THE
NMR
NEWSLETTER

No. 465

June 1997

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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

39th Rocky Mountain Conference on Analytical Chemistry, Denver, Colorado; NMR Symposium, **August 4-7, 1997**: Contact: J. P. Yesinowski, Code 6120, Naval Research Laboratory, Washington, DC 20375-5342; 202-767-0415; fax 202-767-0594; email yesinowski@nrl.navy.mil. See Newsletter 458, 8.

8th Annual Chemagnetics Workshop on Solid State NMR, Estes Park and Ft. Collins, CO, **August 8-9, 1997**; Contact: J. Frye, Otsuka Electronics USA, Inc., 2607 Midpoint Dr., Ft. Collins, CO 80525; (800) 468-7852; Fax. (970) 484-0487; email jimf@chemagnetics.com; See Newsletter 465, 36.

Fourth International Meeting on Recent Advances in Magnetic Resonance Applications to Porous Media, Trondheim, Norway, **Aug. 31 - Sep. 3, 1997**; Contact: John J. Attard, SINTEF Unimed MR-Center, N-7034 Trondheim, Norway. Tel: +47 73 59 89 25; Fax: +47 73 99 77 08; Email:john.attard@unimed.sintef.no.

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

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

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

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

Additional listings of meetings, etc., are invited.

ROSKILDE UNIVERSITY

Professor Poul Erik Hansen, Department of Life Sciences and Chemistry

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Professor B.L.Shapiro
The NMR Nesletter
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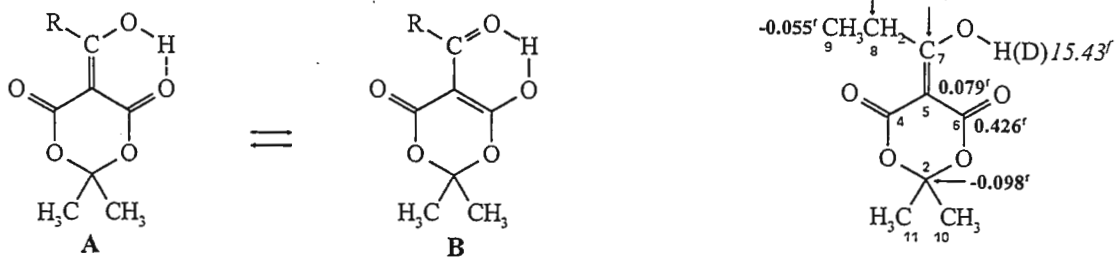
US

April 23 1997 (received 4/30/97)
DATE OUR REFERENCE YOUR REFERENCE

Deuterium Isotope Effects on ^{13}C Chemical Shifts of Ethyl Groups to Probe Tautomerism

Dear Professor Shapiro

For quite a while we have been studying deuterium isotope effects on chemical shifts. Occasionally we come across compounds that are hard to categorize. One recent example is acyl derivatives of Meldrum acids (Fig. 1). This compound exists fully on the enol form, but the question is if it is A or B or an equilibrium between these.



Both solutions are less than good. A has an exocyclic double bond. For B the ester function is enolized. From the isotope effect on chemical shifts (shown in brackets) it is easy to see that A is dominant. However, the very unusual isotope effects at both C-2 and at C-6 indicates tautomerism. A great help is to make R = CH_3CH_2-. In that case we observe a large isotope effect at the CH_2 carbon and a relatively large and **negative** isotope effect at the CH_3 carbon, a carbon four bonds away. Such effects are usually small and positive in aliphatic systems. The negative effect seen at the ethyl derivative of acyl Meldrum acids most likely occurs because of an equilibrium contribution to the isotope effect and therefore supports a tautomeric equilibrium. Ethyl groups can be used in a similar way in other cases. Isopropyl groups etc. are less useful probably because the difference in chemical shifts between the two tautomers are less.

We are strongly interested in similar cases involving enolization of ester or amide groups.

Yours sincerely

Simon Bolvig Poul Erik Hansen

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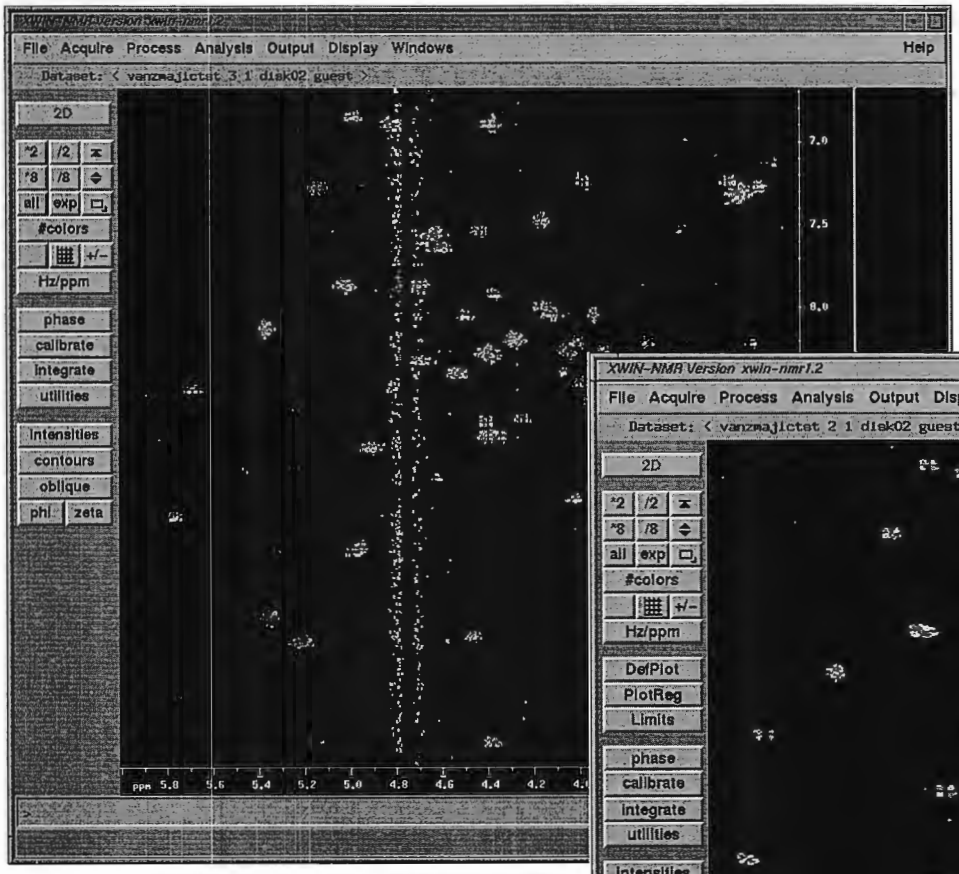

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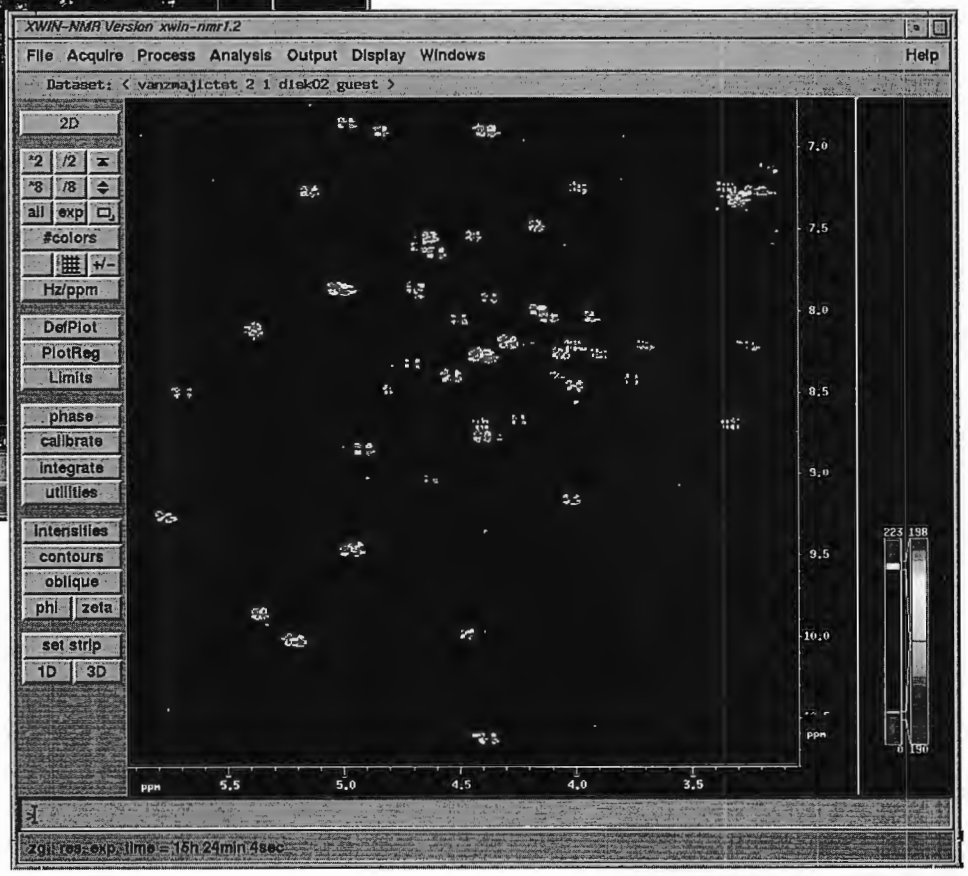
Both experiments were acquired on the Bruker AVANCE 500 equipped with a 5 mm inverse triple resonance (TXI) probe with GRASPTM-III. The sample is 1.5 mM BPTI in 90% H_2O/10% D_2O. The experiment is DQF-COSY.



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!

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Zürich, May 5th, 1997
(received 5/12/97)
2515

Prof. Dr. Bernard L. Shapiro
The NMR Newsletter
966 Elsinore Court
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U.S.A.

χ_1 Conformational Disorder of the Valine Side Chain in Solid Antamanide

Dear Barry,

We have recently investigated the cyclic decapeptide, antamanide, [-Val[1]-Pro[2]-Pro[3]-Ala[4]-Phe[5]-Phe[6]-Pro[7]-Pro[8]-Phe[9]-Phe[10]-] in the solid state and have found interesting environmental effects on the side-chain conformation of Val[1] and on the methyl-group rotation [1]. With the help of techniques to improve the spectral resolution of magic angle spinning (MAS) NMR spectra of fully ^{13}C- and ^{15}N-labelled biomolecules, namely, the TPPM ^{1}H decoupling sequence [2] and homonuclear ^{13}C-^{13}C J-decoupling [3], we were able to make a full resonance assignment. Additionally, we were able to identify two sets of peaks for the valine residue, as illustrated in Figure 1. These two sets of resonances were attributed to the presence of two of the three possible rotamers: I (χ_1=-60°) and II (χ_1=180°) and a stereospecific assignment of the two methyl groups was made for the two rotamers.

