TEXAS A&M UNIVERSITY

NMR NEWSLETTER

No. 432

September 1994

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D-129							
D-14							0.543 (20)
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FORTHCOMING NMR MEETINGS

Fall NMR[2] Meeting, Albuquerque, NM, **October 15, 1994**; Contact: Todd Alam, Dept. of Chemistry, Univ. of New Mexico, Albuquerque, NM 87131; (505) 277-4031 or talam@bootes.unm.edu.

Symposium on "NMR as a Structural Tool for Macromolecules: Current Status and Future Directions, Indianapolis, IN, **October 30 - November 1, 1994**; Contact: Ms. Padmini Nallana, Coordinator, NMR Symposium, Dept. of Physics, Indiana University Purdue University Indianapolis, 402 N. Blackford St., Indianapolis, IN 46202-3273; Tel. (317) 278-1263; E-mail: PADMINI@INDYVAX.IUPUI.EDU; Fax: (3172) 274-2393. See TAMU NMR Newsletter 425, 31.

36th ENC (Experimental NMR Conference), Boston, MA, **March 26 - 30, 1995**; Contact: ENC, 815 Don Gaspar, Santa Fe, NM 87501; (505) 989-4573; Fax: (505)989-1073.

Keystone Symposia on Molecular and Cellular Biology, Frontiers of NMR in Molecular Biology - IV, Keystone, Colorado, **April 3 - 9, 1995**; Organizers: S. W. Fesik, T. L. James, and G. Wagner; Contact: Keystone Symposia, Drawer 1630, Silverthorne, CO 80498; Phone: (303) 262-1230; Fax.: (303) 262-1525..

International School of Biological Magnetic Resonance, 2nd Course: Dynamics and the Problem of Recognition in Biological Macromolecules, Erice, Trapani, Sicily, Italy , **May 22 - 30, 1995**; Contact: Prof. O. Jardetzky, Stanford Magnetic Resonance Laboratory, Stanford University, Stanford, CA 94305-5055; Phone: (415)723-6270; Fax: (415) 723-2253; or, Prof. J.-L. Lefèvre, ESBS, CNRS-UPR9003, Univ. Louis Pasteur, Blvd. Sébastien Brant, F67400 Illkirch Graffenstaden, France; Phone: (+33) 88-655269; Fax.: (+33) 88-655343- See TAMU NMR Newsletter 432, 38.

Continued on page 61

Dr. Barry Shapiro
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21 July 1994 (received 7/26/94)

Dear Barry:

Magnetization transfer in samples with short T_2's.

Normally the mixing time in heteronuclear coherence transfer experiments is a function of the scalar coupling constant. For example in the simplest HMQC experiment it is set to 1/2J, but this may be different depending on the experiment. Such timing is acceptable for samples with long relaxation times relative to 1/2J. However, in samples with short relaxation times care must be taken in ensuring that the transverse magnetization does not decay within the mixing time.

Our group is involved with the assignments of the contact shifted protons in paramagnetic proteins. The contact shifted peaks have short relaxation times (<10ms) and hence broad linewidths (~100Hz). Thus for these protons, only 50% or less of the magnetization may remain at the time of data acquisition of these heteronuclear experiments, with mixing times calculated exclusively from the coupling constants.

Thus there are two important considerations when preparing these experiments. Firstly, the version of chosen experiment with the shortest possible duration should be selected, and secondly, an optimal mixing time relative to the proton relaxation time should be determined.

If one assumes that the transverse magnetization decays in an exponential manner, and that the coherence evolves according to a sine square function which depends on the coupling constant, then a simple function may be evaluated using a spread sheet to calculate the optimim mixing time from a knowledge of T_2 and J. For example with a T_2 of 100ms and a coupling constant of 160 Hz, the standard mixing time for an HMQC experiment is 1/2J=3.13ms. But for protons with a T_2 of 2ms, the optimum mixing time for maximum transfer is 1.25ms, which gives for these protons two times better magnetization transfer than for 3.13ms.

We would like to thank to Dr Wolfgang Bermel for valuable comments. Please credit this contribution to the University of Melbourne subscription (Mark Rizzacasa).

With best wishes,

Bob Brownlee
Bob Brownlee

Maruse Sadek

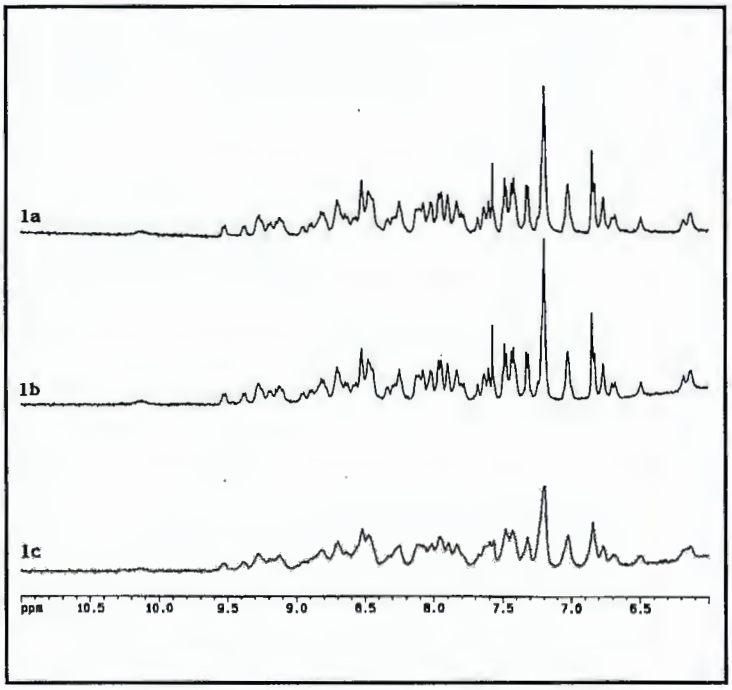
GRADIENT SHIMMING

Optimization of magnetic fields is crucial for high resolution NMR spectroscopy. So far the approach to shimming the field is pragmatic, slow and dependent on the operators skill and endurance. Now Bruker has adapted an auto shimming technique, which is currently used on diagnostic MRI systems, to facilitate the shimming of high resolution samples.

Automatic homogeneity adjustment of the x,y,z shims can be done using a three dimensional phase mapping technique as used by whole body imaging systems. For each automatic iteration, we acquired a 3D data set (64 x 32 x 32 increments) which takes ca. 10 minutes. The known shape and corrections of the actual shim system are then used together with these results to calculate an optimal shim setting for the sample. Following this, a 1D NMR experiment can be done to check the resulting magnet homogeneity.

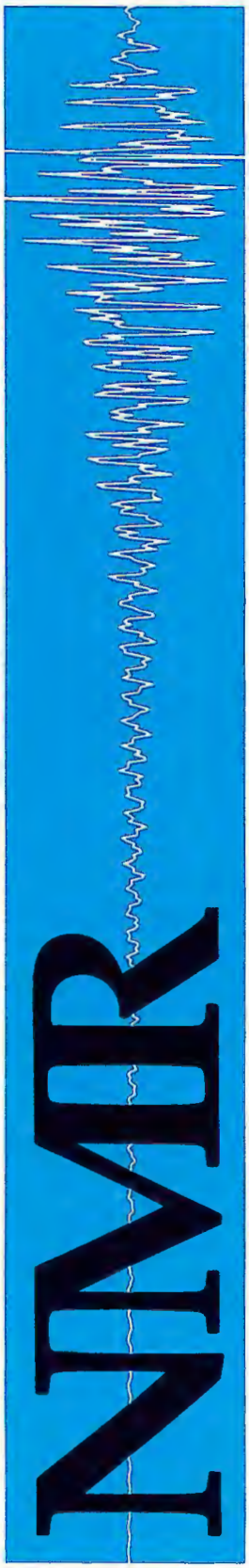
We have applied this technique at 500 MHz using the Bruker 5 mm TXI (inverse triple resonance) GRASP-III (GRAdient SPectroscopy with x,y,z-gradients) probe, together with the BOSS-II shim set (using Z1-Z6 and 12 off-axis gradients) and found that it can dramatically improve the field homogeneity and reduce the time needed to reach optimal field homogeneity.

Our results show that gradient shimming can be extremely successful, quick, and give results that are as good or better than those produced by experienced spectroscopists. As an example, the 1D proton spectrum of 2 mM ubiquitin (90% H_2O/10% D_2O), following conventional shimming is shown in Figure 1a.



We then deliberately misset the major shim values to give the poorly shimmed spectrum shown in figure 1c.

After one iteration of the automatic gradient shimming procedure, we obtained on first try, a spectrum shown in Figure 1b, that is as good as the spectrum shimmed by the spectroscopist.



We then applied the automatic gradient shimming technique to the sucrose water suppression sample (2 mM sucrose in 90%H_2O/10%D_2O, with DSS for reference). The standard Bruker parameters for presaturation of the water resonance and acquisition of the data were used following the gradient shimming. We reached a more than satisfactory result following the third iteration of the automatic gradient shimming, such that the line width of the residual water resonance after presaturation (at 50% height of the DSS signal) is <53 Hz.

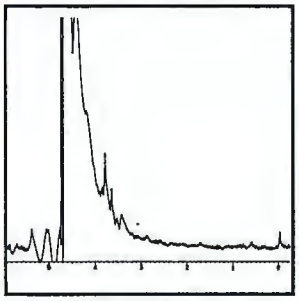


Figure 2a: TIME = 0, shims not optimized.

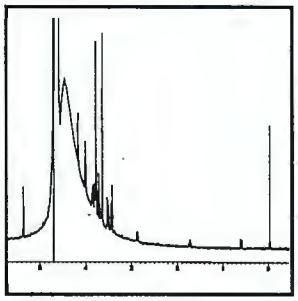


Figure 2b: FIRST iteration.

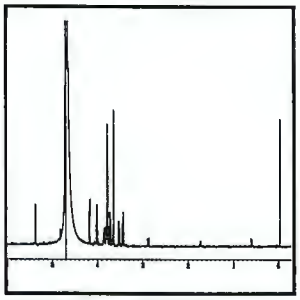


Figure 2c: THIRD iteration.

An additional example is shown for our results using a sample of BPTI (1.5 mM in 90%H_2O/10%D_2O). A 1D proton experiment (16 scans, 2 dummy scans) was acquired with a 1.3 second water presaturation. After a second iteration of the gradient shimming routine, we achieved very respectable results.

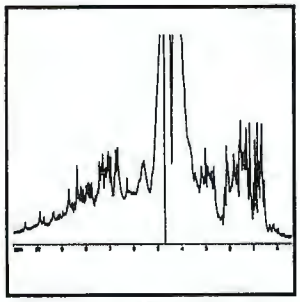


Figure 3a: TIME = 0, using starting shim values.

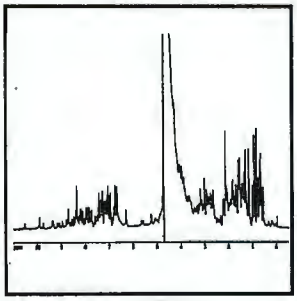


Figure 3b: FIRST iteration, with a clear improvement in the water linewidth.

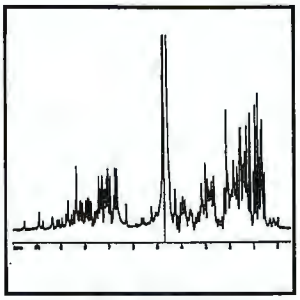


Figure 3c: SECOND and final iteration gives good resolution and linewidth.

These examples show that it is possible to perform successful gradient shimming on 5 mm samples with the Bruker GRASP-III probes within a short period of time and with excellent results. This technique offers promise for the challenges of shimming more difficult sample volumes, such as 2.5, 8.0 and 10 mm samples, or even 5 mm samples with a short filling height.

BII 3/94, P.van Zijl, M. O'Neil-Johnson, S. Sukamar, R. Hurd, P.Webb

S.a.d.i.s.
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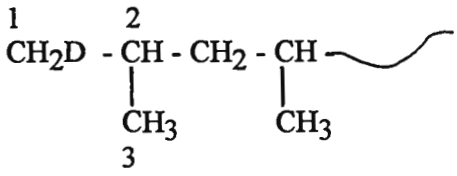
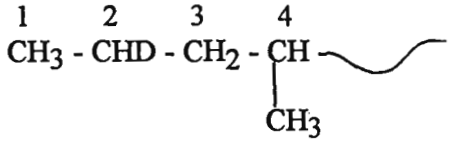
Wissembourg, July 23rd 1994
(received 8/1/94)

Dear Barry,

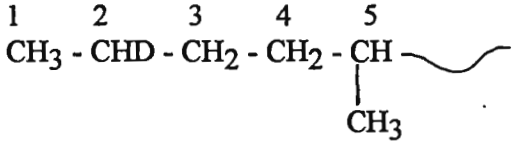
End-Chain structure in polypropylene after deuterium-transfer reaction

Modern supported Ziegler-Natta catalysts produce polypropylene with high regioselectivity and high stereospecificity. The control of molecular weight is obtained by the use of hydrogen which is able to perform the hydrogenolysis of the active metal-carbon bond. Substituting deuterium for hydrogen allows the labelling of the chain ends. The two expected chain ends for a normal insertion are :

(1)  isobutyl type (A)

(2)  n-propyl type (B)

A regioregular insertion forbids propagation. If it is followed by transfer it is expected to produce the following chain end :

 n-butyl type (C)

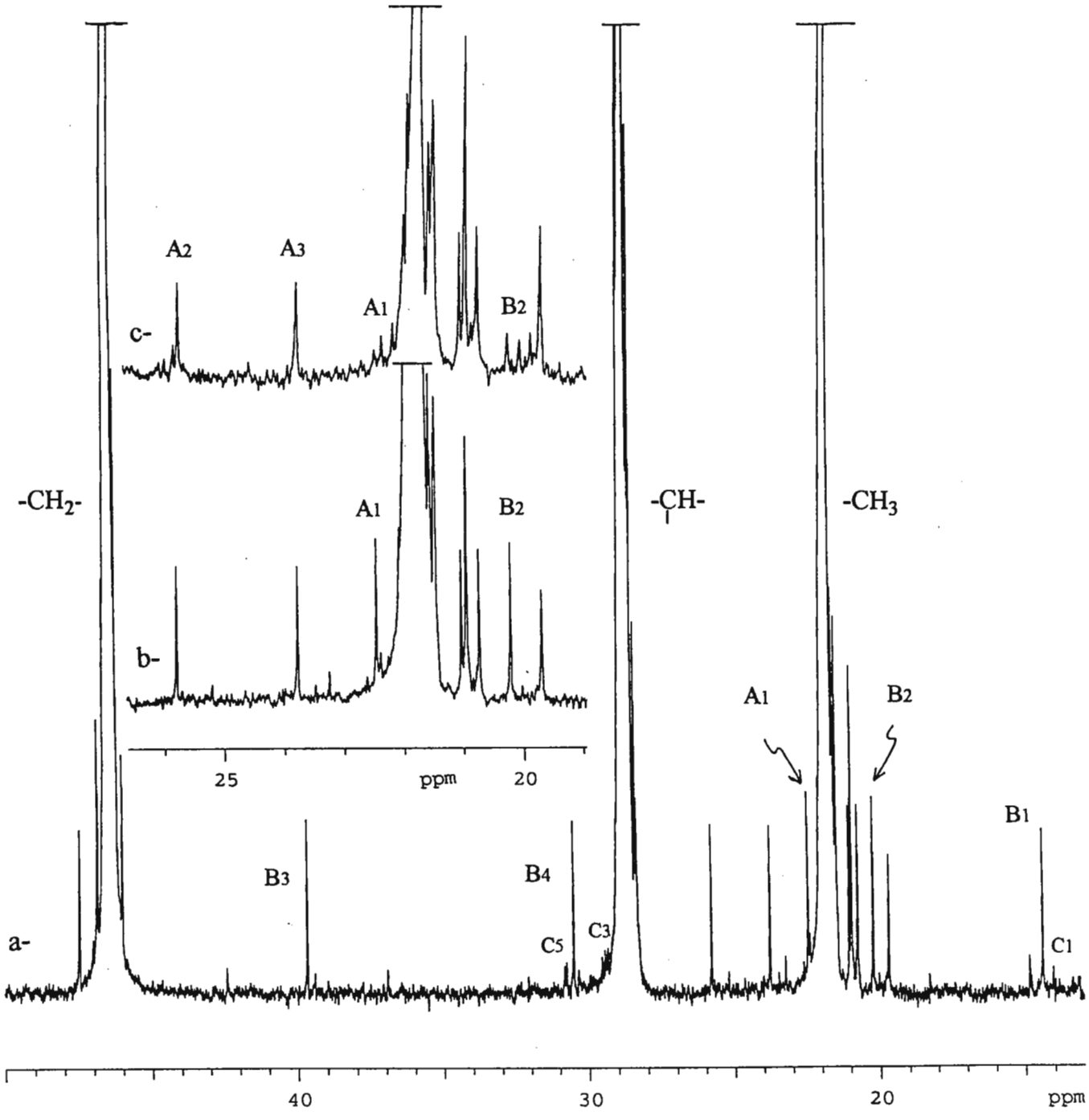
These three end groups are conclusively identified by two ^{13}C NMR spectra obtained at 360K, on a BRUKER AVANCE DRX 400 (100.6 MHz) equipped with a VSP 10 mm probehead, respectively with hydrogen (sample one) and deuterium as transfer agent (sample two).
Shown below are the spectra obtained in 500 scans : a- and b- : sample one, with digital filtering of the solvent mixture Tetrachloroethylene / C6D6 2/1 by volume, c) : sample two, with digital filtering of solvent and methylenic carbon and presaturation of methyne and methylic carbons.
On sample one (see trace a-), all carbons of isobutyl and n-propyl type end groups are clearly identified (A and B) whereas n-butyl end groups are also visible in agreement with preceding studies (T. Tsutsui, N, Kashiwa, A.Mizuno, *Makromol. Chem., rapid. Commun.*, 11, 565,(1990) and J.C. Chadwich, A. Miedma, O. Sudmeijer, *Makromol.Chem.Phys.*, 195, 167, (1994)). But the proportion of the irregular chain ends seems to be very small with the catalytic system used in our study. On sample two (see enlargment c-) compared with b-), the

deuterated carbons of isobutyl and n-propyl type end groups, A1 and B2, are clearly identified which confirms the transfer mechanism.

Sincerely,

M. BOURDONNEAU, M.F. LLAURO*, R.SPITZ** , C. BREVARD

*Laboratoire des Matériaux Organiques à Propriétés Spécifiques / C.N.R.S. B.P 24 69390 Vernaison FRANCE
**Laboratoire de Chimie et Procédés de Polymérisation / C.N.R.S. B.P.24 69390 Vernaison FRANCE



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

(received 8/4/94)
August 1,1994

Subject: **^{25}Mg NMR Relaxation Study of Mg^{2+}-ATP Complexation in Solutions.**

Dear Dr. Shapiro:

In the present study ^{25}Mg NMR relaxations was used directly in order to glean some insight into the nature of chemical exchange in Mg^{2+}-ATP system. It is well known that the ^{25}Mg chemical shift is very insensitive to the formation of complexes[1]. Even at low temperature the chemical shift separation between free Mg^{2+} ion and Mg^{2+}-ATP can not be resolved spectroscopically, but a methed based on linear prediction with singular value decomposition and the minimum Euclidean norm condition has previously been developed in our laboratory in attempts to trace the various modes of frequency correlation in the free induction decay[2]. It has been verified the usefulness of this method by applying it to resolve the chemical shifts of ^{25}Mg on binding processes. Subsequently, after taking advantages of the resolved chemical shift separation as an input to fit the spectra obtained near the null point in inversion recovery experiment, a successive approximation was applied to find the chemical shift separation in the slow exchange limit. Here the complete description of time evolution for longitudinal and transverse relaxations with chemical exchange by density matrix method were invoked in the simulation of the spectra observed in inversion recovery experiments[3]. ^{25}Mg longitudinal and transverse relaxation data in conjunction with the spectral lines obtained near the null point in inversion recovery experiment were used to determine the motional correlation time, the effective quadrupolar coupling constant, and the dissociation rate constant of the Mg^{2+}-ATP complex at various temperature between 270 and 320 K. This study shows that no aggregation of complex has been found with increase of ATP concentration and the complexation process at the temperature range studied is in the moderate exchange regime[4]. With best regards.

