TEXAS A&M UNIVERSITY

NMR NEWSLETTER

No. 406

July 1992

TABLE 1 DEUTERATED SOLVENTS

Cat. No.	Description	Formula	Min. Isotopic Purity (%D)	Density (g/mL)	MP (°C)	BP (°C)	$-\chi_v \times 10^6$ @ (°C)
D-11	Acetic Acid-d_4	CD_3COOD	99.5%	1.1[illegible]	17	118	0.551 (32)
D-120	Acetic Acid-d_4 + TMS		[illegible]				
D-13	Acetone-d_6	CD_3COCD_3	99.9%	0.84	-94	57	0.460 (20)
D-121	Aceonte-d_6 + 1% TMS		99.8%				
D-129							
D-14							543 (20)
D-21							0.611
D-122							
D-130							
D-28	Chloroform-d	$CDCl_3$	99.8%	1.50	-64	62	0.740 (20)
D-31	Chloroform-d + 1% TMS		99.8%				

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Cat. No.	Paper Size	Instrument Model	Type
WCV-100 (S-[illegible])	11" X 26"	HA-100, HA-[illegible]D	Gridded-Two Color
WCV-60 ([illegible])	11" X 26"	A-60, A-60[illegible], A-60D	Gridded-Two Color
WCV-XL ([illegible])	11" X 26"	XL-[illegible] and FT	Gridded-Two Color
WCV-XLFT-[illegible]	11" X 26"	XL-[illegible] and FT	Gridded-Two Color
WCV-XL-200	11" X 26"	XL-200, XL-300, XL-400	Gridded-Two Color
[illegible]	[illegible]	[illegible]	[illegible]
WCV-20 (CFT-20)	11" X 16 3/4"	CFT-20, FT-80, FT-80A	Gridded-Two Color
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FORTHCOMING NMR MEETINGS

34th Rocky Mountain Conference on Analytical Chemistry, Denver, Colorado, **August 8-14, 1992**; Contact: M. C. Goldberg, P.O. Box 25046 MS 424, Lakewood, CO 80225; (303)236-4728.

Eleventh Annual Scientific Meeting and Exhibition, Society of Magnetic Resonance in Medicine, Berlin, Germany, **August 8-14, 1992**; Contact: S.M.R.M., 1918 University Ave., Suite 3C, Berkeley, CA 94704; (415) 841-1899, FAX: (415) 841-2340.

XV International Conference on Magnetic Resonance in Biological Systems, Jerusalem, Israel, **August 16 - 21, 1992**; Contact: Prof. Gil Navon, XV ICMRBS, P. O. Box 50006, Tel Aviv 61500, Israel.; Tel. (972-3) 5174571, Fax: (972-3) 655674/660325.

MRI in the Applied Sciences, Duke University, Durham, North Carolina, **October 25-28, 1992**; Contact: Society of Magnetic Resonmance in Medicine, 1918 University Ave., Suite 3C, Berkeley, CA 94704; (510) 841-1899; FAX: (510) 841-2340.

High Resolution NMR Spectroscopy (a residential school), University of Sheffield, England, **April 1993**[*sic*]; Organizer: Dr. B. E. Mann (Sheffield); For information, contact Ms. L. Hart, The Royal Society of Chemistry, Burlington House, Piccadilly, London W1V 0BN, England; Tel.: 071-437-8656.

Additional listings of meetings, etc., are invited.

Second Notice re 1992-93 Invoices and Subscription Rates

Subscription renewal invoices for the October 1992 - September 1993 year were mailed out on 1 July. If you ought to receive such an invoice, and do not have it in your hands by the time you read this notice, please call or write me promptly. *Payment* of these invoices *must be received* by me *no later than September 10, 1992* to ensure uninterrupted mailing of the Newsletter issues. Please do not delay execution of any necessary paperwork!

Also, please be sure that the instructions on the invoice are followed precisely. In particular, *overseas subscribers* should be careful to see that their name and the invoice number appear on the payment (or, better, that the extra invoice copy which is provided is returned to me with the payment check or money order). Anonymous checks, while otherwise useful, cannot always be credited to the correct account.

The subscription rate for the October 1992 - September 1993 year has been set at US$170.00 for the twelve monthly issues, postpaid. Personal or academic subscriptions will continue to be offered at a 50% discount, at US$85.00. The inexorable rise in costs has necessitated this small increase in the subscription fee.

The new invoices contain entries for *optional* surcharges for First Class or Air Mail Printed Matter mailing. Please adjust the amount you pay accordingly (I trust no one will choose to pay both surcharges.).

Thank you for your understanding and cooperation.

B. L. Shapiro
2 July 1992

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All Newsletter Correspondence
Should Be Addressed To:

Dr. Bernard L. Shapiro
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto, CA 94303, U.S.A.

(415) 493-5971

DEADLINE DATES

No. 408 (September) -------- 21 August 1992
No. 409 (October) -------18 September 1992
No. 410 (November) -------16 October 1992
No. 411 (December)-----20 November 1992

* * * * *

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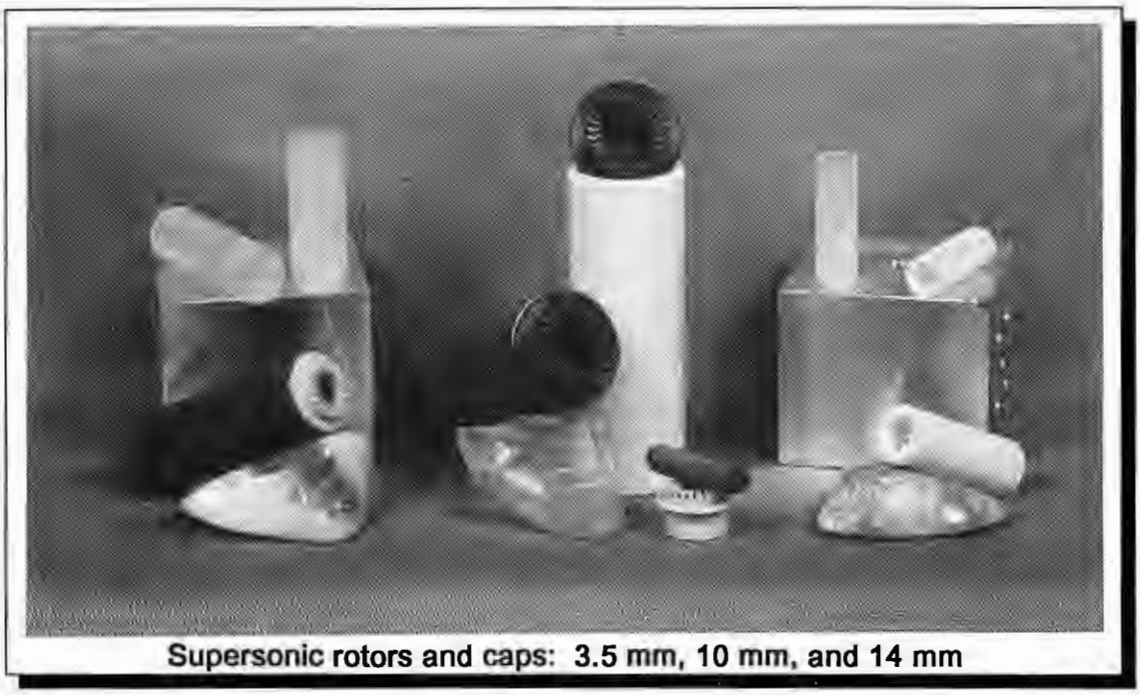

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➢ 1984	Multinuclear Observe for Solids	➢ 1990	Dynamic Angle Spinning
➢ 1985	19 mm MAS	➢ 1991	14 mm MAS at 7 kHz
➢ 1987	CRAMPS Probes	➢ 1992	3.5 mm MAS at 26 kHz
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John Cavanagh Ph.D.
Scientific Associate,
Department of Molecular Biology, MB-2
Phone: 619-554-2801
FAX: 619-554-9822.

(received 5/30/92)
27th May 1992.

Disappearing Peaks in Sensitivity Enhanced Experiments

Dear Prof. Shapiro:

Since publishing several different sensitivity enhanced versions (1-4) of proton detected heteronuclear correlation experiments, in both two and three dimensions, we have received some phone calls asking us questions about these experiments and their optimum implementation. Most questions concern the disappearance of signal in the final spectra.

Two points are worth emphasizing in regard to this. The way in which these experiments work is straightforward. In a conventional HSQC experiment, only one proton in-phase magnetization component is detected, H_X, for example. In the sensitivity enhanced (SE) versions of this experiment (Fig. 1) two orthogonal proton in-phase magnetization components are detected, $-H_X$ and $+H_Y$. Processing of this data alone results in a horrible phase-twisted spectrum. Recording the same experiment but with the phase of ϕ_2 inverted results again in two orthogonal proton in-phase magnetization components, this time $+H_X$ and $+H_Y$. Adding the two sets of data together gives $+2H_Y$, a pure phase correlation spectrum, we denote as ADD. Subtracting the sets of data gives $+2H_X$, also a pure phase spectrum which we call SUB. The ADD and SUB spectra are phase-shifted from each other by 90° in each dimension. Co-addition of ADD and SUB spectra doubles the size of the signal while the noise adds as statistically independent. In theory, a 1.41 increase in sensitivity is realized.

The first thing to note is that to generate the two orthogonal terms, each follows a different pathway. During the refocussing process, the H_X term is derived from longitudinal magnetization while the H_Y term is derived from multiple quantum coherence. These have different relaxation rates and as the system being studied increases in size and the correlation time decreases, the ADD spectrum (H_Y) begins to lose sensitivity. Therefore when processing the ADD and SUB spectra prior to final combination, it is not unusual to see the signal-to-noise ratio of the ADD spectrum slightly less than the SUB spectrum. This causes a small drop in sensitivity enhancement in the whole process.

However, it has been noted in our lab and others, that in some cases there is no signal at all in the ADD spectrum. The explanation for this is that the recommended phase cycling procedure outlined in ref. 2 is not followed correctly. As always, several options exist for the phase cycling in this experiment, especially for the pulses noted ϕ_2 and ϕ_3. The relative phases of the phases of ϕ_2 and ϕ_3 are especially important since they actually determine the signs of the detected orthogonal operators. This means that ϕ_2 and ϕ_3 have to be cycled in parallel in order to maintain a consistent sign relationship between the two detected operators. A quick product operator analysis is not sufficient to check the effect of a phase cycle in these experiments. In many cases an analysis will show that a particular phase cycle is fine and the H_X (SUB) spectrum will appear in its full glory. However it is imperative to remember that the whole point of the SE methods is to collect two components and failure to phase cycle pulses ϕ_2 and ϕ_3 correctly may result in complete cancellation of the H_Y (ADD) spectrum.

In summary, we suggest that the phase cycle shown below in the figure, be employed as it stands when running the SE experiments. It is important that any extra phase cycling be added only after the basic unit has been completed.

Please credit this contribution to the account of Peter Wright.

Best Wishes

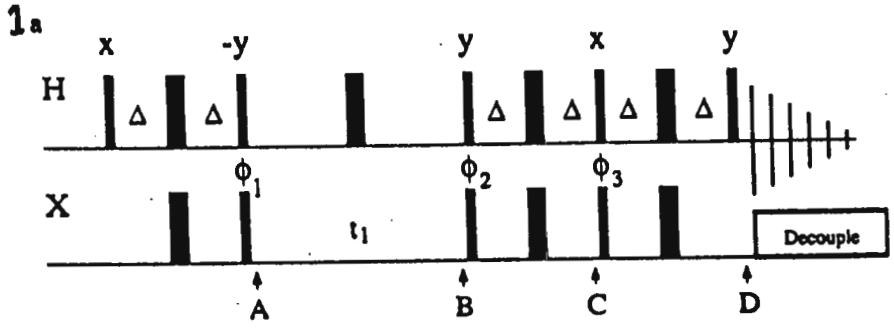
John Cavanagh, Ph.D.

Göran Carlström, Ph.D.

(1) J. Cavanagh and M. Rance, JMR 88, 72 (1990).
(2) A.G. Palmer, J. Cavanagh, P.E. Wright and M. Rance, JMR 91, 429 (1991).
(3) J. Cavanagh, A.G. Palmer, P.E. Wright and M. Rance, JMR 93, 151 (1991).
(4) A.G. Palmer, J. Cavanagh, R.A. Byrd and M. Rance, JMR 96, 416 (1992).

Phase Cycle:

ϕ_1 = x, -x, x, -x ϕ_2 = x, x, -x, -x ϕ_3 = y, y, -y, -y receiver = x, -x, -x, x



Gradient Enhanced Spectroscopy

Phase Sensitive DQF COSY

The selection of multiple quantum coherence with gradients is an effective method for suppressing the water resonance in aqueous solutions and for reducing t_1 noise and other artifacts. A further enhancement is the use of selective water excitation using crafted RF pulses followed by gradient dephasing to attenuate the water signal prior to the coherence selection sequence. This technique has been applied to the phase sensitive gradient enhanced DQF COSY experiment for a 5mM lysozyme sample in 90% H_2O. The data were collected on an Omega PSG 500 equipped with the S-17 Gradient Enhanced Spectroscopy accessory using a 5mm inverse probe.

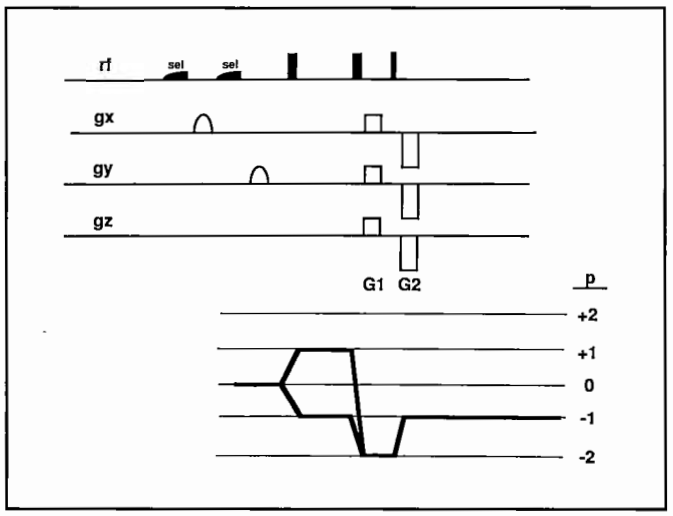


Figure 1

The pulse sequence and the corresponding coherence level diagram for the phase sensitive GE-DQF COSY experiment. The water resonance is selectively excited using a crafted RF pulse followed by a shaped gradient pulse along the X axis which dephases the transverse magnetization. A second selective RF pulse is applied with a dephasing gradient along the Y axis to avoid gradient recalled echoes. This is followed by a phase sensitive GE-DQF COSY sequence.

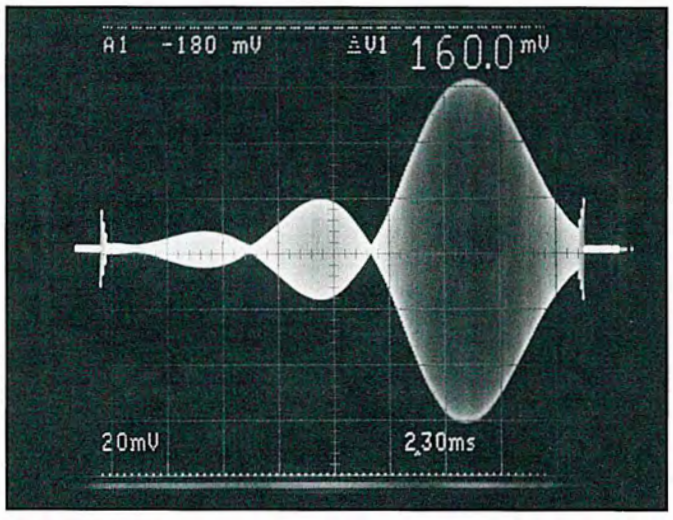


Figure 2

The crafted RF pulse used in this sequence. This pulse was designed using the ''hard pulse approximation'' method. It is characterized by a flat amplitude response in the selected region and minimal excitation in the out of band region. The duration of the selective RF pulse was set to 20 ms, corresponding to an excitation bandwidth of 175 Hz.

