P_{ct} = 0.502$, $P_{cv} = 0.099$, $P_{cs} = 0.073$, $P_{tc} = 0.129$, $P_{tv} = 0.093$, $P_{ts} = 0.108$, $P_{vc} = 0.172$, $P_{vt} = 0.725$, $P_{vs} = 0.071$, $P_{sc} = 0.181$, $P_{st} = 0.634$, $P_{sv} = 0.077$, $P_m = 0.703$. From the model, the copolymer composition can be calculated: (cis)=0.172, (trans)=0.642, (vinyl)=0.087, (styrene)=0.099.

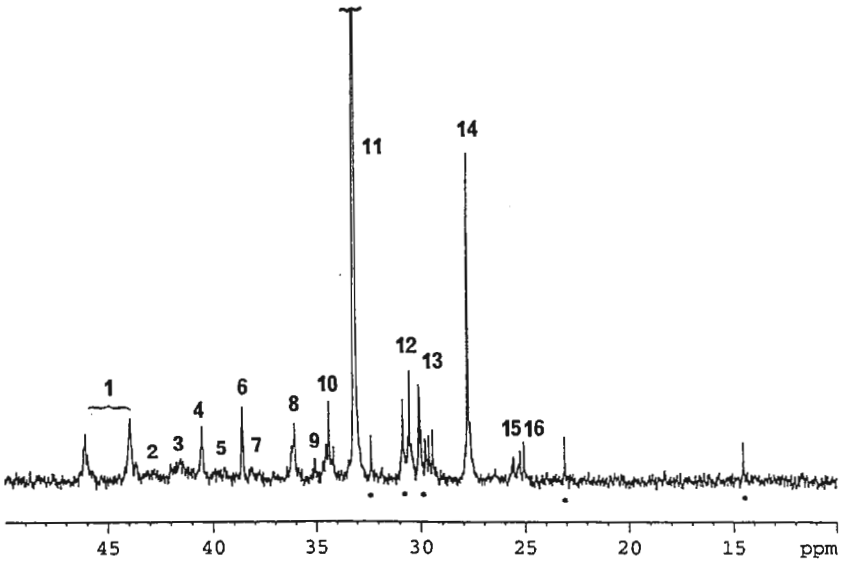


Figure 1. ^{13}C NMR spectrum of SBR at 75.469MHz, aliphatic region

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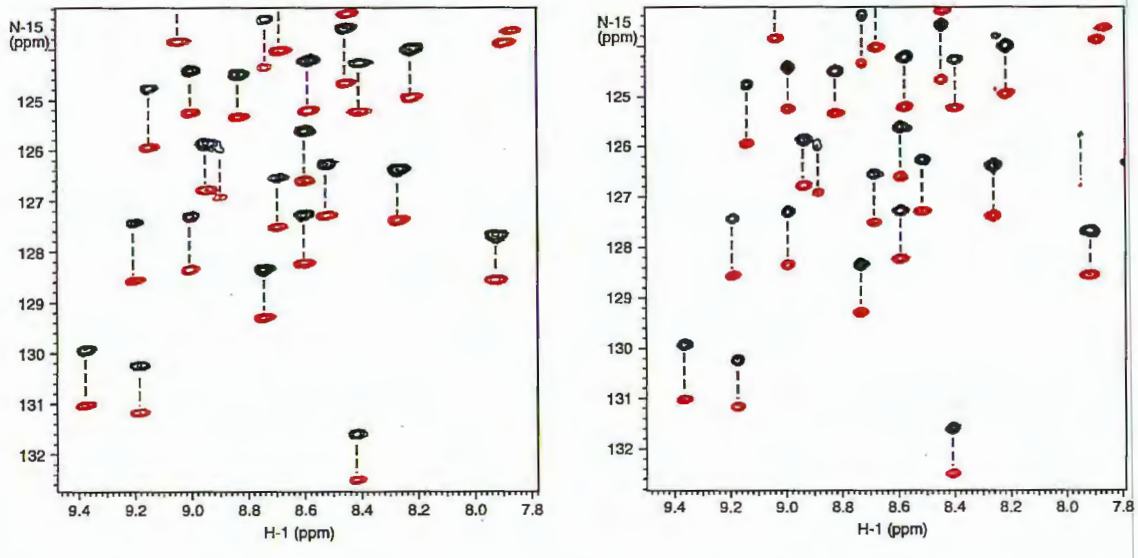
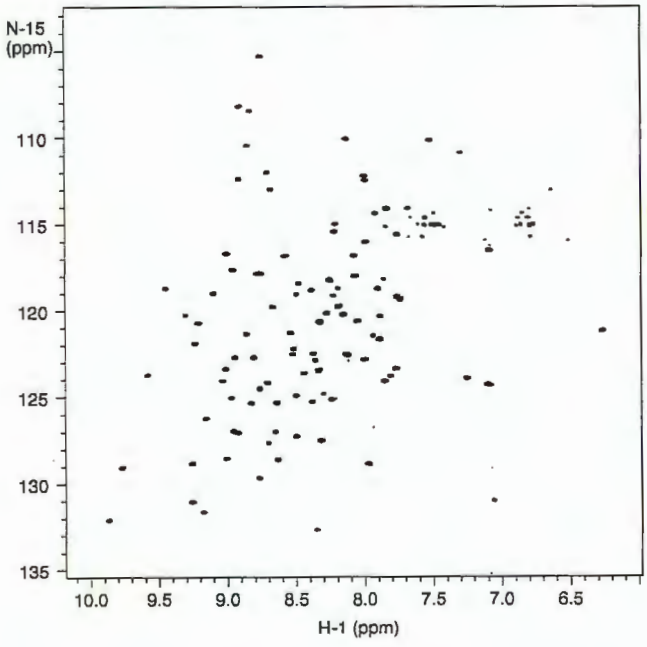


Measurement of residual dipolar N-H couplings at 900 MHz in partially oriented proteins with (right) and without the use of band-selective homonuclear decoupling.

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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ARGONNE NATIONAL LABORATORY
9700 South Cass Avenue, Argonne, Illinois 60439

October 20, 2000
(received 10/23/2000)

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

Re: **Probing Nanoscale Heterogeneity in Complex Carbonaceous Solids**

Dear Barry,

Exploring chemistry and materials' properties on the nanoscale presents significant challenges for characterizing new materials of our scientific age. While newly emerging physical characterization methods have been developed in recent years having potential to probe nanoscale chemical phenomena, such as neutron and x-ray scattering, diffuse-reflectance infrared and NMR spectroscopies, many challenges remain when studying extremely complex, heterogeneous substances.

