THE NMR NEWSLETTER

No. 486

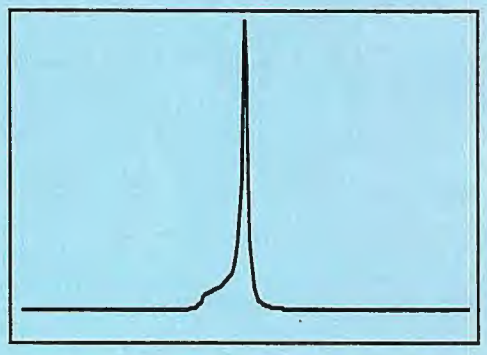
March 1999

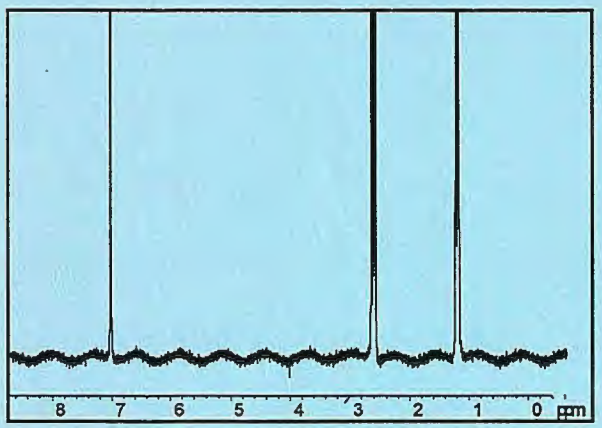
EDUCATIONAL SOFTWARE

SHIMMING SIMULATION AND VIRTUAL SPECTROMETER

now included in all NUTS versions, including ***free***, downloadable demo copies

Shimming Simulation teaches the student how to correct distorted lineshapes. The spectrum at right results from a combination of Z2 and Z3.





The **Virtual Spectrometer** simulates FT-NMR data acquisition, including the effects of common errors such as folding or too high gain setting, shown at left

---

NUTS Lite **$100** Basic 1D processing including:
Import filters for all major data formats, FT, phasing, baseline correction, integration, peak picking, cursor readout of chemical shifts and couplings, inset plots, structures on plots, macros for automated processing, shimming, virtual spectrometer, printing and copying. Does not include support or upgrades.

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NUTS-2D **$750** All 1D features above plus 2D processing and display, including:
Automated processing with macros, stacked plots, projections of all or part of the spectrum, interactive 2D peak picking and labeling, high resolution spectra on edges of contour plots, grid lines on contour plots, user control of contour levels and colors.

NUTS Professional **$1,000** All 1D and 2D features above, plus **Arrayed Mode**
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---

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FORTHCOMING NMR MEETINGS

Spin Choreography - a symposium in appreciation of Ray Freeman, Cambridge, England, **April 8-11, 1999**; web site: http://mchsg4.ch.man.ac.uk/mcmr/RF.html; fax: c/o M.H.Levitt +46-8-15 2187; email: mhl@physc.su.se.

Seventh Scientific Meeting and Exhibition of the Intl. Soc. for Magnetic Resonance in Medicine (ISMRM), Philadelphia, PA, **May 22 - 28, 1999**; Contact: International Society for Magnetic Resonance in Medicine, 2118 Milvia St., Suite 201, Berkeley, CA 94704.

International School of Structural Biology and Magnetic Resonance, 4th Course: Dynamics, Structure and Function of Biological Macromolecules; Erice, Sicily, Italy; **May 25-June 5, 1999**; Contact: Ms. Robin Holbrook, Stanford Magnetic Resonance Laboratory, Stanford University, Stanford, CA 94305-5055; (650) 723-6270; Fax: (650) 723-2253; Email: reh@stanford.edu. See Newsletter 483, 8.

Royal Society of Chemistry: 14th International Meeting on NMR Spectroscpy, Edinburgh, Scotland, **June 27 - July 3, 1999**; Contact: '99NMR14' c/o Mrs. Paula Whelan, The Royal Society of Chemistry, Burlingtom House, London W1V 0BN, England; +44 0171 440 3316; Email: conferences@rsc.org\

SMASH No. 1 (*S*mall *M*olecules *A*re *S*till *H*ot), Argonne, IL, **August 15-18, 1999**; Contact: Ms. Karen McCune , (mccune_karen_a@lilly.com, 317-276-9783) or S. R. Maple (maple_steven_r@lilly.com) or G.E.Martin (gary.e.martin@am.pnu.com) or A. G. Swanson (alistair_swanson@sandwich.pfizer.com. See Newsletter 484, 29

"Applications of NMR to Complex Systems", Symposium at the American Chemical Society Meeting, New Orleans, LA, **August 22-26, 1999**; Contact: R. E. Botto, Symposium Chair, Chemistry Division, Argonne National Laboratory, 9700 South Cass Ave., Argonne, IL 60439; 630-252-3524; Fax: 630-252-9288; E-mail: robert_botto@qmgate.anl.gov

41st ENC (Experimental NMR Conference), Asilomar Conference Center, Pacific Grove, CA, **April 9-14, 2000**; Contact: ENC, 1201 Don Diego Avenue, Santa Fe, NM 87505; (505) 989-4573; Fax: (505) 989-1073; E-mail: enc@enc-conference.org.

Additional listings of meetings, etc., are invited.

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Dr. B.L. Shapiro
NMR Newsletter

Date: 15th February 1999
(received 2/22/99)

Dear Barry,

Magic Angle orientation of a non-mesogenic solute!

We have been working on various quaternary ammonium halides with three long (octadecyl) chains, which exhibit complex thermotropic liquid crystalline behaviour depending on the length and structure of the fourth substituent on nitrogen (1). We have studied in detail, proton and deuterium NMR spectra of *N*-methyl-*N,N,N*-trioctadecylammonium iodide in the presence of non-mesogenic solutes (2). These compounds, due to their very low order parameters, are shown to have potential applications to structure determination. We have recently been looking at the NMR spectra of a new thermotropic mesogen, ethyltrioctadecylphosphonium iodide. We observe that this compound, similar to aforementioned quaternary ammonium compound, exhibits a low order parameter. NMR studies also indicate that the average orientation of the HH dipolar vector of a non-mesogenic solute, CH_2Cl_2 dissolved in this mesogen passes through the "Magic Angle" as the temperature is varied (Fig. 1). The low order parameter and the possibility of controlling the orientation have to be further explored as the phenomenon, if general, may possibly lead to some novel applications and would have implications in structural studies of the dissolved molecules. Further studies on the mesogen and other solutes are in progress.

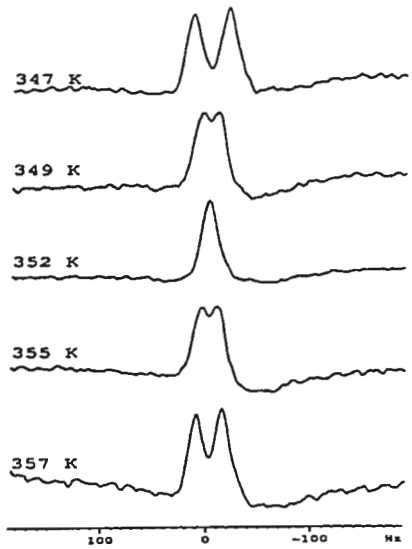

Fig.1 ^1H NMR spectra of CH_2Cl_2 dissolved in the mixture of a thermotropic mesogen, ethyltrioctadecylphosphonium iodide. The H-H dipolar vector orients at "Magic Angle" at 352K as observed by the absence of H-H dipolar splitting.

