THE NMR NEWSLETTER

No. 457

October 1996

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	Frequency Range	Resolution	Switching Time[1]	Phase-Continuous Switching[2]	Rack-Mount Cabinet Dim.[3]	Remote-Control Interface	Price Example[4]
PTS 040	.1-40 MHz	optional .1 Hz to 100 KHz	1-20μs	optional	5¼"H×19"W	BCD (std) or GPIB (opt)	$5,330.00 (1 Hz resol., OCXO freq. std.)
PTS 120	90-120 MHz	optional .1 Hz to 100 KHz	1-20μs	optional	5¼"H×19"W	BCD (std) or GPIB (opt)	$5,330.00 (1 Hz resol., OCXO freq. std.)
PTS 160	.1-160 MHz	optional .1 Hz to 100 KHz	1-20μs	optional	5¼"H×19"W	BCD (std) or GPIB (opt)	$6,495.00 (1 Hz resol., OCXO freq. std.)
PTS 250	1-250 MHz	optional .1 Hz to 100 KHz	1-20μs	optional	5¼"H×19"W	BCD (std) or GPIB (opt)	$7,440.00 (1 Hz resol., OCXO freq. std.)
Type 1 **PTS 310** Type 2	.1-310 MHz	1 Hz	1-20μs	standard	3½"H×19"W	BCD (std) or GPIB (opt)	1 Hz resol., OCXO: $6,425.00 1 Hz resol., OCXO: $5,850.00
PTS 500	1-500 MHz	optional .1 Hz to 100 KHz	1-20μs	optional	5¼"H×19"W	BCD (std) or GPIB (opt)	$8,720.00 (1 Hz resol., OCXO freq. std.)
PTS 620	1-620 MHz	optional .1 Hz to 100 KHz	1-20μs	optional	5¼"H×19"W	BCD (std) or GPIB (opt)	$9,625.00 (1 Hz resol., OCXO freq. std.)
PTS 1000	0.1-1000 MHz	optional .1 Hz to 100 KHz	5-10μs	optional	5¼"H×19"W	BCD (std) or GPIB (opt)	$11,830.00 (1 Hz resol., OCXO freq. std.)
PTS 3200	1-3200 MHz	1 Hz	1-20μs	optional	5¼"H×19"W	BCD (std) or GPIB (opt)	$14,850.00 (1 Hz resol., OCXO freq. std.)
PTS x10	user specified 10 MHz decade	1 Hz	1-5μs	standard	3½"H×19"W	BCD (std) or GPIB (opt)	$3,000.00 (1 Hz resol., OCXO freq. std.)
PTS D310	two channels .1-310 MHz	.1 Hz	1-20μs	standard	5¼"H×19"W	BCD (std) or GPIB (opt)	$8,560.00 (.1 Hz resol., OCXO freq. std.)
PTS D620	two channels 1-620 MHz	.1 Hz/.2 Hz	1-20 μs	standard	5¼"H×19"W	BCD (std) or GPIB (opt)	$13,240.00 (.1 Hz/.2 Hz resol., OCXO freq. std.)

1 Switching Time is dependent on digit (decade) switched; see detailed instrument specifications.

2 For applicable digits, see detailed instrument specifications.

3 Bench cabinets are 17" wide.

4 Prices are U.S. only and include Manual and Remote (BCD) Control; PTS 3200 Digital Front Panel.

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FORTHCOMING NMR MEETINGS

38th ENC (Experimental NMR Conference), Orlando, FL, **March 23 - 27, 1997**; Contact: ENC, 1201 Don Diego Avenue, Santa Fe, NM 87505; (505) 989-4573; Fax: (505) 989-1073.

International Society for Magnetic Resonance in Medicine, Fifth Scientific Meeting and Exhibition, Vancouver, BC, Canada, **April 12-18, 1997**; Contact: ISMRM, 2118 Milvia St., Suite 201, Berkeley, CA 94704, USA; (510) 841-1899; Fax (510) 841-2340; Email: info@ismrm.org.

Symposium on NMR Spectroscopy of Synthetic Macromolcules, ACS National Meeting, San Francisco, **April 13-17,** 1997; Contact: H. N. Cheng or English, A. D. See Newsletter 456, 20.

4th International Conference on Magnetic Resonance Microscopy "Heidelberg Conference in Albuquerque", **Sept. 21-25, 1997**: Contact: E. Fukushima, The Lovelace Institutes, 2425 Ridgecrest Drive SE, Albuquerque, NM 87108-5127; (505) 262-7155; Fax: (505) 262-7043. See Newsletter 449, 37.

Additional listings of meetings, etc., are invited.

University of Illinois
at Urbana-Champaign

School of Chemical Sciences
600 South Mathews Avenue
Urbana, IL 61801

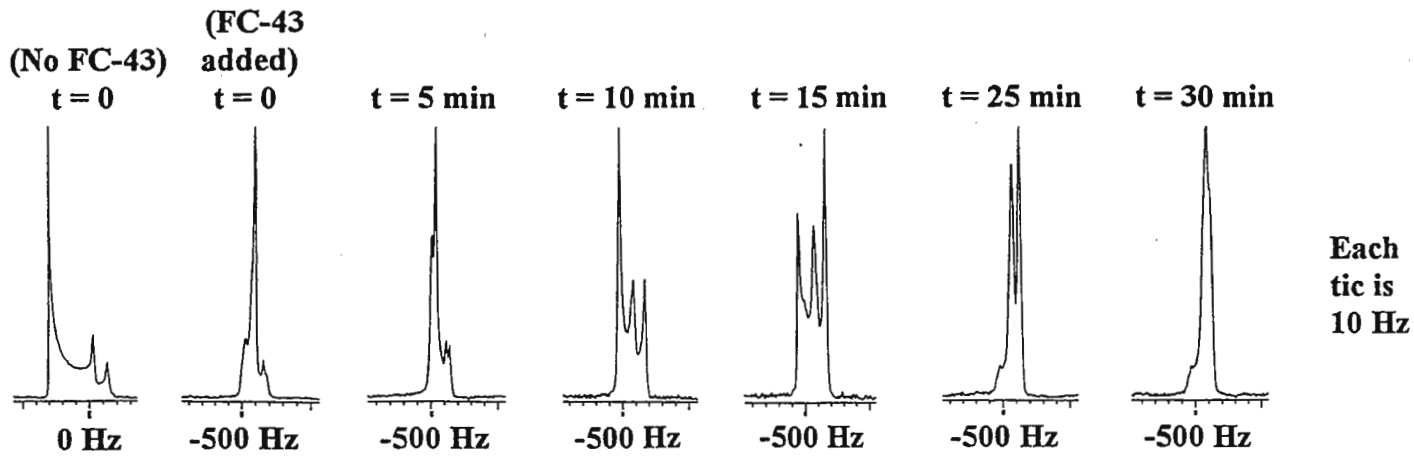
Magnetic Susceptibility Matching Fluid in Microcoil NMR

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

September 25, 1996
(received 9/26/96)

Dear Dr. Shapiro and Readers,

In our studies of NMR microcoils, we employ a perfluorinated organic fluid as a magnetic susceptibility matching medium [1]. Our microcoils are usually composed of 50-μm Cu wire and are wound around fused silica capillary of about 350 μm outer diameter. The coil and capillary region are surrounded by the fluid which greatly reduces linewidth, significantly improves line shape, and enhances S/N about 4-fold due to improved field homogeneity in the sample region. The volume magnetic susceptibility of 3M's Fluorinert FC-43® (χ_{FC}) is 7% smaller than χ_{Water}, 15% smaller than χ_{Cu}, and 40% less than χ_{Silica}. The wire is coated with polyurethane, the capillary with polyimide, and the coil is held in place by cyanoacrylate adhesive. Since these six substances are present in the coil region, χ_{FC} is probably not an optimized magnetic susceptibility match. We have observed that when FC-43 is first applied to the coil region, the proton NMR signal (from 10% H_2O/D_2O) takes as long as three hours to fully stabilize. Shown below are several spectra from the first 30 min of equilibration. All the shims are set to zero and the chemical shift axis is scaled identically in all spectra. The shift in peak location upon application of FC-43 is about -540 Hz. Once the fluid is applied, the probe is reinserted into the 300 MHz magnet and not moved again. The fluid obviously alters the magnetic environment of the sample perhaps by diffusing into the adhesive, into the wire or capillary coatings, into any interstitial spaces between the coil components, or by diffusion of oxygen into or out of the coil region. If the FC-43 is removed and allowed to thoroughly evaporate from the coil, the phenomenon is repeatable.



Please credit this contribution to the account of Dr. Vera Mainz at the Molecular Spectroscopy Lab at the University of Illinois School of Chemical Sciences.

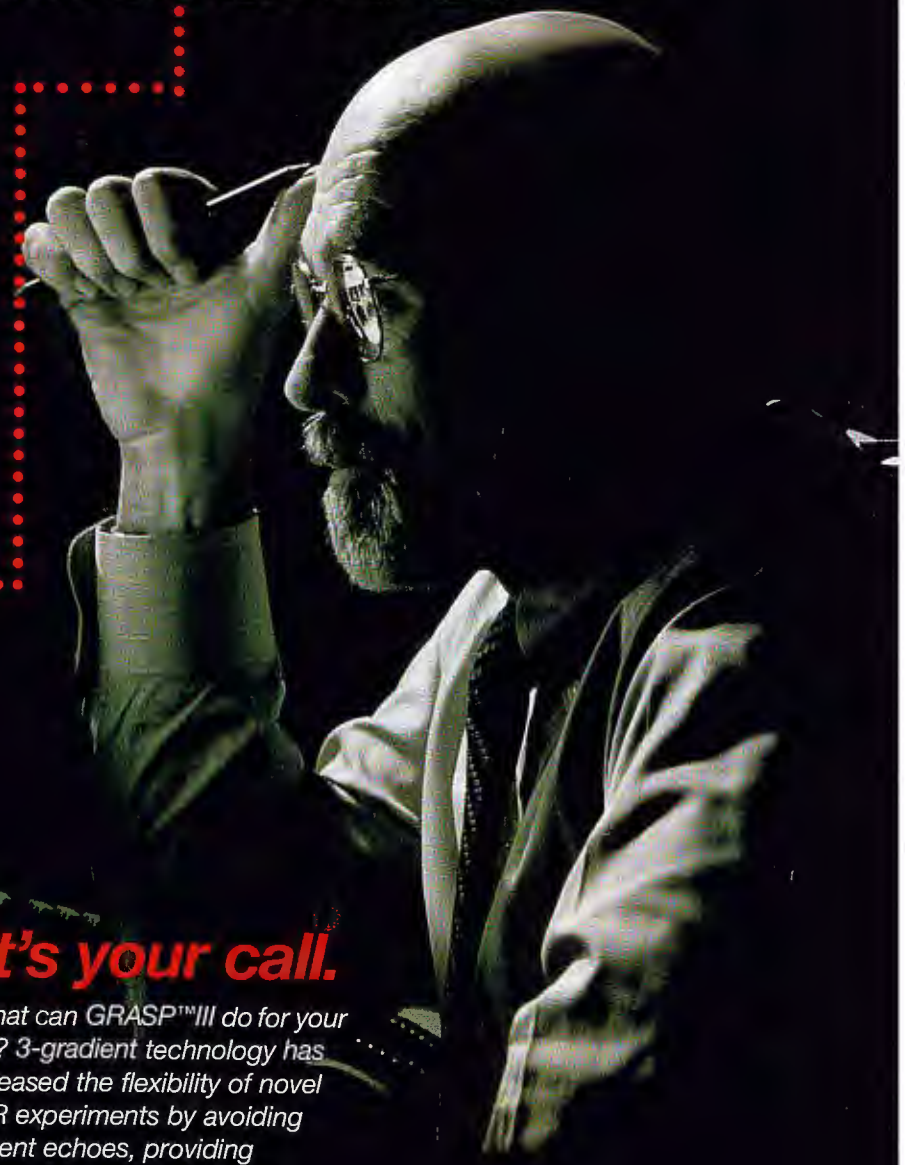
Sincerely, Dean Olson

Dean L. Olson, Postdoctoral Research Associate
Jonathan V. Sweedler, Associate Professor of Chemistry

[1] Olson, D.L.; Peck, T.L.; Webb, A.G.; Magin, R.L.; Sweedler, J.V. *Science* **270**: 1967-1970 (1995).

Take a hard look at the difference.

Expansion of DQF-COSY with magic angle gradient, 1.5 mM BPTI in 90% H_2O/10% D_2O.