Recent work has focused on identifying the nanoscale variation in chemistry of complex carbonaceous solids; in particular, our work has focused on a study of Argonne coals. Coals are characterized as being an "organic rock", comprised of different plant remains and extremely heterogeneous in nature. The question that we would like to address is: "At what length scale does its chemical heterogeneity persist?" Earlier experiments have provided some insight into this question. Previous magnetic resonance imaging (MRI) and scanning transmission x-ray microscopy (STXM) studies (*REB*) had provided detailed, two- and three-dimensional chemical pictographs showing the chemical variation of the organic phases of coal. While the images obtained were impressive, showing a definitive, sharp separation in chemistry along maceral (metamorphosed plant organic matter) boundaries, the maximum pixel image resolution achievable approached 50 nm at best, well above the nanoscale.

Solid-state spin-diffusion NMR techniques are available that allow an investigation of chemical heterogeneity on the nanoscale, 2-50 nm. These methods have been applied successfully to the study of phase-separated polymer blends whose components have a sufficient chemical distinctiveness. The concept behind the experiment is to set up a magnetization gradient, or non-equilibrium magnetization, between the chemically different (as distinguished by CRAMPS methods) protons that reside in different domains, and then to allow the magnetization to equilibrate, or to 'diffuse' between domains. From the time dependence of the diffusion process, one can estimate the size of the domains. In this manner, the scale of chemical heterogeneity of the system may be assessed. The challenge has been to apply the method to an extremely complex substance as coal.

Proton NMR experiments, both inversion-recovery and spin-diffusion types, employing CRAMPS methods were performed on a sample of the Utah Blind Canyon coal (APCS 6), which is largely a bimaceral coal composed of vitrinite with about 7% resinite. Pure maceral fractions were obtained by physical separation methods in order to measure proton NMR spectra and proton relaxation times on the pure maceral phases, which information is needed to perform the analyses. Inversion-recovery data for measuring the longitudinal proton relaxation time, T_1^H, is shown in Figure 1. The T_1^H decays of the resinite and vitrinite polarizations are distinct and the resinite lineshape is extracted from this data based on two characteristics, namely, a) that the aliphatic CRAMPS linewidth for resinite is significantly narrower than its vitrinite counterpart (this linewidth difference is also borne out on the basis of the CRAMPS spectrum of the pure resinite maceral) and b) that distinct T_1^H's of the vitrinite and resinite components can be identified. Thus, knowing the general aliphatic lineshape from the CRAMPS spectrum of the maceral, one can separate the resinite spectrum appropriate to this mixed sample from the inversion recovery data. The resinite spectrum is isolated when the broader aliphatic vitrinite component passes through the null condition. Note in Figure 1 that the resinite polarization does not decay exponentially for all time. Nevertheless, the fact that the T_1^H decays are separable and, in particular, that the resinite component, representing about 10% of the total protons, has an upward rather than a downward deviation from exponentiality, implies that most of the resinite domains exceed 100 nm in size.

We were also interested in the possibility that there might be some resinite domains which were significantly smaller and within spin diffusion (SD) distances. These smaller domains might not show up so well in the inversion recovery experiment which begins with no polarization gradients. Hence, we also took SD data for the Utah coal; results are shown in Figure 2. The CRAMPS preparation chosen produces a modestly negative aromatic and a more strongly positive aliphatic proton polarization. After about 4 ms of SD, equilibration of polarization within each phase leads to an average, slightly-positive initial proton polarization, P_o, for the aromatic-rich protons of vitrinite, along with a more strongly positive, mainly-aliphatic resinite proton polarization. In this manner, an initial gradient of proton polarization is established between the two maceral phases, thereby allowing us to use the CRAMPS lineshape to monitor any subsequent SD between those components.. Inversion of the initial gradient on alternate scans with corresponding addition and subtraction of signals dictates that T_1 effects will cause a decrease in the magnitude of the total signal. Changes in relative polarizations levels, associated with resinite and vitrinite domains, with diffusion time are shown in Figure 2 with P_o representing the polarization at Boltzmann equilibrium. Only data for > 4ms is included because interphase SD dominates in this time regime whereas intraphase equilibration is more important at earlier times. There is a slightly accelerated decay for the resinite polarization and a corresponding, hardly perceptible rise in the vitrinite polarization in the 4 ms to 12 ms range. (We have repeated this measurement with other initial conditions and the existence of a small, but real rise in the vitrinite polarization and a corresponding drop in the resinite polarization is reproduced, albeit the associated magnitudes are not so well reproduced.) Note that the vitrinite polarization levels are about an order of magnitude smaller than those of the resinite since the former are multiplied x7 in Figure 2. However, since the resinite protons are only about 10% of the total protons, SD should exhibit itself in the form of comparable vertical displacements in opposite directions for the two spin-diffusion data sets in Figure 2. The deduction from Figure 2 in terms of mixing is that a maximum of 20% of the resinite protons are in domains small enough (< 6nm) to undergo SD on the timescale of 12 ms or less; however, the majority of the protons of resinite are isolated from those of the vitrinite so that the resinite decay rate seen is indistinguishable from the resinite T_1^H observed in the inversion-recovery experiment. From the similarity of slope for the SD and inversion recovery data for resinite, it is clear that most of the resinite is in domains large enough (> 100 nm) so that SD on the 250 to 1000 ms timescale has insignificant influence.

The NMR spin-diffusion data suggest that there is little or no vitrinite/resinite heterogeneity at length scales between 6 and 100 nm. In this connection, we did background SD experiments on the pure vitrinite macerals in order to determine how long it took before the lineshape stabilizes after the imposition of an aromatic/aliphatic polarization gradient; that time is 4 ms. Hence, the polarization changes in the 4 to 12 ms range are not likely to be attributable to spin equilibration within the vitrinite phase.

The SD results are consistent with the previous images obtained by STXM and suggest that resinite domains are mainly large with clear phase separation, akin to domains found in slowly formed, thermodynamically incompatible polymer blends. It is again consistent with the size of the smallest resin bodies, which reflect the largeness of cavities of epithelial cells (50 μm) in which terpenes are produced and stored, and eventually secreted into even larger structures visible by the naked eye. That the remaining 20% of the resinite protons are within domains of 6 nm or less suggests that low molecular weight terpenoid materials percolated through the surrounding micropores of woody tissue soon after their secretion. Subsequent diagenesis led to their polymerization resulting in resins intimately dispersed within the Utah vitrinite. These findings are entirely in accord with the known paleobotany and geochemistry of resins in coal bearing formations and the unusual chemical nature (H/C ratio, reflectivity and fluorescence) of the vitrinite in the Utah coal.