(1) L. Lu, N. Sharma, G.A. Naganagowda, C.L. Khetrapal and R.G. Weiss, Liquid Cryst. 22(1), 23 (1997).
(2) L. Lu, G. A. Naganagowda, N. Suryaprakash, C. L. Khetrapal and R. G. Weiss, Liquid Cryst. 25(3), 295 (1998).

(S.Vivekanandan) (G.A. Naganagowda) (K.V. Ramanathan)

(C.L. Khetrapal) (R.G. Weiss)

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UNIVERSITY OF CAMBRIDGE
DEPARTMENT OF CHEMISTRY
Cambridge CB2 1EW
United Kingdom

Professor Barry Shapiro,
966 Elsinore Court,
Palo Alto,
CA 94303

27 January 99
(received 2/4/99)

"Three Qubits and a Span*"

Dear Barry,

There is considerable academic interest in the prospect of building a quantum computer using a high resolution NMR spin system. In principle a quantum computer should outperform classical computers in solving certain "hard" computational problems, such as finding the prime factors of a large number. The fact that this could break the standard encryption code used by most banks scarcely concerns us here; *Honi soit qui mal y pense.*

There is a long, long way to go before a practical NMR computer can be implemented with a sufficient number of quantum bits (qubits), but it's fun to try. The ground rules for quantum computers are not those to which NMR spectroscopists are accustomed. We cannot choose a "convenient" initial condition such as Boltzmann equilibrium; a quantum logic gate must be able to deal with an *arbitrary* initial state. It must be able to manipulate one or two chosen spins while returning all coupled neighbours (the "spectators") to their initial condition. Since, in a homonuclear spin system, we are talking about frequency-selective manipulations which take many milliseconds to implement, ensuring that spectators remain inactive during this time is not trivial.

One very useful quantum manipulation is the controlled-not (exclusive-or) gate which has the truth table

$$\alpha\alpha \rightarrow \alpha\alpha$$
$$\alpha\beta \rightarrow \alpha\beta$$
$$\beta\alpha \rightarrow \beta\beta$$
$$\beta\beta \rightarrow \beta\alpha$$

The second spin is flipped if the first spin is in state β but not if it is in state α. The key step in the implementation of this gate is the homonuclear variant of "INEPT" using band-selective π pulses. For a three-spin (RIS) system the pulse sequence of Figure 1 can be used, where the soft pulses are written as ellipses. Spin R is the spectator and returns to its initial state, whereas both I and S acquire phase shifts of $\pm 8\pi J_{IS}\tau$, set equal to $\pm\pi/2$ to achieve the required antiphase configuration of proton vectors. The phase evolution diagram for spin I is shown.

The resulting spectra are shown in Figure 2. (No prize for guessing our favorite molecule.) Multiplet R is unchanged, whereas multiplets I and S show dispersion signals, antiphase with respect to J_{IS}. An overall $\pi/2$ phase shift then creates antiphase absorption.

My accomplices in this work were Hervé Barjat and Noah Linden; a more comprehensive account has been submitted to *Chemical Physics Letters.*

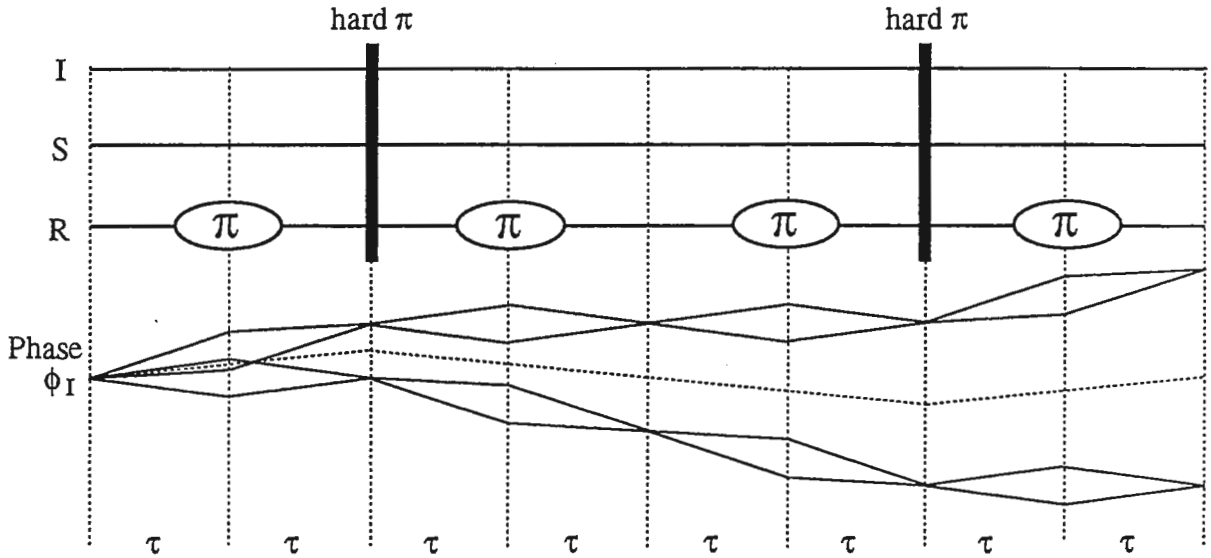
Kindest regards

Ray

Ray Freeman

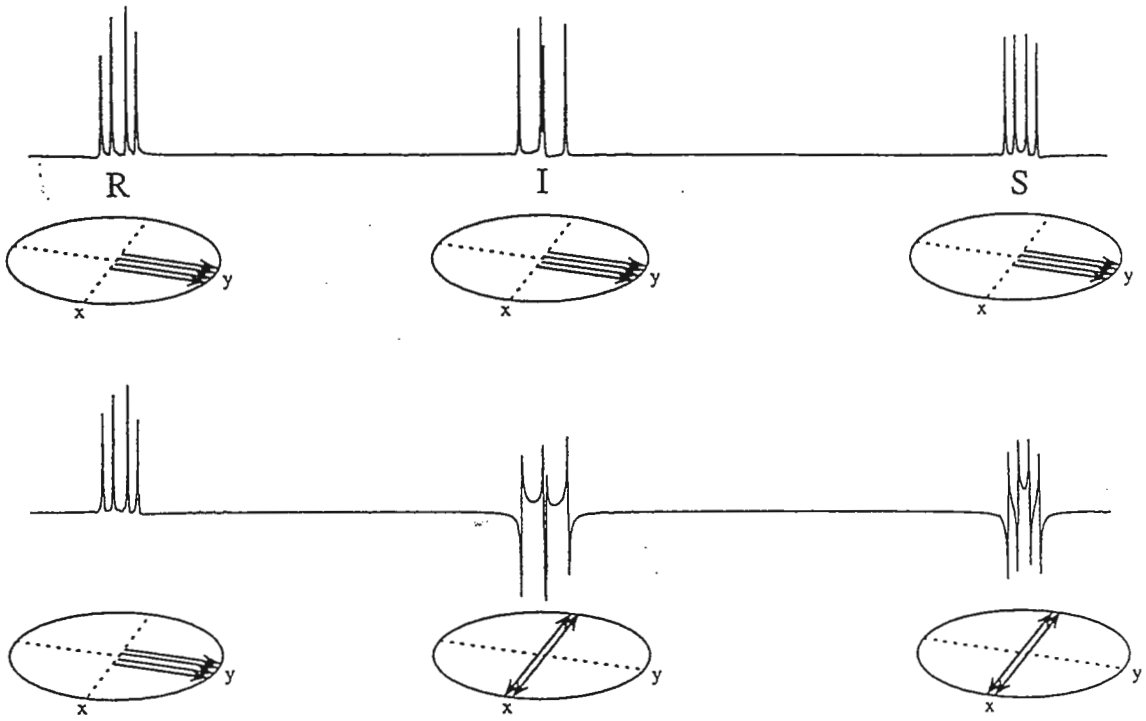
*Slight misquotation re vital statistics of Goliath of Gath, 1 Samuel 17.4