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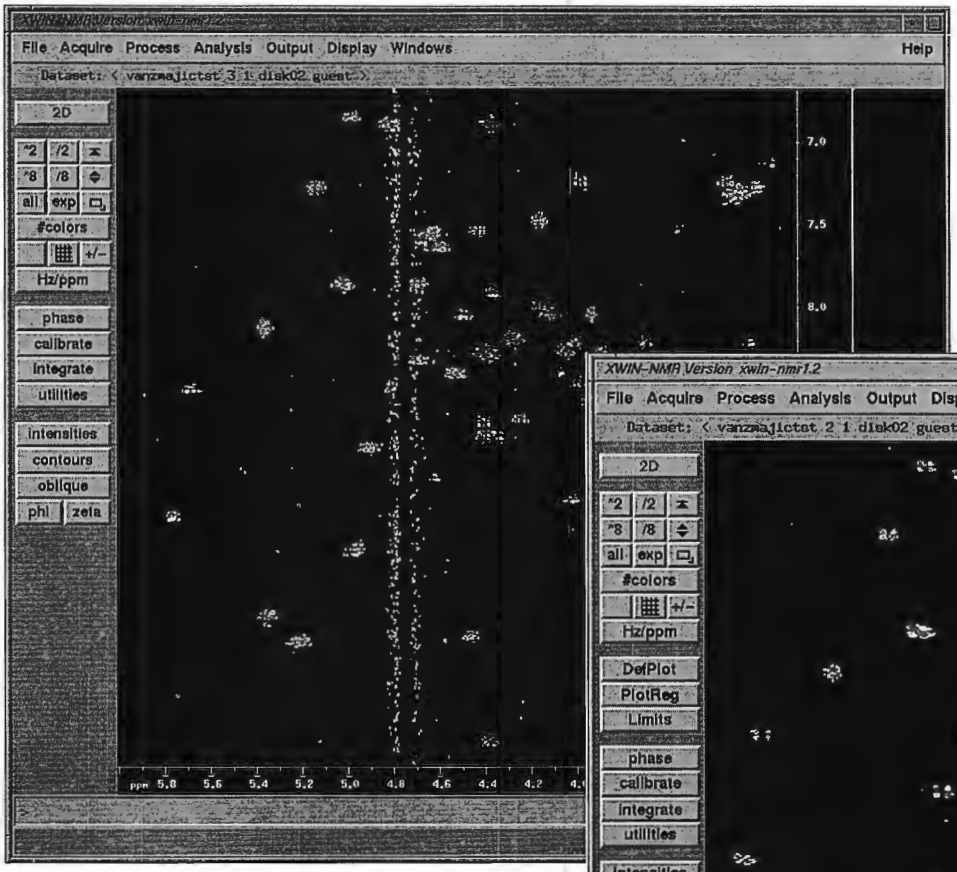
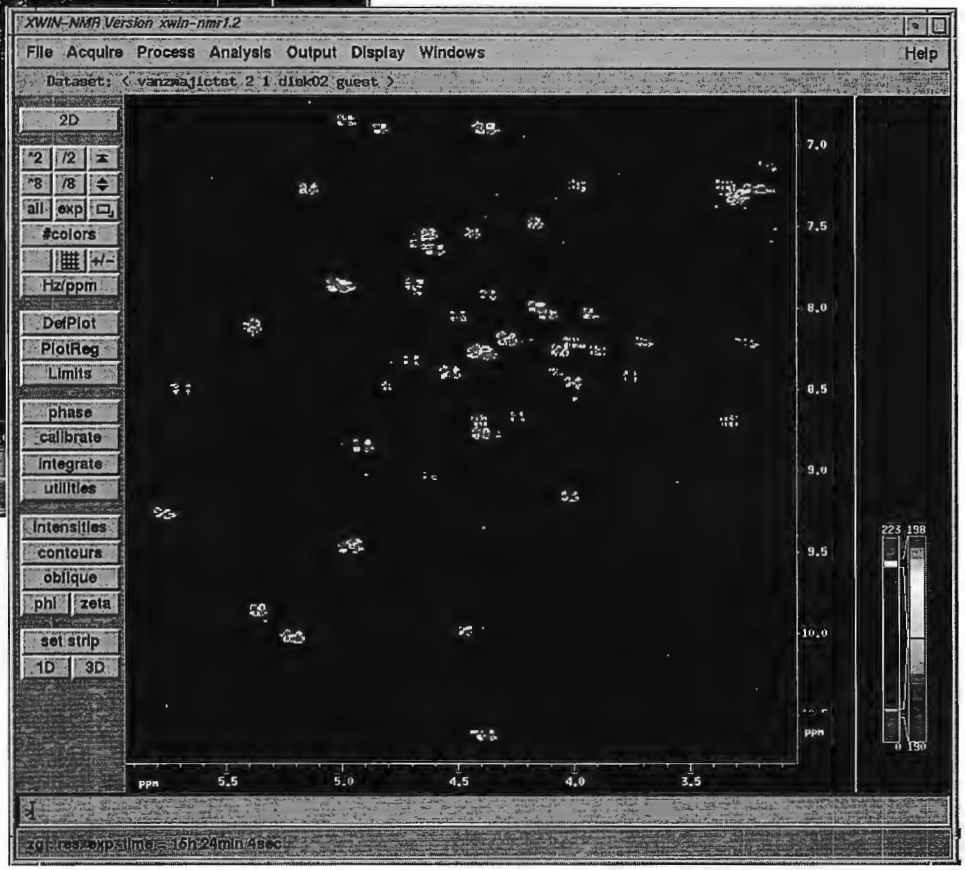


Results with the z-gradient only... residual water ridge is clearly visible and overlaps crosspeaks of interest.

Using the Bruker GRAdient SPectroscopy III (GRASPTM-III) accessory with x,y,z-gradients, an effective gradient at the magic angle can be easily produced by applying three gradients simultaneously. This greatly improves the elimination of residual water by coherence selection in multiple-quantum filtered COSY experiments.

Both experiments were acquired on the Bruker AVANCE 500 equipped with a 5 mm inverse triple resonance (TXI) probe with GRASPTM-III. The sample is 1.5 mM BPTI in 90% H_2O/10% D_2O. The experiment is DQF-COSY.



Results with magic angle gradient... residual water ridge is eliminated! Crosspeaks previously overlapped by the water can be observed and used for correlation assignment. The elimination of the water signal is achieved by coherence selection. No presaturation is used in either experiment!

For complete details or to arrange a demonstration please contact your nearest Bruker representative.

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Prof. ANIL KUMAR

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

(received 9/24/96)
September 13, 1996

Sideband Suppression by VSMASS

"Close your eyes to what you don't want to see"

Dear Barry,

Largest application of solid state NMR is to identify the various ^{13}C chemical sites in a solid powder. For this the standard technique is to enhance sensitivity via cross–polarization and reduce powder broadening by magic angle sample spinning, breaking–up the powder pattern into a centreband and several sidebands. The sidebands are identified by shifting them in a separate experiment with a different spinning speed. Alternately, they are suppressed by application of a series of r.f pulses with well defined intervals,before the accumulation of the signal, using techniques named TOSS, SELTICS etc. These later techniques require time consuming careful experimentation and are utilized only by few experts . Generally people identify the centrebands by the former method and publish spectra, marking asterix on either the centrebands or sidebands.

We have never been able to understand why obtain spectra with sidebands when they are not needed, or in other words why signal average data with the same spinning speed, when the sidebands are not needed. A straight forward signal average of spectra collected with slightly different speeds (difference more than the linewidth) produces, clean, beautiful–looking spectra, with no extra effort or time (same time as a single spectrum with sidebands) as shown in Fig 1(b). We may add that computer control of spinning speed, which should have been implemented by instrument manufacturers long ago, is now available as 'standard' in most of the spectrometers. What is still not done by them is to make the variation of spinning speed, into a single command in the AU programme (of the Bruker software) necessitating us to write a small package, which is available via e–mail from `madhu@physics.iisc.ernet.in`. This idea is also getting printed in Chemical Physics Letters.

Please advise journals not to publish spectra with asterices!!! We should publish clean spectra.

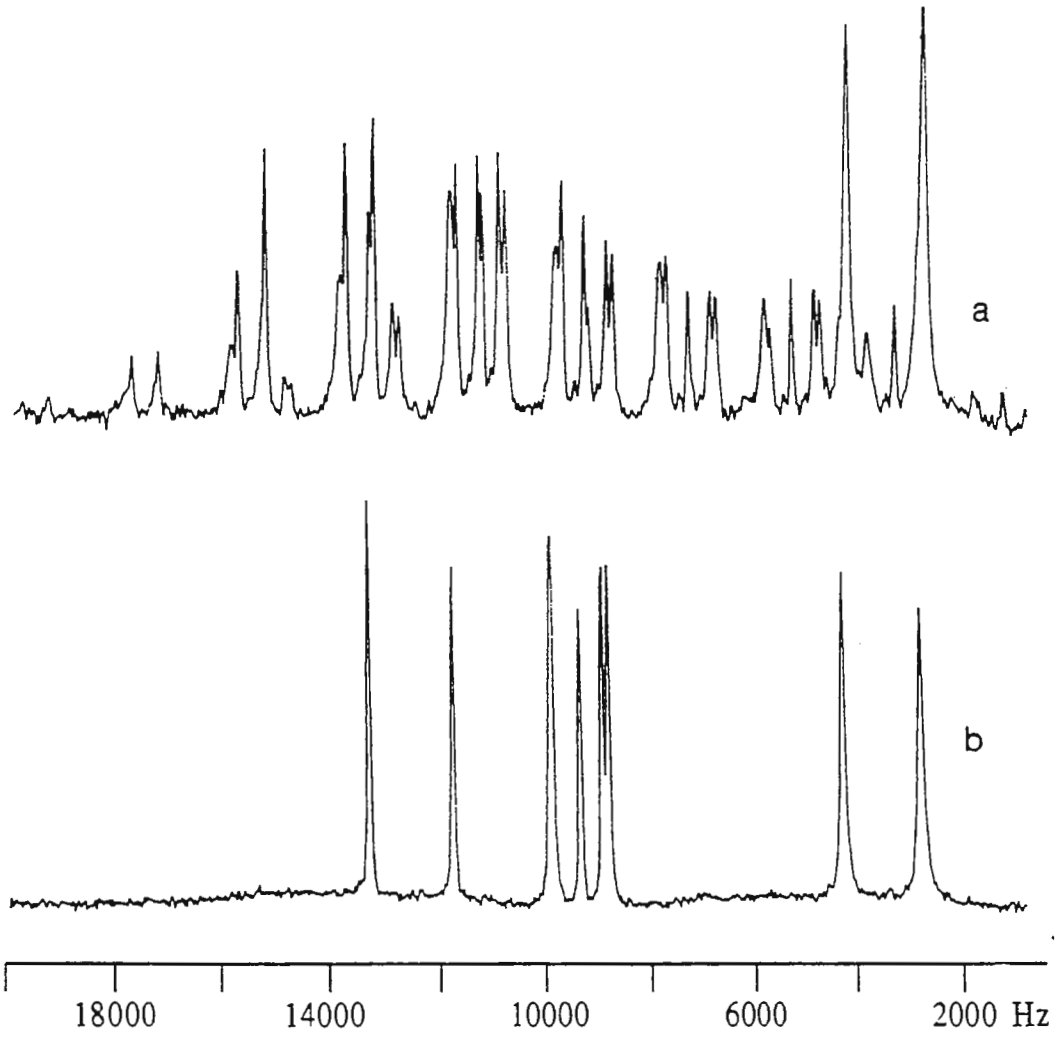

Figure 1

Figure 1: (a) ^{13} CP–MAS spectrum of tyrosine acquired in 1000 scans at a spinning speed of 2500 Hz in DSX–300 spectrometer. The CP contact time was 1 ms and recycle delay was 5 sec. (b) Same spectrum in same time (and same number of scans) with spinning speed varied from 2000 to 4000 Hz in steps of 50 Hz.

With best regards,

Madhu Pratima Anil Kumar

PS: Kindly credit this to the account of our colleague Prof.Khetrapal.

מחלקה לפיסיקה כימית
Chemical Physics Department

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August 22, 1996
(received 8/31/96)

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

NMR Crystallography

Dear Barry

NMR is a powerful complementary tool for crystallographic studies of solids. In particular it provides useful dynamic information that cannot be obtained by X-ray. In some cases NMR can also provide structural data, not available otherwise, especially when no good crystals can be grown, or when they are orientationally, or otherwise, disordered.

We have encountered several such examples in our investigations of solid mono substituted bullvalenes. In solutions these compounds exist as a mixture of rapidly interconverting isomers, while in the solid state they usually crystallize as a single isomer, most often, as the one which is abundant in solution. Thus fluorobullvalene crystallizes as isomer 4 (i.e. as the isomer in which the substitution is at carbon number 4 - see numbering system in the figure), while cyanobullvalene crystallizes as isomer 3.

Bromo- and iodo- bullvalene exist in solution as a mixture of isomer 3 (55%) and isomer 2 (45%). Both these compounds crystallize in the space group Fdd2, with the molecules at sites of C_2 symmetry. Since non of the mono substituted bullvalene isomers has C_2 symmetry the crystals must be orientationally disordered and possibly contain a mixture of isomers; X-ray measurements cannot tell.

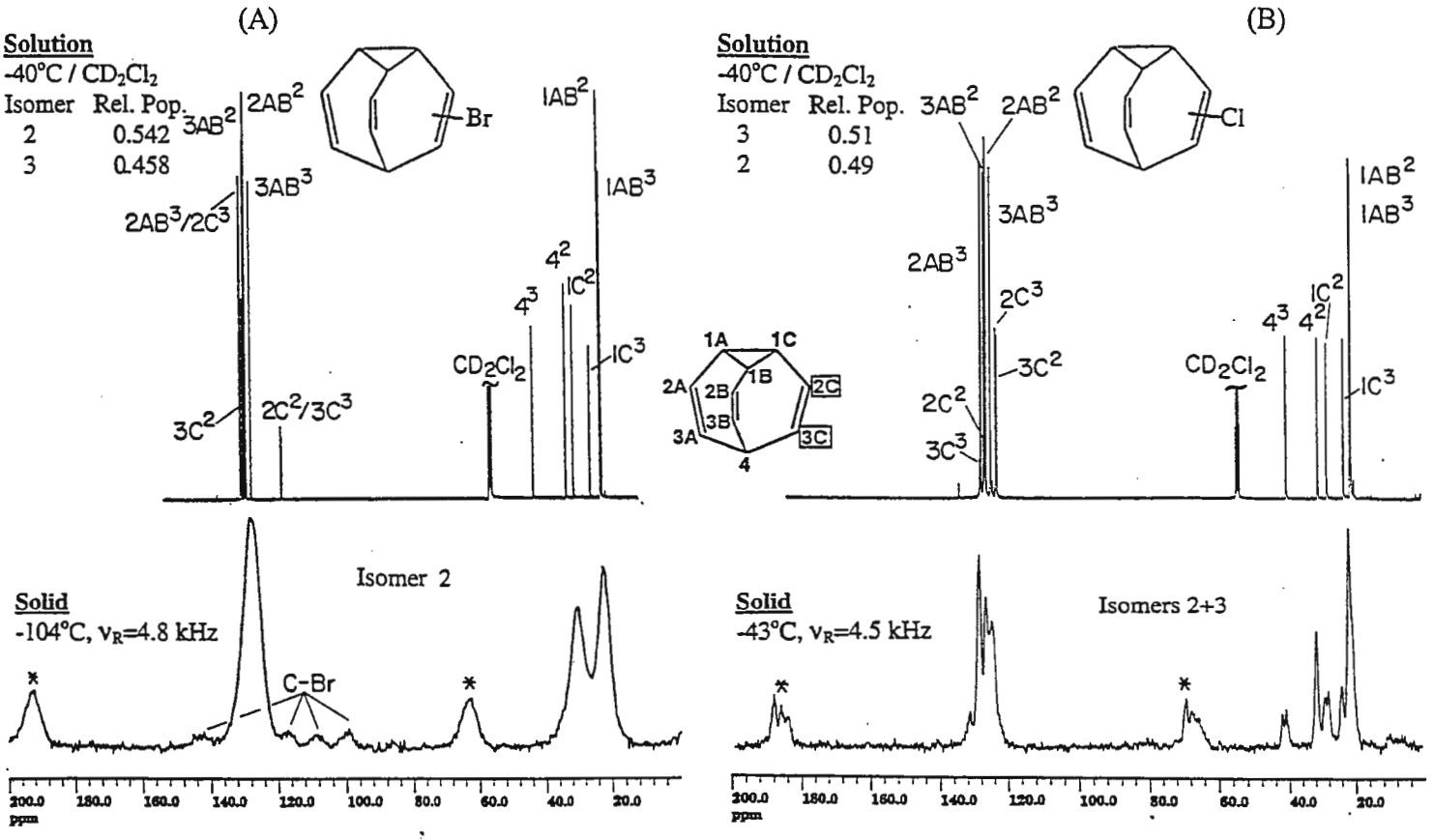
In part A of the figure we compare the solution and solid MAS carbon-13 spectra of bromobullvalene. It immediately transpires that the solid consists entirely of isomer 2, despite the fact that isomer 3 is the dominant species in solution (note the missing of the 4^3 and $1C^3$ peaks in the MAS spectrum). Dynamic 1D and 2D measurements confirm the orientational disorder of the molecules in this system. A similar situations obtains in iodobullvalene.