Sincerely,

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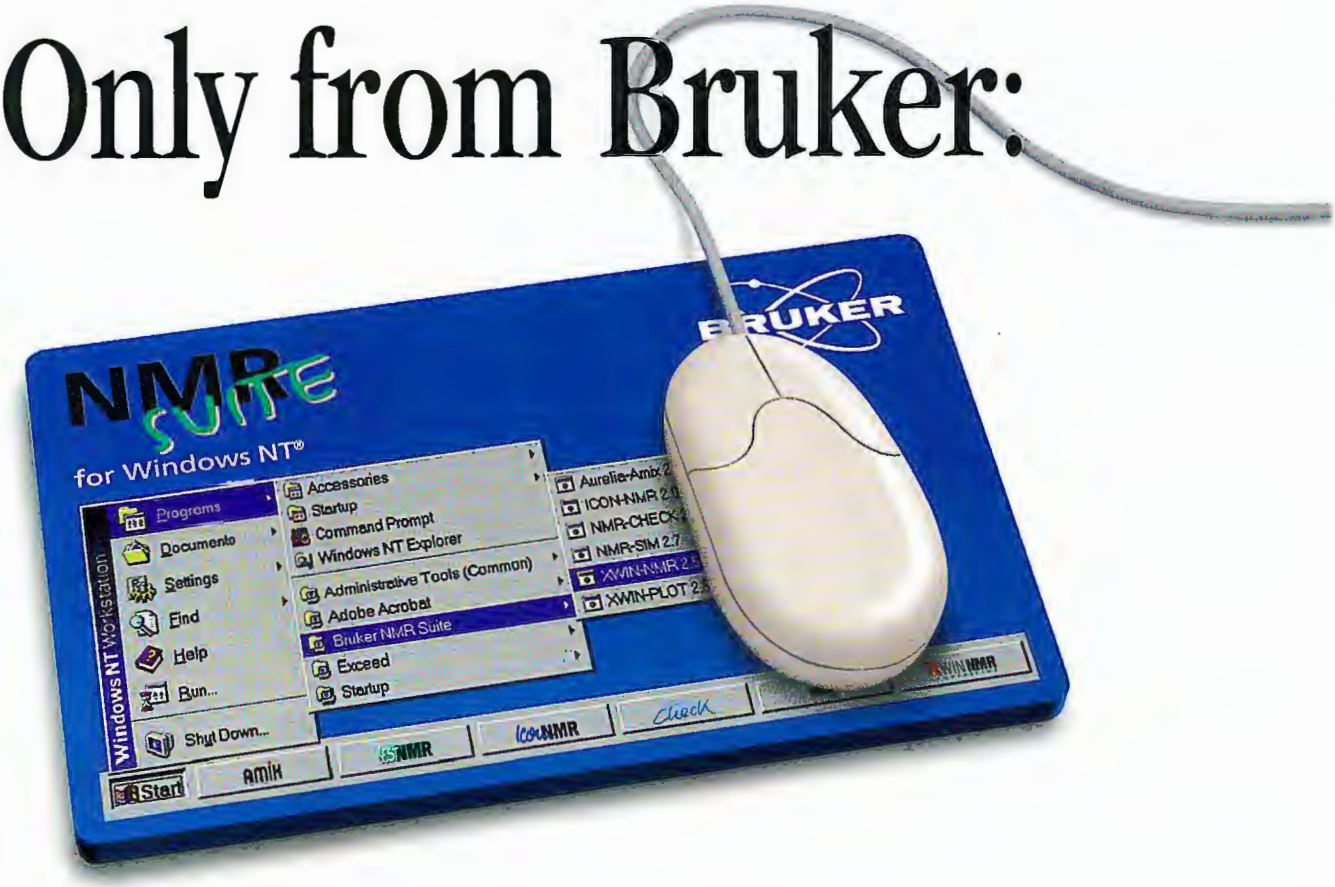

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Answers to some frequently asked questions about NMR Suite™ for Windows NT®:

Q: What exactly is NMR Suite for Windows NT?

A: NMR Suite consists of the following programs:

XWIN-NMR (spectrometer control and data processing)

XWIN-Plot (object oriented plot editor)

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

NMR-Sim (numerical simulation of NMR spectra)

NMR-Check (diagnostic software).

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

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

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

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

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

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

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

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

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

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

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

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

A: The following hardware is recommended:

- Pentium II/266 MHz
- 64 MB ram (128 MB recommended)
- 4 GB disk (SCSI recommended)
- PCI graphics card, 2MB, minimum 256 colors and 1024 x 768 pixel resolution
- 3.5" floppy, 1.44 MB
- CD ROM
- 3 button mouse for PS/2 port
- Keyboard
- 2 ETHERNET cards, 10/100 Mbit 3COM 3C905 PCI bulk
- Windows NT 4.0 workstation installed on NTFS file system, including service pack 3

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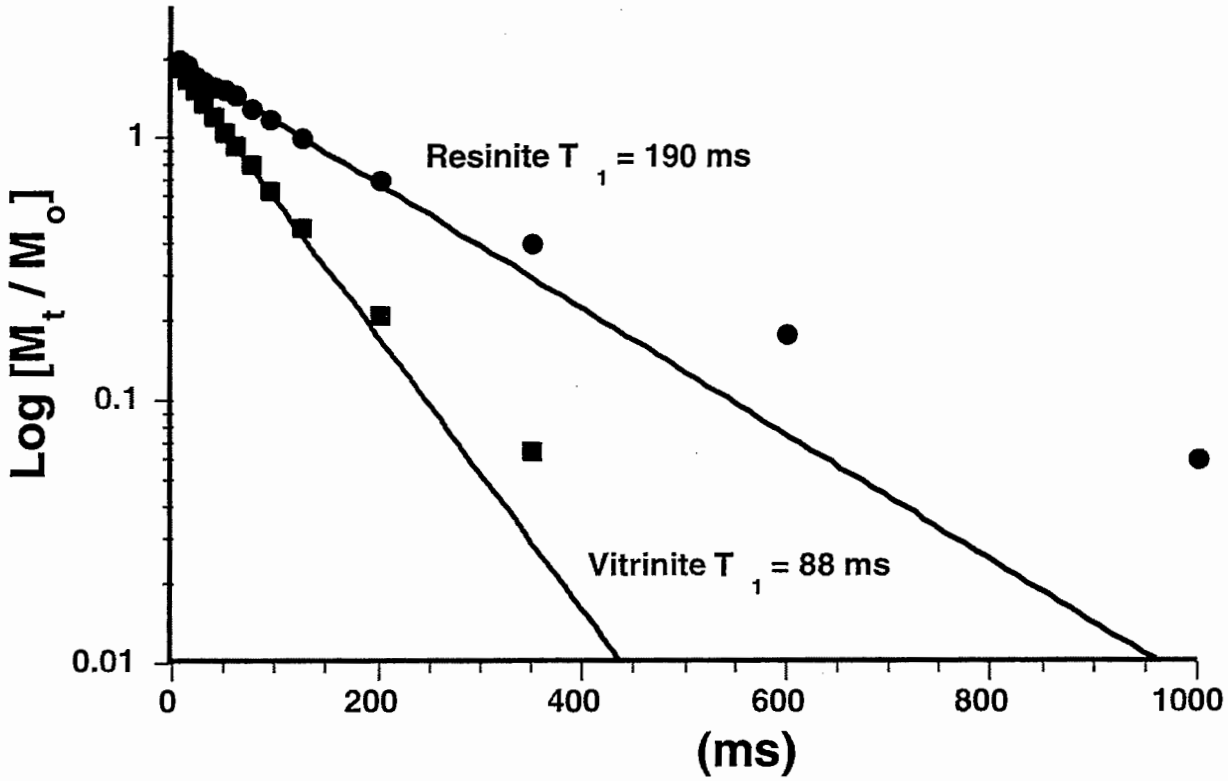