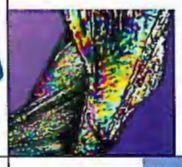
Figure 1. Pulse sequence to prepare antiphase I and S magnetizations. The complexity arises from the need to detect only even-numbered spin echoes

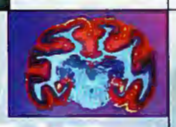


Phase evolution diagram for spin I

Figure 2 . Spectra showing the generation of antiphase dispersion signals for I and S



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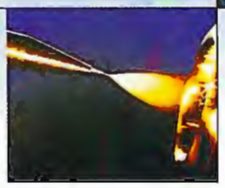

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04103 Leipzig

Prof. Dr. B. L. Shapiro
The NMR Newsletter
966 Elsinore Court
Palo Alto, CAL. 94303
USA

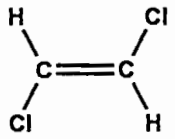
Leipzig, 09.02.99

(received 2/17/99)

Surprises on Symmetry

Dear Barry,

Sometimes we are asked to determine a C,C spin coupling constant in C_2-symmetric molecules, such as *trans*-1,2-dichloroethene **1**. Twelve years ago we have solved this problem by recording a proton coupled INADEQUATE (see *JMR* **1986**, *66*, 555) which revealed the expected AA'XX' spin system of the molecule from which the desired C,C spin coupling could be extracted. However the spectrum was missing two lines and this was explained that the A_2 subsystem of an AA'XX' spin system cannot generate double quantum coherences.



$^1J(C,H)$ 199.1
$^2J(C,H) < 0.2$
$^3J(H,H)$ 12.1
$^1J(C,C)$ 91.9

Asked recently again to perform such a measurement for a compound, which was only available in minor quantities the approach mentioned above was clearly inadequate. A proton INADEQUATE gives predominantly only the signals of the two AA'X spin systems, and the 100 times less sensitive (but desired) lines of the AA'XX' spin system are hidden. We therefore turned to the elegant gradient supported 2QHMBC technique recently communicated by Sørensen et al. (*JMR* **1997**, *124*, 245). We converted their pulse sequence to 1D and left everything off which was not necessary in our case; see Fig. 1. The gradients directly select one proton and two carbon atoms. The resulting spectrum (see Fig. 2) is now missing 4 lines; thus, one of the two AX subspectra of the AA'XX' spin system is missing and it is a very nice spectroscopic exercise to think about this. Fortunately the desired C,C spin coupling constant can be safely extracted manually (see Emsley, Feeney, Sutcliffe, p 392f.) or by spin simulation. We could reproduce the experimental results with the Bruker pulse simulation program NMRSIM.

Sincerely yours

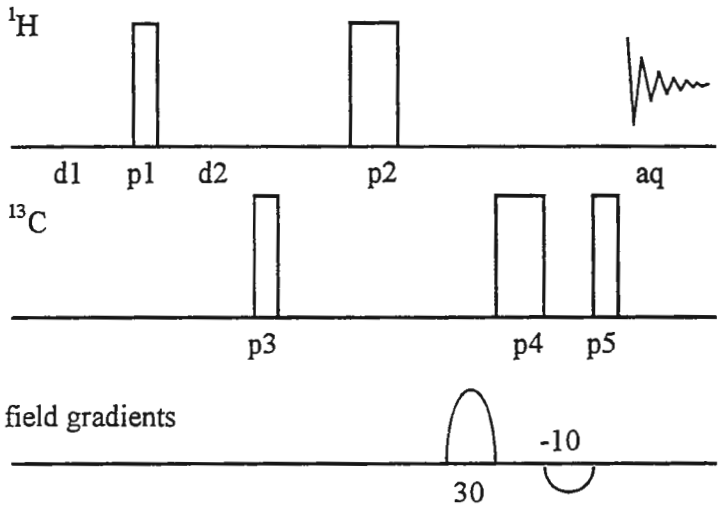

^{1}H
d1
p1
d2
p2
aq
^{13}C
p3
p4
p5
field gradients
30
-10

Fig. 1

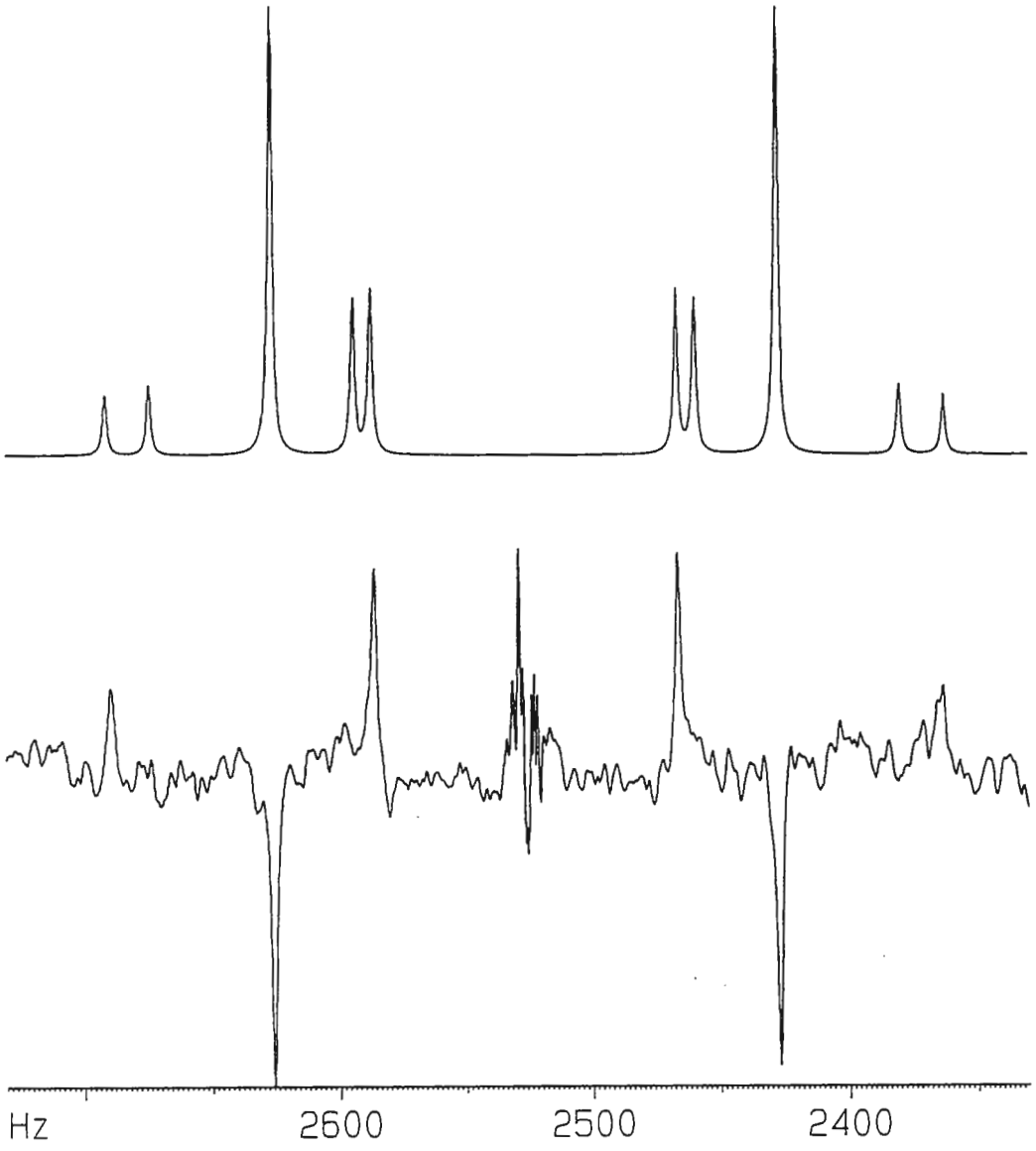
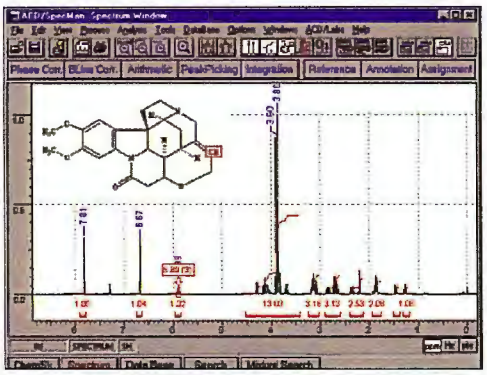