Yours Sincerely,

Lian-Pin Hwang Zhi-Shian Shen

References:
1.Neurohr, K. J.; Drakenberg, T.; Forsén, S. in "NMR of Newly Accessible Nuclei"(Laszlo, P. ed.), Academic Press, New York, 1983,2,229.
2.Lin, Y. Y.; Ge, N. H.; Hwang, L. P. *J. Magn. Reson.* 1994,*103*,1.
3.Price, W. S.; Ge, N. H.; Hong, L. Z.; Hwang, L. P. *J. Am. Chem. Soc.* 1993,*115*,1095.
4.Glonek, T. *International Journal of Biochemistry* 1992, *24*, 1533, and pertinent references therein.

Battelle
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August 5, 1994 (received 8/22/94)

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

Paul - I've moved:
seven years ago.
Barry

Re: 750 Installation at Battelle's Pacific Northwest Labs (PNL)

Dear Barry:

Just a short note to summarize our experiences with our new Varian 750. However, before I start I want to mention the efforts of Oxford and Varian to bring this first system up. They were simply outstanding. Oxford "baby sat" the system from England to PNL. The Oxford installation engineers did an excellent job going over every possible detail associated with the delivery to the laboratory and the subsequent installation. There were many late evening and early morning stays at the lab to get the job done and done well. I know I was part of the escort crew that had to stay with them during the installation. The magnet was brought to field in mid December and then it stabilized over the Christmas holidays. The Varian engineers wasted no time in bringing the rest of the system up to speed. The magnet made line shape & sensitivity specifications by mid January and we had obtained our first DNA data in the same time frame. I have never been associated with a first of its kind effort that went so well! We have enjoyed nearly flawless operation since that time.

We have had more difficulties bringing our chemistry up to speed than we have had with the spectrometer. However, those days are behind us at the present. We are looking at damaged unsymmetrical DNAs and looking forward to examining their complexes with proteins. Likewise, we are enjoying our collaborations with Gary Drobny at the University of Washington via some DNA binding proteins and some interesting metal binding template proteins. Virtually all of the high resolution work is being done by either Mike Kennedy or in collaboration with Mike. However, we have taken the opportunity to examine some solid-state MAS and static experiments on the 750 as well. I look forward to communicating the results of some of these projects to you in the future.

I hope you are maintaining your golf expertise and I look forward to a trip to England later this year to explore any recent efforts by one of our favorite authors.

Sincerely,

Paul Ellis/gw

Paul D. Ellis
Associate Director
Macromolecular Structure & Dynamics

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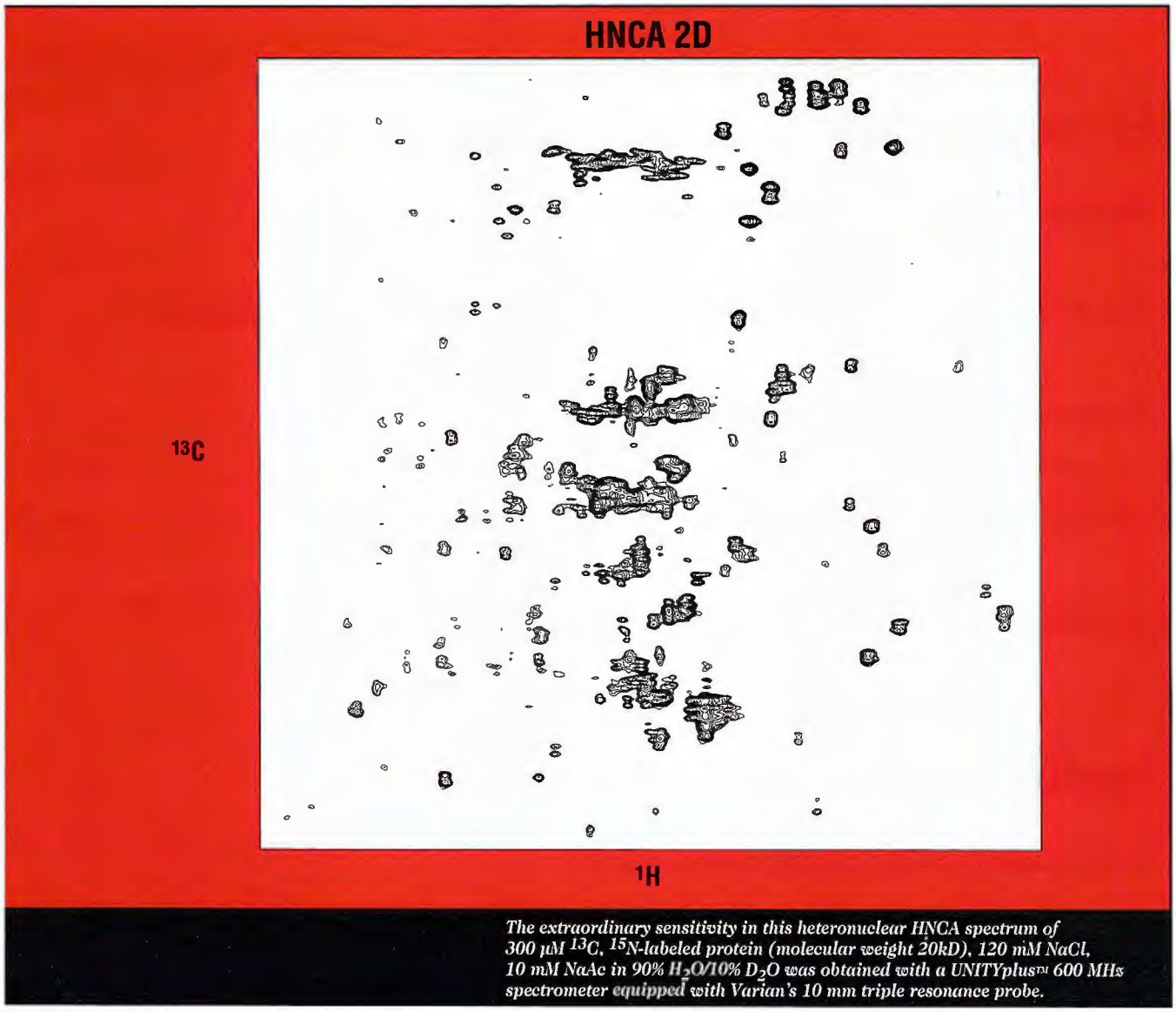

The extraordinary sensitivity in this heteronuclear HNCA spectrum of 300 µM ^{13}C, ^{15}N-labeled protein (molecular weight 20kD), 120 mM NaCl, 10 mM NaAc in 90% H_2O/10% D_2O was obtained with a UNITYplus™ 600 MHz spectrometer equipped with Varian's 10 mm triple resonance probe.

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THE CITY UNIVERSITY OF NEW YORK

July 25, 1994 (received 7/29/94)

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

^{2}H NMR of Damp Tomato Cuticle

Dear Barry:

Although the City University of New York appears to be setting a world record for the delay between a spectrometer purchasing decision and the issuance of the requisite Purchase Orders, we have managed so far to install our Unity*plus* 500 (at Hunter College) and Unity*plus* 300WB (at The College of Staten Island). Our initial experience with the latter 3-channel instrument, which is equipped for full solids operation as well as with a 10-mm broadband liquids probe, is the subject of this contribution.

In conjunction with our efforts to understand the protective functions of the fruit cuticle, we used chambers of controlled relative humidity to swell tomato skins with D_2O. The impact of hydration on the cuticular polymers has been examined with CPMAS ^{13}C NMR ($T_{1\rho}(H)$, $T_1(H)$, ($T_{1\rho}(C)$, $T_1(C)$, high-power-decoupled CPMAS vs. low-power-decoupled DPMAS). In a complementary fashion, the state of cuticle-associated water has been probed with ^{2}H NMR experiments.

A damp cuticle sample containing 1%-by-weight water yielded the quadrupole echo data shown in **Figure 1**, obtained with automated temperature control and acquisition over a period of 12 hours. A narrow central resonance is observed in each spectrum, albeit 200-1000 times broader than bulk D_2O. In addition a broader feature is present; its lineshape indicates some motional averaging even at -10 °C. As the temperature is lowered through the range of plausible growth conditions for tomatoes, citrus fruit, etc., we observe an increasing spectral contribution from the semirigid population of deuterons.

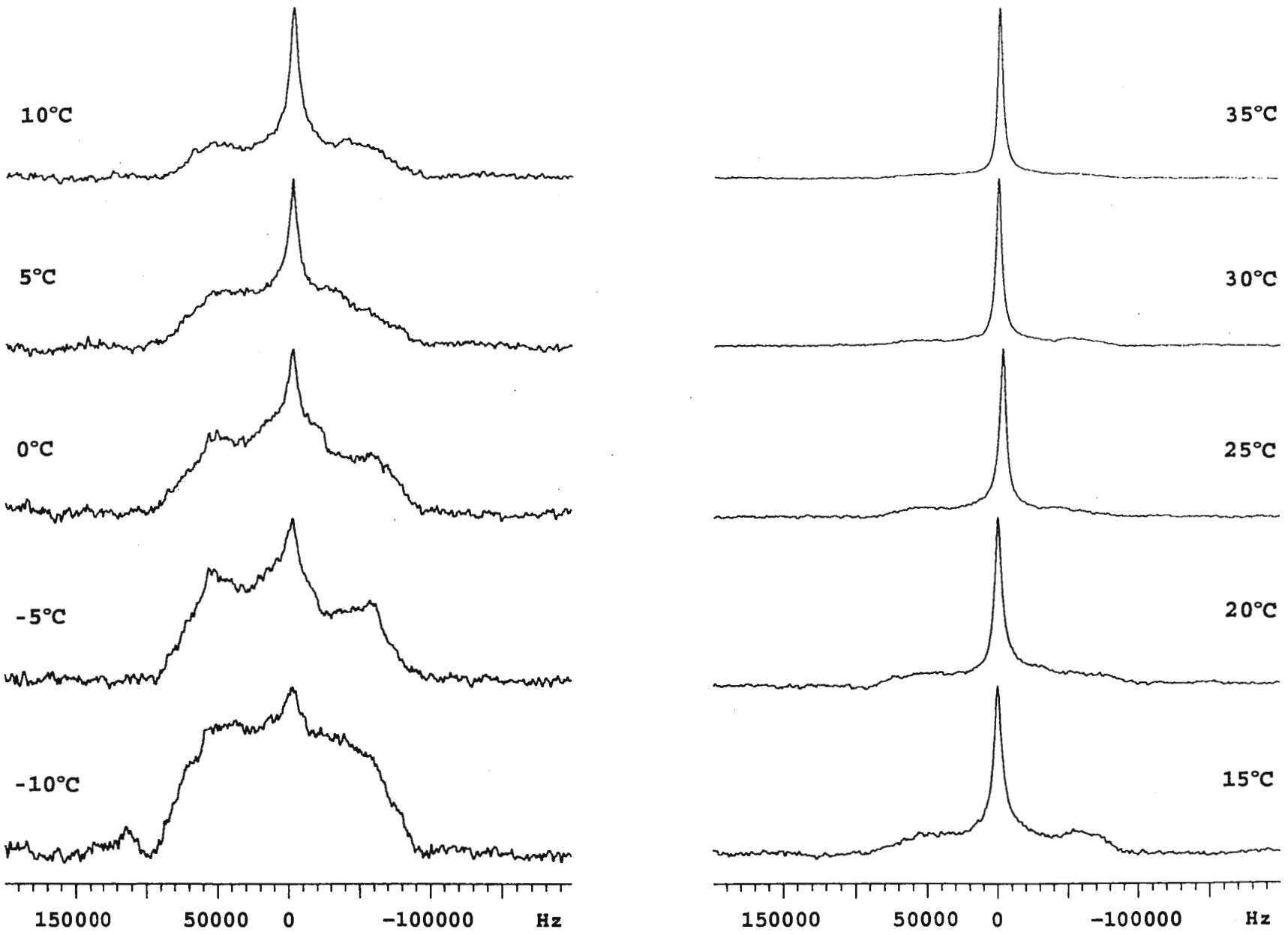
^{2}H NMR has been used to study analogous hydration phenomena in epoxies[1] and fluoropolymer membranes.[2] The narrow peak is attributable to mobile but anisotropically tumbling water absorbed within the tomato cuticle, but what is the broader pattern? It could be a more immobilized population of water in slow exchange, a hypothesis suggested by the way in which the relative intensities change with temperature. The broad pattern could also arise from OD groups produced in the cutin polymer, so that it will be retained in the ^{2}H spectrum when we remove the water. A fuller report will follow when the experiments and analysis are complete...

Very truly yours,

Ruth Stark	Zhen-jia Chen	Frank Corrigan	Leon Van Gorkom
Professor	Ph.D. Student	Undergrad Fellow	Facility Manager

References: 1. L.W. Jelinski, J.J. Dumais, R.E. Stark, T.S. Ellis, & F.E. Karasz (1983) *Macromolecules* 16, 1019; L.W. Jelinski, J.J. Dumais, A.L. Cholli, T.S. Ellis, & F.E. Karasz (1985) *Macromolecules* 18, 1091. 2. R.S. Chen, J.P. Jayakady, S.G. Greenbaum, Y.S. Pak, G. Chu, M.G. McLin, & J.J. Fontanella (1993) *J. Electrochem. Soc.* 140, 889; M. Rankothge, Haryadi, G. Moran, J. Hook, & L. Van Gorkom (1994) *Solid State Ionics* 67, 241

Figure 1. 46 MHz ^2H quadrupole echo spectra of tomato cuticle containing 0.7 mg D_2O. 5-mm Varian wideline probe, P(π/2)=2.4 μs, SW=2 MHz, RD=1 s, plots normalized to the tallest peak.



THE UNIVERSITY OF SYDNEY
DEPARTMENT OF BIOCHEMISTRY
SYDNEY N.S.W. 2006
AUSTRALIA

Dr Glenn F. King
E-mail: gfking@extro.ucc.su.oz.au

TELEPHONE: [61-2]-692-3902
FACSIMILE: [61-2]-692-4726

19th July, 1994
(received 7/28/94)

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

Dear Dr Shapiro,

We were most interested in the report by Rosanna Mondelli in the April 1994 edition of the TAMU NMR Newsletter that her group has used NMR spectroscopy to determine the tertiary solution structure of endothelin-1. However, we were surprised by their claim that "Up to now, the reported structural results have been obtained only from experimental data in DMSO or in media containing organic solvents...". They may not have seen our 1991 report of the structure of the related sarafotoxin-b[1] or our 1992 report of the structure of endothelin-3[2], both determined from NMR data using peptide dissolved in pure water. It could be reasonably argued, however, that since the endothelins are vasoactive peptides, the structures should be derived from peptide dissolved in saline solution, and we have taken this approach in our recent determination of the solution structure of sarafotoxin-c[3].

Mondelli's structure of endothelin-1 is similar to that of endothelin-3 which we published in 1992[2], although there are some notable differences. The overall structure of endothelin-3 (see Figure 1 overleaf), moving from the N-terminus to the C-terminus, consists of an N-terminal extended (non-beta) strand, a relatively well-defined loop, and a stretch of helix followed by a bend which causes the C-terminal residues to lie in close apposition to this central helical domain. The central helix in the endothelin-3 structure is irregular and shorter (residues 9–15) than the α-helix (residues 9–16) reported for the endothelin-1 structure, while the C-terminal segment in endothelin-1 folds back further towards the N-terminal end of the central helix. While the proximity of the Ile 19 γ-methyl group to the Trp 21 indole ring in the endothelin-3 solution structure[3] satisfactorily explains the low frequency NMR shift observed for the Ile/Val 19 γ-methyl protons in all endothelin/sarafotoxin isoforms, it is unclear at this stage as to whether this rationalisation can also be made from Mondelli's endothelin-1 structure.

While it is satisfying that the NMR-derived aqueous solution structures of endothelin-1 and endothelin-3 are rather similar, the recently published crystal structure of endothelin-1[4] is markedly different. It therefore seems that the biologically active conformation of endothelin will remain enigmatic until the structure of peptide bound to its receptor is determined.

Yours sincerely

Glenn F. King

Glenn F. King

R G Mills

Robyn G. Mills

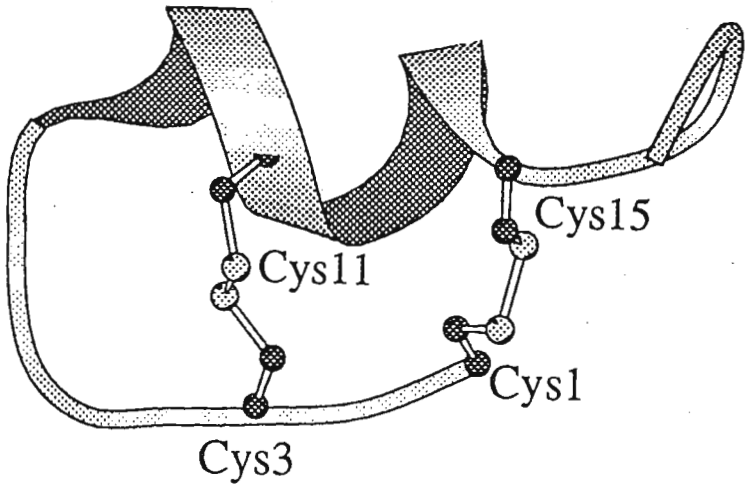


Figure 1: Schematic representation of the NMR-derived solution structure of endothelin-3 generated using the program MOLSCRIPT[5].

References

1. Mills, R.G., Atkins, A.R., Harvey, T., Junius, F.K., Smith, R., and King, G.F. (1991) *FEBS Lett.* **282**, 247–252.
2. Mills, R.G., O'Donoghue, S.I., Smith, R., and King, G.F. (1992) *Biochemistry* **31**, 5640–5645.
3. Mills, R.G., Ralston, G.B., and King, G.F. (1994) *J. Biol. Chem.*, in press.
4. Janes, R.W., Peapus, D.H., and Wallace, B.A. (1994) *Nature Struct. Biol. 1*, 311–319.
5. Kraulis, P.J. (1991) *J. Appl. Cryst. 24*, 946–950

P.S. Please credit this contribution to the account of Prof. Philip W. Kuchel.

POSTDOCTORAL POSITION. Available immediately to study molecular structure and development of plant cuticle polyesters. Requires Ph.D. in chemistry or biochemistry, with experience in one or more of the following areas: analytical method development (MS, HPLC, NMR); biosynthesis, biodegradation, and isolation of plant materials. Send a curriculum vitae, two letters of reference, and copies of pertinent publications to: **Prof. Ruth E. Stark, Department of Chemistry, City University of New York, The College of Staten Island, 2800 Victory Boulevard, Staten Island, NY 10314.** EO/AA/ADA Employer.