Figure 1. $T_1{}^H$ in Utah Coal

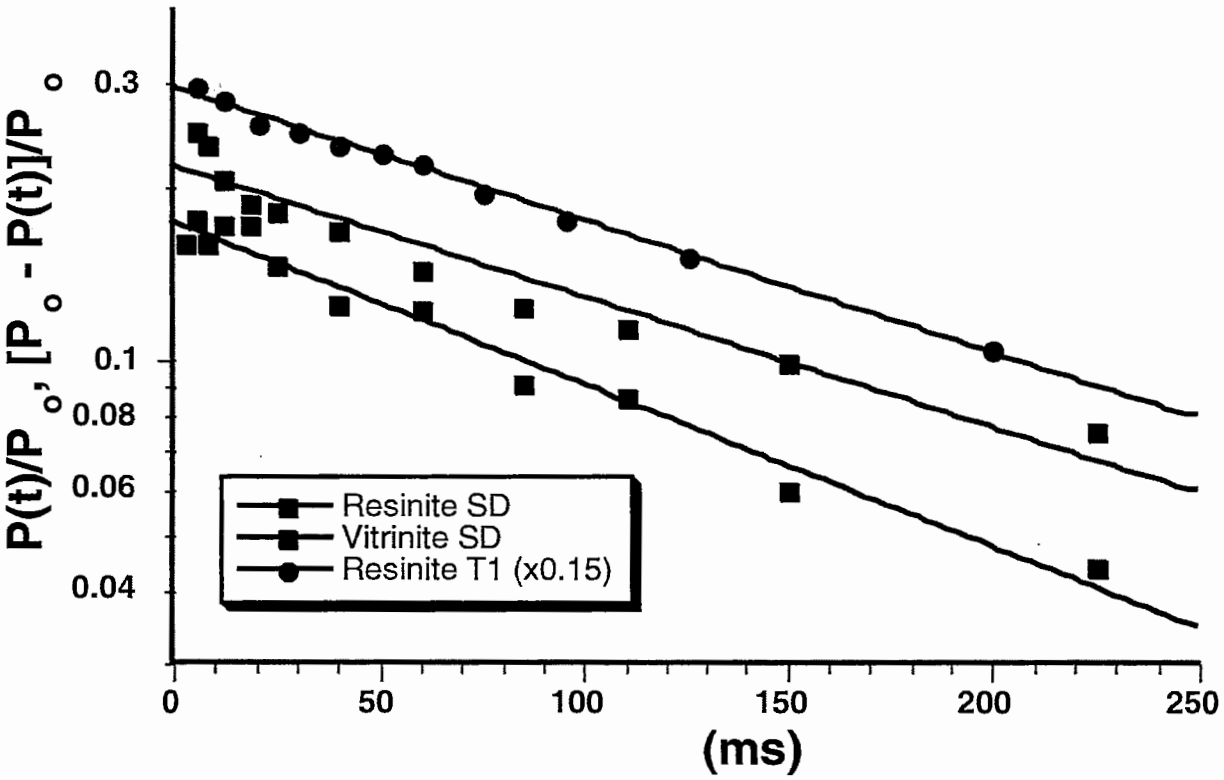

Figure 2. Spin Diffusion in Utah Coal

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DEPARTMENT OF CHEMISTRY

BERKELEY, CALIFORNIA 94720

March 28, 1974

Professor B. L. Shapiro
Department of Chemistry
Texas A & M University
College Station, Texas 77843

Dear Barry:

We have constructed a device which is quite useful for nmr in all types of systems. This has several novel features, e.g.:

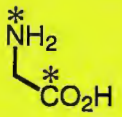
1. Produces a 11166 pulse 387τ sequence with independently adjustable widths and delays. This eliminates all shifts and couplings in any spectrum, including chemical shifts, Kondo shifts, scalar coupling, dipolar coupling, quadrupolar coupling, Knight Shifts, Day Shifts, and Suhl-Nakamura coupling, leaving a simple, clear, easily interpretable spectrum consisting of one sharp line. Analysis of this spectrum is performed automatically.
2. Eliminates automatically large extraneous peaks due to solvents. The solvent to be eliminated is selected by pushbutton out of a collection of 1704 entries. Solvent peaks may also be added.
3. Sensitivity is enhanced by transferring polarization from a large sample maintained in Saclay at 2°K. Polarization is running out so competition is keen and proposals must be submitted for permission to transfer.
4. Operates with 130 frequency synthesizers and a magnetic field calibrated according to the theorem $\omega_o = \gamma H_o$. The lock is external and operates on $^{5}Li - {}^{11}Li$; it is powerful and can be put on another campus.
5. The machine smiles during a spin tickling experiment.
6. Equipped with a 45 KW power transmitter. This is supplied with an answering service to reply to complaints from radio amateurs in Siberia.
7. Optical detection is optional. Sensitivity is so enormous that half a photon has been detected. The other half is being kept spin-locked under close guard of a hyperfine component.
8. The device operates in any frame including the rotating, pulsating, and randomly fluctuating frames.
9. Can do DEFT, TDFT, PRFT, WEFT, LEFT, GLBM, etc., by selecting a combination of four letters. If no experiment exist for a selected combination, the device will invent one and publish a preliminary communication.