Hz
2600
2500
2400

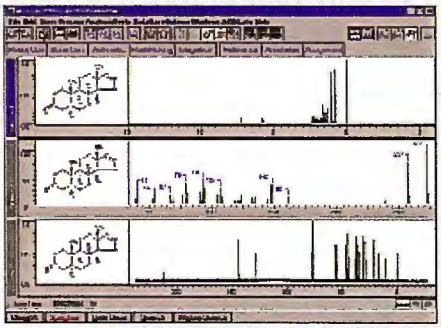
Fig. 2

ACD/NMR Manager

Take raw NMR data directly from the spectrometer and process it using a wide array of tools!



Intuitive interface for all kinds of processing



Tile feature in Spectrum Window lets you view several spectra at ones

- Import the major FID and FT formats, including Varian, Bruker, JEOL, Chemagnetics, WinNMR, Nuts, Lybrics, JCAMP, etc.
- Use zero filling, weighting functions, Fourier Transform.
- Apply automated or manual phase and baseline correction, peak referencing.
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- Create macro programs for completely automated data processing.
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- **New!** - Five varieties of weighting functions.

Release 4.0 ENC '99

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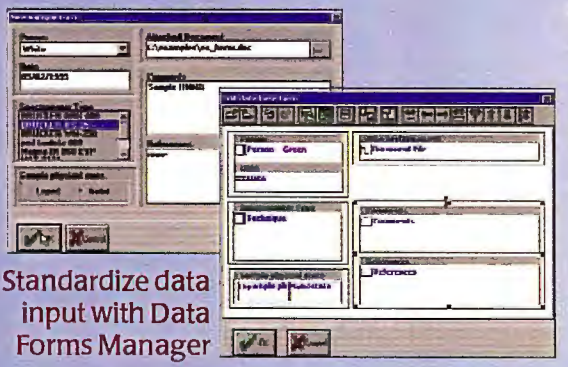
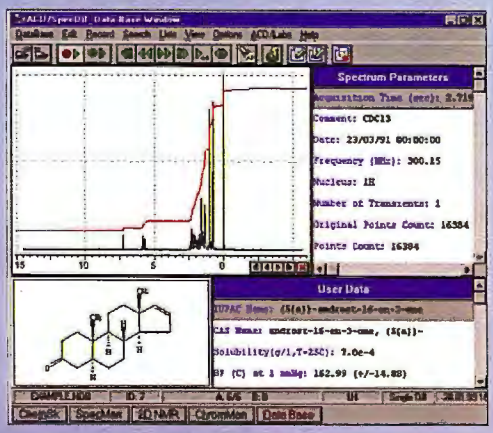


Standardize data input with Data Forms Manager



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  - molecular structure or sub-structure;
  - formula, molecular weight and user-defined fields; and any special fields corresponding to spectrum type (e.g. for mass spectra, the user-defined m/z values and losses from a molecular ion)
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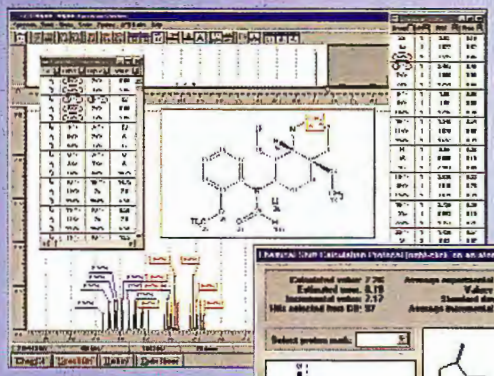
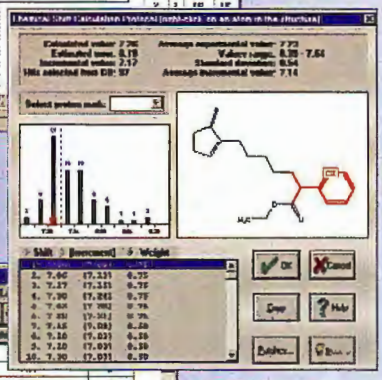
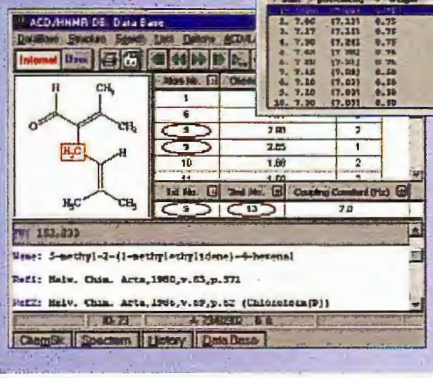


Predicted spectrum



Calculation Protocol Window



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- **NEW!** Include/Exclude exchangeable protons.
- **NEW!** Automatic transfer of predicted shifts to database record. Enter shifts without assignments to the database, too.
- **NEW!** Modifiable weighting in Calculation Protocol Window.
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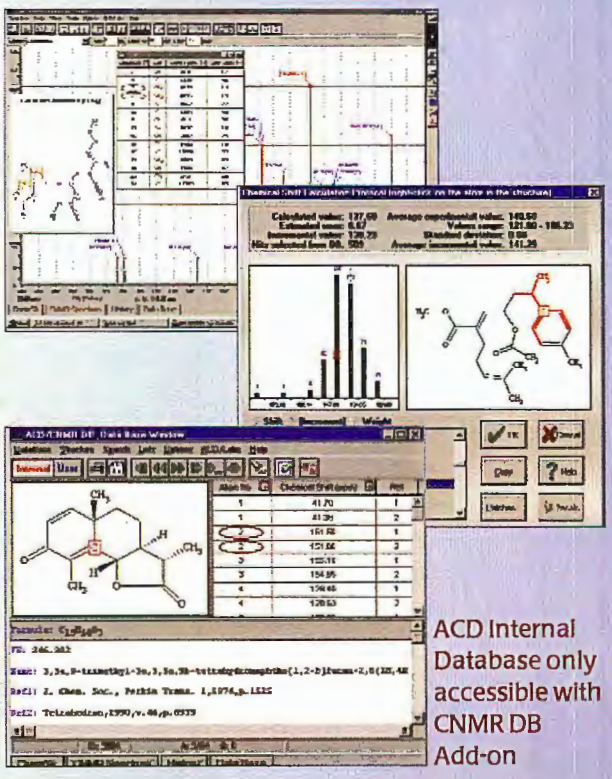
Release 4.0 EHC '99

ACD/CNMR

Calculate the carbon-13 NMR spectrum for any organic molecule to an accuracy of 3 ppm or better.