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DEPARTMENT OF CHEMISTRY

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

July 26, 1994
(received 8/13/94)

Dear Professor Shapiro,

It is our pleasure to return to the group of contributors to the TAMU Newsletter, after significant changes and restructuring in our laboratory. As you may know, George Levy has left the lab, so our direction will now be much more toward NMR applications in biopolymer structure and dynamics, and less in the data processing area. Another part of the changes was an upgrade our aging GN-500 with a Tecmag system in order to gain higher flexibility and better reliability for multidimensional experiments. The software is quite flexible for a variety of data acquisition protocols, and has been a reasonable investment. However, it is no secret that the current version of this software is deficient for 3D and 4D (etc.) data acquisition. We have overcome some of the limitations, and can now run phase sensitive multidimensional experiments. This includes any combination of sequential and simultaneous acquisition schemes using phase cycling for sign discrimination. (Thanks are due to Irene Nooren, who is presently at Utrecht University, Netherlands, for many preliminary tests and experimentation.) A 2D experiment with TPPI incrementation for the remote (t_1) dimension is most straightforward, for it is directly supported by the Tecmag design. A complex incrementation (States-Haberkorn-Ruben method (SHR)) with the usual axial peak suppression requires two-block acquisition due to lack of independent incrementation of individual tables in the current version of the software. The States-TPPI method, which avoids extra phase cycling steps for axial peak suppression while retaining advantages of the complex (simultaneous) data structure, requires a four-block implementation. We have found, however, the States-Redfield combination to be the simplest to use *(1)*. This approach retains - and extends somewhat - the advantages of the former method, but requires no extensive changes in the pulse sequence. Only a one-block acquisition will do the job - with fake "TPPI" incrementation on the appropriate pulses. The only special requirement is that every time increment must be used twice in consecutive cycles relative to that of a SHR incrementation, doubling the table size for time delays. The situation is a bit more difficult in the case of 3D (nD) acquisitions. Theoretically the scheme mentioned above can be extended directly to more than 2D, but with the current software version difficulties occur. We now use a hard wired acquisition method, limited by the buffer size for pulse sequence compilation. For example, we present a 3D-NOE/NOE spectrum of a bulged 26-mer DNA acquired on our system. (Detailed description of the experimental conditions and technical implementation is available on request from C.S. and I.P.). The sample was prepared in Prof. Neocles Leontis' laboratory (Bowling Green University, Bowling Green, OH). Various spectra of this sample have been analyzed by Mike Szafranski in our lab, and structure refinement is in progress.

Traditionally, our laboratory has used off-line data processing, and this is the case for all applications mentioned above. It is particularly necessary because the Tecmag software cannot now handle multi-block data, especially those with a complex acquisition scheme. At the time we are using NMRZ and Triad (Tripos Associates, Inc.) for this purpose. Appropriate file conversion from Tecmag format to *z-ants* (a very simple data structure *(2)*) is done by a home written routine *(3)*, which is available from the authors.

Sincerely,

István Pelczer Chris Spronk Deborah Kerwood Philip N. Borer

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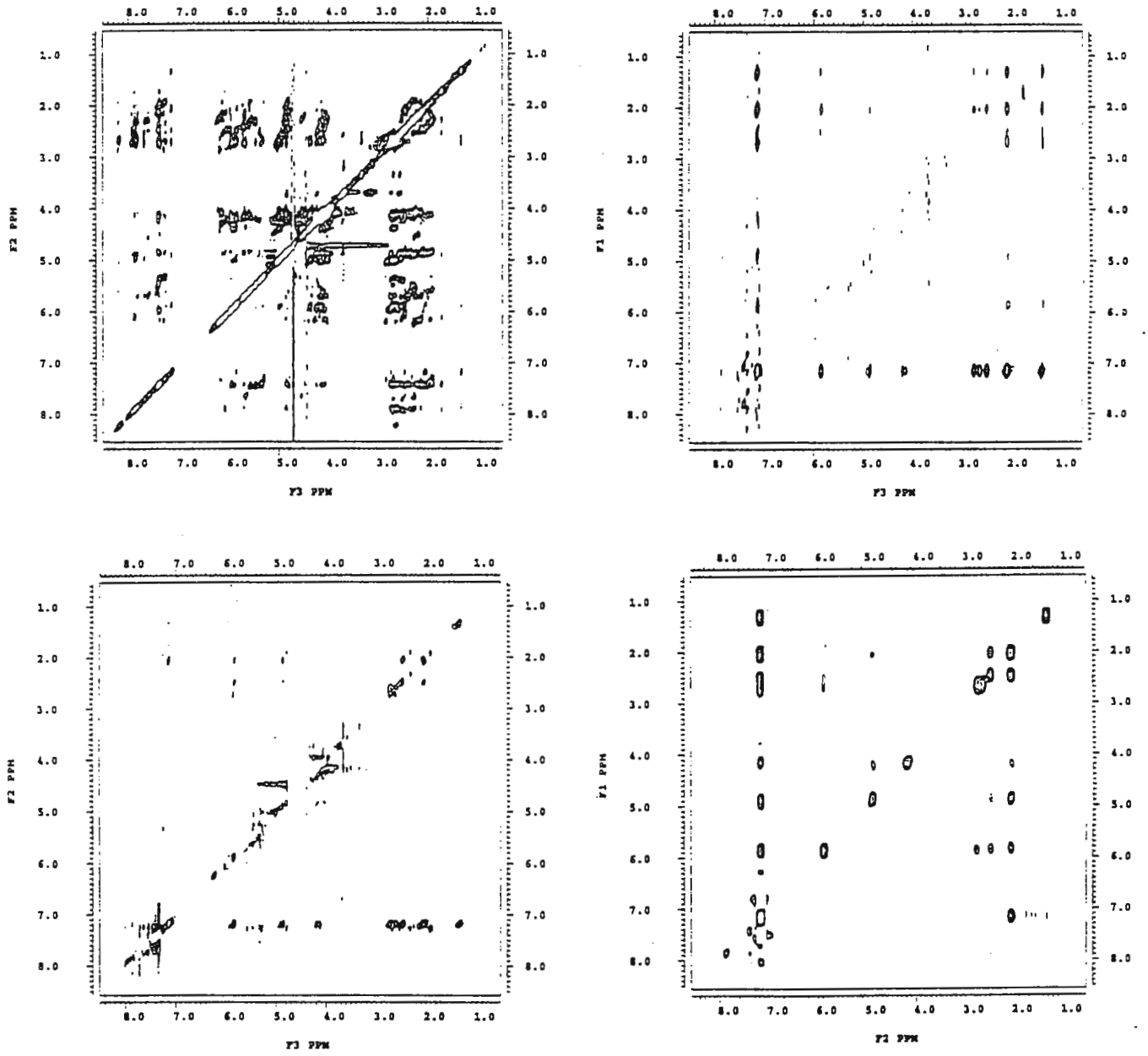

Figure 1. Selected 2D planes from the 3D-NOE/NOE spectrum of the double stranded bulged DNA: d(5'-GGCAGAAGTGCCGC-3')-d(3'-CCGTC--CACGGCG-5'). *(upper left)* First (F3,F2,t1) plane, *(upper right)* F3/F1 plane, *(lower left)* F3/F2 plane, *(lower right)* F2/F1 plane, each of the last three cut at the frequency of the T9,H6 proton (7.15 ppm).

‡ Chris Spronk is on a short leave from Laboratory of Biophysical Chemistry, University of Nijmegen, Toernooiveld 1, 6525 ED Nijmegen, The Netherlands

(1) Bishop, K. D., Pelczer, I.* and James, T. L.; "States-Redfield Phase Incrementation: An Alternative Acquisition Scheme in Multidimensional NMR" 34th ENC, St.Louis, MO, USA, March 14-18, 1993, abstracts: P 53

(2) Pelczer, I., Hoch, J. C., Roggenbuck, M. W., Vaidyanathan, A., Leccarde, M. G. and Borer, P. N.; "Z-ANT Processing; A New Alternative for Multidimensional NMR Data Processing" 33rd ENC, Pacific Grove, CA, USA, March 29 - April 2, 1992, abstracts: WP 188

(3) Huitema, E. and Pelczer, I.: "Conversion software 'tec2nmri' and 'catzants' from Tecmag format to NMRi (*z-ant*) format" Chemistry Department, Syracuse University, 1993

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INSTITUTE FOR CLINICAL AND EXPERIMENTAL MEDICINE
DEPARTMENT OF DIAGNOSTIC AND INTERVENTIONAL RADIOLOGY
HEAD: JAN PEREGRIN, MD, PhD

July 25, 1994
(received 8/15/94)

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

FURTHER STUDY OF ADIABATIC PULSES

Dear Dr. Shapiro,

After first experiences with new adiabatic pulses (created by new pulse generator on IBM/PC, which extends feasibilities of our system - see TAMU No. 420) we paid attention to the detailed study and optimization of these pulses.

For testing we were using at first phosphorus fantoms. The different concentration of Na^+, H^+, PO_4^{3-} determined the pH and consequently chemical shift of the samples. After these preliminary studies we verified our results in vivo on the calf muscle of the volunteers.

Although the adiabatic pulses are obviously used for the better localization in MR spectroscopy, we found them very useful also for simple excitation. We tested the influence of the transmitter voltage and relative value of the frequency modulation of the pulse on the resulting spectra. Great dependence on the value of frequency modulation was observed. The behavior of the adiabatic pulse with a low frequency modulation is similar to the common amplitude modulated pulse. Increasing frequency modulation brings about increasing of the signal-to-noise ratio and broadening of the interval of the transmitter voltage (which is in absolute value much more higher than in case of AM pulses), in which the pulse works. Using surface coils, which radiation is dependent on the distance from the coil, this enables more homogeneous excitation also in the deeper layers of the measured sample. Further increasing of the frequency modulation causes then lowering of the S/N ratio again, which signalized, that the magnetization vector is twice turned about greater angle than is necessary (although the resulting excitation angle is still 90 degrees). Optimized adiabatic pulse brings about 25% - 30% increasing of the S/N ratio comparing with AM pulses. This result was verified also in vivo.

This basic study will help us now optimize and improve the method of 31P spectroscopy of the calf muscle during the exercise. Higher S/N ratio increases sensitivity of the measurement and enables shortening of the time of examination, which is for the spectroscopy of the muscles during exercise very important.

Yours sincerely,

Milan Hájek Vít Herynek

MR Unit, RADP

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42 2 47 28 229

DEPARTMENT OF THE NAVY
NAVAL RESEARCH LABORATORY
WASHINGTON D C 20375-5320

IN REPLY REFER TO:

19 July 1994

Postdoctoral Positions

There are potential postdoctoral opportunities at the Naval Research Laboratory in Washington DC. *Please note the relevant deadlines: now is the time to apply for positions commencing in spring of 1995.* Interests span a number of areas in magnetic resonance.

- We seek individuals with interests in novel applications of pure nuclear quadrupole resonance: NQR imaging and NQR stochastic excitation/detection are possibilities. NQR experience is desirable but not necessary.

- At NRL we have been developing solid state NMR imaging techniques for the analysis of materials. We are particularly interested in proposals that apply advanced solid state NMR imaging techniques to the study of materials: polymers, composites, or ceramics.

- We are also interested in proposals dealing with solid state NMR studies of polymer structure and dynamics. We have an active collaboration with Dr. Mike Roland exploring polymer miscibility, extended chain crystallinity, and the use of xenon as a probe molecule dissolved in polymers.

- We have an effort underway to use optically-pumped spin-exchanged xenon to obtain NMR spectra from diamond surfaces with enhanced selectivity and sensitivity. A postdoctoral position may become available in this area in the fall of 1995.

There are two postdoctoral programs at NRL: the National Research Council (NRC) associateship and the ASEE/NRL postdoctoral fellowship, administered through the American Society for Engineering Education (ASEE). Each program selects applicants on a competitive basis. Relocation and professional travel allowances are provided. *An immutable requirement is that the applicant be a U.S. citizen.*

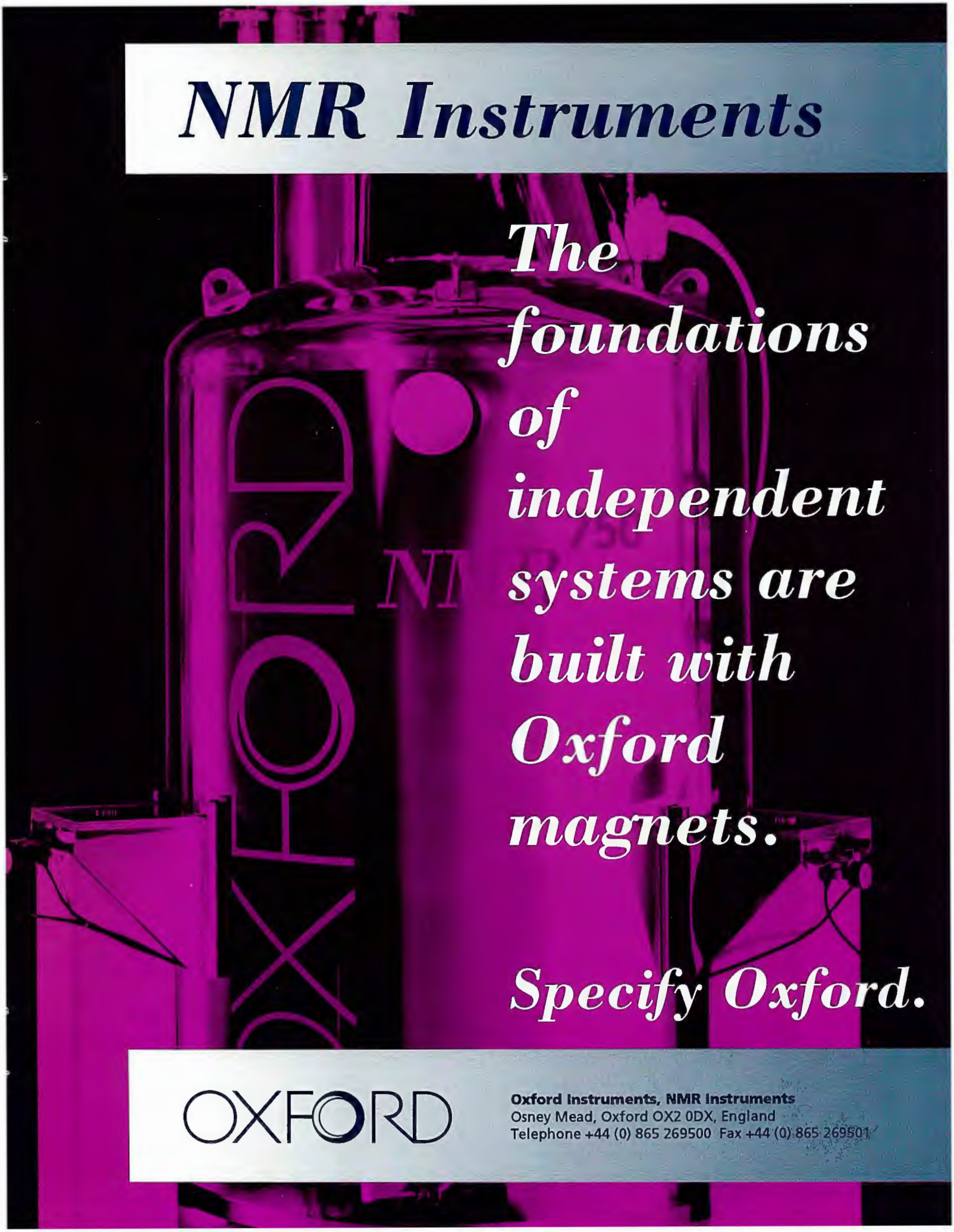
The NRC appointment is for two years, with the possibility of a third year. Contract support from the Office of Naval Research may be available for NRC associates who continue post-tenure research at an academic institution. ASEE/NRL appointment is for one year, renewable for a second and possibly third year. Stipends for both programs presently start at $36K.

For the NRC program, there are two selection cycles yearly; the application deadlines are *15 January and 15 April 1995*. There are four ASEE/NRL award cycles with deadlines of *1 October 1994,and 1 January, 1 April, and 1 July 1995.*

Please encourage anyone interested to contact one of us directly and informally.

Joel Miller	(202) 767-2337	MILLER5@NRLFS1.NRL.NAVY.MIL
Al Garroway	-2323	GARROWAY@NRLFS1.NRL.NAVY.MIL
James Yesinowski	-0415	YESINOWS@NRLFS1.NRL.NAVY.MIL

NMR Instruments



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

(received 7/27/94) July 18, 1994

Three-Axis and 10mm Pulsed Field Gradients

Dear Barry,

We have recently carried out experiments on a UNITY*plus* 600 using a 10 mm triple-resonance probe equipped with Actively-shielded Pulsed Field Gradient capability. In addition, we have developed a three-axis PFG 5mm probe that we have tested at 500 MHz. We are sure that there is considerable interest in the performance of these and would like, therefore, to report the current state of progress.

Figure 1 shows a 10mm 15N-pfg-HSQC on a very dilute 350uM solution of an enriched protein in H2O. Each of the 256 complex t1 increments had 128 transients. Water suppression was excellent, permitting the accurate observation of all amide protons.

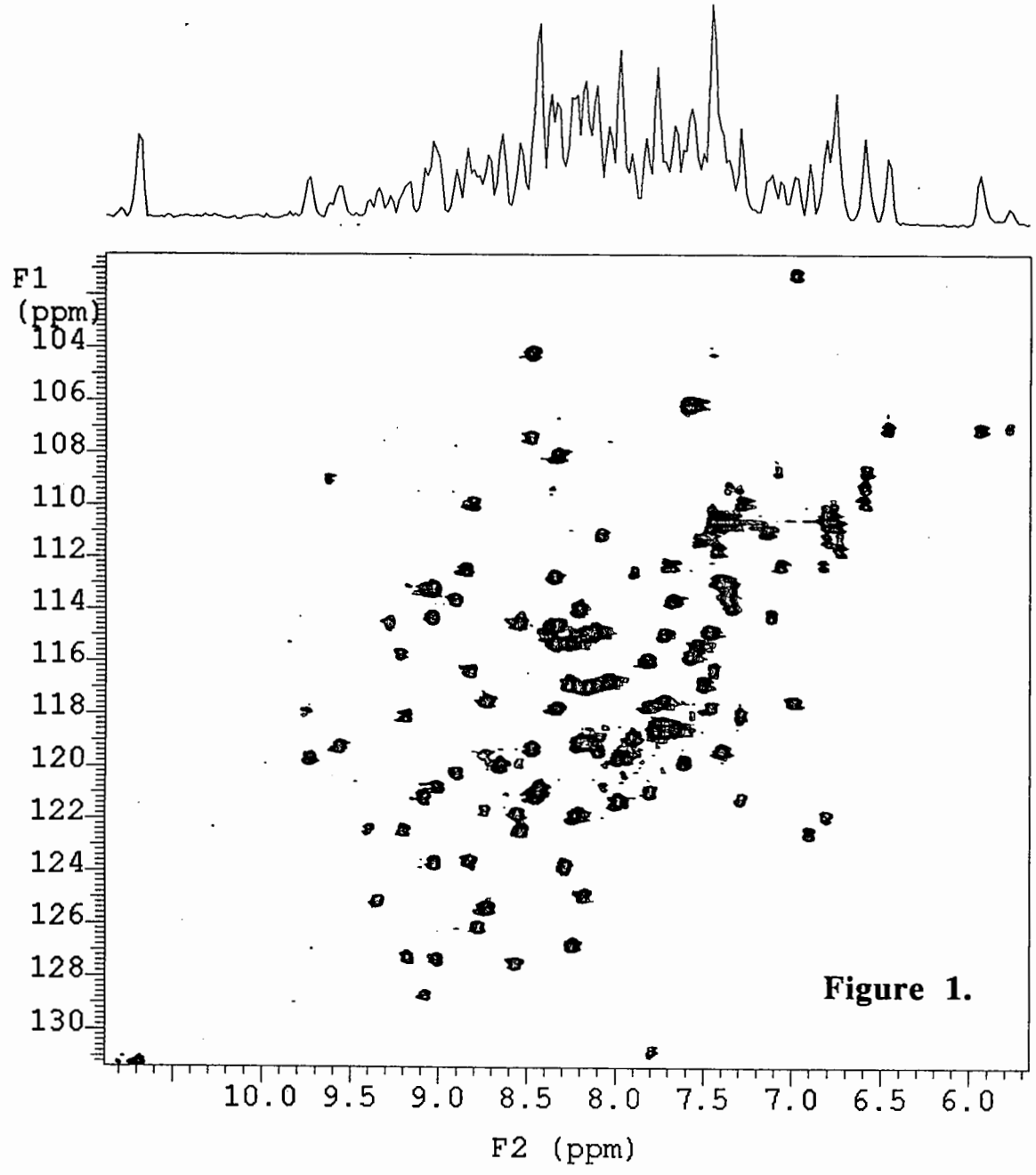


Figure 1.

varian associates / 3120 hansen way, box d-298 / palo alto, ca 94304-1030

The UNITY*plus* console was equipped with our standard low-noise transconductance current amplifier(s). No pre-emphasis was necessary for any of the experiments, in fact no-preemphasis has been needed in any of our PFG experiments to date. The 10mm probe benefited from the higher filling factor in observed s/n. We obtained the expected increases of 2.2x in s/n relative to 5mm performance. For the most demanding experiments on dilute biomolecules the 25% increase in s/n relative to the same solution in an 8mm probe is quite attractive, substantially reducing the time required for attaining a given s/n. Since in many cases one is not sample amount limited but concentration limited, going from 8mm to 10mm does have considerable benefit. Of course, this places greater demands on the probe , magnet and shimming technology.