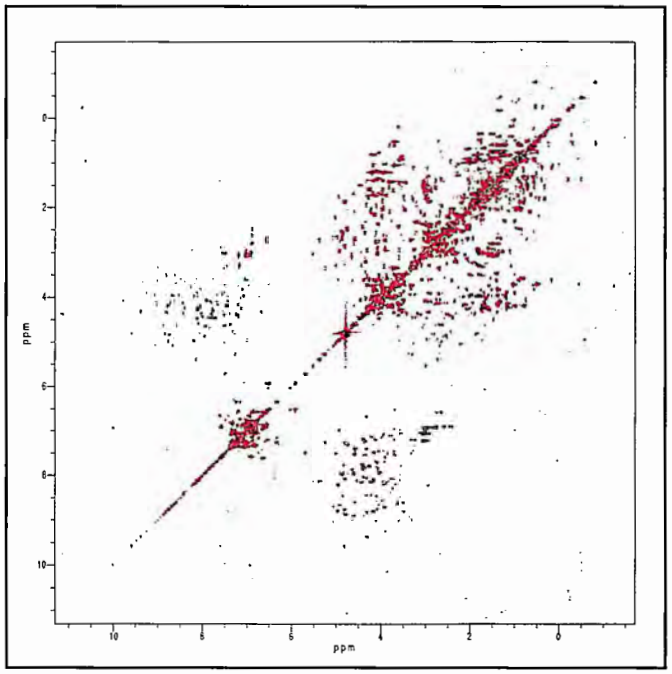


Figure 3

A phase sensitive GE-DQF COSY spectrum of 5 mM lysozyme in 90% H_2O. Thirty-two scans were accumulated for each of the 700 t_1 increments resulting in a total data acquisition time of approximately 9.7 hours. Half-sinusoidal gradient pulses of 20 ms duration with an amplitude of 10 G/cm were used to dephase the excited water signal. Coherence selection was achieved using 2 ms gradient pulses of 17 and 34 G/cm amplitude.

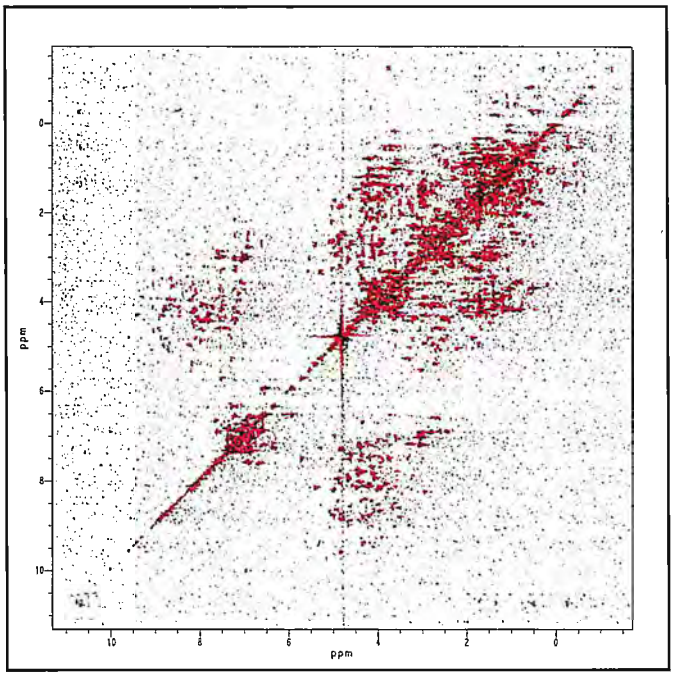


Figure 4

The noise floor contour plot. This spectrum illustrates the efficiency of the water suppression and the absence of t_1 noise and artifacts. The effectiveness of the water suppression stems from two different sources: the dephasing of the water signal after excitation by the selective RF pulse, and the selection of the double quantum pathway using the gradient pulses G_1 and G_2.

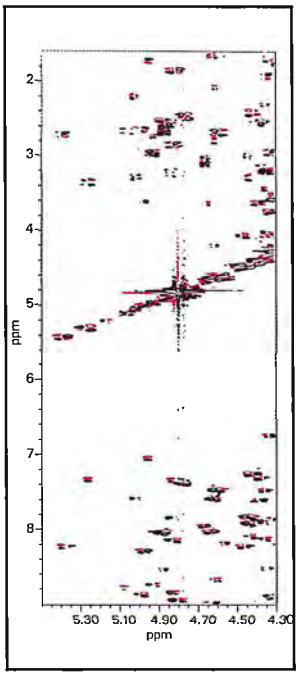


Figure 5

The expansion plot shows good resolution and intensity for cross peaks at the chemical shift of water. The combination of selective RF techniques and actively shielded gradients allows phase sensitive spectra of medium-sized proteins in aqueous solution to be obtained.

GE NMR Instruments

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

May 14, 1992
(received 6/18/92)

Dr. Bernard L. Shapiro
Editor/Publisher
TAMU NMR Newsletter
966 Elisnore Court
Palo Alto, CA 94303

RE: Characterization of Ordered Buckminsterfullerene Co-Crystal $C_{60}\cdot CH_2I_2\cdot C_6H_6$

Dear Barry:

We have obtained the first crystals containing crystallographically ordered (at room temperature), unmodified fullerene (C_{60}) molecules, with chemical composition $C_{60}\cdot CH_2I_2\cdot C_6H_6$. The fullerene molecules are arranged in novel hexagonally close-packed layers, separated by the solvent components. The monoclinic (space group *C2/c*) crystal structure of $C_{60}\cdot CH_2I_2\cdot C_6H_6$ was determined by X-ray crystallography. It should be noted that all buckminsterfullerene carbon atoms readily appeared as distinct peaks on the electron density maps, indicating a high degree of order even at room temperature. In addition to C_{60} and the methylene iodide molecule, a molecule of benzene was located.

Solid-state 25-MHz ^{13}C NMR spectra of $C_{60}\cdot CH_2I_2\cdot C_6H_6$ are shown in the Figure. The MAS spectrum (a) exhibits two sharp resonance lines at 146.7 and 133.1 ppm and a somewhat broad resonance at approximately 35 ppm, corresponding to carbon atoms of C_{60}, benzene and methylene iodide, respectively. The recycle delay time was chosen to enhance the weak carbon signal of methylene iodide, hence integrated signal intensities in the spectrum do not accurately reflect the number of carbon atoms in each chemical environment. Broadening of the carbon resonance of methylene iodide results from residual quadrupole coupling to iodide due to restricted motion in the crystal lattice. The static spectrum (b) of the C_{60} co-crystal recorded at room temperature shows a single Gaussian line at 146.7 ppm with a fwhm = 147 Hz. The C_{60} resonance in the static spectrum recorded at a carbon frequency of 75 MHz has a fwhm = 240 Hz. The narrow line widths observed at both frequencies indicate that the C_{60}molecules in the co-crystal rotate rapidly and *nearly isotropically* relative to the NMR time scale defined by the inverse of the chemical shift anisotropies (CSA) of the aromatic carbons in C_{60}. The CSA powder pattern for solid C_{60} is approximately 200 ppm in width, thus the rotational correlation time of C_{60} in the co-crystal must be short compared to the inverse CSA width, or greater than about 70 μs.

Even though the crystallographic analysis of $C_{60}\cdot CH_2I_2\cdot C_6H_6$ shows the fullerene molecules to be ordered on the X-ray time scale, NMR experiments indicate dynamic effects. Static ^{13}C spectra taken at two field strengths demonstrate that the rotational correlation time of C_{60} in the co-crystal is shorter than 70 μs. This upper limit is at least four orders of magnitude greater than the lower limit, which has to be less than the correlation time of pure C_{60}, i.e., <1 ns. An attractive possibility which is consistent with the crystallographic observation of distinct electron density peaks (ordered atomic positions) would be a restricted (jumping) rotation of the molecule such that each atom spends a considerable amount at or near its equilibrium position ("ratchet motion"). A thorough investigation of the temperature dependence of the ^{13}C NMR lineshape and spin-lattice relaxation time (T_1) to probe the details of molecular motion in $C_{60}\cdot CH_2I_2\cdot C_6H_6$ is planned.

Urs Geiser
Chemistry & Materials
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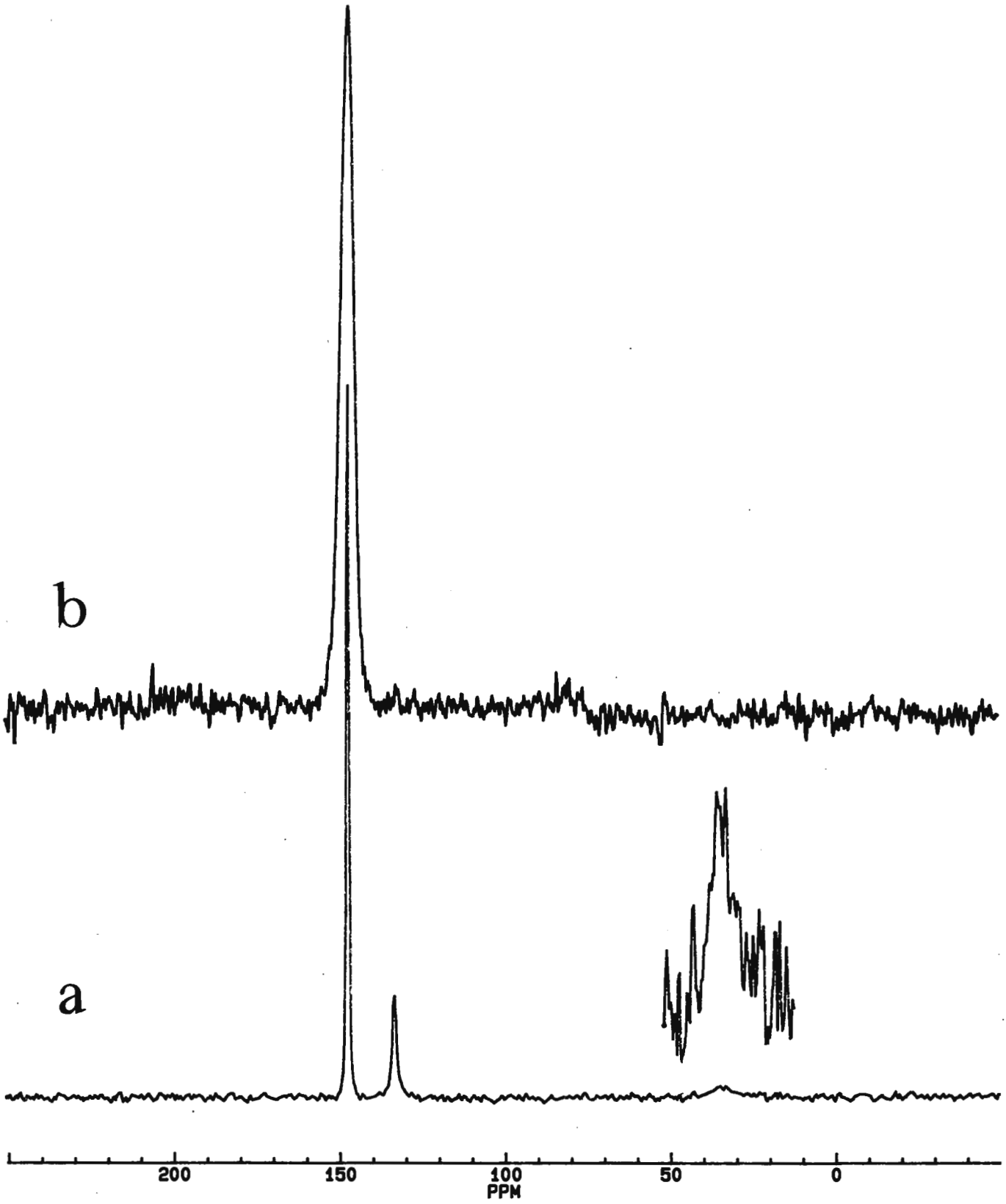
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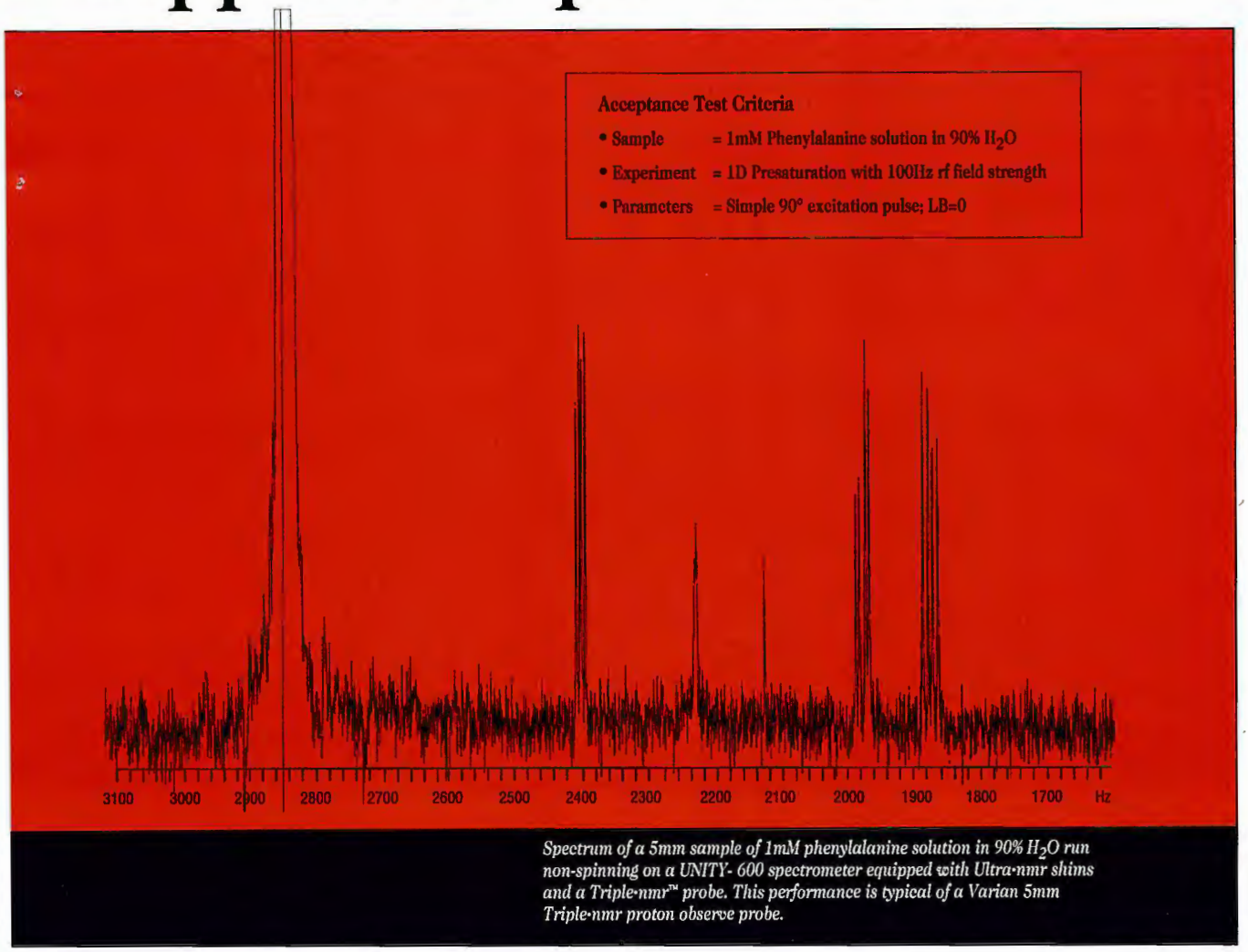

Spectrum of a 5mm sample of 1mM phenylalanine solution in 90% H_2O run non-spinning on a UNITY- 600 spectrometer equipped with Ultra•nmr shims and a Triple•nmr™ probe. This performance is typical of a Varian 5mm Triple•nmr proton observe probe.

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

June 4, 1992
(received 6/15/92)

CAN NMR SUSTAIN THE FOREST ?

Dear Barry,

Sustainable forestry is critically dependent on the production and planting of seedlings. The requirement for 1992 in British Columbia alone is for 237 million nursery seedlings to be produced and planted. The cost of the 600 million seeds required for this process is $340,000. For conifers to germinate, the seeds must be taken out of their dormant state by a process called "stratification", typically a combination of moisture and chilling treatments. However, there is no rapid, simple technique for assessing the readiness of seeds to germinate. We would like to see if NMR offers some insight into physiological changes during stratification and also provide a method to determine whether tree seeds are ready to germinate.

Rutar[1] has shown that magic angle spinning removes line broadening due to differences in magnetic susceptibility in plant seeds. In the case of the seeds we have studied, linewidths are reduced from about 400 Hz to 10 Hz. The spectra in the figure show some of the changes that take place during germination. In the dormant state only signals from mono, di, and trienoic fatty acids are observed. After stratification, the seeds were soaked in water and signals from sucrose can be seen within a few hours. Upon germination, after 10 days in a moist environment, signals from other carbohydrate and protein fragments are observed.

Timing is critical in seedling production and a repeat sowing may not be possible. This method shows great potential in predicting successful stratification and germination.