The χ_1 conformational static disorder found for Val[1] prompted us to also look at the methyl group ^{13}C T_1 relaxation times. We found that in conformation II of valine, the two methyl groups $Val^1\ C_{II}^{\gamma 1}$ and $Val^1\ C_{II}^{\gamma 2}$ rotate with similar jump time constants of 61.3 and 89.0ps, respectively. The other conformer, however, shows a more individual behavior with 31.2ps for $Val^1\ C_I^{\gamma 1}$ and 6.7ps for $Val^1\ C_I^{\gamma 2}$ (see Figure 2). It is interesting to note that though $Val^1\ C_I^{\gamma 1}$ and $Val^1\ C_{II}^{\gamma 2}$ occupy nearly the same position in space, their motional behavior is different by a factor 2.9. The small jump time constant of $Val^1\ C_I^{\gamma 2}$, constrasting with the values of all other methyl groups, indicates that this methyl group must be in a unique surrounding. Putting all of this information together, we propose a possible model which rationalizes the data, shown in Figure 2. The fact that rotamer III is not found can be explained by a strong steric hindrance in a large angular range (on the left of Figure 2) allowing only the proton to be close to that region. The three regions of lower steric hindrance are asymmetric and not exactly differing by 120° in their χ_1 angles. In rotamer I, the methyl group $C^{\gamma 2}$ is situated in a relatively wide potential energy minimum with little hindrance, leading to τ_{jump} = 6ps, being fixed there by the more restrained methyl group $C^{\gamma 1}$ with τ_{jump} = 31ps. The restraints for rotamer II are more pronounced leading to longer values of τ_{jump} of 61ps and 89ps. It is impossible at this point to further specify the nature of the restraining interactions. It is well conceivable that, among others, also the interactions with the water molecules could be relevant.

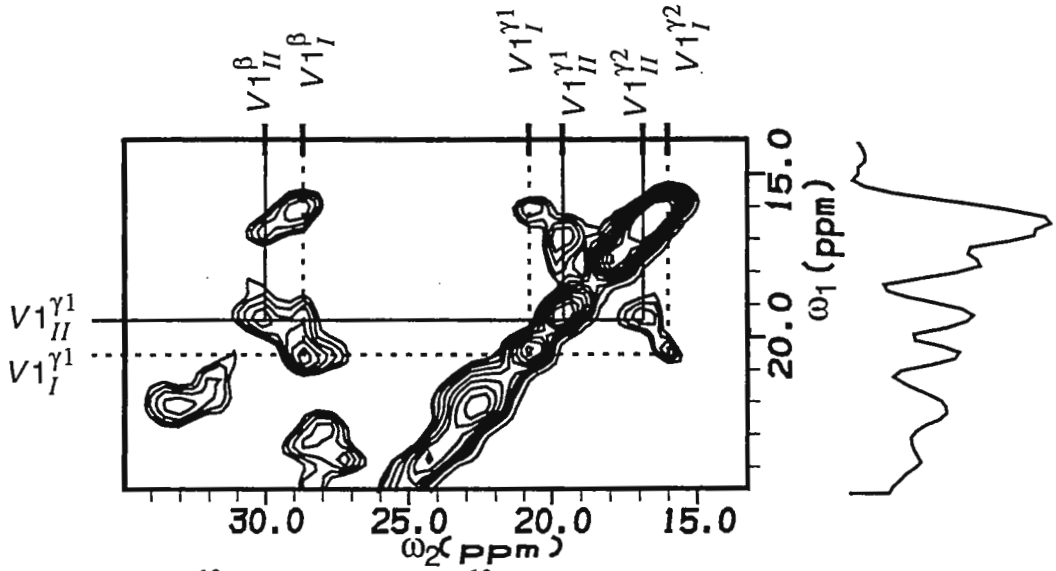

Figure 1. Part of 2D ^{13}C spectrum of fully ^{13}C-labelled antamanide, recorded at 100 MHz carbon frequency and room temperature, displaying the connectivities for the two valine rotamers. The spectrum, where homonuclear J-decoupling is applied in ω_1, was acquired with a mixing time of 32 ms and a spinning speed of 15 kHz.

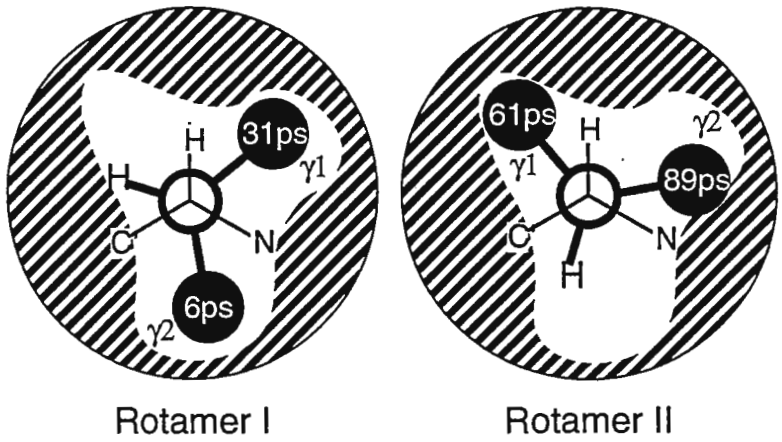

Figure 2. Pictorial representation of the proposed model rationalizing the relaxation data of the two valine rotamers in antamanide in the solid state. The methyl groups are indicated as filled black circles, their rotational correlation times are also given. The hindering potential is indicated by the hatched areas.

Best wishes,

S. K. Straus T. Bremi R.R. Ernst

[1] Straus, S.K., Bremi, T. and Ernst, R.R. (1997) *J. Biomol. NMR*, in press.
[2] Bennett, A.E., Rienstra, C.M., Auger, M., Lakshmi, K.V. and Griffin, R.G. (1995) *J.Chem.Phys.* **103**, 6951-6958.
[3] Straus, S.K., Bremi, T. and Ernst, R.R. (1996) *Chem. Phys. Lett.*, **262**, 709-715.

Prof. Dr. F. H. Köhler
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The NMR Newsletter
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May 15, 1997
(received 5/20/97)

Temperature Measurement in Solid-State NMR Spectroscopy

Dear Dr. Shapiro,

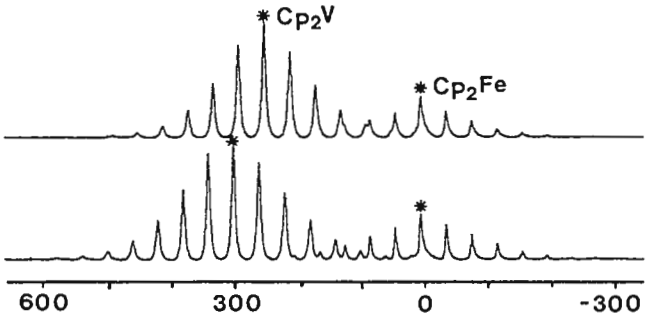
you have to set up experiments yourself in order to believe what careful textbooks on practical NMR spectroscopy tell you about the difficulties to determine the precise temperature at which the data was obtained. This is still true even though dynamic NMR spectroscopy in solution is a fascinating topic since the mid-fifties. Improvements are still being worked out; a recent example is the liquid-medium thermo jacket of W. Dietrich and his colleagues at the University of Bochum which allows to perfectly control the temperature of the entire rotating tube.

In solid-state NMR spectroscopy things are more complicated because, for a given temperature set at the controller, the sample temperature depends (among other things) on the material of the rotor, its weight and diameter, and on the MAS frequency. Clearly, a precise and convenient temperature measurement is possible when the thermometer is placed in the rotor together with the sample. This can be realized by using NMR shift thermometers.

Imagine you are doing a solid-state NMR temperature series with one of the common nuclei like ^{31}P or ^{13}C. Then it would be most useful to have a shift thermometer based on that nucleus; otherwise repeated retuning of the system is necessary. A good signal-to-noise ratio and temperature resolution of the shift thermometer would be also desirable. These conditions make the choice of a suitable sample difficult.

At this point we reasoned that we should try a ^{1}H NMR shift thermometer, because standard MAS probe-heads dispose of a decoupling channel. It turned out that the paramagnetic compound vanadocene ($(C_5H_5)_2V$, S = 3/2) is well suited. Some advantages are: (i) It is commercially available. (ii) It has only one signal with very good S/N, of course. (iii) The signal shifts 1.0 ppm per °C, and its position can be determined precisely (typically better

than 0.1 ppm). (iv) The spectra of interest are not disturbed by the shift thermometer, because the only other NMR signal of vanadocene is that of ^{13}C which appears at -651 ppm (298 K), i.e., far outside the usual range. (v) The temperature may be measured as a signal-shift difference by using (isostructural) ferrocene as an internal diamagnetic standard. How the spectra of this shift thermometer look like is shown in the Figure. The topic has been worked out in more detail including the ^{13}C NMR signal of vanadocene as a thermometer and will be published in *Magn. Reson. Chem.* There we also refer to the very stimulating work of many other spectroscopists who previously offered solutions to the problem of measuring the temperature in MAS NMR spectroscopy.



^{1}H MAS NMR spectra of a mixture of vanadocene and ferrocene (4/1) at 370 K (top) and 305 K (bottom). Scale in ppm; rotational frequency 12 kHz.

Sincerely yours,

(F. H. Köhler)

(X.-L. Xie)

When you need problem-solving power in an NMR probe,

Switching between combinations of nuclei for triple resonance experiments, such as $^{1}H\{^{13}C/^{15}N\}$ and $^{1}H\{^{13}C/^{31}P\}$, is one benefit of Varian's new $^{1}H\{^{13}C/^{15}N/^{31}P\}$ 5 mm PFG Penta Probe. This versatile probe is ideal for biomolecular protein and nucleic acid experiments.

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Professor B.L. Shapiro, Publisher
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May 10, 1997
(received 5/13/97)

BIG-BIRD : New Pulse Sequence Element

Dear Barry:

Recently we have succeeded in generalizing the popular pulse sequence elements of BIRD and TANGO which appear as special cases of our new pulse sequence element dubbed BIG-BIRD (Bilinear Independent Gyrations BIRD) (*J. Magn. Reson.* **125**, 202-206, 1997). It also generalizes the pulse sequence element BANGO since not only independent and arbitrary flip angles but also arbitrary phases can be selected for protons attached/not attached to an NMR active heteronucleus.