This work was hardly supported.

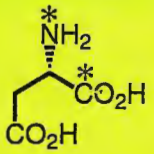
Best regards,

Alex Pines

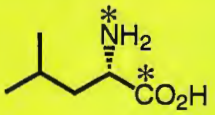
AP:crh



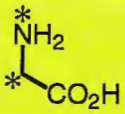
Glycine-1-^{13}C,^{15}N
>99 atom % ^{13}C
>99 atom % ^{15}N



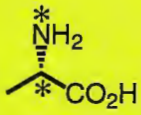
L-Aspartic Acid-1-^{13}C,^{15}N
>99 atom % ^{13}C
>99 atom % ^{15}N



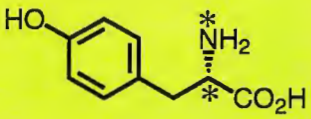
L-Leucine-1-^{13}C,^{15}N
>99 atom % ^{13}C
>99 atom % ^{15}N



Glycine-2-^{13}C,^{15}N
>99 atom % ^{13}C
>99 atom % ^{15}N



L-Alanine-2-^{13}C,^{15}N
>99 atom % ^{13}C
>99 atom % ^{15}N



L-4-Hydroxyphenylalanine-2-^{13}C,^{15}N
>99 atom % ^{13}C
>99 atom % ^{15}N

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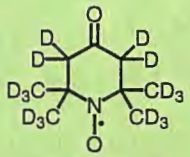
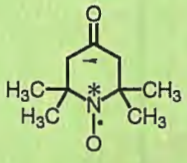


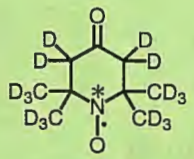
TEMPONE-d_{17}

>97 atom % D



TEMPONE-^{15}N

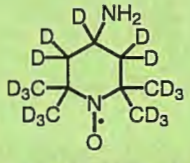
>99 atom % ^{15}N



TEMPONE-$^{15}N,d_{16}$

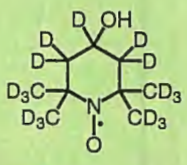
>98 atom % D

>99 atom % ^{15}N



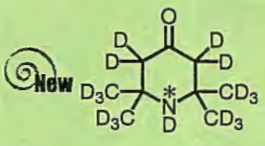
TEMPAMINE-d_{17}

>98 atom % D



TEMPOL-d_{17}

>97 atom % D



4-Oxo-2,2,6,6-tetramethylpiperidine-d_{17},1-^{15}N

(Spin Label Intermediate)

>97 atom % D

>99 atom % ^{15}N

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25 October 2000 (received 11/3/2000)

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

"Quantitative MR Imaging of Muscle Atrophy"

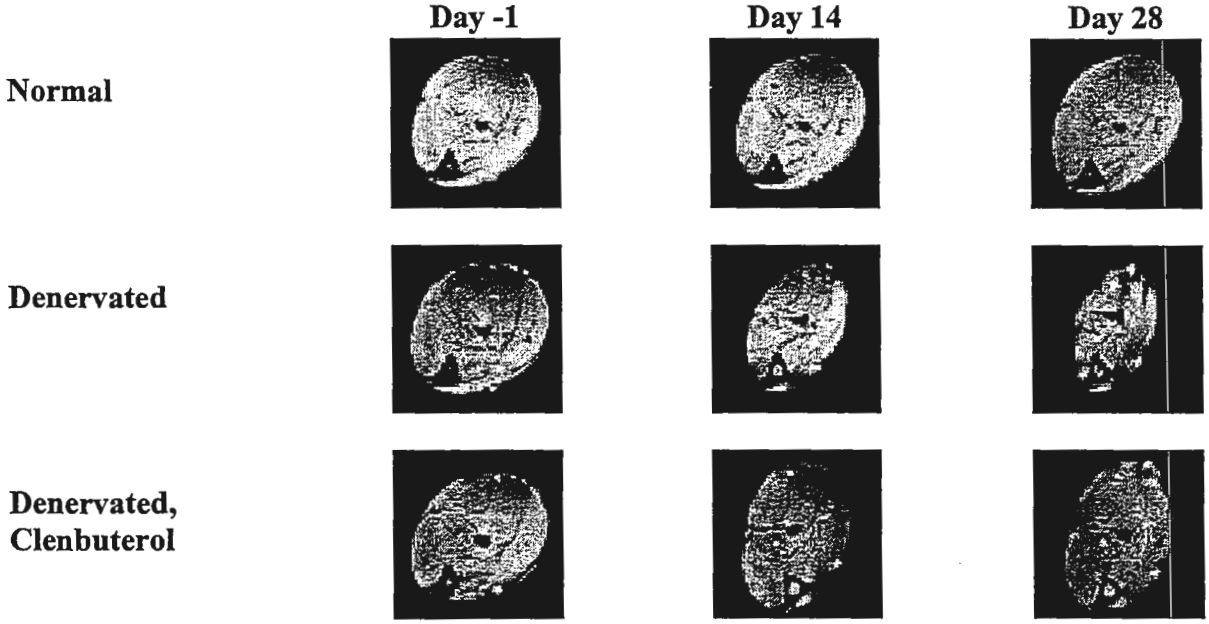
Dear Dr. Shapiro,

For several years now, we have been using MR imaging to quantify the changes in muscle morphometry, which accompany denervation-induced muscle atrophy in the rat. Resection of the sciatic nerve produces a reproducible atrophy of the muscles of the lower leg [1]. Prior to the use of MRI, quantification of muscle atrophy required sacrifice of the animal, dissection of the muscle, and determination of its wet weight. This precluded serial studies of a population of animals, and in fact, some time course studies were not being conducted due to limited resources. MRI opened the possibility of conducting these studies with far fewer animals and less personnel time.

I developed this approach for image acquisition. For optimal signal-to-noise and image localization, my group has been using a small loop coil constructed for us by Paul Morris (Morris Instruments, Ottawa). The coil is affixed to the underside of a plastic platform which has a 40 mm hole cut into it. A rat is anesthetized with 1-2% isoflurane in oxygen delivered via a small facemask, and one or both legs, depending on the study, are dropped through the hole for MRI. Because the muscle is fairly uniform, tissue contrast is not an issue and the only sources of susceptibility artifacts are the bone-muscle interfaces. Hence, we can use a very rapid steady-state gradient echo sequence with short echo times and obtain images of good quality with sufficient spatial resolution in just a few seconds. In fact, the time required to anesthetize and position a rat is longer than the time required to obtain the images!