Sincerely,

Caroline Preston

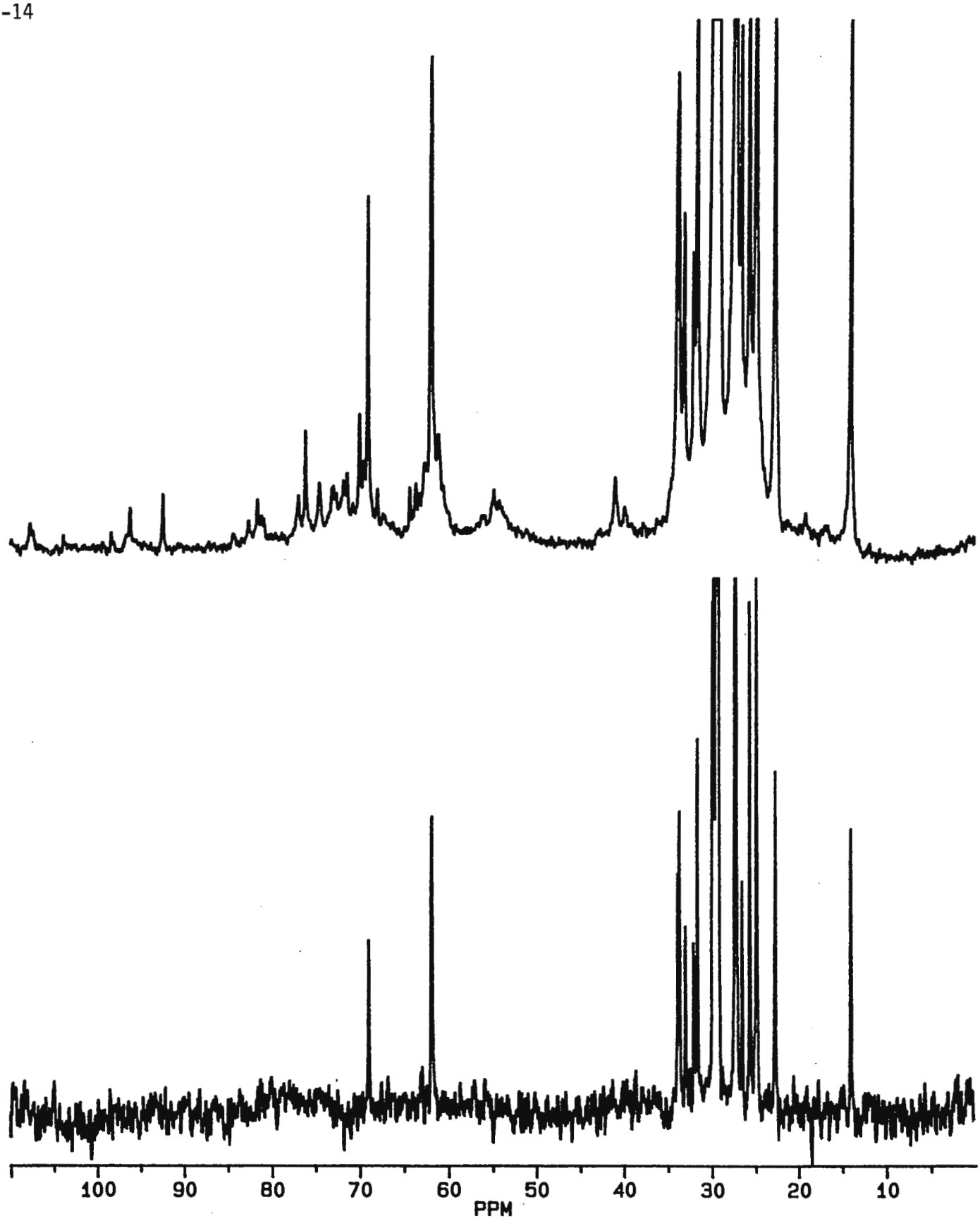
Brian Sayer

Caroline M. Preston
Pacific Forestry Centre
Victoria, B.C.

Brian G. Sayer

1. V. Rutar, J. Agric. Food Chem. 1989,37, 67-70.

Please credit this contribution to Alex Bain's account.



Top. Part of the ^{13}C spectrum of germinating *Picea Sitchensis* seeds. Bottom. Part of the ^{13}C spectrum of a *Picea Sitchensis* seed.

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

May 28, 1992
(received 6/4/92)

Dear Dr. Shapiro:

We recently received the first pulsed field gradient (PFG) triple resonance probe from Varian. The probe and associated hardware are part of our UNITY 500 MHz NMR system. As our work focuses on molecular structures important to the understanding and control of carcinogenesis we have begun investigating the utility of field gradients for solvent suppression and coherence selection in 2D and 3D experiments. We would like to provide some practical insight into setting up PFG experiments.

The reproducibility and accurate control of gradient pulse areas is critical for best PFG results. In setting up the ^{1}H-^{15}N HMQC experiment we were delighted to find that the amplifier driving the PFG is remarkably stable and linear. In the PG-HMQC experiment the applied gradients are scaled by the ratio of the ^{1}H single quantum and ^{1}H-^{15}N double quantum frequencies which is theoretically a factor of 1.1014 when using the two gradient pulse version of the HMQC experiment. Mismatching the area of the gradient pulses results in severe attenuation of signals arising from the desired coherence pathway. For gradient strengths of 30 G/cm and a standard sample length, a 1 microsecond mismatch of the gradients results in only 94% of the signal remaining while with a 2 microsecond mismatch only 22% of the signal remains. We are happy to report that gradient areas are reproducible to levels far below this level and can easily be controlled to such fine specifications. The observed results are close to the theoretical model which assumes linear gradients across the sample region and a uniform excitation and detection of spins along the gradient axis. In figure 1 is a series of spectra showing the first increment of a series of ^{1}H-^{15}N HMQC experiment with gradient time arrayed. The experiment was performed on a (Ala)^{13}C(Gly)^{15}N(Gly) sample in DMSO. The first gradient in all cases is 5 msec in duration and 30 G/cm in strength. The step sizes are about 2 parts per ten thousand in gradient pulse area. In figure 2 is PG-HMQC ^{1}H-^{15}N spectrum of ^{15}N enriched

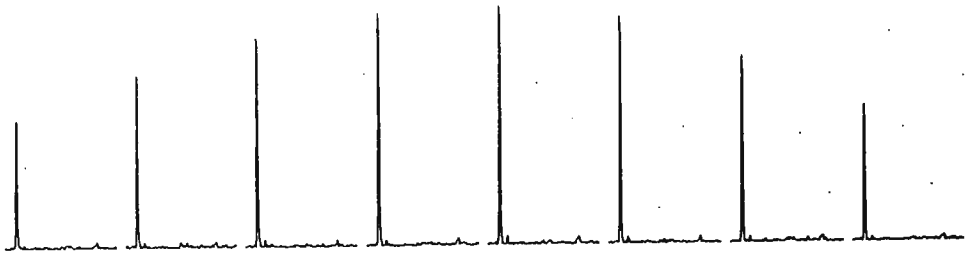
nisin. We are excited about the potential for this device for improving existing experiments and opening up new avenues of research here at UNMC.

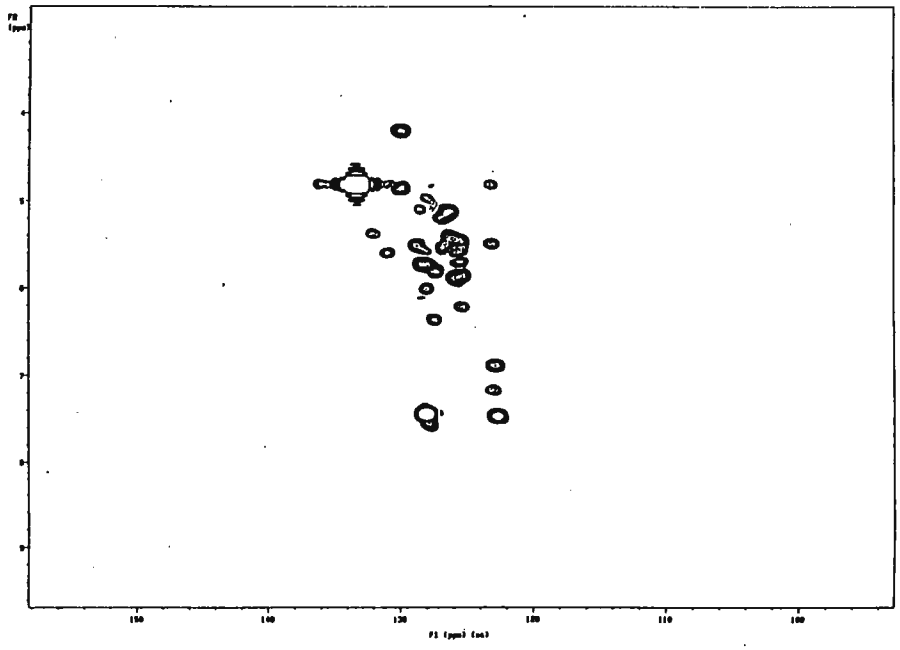
Sincerely yours,

William H. Gmeiner
Assistant Professor
Eppley Cancer Institute, UNMC

Timothy Saarenin, Ph.D.
Product Manager
Varian, Inc.

P.S. - Please start a subscription for Dr. Gmeiner with this contribution.





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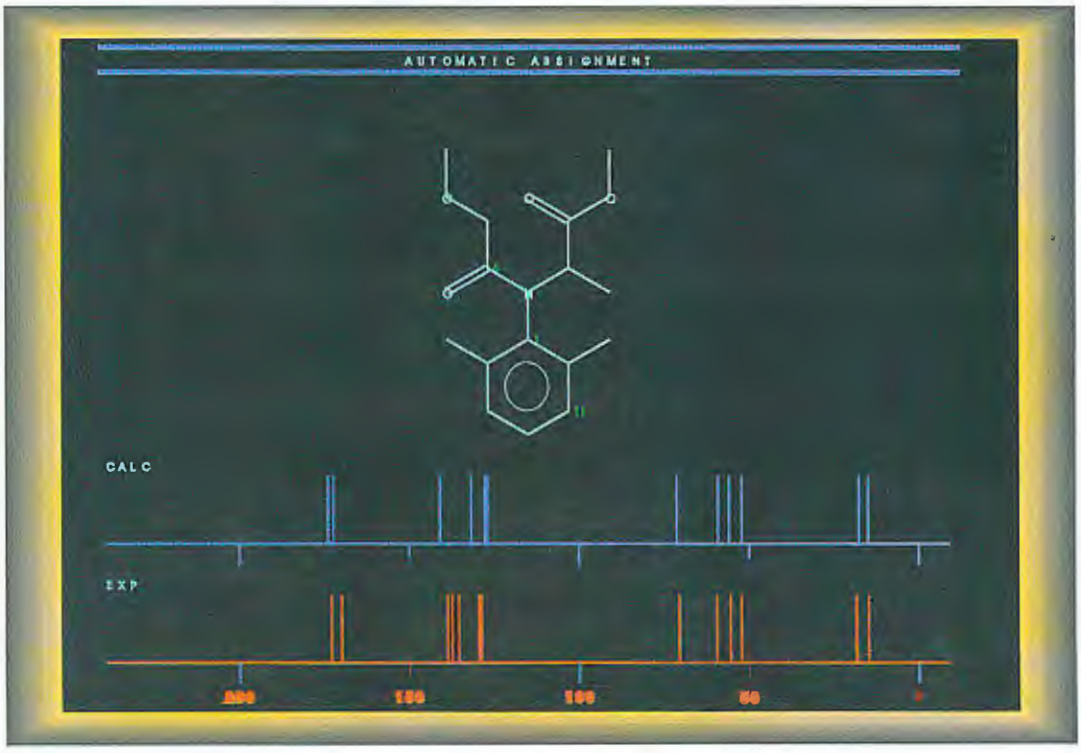

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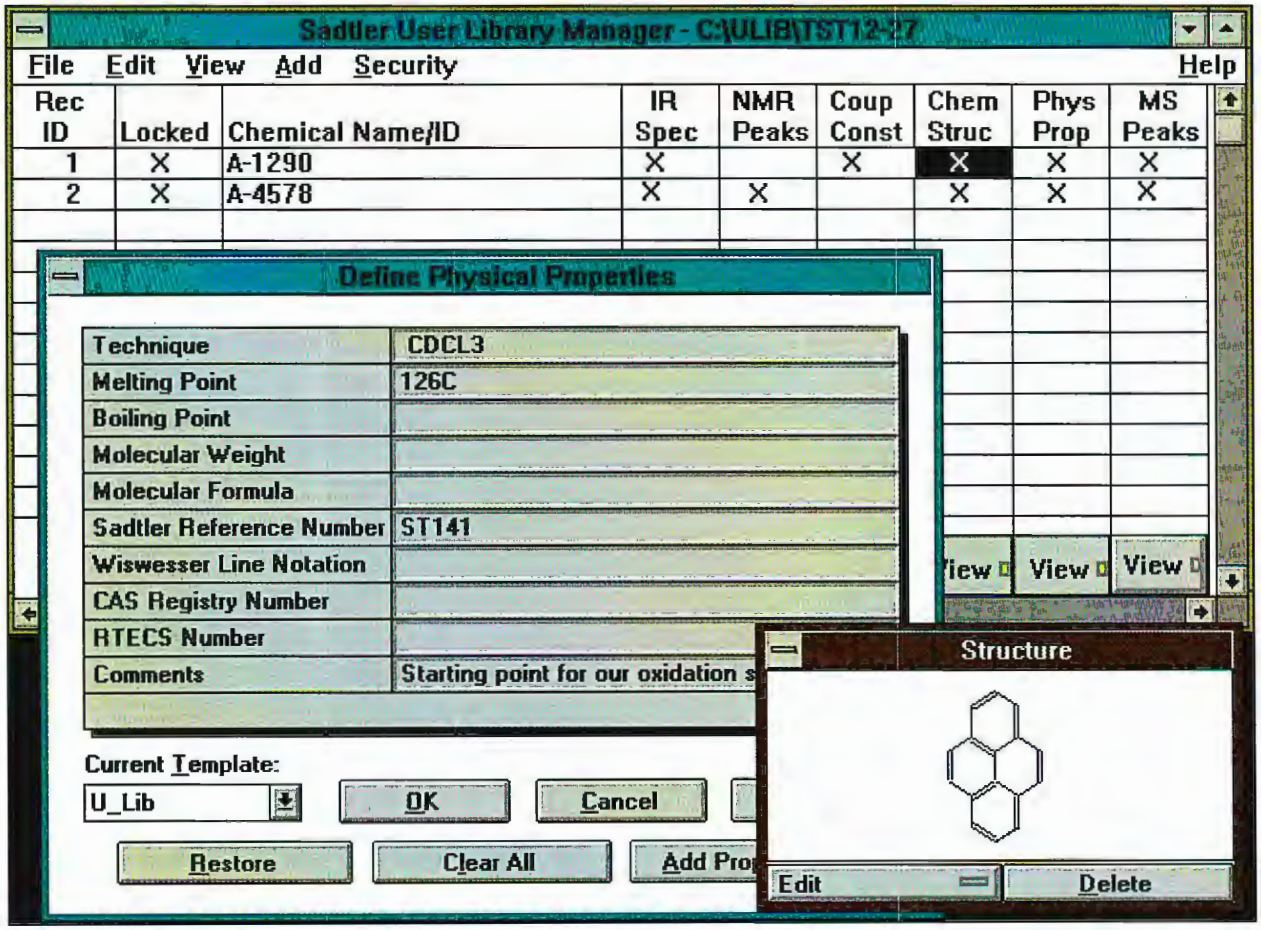

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24 May 1992
(received 5/30/92)

Professor Bernard L. Shapiro
TAMU NMR Newsletter
966 Elsinore Court, Palo Alto
California 94303 USA

Observation of splittings in ^{13}C CP/MAS spectra due to J(^{14}N,^{13}C)

Dear Barry,

In high-resolution ^{13}C CP/MAS studies of solids one generally observes asymmetric doublets with relative intensities of 2:1 for carbon nuclei which are directly bonded to ^{14}N. This phenomenon is now well understood and arises because of a breakdown of the high-field approximation.[1] Qualitatively, the ^{14}N Zeeman levels are strongly perturbed by the quadrupolar interaction; hence, the ^{14}N,^{13}C direct dipolar interaction, D, is not averaged to zero by MAS. The splitting in the asymmetric doublet is proportional to $\chi(^{14}N)D/\nu(^{14}N)$, where all symbols are standard. Typical residual splittings are on the order of 0-300 Hz for spectrometers operating at ^{1}H frequencies of 150-300 MHz. Usually indirect ^{14}N,^{13}C spin-spin coupling constants, J's, are not observed in the solid state because these coupling constants are generally much smaller than typical ^{13}C CP/MAS line widths.

Analysis of the ^{13}C CP/MAS spectrum of a highly crystalline sample of $[(n\text{-}C_3H_7)_4N]$ $[Cd(SCN)_3]$,**1**, indicates the presence of three non-equivalent thiocyanate groups (see fig.1), consistent with the known trigonal-bipyramidal structure of this compound.[2] The most interesting feature of the spectrum is that the line shape of one of the carbons, C_{eq}, exhibits a splitting which is due to indirect ^{14}N,^{13}C spin-spin coupling. Since only the portion of the ^{13}C line shape associated with the nitrogen $m_I = +1$ and -1 spin states is affected by the J interaction, the observed splitting is amplified by a factor of two. The interpretation of the ^{13}C CP/MAS spectrum at 4.7 T was also confirmed by measurements at 9.4 T using our new wide-bore AMX-400 spectrometer. To our knowledge this is the first reported observation of a splitting due to J(^{14}N,^{13}C) in the solid state. Further details will appear in the new journal, Solid State NMR.