As a second example consider chlorobullvalene, which exhibits an entirely different behavior. This compounds melts at 14°C and no X-ray study of its crystalline state was reported. The NMR spectra (see part B of the figure) show that both, in solution and in the solid state, chlorobullvalene exists as a 1:1 molar mixture of the 2 and 3 isomers. Moreover the splitting of some of the peaks suggest that there are several types of molecules, at least of isomer 3, in the unit cell. Dynamic studies indicate that isomer 2 undergoes a degenerate rearrangement and likewise isomer 3, but there is no interconversion of the 2 and 3 isomers on the NMR time scale.

With best wishes

R. Poupko H. Zimmermann L. Olivier K. Müller C. Krieger Z. Luz

Below are carbon-13 solution (top) and solid state MAS (bottom) spectra of bromobullvalene (A) and chlorobullvalene (B). The peak assignment is explained with the help of the inserted formula. The substitution sites for the 2 and the 3 isomers are respectively carbons 2C and 3C, which are enclosed in squares in the formula.



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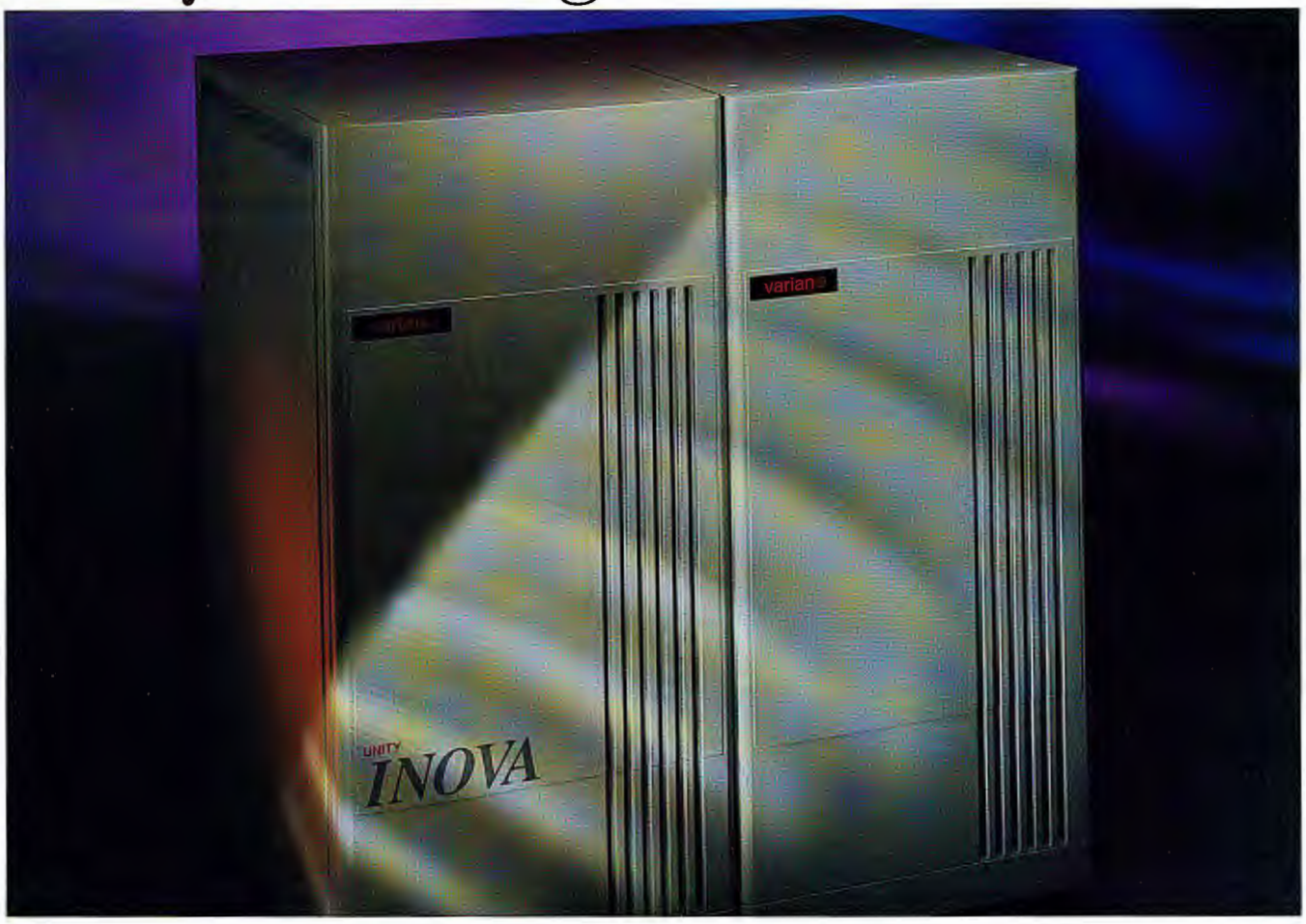

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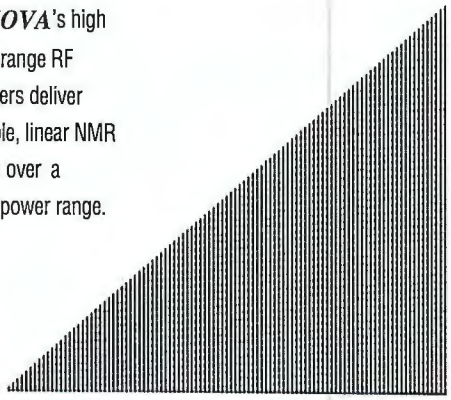
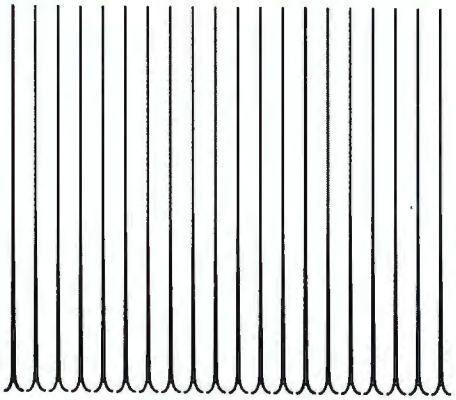


Stable, shaped RF pulses are a hallmark of the UNITY*INOVA* linear RF system, which delivers high stability shaped pulses such as these 15 µsec gaussian pulses.



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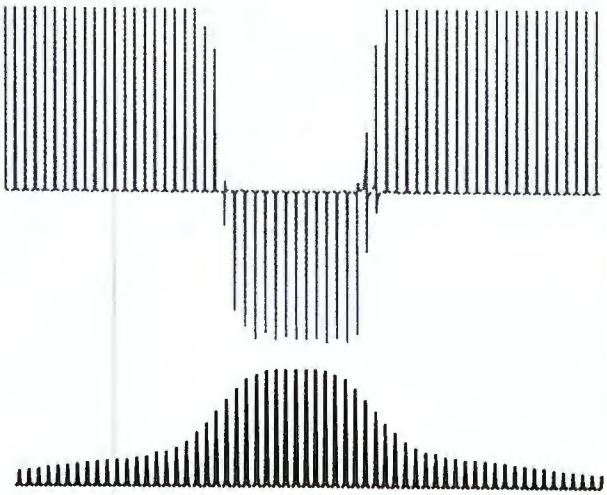

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Dr. Barry Shapiro
The NMR Newsletter
966 Elsinore Court
Palo Alto, CA 94303

September 16, 1996
(received 9/20/96)

Dear Barry,

Processing of n-dimensional data sets (n > 2) acquired using Varian instrumentation requires third party software. Our laboratory is licensed for an older version of FELIX (v. 2.05) that has proved adequate to date. Our desire to commit to software that we perceive as having a long-range plan for support and growth and that is reasonably priced to the academic community has led us to investigate NMRPipe (ver. 6/25/96, from Frank Delaglio, delaglio@speck.niddk.nih.gov). As mentioned before in this forum, (Duncan M. Smith, July, 1996), NMRPipe requires extensive command-line entry or script generation for the conversion of vendor specific data to the data format compatible with NMRPipe. Additional script generation is then required for data processing to be done using NMRPipe. Frank Delaglio has incorporated Tcl/Tk/X11 graphical interfaces into his software package that make the processing of data a snap. To facilitate the data conversion process, Dave Babcook in our group has written a macro within VNMR that takes the Varian FID from the files interface, loads the selected data set into the current experiment, reads the parameters from that data set and generates an appropriate conversion script to create the NMRPipe data set. This macro is invoked by entering the macro name (i.e. vnmr2pipe) at the VNMR command prompt after having selected one Varian FID directory while in file select mode. Two versions of the macro do the same procedure, one is completely automatic except for a user prompt to enter the number of dimensions, the other is interactive and confirms every step with the user. If interested in obtaining this macro, please send a message by e-mail to dbabcook@unmc.edu and the macros will be sent to the return address.

Sincerely yours,

David M. Babcook

William H. Gmeiner

Biophysical Faculty Positions
The University of Texas Medical Branch
Galveston, Texas

In continuing development of Structural Biology at the University of Texas Medical Branch, we are pleased to announce two tenure-track faculty openings at the Assistant Professor level.

1. Nuclear Magnetic Resonance Spectroscopy - We seek candidates with strong backgrounds in NMR spectroscopy who make extensive use of NMR to solve important biological problems. Successful candidates are to establish independent research program that may involve determinations of macromolecular structures and/or innovative application of NMR spectroscopy in the study of biological macromolecules. The NMR facility at UTMB consists of new 400, 600 and 750 MHz Varian Spectrometers housed in a newly renovated building.

2. Biophysical Chemistry - Candidates with strong backgrounds and expertise in biophysical chemistry are sought. The successful candidate must establish an independent research program to investigate an important biological problem that involves macromolecular interactions. Center facilities include analytical ultracentrifugation, electrospray mass spectrometry and dynamic light scattering apparatus.

Candidates for both positions must have outstanding potential in research with a record and stature in their field of expertise. They are expected to attract funding from national agencies and to fully participate in teaching and service with the Department of Human Biological Chemistry and Genetics. Affiliations with The Sealy Center for Structural Biology, other UTMB Centers and relevant Departments are possible.

Structural Biology currently consists of 10 biophysical faculty members in areas of NMR spectroscopy, x-ray crystallography, computational biology, and biophysical chemistry. Facilities in areas of NMR spectroscopy, computation, x-ray crystallography, and biophysical chemistry are directed by Drs. David Gorenstein, Werner Braun, Robert O. Fox and James Lee, respectively. Structural Biology received financial support from UTMB and endowed support from the Sealy Center for Structural Biology. Facilities consist of a newly renovated building and additional laboratories, along with excellent computational, NMR, crystallographic, and biophysical equipment.