Figure 2 shows the substantial effect on residual water in a gradient DQCOSY t1 increment. Here, the gradient angle is varied from along z in the spectra at the left, to along y in the spectra at the right. Ten trials were performed at each angle and are plotted side-by-side to assess the reproducibility of the water suppression. A clear minimum at 54 degrees is noted, the value of the magic angle. This increased suppression is obtained using only two of the gradients available in this 5mm triple-resonance triple-axis PFG probe. This probe had the same X RF performance as observed with the z-axis triple-resonance PFG probe.No degradation of proton 90 degree pulse or sensitivity was observed.

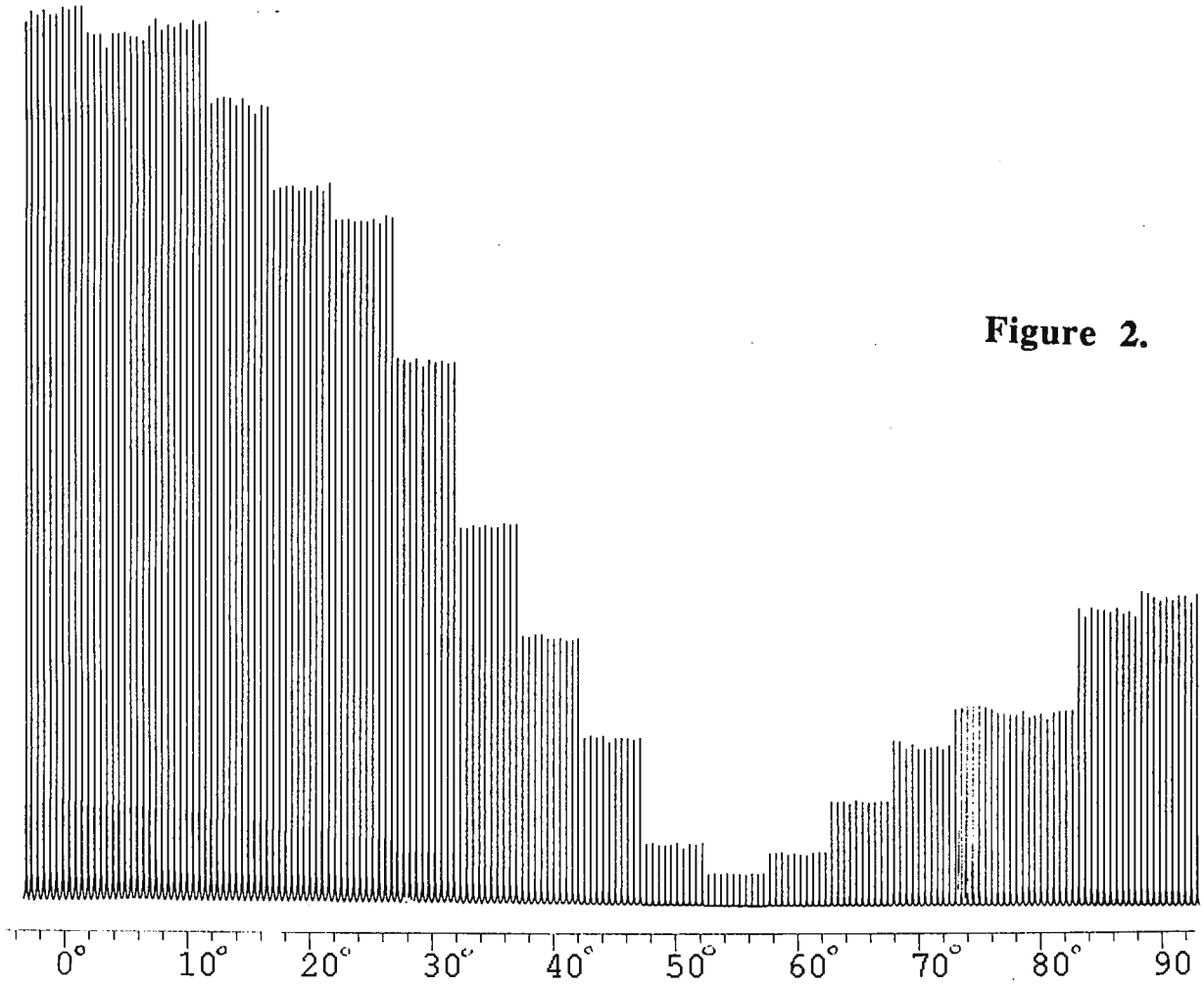
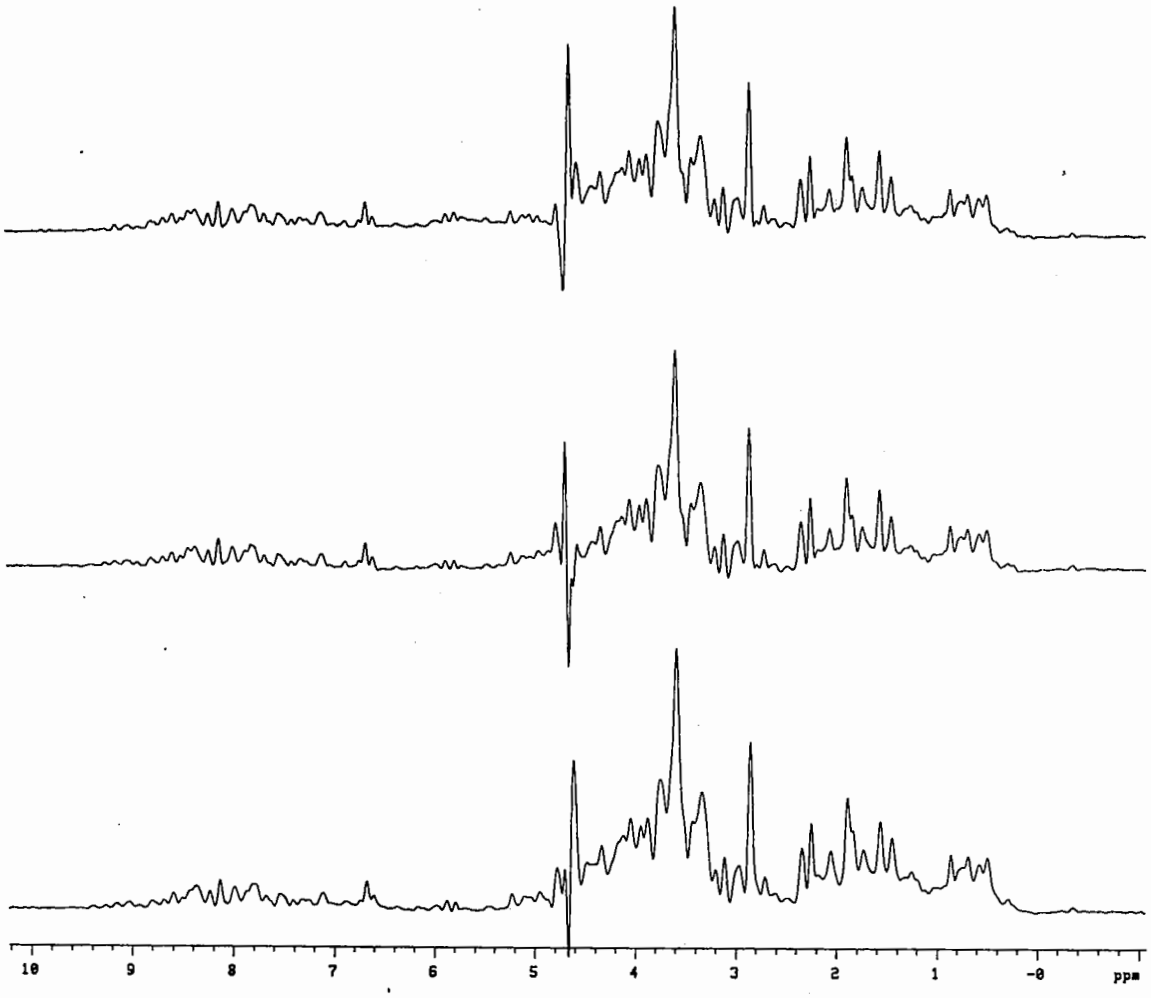


Figure 2.

varian associates / 3120 hansen way, box d-298 / palo alto, ca 94304-1030

The data below show the first increment of an isotope-edited NOESY experiment. For the same overall gradient strength, equivalent performance is noted for a gradient applied at the magic-angle (upper- two gradient axes used), along the z-axis only using the three-axis probe (middle), and along the z-axis only using a z-axis-only probe (lower). This equivalence in performance permits the user to leave a three-axis probe in the magnet without suffering performance loss for work that uses only z-axis gradients or no gradients at all



Sincerely Yours,

George Gray
NMR Applications Lab

Debbie Mattiello
NMR Research Department

Mike Cummings
NMR Probe Department

Mayo Foundation Rochester, Minnesota 55905 Telephone 507 284-2511

Mayo Clinic Mayo Medical School
Mayo Graduate School of Medicine

August 12, 1994
(received 8/18/94)

Errors in Two Dimensional Exchange Spectroscopy

Dear Barry:

Quantitative two-dimensional exchange spectroscopy (i.e. EXSY, NOESY and all their derivatives) have became so popular that a word of caution is warranted. Namely, there is a tendency to overinterpret quantitative exchange spectra and a feeling that stronger cross-peaks (longer mixing time) yield better (more precise) result upon full matrix analysis (FMA). We have calculated propagation of errors in a multispin system from the experimental peak volumes, $V \pm \Delta V$, into magnetization exchange rates, $L \pm \Delta L$. If volume errors, ΔV, are random, uniform and uncorrelated then the error of the exchange rate constant, ΔL_{ij}, is given by:[1]

$$|\Delta L_{ij}| = \frac{\Delta V}{V_o} \sqrt{\sum_{r=1}^{n}\sum_{v=1}^{n} u_{ir}^2 u_{jv}^2 \left[(1-\delta_{\lambda_r\lambda_v}) \left(\frac{\lambda_r - \lambda_v}{e^{-\lambda_r\tau_m} - e^{-\lambda_v\tau_m}} \right)^2 + \delta_{\lambda_r\lambda_v} \left(\frac{e^{+\lambda_r\tau_m}}{\tau_m} \right)^2 \right]} \qquad (1)$$

with ΔV - the average volume error; V_o - volume of a single spin diagonal at $\tau_m = 0$; τ_m - mixing time; u_i - eigenvectors of the dynamic matrix, L; λ_i - eigenvalues of the dynamic matrix; δ_{ij} - Kronecker δ ($\delta_{ij} = 1$ for $i = j$ and 0 for $i \neq j$); n - number of spin sites. Because eigenvectors and eigenvalues are already calculated in a full matrix analysis, Eq. (1) is easy to use. Error function has a minimum at some optimal mixing time which position depends on topology of the magnetization exchange network. Thus, well developed cross-peaks (longer mixing time) do not guarantee better precision in subsequently calculated magnetization exchange rates. We have verified validity of Eq. (1) by a sequence of NOESY experiments recorded for a range of mixing time on a rigid dipeptide, cyclo(Gly-Pro). The dipeptide was dissolved in a cryoprotective mixture, 1:3 v/v H_2O/DMSO, in which at -30° C the dipeptide overall correlation time was 4 ns, like for small protein at room temperature. Scattering of the cross-relaxation rates obtained from the FMA around the value generated from the known geometry clearly shows minima in the range $\tau_m = 100 - 200$ ms. As equation (1) indicates, there is not much freedom in selecting mixing time for optimal results at a given signal-to-noise ratio. Obviously, only with infinitely small peak volume errors, optimization of mixing time is irrelevant.

1. S. Macura, J. Magn. Reson. B, **104**, 168 (1994)

Sincerely yours,

Nenad Juranić
INTERNET:juranic@mayo.edu

Slobodan Macura
INTERNET:macura@mayo.edu

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for Aqueous samples

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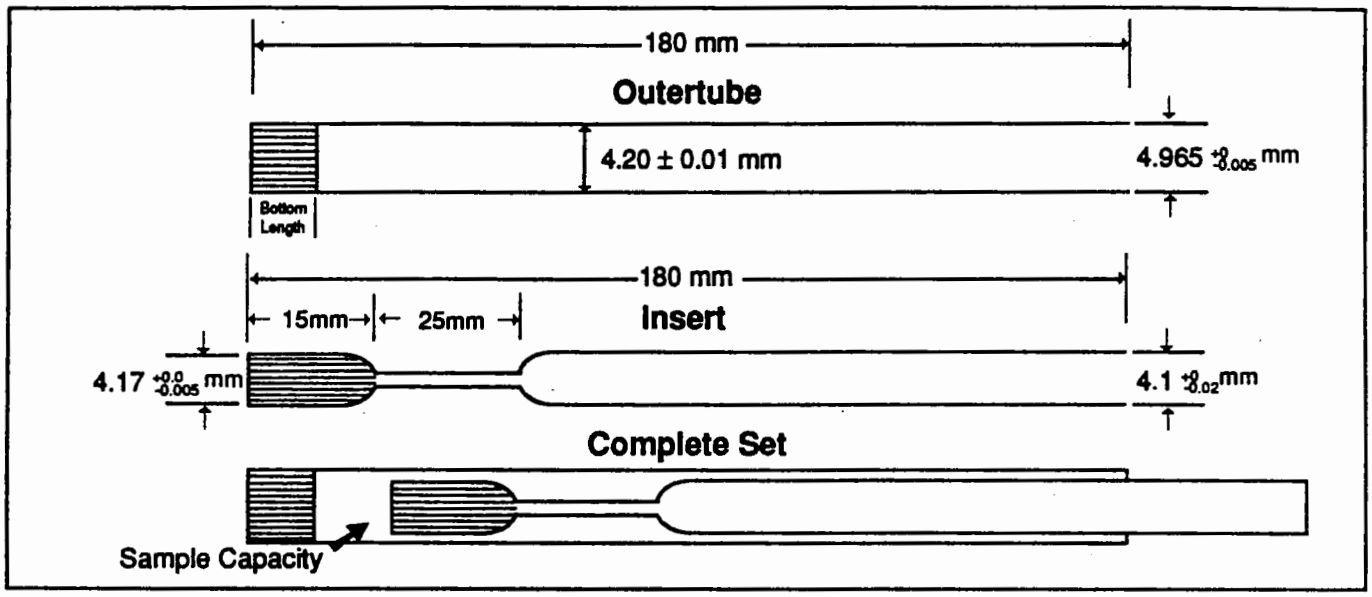

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Complete Set	Insert length (mm)	Insert ID (mm)	Insert OD (mm)	Outertube length (mm)	Outertube ID (mm)	Outertube OD (mm)	Bottom* length (mm)
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BMS-005V	180	2.6	4.1	180	4.2	4.965	15

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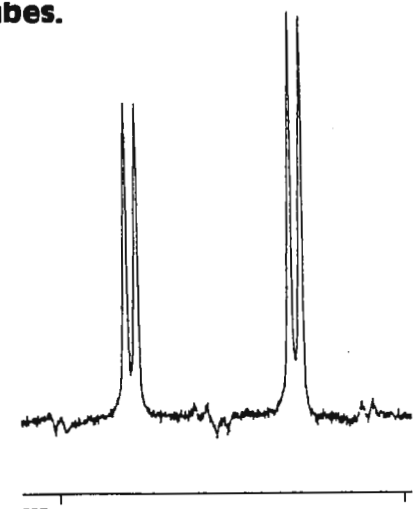
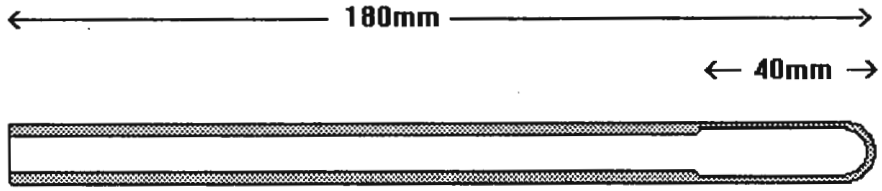
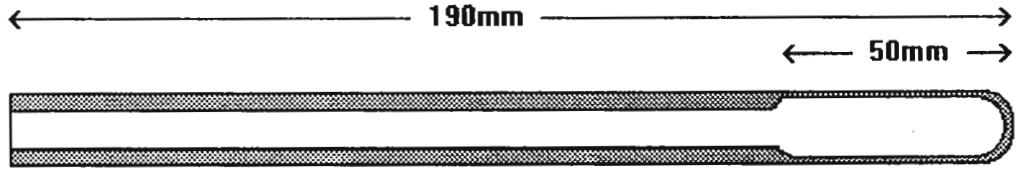


Fig.1a Fig.1b

The spectra of 20mm sucrose in D_2O were obtained with a single scan without apodization prior to Fourier transformation on a Bruker AMX-600 spectrometer at 298 K. By using Shigemi high quality 5mm standard tube (Fig.1a) and the Shigemi highly sensitive thin wall 5mm tube (Fig.1b), the spectra confirms a sensitivity enhancement of about 10%.



PST-001 and PST-002



ST8-001,ST8-002, ST10-001, and ST10-002

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8	ST8-001	0.25	40/ 8	8.00 + 0.00 - 0.01	7.52 ± 0.01	$31.00	$28.00
	ST8-002	0.25	50/15	8.00 + 0.00 - 0.01	7.52 ± 0.01	$27.00	$25.00
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DEPARTMENT OF THE NAVY
NAVAL RESEARCH LABORATORY
WASHINGTON D C 20375-5320

IN REPLY REFER TO:

19 August 1994
(received 8/24/94)

A DIAMOND IS NOT (FOREVER)$^{1/2}$

Dear Barry:

Spin-lattice relaxation of ^{13}C in diamond can be surprisingly efficient when paramagnetic atoms of substitutional nitrogen (so-called P1 centers) are present, as Mark Henrichs, Mike Hewitt and co-workers showed a number of years ago (*J. Magn Reson.*, **58**, 85 [1984]). They observed, using MAS to remove spin-diffusion, ^{13}C magnetization recovery going as $t^{1/2}$ at short recovery times. A number of theoretical treatments have predicted this as the short-time behavior of a more general form $\exp(-t/T')^{1/2}$.