Yours sincerely,

Roderick E. Wasylishen
Professor of Chemistry

Klaus Eichele
Killam PDF

1. For example see R.M. Dickson, M.S. McKinnon, J.F. Britten, and R.E. Wasylishen, Can.J. Chem. **65**, 941, 1987.
2. M.Taniguchi and A. Ouchi, Bull.Chem.Soc.Jpn. **62**,424,1989.

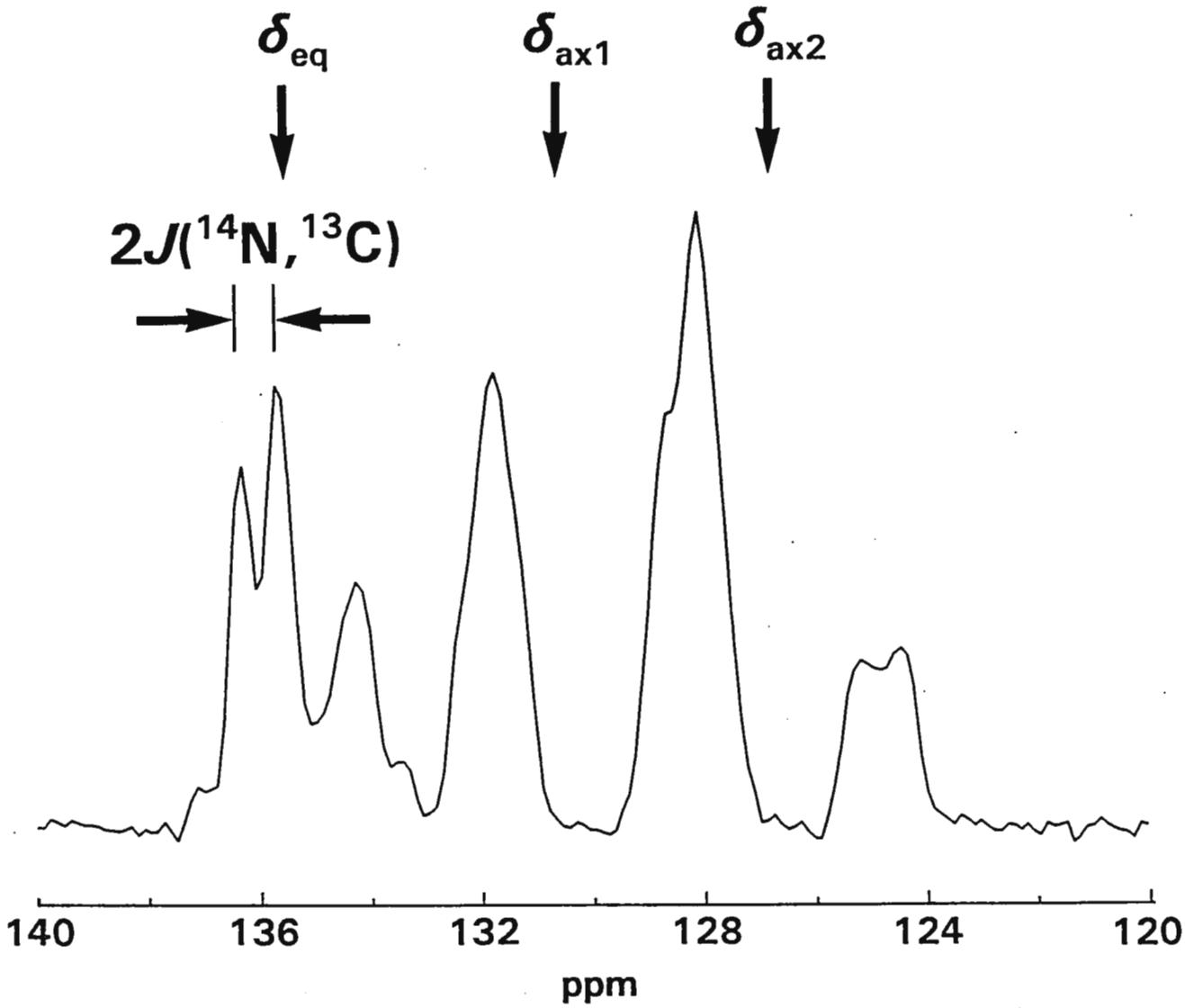

Fig. 1: ^{13}C CP/MAS spectrum of $[(n\text{-}C_3H_7)_4N][Cd(SCN)_3]$, showing the peaks due to the carbons of the three non-equivalent thiocyanate ligands. The positions of the isotropic chemical shifts are indicated by arrows. The carbon nucleus of the equatorial ligand displays splitting due to $^1J(^{14}N,^{13}C)$ = 16 Hz and is flanked by satellites due to $^2J(^{111/113}Cd,^{13}C)$ = 75 Hz.

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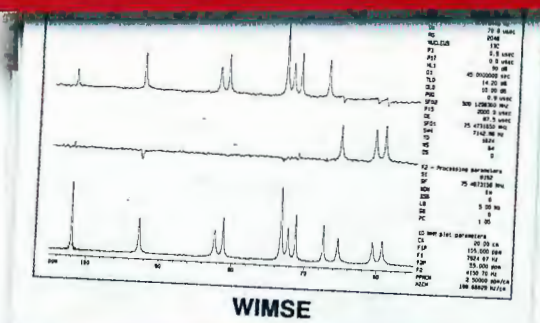

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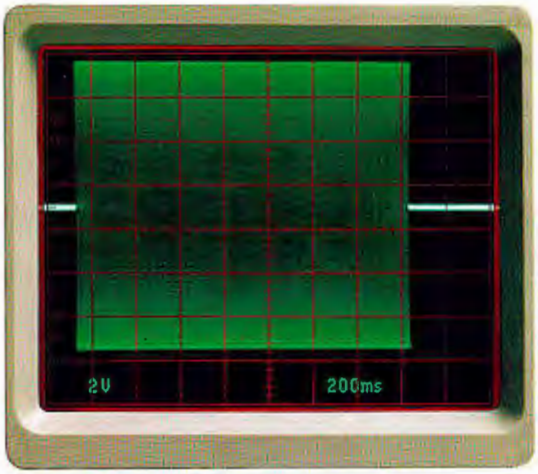

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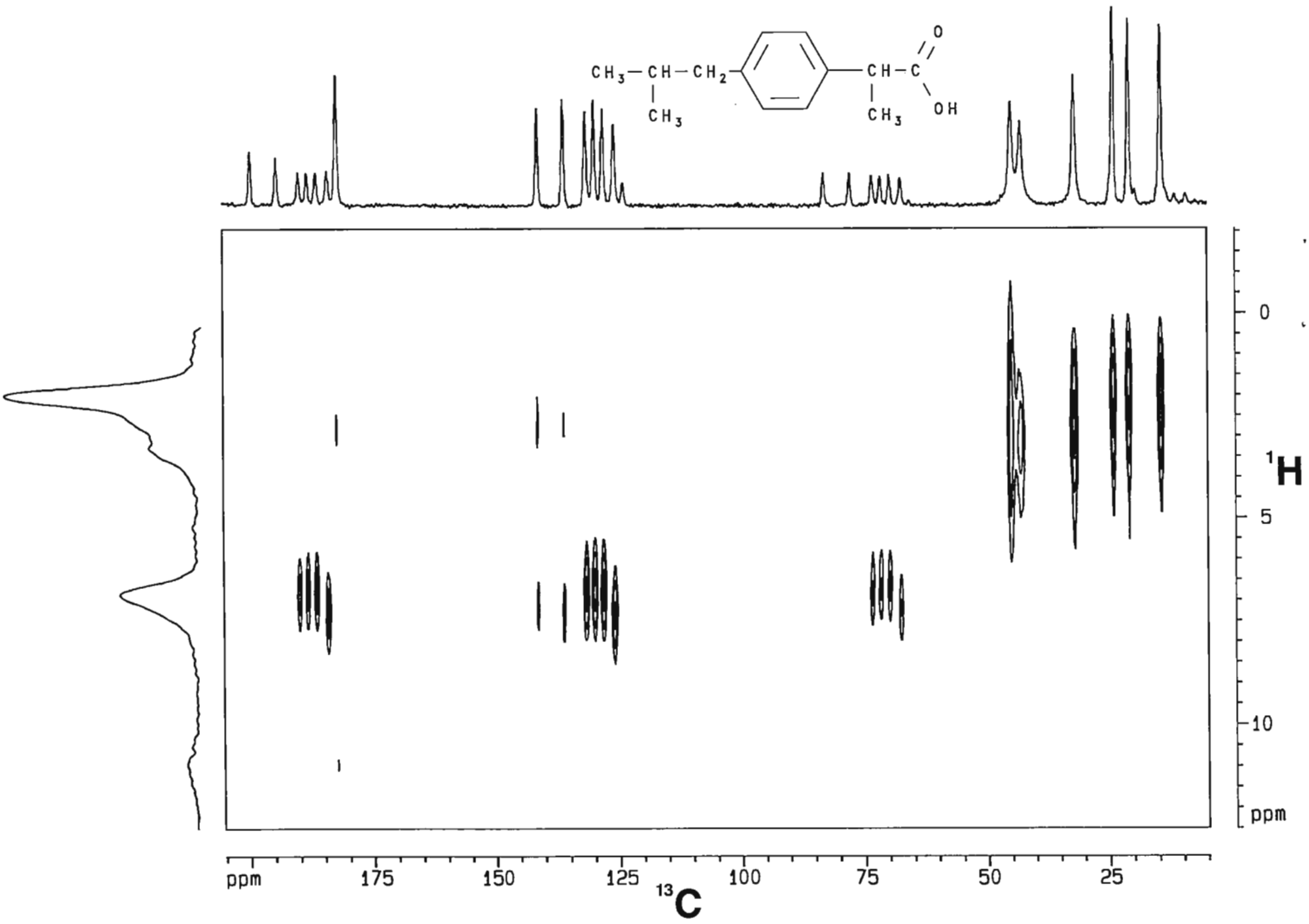

Solid State 2D HETCOR of Ibuprofen

In this spectrum the solid state 2D HETCOR experiment, developed in the Bruker applications laboratory, has been applied to the pharmaceutical compound ibuprofen. For comparison, 1D ^{13}C CPMAS and BR-24 ^{1}H CRAMPS spectra are displayed along the axes in place of the usual 2D projections. Strong cross peaks are visible for all of the directly bonded proton-carbon groups, as well as first order sidebands for the four aromatics. In addition, each of the three non-protonated carbons is shown to correlate to at least two long range protons. Note that minute differences in proton chemical shift, impossible to resolve in the 1D CRAMPS spectrum, are clearly visible in the 2D spectrum. 2D HETCOR requires simultaneous, fully windowless pulse sequences to be applied to both protons and carbons during the evolution and mixing periods. All the data shown here was obtained and processed on a Bruker ASX-300.

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Department of Physics

Dr. Barry L. Shapiro
TAMU Newsletter
966 Elsinore Court
Palo Alto, CA 94303

(received 6/15/92)

DIPOLAR FILTERED PROTON NMR AND SiH_2

Dear Dr. Shapiro,

The proton NMR line for hydrogenated amorphous silicon usually shows two principal components: a 25 kHz FWHM Gaussian from more clustered hydrogen and a 4 kHz FWHM Lorentzian from less clustered hydrogen. At low temperatures there also is a 176 kHz doublet[1] from trapped o-H_2. When appreciable SiH_2 exists there is a 14.6 kHz doublet[2], which is difficult to resolve in the presence of strong dipolar coupling to nearby hydrogens. Recently, we have developed a dipolar filtering technique which can improve the structure resolution of proton NMR for a-Si:H. The filter is a Jeener-Broekaert three-pulse sequence: 90_x-τ-α_y-τ_1-β_y-t-echo. Right after the third pulse a JB echo may be formed and at $t=\tau$ a stimulated echo is superimposed as shown in Fig.1a. Our studies have been focused on the stimulated echo. We have found that for a-Si:H, hydrogens in the more and less clustered phases, o-H_2, or SiH_2, have very different T_2 and T_{1d}. These differences provide a good way to eliminate those components which have small T_2 or T_{1d}. With this dipolar-filtering technique, the 14.6 kHz Pake doublet of SiH_2 has been resolved up to 220 K. In Fig.1b $\alpha=\beta=45^o$ and the Fourier transform was performed on the stimulated echo at $t=\tau$. To see the 14.6 kHz doublet for SiH_2, we use both the $T_2(\tau=50\ \mu s)$ and $T_{1d}(\tau_1=20$ ms) filters to cut down the signal from the clustered hydrogen which has a short T_2(about 20 μs) and the signal from the less clustered hydrogen which has a short T_{1d}(about 10 ms). Because SiH_2 and o-H_2 have longer T_2 and T_{1d}, both of them pass the two filters. In Fig.1b, at T=7 K there are two doublets: one for SiH_2(14.6 kHz) and the other for o-H_2(176 kHz). As temperature increases the 176 kHz doublet is motionally narrowed but there is no motional narrowing effect upon the SiH_2 doublet even at T=220 K.

1. J.B.Boyce and M.Stutzmann, Phys.Rev.Lett. 54, 562(1985).
2. J.B.Boyce, *Hydrogen in Disordered and Amorphous Solids*, G.Bambakidis and R.C.Bowman,Jr. eds. (Plenum Press, 1986) p.111.

Sincerely,

P.H.Chan

R.E.Norberg

Washington University
Campus Box 1105
One Brookings Drive
St. Louis, Missouri 63130-4899
(314) 889-6276

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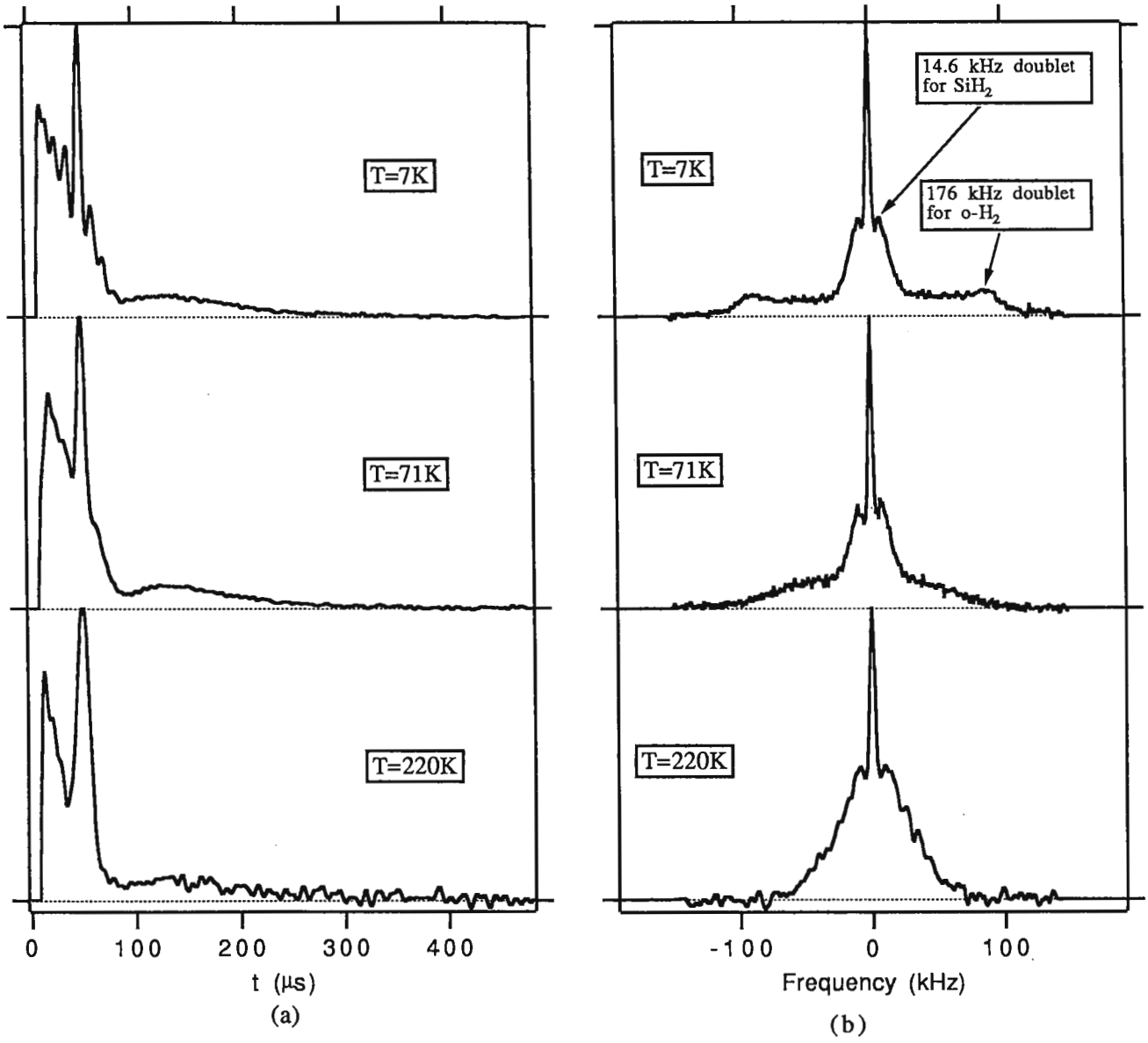

Fig. 1 (a) transient shapes of echoes at T=7K, 71K and 220K respectively. The JB echo is formed right after the third pulse and the stimulated echo is formed at $t=\tau$. (b) FT spectra of the stimulated echoes for SiH_2 and o-H_2. Motional narrowing can be seen for o-H_2 but no motional narrowing for SiH_2. The pulse sequence is 90_x-τ-45_y-τ_1-45_y with τ=50μs and τ_1=20ms. The FT begins at the $t=\tau$ peak of the stimulated echo.