Applications should include a complete curriculum vitae, a description of research interests and names and addresses of at least three references. Review of applications will continue until the positions are filled. Applications should be addressed to:

Dr. James C. Lee
Structural Biology Search Committee
Department of Human Biological Chemistry and Genetics
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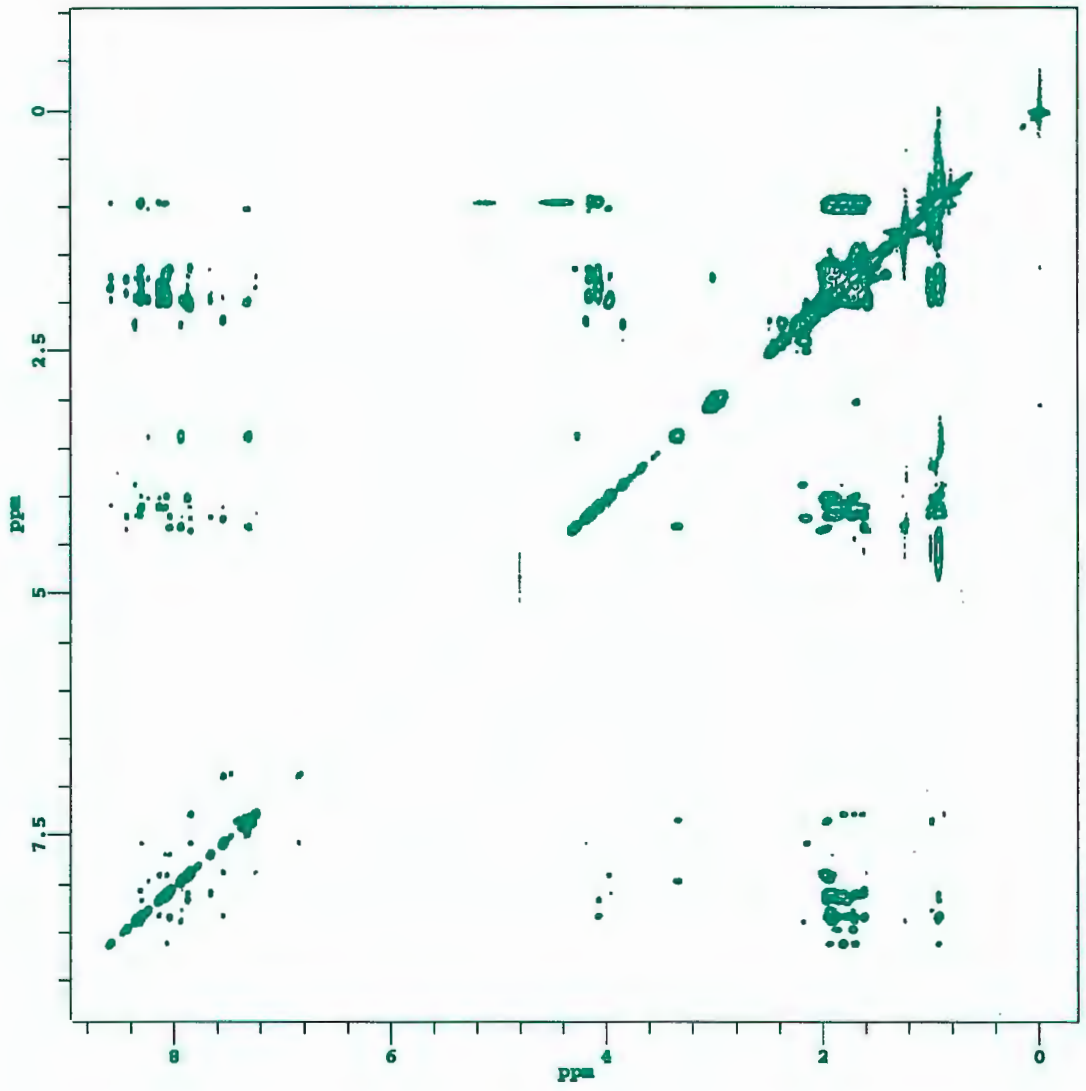

This spectrum was acquired as 2048 rows of 512 hypercomplex points each. Thirty-two scans per row were used to achieve the excellent signal-to-noise seen here.

NOESY SPECTRUM WITH FLIPBACK WATER SUPPRESSION OF A DILUTE PEPTIDE IN MICELLES

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The spectrum shown here is of 2mM PIF in sodium dodecylsulfate-d_{25}. PIF is a peptide of eighteen residues, and sodium dodecylsulfate-d_{25} is a micelle surfactant. The solvent mixture is 90% H_2O, 10% D_2O.

Absolutely no t_1 ridges nor any other artifacts are present. The impressive resolution of the fine structure shown here attests to the CMX Infinity's ability to deal with systems with difficult dynamic range requirements. This, combined with the superb water suppression as demonstrated here, makes the CMX Infinity Spectrometer an excellent choice for biological applications.

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

September 18, 1996
(received 9/23/96)

Signal Enhancement as a Measure of Molecular Motion in Asphalts

Dear Barry:

An important feature in the NMR spectra of any material is the increase in the signal-to-noise ratio as the temperature is decreased. The increase in the signal is due to the difference in the population of nuclear spins in the ground state relative to a higher energy state. That is, as the temperature is lowered, the number of spins increases in the ground state increasing the spin differences between the energy states resulting in an increase in the intensity of the NMR signal. The increase in signal can be predicted from the Boltzmann distribution equation and nuclear spin theory. The total spin magnetization, M_o, at any given temperature is given by equation 1.[1]

$$M_o = N\gamma^2\hbar^2 B_o / 4kT \qquad (1)$$

Sullivan and Maciel[2] used equation 1 to show that the increase in the NMR signals for Powhatan #5 coal at temperatures below 21°C is due only to the Boltzmann factor (ratio of the absolute temperatures). Coal is a very rigid solid without any apparent or significant molecular motion in the range of 10 to 50 kHz throughout the low temperature range. Figure 1 shows the change in the CP/MAS spectra of asphalt AAA-1 obtained at temperatures of 20 and -45°C. For the same set of conditions, the signal-to-noise ratio in the NMR spectrum at -45 is considerably better than for the spectrum taken at 20°C. Note also that the signal of the methylene carbons (32 ppm) at -45°C is greatly enhanced relative to the signal at 20°C. The signal enhancement is greater than that predicted by the Boltzmann Factor.

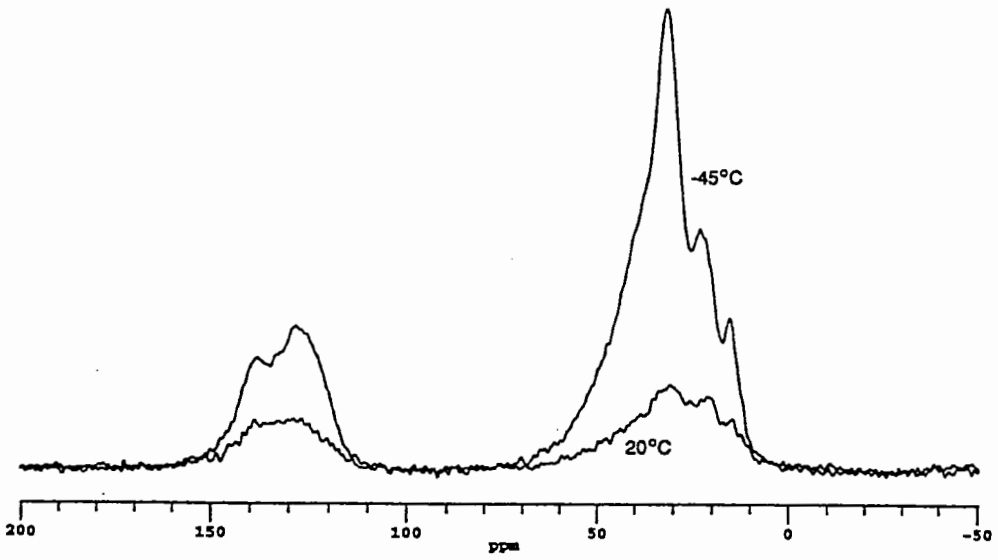

Fig. 1. Carbon-13 CP/MAS Spectra of Asphalt AAA-1 at 20 and -45°C

Figure 2 shows the NMR molecular-mobility/temperature profile plots of the aliphatic area ratios as a function of temperature for three asphalts. Also shown in Figure 2 is the plot of the absolute temperature ratios relative to 293°K as a function of temperature.

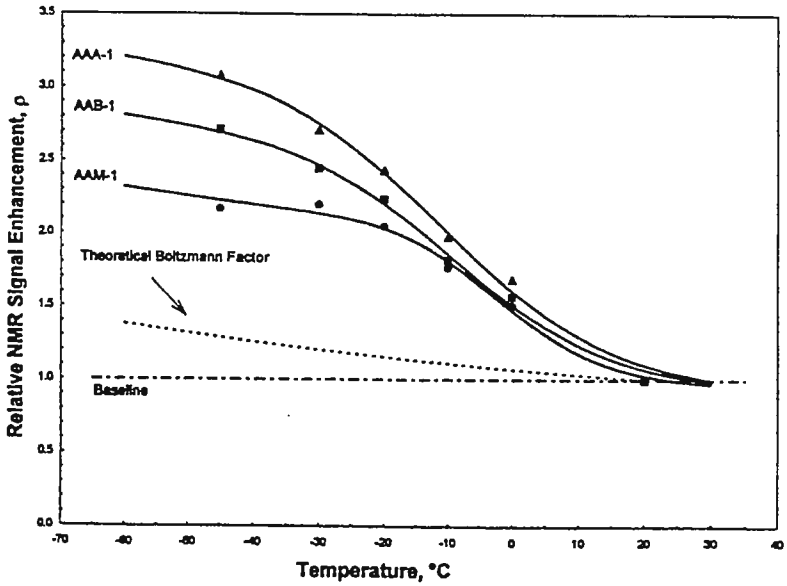

Fig. 2. NMR Molecular-Mobility/Temperature Profile Plot for Aliphatic Carbons in Asphalts AAA-1, AAB-1 & AAM-1

The ratio of the integrated areas for the aliphatic carbons of the three asphalts differ significantly from coal and from the signal enhancement due to the Boltzmann factor. These differences are the result of extensive molecular motions in asphalts which prevent effective cross-polarization of the carbon and hydrogen spins. However, as the temperature decreases, the molecular motion decreases, the molecular structure of asphalt becomes more rigid-like, the cross-polarization mechanism becomes more effective, and more carbons are observed resulting in an increase in the integrated area ratio with decreasing temperature. Thus, the area ratio can be defined as a molecular rigidity parameter. That is, as the temperature decreases, the molecular structure of asphalts becomes more rigid-like.

Asphalt AAA-1 shows a greater enhancement of the aliphatic carbon NMR signal over the temperature range from +20 to -45°C than asphalt AAB-1 which, in turn, shows a greater enhancement than asphalt AAM-1. The greater the relative enhancement at any given temperature the more molecular motion involved for the asphalts. Thus, the extent of segmental and rotational motions of the aliphatic carbons in the asphalts can be ranked as follows: AAA-1 > AAB-1 > AAM-1. This ranking is in the same relative order as the glass-transition temperature, viscosities, and various other rheological properties.

The distinction among asphalts based upon the extent of molecular motions over the temperature range of 65°C for the aliphatic carbons suggests that the NMR mobility/temperature profile methodology may be useful to study aging, steric hardening, and low temperature physical hardening as it affects the motions of the aromatic and/or aliphatic carbons in asphalts.

References

1 Harris, R. K., 1983, "Nuclear Magnetic Resonance Spectroscopy," Pitman Publishing Inc., Marshfield, MA, p. 9.

2 Sullivan, M. J., and G. E. Maciel, 1982. Spin Dynamics in the Carbon-13 Nuclear Magnetic Resonance Spectrometric Analysis of Coal by Cross Polarization and Magic-Angle Spinning, Anal. Chem., 54, 1615-1623.

Sincerely,

Daniel A. Netzel

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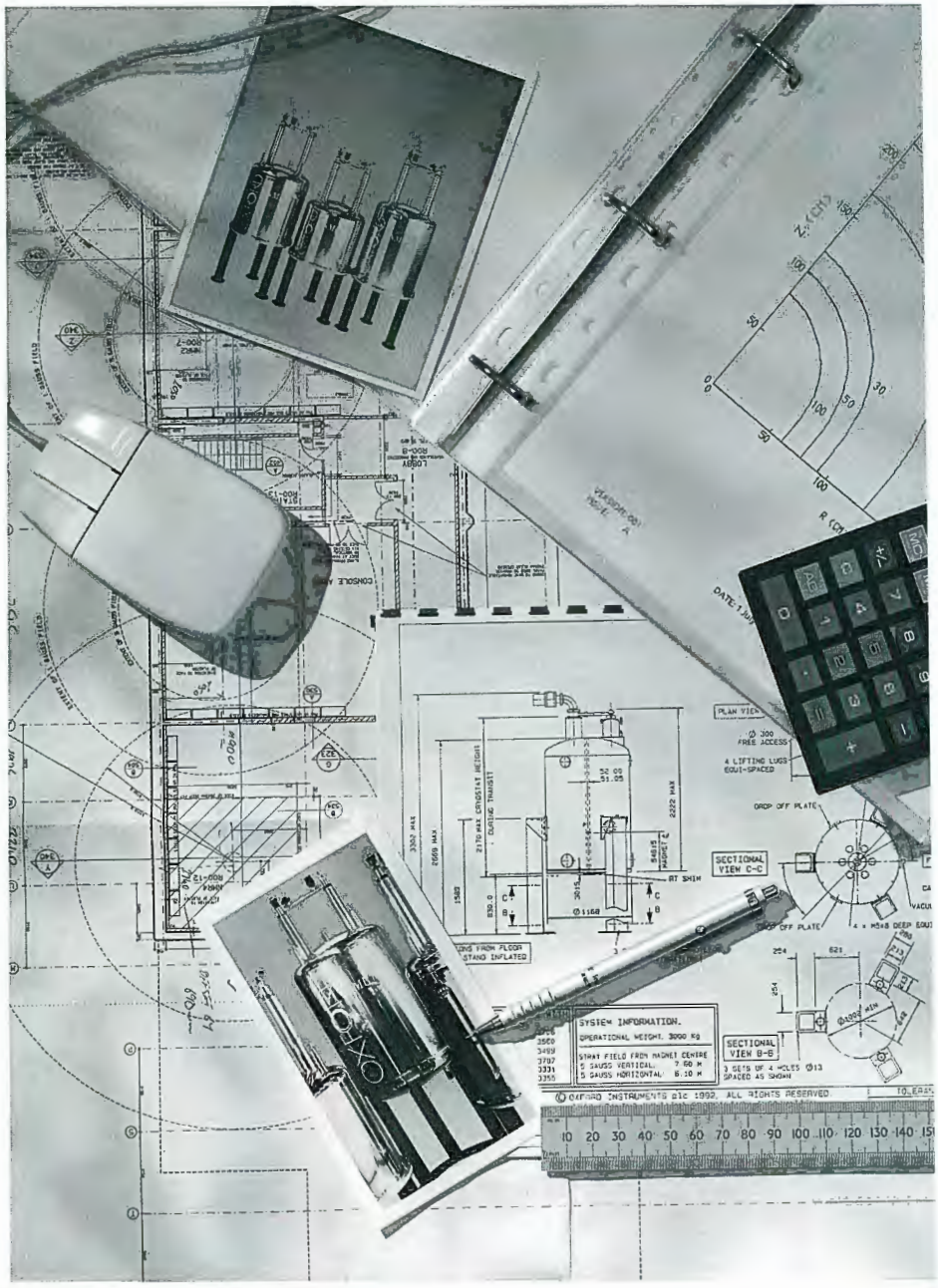

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500	51	10	150	3.2
400	54	8	365	2.8
360	54	8	365	2.8
300	54	3	365	2.8
270	54	2.7	365	2.8
200	54	2	365	2.8
100	54	1	365	2.8
500	89	15	120	3.4
400	89	10	180	2.8
360	89	10	365	2.8
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Dear Barry:

In the late 1970's I was a graduate student in Bob Vaughan's group at Caltech; it was quite fashionable in those days to perform "dipolar oscillation spectroscopy" or "separated local field spectroscopy". In these experiments the time-dependent dipolar coupling between two nuclei *in a powder sample* was measured and compared to an equation such as

$$\text{signal} \propto 1 - \frac{1}{2}\int_0^{\pi} \cos[2\pi D(3\cos^2\theta - 1)t]\sin\theta d\theta \ ,$$

where D is the dipolar coupling constant (which contains the internuclear distance to the $-\frac{1}{3}$ power), t is the amount of time under which dipolar evolution occurs, and θ is the angle between the internuclear vector and the applied magnetic field. A quick look at my old lab notebook shows that I evaluated this integral numerically with a programmable (and quite expensive) calculator; it took about **15 hours** to calculate six choices of the Dt product; these six points were used, with pencil, graph paper, and french curve, to generate a "dipolar evolution curve."