BIG-BIRD is a four-pulse sequence element, as shown in Figure 1, and does not necessitate the use of selective rf pulses. The flip angles and phases of the first and last rf pulses (α_μ, γ_η) on the proton channel are calculated in such a way that the cumulative effect of the pulse sequence element generates the desired flip angles and phases for I and I{S} spin systems:

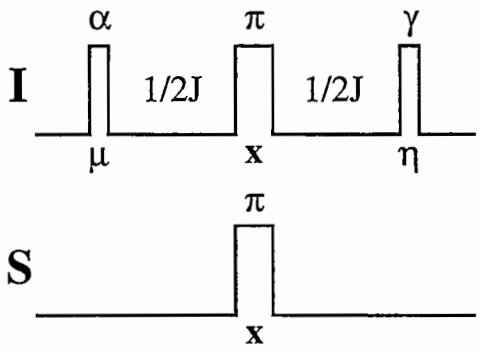

Figure 1

$$\begin{cases} I : \left(\beta^{I}\right)_{\varphi^{I}} \\ I\{S\} : \left(\beta^{IS}\right)_{\varphi^{IS}} \end{cases}$$

Figure 2 illustrates the enhanced capability obtained with BIG-BIRD in being able to arbitrarily and independently vary the phases and flip angles for the two classes of protons. A series of one-dimensional spectra was recorded for different combinations of desired flip angles (β^I, β^{IS}) and phases (φ^I, φ^{IS}) for I and I{S} spin systems, respectively. These spectra were recorded on a sample containing 1% iodomethane with about 60% ^{13}C-labeling in $CDCl_3$. All spectra were phase-corrected with the same zero- and first-order phase corrections and plotted with the same scaling factor.

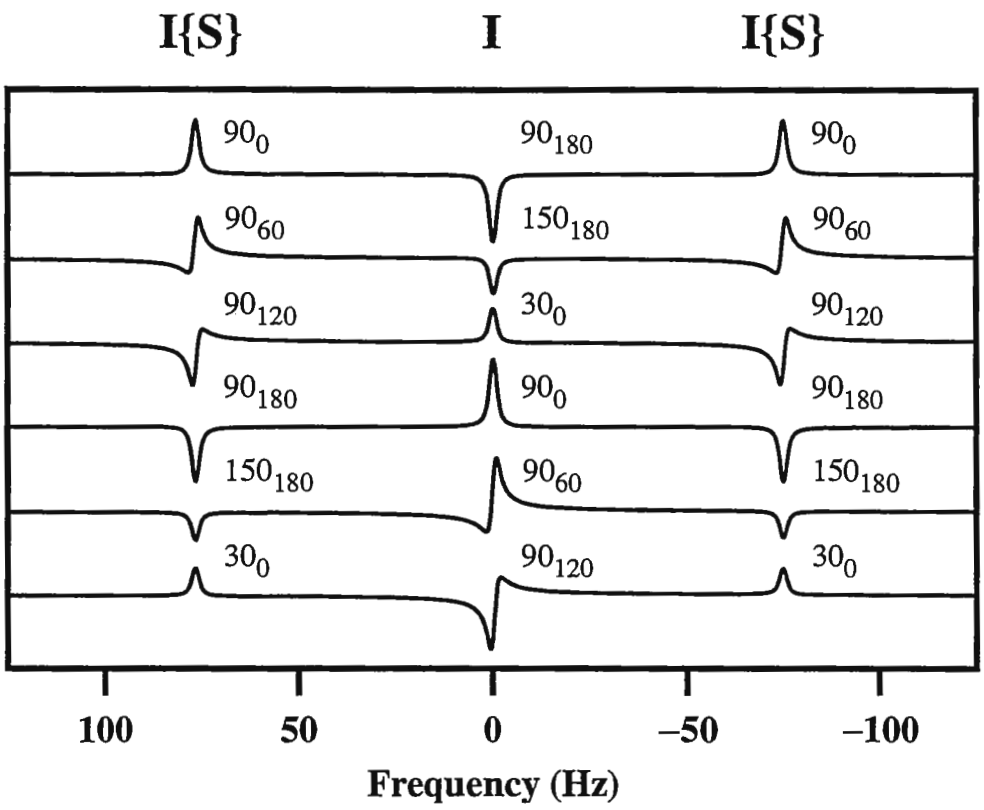

Figure 2

BIG-BIRD opens up a new avenue for simultaneously applying independent phase cycles to different classes of protons. We are currently evaluating applications which could exploit this new feature.

Sincerely yours,

Jacques Briand

Jacques Briand & Ole W. Sørensen (Carlsberg Laboratory, DK-2500 Valby, Denmark)

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Cornell University

Biotechnology Program
130 Biotechnology Building
Ithaca, NY 14853-2703 USA
(607) 255-2300

Porcupine Surgery!

April 24, 1997
(received 4/28/97)

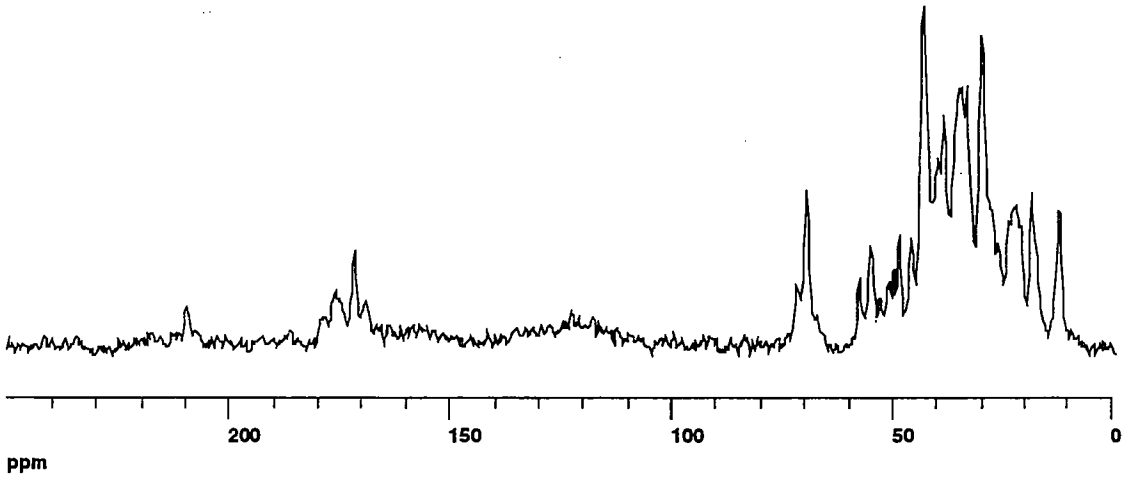
Dr. B. L. Shapiro
The NMR Newsletter
966 Elsinore Court
Palo Alto, CA 94303

Dear Barry,

Recently a rather unusual sample passed through our hands which we thought might be of interest to NMR Newsletter readers. Our program was contacted by Dr. Prowten on behalf of a prehensile tail porcupine at the Buffalo Zoo which had some intestinal difficulties. The porcupine in question apparently goes through this problem every year, and has it resolved surgically by removal of a mass known as a bezoar. Webster's dictionary defines this as:

Bezoar \Be"zoar\, n. [F. b['e]zoard, fr. Ar. b[=a]zahr, b[=a]dizahr, fr. Per. p[=a]d-zahr bezoar; p[=a]d protecting + zahr poison; cf. Pg. & Sp. bezoar.] A calculous concretion found in the intestines of certain ruminant animals (as the wild goat, the gazelle, and the Peruvian llama) formerly regarded as an unfailing antidote for poison, and a certain remedy for eruptive, pestilential, or putrid diseases. Hence: Any antidote or panacea.

While we did not make any attempt to evaluate the material's value as an antidote for poison, we did collect a ^{13}C CP/MAS spectrum. The spectrum was acquired on a home-built spectrometer with a Doty Scientific H-X-Y narrow-bore probe operating at 90.6 MHz for ^{13}C and is the result of 1024 accumulations. The spinning speed was 8 kHz and the proton decoupling field strength was 90 kHz.



We are pleased to report that the porcupine is doing well after its surgery. While we are not eagerly awaiting an outbreak of a pestilential or putrid disease, we wonder if it might help the Ithaca weather.

Please credit this contribution to Cathy Lester.

Sincerely,

Carl Michal

L. W. Jelinski

Carl Michal Lynn Jelinski

Mobil Technology Company

MARKETING, REFINING AND CHEMICAL
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April 30, 1997
(received 5/5/97)

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

Simplified Sample Preparation for Improved Sensitivity and Resolution in ^{1}H MAS NMR of Zeolites

Zeolites contain several distinct hydrogen species. In particular, hydrogen atoms are associated with framework aluminum sites or acidic Al(OH)Si sites, non-framework aluminum sites, silanols, ammonium ions, and adsorbed water. ^{1}H NMR has the potential to study these different protons and provide information on zeolite acidity, structural defects, and dealumination/realumination mechanisms. However, since the protons of interest are relatively dilute, ^{1}H NMR studies of zeolites are complicated by adsorbed water. Careful sample dehydration is required to remove the water resonance(s) that would otherwise mask the resonances of interest. In addition to sample preparation considerations, line broadening by chemical shift anisotropy and dipolar couplings complicates ^{1}H NMR studies. Substantial improvement in resolution on dehydrated zeolites can be achieved using CRAMPS and MAS techniques.

Sample preparation procedures commonly reported in the literature for both ^{1}H MAS and CRAMPS studies include dehydration of the sample at high temperature and reduced pressure followed by flame sealing the sample in a glass ampoule. The sealed ampoule prevents sample rehydration and can be inserted into an NMR rotor for analysis. The two major drawbacks to this approach are the difficulty in uniformly sealing the glass ampoule and that the glass ampoule limits the rotation rates to about 2-3 kHz. We have developed a greatly simplified and effective procedure that involves dehydrating the material of interest *directly* in a ZrO_2 MAS rotor, without the need of glass ampoule or flame seal. The detailed procedures are: 1) sample is packed tightly into a 4 mm (o.d.) ZrO_2 rotor, 2) the uncapped rotor is placed in a 10 mm (o.d.) glass tube fitted with a J. Young valve (these tubes and valves are commercially available from Wilmad), 3) the assembly is connected to a vacuum line and mounted in a tube furnace, 4) sample is dehydrated under vacuum ($<10^{-4}$ torr) at a rate of 1°C/min to 70°C, held at 70°C for 1 hour, then ramped at 3°C/min to 400°C, held at 400°C for 10 hours, after which the furnace is shut off and the sample allowed to cool back to room temperature under vacuum, 5) the J. Young valve is closed under vacuum, and 6) the tube assembly containing the rotor is removed from the vacuum line and transferred to a dry box where the rotor is capped with the standard Kel-F cap.