The time required for image analysis is a major concern for these types of studies. Determination of muscle wet weight may appear to be time-consuming, but the talented people working here can do this very quickly. Hence the determination of an MRI-based correlate to wet weight, such as muscle cross-sectional area or muscle volume, has to be accomplished in a similar timeframe. Given the number of animals on a study, and the number of images acquired from each animal, data reduction can be a daunting task. To simplify this, then, my first approach relied on determination of muscle cross-sectional area (CSA) from a single slice, which could be reproducibly placed between the points at which the fibula attached to the tibia. In fact, based on a validation study, we were excited to find that single-slice muscle CSA correlated very well with total muscle wet weight. We then applied this single-slice methodology to the time-course study of the effects of a benchmark compound, clenbuterol, which is known to inhibit muscle atrophy. The figure demonstrates the effect. Quantitative estimates of clenbuterol's anti-atrophy effect, based on muscle wet weight, were known from earlier studies. Relative to those results, we found that CSA *overestimated* the effectiveness of clenbuterol. In subsequent work, we found that the effect of

clenbuterol is *anisotropic* over the leg, because different muscle groups respond differently to the drug. Hence, a single-slice approach could *not* provide an accurate assessment of the effect of clenbuterol. I then developed a multislice protocol to allow the determination of total muscle volume via the Cavalieri principle [2]. This was successful. Detemination of clenbuterol's protective effect from MRI-derived muscle volume agreed very well with that determined from wet weight.



The need for volume estimates presented one final problem. The multislice approach did not appreciably increase the total image acquisition time, but the image analysis time grew dramatically! As a result, I became quite proficient at outlining muscle borders "by hand" using the computer mouse, all the while developing carpal tunnel pain and drinking coffee to stay awake. This was not particularly fun and certainly not time-efficient, so I commissioned a member of P&G's corporate image analysis group, Mr. Bryan Towne, to write an image analysis application to do much of the work for me. Bryan was quite successful. His application finds the muscle borders semi-automatically, computes total muscle volume, and writes the results, along with data tracking information extracted from the image file, to an Excel spreadsheet, which can be sent directly to biostatistics.

So, in summary, we are well positioned to conduct additional studies of muscle atrophy using MRI. We have already applied this to studies other models of muscle atrophy and to studies of different clenbuterol dosing regimens, and we have just begun to translate the methodology for use with mice.

Sincerely,

Mike Cockman
cockman.md@pg.com

[1] Zeman RJ, Ludemann R, Etlinger JD: Clenbuterol, a Beta2-Agonist, Retards Muscle Atrophy in Denervated Muscles. *Am. J. Physiol.* 1987; 252:E152-E155.

[2] Elliott MA, Walter GA, Gulish H, Sadi AS, Lawson DD, Jaffe W, Insko EK, Leigh JS, Vandenborne K: Volumetric measurement of human calf muscle from magnetic resonance imaging. *MAGMA* 1997; 5:93-98.

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5 mm	541-PP-8	800	8 in	0.015"	0.00015"	0.00015"	28.33	+100	25.49
5 mm	535-PP-7	500	7 in	0.015"	0.0005"	0.00025"	15.25	+100	13.35
5 mm	535-PP-8	500	8 in	0.015"	0.0005"	0.00025"	16.75	+100	14.65
5 mm	528-PP-7	400	7 in	0.015"	0.0010"	0.0005"	10.85	+100	9.35
5 mm	528-PP-8	400	8 in	0.015"	0.0010"	0.0005"	11.95	+100	10.25
5 mm	507-PP-7	360	7 in	0.015"	0.0020"	0.0010"	7.15	+100	6.15
5 mm	507-PP-8	360	8 in	0.015"	0.0020"	0.0010"	8.00	+100	6.90
5 mm	506-PP-7	100	7 in	0.015"	0.0020"	0.0020"	5.65	+100	4.90
5 mm	506-PP-8	100	8 in	0.015"	0.0020"	0.0020"	6.55	+100	5.80
5 mm	506-IM-7	100	7 in	0.015"	0.0020"	0.0020"	3.85	+100	3.45
5 mm	506-IM-8	100	8 in	0.015"	0.0020"	0.0020"	4.05	+100	3.65
5 mm	WG-5MM-THRIFT-7*	60	7 in	0.015"	nominal	nominal	1.49	+100	1.30
5 mm	WG-5MM-THRIFT-8*	60	8 in	0.015"	nominal	nominal	1.75	+100	1.58
*N51 glass						(1 pk=100 ea)	1-4 pks	5-9 pks	10 pks
5 mm	HIP-7*	60	7 in	0.015"	nominal	nominal	$95.00	$90.00	$85.00
5 mm	HIP-8*	60	8 in	0.015"	nominal	nominal	105.00	100.00	95.00

Size	Product Number	MHz	Length	Wall Thickness	Concentricity	Camber	Each	Qty.	Bulk Each
3 mm	335-PP-7	500	7 in	0.015"	0.0005"	0.00025"	$11.75	+100	$10.00
3 mm	335-PP-8	500	8 in	0.015"	0.0005"	0.00025"	12.90	+100	10.90
3 mm	327-PP-7	400	7 in	0.012"	0.0010"	0.0010"	9.10	+100	7.80
3 mm	327-PP-8	400	8 in	0.012"	0.0010"	0.0010"	10.10	+100	8.60
3 mm	307-PP-7	360	7 in	0.012"	0.0020"	0.0010"	8.45	+100	7.20
3 mm	307-PP-8	360	8 in	0.012"	0.0020"	0.0010"	8.95	+100	7.70

Size	Product Number	MHz	Length	Wall Thickness	Concentricity	Camber	Each	Qty.	Bulk Each
10 mm	513-7PP-7	400	7 in	0.018"	0.0015"	0.0005"	$23.25	+25	$20.95
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5 mm	528-JY-8	400	8"	0.015"	0.0010"	0.0005"	75.70	+10	68.10
5 mm	507-JY-7	360	7"	0.015"	0.0020"	0.0010"	72.45	+10	65.25
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October 26, 2000
(received 10/26/2000)

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

Biopolymer Kuhn length and $T_{1\rho}$ of ^{23}Na

Dear Barry,

Sodium ions in aqueous biopolymer systems (and biological tissue) are hydrated and are dissolved in the water. They diffuse rapidly, about half as fast as in dilute aqueous solution. Also, they reside in two basically different types of environments: (a) the isotropic "liquid" host, and (b) the anisotropic interface at the "surface" of the relatively immobile macromolecules. The motions in (a) are rapid, similar to those in a bulk liquid. In (b) the motions are much slower than in (a) and the geometry of the local surroundings is anisotropic with respect to a director defined by the local macromolecular interface.