The alphabet soup of new pulse schemes aimed at determining internuclear distances in solids, such as SEDOR, REDOR, TEDOR, TRAPDOR, REAPOR, etc., inevitably involve comparing data sets to equations such as the one shown above. It is interesting to ask how one best compares data to such models. This letter describes my group's success with Mathematica and some clever mathematics published recently by our colleague Karl Mueller at Penn State.*

Mathematica is a software package for numerical and symbolic computation, as well as graphics and sound presentation. It is often compared, and contrasted, to other popular software such as MATLAB, MathCad, and Maple. Although I find Mathematica to be quite lovely, I am not trying to sell it here. I am hoping to remind you how software of this type can make your analysis of NMR problems more productive.

How does Mathematica, and their ilk, deal with solving equations such as the one shown above? First, one types the equation in the form of a command, like this:

$$\text{curve} = 1 - 0.5\text{Integrate}[(\text{Cos}[2\pi\text{Dt}(1 - 3(\text{Cos}[\theta])^2)])\text{Sin}[\theta], \theta, 0., \pi]$$

Mathematica immediately obliges the user by returning the *symbolic* solution to the integral; the output to the user looks like

$$1 - 0.5\left(\frac{-\left(\cos(2\,Dt\,\pi)\,FresnelC(-2\sqrt{3}\sqrt{Dt}) + FresnelS(-2\sqrt{3}\sqrt{Dt})\,\sin(2\,Dt\,\pi)\right)}{2\sqrt{3}\sqrt{Dt}} + \frac{\cos(2\,Dt\,\pi)\,FresnelC(2\sqrt{3}\sqrt{Dt}) + FresnelS(2\sqrt{3}\sqrt{Dt})\,\sin(2\,Dt\,\pi)}{2\sqrt{3}\sqrt{Dt}}\right) .$$

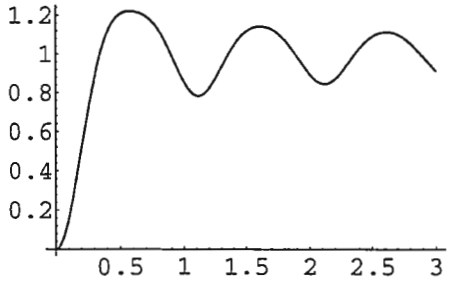
Notice that the output tells us that the integral yields so-called "Fresnel integrals". One can instruct Mathematica to numerically evaluate **and plot** these integrals as a function of Dt, thereby generating the desired "dipolar evolution curve". On my 90Mhz Pentium machine this calculation and graphing takes **181 seconds** for a given value of the internuclear distance, approximately 300 times faster than the top-of-the-line 1979 calculator, not including the large amount of time it took me to program the calculator (in BASIC) and generate a hand-drawn plot.

* Karl T. Mueller, Jour. Mag. Res. **A113** 81 (1995)

Alternatively, one could turn to Karl's paper and discover that the Fresnel integrals may be replaced with an infinite series of Bessel functions. Karl shows in his paper that the series solution converges rapidly: keeping only the first five terms in the series is accurate to within 0.1%. Using Karl's formulas, then, I ask Mathematica to evaluate

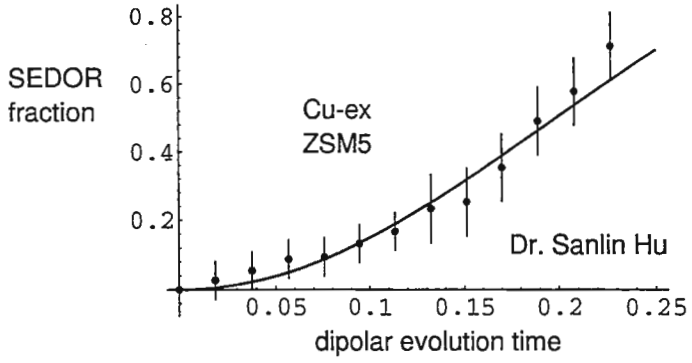
$$1-\text{BesselJ}(0, 3\pi x)\cos(\pi x) + 2\Big(0.333333\,\text{BesselJ}(1., 3\pi x)\cos(0.5\pi + \pi x) + 0.0666667\,\text{BesselJ}(2., 3\pi x)\cos(1.\pi + \pi x) + 0.0285714\,\text{BesselJ}(3., 3\pi x)\cos(1.5\pi + \pi x) + 0.015873\,\text{BesselJ}(4., 3\pi x)\cos(2.\pi + \pi x) + 0.010101\,\text{BesselJ}(5., 3\pi x)\cos(2.5\pi + \pi x)\Big)\ ,$$

where x is the product Dt. This approximation took my Pentium machine **10.8 seconds** to generate and plot, which is a factor of 18 in time savings over the numerical evaluation of the Fresnel integrals! The result is the "SEDOR" curve shown below



As Karl points out in his paper, the real advantage to these analytical expressions is that Mathematica has built-in nonlinear fit algorithms so that the expression for the SEDOR curve may be fit to actual data, with the resulting "best fit" yielding the dipolar frequency, and thus the internuclear distance. All this can be done in moments with your personal computer.

As an example, the graph below shows actual SEDOR data and best fit using Mathematica and Karl's series solution, taken by my postdoc Sanlin Hu. The range of the dipolar evolution time is much smaller than that for the graphs above, and the computational time is correspondingly much faster. Indeed, the fit below with D as the floating variable took my Pentium **0.88 seconds** to evaluate (it took just another few other keystrokes to generate the plot). The data are from Cu-Al double resonance experiments on a copper-exchanged zeolite, and establish that the Cu(1) atoms are just over two angstroms away from the aluminum atoms, in excellent agreement with computational quantum theory estimates.



Mathematica is not unique; there are other packages that claim similar "user-friendliness." My point is that if you haven't been using these packages to explore mathematical analysis of your NMR data, you may be missing out on very productive, and powerful, extensions of your work. As it turns out, these packages also serve the purpose of reinforcing the fundamentals of mathematics and analysis that were once a part of our intellectual training, but may have rusted a bit over the years. In closing I note that Mathematica is a powerful teaching tool also, and my teenage children, as well as my graduate students, have benefitted by exploring worked examples in Mathematica textbooks. *

my best wishes to all,

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September 18, 1996
(received 9/21/96)

Dr. B. L. Shapiro
The NMR Newsletter
966 Elsinore Court
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USA

Suspension NMR Spectroscopy of Phosphines Immobilized on Silica

Dear Dr. Shapiro,

The immobilization of homogeneous catalysts on inert supports is of growing interest, because the advantages of homogeneous and heterogeneous catalysis, like high selectivity and easy recycling, can in principle be combined. Since most metals form stable phosphine complexes, bifunctional phosphines like $Ph_2P(CH_2)_3Si(OEt)_3$ are often used as linkers [1]. However, insiders will agree with me, that the subject is literally very tricky, and a lot of basic research is required.

The most powerful analytical tool for characterizing surface-immobilized species is CP/MAS NMR spectroscopy. However, it is somewhat time consuming and expensive, when a large number of samples has to be measured. Therefore, as an alternative, we investigated ^{31}P suspension NMR spectroscopy [2] for a rapid check of the surface-modified silicas under "realistic" (wet) conditions.

Indeed, suspension NMR spectroscopy offers several advantages: a) The materials do not have to be dried prior to the measurement. b) Reliable quantitative information can be extracted from one spectrum. For example, the ratio of oxidic impurity S to immobilized phosphine (see Fig. 1, top trace) is correct. c) There are no rotational sidebands that could lead to overlapping. d) The measurement times are short. For example, the suspension NMR spectrum of Fig. 1 was recorded in about 15 minutes, while the CP/MAS spectrum of a sample with equal surface coverage (Fig. 1, lower trace) required three hours. e) The low power mode of any routine NMR machine suffices.

From a systematic study, some general trends emerge [2]: 1. The lower the viscosity of the solvents (or better: the suspending liquids), the narrower are the lines. This is due to the enhanced mobility of the surface-attached species. 2. Increasing polarity of the solvents reduces the linewidths, because the phosphine moieties are detached from the surface. 3. The average pore or particle size does not influence the linewidth.

The example of Fig. 1 shows, that under optimal conditions the suspension NMR signals can be nearly as narrow as the ^{31}P CP/MAS NMR resonances!

Please credit this contribution to Prof. F. H. Köhler's account.

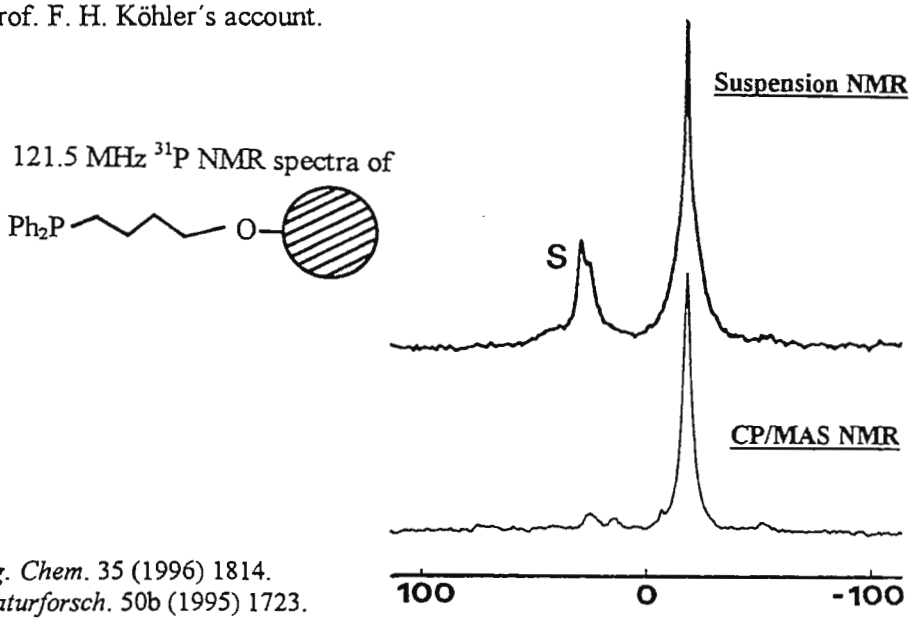

Fig. 1: 121.5 MHz ^{31}P NMR spectra of Ph_2P–O–⟨hatched⟩

With best regards,

Janet Blümel

(Dr. habil. Janet Blümel)

[1] K. D. Behringer, J. Blümel, *Inorg. Chem.* 35 (1996) 1814.
[2] K. D. Behringer, J. Blümel, *Z. Naturforsch.* 50b (1995) 1723.

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NMR studies of the ISL Homeodomain; position available

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

Monday, September 16, 1996
(received 9/21/96)

Dear Barry,

One of us (Sybren Wijmenga) has recently moved from Nijmegen University in the Netherlands to Umeå University in Sweden. Here at the Department of Medical Biophysics he is in the process of setting up a laboratory for structural studies of biological macromolecules by means of high resolution NMR. The main stay of the instrumentation is going to be a fully equipped Bruker 600 MHz DRX spectrometer, to arrive in December of this year. A 500 Mhz Bruker AMX2 spectrometer is already available. The instrumentation is shared with the Department of Organic Chemistry. We seek now a young graduated NMR scientist with preferably some post-doctoral experience whose task will be the management of the NMR instrumentation. Also, he or she can carry out his or her own research and/or participate in the ongoing projects. Please note the attached announcement for further details.

As you can imagine at this point we have a lot of plans for projects. We are particularly enthousiastic since at the University reside a number of well known molecular biology groups, who have a strong interest in complementing their research with structural data from NMR methods. These projects involve structure determination of nucleic acids as well as proteins, and also the study of their interactions. Apologies for the generality. That is how it is when plans exist, but most data still have to be produced.