Preparing the sample in this manner has several advantages over other methods. Since there is no flame or epoxy seal the sample can be easily recovered and the rotor, cap, and J-Young valve/tube assembly can be reused. Several samples can be dehydrated simultaneously in the valve/tube assembly. Of particular importance is that more sample can be packed into the rotor because no volume is taken up by the glass ampoule, resulting in improved sensitivity. More importantly, we routinely record ^{1}H spectra on samples prepared directly in the rotor at a spinning speed of 15 kHz and show significant enhancement in spectral resolution compared to previously published work on samples prepared in glass ampoules where rotor spinning speeds are limited to ~2-3 kHz. This is due to the effective averaging of the CSA and dipolar couplings at the higher spin rates. Our experiences with this procedure show that the cap makes a tight seal with the rotor and prevents rehydration of the sample. To further reduce the risk of water adsorption, samples are stored in the glove box and we typically use N_2 as the spinning gas.

Gordon J. Kennedy

Gordon J. Kennedy

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14 May, 1997
(received 5/16/97)

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

A Direct Solution for Resolving Highly Overlapped Spectra Using PGSE NMR

Dear Dr. Shapiro:

We have recently reported [1,2] the application of a multivariate statistical analysis called generalized rank annihilation method (GRAM) for use with pulsed gradient spin echo (PGSE) NMR data. This method, which is based upon the work of Kubista [3], provides a direct solution and is, therefore, not dependent upon iterative fitting of multiexponential functions. Kubista showed that an exact analytical solution may be obtained when two data sets are available with proportional signal intensities.

$$\mathbf{A} = \mathbf{CP}$$
$$\mathbf{B} = \mathbf{C}\beta\mathbf{P}$$

A and **B** are data matrices of size *cv*; *c* is the number of spectra and *v* is the number of spectral data points. **C** (size *cn*) and **P** (size *vn*) are the signal intensities and pure spectra matrices of *n* components. The matrix β is a diagonal matrix (size *nn*) and describes a scaling factor between **A** and **B**.

PGSE NMR data sets fit the bill. The experiment involves collecting several spectra while varying the applied magnetic field gradient, g. The signal from the pure components decays as a decreasing exponential depending upon their diffusivity in the following manner.

$$\text{Signal}_i = e^{-Di \cdot f(g)}$$

where i represents an individual pure component, D is the diffusivity, and f(g) is a function of the gradient area and depends upon the pulse sequence used. (Actually, one needs to vary g^2 in equal steps for the analysis). Now, we split the data set into two, **A** and **B**. This is simply done by the following: **A** comprises spectra 1 to (n-1) and **B** comprises spectra 2 to n. Since the signal depends upon an exponential, there is naturally a proportional scaling factor which relates **A** and **B**. Refer to the table below. We can therefore solve for β, **C** and **P**. We call this direct exponential curve resolution algorithm or DECRA. (I know, ... another acronym!)

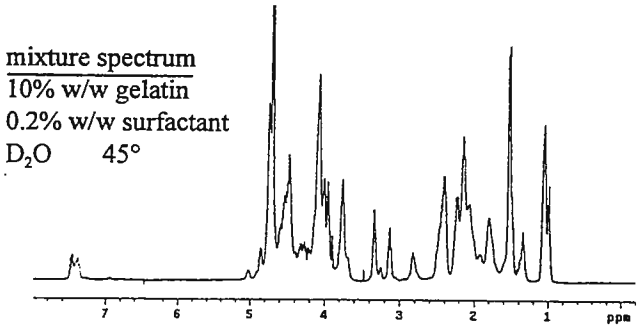
Representative signal decay data for a sample containing 2 components

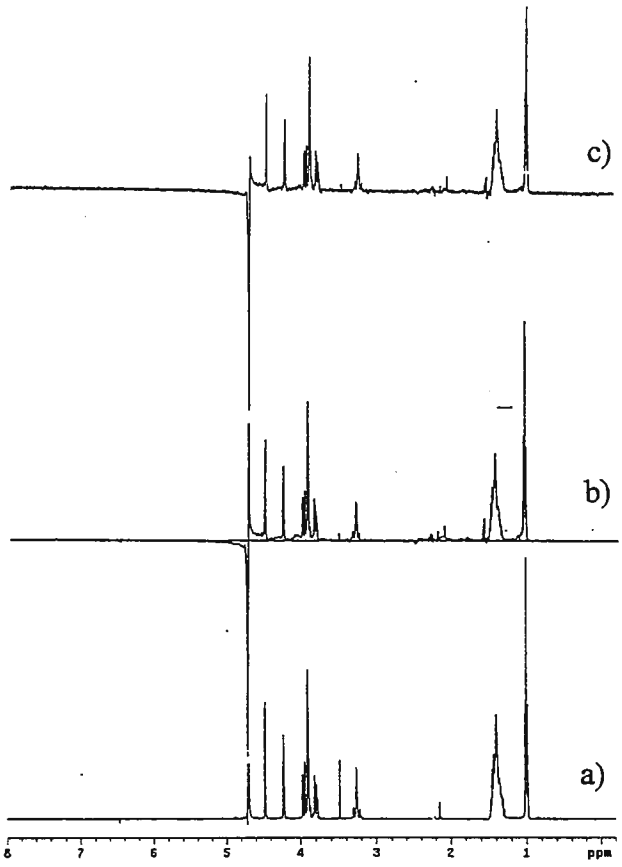
spectrum #	component data		**A**		**B**	
1	27	8	27	8	9	4
2	9	4	9	4	3	2
3	3	2	3	2	1	1
4	1	1				

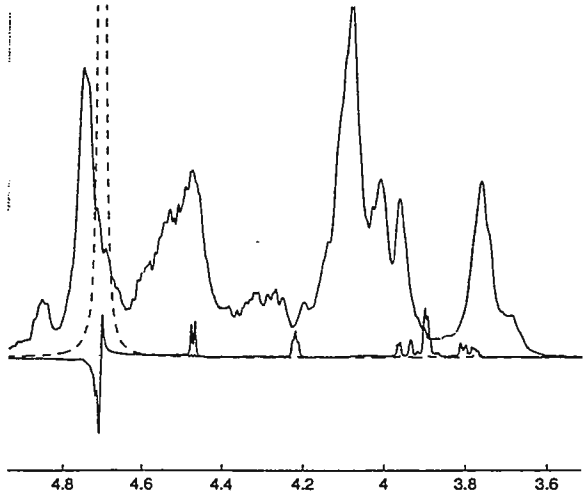
$$\beta = \begin{pmatrix} \frac{1}{3} & 0 \\ 0 & \frac{1}{2} \end{pmatrix}$$

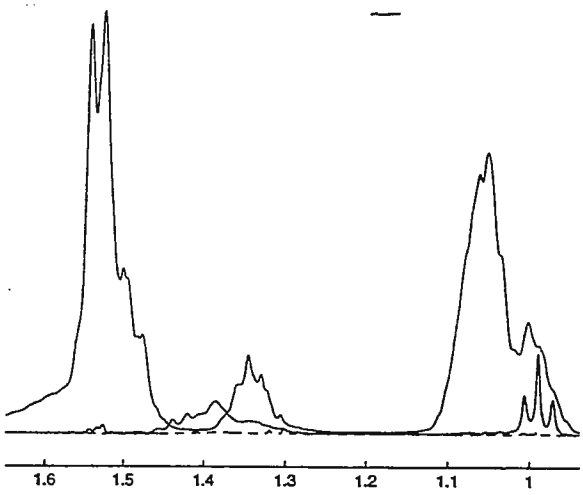
GRAM has previously been applied to PGSE NMR data, [4] but the data was not split in such a manner. Instead, a more elaborate data acquisition scheme was used which compromised the lineshape.

An example is shown that represents a nonionic surfactant, (di-(C6-Glu); $(C_5H_{11})_2C(CH_2NHC[CHOH]_4CH_2OH)_2$) at 0.2% w/w in an aqueous gelatin (10% w/w) solution. The data was acquired on a Varian Inova 400 instrument with a standard PFG accessory. Despite the high overlap, DECRA is able to successfully resolve all three components and their respective diffusivities with minimal artifacts. The data set is comprised of 30 spectra containing 9063 real data points each. The algorithm runs as a MATLAB script on a Pentium-200 and windows 3.1. The processing requires input of both the range of spectra and number of components, *n*. For this example, processing time is less than 5 seconds! The entire spectrum is used in the analysis, even the noise! Furthermore, since it does not require an iterative fitting procedure, only a small number of spectra are needed for the analysis. The resolved surfactant spectrum is shown for the analysis of only 4 spectra (i.e. **A** comprises spectra 1, 5, 9and **B** comprises spectra 5, 9, 13). The result is very comparable to the analysis of the full data set.









Expansions of the spectral composition of all three resolved components at $g^2 = 0$.

Resolved surfactant spectrum using DECRA. a) reference
b) spectra 1-30 used in the analysis c) spectra 1, 5, 9, 13 used

[1] B. Antalek, W. Windig, J. Am. Chem. Soc. 118, 1996, 10331-10332.
[2] W. Windig, B. Antalek. Chemom. Intell. Lab. Sys. (in press)
[3] M. Kubista, Chemom. Intell. Lab. Sys. 7 (1990) 273-279.
[4] D. Schulze, P. Stilbs. J. Magn. Reson. Ser A 105, 1993,54-58.

Sincerely,

Brian Antalek
bantalek@kodak.com

Willem Windig
windig@kodak.com

OXFORD

Technical Specifications

200 - 600MHz Narrow Bore NMR Magnet Systems

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

Advanced Design and Manufacture

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

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

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- All Oxford Instruments magnets feature a virtually unlimited lifetime (with proper maintenance and cryogen service)

See reverse for full technical specifications

Specifications

Specification	System Type								
Magnet	**200/54**		**300/54**		**400/54**	**500/51**		**600/51**	
Operating Field (Tesla)	4.7		7		9.4	11.7		14	
NMR Operating Frequency (MHz ^{1}H)	200		300		400	500		600	
Field Stability (Hz/hour ^{1}H)	<2		<3		<8	<10		<10	
5 Gauss Stray Field Contour						**Standard**	**Shielded**	**Standard**	**Shielded**
Axial (Metres)	1.75		2.2		2.8	3.5	1.8	4.0	2.5
Radial (Metres)	1.5		1.7		2.2	2.8	1.3	3.2	1.75
Cryostat	**Standard**	**Compact**	**Standard**	**Compact**					
Standard Cryostat Minimum Helium Refill Interval (Days)	235	80	235	80	183	150		150	
Standard Cryostat Helium Refill Volume (Litres)	79	26	79	26	62	52		80	
Year Hold Cryostat Option Available	✓	X	✓	X	✓	X		X	
Nitrogen Refill Interval (Days)	14	14	14	14	14	17		18	
Nitrogen Refill Volume (Litres)	61	32	61	32	61	84		131	
Nominal Room Temperature Bore Diameter (mm)	54	54	54	54	54	51		51	
Minimum Operational Ceiling Height (Metres)	2.9	2.5	2.9	2.5	2.9	3.1		3.4	
System Weight (kg) Including Cryogen's	315	120	325	133	380	533		1100	

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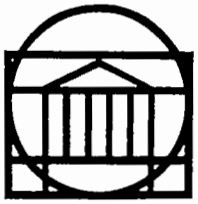
Version: 003 Issue: B May 1997



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

(received 5/22/97)
May 19, 1997.

re: Magnetic Relaxation Dispersion of Aqueous Ionic Solutes

Dear Barry,

Recently, we reported to you our construction of a novel instrument that makes use of two independent and shielded magnets: a 7T superconducting solenoid, and a 4- inch electromagnet producing a variable field from 0 to 1.6T. Pneumatically shuttling a sample between these two fields permits direct detection of the magnetic field dependence of the nuclear spin-lattice relaxation rate, or nuclear magnetic relaxation dispersion (MRD), by polarizing the spins in the high field, rapidly shuttling the sample to the low field for a variable time, and returning the sample to the high field where the remaining magnetization is detected with high resolution and sensitivity. Interpreted in terms of relative diffusion, chemical binding and molecular rotation, the MRD profile can be related to the various fluctuations that drive magnetic relaxation, and as such is a powerful probe of liquid phase molecular dynamics. Having now invented a suitable high-field multi-nuclear probe and sample containment system, we have recorded MRD profiles of ionic solutes in aqueous phases containing proteins, phospholipid vesicles and paramagnetic relaxation agents.