We have found a number of surprises in our detailed quantitative NMR and EPR study of ^{13}C spin-lattice relaxation in several synthetic diamond powders containing such paramagnetic centers that have been provided to us by Dr. Karen McNamara of GE. First of all, the relaxation rate predicted by the standard relaxation formula found in Abragam (p. 380) is much too small when the electron longitudinal relaxation time (measured to be of the order of a ms) is used as the correlation time. The explanation is that since fluctuations in S_z of the electron are what matters in the relaxation process, not only electron T_1 processes but also electron-electron spin flip-flops (via dipolar interactions) of the form S_+S_- need to be considered, particularly at high concentrations of electrons (in our case ca. 10^{19}-$10^{20}/cm^3$). These electron-electron interactions give rise to a calculated electron correlation time of the order of 0.1 μs that can more quantitatively explain the rapid spin-lattice relaxation we observe (on the order of minutes).

One thing leads to another. We had observed ^{13}C relaxation behavior in both static and MAS samples that fit well to a "stretched-exponential" of the form $\exp(-t/T')^{\alpha}$, where the "stretched exponent" α was typically between 0.54 and 0.70. Although a "single-center" model of nuclear relaxation by uniformly-disposed paramagnetic spins has been used in the past, a "multi-center" model seems more appropriate (see Figure). We reasoned that the randomly-situated paramagnetic spins in a multi-center model would produce a *distribution* of electron correlation times that might account for stretched-exponential behavior (just as certain types of distributions do in many other physical phenomena). We are calculating the *distribution of the strongest electron-electron dipolar coupling* and using this to approximate the (inverse) electron correlation time. The resultant distribution of correlation times can be used to calculate the magnetization recovery for an ensemble of ^{13}C nuclei that are assumed individually to follow single-exponential relaxation behavior (as would be expected for static samples with significant spin-diffusion). We have found using a trial (inexact) distribution that stretched-exponential behavior with $\alpha = 0.64$ can be predicted! (non-factorial punctuation).

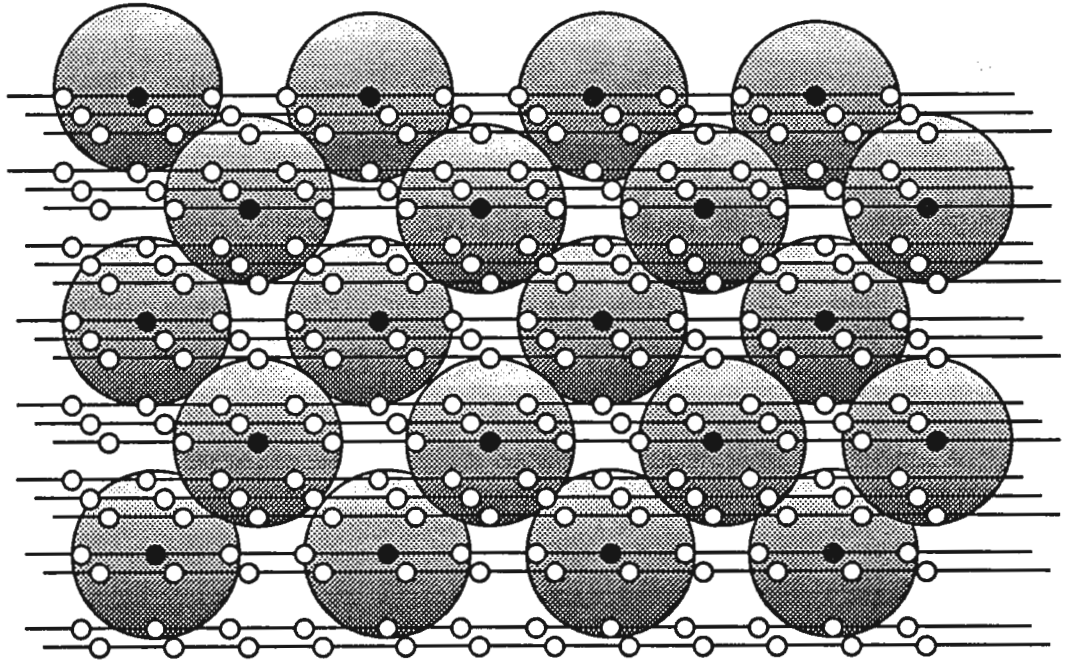
Since past treatments seem to gloss over the angular dependence of the electron-nuclear relaxation rate ($\sin^2\Theta \cos^2\Theta$), we have investigated this aspect of relaxation as well, including the averaging effects of MAS. I would be grateful if any TAMU readers can bring to our attention any unfamiliar literature treating the above points. My e-mail address is "yesinows@nrlfs1.nrl.navy.mil." A full account of this work is being written up for submission to *Physical Review B.*

Sincerely yours,

Blane

D. Blane Baker
NRC Postdoctoral Fellow

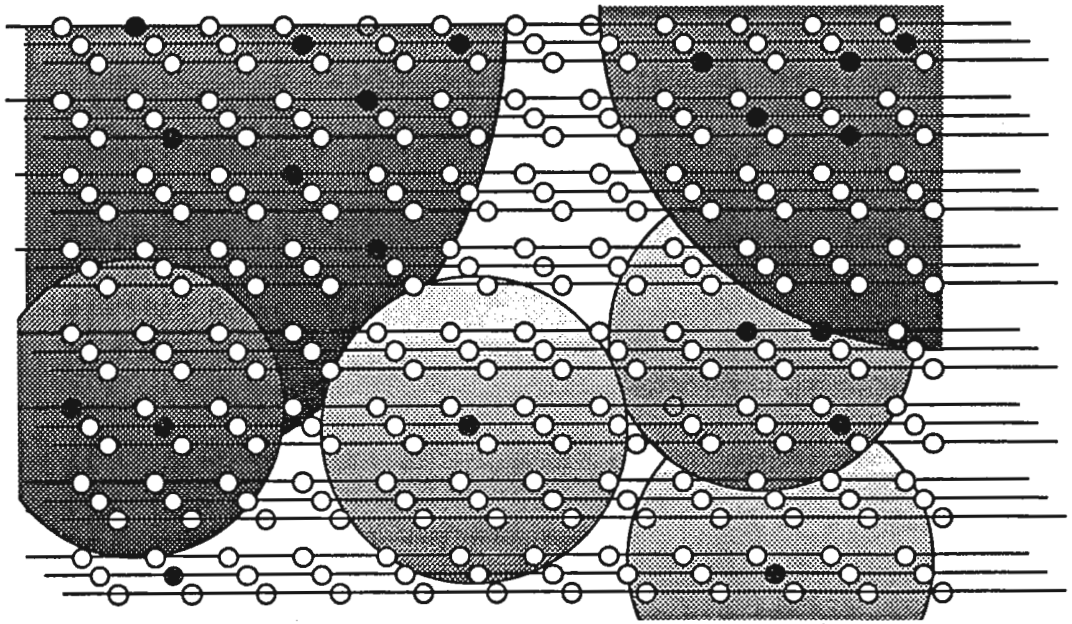
James P. Yesinowski
Materials Chemistry Branch



SINGLE-CENTER RELAXATION

● = PARAMAGNETIC CENTER

○ = LATTICE SITE



= SPHERE OF RELAXATION INFLUENCE

MULTI-CENTER RELAXATION

CENTRE FOR NUCLEAR MAGNETIC RESONANCE

Department of Chemistry
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DR. OLIVER W. HOWARTH

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PROF. RAY DUPREE

Prof. Bernard L. Shapiro
966 Elsinore Court
Palo Alto
California 94303

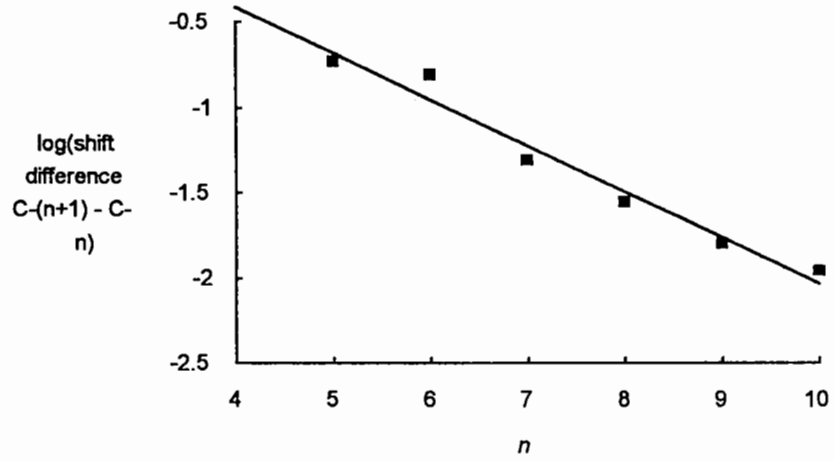
27th July 1994
(received 8/1/94)

Watching charge move down saturated polymethylene chains

Dear Barry,

Last time I wrote, I reported our discovery that CH=CH bonds act as reporters for the displacement of charge down the chains of monounsaturated long-chain esters. We have shown that this effect arises from a through-bond (inductive) interaction rather than a through-space (electric field) one, both from the way the CH=CH ^{13}C shift difference depends on chain length, and also from the fact that its sign does not change when you reverse the ester grouping. This work has now been published[1], and we have since shown that the same attenuation factor of +1.75 per intervening methylene arises even if you change either the reporter group, or the dipole at the end of the chain. Thus the reporter group can be C≡C, or even the 1,4 carbons of a selenophene ring, and the dipole can be OH, OCOR, COOR or COOH. We can also support the effect semi-quantitatively by semi-empirical calculations at the AM1 level.

In the course of this work we studied a fascinating paper on the ^{13}C shifts in fully saturated long chain acids, by Bengsch *et al.*[2] At first sight, these shifts do not seem to form any obvious pattern, because end-effects are clearly visible for the first and last five or so carbons in the chain. These end-effects arise from a combination of through-bond and of γ-*gauche* interactions. However, the authors showed that the central carbons form an unexpected pattern, in that they come to a 'band head' around C_{n-6}, for an n-carbon chain. The position of this 'band head' is close to 29.6 ppm. We believe that these shifts reflect the same tiny charge gradient that we detect *via* unsaturated reporter groups. The shifts at saturated carbon are only about 5% as sensitive to charge as those at unsaturated carbon. Nevertheless, if we plot the log of successive saturated carbon shift differences against chain position (from the acid moiety) we once again get a straight line. Its slope corresponds to + 1.85 per intervening methylene. This is within experimental error of the 1.75 value above, and hence provides an explanation of the otherwise curious observation of a 'band head'.



Convergence of the shift difference between successive carbons in stearic acid (C_{16})

Best wishes

Oliver

1 Bianchi, G.; Howarth, O. W.; Samuel, C. J.; Vlahov, G. *J. Chem. Soc., Chem. Commun.* **1994**, 627-628.
2 Bengsch, E.; Perly, B.; Deleuze, C.; Valero, A. *J. Magn. Reson.* **1986**, *68*, 1-13.

DuPont Central Research and Development
Experimental Station
P.O. Box 80356
Wilmington, Delaware 19880-0356

DuPont Central Research and Development

August 12, 1994
(received 8/15/94)

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

NMR Symposium
at the
37th Rocky Mountain Conference
on Analytical Chemistry

Dear Barry:

Next year, the Rocky Mountain NMR Symposium will take place Monday - Thursday, **July 24 - 27, 1995** at the **Hyatt Regency Denver** in **Denver, Colorado.**

The program will consist of invited lectures and contributed papers for oral and poster presentations on NMR research in areas such as solid-state NMR, spin dynamics, new techniques, polymers, inorganic chemistry, environmental science, materials science, catalysis, surfaces and interfaces, materials imaging, dynamic polarization, etc.

The Symposium is organized by Alexander J. Vega - chair, Joel R. Garbow - co-chair, Lucio Frydman, Bernard C. Gerstein, Jeffrey A. Reimer, Hans Thomann, Robert A. Wind, and James P. Yesinowski.

For further information I can be contacted at DuPont Central Research and Development, P. O. Box 80356, Wilmington, DE 19880-0356; phone: (302) 695-2404; fax: (302) 695-1664; e-mail: vega@esvax.dnet.dupont.com.

Best wishes,

Lex

Alexander J. Vega

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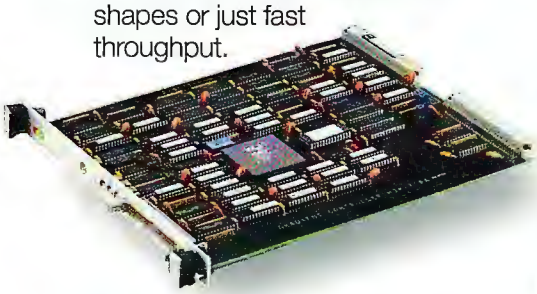

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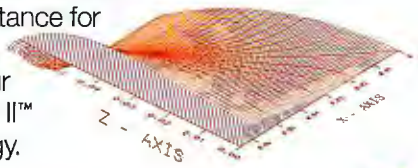

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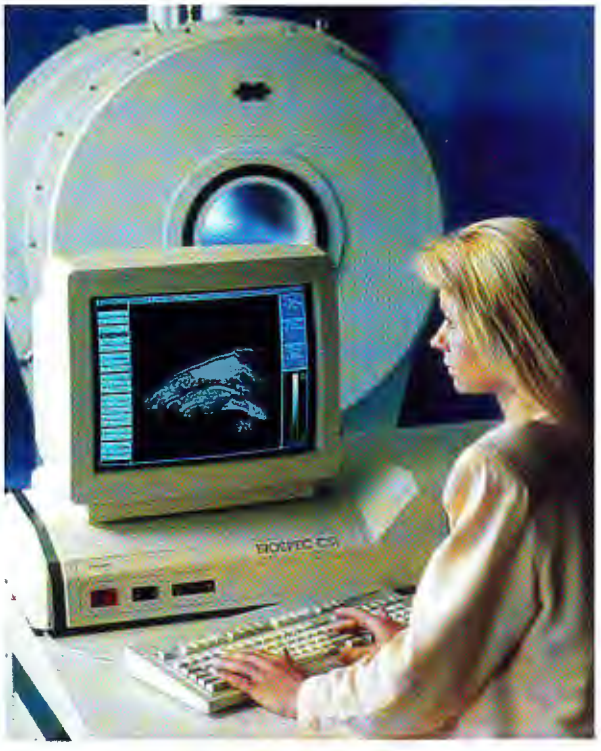

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IUPUI

July 27, 1994
(received 8/1/94)

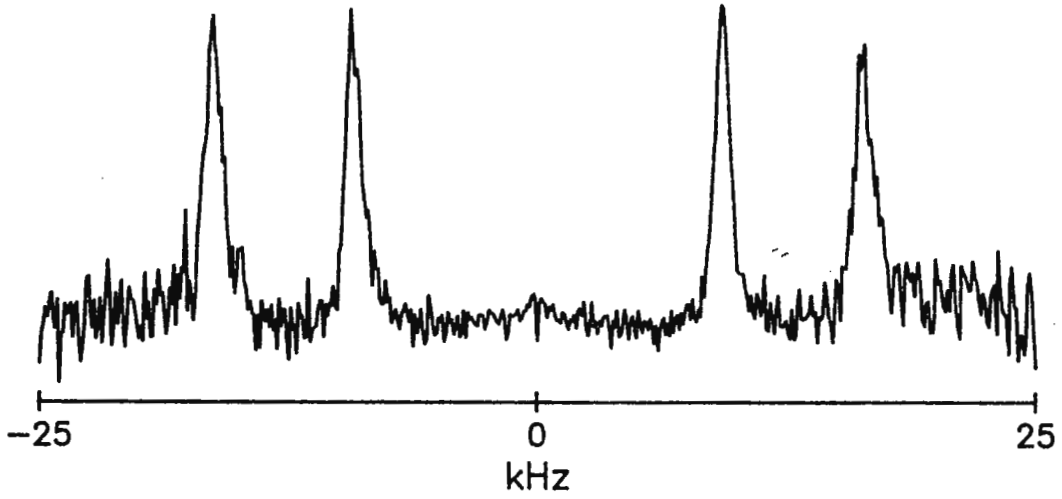
Dr. Bernard L. Shapiro
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto, CA 94303

Depaking by Fast Fourier Transform

Dear Dr. Shapiro:

The generation (depaking) of aligned spectra from powder pattern signals associated with orientational dependent Hamiltonians which scale as $P_2(\cos\theta)$ is a useful technique in studies of membranes. We have recently devised a procedure which, unlike previous methods [1,2], is neither iterative nor dependent upon the inversion of large matrices. Our approach is to employ weighting functions in time and frequency domains, which enables evaluation of an oriented spectrum directly from the FID by FFT. Execution is consequently hundreds of times faster than prior methods.

An example of an oriented ^2H NMR spectrum derived by our procedure for a hydrated sample of potassium [9,10-$^2H_2$] oleate in the smectic mesophase is shown. It agrees well with the aligned spectrum obtained by the conventional iterative method, but this 4K dataset took on the order of 1 second to attain on a 486 computer operating at 33 MHz. This speed offers the opportunity to depake online.



We have recently submitted a paper to *J. Mag. Res.* and we will be pleased eventually to provide details.

1. E. Sternin, M. Bloom, and A. L. Mackay, *J. Magn. Reson.* **55**, 274 (1983).
2. K. P. Whittall, E. Sternin, M. Bloom, and A. L. Mackay, *J. Magn. Reson.* **84**, 64 (1989).

Please credit this contribution to the account of B. D. Nageswara Rao.

Yours sincerely,

M. Alan McCabe Stephen R. Wassall

DEPARTMENT OF PHYSICS

LD 3154
402 N. Blackford Street
Indianapolis, Indiana
46202-3273

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International School of Biological Magnetic Resonance, 2nd Course: Dynamics and the Problem of Recognition in Biological Macromolecules

ETTORE MAJORANA CENTRE FOR SCIENTIFIC CULTURE

Erice, Trapani, Sicily, Italy
22-30 May 1995

Preliminary Program and Lecturers

<u>Basic NMR Methods for Structure and Dynamics Studies</u>	
Basics of Liquid State NMR Spectroscopy	Hartmut Oschkinat, EMBL Heidelberg, Germany
Basics of Solid State NMR Spectroscopy	Stanley Opella, University of Pennsylvania, USA
Heteronuclear Relaxation and Molecular Dynamics	Gerhard Wagner, Harvard Medical School, USA
Proton Exchange and Internal Dynamics	Walter Englander, University of Pennsylvania, USA
Methods for Isotopic Labeling of Biomolecules	John L. Markley, University of Wisconsin, USA
<u>Simulated and Observed Molecular Dynamics</u>	
Molecular Dynamics and Interaction Simulation	Martin Karplus, Harvard University, USA
Simulating Protein and Nucleic Acid Molecular Dynamics	Michael Levitt, Stanford University, USA
Dynamics Observed in the *trp* Repressor	Oleg Jardetzky, Stanford University, USA
Dynamics of Peptides Observed by NMR and Other Physical Methods	Rudolf Rigler, Karolinska Inst., Stockholm, Sweden
<u>Dynamics of Polysaccharides</u>	Robert G. Shulman, Yale University, USA
<u>Protein-Small Molecule Interactions</u>	
Enzyme-Ligand and Inhibitor Interaction	Gordon C. K. Roberts, University of Leicester, UK
Structural and Dynamical Consequences of Ion Binding on Metalloproteins	Sture Forsén, University of Lund, Sweden
<u>Protein Motion and Folding</u>	
Protein Structure and Dynamics Studied by Spectral Editing	John L. Markley, University of Wisconsin , USA
Pathways of Protein Folding	Christopher M. Dobson, Oxford University, UK
<u>Nucleic Acids and Protein-Nucleic Acid Interactions</u>	
Plasticity of DNA Structure and Mutagenesis	Jean-François Lefèvre, Louis Pasteur University, France
Structure and Dynamics of RNA	Cornelius Hilbers, University of Nijmegen, NL
Interaction between *trp* Repressor and *trp* Operator	Oleg Jardetzky, Stanford University, USA
NMR Studies of Protein-Nucleic Acid Complexes	Rolf Boelens, Utrecht University, NL
<u>Protein-Protein Recognition</u>	
Protein A and G Interactions with Antibodies	Gordon C. K. Roberts, University of Leicester, UK
Antigen and Superantigen Recognition by MHC Class II Receptor	Theodore Jardetzky, Northwestern University, USA
Inhibitor-Protease Interactions	Tad Holak, Max-Planck-Institut, Munich, Germany
Crystallographic Studies of Protein-Protein Interactions	Rudolf Ladenstein, Karolinska Inst., Stockholm, Sweden
<u>Protein-Lipid Interactions</u>	
NMR Studies of Protein Membrane Interaction	Stanley Opella, University of Pennsylvania, USA

PURPOSE OF THE SCHOOL

The School will be devoted to the analysis of the dynamic behavior of biological macromolecules by Nuclear Magnetic Resonance and the implication of dynamics on the mechanism of recognition between macromolecules or macromolecules and small substrates. The subjects listed above will be explored in detail during the course. Students are encouraged to submit abstracts and some student research will be selected for presentation in workshops.