Water molecules and sodium ions exchange between the interface and the liquid. Translational diffusion in the liquid transports the ions to the interfaces of different macromolecules and the exchange brings the ions into these different interfaces. When the vicinal interfaces are locally ordered to some extent (i.e., are not randomly oriented), the diffusing ions retain a residual electrostatic field gradient (efg) from the nonaveraged-out anisotropy of the interfaces for a period of time (τ_a). This time is related to the range of local order through the self diffusion coefficient in the liquid. If τ_a is greater than the reciprocal of the residual quadrupole coupling constant due to the residual efg, quadrupole splitting appears in the NMR spectrum.

Expressed in different terms, a change in orientation of macromolecules encountered by a given diffusing sodium ion is accompanied by a change to a new local quadrupole frequency because it can exchange with a macromolecular interface of different orientation. The correlation time of this quadrupole frequency depends on the length of time that the diffusing ion experiences a given orientation of interface in B_0 before it encounters an interface with a greatly different orientation. This correlation time is related, through the diffusion coefficient, to the distance over which this orientational change occurs. Long-range non-uniformities can cause the residual efg to have several components with different correlation times.

These correlation times, together with the associated residual efg's, affect the NMR relaxation times of the ions; the transverse relaxation time and the spin-lattice relaxation time in the rotating frame ($T_{1\rho}$) are especially affected. Because the value of $T_{1\rho}$ depends on the value of ω_1, and because ω_1 can be

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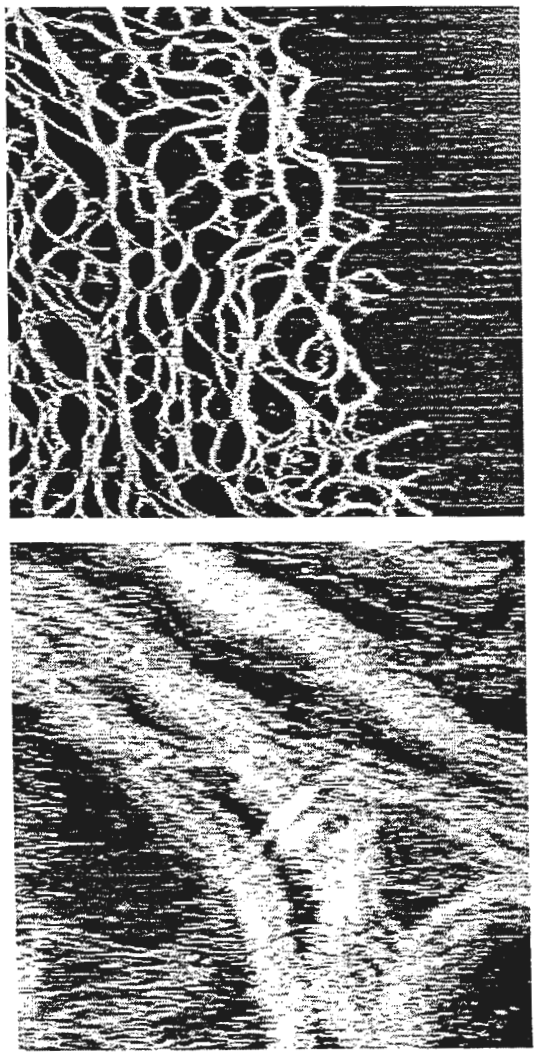
adjusted in the range 1 – 100 kHz, this measurement can be used to measure τ_a values in the range from 1 ms to 10 μs. Since diffusion coefficients in aqueous solutions are of the order of 10^{-5} cm^2/s, the $T_{1\rho}$ experiment may detect local order in the range 10^3 to 10^4 angstroms.

It is well known that the macromolecules in tissue are not randomly oriented and that residual quadrupole interaction has been reported for sodium in tissues such as cartilage, skeletal muscle, and brain. Also, residual quadrupole interaction that causes splitting in the ^{23}Na NMR spectrum has been reported for much simpler systems, such as aqueous biopolymers including κ-carageenan gels and xanthan gels. I have undertaken ^{23}Na NMR relaxation experiments on xanthan gels to determine just what such measurements can tell us about the arrangement of the macromolecules. Xanthan is a ubiquitous, thoroughly-characterized component of sauces that most people enjoy.

The two figures below are AFM images of xanthan molecules, taken from the Internet.



The upper image size is 1.5 micron by 1.5 micron, and the lower image size is 0.2 by 0.2 micron. The lower image shows several "threads" composed of many helical macromolecules.

Xanthan is an intrinsically stiff polyelectrolyte with persistence length 1000 – 1200 angstroms and a Kuhn length (distance along the macromolecular backbone before which the director changes drastically in orientation) of approximately 2000 angstroms. The upper image shows a definite tendency of the

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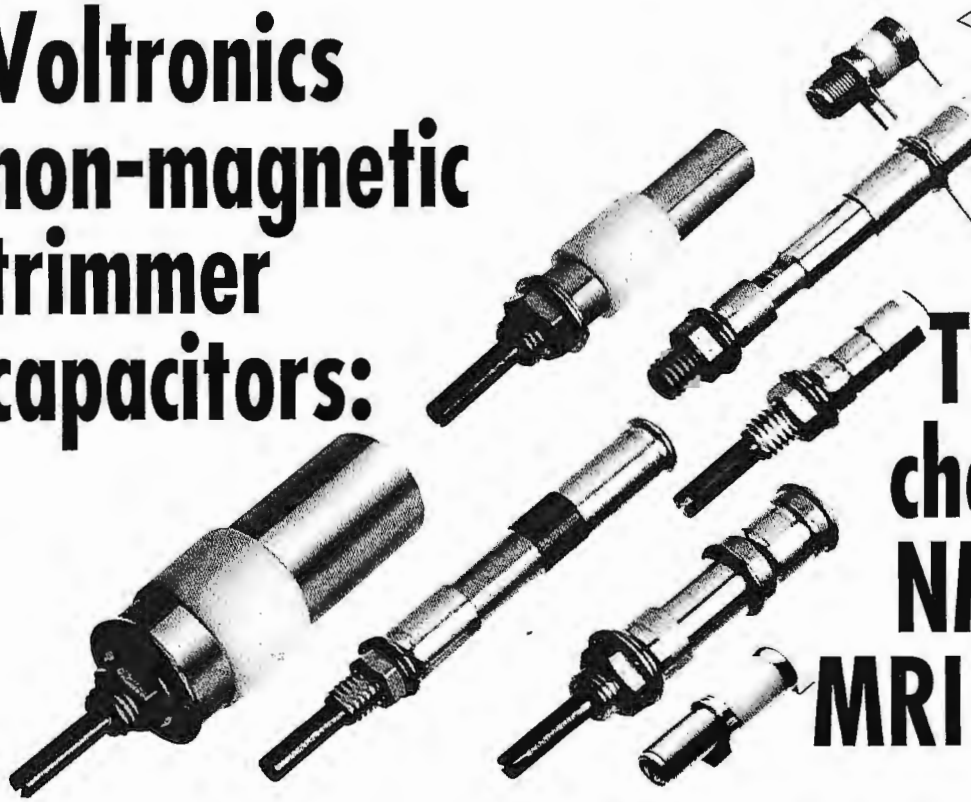