In solutions containing paramagnetic compounds, the nuclear spin may relax by scalar Fermi contact and dipolar couplings with the unpaired electrons. Scalar coupling can only be effective with the formation of a chemical bond, and is thus modulated by the lifetime of the complex. Alternatively, relaxation via dipole-dipole interactions is constrained by the separation of the spin centres. Long-range intermolecular contributions are correlated by the low-frequency relative translation of the spin bearing particles, while intramolecular relaxation depends upon the rotational diffusion of the complex owing to the rapid reorientation of the vector separating the spins. The relative time scales of rotation, translation and chemical exchange must be considered.

Below are the MRD data for $^{7}Li^{+}_{(aq)}$ relaxed by two kinds of paramagnetic species: $Mn^{2+}_{(aq)}$ and the stable nitroxide free radicals 4-hydroxy-TEMPO (at pH=7.0), 4-amino-TEMPO (pH=4.0), and 3-carboxy-PROXYL (pH=10.0). In lieu of a detailed theoretical account, several qualitative observations can immediately be made. Both sets of data display the effects of relaxation due to electron-nuclear dipole-dipole interactions, and that due to the efficient intramolecular coupling mechanisms inherent to a H-bonded complex of finite lifetime. The effects of translational diffusion are readily apparent from the nitroxide data at small frequencies, where changes in pH, charge, and free radical moiety alter the diffusive characteristics of the dipolar interaction.

Continuing through higher frequencies disperses the Fermi scalar relaxation, which passes through an inflection at the inverse of the mean lifetime of a bonded complex, until the three sets of data converge, further implying the existence of a rotationally correlated species. Given the fact that $Mn^{2+}_{(aq)}$ bears S=5/2, it should be expected that the intermolecular dipolar interactions are more effective in this case; conversely, formation of a complex will be hindered by the larger positive charge and the presence of coördinated water molecules. In both cases, dispersion of any contribution modulated by rotational diffusion (including coupling of the nuclear magnetic quadrupole moment to an electric field gradient) will appear at much higher frequency, since rotational correlation times of tens of picoseconds are expected for molecules this size. Note that $\omega_e=1694\omega_L$.

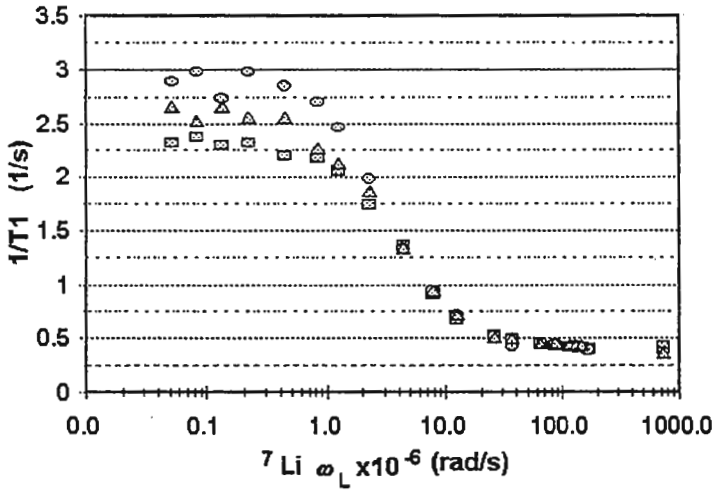

Figure 1. $^7Li^+_{(aq)}$ MRD by nitroxide free radical:
■ = 4-amino-TEMPO
▲ = 3-carboxy-PROXYL
● = 4-hydroxy-TEMPO
$[Li^+]$ = 2.0M
[N-O] = 10.0mM

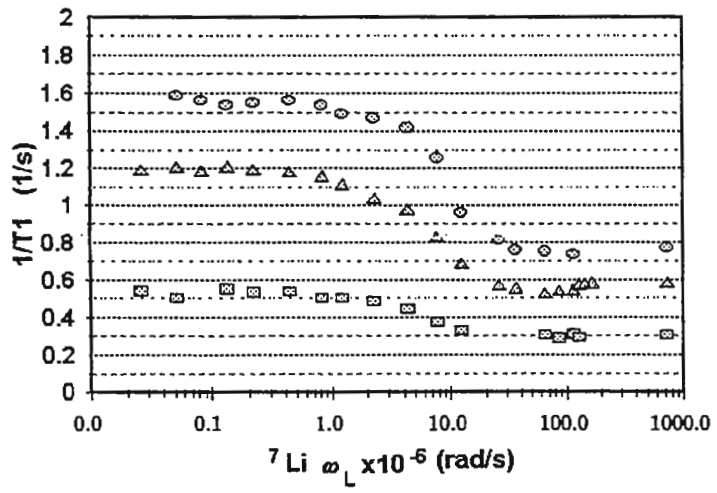

Figure 2. $^7Li^+_{(aq)}$ MRD by $Mn^{2+}_{(aq)}$:
■ $[Mn^{2+}]$=1.0mM
▲ $[Mn^{2+}]$=2.5mM
● $[Mn^{2+}]$=3.5mM
$[Li^+]$ = 2.0M

Best wishes,

Timothy Dinesen Shawn Wagner Robert Bryant

The NMR Revolution advances...

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Combinatorial Chemistry:

Bruker has the solution!

Bruker now offers an NMR accessory specifically designed for combinatorial chemistry analysis.

BACKGROUND

Until now, the search for new medicinal chemicals has been an exercise in tedium. Working with solutions in test tubes, chemists could only conveniently assemble new molecules one at a time, typically at the rate of one or two per week. Now a new approach, "Combinatorial Chemistry," is allowing properly equipped laboratories to produce new compounds at the rate of 100 to 200 per week.

Based on techniques developed in the 1960's, and re-discovered in the early 1980's by Leznoff,[1] Frechet[2] and others, combinatorial chemistry is a technique by which molecular fragments are assembled in multiple combinations on a polymer bead substrate to produce thousands of new molecules almost overnight.

The traditional method of analyzing compounds directly attached to polymer supports requires that the compound be removed from the resin, which is time consuming and may also structurally alter the reaction product. On the other hand, NMR assignment of compounds "on resin" using typical high resolution techniques is difficult or impossible because the polymer beads restrict molecular motion and cause the resonances to be broadened by direct dipolar coupling.

THE CCA ACCESSORY

The Bruker **C**ombinatorial **C**hemistry **A**ccessory (CCA) overcomes these limitations by using **M**agic **A**ngle **S**pinning (MAS) to eliminate dipolar couplings and produce high resolution NMR spectra of "on resin" compounds.

The special Bruker CCA MAS probe is capable of better than 1 Hz resolution (full width at half height). For combinatorial chemistry samples, this is more than adequate to ensure that the spectral resolution will not be instrument limited.

The unique new Bruker CCA accessory includes a MAS probe optimized for high resolution measurements, MAS control unit, and MAS sample changer with 20 sample capacity (larger capacity optional).

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- Pneumatic insertion and ejection of the sample rotors
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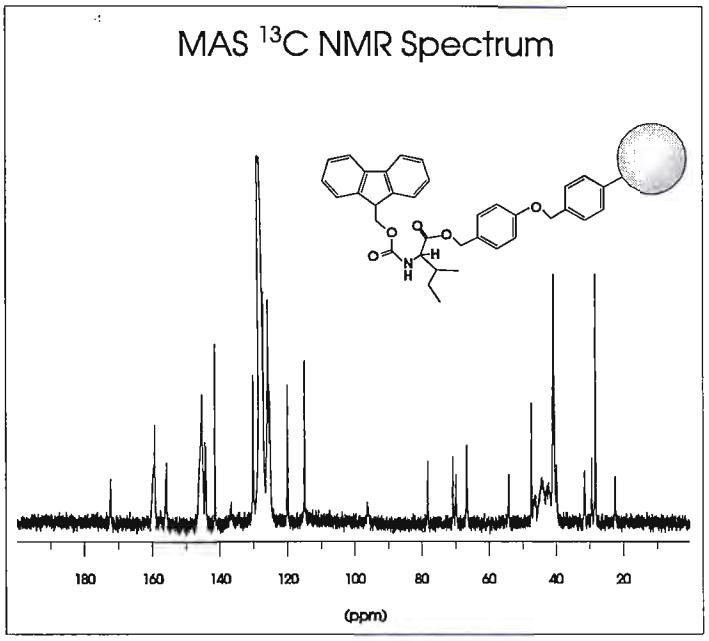
- ^{1}H and BB (^{15}N - ^{31}P) observation on the same probe
- Optional internal or external ^{2}H lock



The Bruker CCA accessory is available for all *AVANCE* 400 and 500 MHz standard bore NMR systems. On special request, it can also be offered at other frequencies and bore sizes. Also available is the new Bruker HFX accessory, which enables the probe to be used for X observation with simultaneous decoupling of ^{1}H and ^{19}F.

Please contact your local Bruker representative for more information.

Figure 1: ^{13}C MAS spectrum of a typical compound [3].