One project has already produced some nice results. This project has been initiated and conducted by two of us, viz. Gity Behravan and Per-Olof Lycksell, at the Department of Medical Biophysics and Sybren Wijmenga has been involved before moving. It concerns the Homeo- and LIM-domains of the Insulin gene transcription factor ISL-1. *In* vitro translated ISL-1 was shown to bind to the TAAT motif present in the insulin gene enhancer in rat. ISL-1 is also involved in the regulation of the amylin and proglucagon genes. The Homeo-domain binds DNA specifically, most likely via a helix-turn-helix motif. The role of the LIM-domain seems to be the modulation of the DNA binding. Efficient expression systems have been set up for producing sufficient quantities of both the Homeo-domain, a.o. doubly ($^{13}C/^{15}N$) labeled material of a 74 amino acid residues long construct, and the LIM-domain for NMR studies and characterisation of the DNA binding. The figure shows a ^{15}N HSQC spectrum of the ^{15}N labeled free Homeo-domain. Furthermore, of this construct the suite of triple resonance spectra necessary for assignment has been recorded. In the near future we hope to complete the assignment and have a structure available of the free Homeo-domain. At present it is already known

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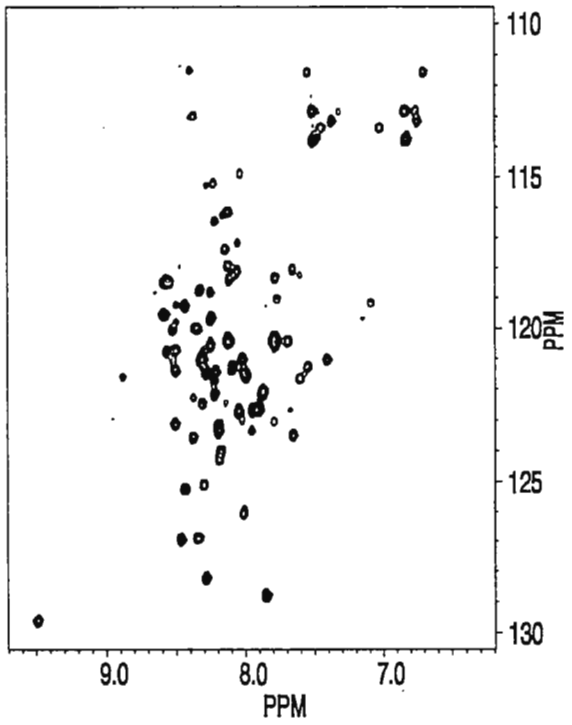
Figure 1

^{15}N-^{1}H HSQC of Homeo-domain.



from the CD and NMR data that the free Homeo-domain has a significant helix content consistent with a helix-turn-helix type of fold. Initial studies on the Homeo-domain/DNA complex clearly show strong binding of DNA to the Homeo-domain. Most interestingly considerable stabilisation occurs of the Home-domain on complexation.

Gity Behravan Per Olof Lycksell Sybren Wijmenga

Please credit this contribution to the account of professor Ulf Edlund.

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Further information can be obtained from Professor Sybren Wijmenga, Department of Medical Biophysics, Umeå University, S-901 87 Umeå, Sweden, tel.: +46-90-167403/165234, e-mail: sybren@indigo.chem.umu.se and/or Professor Ulf Edlund, Department of Organic Chemistry, Umeå University, tel.; +46-90-166933, e-mail: ulf.edlund@chem.umu.se.

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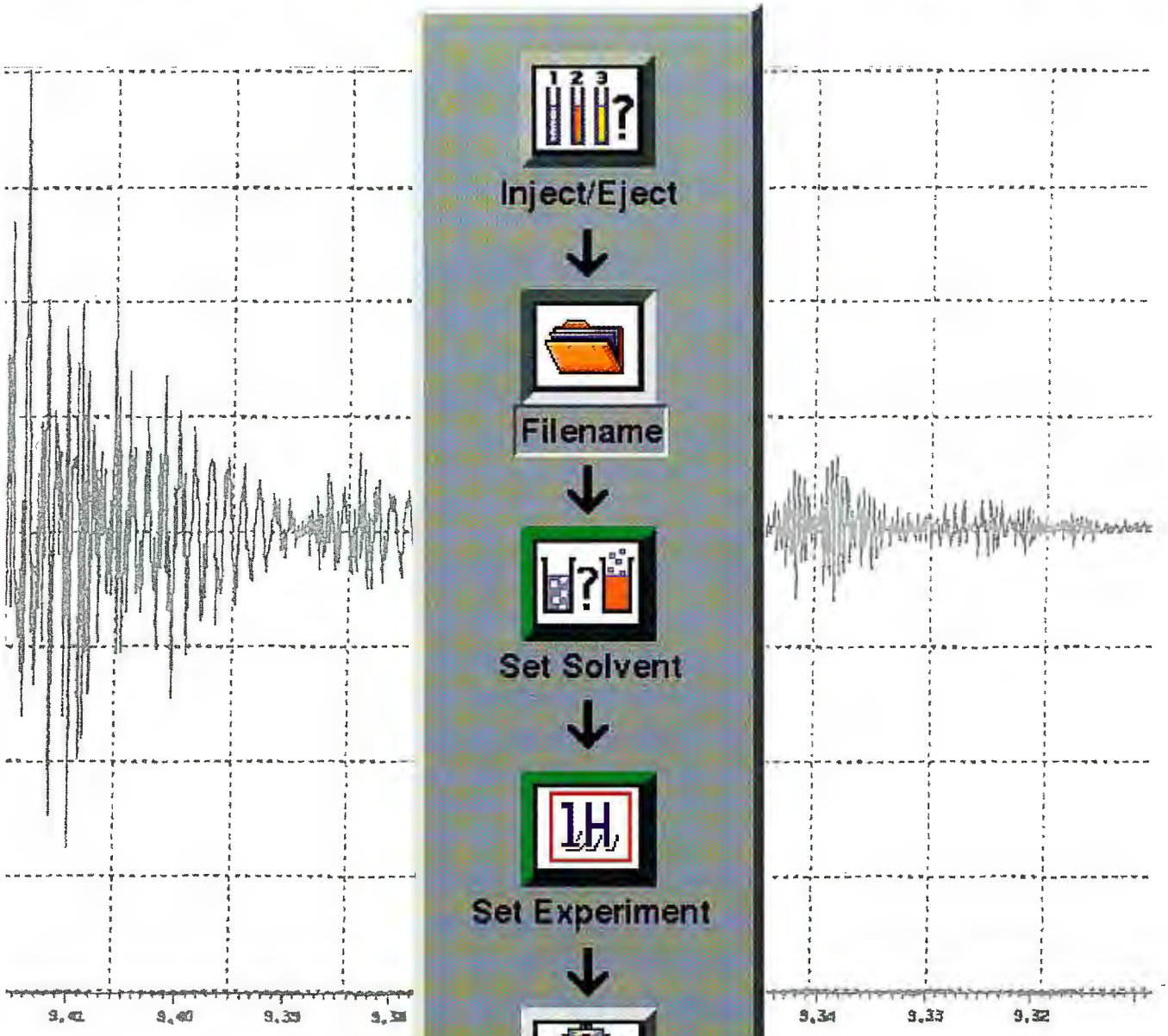

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September 10, 1996
(received 9/20/96)

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

^{29}Si CP-MAS NMR INVESTIGATION OF THE *IN-SITU* GENERATION OF SILICA REINFORCEMENT IN MODIFIED POLYDIMETHYLSILOXANE (PDMS) ELASTOMERS

Dear Barry:

The *in-situ* generation of silica particles by the sol-gel method provides an alternative route to prepare reinforced polydimethylsiloxane polymers. We have begun a systematic study of the *in situ* growth of the silicate reinforcement phase within the PDMS elastomer. Reactive functional groups were incorporated into the elastomeric matrix backbone in order to modulate the degree of interaction between the matrix and filler phases.

The backbone of PDMS was modified by the addition of one reactive trifunctional silicon for every 10 normal difunctional silicon repeat units: $(Me_2SiO)_{10}(MeOMeSiO)_1$. The silica reinforced elastomeric materials were prepared by mixing this matrix polymer, in the presence of dibutyltin dilaurate catalyst (0.2 weight %), with tetraethoxysilane (TEOS) in amounts sufficient to fill the matrix to 10 weight % silica. We used solid state MAS ^{29}Si NMR spectroscopy and ^{29}Si NMR relaxation times to determine the extent to which the two phases are chemically and physically coupled.

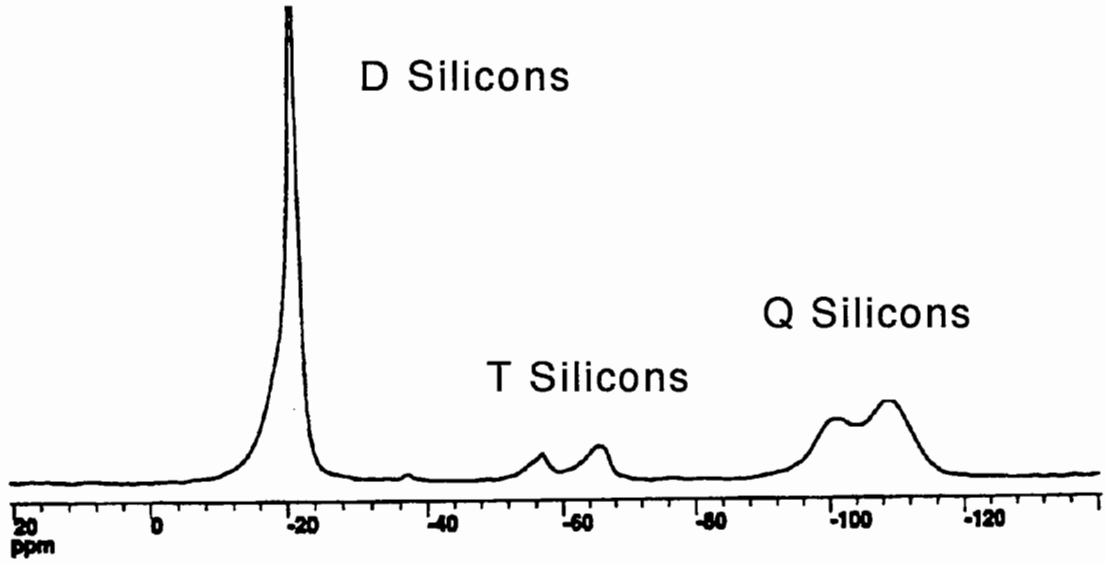

Fig 1. The solid state CP-MAS ^{29}Si NMR spectra of the $(Me_2SiO)_{10}(MeOMeSiO)_1$ + Si $(OEt)_4$ material showing the D, T and Q silicon species.

The ^{29}Si NMR spectrum of $(Me_2SiO)_{10}(MeOMeSiO)_1$ filled with TEOS is shown in Fig 1. The resonance at -21.4 ppm corresponds to the D (difunctional) backbone silicons, the resonances at -56.9 and -65.3 ppm correspond to the T^2 and T^3 (T is the trifunctional silicon and the superscript refers to the number of bridging oxygens) backbone silicons which are functionalized and the resonances at -100.7 and -108.6 ppm correspond to the Q (tetrafunctional) silicons associated with the formation of the silicate filler from TEOS.

The spectra were recorded as a function of cross-polarization time so that the relative product distributions for each silicon species could be determined. The experiments were only carried out to 15 ms so the longer $T_{1\rho H}$ relaxation times were not measured. The magnetization buildup was fit by a single exponential function where the time constant was set equal to the cross-polarization relaxation time. The shapes of the resonances corresponding to the T and Q silicons were independent of time for cp times ranging from 7 to 15 ms. The long plateau region exhibited by the signal intensities, coupled with a constant spectral shape for each silicon species gave us confidence that the spectrum components for the T and Q silicon species were at least semiquantitative.

The deconvolution of the Q resonances showed that the extents of reaction of these silicons ranged from 86 to 92 %. These values are somewhat higher than that observed for neat acid-catalyzed TEOS sol-gels. The greater extent of reaction of TEOS may be due to its increased mobility when dispersed in the elastomeric matrix. The T silicons begin the reaction as T^2 silicons and condense to form T^3 silicons. The extents of reaction of the T silicons are approximately 65 % for the material investigated.

The extent to which the various phases are physically coupled can be probed by examining the cross-polarization times of each phase. The cross-polarization times of the T and Q silicons (2 ms) are similar to each other indicating that the the reactive T silicons and the Q silicons have similar mobilities. The cross-polarization times of the D backbone silicons (4 ms) are considerably longer than those of either the T or Q silicons demonstrating that on the average, these silicon possess greater mobility than the silicons with higher functionalities.

This work supported by the United States Department of Energy under contract DE-AC04-94AL8500.

With best regards,

S. Prabakar S.E. Bates T. A. Ulibarri R. A. Assink

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9 September 1996
(received 9/14/96)

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

RE: XENON-PROTON CROSS-POLARIZATION

Dear Barry:

We have made first attempts to investigate the practical aspects of transferring magnetization from optically pumped ^{129}Xe gas with high nuclear spin polarizations to protons in aqueous solutions. Our experiments were made possible by a collaboration between the Princeton Groups of W. Happer and G. Cates and the University of Virginia Radiology Department (James Brookeman and collaborators) and the pulmonary group headed by Dr. Thomas Daniels for the purpose of imaging human lung. We had access to hyperpolarized ^{129}Xe samples for just over a week and conducted simple experiments following the work in magnetically dilute nonaqueous systems by Pines and collaborators.

^{129}Xe polarized to the level of 2% contained in glass containers treated with dichlorodimethyl silane was shaken rapidly with solutions of L-tyrosine, α-cyclodextrin, β-cyclodextrin, and apomyoglobin. Labile protons were out-exchanged prior to the experiment to minimize ^1H exchange into the D_2O as well as maximize solute ^1H relaxation times. Immediately following a vigorous shaking of the D_2O solution, which was injected into the sample bulb, the 3 mL aquous sample was placed in a 4.7 T horizontal magnet (SISCO) and the ^1H or ^{129}Xe spectrum recorded within seconds. We were searching for large intensity changes and took ^1H spectra using 5° pulses every 2.1 s for five minutes. In no case did we detect a significant enhancement of the proton spectrum similar to that reported by Navon et al. Science 271, 1846 (1996) for any of the solutes listed.