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macromolecular chains to lie along the vertical direction. Also, visual inspection shows that both images are consistent with the above persistence and Kuhn lengths.

The ^{23}Na NMR spectrum of an aqueous 20% xanthan sample is a powder pattern that is characterized by a residual quadrupole coupling constant (QCC) of 5376 Hz. $T_{1\rho}$ measurements yield a correlation time of 36.3 μs. The distance that a sodium ion diffuses in any given direction during this time is 2320 angstroms, which is comparable to the reported Kuhn length. This level of agreement is gratifying.

The above correlation time is sufficiently short so as to "wash out" the powder pattern spectrum from the QCC of 5376 Hz. Use of the Ostroff-Waugh multiple-pulse sequence results in another, much longer, correlation time, 1.3 ms. This is interpreted as a time dependence of the quadrupole frequency, from the above QCC, that arises from diffusional migration of the sodium ions among different orientations of the "crystallites" that contribute to the powder pattern spectrum. This value of correlation time corresponds to the occupation time for a diffusing ion to remain in a sphere of 3.56×10^{-4} cm. This distance corresponds to the diameter of a crystallite, or ordered domain of macromolecules, of 18 Kuhn lengths.

Conventional T_1 and T_2 measurements at two different B_0 values yield a short correlation time of 1 ns. Of course, with each correlation time there is an associated value of QCC.

Also, solid echo and multiple quantum filter measurements were made. The results of simulations made with the above correlation times and QCC values agree very well with these measurements.

A long paper that includes the above experiments and describes them in excruciating detail has been submitted to the **Journal of Magnetic Resonance**.

Sincerely,

Don

Donald E. Woessner, Ph.D.
Adjunct Assistant Professor of Radiology
donald.woessner@utsouthwestern.edu

THE NMR NEWSLETTER

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The notice below was intended to expedite the receipt of the technical contributions to the Newsletter. However, our local internet service provider and cable modem company have been having problems, and we have been without e-mail or internet access for extended periods many times during the past few months. Please bear with us until this situation is resolved. Until then, it might be well to back up your submissions by sending a duplicate copy by 'snail-mail'.

'New' Way to Submit Technical Contributions.

22 August 2000; also 7 September 2000

As a step in modernizing the Newsletter, we are now asking that, when possible, technical contributions are submitted as **attachments to e-mail messages**. The attachments should be in WORD7 or WORD2000 formats, as **.doc files**. We will attempt to accommodate other formats as well, but please check with us in advance. (If the WORD or other pre-approved format is not available, we will reluctantly accept plain ASCI II text files or contributions embedded in standard e-mail, in which case formatting will be at our discretion.)

Graphics (including letterheads), drawings, spectra and your signatures should be embedded in the WORD files or included as separate file attachments when possible. If letterhead is not available as a graphic file, we will attempt to create a reasonable facsimile. To that end, please provide a faxed copy of the letterhead.

In any event, please include your preferred **e-mail address** below the signature line in your contributions.

Many thanks for your cooperation.

Barry Shapiro.
shapiro@nmrnewsletter.com

Forthcoming NMR Meetings, continued from page 1:

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.

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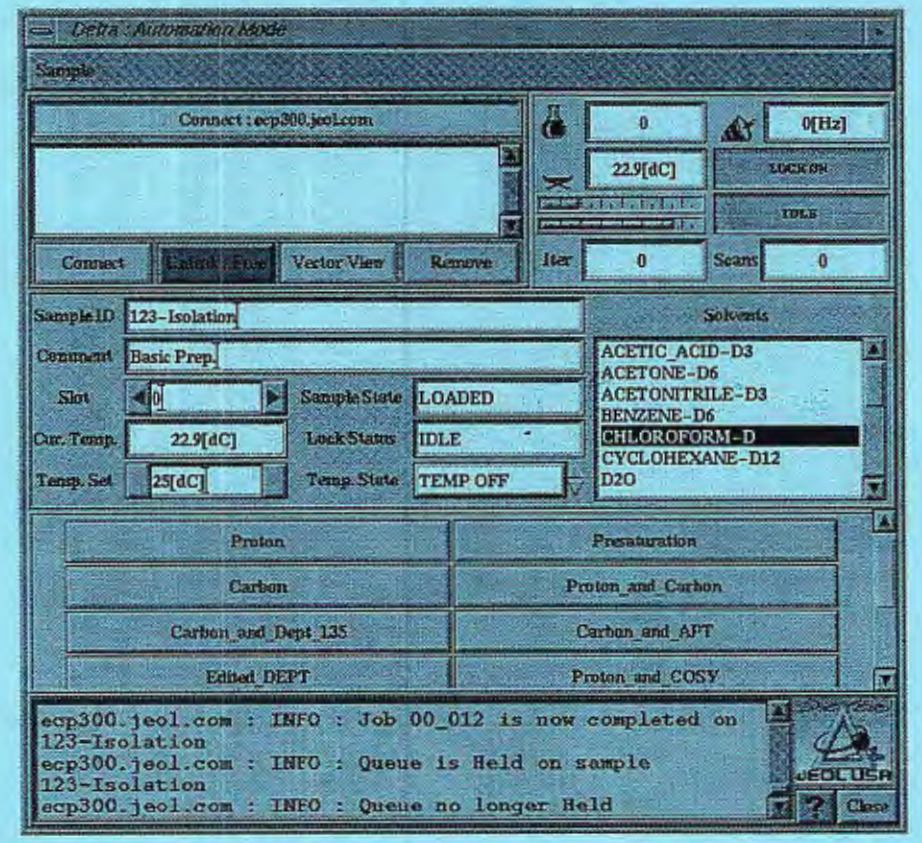

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