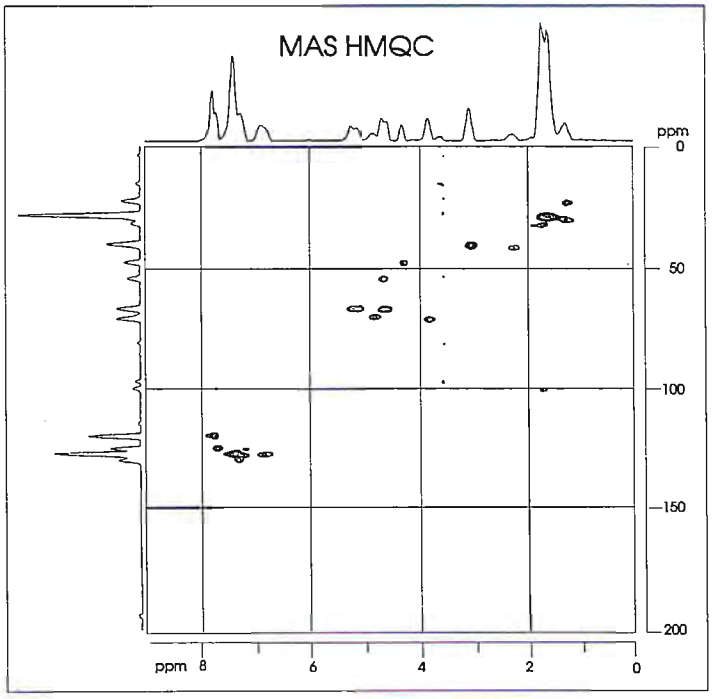


Figure 2: A two dimensional HMQC spectrum of the compound shown in Figure 1.

[1] C.C. Leznoff, Acc. Chem. Res. **11**, 327 (1978)
[2] J.M.J. Frechet, Tetrahedron **37**, 663 (1981)
[3] R.C. Anderson, M.A. Jarema, M.J. Shapiro, J.P. Stokes and M. Ziliox, Bruker Report **142/96**

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Bethesda, MD 20892-0520
May 7, 1997 (received 5/12/97)

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

BIOMOLECULAR SOLID STATE NMR TECHNIQUES

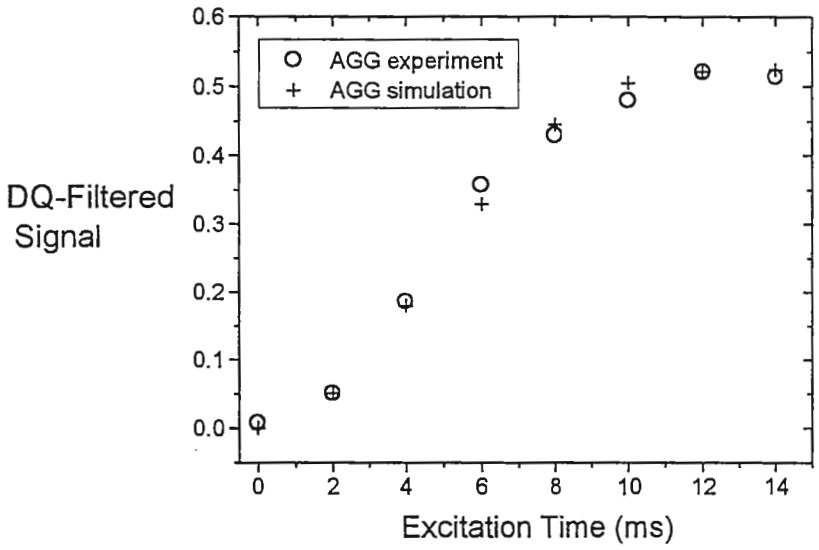
Dear Barry:

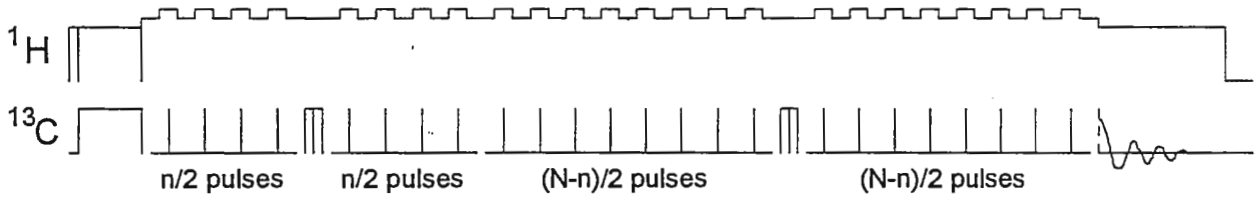
The standard approach to structural studies of (unoriented) biopolymers in solid state NMR has been to introduce pairs of specific isotopic labels and to use dipolar recoupling techniques (e.g., rotational resonance, REDOR, DRAMA, or some outgrowth of these techniques) in combination with magic-angle spinning to measure a specific internuclear distance. But dipolar recoupling measurements can be difficult to execute because they demand very high rf powers, very homogeneous rf fields, carefully tuned rf pulses, or very fast MAS, or difficult to analyze because of uncertainties regarding the effects of pulse imperfections, incomplete proton decoupling, and spin relaxation. This is particularly true when the distances of interest are long, when the labeled nuclei have large chemical shift anisotropies, and when the experiments are carried out in high fields or at low temperatures. Inevitably, the experiments that are most interesting to us require long distances, large CSA, high fields, and low temperatures. In addition, sensitivity is always the dominant consideration and our protein and peptide samples are concentration-limited, so we can not work with restricted sample volumes or small rotors and thereby improve the rf homogeneity, rf field strengths, or spinning speed. What we need then are alternative techniques that are experimentally simple and that provide structural information in a robust fashion under nonideal conditions.

One such technique is two-dimensional magic-angle spinning exchange spectroscopy. We have shown that rotor-synchronized 2D MAS exchange spectra of peptides with ^{13}C labels at pairs of carbonyl sites, acquired at relatively low spinning speeds, permit the determination of peptide backbone conformations, as specified by ϕ and ψ dihedral angles (J. Am. Chem. Soc. **118**, 8487 (1996); J. Chem. Phys. **105**, 7915 (1996)). In the fully-exchanged limit, the information in the 2D MAS exchange crosspeaks is angular in nature, since the spectra depend on the relative orientations of the CSA tensors at the two labeled sites rather than on the distance between the labels. Good results are obtained at relatively low rf powers, at high fields (including 17.6 T), at low temperatures, and without worrying about rf homogeneity, phase transients, etc. Both ϕ and ψ can be determined from a single spectrum of a single sample, and information about conformational distributions can be obtained in the case of disordered or partially-folded peptides. We are currently using this technique to study the conformation of a peptide from the V3 loop of the HIV-1 envelope glycoprotein gp120 when bound to an anti-gp120 antibody in frozen solution, among other things. (Incidentally, our structural measurements on frozen solutions of peptides and proteins would not be possible without Chemagnetics MAS probes, which allow us to change samples at -140° C and spin them stably, reproducibly, and reliably at low temperatures for weeks at a time.)

Distance measurements are still important, however, for example as a means of distinguishing between distinct conformations that happen to give similar 2D MAS exchange spectra. In particular, we would like to perform carbonyl-carbonyl distance measurements on the same doubly-labeled samples that we prepare for 2D MAS exchange measurements. To this end, we have developed a somewhat new approach to dipolar recoupling, based on the incorporation of rf-driven recoupling (RFDR) into a constant-time double-quantum filtering technique. The RFDR sequence (Bennett, Ok, Griffin, and Vega, J. Chem. Phys. **96**, 8624 (1992)) consists of a train of n 180° pulses, one in every two rotor periods. Double-quantum filtering is accomplished by placing a pair of 90° pulses in the middle of

the RFDR train and phase-cycling the pulses appropriately. To construct a constant-time technique, a second RFDR train, comprised of N-n 180° pulses with N fixed, is placed after the first RFDR train. The phases of the 90° pulses in the second train are set so that both the chemical shifts and the dipole coupling for the spin-1/2 pair are refocussed. The pulse sequence is shown schematically below, along with experimental and simulated plots of the build-up of double-quantum filtered ^{13}C NMR signal from doubly-labeled *Ala-*Gly-Gly, a favorite model compound. The build-up rate depends primarily on the carbonyl-carbonyl distance, which is determined by ϕ, but also depends significantly on ψ.





This constant-time double-quantum excitation method has the advantages that (1) the carbon pulses are sparse, so that effects of pulse imperfections are minimized and very high proton decoupling powers are unnecessary, (2) dephasing from residual proton couplings and spin relaxation has almost no effect on the shape of the build-up curve, and (3) the CSAs can be large. More details will be given in a forthcoming publication.

Sincerely,

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

May 7, 1997
(received 5/13/97)

Practical Aspects of Gas Phase NMR

Dear Barry,

We have lately been finding a variety of practical applications of gas phase NMR. In contrast to the interesting but rather more exotic examples involving hyperpolarized xenon, we have focused on the simple expedient of using gas phase NMR for following the reaction chemistry of small molecules. We believe that this technique has simply been largely overlooked as an analytical method, perhaps because of the following two perceptions: (i) the concentration of spins is too low to readily afford an NMR signal, and (ii) the relaxation times must be quite long (by extension from solution work with small molecules). In fact, it is well known that spin rotation is the dominant relaxation mechanism for spins-1/2 in the gas phase, and at around 1 atm pressure, T_1's tend to be approximately 1 second for protons and tens of milliseconds for fluorine.

In order to do kinetics, we need to restrict the sample to the thermostatted region of the probe. We do this by sealing off a 10 or 12 mm diameter ampoule which is about 2 inches long and which has a 5 mm diameter extension for attachment to a vacuum line. Once the gases of interest have been condensed in, the 5 mm extension is sealed with a torch and the stub attached to a normal 5 mm tube by means of some heat-shrink tubing. For most gas samples at 1 atm pressure, the ampoule volume contains several milligrams of material, so it is trivial to get good S/N for protons or fluorine in a single pulse. We have used this approach to study the kinetics of a number of unimolecular thermolyses, and have found that extensions to systems involving heterogeneous catalysis also work quite well (see *J. Am. Chem. Soc.* **118**, 10000 (1996)).

In order to support this work, we recently obtained a 10 mm high-temperature probe from Nalorac. The probe is a "dual broadband" and consists of an inner coil for $^{15}N - ^{31}P$ and a decoupler coil that is tunable between ^{1}H and ^{19}F. On our widebore 360 we get a little over 200:1 on ASTM ^{13}C, which might not sound great until you realize that this is from a probe that can go to 400°C using air (i.e., dry nitrogen) cooling alone. Figure 1 shows the results we obtained on a test sample calculated to have (at 25°C) 0.5 atm $^{13}C_2H_4$ and 0.5 atm $^{13}CH_3Br$. In each case, the spectra result from signal averaging for approximately 2 minutes (1024 scans). The greater efficiency of spin rotation for ethylene than for

bromomethane accounts for its broader line. Clearly, Mr. Boltzmann has something to say about sensitivity at high temperature and the nature of kinetic studies that require ^{13}C observation.