ACD Internal Database only accessible with CNMR DB Add-on

- Calculates spin-spin interactions, simulates off-resonance, DEPT, J-modulation and much more
- Prediction based on an internal data file with over 900 000 experimental chemical shifts.
- Self-training system lets you create your own database of chemical shifts, for improved prediction accuracy
- **NEW!** Data Forms Manager streamlines and standardizes record entry as you build your own databases.
- **NEW!** Include multiple user databases in system training
- **NEW!** Modifiable weighting in Calculation Protocol Window.
- **NEW!** Enter ^{13}C-X coupling constants, where X = ^{19}F, ^{31}P, etc.
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Southwestern Graduate School
of Biomedical Sciences
Southwestern Allied Health Sciences School

February 16, 1999
(received 2/22/99)

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

Beware of Memory Managers

Dear Barry,

In carrying out my research on ^{23}Na NMR in incompletely disordered aqueous systems such as biological tissue, I do simulations of FID's from various pulse sequences as well as analysis of relaxation time experiments. Some of these simulations are carried out with John Waugh's excellent program ANTIOPE. However, I know of no programs for common personal computers (i.e., IBM clones) which can do most of my simulations. Consequently, I write my own programs. I compile them with QuickBasic 4.5 under DOS. QuickBasic is versatile, easy to use, and has powerful graphics capabilities. The source code, which can be written in ASCII, is easily modified to fit new situations and is similar to the FORTRAN that I used a long time ago with the venerable punched cards.

To carry out an accurate simulation of NMR signals with random motions that cause random nuclear spin interactions, it may not be feasible to derive an analytical mathematical expression. Instead, it is necessary to repeat a calculation a large number of times with values of motional parameters chosen randomly by use of a random number generator and average the results. This can involve many tens of thousands, or even hundreds of thousands, of repetitive calculations. Even with modern PC's with fast chips (such as my Clone 400 MHz Pentium II PC), considerable time is required for the simulation. The same situation holds for the pedagogical QuickBasic programs for teaching relaxation phenomena that have been published recently in ***Concepts in Magnetic Resonance*** by Olivieri. Ditto for my CORVUS programs that are described in the July 1998 issue of the ***Journal of Magnetic Resonance***. While working with programs compiled with QuickBasic, I found a phenomenon that may interest others who carry out long calculations.

This phenomenon involves the effect of memory managers on the speed of calculations. The effect has been the same when PC's from many different manufacturers were tested. It is readily illustrated with the Savage Benchmark program that I copied (and embellished a little) from a magazine several years ago. It is the simple, computational-intensive program listed below:

```
10 REM            SAVAGE BENCHMARK
20 DEFDBL A
30  SCREEN 12
40  COLOR 14
50 LOCATE 10, 1: PRINT "The SAVAGE Benchmark Test"
60 COLOR 12
```

5801 Forest Park Rd. / Dallas, Texas 75235-9085 / (214)648-8186 Telefax (214)648-8181

```
70 LOCATE 12, 1: PRINT "Now computing for 649,351
     repetitions..."
80 Y0 = TIMER
90 A = 1!
100 FOR I = 1 TO 649350!
110 A = TAN(ATN(EXP(LOG(SQR(A * A))))) + 1!
120 NEXT I
130 Y1 = TIMER
140  COLOR 10
150 LOCATE 14, 1: PRINT "Numerical Result ="; A
160 COLOR 11
170 LOCATE 16, 1: PRINT "Seconds Elapsed ="; Y1 - Y0
180 COLOR 7
190 DO
200 LOOP WHILE INKEY$ = ""
210 END
```

The number of repetitions was chosen so that the test would take 10.0 seconds on a 266 MHz Pentium II computer under the condition of greatest speed.

I found that the speed of this test (and other programs compiled under QuickBasic) depends critically on installed memory managers. The time of 10.0 seconds was obtained with the Microsoft memory manager **EMM386.EXE** ***not*** installed. If **EMM386.EXE** is installed, 27 seconds is required on the above computer. This 2.7-fold longer time is not insignificant when doing simulations that may take an hour or so.

Windows 3.1, Windows 95, and, presumably, Windows 98 automatically loads **EMM386.EXE** on boot-up. Thus, such programs run slow from the **DOS PROMPT** of all these windows. The same factor of 2.7 holds for running under the **DOS PROMPT** of these windows. The same factor seems to hold for 386, 486, Classic Pentiums, and Pentium II's. I have not tested on a Pentium with MMX. The MMX feature on chips may be an additional factor that influences the speed of QuickBasic.

The answer seems to be to use a computer boot-up that does not load **EMM386.EXE** and run the programs directly under DOS. This is easy to do for computers that run Windows 3.1. Just set up a multiple-boot menu that contains an option in which the **AUTOEXEC.BAT** file does not load **EMM386.EXE**. In some computers/set-ups under Windows 95, **EMM386.EXE** can be disabled under the **DOS PROMPT** by using the DOS 7.0 command **EMM386 OFF** and then running the program in DOS. This does not always work. With my two Pentium II computers, with Windows 95, version 2.0 and version 2.5 (the ultimate), I worked with the technician at the manufacturer (Clone Computer Corp., in Dallas) to get an appropriate double-boot set up. I did this because I am relatively computer-illiterate and I wanted to make sure that I could run the programs fast.

EMM386.EXE is not the only memory manager that slows QuickBasic programs. Using a 90 MHz Classic Pentium with several different memory managers, the degree of slowdown was found to depend on the memory manager. All of the other different memory managers ran the Savage Benchmark even slower than **EMM386.EXE**. With one, the slowdown factor was 10!

Even with no memory manager, the program running time depends on the CPU chip. Here are

the running times for the above Savage Benchmark program: Classic 166 MHz Pentium, 8 seconds; 266 MHz Pentium II, 10.0 seconds, and 400 MHz Pentium II, 6.36 seconds. Why did the 266 MHz Pentium II run the program ***slower*** than the 166 MHz Classic Pentium? The Clone technician thought that the MMX technology may actually slow down some operations.

The whole problem of running time of long simulations is even more complex than the examples that I gave above. Using a standard set of parameters, I ran John Waugh's program ANTIOPE to calculate powder patterns. The use of **EMM386.EXE** did also slow the running, but by a paltry several percent instead of the enormous factor of 2.7. Also, comparing run time on the 266 MHz Pentium II versus that on the 166 MHz Classic Pentium, the 266 MHz Pentium II ran 2.3 times ***faster***. This is even much faster than the ratio of clock speeds, which is 1.6. ANTIOPE is compiled under FORTRAN. Why is there such a great difference of performance between QuickBasic and FORTRAN?