NMR has reached the stage where both three-dimensional structure of macromolecular complexes and dynamics can be studied quite accurately. An integrated approach between these two aspects should be utilized more and more in future research. The proposed course will bring together the interesting features of such an integrated approach.

VENUE

The Ettore Majorana Centre for Scientific Culture was founded in 1963 in the pre-medieval mountain town of Erice near Palermo as a Conference Centre, taking its inspiration from the Italian Physicist, Ettore Majorana. The Centre's lecture halls are located in two restored monasteries and the ancient Palazzo Ventimiglia. School participants are housed in the Centre Institutes or local hotels and meals are taken at local restaurants.

GENERAL INFORMATION

Prospective participants should apply to either:

Prof. Oleg Jardetzky
Stanford Magnetic Resonance Laboratory
Stanford University
Stanford, CA 94305-5055
USA
fax: +415/723-2253
phone: +415/723-6270
jardetzky@camis.stanford.edu

or

Prof. Jean-François Lefèvre
ESBS, CNRS-UPR9003
Université Louis Pasteur
Blvd. Sébastien Brant
F67400 Illkirch Graffenstaden
France
fax: +33/88 65 53 43
phone: +33/88 65 52 69
lefevre@bali.u-strasbg.fr

stating: (1) date and place of birth, nationality, qualifications and present position; (2) address, fax and phone numbers and email address; and (3) list of publications.

Applicants interested in submitting unpublished results should send the title and an abstract of about 200 words. Selected papers will be presented and discussed in special sessions.

The total fee, including full board and lodging (arranged by the School) will be approximately US $1,000. Limited financial aid available. Participants should arrive by 5 p.m. on the 22nd.

THE CLOSING DATE FOR RECEIPT OF APPLICATIONS IS MARCH 20, 1995. NO APPLICATION FORM IS REQUIRED.

Attendance will be limited to ~75 students, to be selected by the Co-Directors. Further details will be mailed with the acceptance letter.

Oleg Jardetzky
Co-Director of the School

A. Zichichi
Director of the Centre

Jean-François Lefèvre
Co-Director of the School

United States Department of Agriculture
Agricultural Research Service
Midwest Area National Center for Agricultural Utilization Research
1815 North University Street
Peoria, Illinois 61604
Telephone: 309-685-4011
FAX: 309-681-6686

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

(received 8/5/94)

Solution to Low Line Voltage on an MSL

Dear Barry,

We have recently had trouble with low voltage in our lab. The "220"(208 volts) lines can be as low as 198 volts routinely. Although the NMR spectrometers can operate at this voltage I experience some power droop in the output of the amplifiers which is unacceptable for CP. In fact, this is how I became aware of the problem with the low voltage.

I noticed during CPMAS setup that the intensity of the FID was not stable. I traced this problem to a variable power droop in most of the amplifiers. Thus it was impossible to maintain the match in proton and carbon power levels. The voltage at my switch box was compared to the voltage at the transformer and we found that the problem was not in the way it was wired, rather the whole wing had the low voltage. Monitoring of the voltage over a period of weeks showed that the voltage remains low but fairly stable. I have been able to convince the building engineer to replace the 50 year old transformers for my wing with new ones capable of supplying the ever increasing demand for electricity (this had been planned to coincide with renovation of the wing but the timetable for the renovation has been severely slowed due to budget cuts). I have been assured that the work will be completed before the end of the year but I still needed a temporary solution.

Since the voltage was stable I did not need a motor generator or a switching transformer. Fortunately my MSL has a transformer to accomodate several countries power grids so I turned off the three phase switch box to change the wires in the MSL's transformer from the 208 volt taps to the 195 volt taps. This allows the MSL to run with only a 2% over voltage rather than the 5% under voltage (which ruins sensitive experiments) all at no cost. The cost issue was very important for the temporary solution since a permanent fix was coming. Everything has been working fine for weeks now and the only problem I foresee is if everyone in the wing turns everything else off and CILCO supplies an over voltage.

Sincerely,

Arthur R. Thompson

Arthur R. Thompson

Duke University

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

August 17, 1994
(received 8/23/94)

$^{19}F - {}^{13}C$ 2D NMR Determination of Coupling Constant Sign

Dear Barry,

In this contribution I continue the 2D NMR explorations of determination of coupling constant signs. Using 1H-13C 2D NMR on the fluorinated anesthetic, halothane ($CF_3CHClBr$), the HC correlation was previously (1) shown to be modulated by coupling from the three equivalent fluorine nuclei (passive M spin) and appear as a group of four cross peaks separated by $3J_{HF}$ and $^nJ_{CF}$. In the direct bond map as processed with Varian software, the four cross peaks oriented with positive slope. Thus $3J_{HF}$ and $2J_{CF}$ have the same relative sign in halothane. However, in the analogous multi-bond map, the four cross peaks oriented with negative slope and $3J_{HF}$ and $1J_{CF}$ were determined to have opposite relative signs.

I wondered if a different view of coupling effects might be achieved by 19F-13C 2D NMR. Since the 19F NMR scale (300 ppm) is considerably wider than that of 1H (10 ppm) and even wider than that of 13C (200 ppm), adequate 19F/13C pulse power and digitization of cross peaks were issues to consider. Also this appears to be relatively new ground as there is a paucity of 19F-13C 2D data in the NMR literature. Nevertheless, I forged ahead and after the usual wrong turns and finally the appropriate setup and calibrations on a Varian Unity 500 began recording my first successful 19F-13C 2D spectra over the past year.

Figure 1 shows the 19F-13C direct bond map for halothane recorded at high digitization, with projections to emphasize the resolution. A group of two cross peaks is obtained. The peaks are separated by $3J_{HF}$ and $2J_{HC}$, and are seen to orient with negative slope i. e. the higher frequency 19F resonance near -77.35 ppm has the lower 13C frequency near 121.32 ppm. $3J_{HF}$ and $2J_{HC}$ are hence determined to have opposite relative signs.

Figure 2 shows the multi-bond map correlating the fluorines to the CH group. This appears as two cross peaks separated by $3J_{HF}$ and $1J_{HC}$ and tilted with positive slope. The higher frequency 19F resonance near -77.35 ppm occurs at the higher 13C position near 50.7 ppm. $3J_{HF}$ and $1J_{HC}$ are thus determined to have the same relative sign. Some of this work has recently appeared (2).

Sincerely,
Anthony A. Ribeiro Tony

1. TAMU NMR Newsletter 422 p. 29 (Nov. 1993).
2. J. Magn. Reson. 107 158 (1994).

Figure 1.

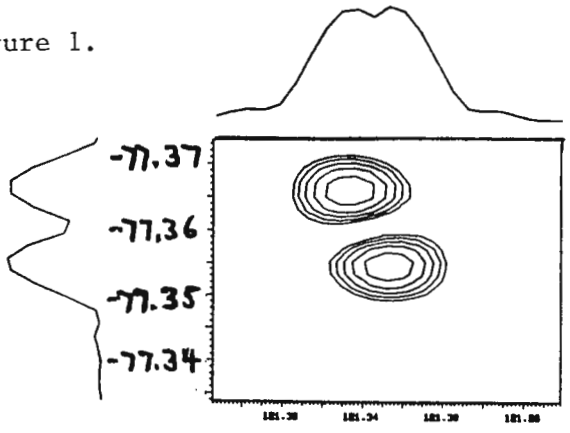
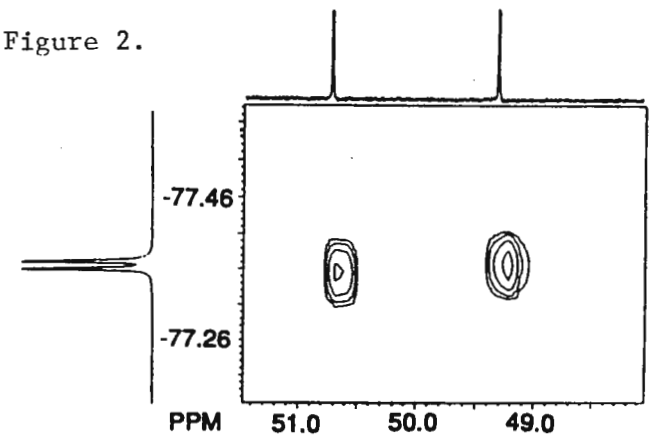


Figure 2.



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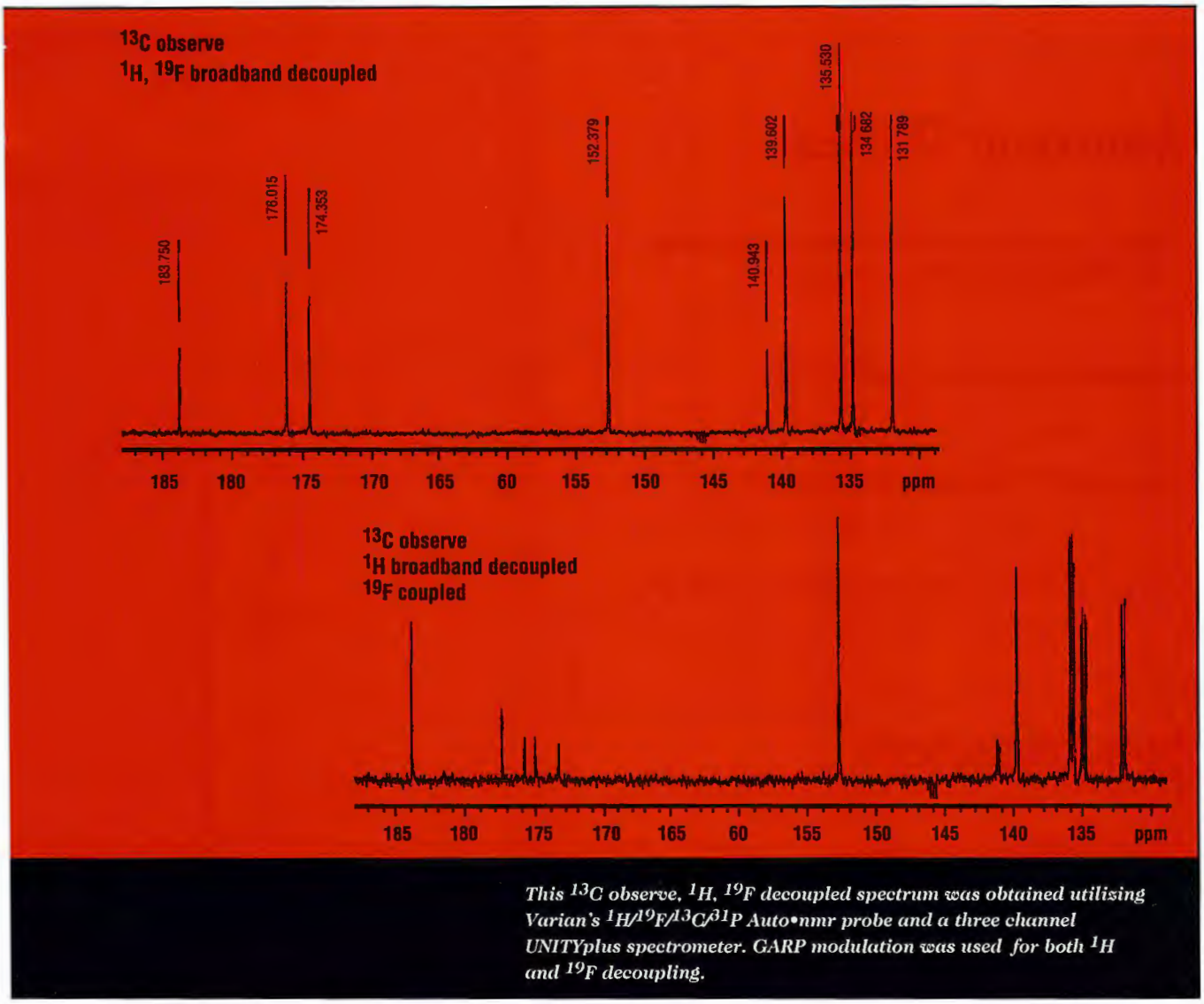

This ^{13}C observe, ^{1}H, ^{19}F decoupled spectrum was obtained utilising Varian's ^{1}H/^{19}F/^{13}C/^{31}P Auto•nmr probe and a three channel UNITYplus spectrometer. GARP modulation was used for both ^{1}H and ^{19}F decoupling.

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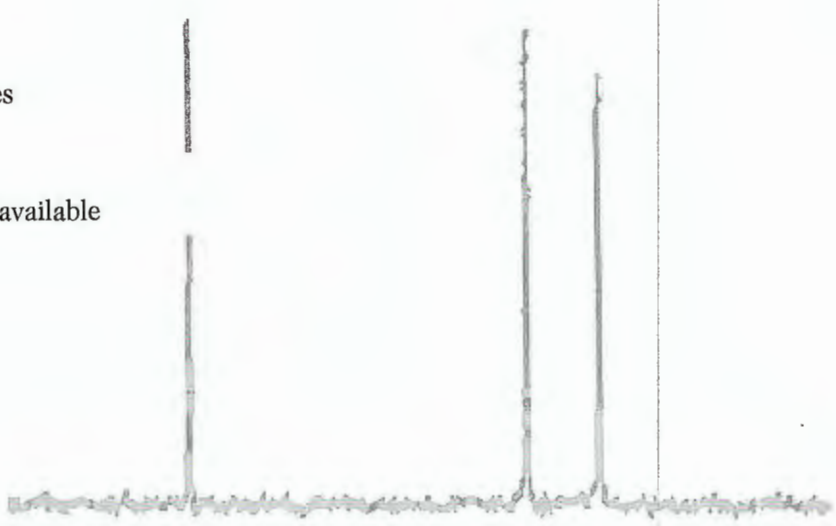

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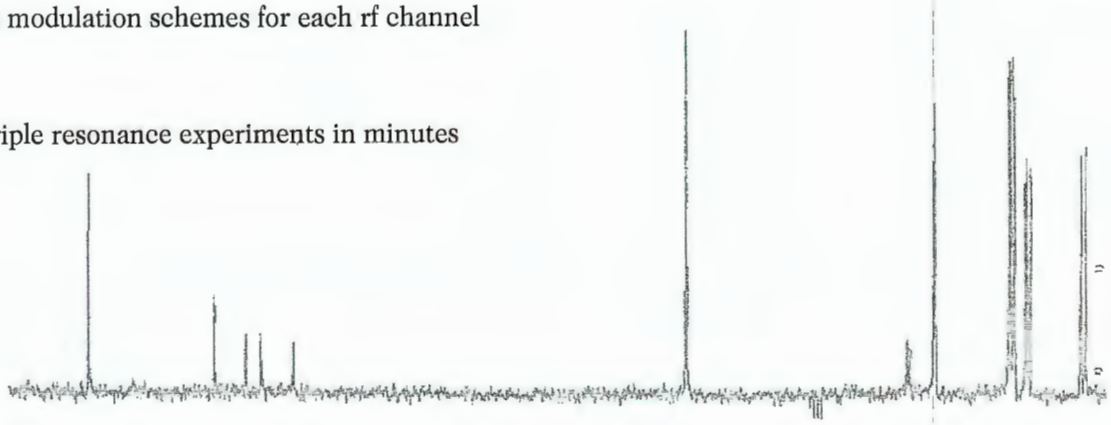

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MAG-8074/499

Carnegie Mellon University
Department of Chemistry
4400 Fifth Avenue
Pittsburgh, PA 15213

Tue Aug 9 13:53:59 EDT 1994
(received 8/15:43.63/94)

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

Dear Barry,

Your gentle(?) nudging has finally moved me to write you about the latest things going on here. As you know, I am gradually winding down my NMR activities, but still am searching for the epiphany, wherein all the central phenomena of NMR suddenly fall into place, and the observations make sense.

Recently, while fatootsing* around among the relations between the various dipolar relaxation rate equations, I noticed something on which I haven't seen any comments, although it seems to me exceedingly unlikely that it has gone unnoticed. The expressions familiar to all ardent fans of relaxation are:

$$\rho_1 = K^2[J(0) + 3J(\omega) + 6J(2\omega)]$$

$$\rho_2 = K^2[2.5J(0) + 4.5J(\omega) + 3J(2\omega)]$$

$$\sigma_1 = K^2[-J(0) + 6J(2\omega)]$$

$$\sigma_2 = K^2[2J(0) + 3J(\omega)]$$

Just looking at this one can easily see that

$$\rho_1 = \sigma_1 + \sigma_2$$

$$\rho_2 = 0.5\sigma_1 + 1.5\sigma_2$$

and

$$\sigma_1 = 1.5\rho_1 - \rho_2$$

$$\sigma_2 = -0.5\rho_1 + \rho_2$$

Since these are all connected together, one can presumably measure any two and calculate the others from these. For example, one could measure just the longitudinal ρ_1 and σ_1, and calculate the transverse ones, from

$$\sigma_2 = \rho_1 - \sigma_1$$

$$\rho_2 = 1.5\rho_1 - \sigma_1$$

---

* For a definition of this old Bronxian participle, consult Prof. Natalie Mayer, (408) 738 1672

Somehow everything that is going on, dipolar-relaxationwise, is reduceable to just two fundamental processes. It's not clear how to identify them. However it is interesting to diagonalize the coefficent matrix above:

$$\begin{vmatrix} 1 & 1 \\ 0.5 & 1.5 \end{vmatrix}$$

The eigenvectors give the new combinations:

$$\rho_1+2\rho_2 = 2\sigma_1 + 4\sigma_2$$

$$2(\rho_1 - \rho_2) = (\sigma_1 - \sigma_2)$$

From the first of these one gets

$$(\sigma_1 + 2\sigma_2)/(\rho_1 + 2\rho_{2)} = 0.5$$

which, I think, says that the Overhauser effect along the axis at the magic angle is always 0.5

Has someone checked this all out already? It looks to me like it offers some new and interesting ways to get pairwise distances and correlation times out of NOESY and ROESY(!!@*) spectral data.