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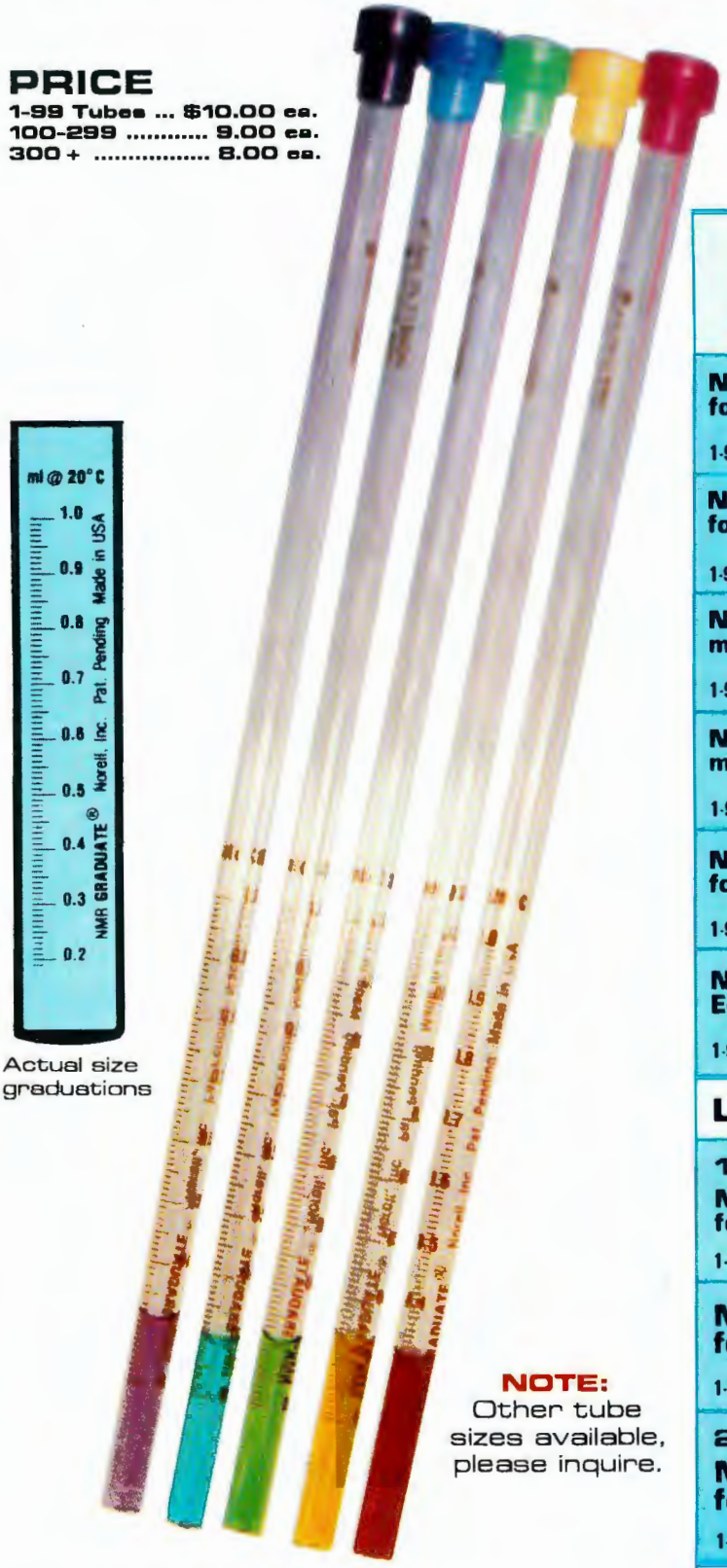

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TLC60115	Lithium-^{6}Li Fluoride 95%	1g 5g 10g	$70.00 $225.00 $350.00
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Natural Abundance: 92.58%
Nuclear Spin: 3/2; Frequency at 11.75T: 194.365 MHz

Catg. No.	Compound	Quantity	Price
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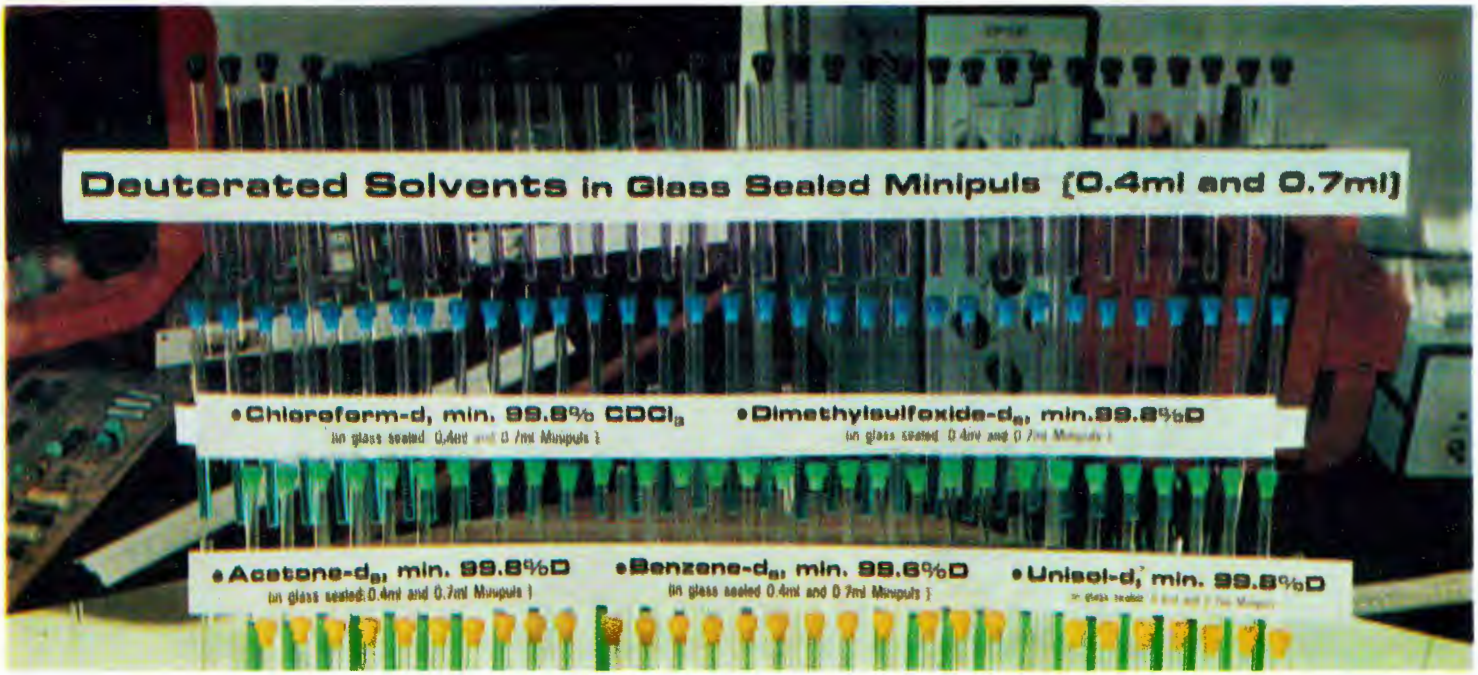

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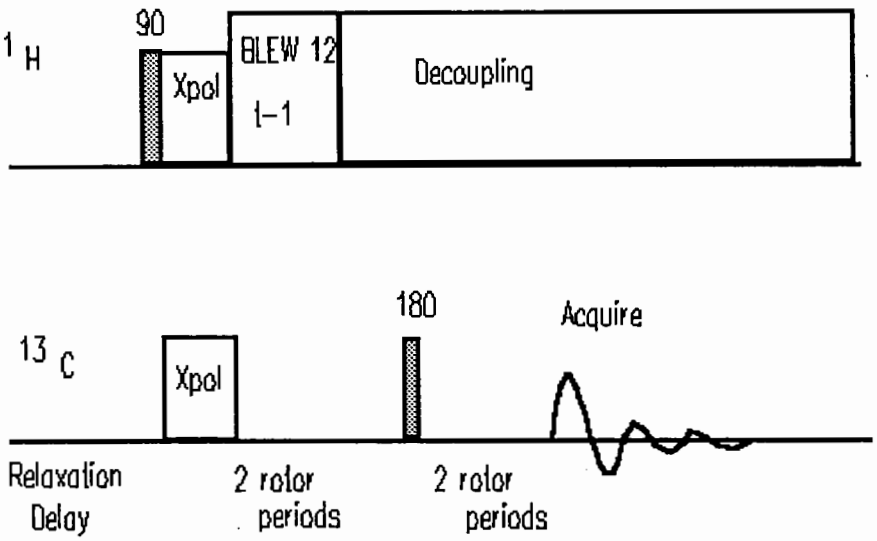
June 10, 1992 (received 6/11/92)

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

Separated Local Fields - Application to Starch

The separated local fields technique[1] is a solid state 2D method which displays a ^{13}C spectrum along the F-2 dimension and C-H dipolar coupling information along the F-1. With help from Doug Burum from Bruker and Chuck Bronnimann from Chemmagnetics, I was able to run the sequence on our Varian VXR -400. The sequence is shown below.

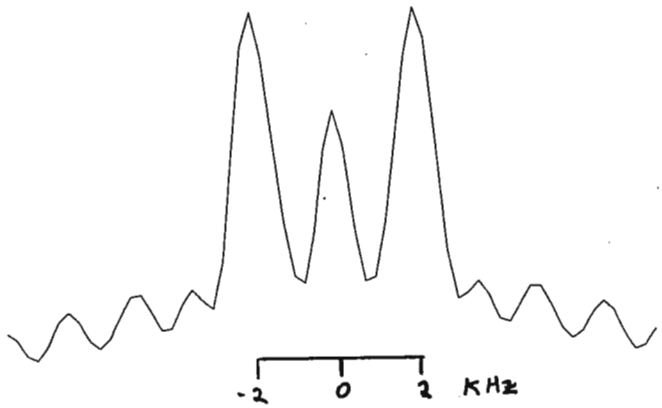


Using BLEW-12 proved to be feasible and it is supposedly more fool-proof than other multiple pulse dipolar decoupling sequences. In order to keep the t1 increments short , it was necessary to use high decoupling power (about 80 Watts), resulting in a 90^{o} pulse of 4 usec. or less. This corresponds to an F-1 width of 21 kHz. This 90^{o} pulse must be calibrated for the BLEW-12 to be effective.

Although starch does not have a large CSA, it was still necessary to spin at 2 kHz on our instrument to get a satisfactory presentation in the F-2. The time between the cross polarization and the ^{13}C refocussing pulse and that between the pulse and acquisition was 2 rotor periods, 1000 usecs. This allows 20 t1 increments and still retain acceptable sensitivity.

To get proper phasings in F-1, it is necessary to record the first FID with t1=0 or no BLEW-12 cycle and then to use 1....N cycles. It is also necessary to discard the imaginary part in the Fourier transfromation down the t1 axis.

An F-1 slice down F-2 = 103 ppm, C-1, is shown below. Slices through the other carbon resonances, with the exception of C-6 look similar. It is hard to compare the pattern with the results reported by Schaefer et al.,[2,3] since the sample was spun a little faster and BLEW-12 shrinks the dipolar pattern more than WAHUHA. However, the spectrum probably indicates that the anhydroglucose units of Waxy 1 starch are not experiencing motions of significant amplitude. 180° spin flips can be ruled out from published x-ray data.



Gary Juneau

1. M. G. Munowitz, R. G. Griffin, G. Bodenhausen, T. H. Huang, J. Am. Chem. Soc., **103,** 2529-2533, (1981)
2. J. Schaefer, E. O. Stejskal, R. A. McKay, W. T. Dixon, Macromolecules, **17,** 1497-1489, (1984)
3. J. Schaefer, M.D. Sefcik, E. O. Stejskal, R. A. McKay, W. T. Dixon, R. E.Cais, Macromolecules, **17,** 1107-1118, (1984)

PETROLEUM RECOVERY INSTITUTE

Dr B L Shapiro
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto, California
USA 94303

May 21, 1992
(received 6/1/92)

Miscellaneous Applications of Materials MRI

Dear Barry,

The installation of our Bruker Biospec-X 24/30 imaging system has been completed for some time now and we have reached a steady-state operation at the moment. Despite being the first to acquire this model with its associated (and impressive) image processing software it has been remarkably well-behaved under the circumstances. Down time since installation (more than 5 months now) has been 0%.

For the moment I just thought that I would give a brief overview of some of the applications encountered to date and show a few images (although it is difficult to do justice to the data when it's reproduced).

In anticipation of summer (since it's snowing outside as I write this) I include on the next page an image of the core of an uncooperative golf ball. The heterogeneity apparent in the back projection image was indeed found to be real from subsequent visual inspection of the core after cutting it up.

We have also been routinely running core samples under a variety of conditions. We now have one homebuilt core holder (about 11.7 cm diameter and almost 140 cm long) operated at ambient temperature up to 1000 psi. Our newly acquired Temco MRI cell is in testing and is capable of operation at 5000 psi and 300F. Other specialty holders are under design by our talented shop personnel. Rudimentary variable temperature capabilities have been added for several probes while we design a more elegant system for future use, particularly with polymer samples.

Other images shown in this communication include: (i) a comparison of a long echo time image of a rubber by MSME with that obtained by back projection at much shorter echo times, (ii) an oil saturated core, and (iii) a spin echo image of a human hand.

Other applications have included imaging of seeds, a wide variety of polymer composites/blends, flowers, fruit, wood, plants, sludges and emulsions.

More details will follow in our next contribution.

Dave

Dave Axelson
Group Leader
Founders Imaging Centre

Dave - Snow on 5/21 (or even 21/5)!! Now I remember why I left the old sod for more clement climes. Re your image of the golf ball (sure it's not a snowball?): It will putt more trulyful if it was more uniform and spherical.

Barry.

Petroleum Recovery Institute
3512 - 33 Street N.W., Calgary, Alberta, Canada
T2L 2A6

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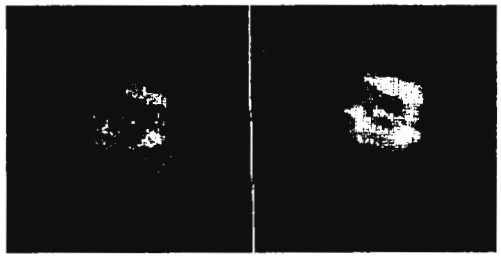
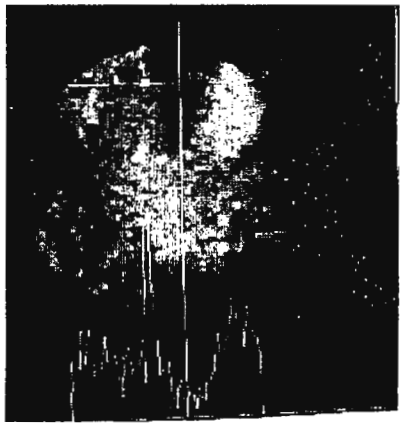
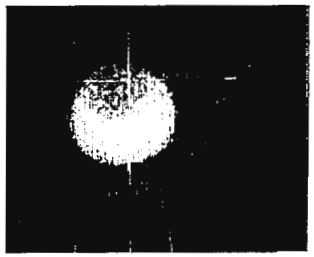


Rubber
Left side: MSME (TE = 15 ms)
Right side: Back projection (TE = 2.8 ms) Both images have been scaled to be directly comparable.

MSME of hand



Oil saturated core.
MSME.