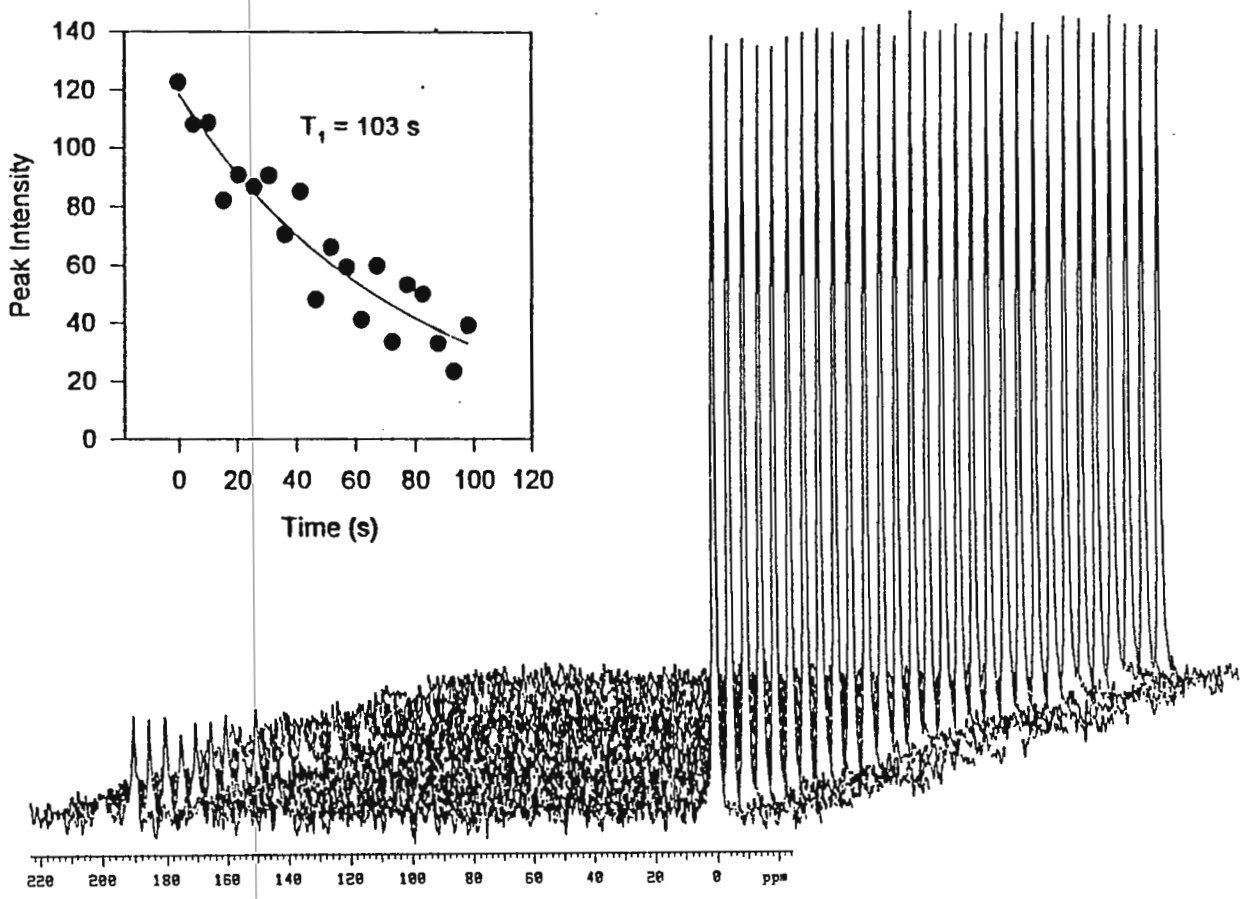
The xenon spectrum was monitored in separate experiments using samples prepared in the same way. The ^{129}Xe T_1 in the D_2O solution was approximately 600 s and the xenon T_1 values in L-tyrosine and β-cyclodextrin solutions were similar and longer than 100 s. The T_1 of the gas phase signal above the D_2O solution was long and the decay dominated by the effects of the 10.8° sampling pulses used to monitor it. Both α-cyclodextrin and myoglobin bind xenon. The relaxation rate of ^{129}Xe in the apomyoglobin solution was so rapid that no ^{129}Xe resonance could be detected in the solution following mixing although the gas phase peak verifies that the polarization was not inadvertently lost at the glass surface. The α-cyclodextrin solution shows a measurable decay of the ^{129}Xe polarization with at T_1 of 103 s as shown in the Figure below. The chemical shift reference is taken as the gas phase signal. These relatively short ^{129}Xe relaxation times demonstrate efficient coupling to the solute protons which serve as relaxation agents for the xenon. In spite of these observations, no proton signal enhancement was observed with single pulse experiments and the finite mixing times employed. Given the solution mixing times and sample positioning times of order 10 seconds, if the proton polarization was enhanced significantly, the solute ^1H polarization relaxed to Boltzmann levels more rapidly than we were able to detect the ^1H spectrum.

The ^{129}Xe-^1H cross-relaxation rate for these samples is that appropriate to the fringe field of the 40 cm-4.7 T magnet. The remaining contact occurs at 4.7 T. No match conditions were created, either at zero

field or using rf fields (Hartmann-Hahn) both of which may make the transfer rate more favorable. In the β-cyclodextrin and L-tyrosine solutions, no effective magnetic coupling was observed. Thus, transient or collisional interactions are unlikely to be effective as practical cross-relaxation vehicle for proton rich solutes.

Although these experiments were disappointing, they do not by any means eliminate the possibility that significant enhancements may be observed with higher xenon polarization, more efficient sample mixing, and a magnetization transfer conducted under some kind of matched resonance condition.

Exponential Fit for ^{129}Xe in 20mM α-Cyclodextrin/D_2O



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Date: August 23, 1996
(received 9/9/96)

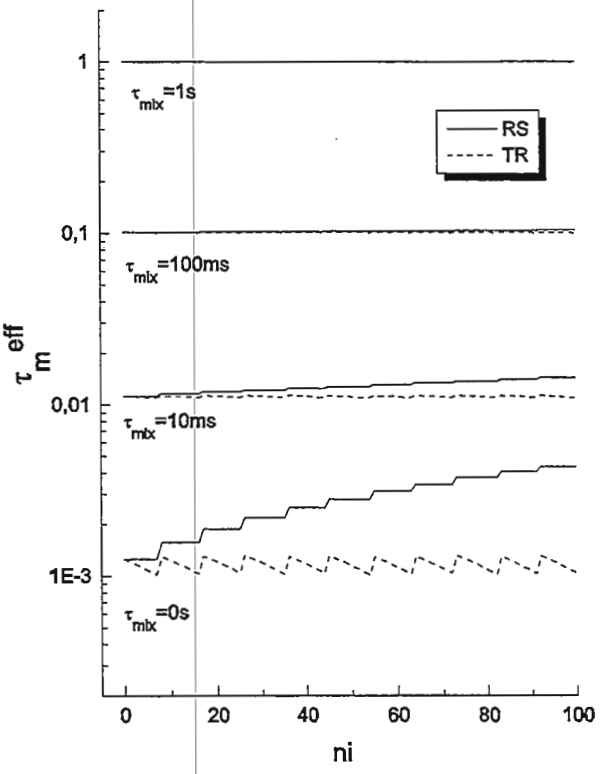
Dear Prof. Shapiro,

we are currently working with Solid-State Exchange experiments to investigate slow molecular dynamics in organic solids and polymers /1/. Since we are interested in time constants of the processes rather than in its geometry (jump angles etc.), we use the ^{13}C-2D-MAS exchange technique introduced by Veeman /2/ and modified by Hagemeyer /3/. This method gains a much higher spectral resolution and better signal-to-noise compared to the static experiments /2/, however, it provides only limited information about the geometry of the process. We like to tell you about our experiences in using the necessary spectrometer-hardware.

The pulse- sequence used for this method is basically the well-known NOESY-sequence where the appearence and the growth of cross-peaks indicate the existence of dynamic processes and its time-constants. One absolutely necessary feature of the 2D-MAS technique is the rotor-synchronization of the mixing time, i.e., the $\pi/2$- pulses before and after the mixing time must be performed at the same phase of the MAS-rotor to avoid crosspeaks that are not due to the exchange but result from artefacts of the method. In other words, the mixing time must be set to either an integer number of the rotation period ($\tau_m=N\cdot T_R$, rotor-synchronization, RS) or to an integer number minus the actual time-increment t_1 of the 2D-experiment ($\tau_m=N\cdot T_R-t_1$, time reversal, TR). Both types of experiments are necessary to obtain phase-sensitive 2D-MAS-spectra /3/,/4/. It should be emphazied that it is absolutely not sufficent to just calculate $N\cdot T_R$ and set this delay as mixing time because after long mixing times (hundreds or thousends of rotor-cycles) even small deviations will result in a substantial mismatch of the rotor-synchronization and thus in unwanted cross-peaks and/or in phase-twisting.

Most of the commercial spectrometers usually do not provide the hardware for doing rotor-synchronization, however, at most instruments there is the opportunity to feed an external trigger into the pulse-programmer. Now, the signal from the reader of the MAS-spinning speed can serve as this trigger providing information about the rotor-phase to the pulse-program. However, the actual problem is that the timing of the pulse-sequence does not allow to put the trigger just before the two $\pi/2$-pulses, i.e. let the $\pi/2$-pulse before the mixing time wait for the trigger because this would lengthen the t_1-increments of the 2D-experiment and it would give rise to completely distorted 2D-spectra. Thus, the following procedure is normally used /5/: at the beginning, the pulse-sequence is just waiting for the external trigger from the MAS-rotor. This defines the initial phase of the rotor, lets say 0 degree. Now, the pulse-sequence continues with either a first $\pi/2$- pulse or Cross-Polarization (lets say, this takes t_A seconds) and the current t_1-increment. The rotor acquired during this time a phase angle of $\phi_A=\omega_R\cdot(t_A+t_1)$ (ω_R being $2\pi\cdot$spinning frequency). Now, the sequence continues with another $\pi/2$-pulse (that takes the magnetization from the x-y-plane into the z-direction) and a predefind mixing time. Rotor-synchronization requires the next $\pi/2$-pulse to appear at exactly a rotor-phase of ϕ_A. This is achieved by waiting for the external trigger which corresponds to the initial rotor-phase of 0 degree. This delay does not harm the experiment since the magnetization is stored in I_Z. Adding a delay of t_A+t_1 ensures the desired phase-angle of ϕ_A. Now, another $\pi/2$-pulse brings the magnetization

back to the x-y-plane and the fid is acquired. It should be noted the condition for time-reversal ($\tau_m = N \cdot T_R - t_1$) can be obtained in a similar manner just by waiting t_A rather than $t_A + t_1$ after the 2nd rotor-trigger. This makes the time-reversed experiments less sensitive to the problem described below as can be seen from the figure.



Although this procedure is well known, the literature have paid less attention so far to the effective mixing time τ_m^{eff} created by this sequence. It is obvious that τ_m^{eff} is larger than the nominal mixing time and it is not constant throughout the 2D-experiment (since the procedure makes use of the current t_1 which changes during the experiment). In particular, the effect is very strong for short mixing times (τ_m beeing comparable to T_2). To illustrate the effect, we calculated τ_m^{eff} for both rotor- synchronized and time- reversed experiments for nominal mixing times of 0, 10ms, 100ms and 1s. The parameters we have used are: Spinning speed 3kHz, length of cross-polarization 1ms, $\pi/2$=4µs. ni is the number of the t_1-increment which itself was set to Δt_1=31µs. ni=50..150 is necessary to avoid truncation in ω_1 (depending on the line-width of the peaks). The discontinuities of the graphs appear each time when $t_A + t_1$ exceeds $N \cdot T_R$; the step-height is equal to 1/(spinning speed) = 333µs.

The distribution of τ_m^{eff} might lead to unwanted crosspeaks and phase-distortions of the 2D-MAS peaks. Since τ_m^{eff} should be plotted on a logarithmic scale, it is obvious that for short mixing times there is a substantial distribution throughout a single 2D-experiment while for longer mixing times, this effect is less important. It should be mentioned the problem of τ_m^{eff}-distribution does not play any role in 1D-exchange experiments since the t_1 is choosen at a fixed value (for example $t_1 = 0.5 \cdot T_R$, /6/) or it is replaced by a special preparation of the spin-system like TOSS /7/.

/1/ Domberger, Reichert, Garwe, Schneider, Donth, J.Phys.:Cond.Matter **7**,7419(1995)
/2/ DeJong, Kentgens, Veeman, Chem.Phys.Lett. **109**, 337 (1984)
/3/ Hagemeyer, Schmidt-Rohr, Speiss, Adv.Magn.Reson. **13**, 85 (1989)
/4/ Titman, Luz, Spiess, J.Am.Chem.Soc. **114**, 3756 (1992)
Reichert, Olender, Poupko, Zimmermann, Luz, J.Chem.Phys., **98**, 7699 (1993)
/5/ Hagemeyer, Thesis, Mainz 1990
/6/ Gerardy-Montouillout, Malveau, Tekely, Olender, Luz, J.Magn.Reson. (submitted)
Reichert, Zimmermann, Poupko, Luz, J.Magn.Reson. (submitted)
/7/ Yang, Schuster, Blümich, Spiess, Chem.Phys.Lett., **139**, 239 (1987)

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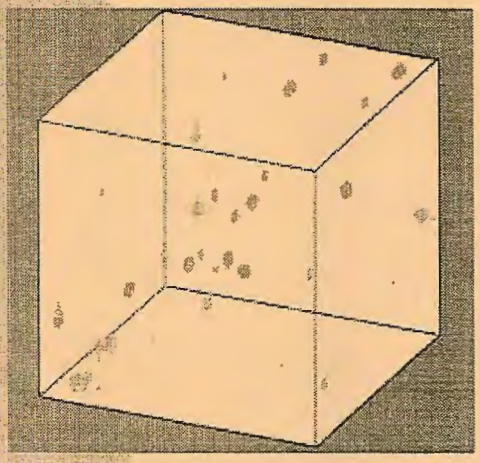

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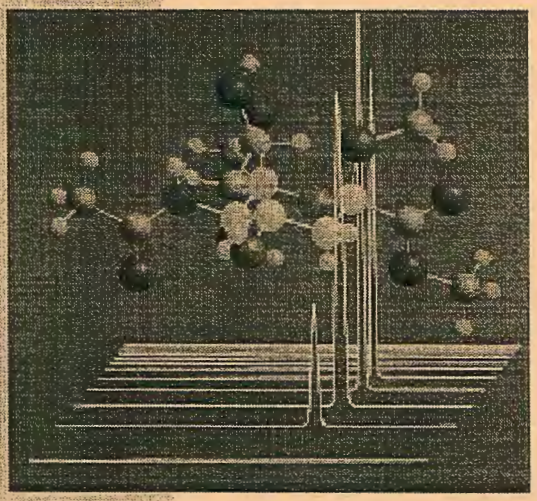