Sincerely

Aksel A. Bothner-By
University Professor Emeritus

Protein Engineering Network of Centres of Excellence
713 Heritage Medical Research Centre
University of Alberta
Edmonton, Alberta
T6G 2S2

Telephone (403) 492-6540
Fax (403) 492-1473

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

Nearest Neighbor Effects on Random Coil Chemical Shifts

Dear Barry,

These days there is an increasing interest in using ^{1}H, ^{13}C and ^{15}N chemical shifts to refine or characterize the structure and dynamics of peptides and proteins. Critical to the success of many of these approaches is the availability of tables describing the so-called "random coil" chemical shifts of the 20 common amino acids. Historically random coil chemical shifts have been measured using short (four or five residue) substituted linear peptides in a variety of solvent conditions referenced against an equally large number of different (obsolete?) chemical shift standards. The result is kind of a hodge-podge of "random coil" chemical shift tables (particularly for ^{13}C and ^{15}N data) which has led to a rather confusing situation for those of us interested in chemical shift analysis.

About 3 years ago, we embarked on a project to measure the "random coil" ^{1}H, ^{13}C and ^{15}N chemical shifts of not only all 20 common amino acids, but also all 400 possible pairs of amino acids. The idea behind looking at amino acid pairs arose from our long-standing interest in understanding the influence of nearest neighbor or sequence effects on random coil chemical shifts. Back in the 1970's a number of people noticed the influence that proline had on the ^{13}C chemical shifts of amino acids which preceded it. More recently Glushka et al. (1989) described a similar kind of nearest neighbor effect on ^{15}N chemical shifts. We decided to check these observations out in more detail to see if, in fact, they were true for all 20 amino acids and for all (^{1}H, ^{13}C and ^{15}N) nuclei.

We chose to work with a protected hexapeptide model with the sequence: Acetyl-Gly-Gly-Xxx-Yyy-Gly-Gly-amide. Where Xxx and Yyy are any two of the 20 common amino acids. So far, we have managed to measure all observable ^{1}H, ^{13}C and ^{15}N chemical shifts for two sets of peptides: GGXAGG and GGXPGG (using the standard one-letter code; X=any amino acid). This "little" project required the synthesis, purification and characterization of more than 50 different peptides (to get everything just right) and approximately 1500 hours of NMR time!

Was it worth it? You be the judge. In Table 1 (see next page) we present the results showing the effects that proline has on the chemical shifts of peptide backbone nuclei. In Table 2 we present the results showing "random coil" chemical shifts for alanine when preceded by the 20 common amino acids. From Table 1 we can clearly conclude that proline (when on the C terminal side of a residue) affects the chemical shifts of all 20 amino acids for all measurable nuclei (^{1}H, ^{13}C and ^{15}N). Equally interesting, from Table 2 we can see that when any of the 20 common amino acid residues precedes alanine, its ^{1}HN and ^{15}N-amide chemical shifts can be significantly affected. We expect this "amide" effect will be observable for the other 19 amino acids as well.

To conclude, it appears that random coil chemical shifts do depend on sequence context and that nearest neighbor effects, be they steric or electronic in origin, do have a significant role to play in determining the chemical shift of amino acids in peptides and proteins.

Glushka, J., Lee, M., Coffin, S. and Cowburn, D. (1989) *J. Am. Chem. Soc.*, **111**, 7716-7722.

All the best,

David

David S. Wishart

Brian

Brian D. Sykes

Table 1: Effects of Proline on the Chemical Shifts of Peptide Backbone Nuclei.

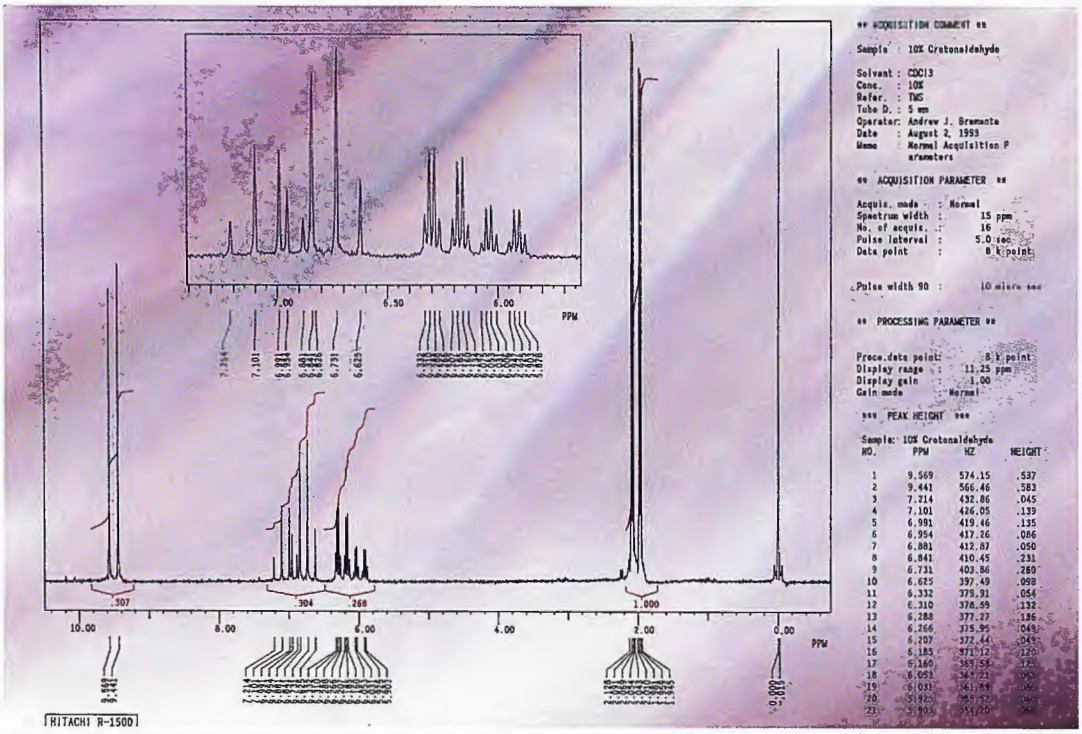
Residue	Hα	Amide H	Cα	C=O	Amide N
Ala	-0.30	0.05	2.0	1.9	-1.2
Cys(reduced)	-0.26	0.02	1.8	1.7	-0.6
Asp	-0.27	0.03	1.9	2.0	-1.0
Glu	-0.29	0.03	2.4	1.9	-1.5
Phe	-0.28	0.17	2.1	1.4	-1.1
Gly	-0.17	0.12	0.6	0.4	-0.3
His	-0.27	0.05	1.7	1.5	0.0
Ile	-0.30	-0.06	2.4	1.4	-1.8
Lys	-0.28	0.11	2.0	1.8	-1.2
Leu	-0.29	0.02	2.0	1.9	-0.8
Met	-0.34	0.03	2.1	1.7	-1.1
Asn	-0.26	0.03	1.8	1.6	-0.3
Pro	-0.31	-----	1.8	5.9	-----
Gln	-0.31	0.03	2.0	1.6	-0.8
Arg	-0.31	0.03	2.0	1.8	-0.8
Ser	-0.31	0.05	1.9	1.5	-0.9
Thr	-0.26	0.00	2.0	1.5	-2.4
Val	-0.32	0.01	2.4	1.4	-1.3
Trp	-0.33	0.16	1.8	1.3	-0.9
Tyr	-0.29	0.02	2.1	1.1	-0.5
Average	-0.288	0.047	1.94	1.55	-0.95
Std. Dev.	0.036	0.056	0.37	1.04	0.56

Chemical shift differences calculated as $\delta X_A - \delta X_P$ where δX_A is the chemical shift of residue X in GGXAGG and δX_P is the chemical shift of resiude X in GGXPGG.

Table 2: Random Coil Chemical Shifts for Alanine when preceeded by the 20 Common Amino Acids.

Residue	Hα	Amide H	Cα	C=O	Amide N
Ala	4.33	8.34	52.6	178.5	123.2
Cys(reduced)	4.35	8.51	52.8	178.3	126.7
Asp	4.33	8.38	52.9	178.4	124.8
Glu	4.34	8.44	52.7	178.4	125.2
Phe	4.31	8.38	52.5	178.0	126.4
Gly	4.35	8.30	52.7	178.5	124.0
His	4.37	8.47	52.7	178.2	125.8
Ile	4.34	8.47	52.7	178.3	128.2
Lys	4.34	8.42	52.6	178.3	125.6
Leu	4.32	8.36	52.7	178.4	125.0
Met	4.34	8.40	52.7	178.3	125.1
Asn	4.34	8.38	52.8	178.3	124.7
Pro	4.33	8.50	52.7	178.6	124.4
Gln	4.34	8.44	52.7	178.4	125.3
Arg	4.35	8.44	52.6	178.3	125.4
Ser	4.37	8.42	52.7	178.3	125.9
Thr	4.36	8.43	52.7	178.3	126.4
Val	4.34	8.48	52.7	178.4	127.9
Trp	4.23	8.26	52.3	179.0	126.8
Tyr	4.31	8.35	52.4	177.9	126.8

^{1}H and ^{13}C chemical shifts directly referenced to internal DSS at 25 °C, pH ~5.0. ^{15}N chemical shifts indirectly referenced to external $NH_{3(l)}$ at 25 °C.



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29 June 1994
(received 8/27/94 [sic])

Professor Bernard L Shapiro
966 Elsinore Court
Palo Alto
CA 94303

Dear Barry,

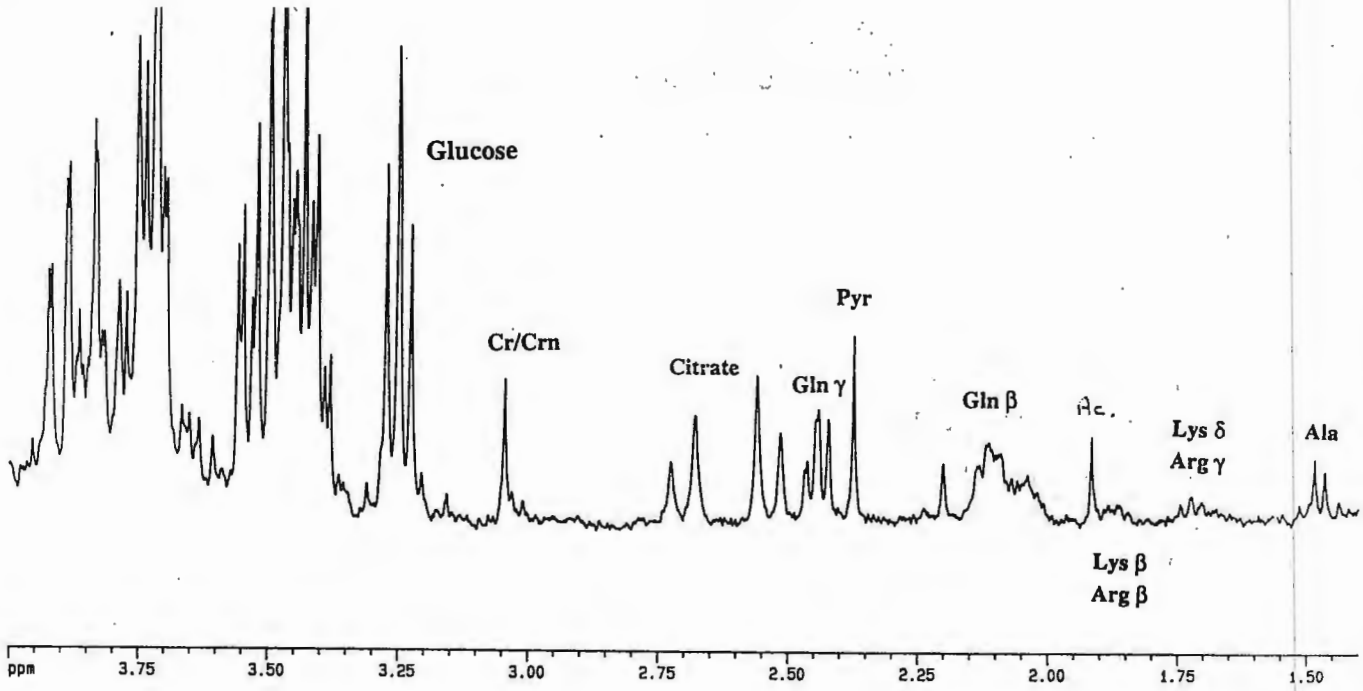
Many thanks for the reminder! Assisted by our colleagues Mike James and Nigel Wood in our Neurosciences Division, we have been examining cerebrospinal fluid (CSF) from rats which have been subjected to photothrombotically induced cerebral ischaemia. This involves injecting a photo-activable dye like rose bengal into the animal and shining light onto stereotaxically located positions on the skull. Photolysis of the dye produces oxygen free radicals which cause platelet agreggation and rapid localised thrombus production. Usually the size of the lesions are measured histologically post-mortem. Changes in the constitution of CSF as a result of ischaemia may provide an alternative, relatively non-invasive, index of the severity of stroke damage and the efficacy of drug treatment.

The Figure shows part of a 360MHz spectrum from CSF from a control (ie unstroked) male hooded Lister rat. The largest metabolite peak in the spectrum is due to lactate and is not shown for the sake of clarity. Other metabolites which make a significant contribution are Alanine, Lysine, Arginine, Acetate, Glutamine, Pyruvate, Citrate and Glucose. We are currently using 2-D methods to assign the large number of minor metabolite signals obscured by glucose resonances between 3.25 and 4 ppm.

Best wishes,

Yours sincerely

David G Reid David A Middleton Michael A Yates



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2514-07,-08,-09	360	.3937	+.0000"/-.0005"	.3569	± .0005"	≤ .0015"	≤ .0005"
2513-57,-58,-59	150	.3937	+.0000"/-.0005"	.3569	±.0005"	≤ .0020"	≤ .0010"
2513-37,-38,-39	80	.3937	+.0000"/-.0005"	.3569	±.0005"	≤ .0030"	≤ .0015"
2513-17,-18,-19	60	.3937	+.0000"/-.0005"	.3569	±.0005"	≤ .0050"	≤ .0020"
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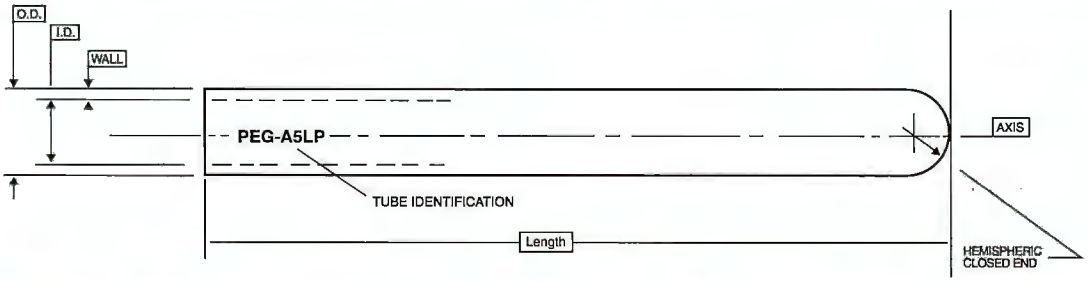

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2506-07	5mm/7"	★22.50	**2506-08**	5mm/8"	★24.75	**2506-09**	5mm/9"	★ 27.25
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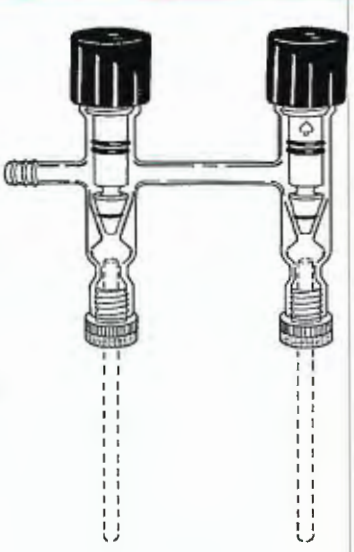
8731 MANIFOLD, Tip-Off ♠



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		Glass Only		Tip-Off Bushing		Tip-Off Port O-Rings	
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1	10mm	**-19**	54.95	**8731-80**	21.80	**7855-716**	15.07
4	5mm, 5mm, 8mm, 10mm	**-22**	199.14	**See Above**	—	**See Above**	—

	Code	Each
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Replacement Plug O-Rings/set	**8194-86**	2.62

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DEPARTMENT OF CHEMISTRY

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

August 19, 1994
(received 8/26/94)

States-Redfield phase incrementation vs. true States-TPPI

Dear Professor Shapiro,

Thank you very much for sending me the previous issues of TAMU NMR Newsletters; a lot to read, a lot to learn. Right away I have run across a letter from Glaxo Inc., Research Institute (TAMU 425-9; FELIX Processing of Bruker States-TPPI Data). I would like to contribute to this issue of data processing and extend their valuable comments with the following general remarks.

In the Glaxo letter a FELIX macro is presented which will do the conversion of data acquired by the phase cycling supplied in the Bruker program into that of conventional, e.g. "true" States-TPPI structure *(1)*. This macro does the same as a special post-acquisition processing step in the Bruker software which is closely related to the so-called Redfield-trick *(2)*. The only difference is that in the original Redfield-Kunz approach, inversion of every other two points has taken place along the sequentially acquired ("real") vector, while in this case every other complex pair should be inverted along the vector acquired in simultaneous fashion. With no inversion the spectral window seems to be shifted away by a half a spectral width leading to serious folding/aliasing.

We have developed and have been using such acquisition scheme on GE instruments (GN and Omega) using off-line software of the late NMRi (now absorbed by Tripos, St.Louis, MO). Due to the close relationship to the Redfield-Kunz acquisition scheme and that of additional processing required we have termed this phase cycling method as ***States-Redfield phase incrementation*** *(3)*. In a typical pulse sequence phase cycling for the two alternative incrementation schemes will proceed as follows (incrementing preparatory phases *(4)*; other combinations are possible, too):

States-Redfield	*States-TPPI (1)*
...	...
increment preparatory phase(s)	increment preparatory phase(s)
loop back, times 2	loop back, times 2
increment preparatory phase(s)	increment preparatory phase(s)
	increment receiver phase
	increment receiver phase
increment time delay	increment time delay
loop back, times N	loop back, times N
...	...

The current "States-TPPI" method in the Bruker software uses the States-Redfield acquisition scheme followed by appropriate inversion of points hidden from the user. True States-TPPI data need no inversion, consequently should be processed with the processing flag set to "States" (MC2=RSH) in the Bruker software (just a straight complex FT). The States-Redfield scheme is simpler to implement in a pulse sequence, especially for experiments of high dimensionality. Also, it may offer a simpler data processing route if axial peaks were to be removed through time domain filtering prior to regular processing *(3)*.

Room 1-014 | Center for Science & Technology | Syracuse, New York 13244-4100 | 315-443-2925 | FAX 315-443-4070

Processing usually does not pose a problem provided the "flags" are set consistently (does anyone remember the closely related "*W*" flag - e.g., Wüthrich-type phase incrementation flag for processing - in the old DISNMR software?) and data acquisition and processing is done in the same environment. Off-line processing has to pay additional attention to this feature and offer capabiliy for independent Redfield-Kunz type inversion, especially if home-written experiments are mixed with "standard" library items. Please, find a complete set of acquisition schemes, related processing requirements and characteristic features of spectra summarized in **Table 1** *(3)*.