Golf ball core
Back projection (TE = 2.8 ms)

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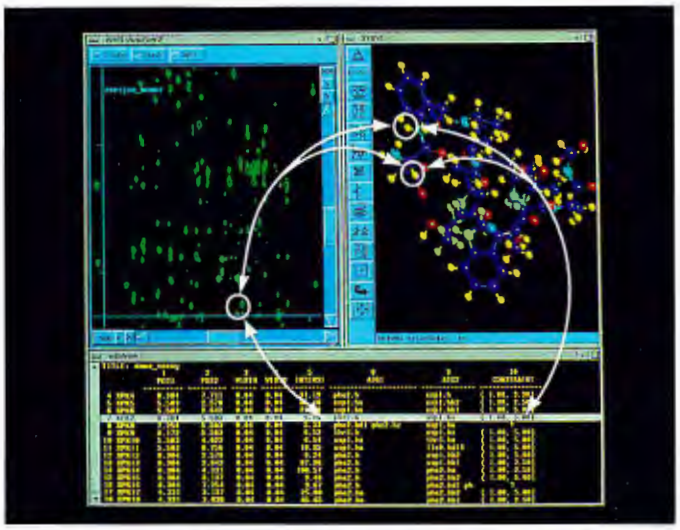

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June 2nd, 1992
(received 6/8/92)

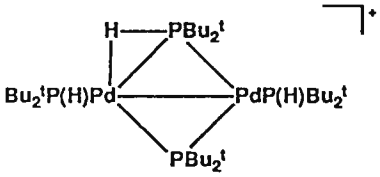
0400.83

Prof. B.L. SHAPIRO
966 Elsinor Court
Palo Alto, California 94303
USA

^{31}P-Solid-state NMR, a complementary method to X-ray Crystallography

Dear Barry,

Agostic bonding, where EH bonds, E = C, N, Si, coordinate to transition metals, continue to be of interest and we have recently reported[1] a new type of palladium(I) dimer containing an example with E = P. The presence of a Pd-H-P bridge in solution was established on the basis of ^{31}P-^{1}H correlations and one- and two-dimensional ^{31}P{^{1}H} heteronuclear Overhauser spectroscopy. Unfortunately, the solid state structure obtained by X-ray crystallography was not as conclusive as the NMR results, showing centrosymmetrical molecules. This difference might originate from either a different bonding mode in the solid state or disordering in the crystal. To fill the missing gap between solid- and solution-state, we have employed ^{31}P-CPMAS techniques (see figure). The results demonstrate that:



a) the molecule contains four non-equivalent phosphorous atoms with isotropical shifts of ca. 445, 230, 60 and 40 ppm, respectively, in close agreement with the solution data of 455, 217, 52 and 48 ppm for the phosphide, the bridging and the two terminal phosphines, respectively.

b) the high field and the one resonating at ca 230 ppm (drawn in black in the figure) have directly attached protons.

In summary it appears i) that the solution and the solid phases of $[Pd_2(\mu\text{-}PBu_2^t)(\mu\text{-}PHBu_2^t)(PHBu_2^t)_2]^+$ contain the same type of molecules with analogous bonding modes and ii) that solid-state NMR spectroscopy can give valuable and complementary information with respect to X-ray crystallography, especially in cases where static disordering is present in the crystals,therefore masking important features of the molecular structure.

[1] A. Albinati, F. Lianza, M. Pasquali, M. Sommovigo, P. Leoni, P.S. Pregosin and H. Rüegger, *Inorg. Chem.* **1991**, *25*, 4691.

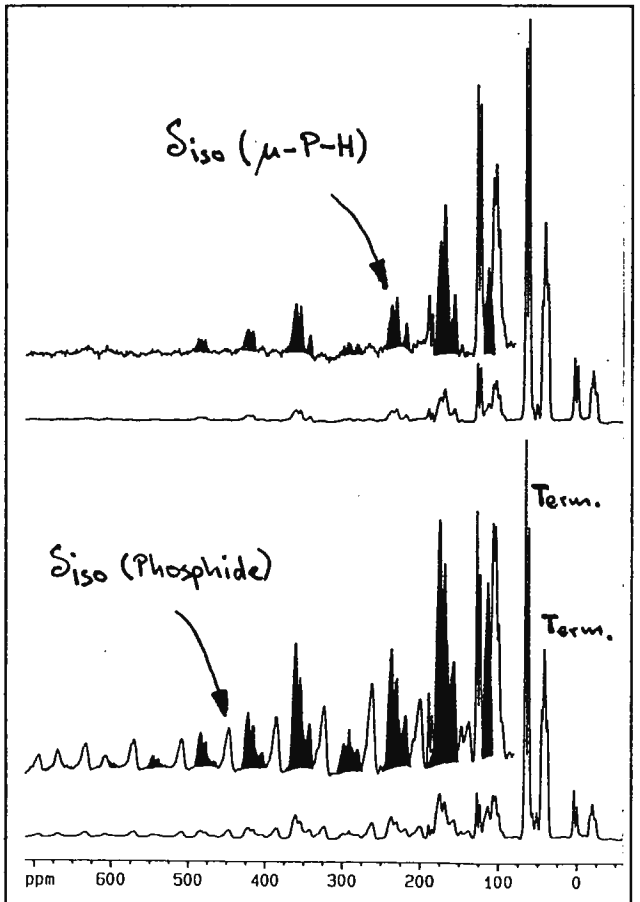


Figure: ^{31}P-CPMAS spectra of $[Pd_2(\mu\text{-}PBu_2^t)(\mu\text{-}PHBu_2^t)(PHBu_2^t)_2]^+$ recorded at 162.0 MHz with ν_{rot} = 10 KHz. The resonances of the bridging phosphine are marked in black. Lower traces: CP-time = 8 msec. All phosphorus resonances are visible. Upper traces: CP-time = 80 μsec. Only protonated phosphorus atoms show up.

Best wishes

Prof. Paul S. Pregosin

Dr. Heinz Rüegger

ROWLAND INSTITUTE FOR SCIENCE
100 CAMBRIDGE PARKWAY
CAMBRIDGE, MASSACHUSETTS 02142

June 10, 1992

Dear Dr. Shapiro:

Two postdoctoral positions in computational aspects of biomolecular nmr, including modern spectrum analysis, visualization, and computer-aided assignment, are available as part of a collaboration between Harvard Medical School and the Rowland Institute for Science. While experience in computational aspects of NMR is preferred, we are also considering candidates with computational experience in other areas of physical science. Interested candidates should transmit their CV and the names of two references to me at the address above, or to Gerhard Wagner at the Department of Biological Chemistry and Molecular Pharmacology, Harvard Medical School, 240 Longwood Avenue, Boston, Massachusetts 02115.

Sincerely,

Jeffrey C. Hoch

Jeffrey C. Hoch, Ph.D.
Fax: (617) 497-4627

JCH/lrp

UNIVERSITY OF OREGON

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Eugene, OR 97403-1229
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EMAIL: dahlquist@nmr.uoregon.edu

Dr. Bernard L. Shapiro
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto, CA 94393

May20, 1992
(received 6/6/92)

In situ pH Measurements for Protein NMR

Dear Barry,

We have been investigating the acid unfolding of sundry T4-lysozyme protein variants using ^{15}N or ^{13}C-edited proton spectra to monitor the process. We were disgusted to observe that the combination of the acidic titration conditions, KCL leakage from the pH electrodes, and accumulation of "partially unfolded" proteins lead to the rapid demise of both the NMR sample and the pH electrode. This is particularly a problem with T4 lysozyme.

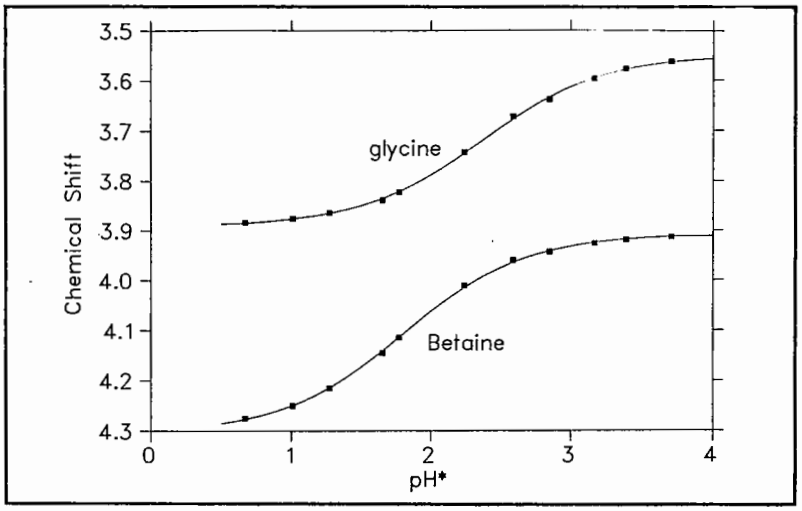

Figure 1. ^{1}H Chemical Shift of pH Indicator Proton Resonances.

We have found that the pH of the solution can be conveniently monitored by ^{1}H-NMR, thus eliminating the need for repeated glass electrode measurements of pH and the associated aggregation of protein and the proliferation of ruined pH electrodes. We use a combination of 100 μM glycine and 100 μM betaine (N,N,N-trimethyl glycine) added to the NMR sample. The ^{1}H resonance of the C_α protons of either amino acid features an easily measured titration shift in the acid range (pH 1 to 3) as a result of carboxyl group protonation as shown in Figure 1. The addition of <u>both</u> compounds provides an internal consistency check of the pH calibration as well as extending the useful pH indicator range. Although the ^{1}H resonances of glycine or betaine may sometimes overlap with protein resonances their posititions can almost always be determined since the small molecule linewidths are substantially narrower than those of the protein.

We also use ^{31}P shifts of various phosphates to monitor pH near neutrality. This can be done very conveniently when using a "reverse detection" (RPT) probe for measurements on pH-sensitive samples of protein/DNA complexes. The X-channel of the RPT probe, normally used for decoupling, is re-tuned for observation of ^{31}P spectra, without the need to remove the sample from the magnet.

Sincerely,

F.W. Dahquist

Eric Anderson

PARKE-DAVIS
Pharmaceutical Research Division
Warner-Lambert Company

(received 6/1/92)

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

Assigning the carbon chemical shift of dioxane in water

Dear Prof. Shapiro:

Recent reports on secondary structure dependent carbon chemical shifts in peptides[1] and proteins[2] have increased peoples' interest in using ^{13}C shifts as a structural diagnostic tool. In the study of such systems, dioxane is a commonly used internal reference standard. The carbon chemical shift of neat dioxane referenced with respect to internal TMS has been reported in literature[3] to be 67.8 ppm and there are many examples in the field of protein NMR where internal dioxane was referenced to this value. Recently we assigned the carbon chemical shifts for the 20 common amino acids in D_2O and D_2O solutions containing 10%, 20% and 30% v/v acetonitrile or trifluoroethanol. Since we were using organic co-solvents we thought we should check the carbon chemical shift value of our internal standard, dioxane. Surprisingly, we found the dioxane peak in water to resonate at 66.6 ppm with respect to external TMS! After further investigation we realized that there is, indeed, a 1.2 ppm difference in the carbon chemical shift of dioxane in water and neat dioxane. This discrepancy was apparently overlooked in earlier literature and has since been perpetuated. We also found that TSP resonates at -2.8 ppm relative to external TMS (Figure 1).

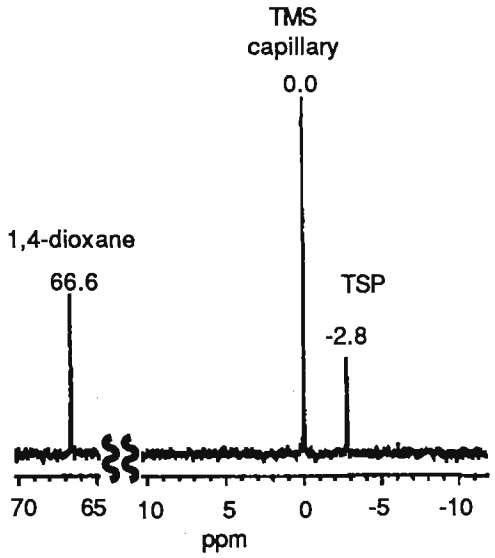

Figure 1. 125 MHz ^{13}C spectrum of 4ul dioxane in 400 ul D_2O containing internal TSP and external capillary TMS. The TMS signal is set to 0 ppm.

Please credit this contribution to D. Omecinsky.

Sincerely yours,

D. Omecinsky M. D. Reily V. Thanabal

1. M.D. Reily, V. Thanabal and D. Omecinsky, *JACS*, (1992) in press.
2. S. Spera and A. Bax, *JACS*, **113**, 5490-5492 (1991).
3. K. Wüthrich, *NMR in Biological Research: Peptides and Proteins*, North-Holland, Amsterdam, 1976, p. 164.

Model R-1500
FT-NMR Spectrometer

The Model R-1500 60 MHz Fourier Transform NMR Spectrometer. (Photo provided through the generous courtesy of the Department of Chemistry Teaching Laboratory, University of Massachusetts, Amherst, MA.)

- **FT system features high sensitivity, excellent resolution and accurate chemical shift measurements.** With field stability ensured by ^{2}D lock, the Model R-1500 delivers performance typically associated with higher field instruments.
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- **The R-1500 permanent magnet provides** minimal, low-cost maintenance.

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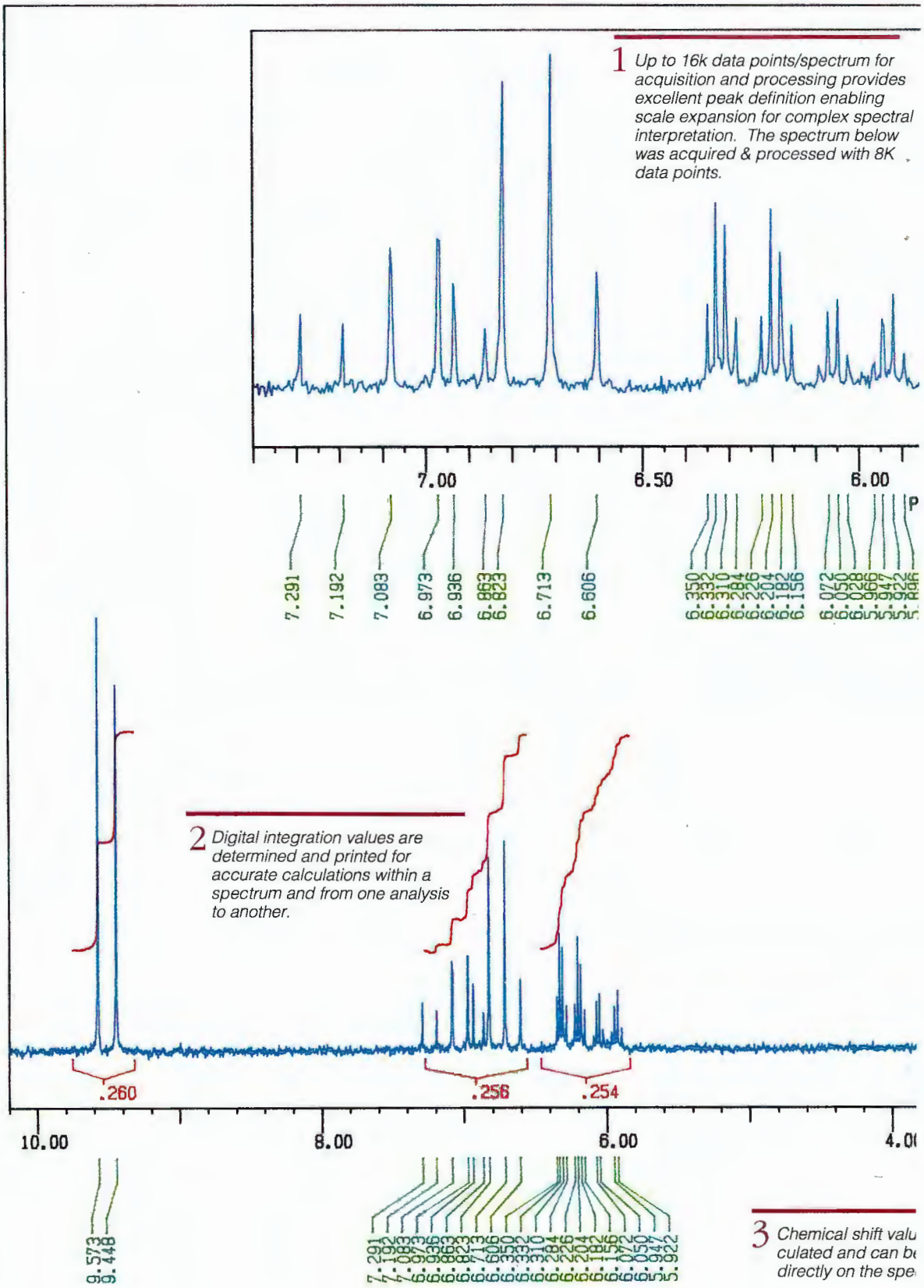
Hitachi Instruments, Inc.