I welcome advice, solutions, comments, etc. Just do not tell me to use C.

Question: does LINUX run QuickBasic programs? If so, does it run them faster than DOS with the same processor?

Best regards,

Don

Donald E. Woessner
Adjunct Assistant Professor of Radiology
dwoess@mednet.swmed.edu

UNIVERSITY OF MISSOURI-ROLLA
Missouri's Technological University

Frank D. Blum
Department of Chemistry
142 Schrenk Hall
Rolla, MO 65409-0010
573.341.4451 (or 4420)
fblum@umr.edu

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

(received 2/20/99)
Feb. 16, 1999

Deuterium Wide Line NMR of Acrylic Silane Coupling Agents in Composites

Dear Barry,

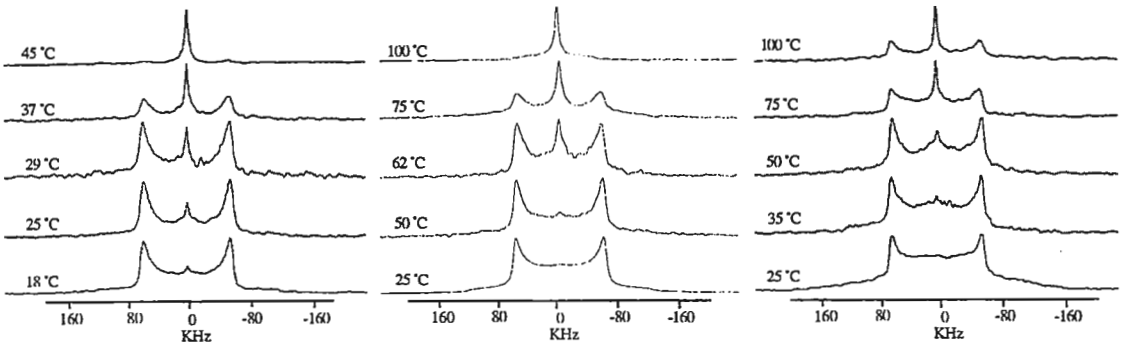
In order to study the influence of the inorganic matrix (or the organic polymer) on the dynamics of APMS-*d* (CH_2=CD-C(O)-O-CH_2-CH_2-CH_2-Si-$(OCH_3)_3$) at an interface using deuterium wide-line NMR, several composites were prepared. To prepare the organic-inorganic hybrid composite (OIC), first, APMS was polymerized with methyl acrylate (MA) and this was referred to as an APMS-MA. Then tetramethoxysilane was added to form an OIC and this composite was referred to as TMOS-APMS-MA. Another APMS sample was prepared with fumed silica. APMS-*d* was immobilized on the silica surface (of fumed silica) and then polymerized with MA. This was referred to as Silica-APMS-MA.

Deuterium wide-line spectra (below) of these samples showed a static quadrupole powder pattern at approximately room temperature. When the acrylic group of APMS was polymerized with methyl acrylate (MA), with or without an inorganic polymer, the motion of the segments having deuteron as a polymer backbone created a powder pattern at around room temperature. The powder pattern of APMS-MA had already started to become distorted at 18 °C. However, the initial temperature of collapse for the powder pattern of an OIC sample (TMOS-PMA-APMS) shifted to a higher temperature and the temperature range for collapse for the powder pattern broadened. A similar phenomenon occurred with a composite having fumed silica (Silica-PMA-APMS). The presence of a rigid inorganic polymer slowed the motion of the organic polymers and broadened the temperature range at which the Pake powder pattern collapsed.

Sincerely Yours,

Hyoryoon Jo

Frank D. Blum



APMS-MA TMOS-APMS-MA Silica-APMS-MA

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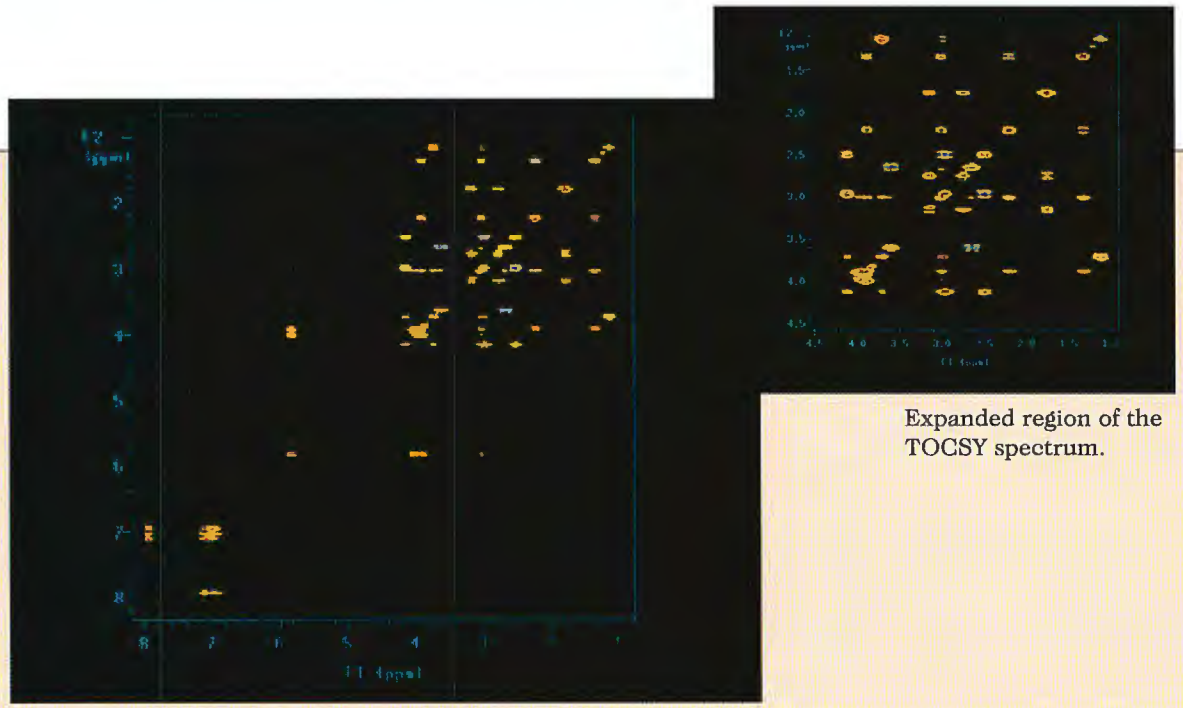
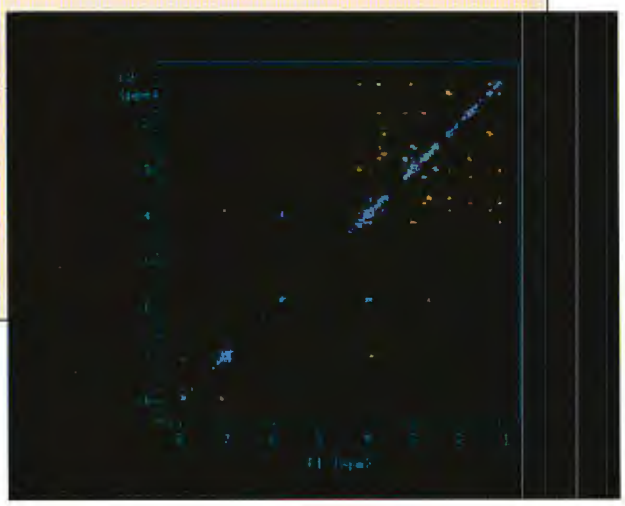


Expanded region of the TOCSY spectrum.