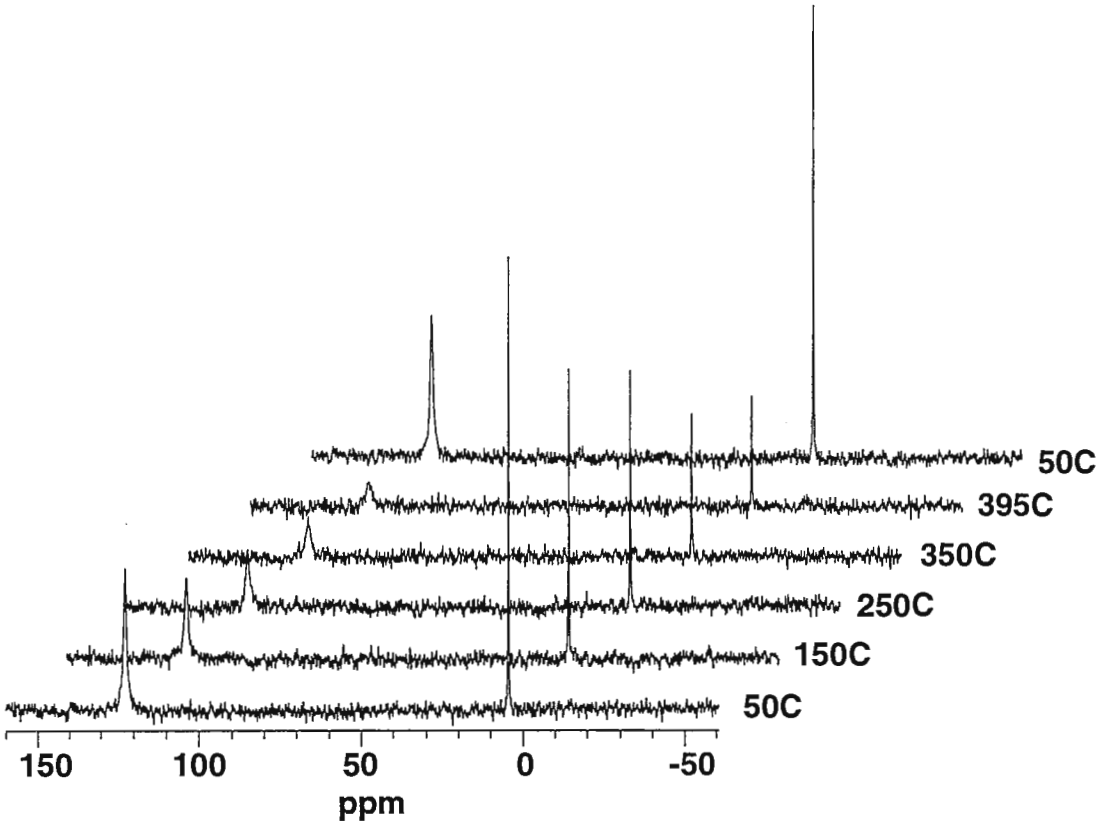


Figure 1. Gas phase ^{13}C NMR spectra as a function of temperature

With sincere best wishes,

Chris Paul Steve

D. Christopher Roe Paul Krusic Steve Hill

LONG ISLAND UNIVERSITY BROOKLYN CAMPUS UNIVERSITY PLAZA, BROOKLYN, NEW YORK 11201

May 21, 1997 (received 5/27/97)

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

High Resolution ^{1}H NMR nOe Difference Studies of Erythromycin A in Water

Dear Barry,

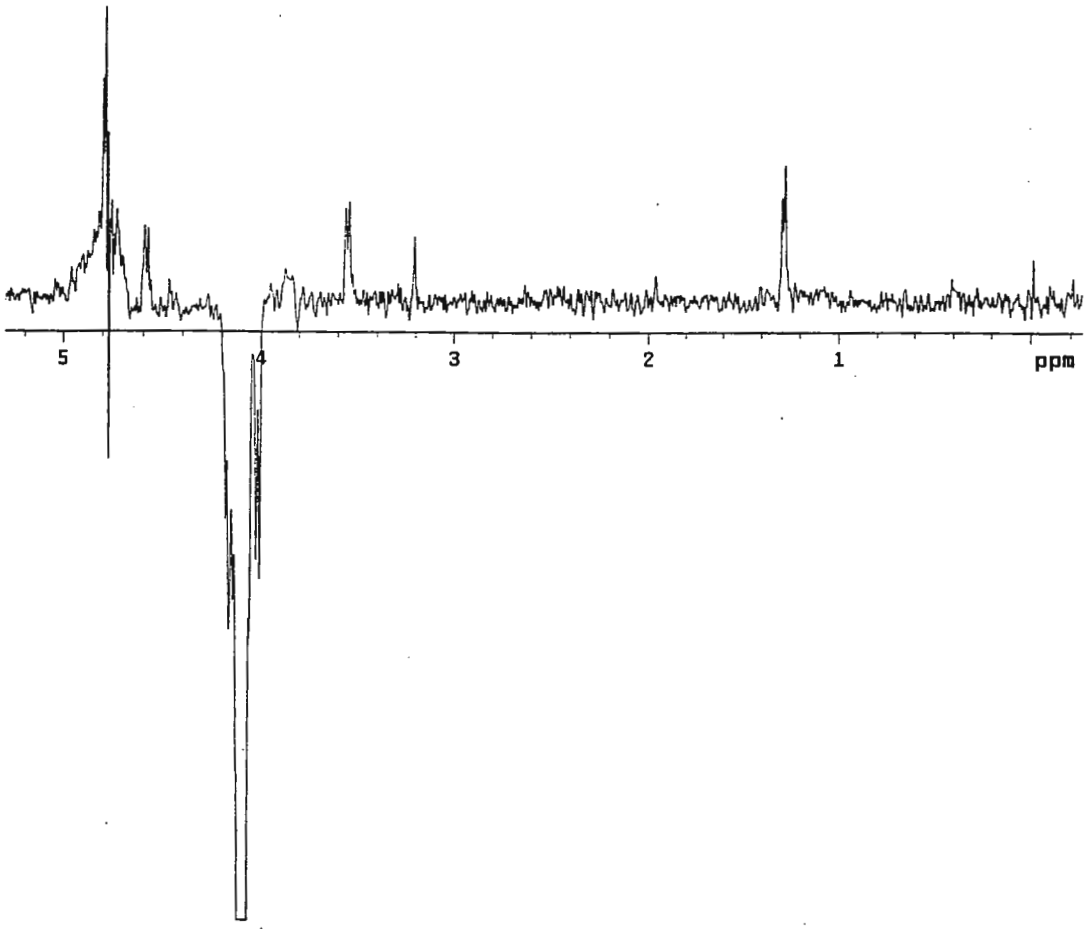
Recently, we have become interested in looking at the solution structure of erythromycin A in water. It has been shown (J. R. Everett, E. Hunt, and J. W. Tyler, *J. Chem. Soc. Perkin Trans.* **2**, 1481-1487, 1990.) that the actual keto-hemi-acetal equilibria associated with erythromycin A depend on the polarity of the solvent. Surprisingly, to our knowledge, there have not been any previous NMR studies with this compound in water. Thus, we are starting to investigate the solution conformation of this compound and the associated hemi-acetal tautomers. Formation of the hemi-acetal is believed to be responsible for resistance to this antibiotic by bacteria. An understanding of the solution structures of the related tautomers at physiological conditions is a prerequisite to the rational design of new drugs void of bacterial resistance. On a recent visit with Boban John of the Varian applications lab in Palo Alto, CA, we used a recently published ("One-Dimensional NOE Exps ..." by K. Stott, J. Keeler, Q.N. Van & A.J.Shaka, *J. Magn. Reson.* **125**, 302-324 , 1997) pulse sequence to obtain a 1D nOe difference ^{1}H spectrum of erythromycin A in water. In addition to the need to selectively excite a peak close to water with solvent suppression, erythromycin A poses the added challenge that all of the peaks in the ^{1}H NMR spectrum are upfield of 5.2 ppm, so that setting the transmitter on water requires some additional flexibility in acquisition and processing. Shown on the next page is a 1D nOe difference ^{1}H spectrum acquired on a Varian Unity INOVA 400 using water presaturation and Z-gradient selection for water suppression with a γH2 of 55 Hz and a shaped pulse for the selective excitation of the proton attached to C-5'' of the cladinose sugar unit of erythromycin A at 4.12/4.07 ppm. The VNMR subroutine, "Pandora's Box", was used to select the target multiplet and the optimum shape/bandwidth (γH2 = 25 Hz) of the selective excitation pulse. Preliminary results indicate that nOe's are seen at 4.61/4.58 ppm, 3.56/3.54 ppm, 3.21ppm (hemi-acetal only), and 1.29/1.27 ppm corresponding, respectively, to protons attached to C1', C5, C4", C6" and C6'. These dipolar couplings are consistent with 2D ROESY data. Split peaks are seen for the 9-ketone and 9,12-hemi-acetal tautomers.

Sincerely,

Fernando Commodari, Ph.D.
Assistant Professor
Long Island University
Brooklyn, NY 11201

George Sclavos

LIU-Brooklyn



1D ^{1}H nOe difference spectrum of erythromycin A in 90% water and 10% $^{2}H_2O$ acquired with a Z-axis gradient triple-resonance probe at 9.4 T in 128 transients. The proton attached to C-5'' of the cladinose sugar unit of erythromycin A at 4.12/4.07 ppm was selectively excited with a hyperbolic secant pulse. nOe's are seen at 4.61/4.58 ppm, 3.56/3.54 ppm, 3.21ppm (hemi-acetal only), and 1.29/1.27 ppm corresponding, respectively, to protons attached to C1', C5, C4", C6" and C6'. Split peaks are seen for the 9-ketone and 9,12-hemi-acetal tautomers.

******Postdoctoral Position******

A postdoctoral fellowship is available starting September, 1997 at LIU-Brooklyn to carry out high resolution NMR studies on erythromycin A and derivatives of erythromycin A and/or peptidomimetics. The appointment will be at the Adjunct Assistant Professor level involving teaching of a class and the associated laboratory. Resources available for research include a new three-channel Varian INOVA 400 and a Silicon Graphics Octane. This is a unique opportunity for an individual to gain teaching experience while carrying out post-doctoral research in New York city (www.brooklyn.net/ashkenaz/brooklyn.html).