1 Up to 16k data points/spectrum for acquisition and processing provides excellent peak definition enabling scale expansion for complex spectral interpretation. The spectrum below was acquired & processed with 8K data points.
7.00
6.50
6.00
7.291
7.192
7.083
6.973
6.936
6.823
6.713
6.606
2 Digital integration values are determined and printed for accurate calculations within a spectrum and from one analysis to another.
.260
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.254
10.00
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6.00
9.573
9.448
3 Chemical shift valu
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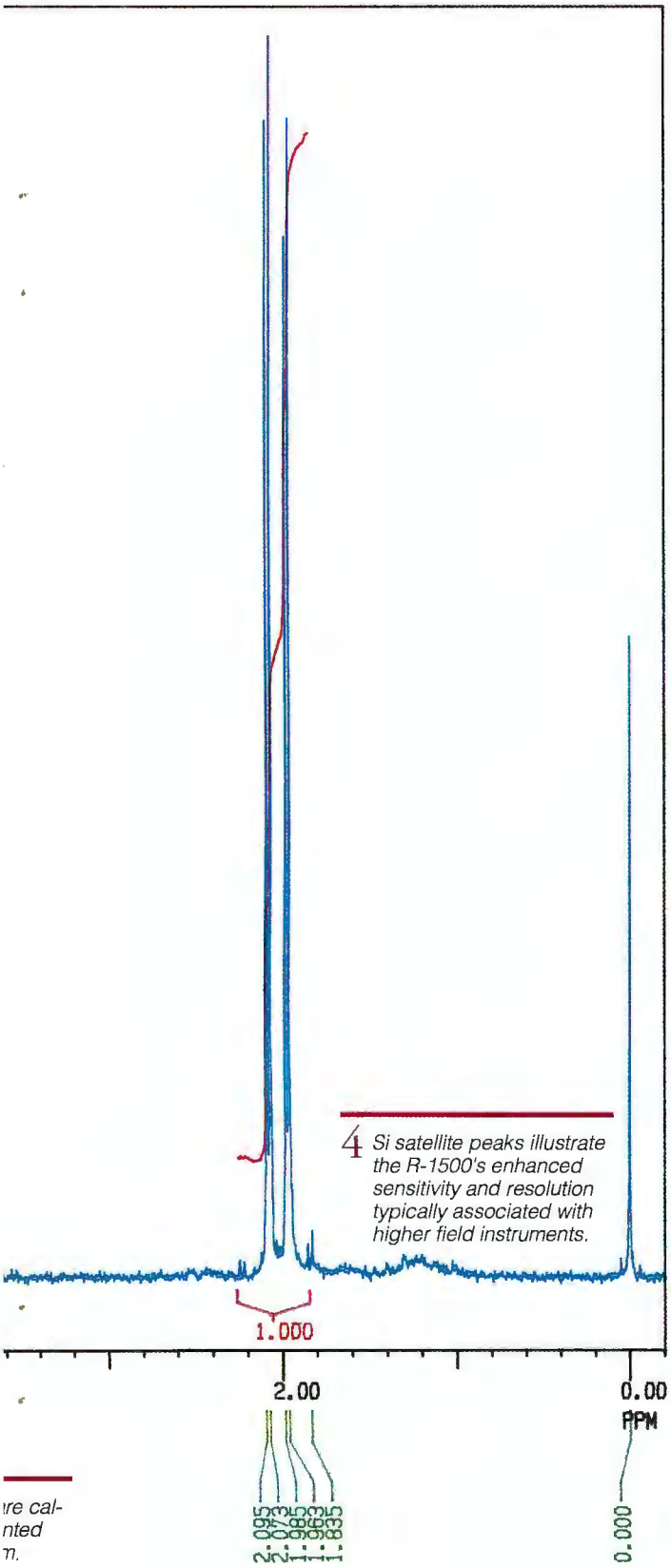


4 *Si satellite peaks illustrate the R-1500's enhanced sensitivity and resolution typically associated with higher field instruments.*

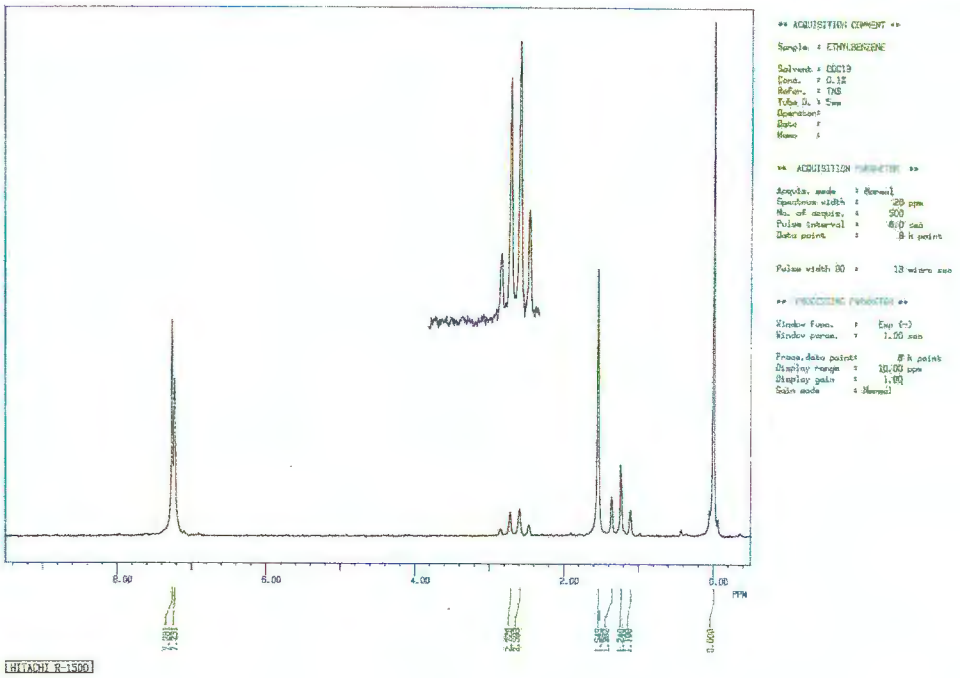
re cal-
nted
n.

```
** ACQUISITION COMMENT **

Sample  : Crotonaldehyde

Solvent : CDCL3
Conc.   : 4%

Tube D. : 5mm
Operator:
Date    : Feb 16 '88
Memo    :


**  ACQUISITION PARAMETER  **

Acquis. mode     : Normal
Spectrum width   :       15 ppm
No. of acquis.   :       16
Pulse interval   :      6.0 sec
Data point       :        8 k point


Pulse width 90   :       10 micro sec


**  PROCESSING PARAMETER **


Proce.data point:        8 k point
Display range    :    10.40 ppm
Display gain     :     1.00
Gain mode        : Normal
```

5 *Acquisition and processing parameters are stored and can be printed automatically so that all data and operating conditions are linked. Additionally, peak height tables including ppm to Hz conversion values can be printed.*

Technical Specifications

This spectrum of 0.1% ethyl benzene illustrates the excellent sensitivity and resolution obtained with the Model R-1500 FT-NMR. A S/N ratio of 77.5 was calculated for the quartet shown in the expanded portion of the spectrum. The silicon satellite peaks seen at the base of the TMS peak underscore the R-1500's outstanding resolution.



Nucleus	^1H
Resonance Frequency	60 MHz
Field Strength	1.41 Tesla; permanent magnet
Field System	Internal deuterium lock
Resolution	Better than 0.4 Hz
Sensitivity	Better than 20 (1% ethyl benzene, single pulse)
	Better than 120 (1% ethyl benzene, 150 pulses)
Pulse Sequence	90^0, 180^0 -t-90^0 (T_1, WEFT)
T_1 Measurement	Inversion recovery method
No. Of Acquisition Points	30,000 or more (12 bit accuracy)
Data Density	1, 2, 4, 8, 16K points per scan
Decouple Modes	Homo decoupling; gated decoupling
Output	4-pen, multicolored printer/plotter for A3/A4
Double Resonance	Standard
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June 11, 1992
(received 6/15/92)

Dr. B. L. Shapiro, Editor
TAMU NMR News Letters
966 Elsinore Court
Palo Alto, CA 94303

Dear Barry:

"TANGO with Spin-lock Pulses in Suppression of ^{1}H-^{12}C Magnetization."

The cyclic peptide [H-Tyr-D-Pen-Gly-Phe-D-Pen-OH]enkephalin (DPDPE), a highly potent delta-opioid selective agonist, has been studied in our laboratory by 1D and 2D ^{1}H-NMR spectroscopy for the determination of molecular conformation. However, due to overlap of some signals in ^{1}H spectrum and a need to obtain optimal information for conformational analysis, we undertook inverse proton detected C, H-correlation NMR studies.

Currently, the main difficulty encountered in inverse experiments is elimination of the large background proton (^{1}H-^{12}C) magnetization. Suppression based exclusively on phase cycling methods, imposes as a prerequisite, a spectrometer with high stability and an optimal dynamic range.[1] Significant improvements can be achieved with pulse sequences that suppress the unwanted signals with the use of a BIRD pulse.[2] However, in macromolecules, the sensitivity is reduced by spin-diffusion between protons bound to ^{13}C and ^{12}C. A different approach, employing a homospoil or spin-lock pulse at the end of a spin-echo preparation period, has been proposed to destroy the undesired ^{1}H-^{12}C magnetization.[3] However, this method requires an additional inversion pulse pair during the refocusing period for experiments such as HMQC-NOESY, HMQC-TOCSY, etc. We have observed that pulse sequences employing a preparatory TANGO pulse (acts as a 90° pulse on ^{1}H-^{13}C), followed by a short spin-lock period, can provide suppression even with relatively unstable spectrometers. This method has the inherent advantages of a pulse sequence incorporating a spin-lock pulse, with the additional merit of using fewer pulses. Since the undesired magnetization is dephased by the spin-lock pulse, an efficient suppression can be achieved regardless of the actual range of proton T_1 values. In addition, the receiver gain can be increased significantly, resulting in a substantial increase in signal to noise.

Sincerely yours,

Victor J. Hruby Katalin E. Kover Om Prakash

1. J. J. Titman, D. Neuhaus and J. Keeler, J. Magn. Reson. 85, 111 (1989).
2. A. Bax and S. Subramanian, J. Magn. Reson. 67, 565 (1986).
3. G. Otting and K. Wüthrich, J. Magn. Reson. 76, 569 (1988).

406-48

United States Department of Agriculture | Agricultural Research Service | North Atlantic Area Eastern Regional Research Center | 600 East Mermaid Lane Philadelphia, Pennsylvania 19118

April 27, 1992

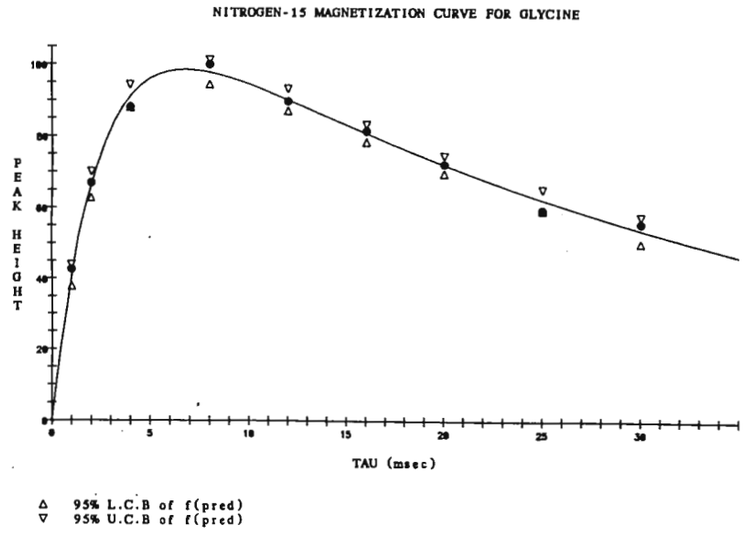
Dr. Bernard Shapiro
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto, CA 94303

NITROGEN-15 CP MATCHING USING NITROGEN 15 LABELLED GLYCINE

Dear Dr. Shapiro,

In the cross-polarization experiment one needs to achieve the Hartman-Hahn matching using a suitable compound. For solid-sate carbon-13 NMR spectroscopy adamantane is used. For solid-state nitrogen-15 NMR spectroscopy nitrogen-15 labelled ammonium nitrate has been used. In this laboratory we have had little luck matching with this compound. We have found that even though a match has been achieved using ammonium nitrate we obtain less than satisfactory natural abundance nitrogen-15 spectra. Part of the problem is the matching condition for ammonium nitrate appear to be very dependent on spinning rate.

In seeking a more suitable compound for matching nitrogen-15 labelled glycine was tested. It turned out that it was easy to acheive the match on this compound. The magnetization curve is shown below. T_{CH} is 2.35 msec and T_{1rho} is 34.5 msec. It is possible to match on this compound using a 5 msec contact time and with a 2 to 3 second recycle time.



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Sincerely yours,

Robert L. Dudley

Robert L. Dudley

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Jeffrey A. Reimer, Associate Professor

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REIMER@GARNET.BERKELEY.EDU
510-642-8011

Sir: June 15, 1992 (received 6/18/92)

We have recently been investigating the dynamics of deuterated pyridine adsorbed on powders with ^{2}H-NMR. The lineshapes with which we fit our data are generated by averaging the electric field gradient tensor of each deuteron over the path of the motion, e.g. rotations about pyridine's C_{2v} axis. Calculations of this type result in spin-1 powder patterns whose effective asymmetry parameter, η^*, depends upon the symmetry of the motion. We have considered low-symmetry, fast motions, such as incomplete rotations or 2-site flips about pyridine's C_{2v} axis, which, in general, yield asymmetric lineshapes ($\eta^* \neq 0$). Wittebort et al.[1] have documented calculations of low-symmetry motions neglecting the *inherent* asymmetry of the electric field gradient, η. For C-D bonds, $\eta \cong 0.05$. Wittebort et al. note general trends in η^* and the averaged quadrupolar coupling constant, QCC_{ave}, as a function of rate of molecular motion; these trends should not be a strong function of η. However, in *fitting* experimental data with calculated lineshapes to extract parameters of *fast* molecular motion, we have found that neglecting η results in small but significant differences in the extracted η^*, QCC_{ave}, and motional parameters.

The effect of η on the calculated lineshape parameters is shown in Figure 1. We have calculated the principle components of the averaged electric field gradient tensor for the α-deuteron of pyridine undergoing C_{2v} rotations through an angle in the range $0 \leq 2\phi \leq 360°$, for the cases of $\eta = 0.05$ and $\eta = 0$. The differences between the respective averaged tensor components for the two cases are plotted as a function of 2ϕ. Clearly, the differences, which are in range 0 - 3kHz, are large enough to influence goodness-of-fit tests in least-squares fitting algorithms. Neglecting η can therefore result in the reporting of inaccurate motional parameters, such as a rotation or flip angle.

Please credit this contribution to Raychem.

1. Wittebort, R.J., Olejniczak, E.T., Griffin, R.G. *J. Chem. Phys.* **86** (1987) 5411.

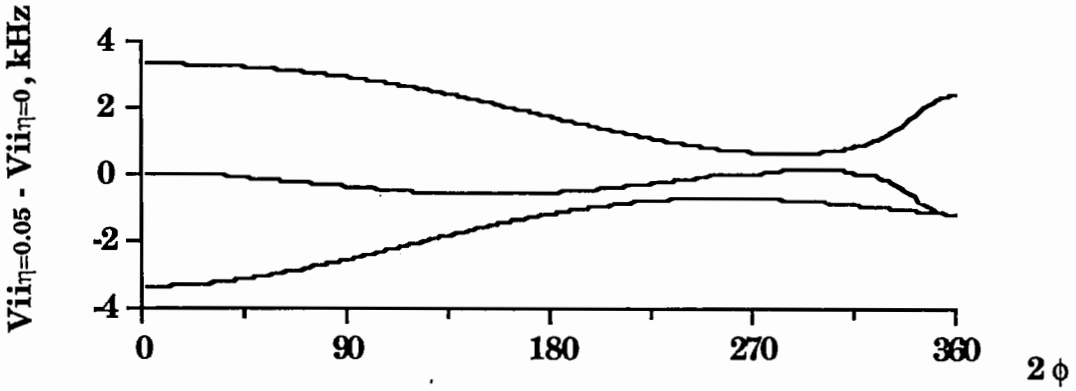

Figure 1: Plot of the differences between the three principle components of motionally averaged tensors calculated for $\eta = 0.05$ and $\eta = 0$.

Sincerely,

Phillip A. Armstrong
Research Associate

Jeffrey A. Reimer
Associate Professor

OHIO STATE UNIVERSITY

Department of Chemistry

Professor Gideon Fraenkel

Phone 614-292-4210
FRAENKEL@OHSTPY
FRAENKEL@MPS.OHIO-STATE.EDU

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(received 6/18/92)

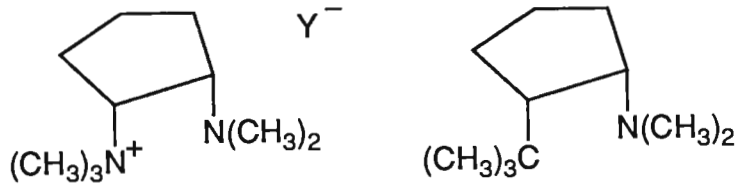
June 11, 1992

Rotation and Inversion in amino-ammonium salts

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

Dear Barry:

We have been working with different kinds of t-amines particularly cis-vicinal t-diamines as potential ligands for lithium and unaccountably, brain receptors. The salts **A** and **B** and amine **C** exhibit interesting dynamic behavior. At 200K ^{13}C NMR of $N(CH_3)_3^+$ in **A** and **B** in acetone-d_6 shows up as



A, $Y^- = I^-$ **B**, $Y^- = CF_3SO_3^-$ **C**

a 1:1:1 triplet (unequally spaced) with $N(CH_3)_2$ as an equal doublet. This doublet also appears in the carbon spectrum of **C**. With increasing temperature, the two NCH_3 multiplets in **A** and **B** each signal average to single lines at their respective centers. Clearly, the changes in the N^+-$^{13}CH_3$ resonance come from different rotation rates about the CH-$N^+(CH_3)_3$ bond while the $N(CH_3)_2$ resonance behavior is due to the rate of inversion at nitrogen with rotation around the C-$N(CH_3)_2$ bond. The latter effect also appears in the ^{13}C NMR of **C**. Activation parameters for nitrogen inversion in **A**, **B**, and **C** are respectively $\Delta H^{\neq}$(kcal/mol) of 16.7, 14.3 and 13.5 with $\Delta S^{\neq}$(eu) of 10.4, 3.5 and 9.7. These $\Delta H^{\neq}$ values are somewhat high for inversion, perhaps due to the crowded nature of these species. Much faster is rotation about CH-$N^+(CH_3)_3$ where for **A** and **B** we find $\Delta H^{\neq}$ (kcal/mol) of, respectively, 4.7 and 3.8 with $\Delta S^{\neq}$(eu) of -23.9 and -28.2. Rotation about the C-N^+ bond must be accompanied by an increase in solvation of the ion-pair in acetone-d_6. Perhaps in the transition state the two ions are further apart, 1hence requiring more solvation. Comments from interested readers are welcomed. Notice the bitnet address for easy cost-free communication. You never put out a list of our E-mail addresses. Do you accept contributions via bitnet?