With my best regards,

István Pelczer

Table 1

Time increment	*Phase incr.*	*Receiver*	*FT*	*Axial pks.*	*Modulated peaks*
Redfield-Kunz*					
t	x	x	real	center	shifted
t + 1Δ	y	x			
t + 2Δ	x	-x			
t + 3Δ	y	-x			
TPPI					
t	x	x	real	edge	normal
t + 1Δ	y	x			
t + 2Δ	-x	x			
t + 3Δ	-y	x			
States-Haberkorn-Ruben					
t	x	x	complex	center	normal
t	y	x			
t + 2Δ	x	x			
t + 2Δ	y	x			
States-TPPI					
t	x	x	complex	edge	normal
t	y	x			
t + 2Δ	-x	-x			
t + 2Δ	-y	-x			
States-Redfield*					
t	x	x	complex	center	shifted
t	y	x			
t + 2Δ	-x	x			
t + 2Δ	-x	x			

where $\Delta = 1/(2 \times SW)$

* Needs inversion of every other pair of data points during processing in order to get normal spectral arrangement.

(1) Marion, D., Ikura, M., Tschudin, R. and Bax, A., *J. Magn. Reson.*, 85(1989)393.

(2) Redfield, A. G. and Kunz, S. D., *J. Magn. Reson.*, 19(1975)250.

(3) Bishop, K. D., Pelczer, I.* and James, T. L.; "States-Redfield Phase Incrementation: An Alternative Acquisition Scheme in Multidimensional NMR" 34th ENC, St.Louis, MO, USA, March 14-18, 1993, abstracts: P 53 (I.P., K.D.B., T.L.J.; manuscript to be submitted to *Concepts in NMR*)

(4) Griesinger, C., Sørensen, O. W., and Ernst, R. R., *J. Magn. Reson.*, 84(1989)14.

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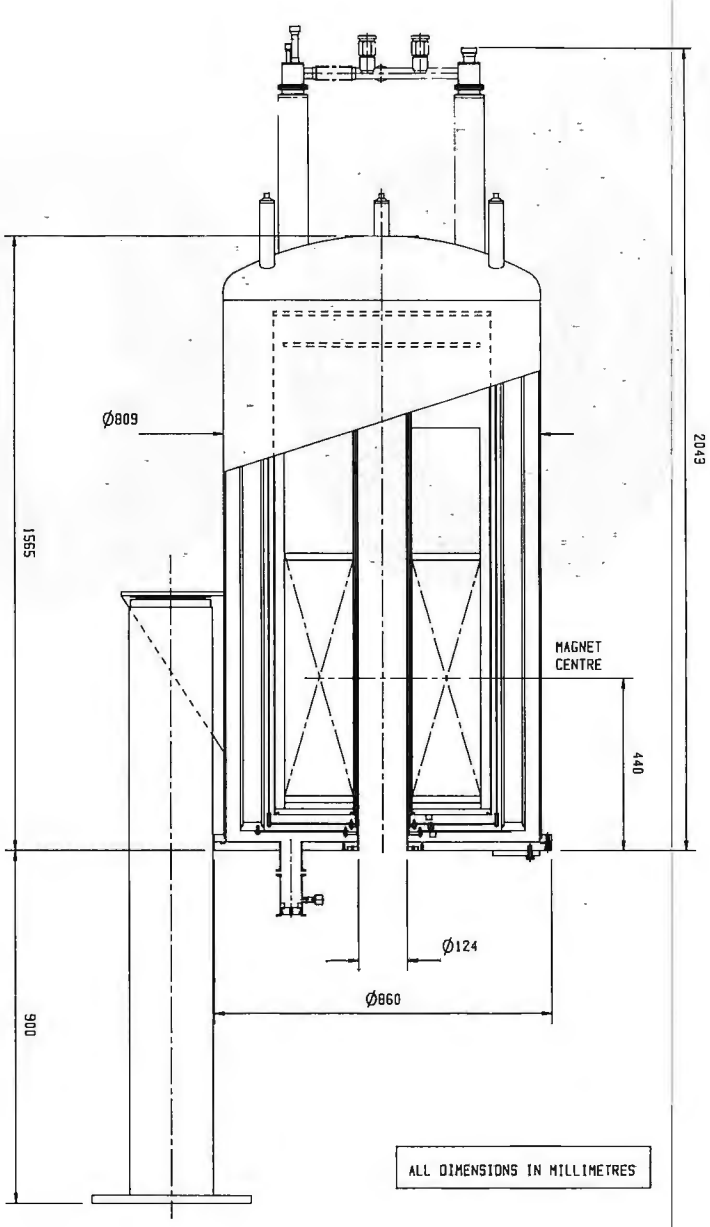

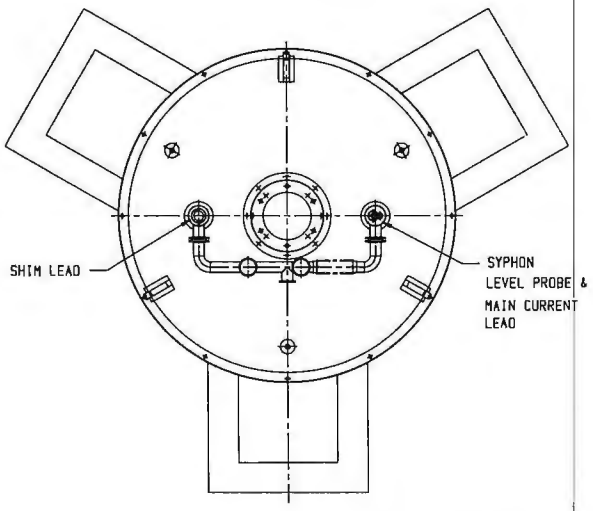

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The Florida State University
Tallahassee, Florida 32306-3006
FAX: 904-644-8281
Department of Chemistry

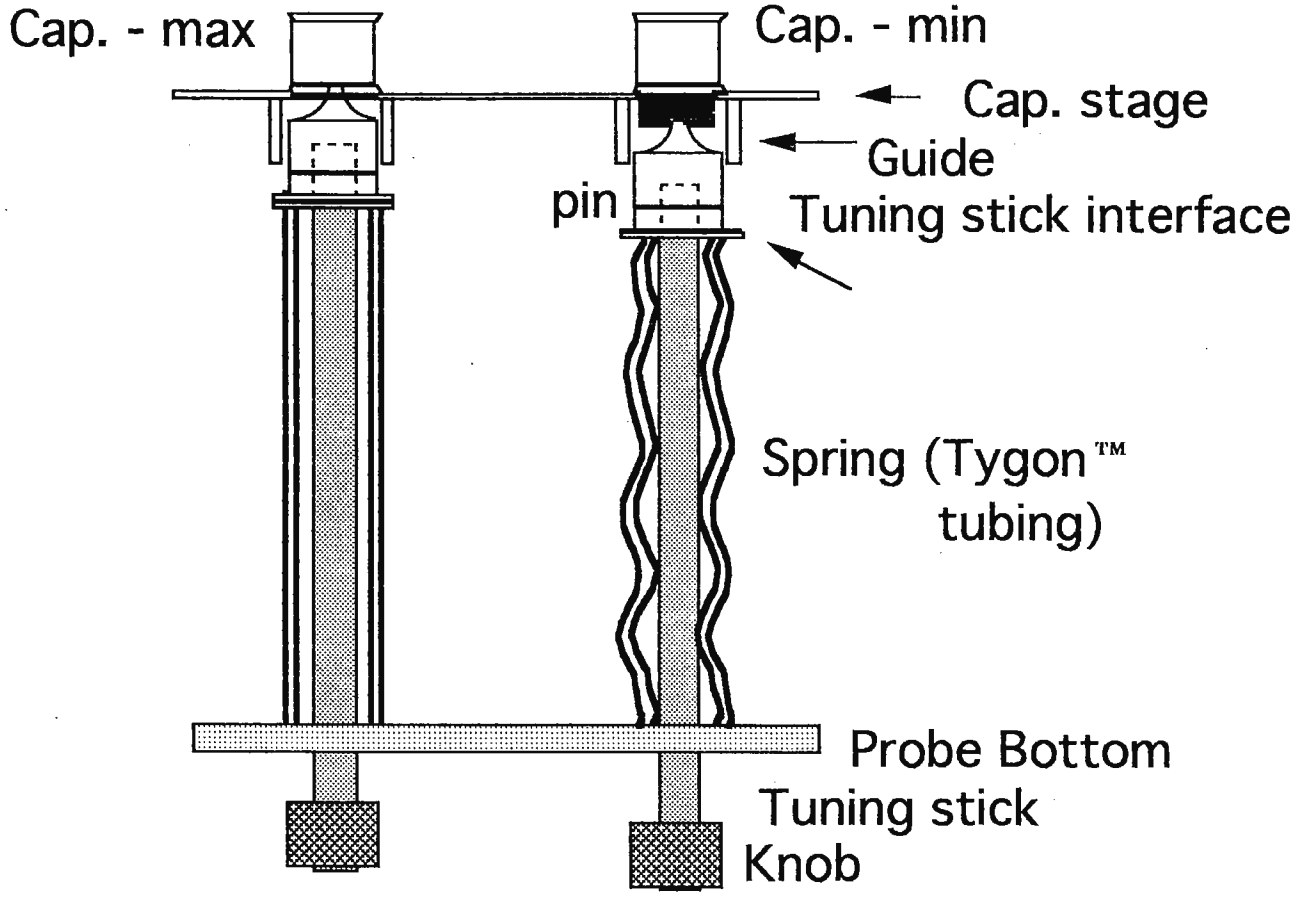
July 28, 1994
(received 8/1/94)

Dear Barry:

In building probes for NMR spectroscopy, we frequently have found that tuning capacitors have pistons whose tuning shafts travel in and out of the capacitor body. A tuning rod or shaft is incorporated to remotely tune these capacitors from outside the magnet with the use of non magnetic springs for flexible tensioning. Since obtaining small quantities of non-magnetic (copper-beryllium) springs proper for this task is not easily done, we have found a substitute "compression spring" which is non-magnetic and can be "cut to fit".

In the last solid state probes that we have built, tuning rods have been constructed with 3/16" diameter fiberglass or phenolic rod with 1/4" ID Tygon™ tubing slipped over its entire length. Slip washers at the top end, a suitable brass coupling to the capacitor and a knob complete the assembly. The compression of the tubing as the capacitor tuning shaft gets longer provides the necessary tension throughout the tuning range. For smaller probes or with less space available, smaller combinations of this arrangement should work equally as well.

R. Rosanske
nmrlab@chem.fsu.edu



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

July 26, 1994
(received 7/29/94)

Dear Barry,

MR MICROIMAGING OF AN AMPHIBIAN OOCYTE DURING MEIOTIC DIVISION

The cell nucleus is generally considered to be permeable to water and ions. Aguayo et al. (Nature **322**, 190, 1986) used MR to image *Xenopus* oocytes, and assigned a bright spot in the image to the cell nucleus. The differences in image intensity between the cytoplasm and the nucleus were proposed to result from proton density variations and/or differences in relaxation times. We have extended this study using *Rana pipiens* oocytes and have utilized both MR microimaging and measurement of relaxation times (T_1, T_2) for intracellular water to examine the hydration state of the nucleoplasm and cytoplasm. We confirmed the assignment of the bright spot to the cell nucleus both by demonstrating its disappearance in enucleated eggs and by following nuclear breakdown during meiotic division. The ovarian oocyte is arrested in prophase I, and can be induced to resume the first meiotic division by addition of progesterone. Following stimulation with progesterone, the oocyte resumes the first meiotic division, the nucleus rises to the surface of the animal hemisphere and the nuclear membrane undergoes fragmentation. These changes could be followed by MR microimaging.

Because of the large size of the oocyte and the slowness of the diffusion process, the exchange of intracellular water with extracellular water and between cytoplasm and intracellular compartments is slow on the NMR time scale. Figure 1 shows oocyte images obtained using a 0.5 mm slice thickness with in-plane resolution of 80 μm. The upper panel in Fig. 1 shows the contrast between cytoplasmic and nucleoplasmic compartments of a prophase amphibian oocyte. A τ_e (spin echo time) of 18 msec and τ_r (recovery time) of 500 ms were used to obtain this image. The bright field surrounding the oocyte represents the extracellular drop of Ringer's solution and the intracellular bright sphere corresponds to the oocyte nucleus. A much darker area is also apparent in the yolk platelet-rich region of the vegetal hemisphere. We found that neither shortening τ_e to 9 ms nor lengthening τ_r to 2 s reduced the observed contrast. The contrast between the nucleus and the cytoplasm must therefore arise in part from a difference in the water proton density between the nucleus and the cytoplasm, although T_2 differences may contribute to the contrast as well. The oocyte can be manually enucleated by making a small slit in the animal hemisphere and pressing the sides of the oocyte with forceps. The intact nucleus

is extruded from the oocyte, the plasma membrane reseals and the resting membrane potential reappears within minutes. A proton spin echo image of manually enucleated oocytes indicates a loss of the intracellular bright field .

Upon addition of 1 μM progesterone, the nucleus begins to rise to the surface of the animal hemisphere. By 8-10 hrs the nuclear membrane breaks down and the nuclear contents equilibrate with the oocyte cytoplasm. Fig. 1 also depicts proton spin echo images of oocytes with nuclei at the surface of the animal hemisphere (middle panel) and following fragmentation of the nuclear membrane (bottom panel). The middle panel illustrates the physiological state in which the nucleus has risen to underlie the cortex of the animal hemisphere. The image portrays three oocytes in the field of view and the nucleus can be clearly seen in two of the three oocytes. In contrast, the bottom panel shows that no bright field is present within the oocyte following disappearance of the nuclear membrane.

Fig. 2 illustrates a multiexponential fit of the semilog plot of an inversion recovery T_1 data from an oocyte suspension. The T_1 magnetization recovery curves of follicular oocytes can be fitted to a sum of three exponentials, representing the extracellular (T_1 = 2.9 s), cytoplasmic (T_1 = .8 s) and the nuclear (T_1 =1.6 s) water compartments. The smallest water compartment (approx. 10%) disappeared 8-10 h after the addition of progesterone to the medium. This corresponds to the time of nuclear breakdown (see Fig 1) and demonstrates that this compartment is the nucleus.

Our data demonstrate that the nucleus is much more hydrated than the cytoplasm in the prophase *Rana pipiens* oocyte. Furthermore, the spatial variation in cytoplasmic image intensity indicates that there are graded variations in the hydration state throughout the cytoplasmic compartment, most notably between the yolk platelet-rich region and the rest of the cytoplasm (top panel, Figure 1). The MR images are consistent with the low cytoplasmic water content and the relatively anhydrous nature of the yolk platelets. Microdissection of cytoplasm and nucleoplasm from frozen oocytes indicated the water content of the nucleus to be about 80% (gm H_2O per ml nucleus), whereas that of the cytoplasm was only about 50%. The difference in hydration state between oocyte nucleoplasm and cytoplasm appears to be largely due to a Donnan-type effect in which large impermeant anions and their counter ions within the nucleus produce a nearly two-fold increase in nuclear water content.

Sincerely,

Gene Morrill | Adele B. Kostellow | Raj K. Gupta

Figure 1

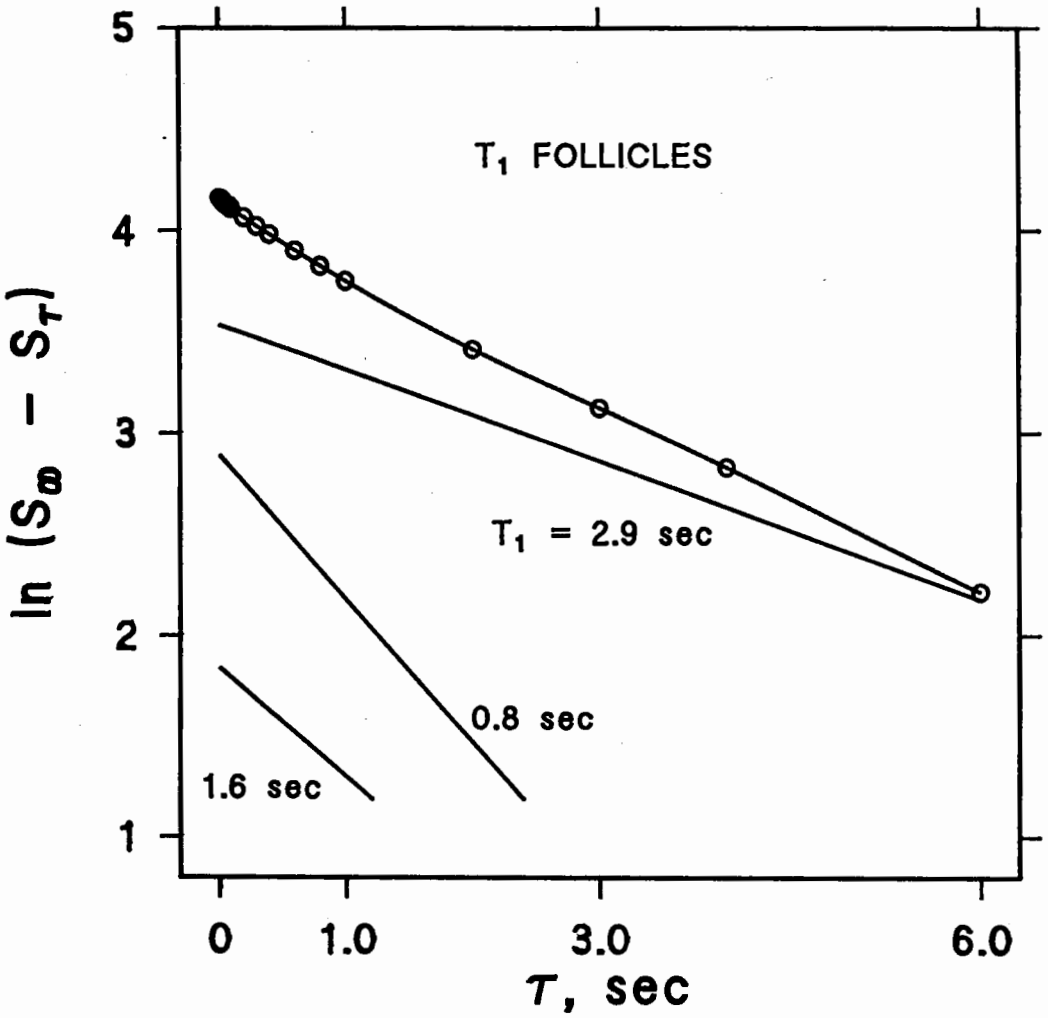

Figure 2

FORTHCOMING NMR MEETINGS, *Continued from page 1.*

12th International Meeting on NMR Spectroscopy, Sponsored by the Royal Society of Chemistry, Manchester, England, **July 2 - 7, 1995**; Contact: Dr. J. F. Gibson or Ms. G. B. Howlett - See TAMU NMR Newsletter 415, 5; Phone: (44-71) 437-8656; Fax: (44-71) 437-8883.

ISMAR 1995, Sydney, NSW, Australia, **July 16-21, 1995**; Contact: Dr. Les. Field, Dept. of Organic Chemistry, Univ. of Sydney, Sydney, NSW 2006, Australia. Phone: +61-2-692-2060; Fax: +61-2-692-3329; Email: ismar-95@biochem.su.oz.au Also, see TAMU NMR Newsletter 419, 26.

NMR Symposium at the 37th Rocky Mountain Conference on Analytical Chemistry, Denver Colorado, **July 24-27, 1995**; Contact: Dr. Alexander J. Vega, DuPont Central Research and Development, P.O. Box 80356, Wilmington, DE 19880-0356; Tel. (302) 695-2404; Fax: (302) 695-1664; e-mail: vego@esvax.dnet.dupont.com. See TAMU NMR Newsletter 432, 34.

Additional listings of meetings, etc., are invited.

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Deadline Dates

No. 434 (November)	21 Oct. 1994
No. 435 (December)	18 Nov. 1994
No. 436 (January)	16 Dec. 1994
No. 437 (Feb.)	27 January.1995

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