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References: (1) Dorn, H.C. *Anal. Chem.* **1984**, *56*, 747A. (2) For recent reviews, see: (a) Hofmann, M. et al. *LaborPraxis Med.* **1993**, *17*, 36. (b) Braumann, U. et al. *GIT Fachz. Lab.* **1994**, *38*, 77. (c) Spraul, M. et al. *Anal. Proc.* **1993**, *30*, 390. (d) Spraul, M. et al. *Bruker Rep.* **1990**, 12. (3)(a) Seddon, M.J. et al. *J. Pharm. Biomed. Anal.* **1994**, *12*, 419. (b) Spraul, M. et al. *Methodol. Surv. Bioanal. Drugs* **1994**, *23*, 21. (c) Spraul, M. et al. *Anal. Chem.* **1993**, *65*, 327. (d) Spraul, M. et al. *J. Pharm. Biomed. Anal.* **1993**, *11*, 1009. (e) Wilson, I.D. et al. *J. Chromat. A* **1993**, *617*, 324. (f) Spraul, M. et al. *J. Pharm. Biomed. Anal.* **1992**, *10*, 601. (4) Roberts, J.K.; Smith, R.J. *J. Chromat. A* **1994**, *677*, 385. (5) Johnson, S. et al. *J. Chem. Soc., Perkin Trans. 1* **1994**, 1499. (6)(a) Albert, K. et al. *Anal. Chem.* **1989**, *61*, 772. (b) Also see references 2-12 from reference 2d.

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Note: *The chemical purity of these products is equal to or better than that of the HPLC grade, protonated solvents. The percent purity assigned represents the deuterium content.*

Acetonitrile-d_3, 95+ atom % D
44,947-4 — **100mL $185.00; 1L $1350.00**

Deuterium oxide, 90 atom % D
43,577-5 — **25g $17.50; 100g $49.00**

Deuterium oxide, 10 atom % D
43,578-3 — **100g $7.50**

Methyl-d_3 alcohol-*d*, 95+ atom % D
44,946-6 — **100mL $300.00; 1L $2150.00**

HPLC Grade Protonated Solvents

Acetonitrile, 99.9+%, HPLC grade
27,071-7 — **100mL $18.10; 1L $34.55; 2L $51.90; 6 x 1L $174.90; 4 x 2L $156.00; 4 x 4L $311.60; 18L $262.50**

Ethyl alcohol, reagent, denatured, HPLC grade
27,074-1 — **100mL $15.05; 1L $22.30; 2L $33.40; 6 x 1L $113.10; 4 x 2L $100.40; 4 x 4L $174.20**

Methyl alcohol, 99.9+%, ACS HPLC grade
27,047-4 — **100mL $14.20; 1L $18.70; 2L $28.05; 6 x 1L $92.40; 4 x 2L $84.00; 4 x 4L $128.20; 18L $110.25**

Toluene, 99.8%, HPLC grade
27,037-7 — **100mL $14.10; 1L $18.40; 2L $27.65; 6 x 1L $93.00; 4 x 2L $82.80; 4 x 4L $147.80**

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Central Research
NMR Spectroscopy

May 20, 1997
(received 5/22/97)

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

...just one of those days

Dear Dr. Shapiro,

The date was April 24, 1997. The time, 8:30am. I was very excited to begin real work on our new three channel, triple-gradient Bruker DRX-500 equipped with an actively-shielded Magnex magnet. The magnet had been energized the previous week, our new probe was installed, and we were ready to go. I pressed the space bar to turn the computer monitor on, pressed the sample eject air, and watched the monitor brighten while I waited for the air. I gasped in horror - the monitor screen background was PINK!

Now, for any readers with current Bruker spectrometers with an SGI workstation front-end, you know that the standard screen background is BLUE, not pink. Pink can arise from deliberately changing the screen background, or from a change in the apparent magnetic field in the vicinity of the computer monitor. A quick degauss and the monitor was BLUE again, telling me that I no longer had 143 amps running through the field coil.

Lesson #1. If your monitor screen turns pink, the magnet has probably quenched.

The date was still April 24, 1997. The time, 10:00am. We had a Sparc 5 monitor from our Varian Unity+400 which had begun to seriously distort because it was in a 5 gauss field and had been that way for about a year and a half. The screen degausser no longer helped, but I had brought in from home a Radio Shack VCR tape degausser which I thought would do the job. I unplugged the monitor, moved it a safe distance away from the computer (and hard drive), and thoroughly degaussed the monitor case. I plugged the monitor back in - and knew something had gone terribly wrong!

Instead of a gradual polychromatic distortion, I had FIXED the edge distortion effects. I had however, created a series of closely-spaced black lines in the center of the monitor screen.

Lesson #2. Don't use a VCR tape degausser for a monitor screen.

The date was still April 24, 1997. The time, 2:00 pm. Now my attention turned to Macintosh software. I had installed a driver for a Kodak DC20 digital camera in our ongoing effort to create a useful intranet web page for the NMR group (we use the camera to generate how-to pictures for the web site). The PhotoEnhancer software that comes with the camera installs the driver and AppleGuide extensions to make for easy help page access. The camera works fine and makes the process of importing graphics quite simple. I reboot the Macintosh and...

Yes, the computer failed to boot. I begin the process of finding the conflicting drivers, assuming that the DC20 driver must remain and that it is the culprit. Yet, two hours later I am no closer to a working computer system. In absolute desperation I remove the AppleGuide extensions, and everything works!

Lesson #3. Never assume anything about Macintosh extensions.

Sincerely, Walt

Walter Massefski, Jr.
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THE NMR NEWSLETTER

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The subscription rate for the 1997-98 year will remain at $190.00, with the usual 50% discount for academic or personal subscriptions.

B. L. Shapiro
2 June 1997

8th Annual Chemagnetics Workshop on Solid-State NMR

The 8th Annual Chemagnetics Workshop on Solid-State NMR, Aug. 8-9, 1997, is aimed at NMR spectroscopists who enjoy informal presentations with generous time for individual discussions. Most of the talks will focus on "nuts & bolts" experimental details and what the speaker would like to have known before starting work. The talks will be at The Elkhorn Lodge in Estes Park, Colorado. The Saturday, August 9, lab sessions will be at the Chemagnetics site in Ft. Collins. Attendance is NOT restricted to owners of Chemagnetics products. For more information, please browse at:
http://www.otsuka.com/html/workshop/entry.html.

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Palo Alto, CA 94303.

(415) 493-5971* - *Please call only between 8:00 am and 10:00 pm, Pacific Coast time.*

Deadline Dates

No. 466 (July)	27 June 1997
No. 467 (Aug.)	25 July 1997
No. 468 (Sept.)	22 Aug. 1997
No. 469 (Oct.)	26 Sept. 1997
No. 470 (Nov.)	24 Oct. 1997

* Fax: (415) 493-1348, at any hour. Do not use fax for technical contributions to the Newsletter, for the received fax quality is very inadequate.

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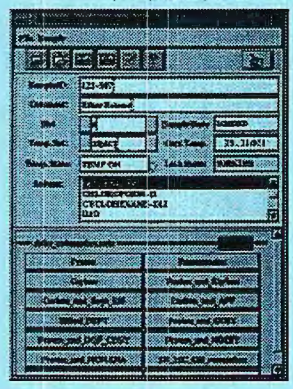
How To Run JEOL's Eclipse+ Spectrometer



Step 1: Enter your sample name and the solvent.

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

Step 3: Walk away with your data.

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

- ✔ Auto Probe Tuning (with AutoTune Broad Band Probe)
- ✔ Auto-sample Control (with AutoSample Changer)
- ✔ Auto Selection of Spectrometer Conditions
- ✔ Auto Baseline Correction
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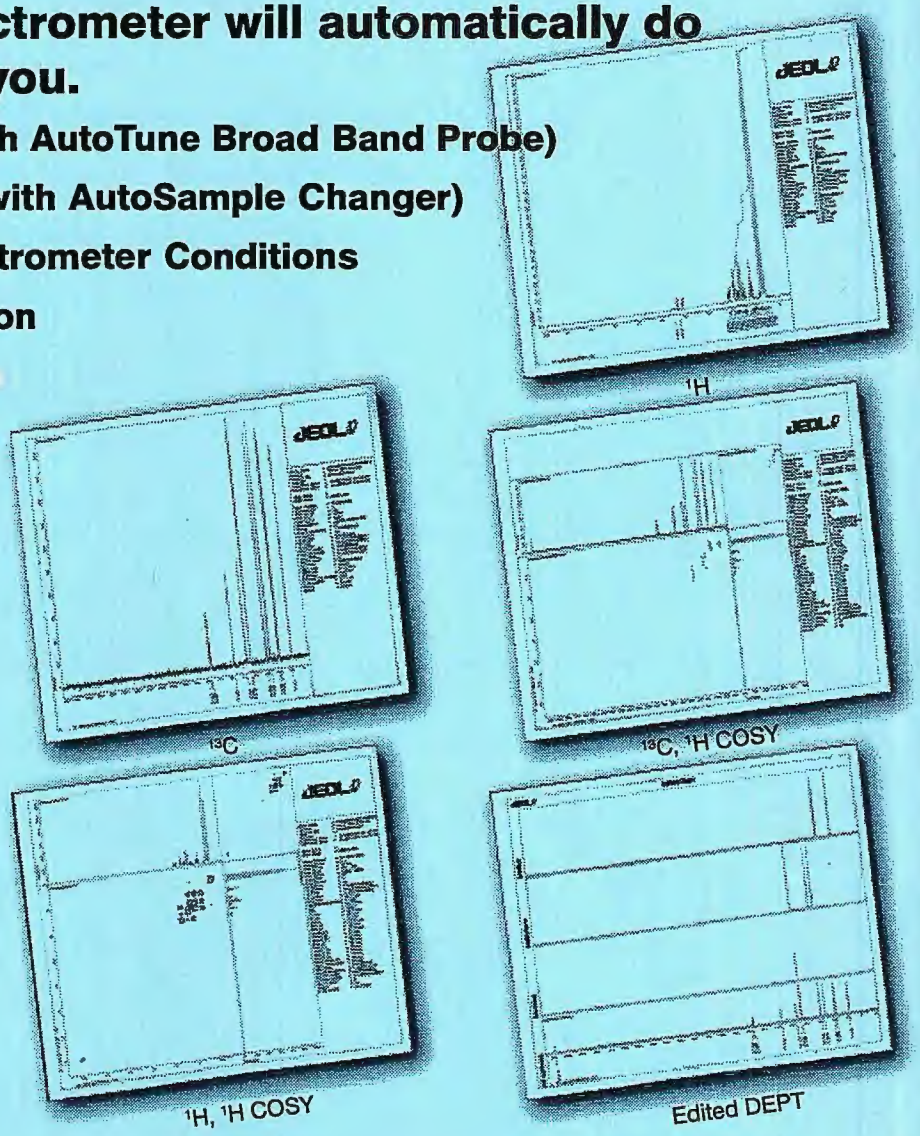

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