I trust this finds you thriving and keeps the **dreaded pink note** from our mail box.

My entire group sends their very best regards.

Yours sincerely,

Gideon

Sharon E. Boyd

Gideon Fraenkel
Professor of Chemistry

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GF:jlp

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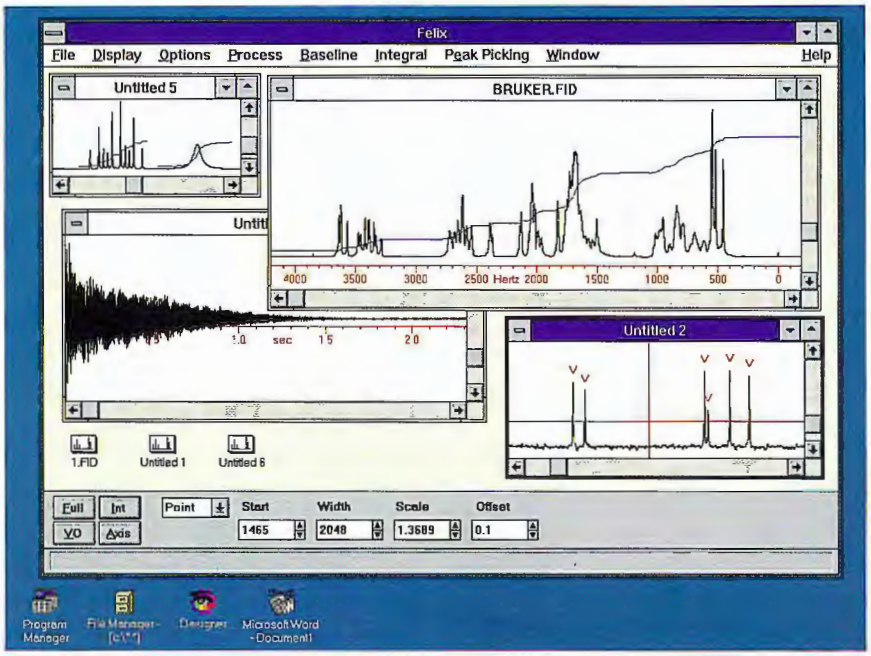

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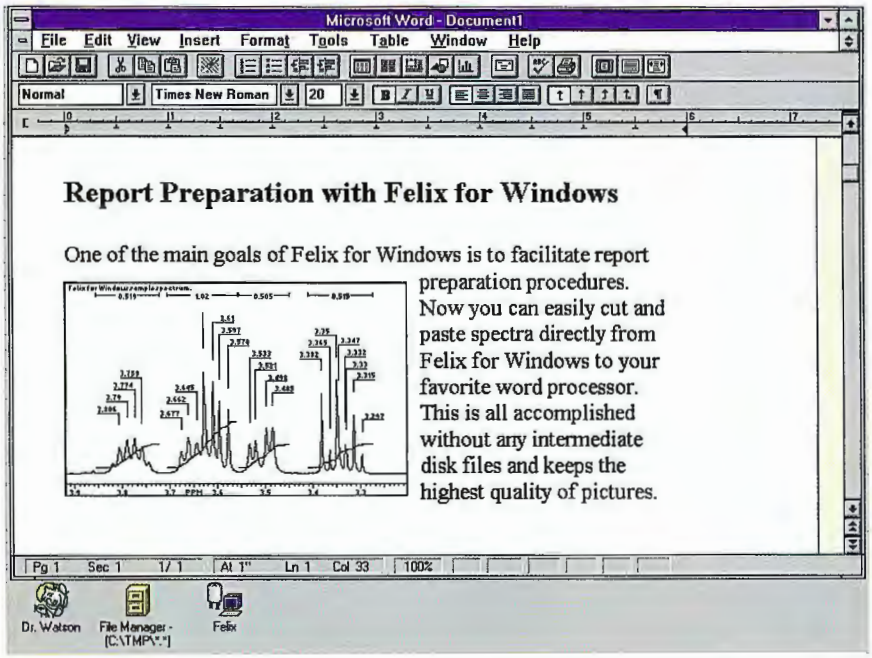

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Dr. B.L. Shapiro
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto, CA 94303

June 15, 1992
(received 6/22/92)

Dear Barry:

INADEQUATE or what?

When I was a graduate student, working with Ray Freeman and members of his group, including Stewart Kempsell, David Max, Malcolm Levitt, Tom Frenkiel and Gareth Morris, we were very excited about the development of the INADEQUATE experiment, which permitted us to measure long-range carbon-carbon couplings in natural abundance samples. Unfortunately, this application of the INADEQUATE experiment was overshadowed by its more mundane use for making ^{13}C resonance assignments. I also must admit that measuring these long range couplings with INADEQUATE was always a painstaking experience. Even at that time we were not optimistic about measuring $^{3}J_{CC}$ couplings much smaller than ~1 Hz. However, the idea never died completely: whenever I ran into some of my old colleagues we spent endless hours discussing the prospects and possible uses of such experiments.

More recently, the possibility of applying such experiments to ^{13}C-enriched proteins revived the idea. The main problems in the application to uniformly (>95%) ^{13}C-enriched proteins are the large natural line widths of ^{13}C in proteins, the very complex structure of the ^{13}C multiplet, and its relatively low sensitivity. After many more endless discussions we (my two colleagues wish to remain nameless, for the time being) found a solution to these problems. The details go too far for this short letter, but the bottom line is as follows: Forget about anything else but methyl carbons and use their strongly non-exponential T_2 relaxation to fish out the narrow component of its line shape; use 2D or 3D NMR to remove the overlap and use ^{1}H detection to enhance the sensitivity. More details will soon appear in JACS.

To make a long story short, the experiment works fine, and a small region of the spectrum from which the J values were measured is shown in Figure 1. A total of well over 100 J couplings were measured in an overnight experiment for the protein calmodulin, complexed with a 26-residue peptide. Note that the molecular weight of this complex is ~20 kDa, with C_α natural line widths of ~15 Hz (T_2 ~20 ms). Measured J couplings range from 0.7 Hz to 3.6 Hz and show the expected *gauche* (~1 Hz) *trans* (~3.1 Hz) difference for the J coupling. Consequently, for protein jocks these couplings contain very valuable information. For example, The $^{3}J_{CC}$ coupling between C_δ and C_α in isoleucine residues specifies the dihedral angle χ_2, which can be difficult to determine unambiguously by other methods. It also allows us to make stereospecific assignment of C_δ methyl groups in leucine and valine residues, and to measure χ_2 in leucines.

For proteins larger than ~10 kD, the method is restricted to couplings involving at least one

methyl carbon or carbons of residues with substantial internal mobility. However, for small ^{13}C-enriched proteins with narrower ^{13}C line widths, ^{13}C-^{13}C long range couplings involving non-mobile non-methyl carbons are also accessible.

Kindest regards,

Ad Bax N.N. N.N.

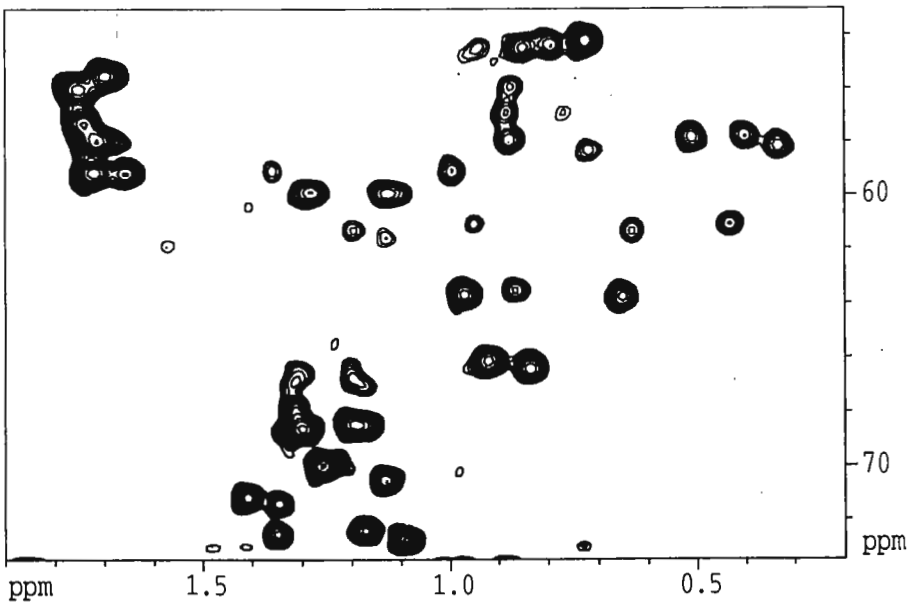
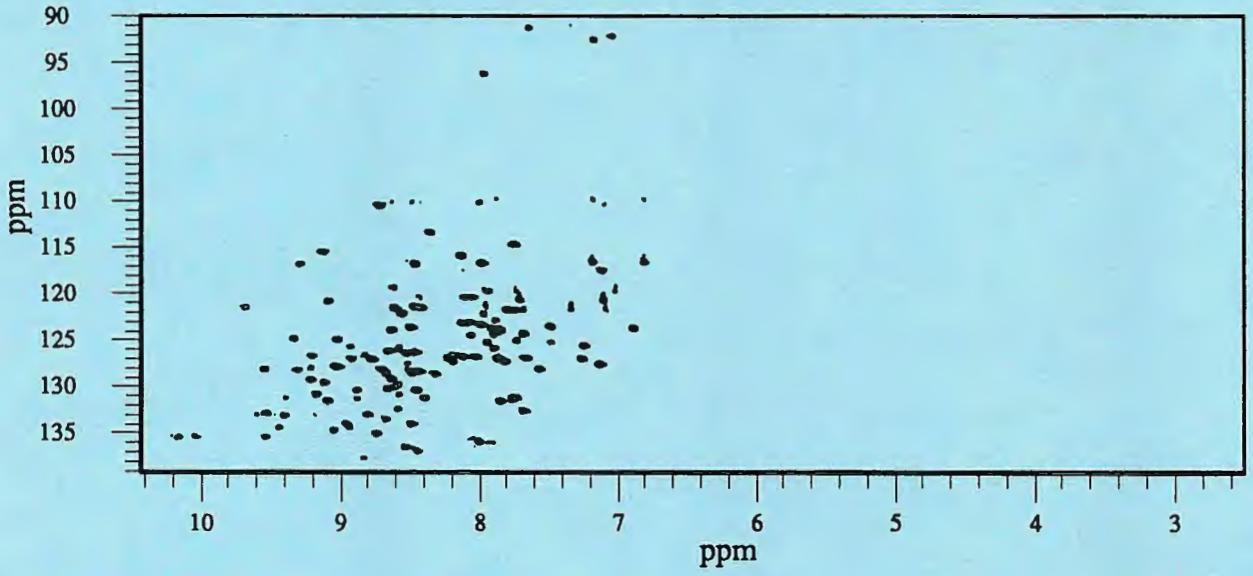


Figure 1. Small region of the ^{1}H-detected ^{13}C-^{13}C shift correlation spectrum of the calmodulin-peptide complex, showing the cross peaks between methyl and C_α resonances. The intensity of these correlations is related in a straightforward manner to the size of the long range coupling.

Gradient Techniques for Large Proteins

Inverse detected ^{15}N–^{1}H NMR techniques in conjunction with ^{15}N labelling have proved to be an invaluable tool for assigning the ^{1}H resonances of proteins in solutions. However, the solvent saturation used in the phase cycled versions of these experiments often results in a decreased peak intensity due to exchange processes. Gradient selection of multiple quantum pathways eliminates the water signal and provides an attractive alternative to traditional phase cycled methods for the following reasons:

- ***Superior Water Suppression*** – Single quantum peaks such as the water resonance are eliminated while correlations which occur at the frequency of the solvent resonance are routinely observed. The undesirable effects of traditional presaturation are also avoided.
- ***Fewer Artifacts*** – Gradient selection conveniently avoids the t_1 noise and cancellation artifacts commonly found in phase cycled experiments.
- ***Greater Sensitivity*** – Since the suppression of undesired signals occurs *prior* to acquisition the receiver gain may be increased.



A phase sensitive GE–HMQC spectrum of a ^{15}N enriched protein of approximately 27 kDa (0.1 mM in 90% H_2O). The data were collected on an Omega 500 system equipped with the S–17 Gradient Enhanced Spectroscopy accessory using a 5 mm Inverse probe. The S–17 GES accessory is a three–axis actively shielded gradient set. This spectrum demonstrates that gradient techniques can be successfully applied to relatively large proteins in dilute solution.

GE NMR Instruments

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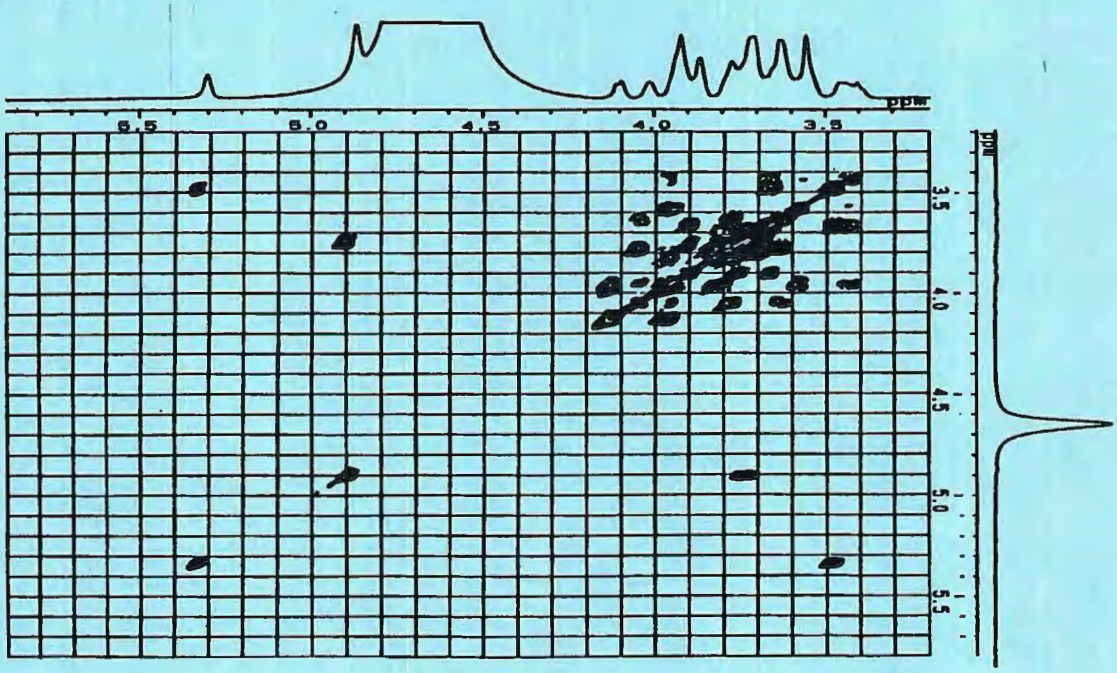
FROM JEOL

PULSED GRADIENT PROBE

Double Quantum Filtered *COSY* with NO presaturation and one scan per slice.



JEOL's gradient probe is tuned to observe ^{1}H with ^{15}N to ^{31}P decoupling and is designed to be used for reverse detection experiments. The above data were collected on an **ALPHA** 500 with the pulsed field gradient accessory. The sample is stachyose in 90% H_2O. By comparing the 1D spectrum plotted at the top with the 2D data, one can see the magnitude of the water suppression afforded by the gradient probe. It is also worthwhile to note that each slice was obtained in one scan and that the double quantum filtering was accomplished with the gradients. This 512 by 512 matrix was obtained in approximately ten minutes.

If you would like to get more information about JEOL's **ALPHA,** please contact us at one of our offices listed below.

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