Spinlock experiments, such as this TOCSY spectrum of strychnine, are routinely performed with ***MERCURY***.



MERCURY features advanced experiment capability, as is demonstrated in this high-quality ROESY spectrum.

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Professor John C. Lindon
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Biomedical Sciences Building
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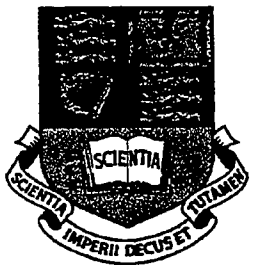
London SW7 2AZ

Tel: 44-(0)171-594-3194
Fax: 44-(0)171-594-3066
E-mail: j.lindon@ic.ac.uk



Imperial College
OF SCIENCE, TECHNOLOGY AND MEDICINE

2 February 1999
(RECEIVED 2/16/99)

Dr. B.L. Shapiro
The NMR Newletter
966 Elsinore Court
Palo Alto, CA 94303-3410
USA

Dear Barry,

Heating effects in high resolution ^{1}H MAS NMR spectra of tissues

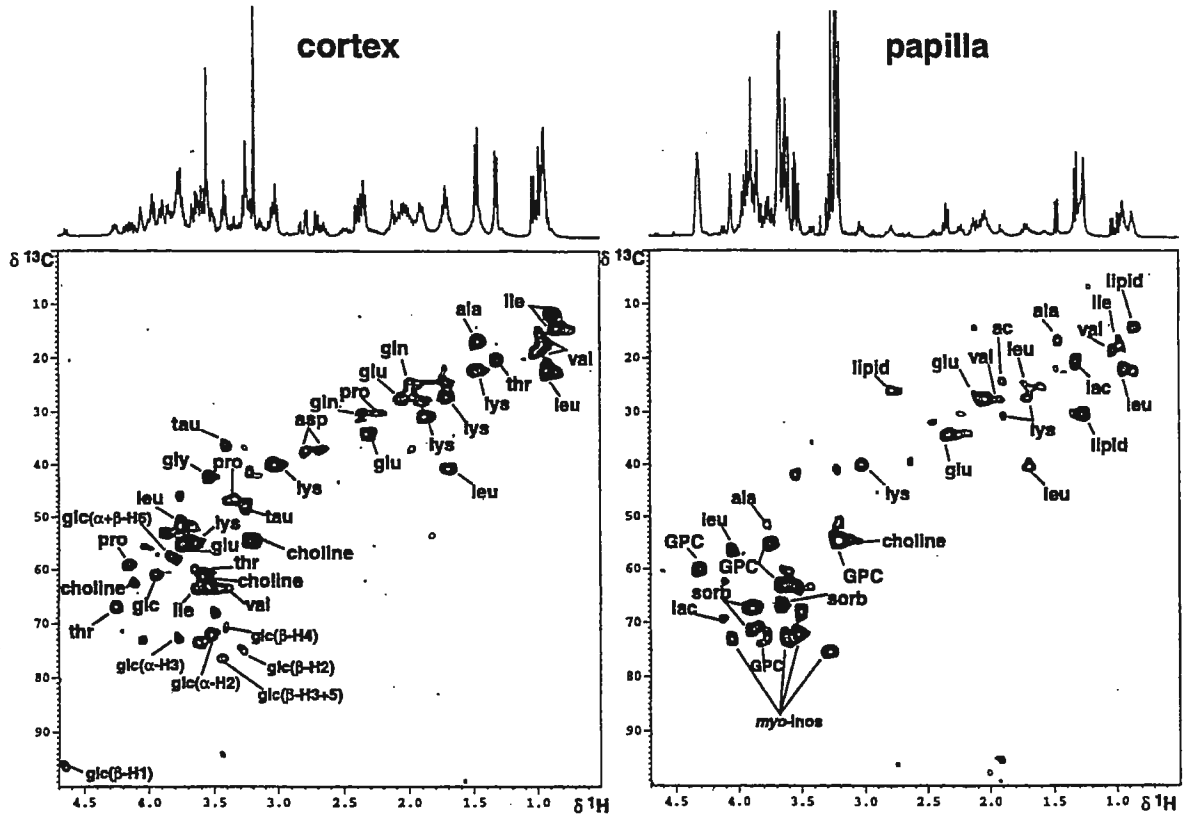
We have been measuring ^{1}H MAS NMR spectra of human and animal tissue and correlating the biochemical information seen with that obtainable from biofluids such as urine and blood plasma. We have made quite detailed examination of rat kidney outer and inner cortex, medulla and liver, plus various human tissues such as tumours from brain, kidney, ovary and prostate using samples as small as 10 mg (pin head size biopsies). The spectra are remarkably sharp and examples from rat kidney are shown below. Most of this work has been done at 400 or 500 MHz and spinning at around 4 kHz is sufficient to move the spinning sidebands out of the spectral region. However, sometimes it is necessary to spin faster to remove dipolar couplings from less mobile component such as some lipids and we have been up to around 12 kHz for this purpose. At these higher spinning speeds we suspected that frictional heating might occur and luckily we have an internal thermometer in the ^{1}H NMR spectra. We observed the effect of heating because when we are locked using D_2O, the ^{1}H spectrum appears to shift as the spinning speed is increased. Of course this is because the D_2O resonance is temperature dependent. We had earlier shown that the ^{1}H chemical shift in ppm between the resonance from the H1 proton of α-glucose and that of HDO can be fitted to a quadratic as a function of temperature (*NMR in Biomedicine*, 7, 243-247 (1994)).

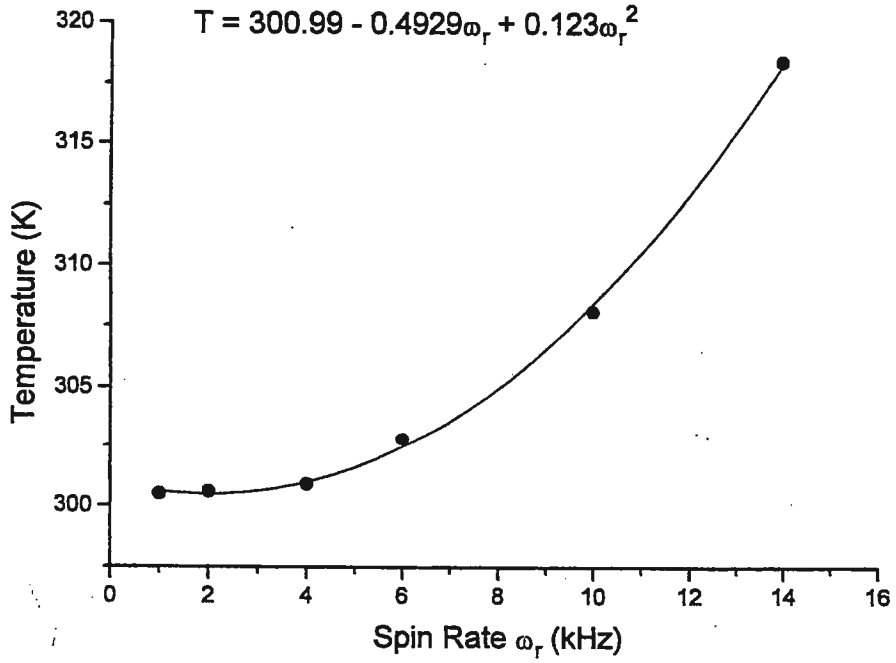
This gives us a means of measuring the temperature inside the tissue sample and shown below is a plot of temperature versus spinning speed using a sample of human prostate tissue. This can also be fitted to a quadratic in ω_r. Thus, keeping to around 4 kHz spinning speed should not cause any problems of heating. It is of course also possible to cool the NMR probe to minimise any effects. We have found that tissue spectra from kidney medulla are stable for several hours whilst significant changes occur in other tissues within about 1 hour, according to how metabolically active they are. Allowing an hour for measurement per tissue allows us to get a range of 1-D and 2-D spectra in this time. For example, shown