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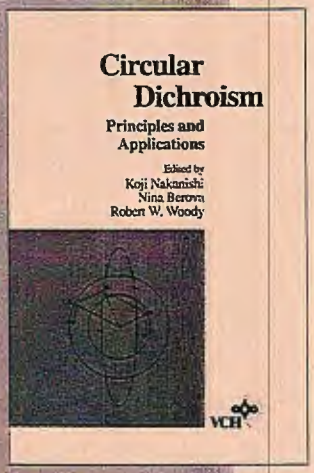

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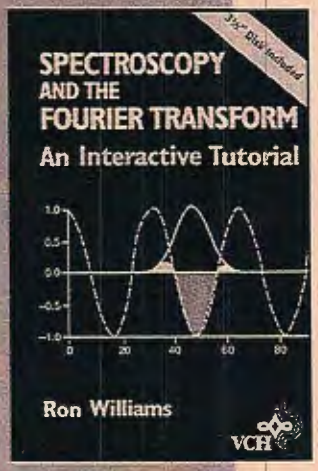

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Dr. B.L. Shapiro
The NMR Newsletter
966 Elsinore Court
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August 22, 1996 (received 8/27/96)

Lability of Polyanion-Gelatin Binding Investigated with PGSE-NMR

Dear Dr. Shapiro:

Polyanions behave as excellent viscosifiers for gelatin solutions and are used in various film and paper products as coating aids. Despite its overall negative charge above the isoelectric point (pH_{iso}), gelatin binds to high molecular weight polyanions to form soluble complexes. Pulsed-gradient spin-echo (PGSE) NMR was employed to investigate the self diffusion of gelatin in solutions of gelatin / poly(styrene sulfonate) (NaPSS) and gelatin / poly (2-acrylamido-2-methylpropane sulfonate) (NaPAMS). Although the number of negative charges per chain segment is the same for each type of polyanion, they provide significantly different rheological behavior in gelatin solutions. The basis for this is not well known.

One advantage of PGSE NMR is the ability to precisely control the time allowed for diffusion during the measurement. We varied this diffusion time (Δ) between 500 and 2000 ms and collected the gelatin signal attenuation (which is proportional to the self-diffusion of gelatin) for both solutions.

Little to no difference is observed for the NaPSS data as a function of Δ. The NaPAMS data, however, bears a striking contrast. The attenuation behavior for gelatin in the gelatin/NaPAMS solution approaches that of free gelatin as Δ increases. This behavior is interpreted in the context of chemical exchange. The binding time for gelatin to NaPAMS is much *shorter* than that to NaPSS. A model[1] was established to describe the signal attenuation to include this exchange and the data fitted accordingly. This exchange phenomenon provides an additional link to the description of the rheology.

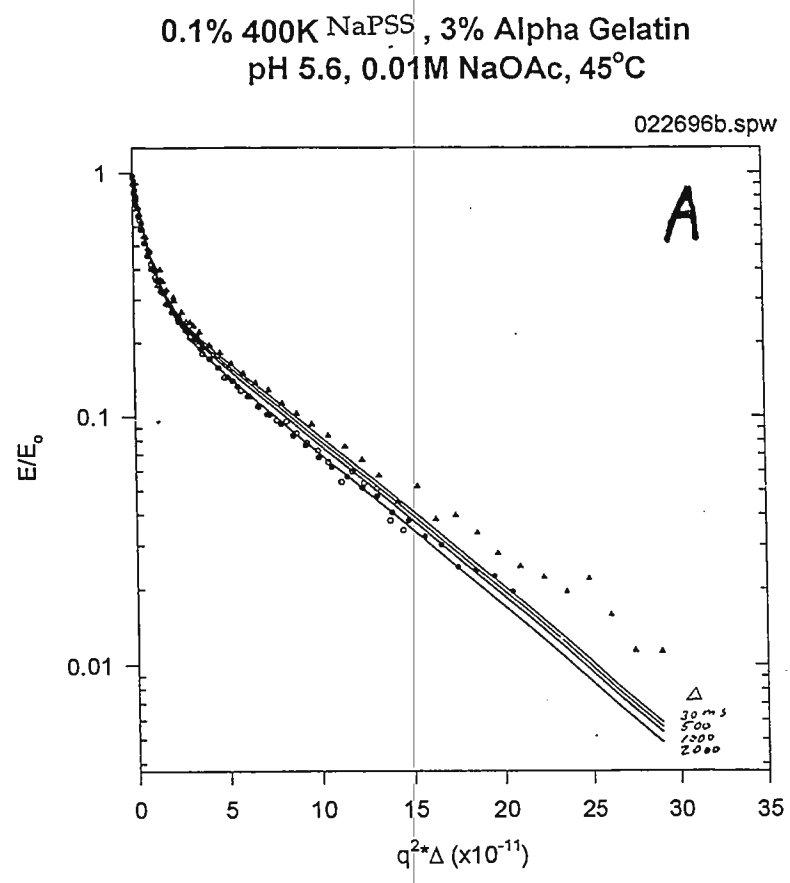
It is important to note a few points. The T_1 and T_2 behavior of the gelatin in these solutions was investigated and found to be similar to that of solutions without polymer. Narrow molecular weight fractions of gelatin (called alpha gelatin) and polymers were used to eliminate complications due to polydispersity. Two to three decades of attenuation was collected for sufficient range in data. The polyanion concentration for these solutions is below the overlap concentration (c*).

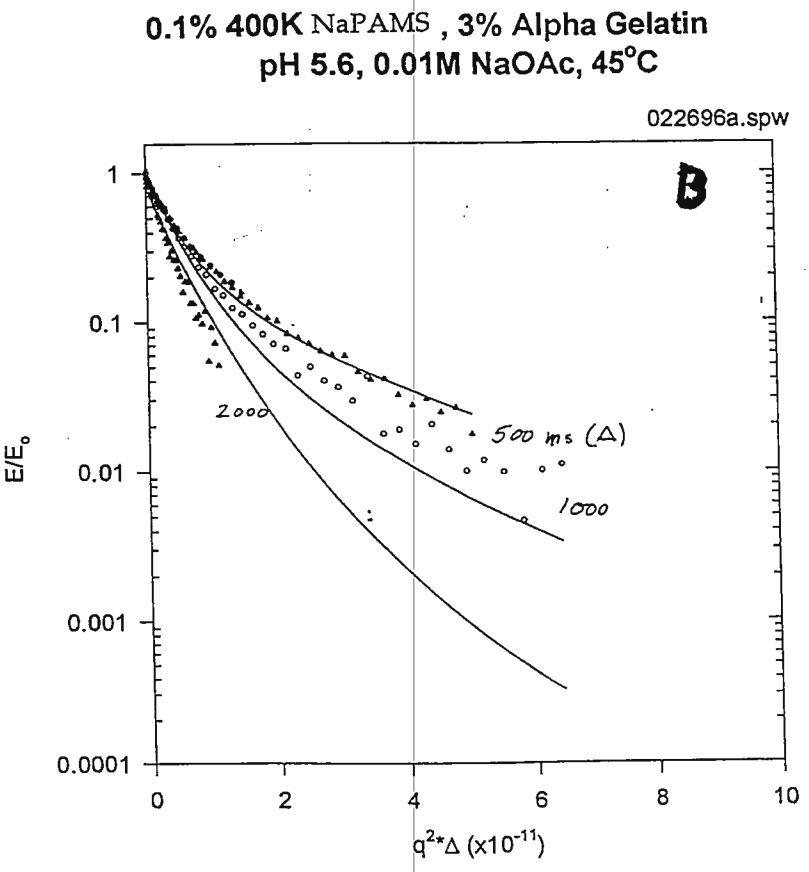
Sincerely,

Brian Antalek

Brian Antalek

1. For further information consult: (a) Callaghan, P.T. *Principles of Nuclear Magnetic Resonance Microscopy;* Clarendon Press: Oxford, 1991; pp 405-407. (b) Johnson, Jr., C.S. in *Nuclear Magnetic Resonance Probes of Molecular Dynamics*, Robert Tycho, ed.; Kluwer Academic Publishers: Boston, 1994; pp 476-478.





PGSE NMR gelatin signal attenuation plots for two aqueous solutions are represented. Data is obtained for three values of diffusion time (Δ): 500, 1000, and 2000 ms. The curves are calculated from a model incorporating chemical exchange of gelatin between two sites, bound to the polymer and free. The results of the fitting are given below in the table. These attenuation functions plotted in semilog format reveal two decay time constants. The fast time constant is interpreted as the gelatin which is free in solution. The slower component is that due to the gelatin bound to the much larger polyanion-gelatin complex. Clearly a difference in the behavior of the gelatin diffusion in both solutions is seen as a function of Δ.

Fitting results:

model parameter	(A) gel/NaPSS	(B) gel/NaPAMS
k (s^{-1})	<0.1	4.2
R	0.55	1.4
D_q (10^{-11} m^2/s)	0.14	0.3
D_p (10^{-11} m^2/s)	1.4	5.0

Parameter descriptions:

k = rate constant for gelatin exchanging *from* complex

R = molar ratio of bound gelatin to free gelatin

D_q = diffusion coefficient for bound gelatin (polymer-gelatin complex diffusion)

D_p = diffusion coefficient for free gelatin

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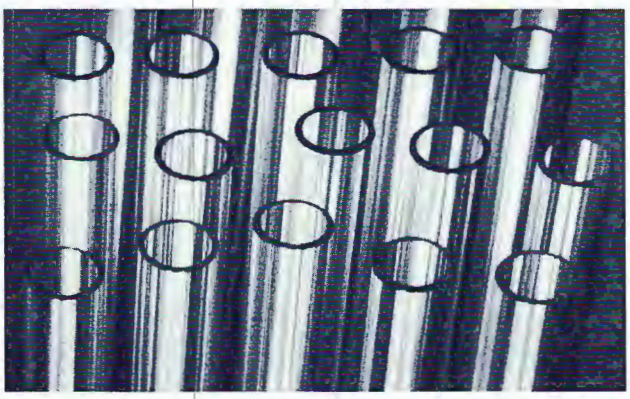

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(Slightly revised October 1996)

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

B. L. Shapiro
October 1996

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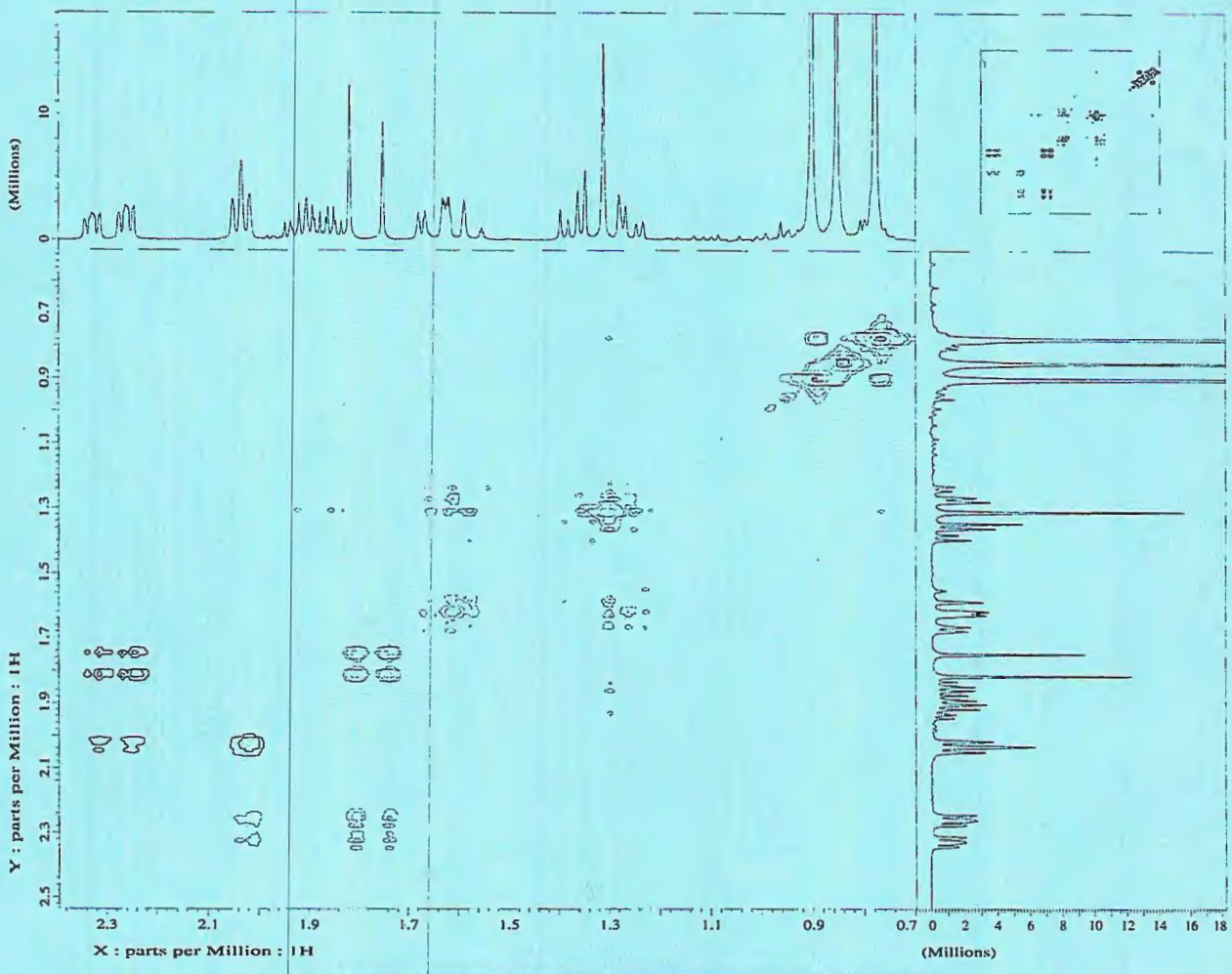

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