below is the ^{1}H-^{13}C HMQC MAS NMR spectrum of rat kidney cortex and papilla. We believe that the ^{1}H MAS NMR approach offers widespread possibilities for studies of tissue damage and disease.

Best wishes,

John Lindon Jeremy Nicholson Elaine Holmes





BICELLE PREPARATION

Buffer: An effective and convenient method for preparing bicelles makes use of a buffer solution containing 10mM phosphate buffer, pH 6.6, 3.0 mM sodium azide, 93% H_2O (HPLC-grade), 7% D_2O (99.9%). Below, this solution will simply be referred to as buffer.

Bicelle Formation: DMPC/DHPC stock solutions containing a total of 15% w/v (150mg lipid/ml) are prepared as follows:

Add buffer to the lyophilized lipid mixture

50mg lipid mixture, 280µg buffer

200mg lipid mixture, 1130µg buffer

Let the mixtures hydrate at room temperature (18-22°C) for several hours.

Lipid mixtures with a "q" of 2.8 - 3.0, the hydration is complete in 2 - 3 hours.

Lipid mixtures with a "q" of 3.25 - 3.5 require 24 hours for complete hydration.

Accelerated hydration (one hour) may be effected by heating any mixture to 40°C for 10 minutes and cycling to 18°C twice, then briefly vortexing.

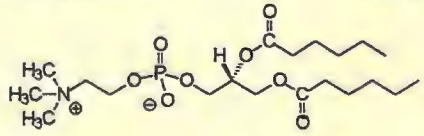
Protein-Bicelle Mix: Two volumes of protein solution are added to one volume of bicelle solution.

Tjandra, N., & Bax, A., "Direct Measurement of Distances and Angles in Biomolecules by NMR in a Dilute Liquid Crystalline Medium" *Science* (1997) **278**:1111-1113.

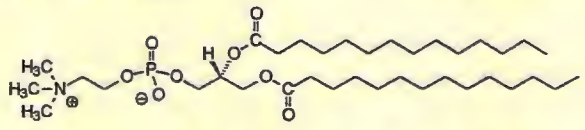
Ottiger, M., & Bax, A., "Characterization of Magnetically Oriented Phospholipid Micelles for Measurement of Dipolar Couplings in Macromolecules" *J. Biomol. NMR* (1998), in press.

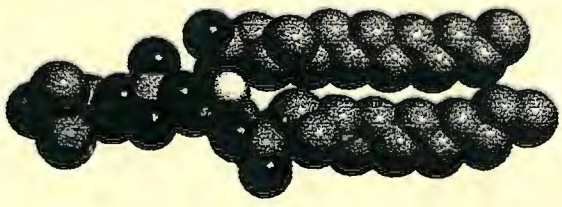
BICELLE FORMING LIPIDS

1,2-dihexanoyl-*sn*-glycero-3-phosphocholine (DHPC)
Cat No. 850305 **MW 453.51**



1,2-dimyristoyl-*sn*-glycero-3-phosphocholine (DMPC)
Cat No. 850345 **MW 677.95**





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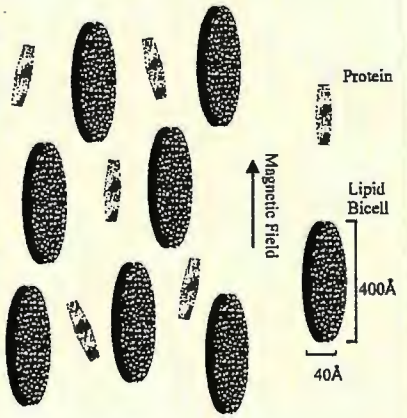

Two of the most common phospholipids used for bicelle formation are **1,2-Dimyristoyl-*sn*-Glycero-3-Phosphocholine (DMPC)** and **1,2-Dihexanoyl-*sn*-Glycero-3-Phosphocholine (DHPC)**. Preparation of defined mixtures of these lipids can be technically difficult and time consuming. Also, specialized equipment is required for handling the materials due to the hygroscopic nature of DHPC. To assist researchers in utilizing this technique, Avanti now offers these components in **pre-mixed units ready for hydration.** Just add buffer and protein solution and you can be ready to take measurements in less than 1 hour.

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3.0				790573
3.25				790574
3.5				790575

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DEPARTMENT OF THE NAVY
NAVAL RESEARCH LABORATORY
4555 0VERLOOK AVE SW
WASHINGTON DC 20375-5320

IN REPLY REFER TO:

February 12, 1999
(received 2/22/99)

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

Deuterium NMR and Residual Strain in a Natural Rubber Double Network

Dear Dr. Shapiro:

We have found evidence of a slight residual strain in a sample of a natural rubber double network (DN) using deuterium NMR. Double networks are cross-linked elastomers in which the cross-linking is performed in two separate steps. The first cross-linking is performed in the typical fashion in the absence of any applied stress. The second cross-linking step, however, is performed while the elastomer is stretched to about 400% elongation. When the cross-linking is complete and the elastomer is released from it's restraining device, it is found through birefringence that there is a residual strain existing in the elastomer. The degree of strain is slight; birefringence measurements indicate approximately a 3% residual strain.

We chose to examine these DNs using deuterium NMR of solvents swollen into the elastomer. This technique has been shown to be a simple yet powerful way of determining molecular structural order in strained elastomers (Samulski, E.T. *Polymer*, **1985**, 26, 177). Preliminary deuterium NMR experiments using benzene-d_6 as a swelling agent, however, showed no evidence of residual strain in these elastomers. We then examined a natural rubber single network with benzene-d_6, hexane-d_{14}, and chloroform-d as a function of uniaxially applied stress. We found that the different solvents had different degrees of sensitivity to molecular order and that chloroform was the most sensitive of these solvents to the applied stress in natural rubber. When we examined the DN swollen with chloroform we saw quadrupolar splitting in the unstressed sample. Figure 1 compares the results we obtained when examining the unstrained DN with benzene and with chloroform where the evidence of a residual strain is clearly evident using chloroform.

Deuterium NMR was then carried out on a uniaxially deformed double network. We observe a slight decrease of the splitting at first and then an increase in the splitting with an increase in applied stress (Figure 2). These results indicate that we are observing a residual compressive strain that is relieved with the application of an extension force. This result is also supported by birefringence observations.

This methods illustrates that with the proper selection of solvent, slight strains can be resolved.

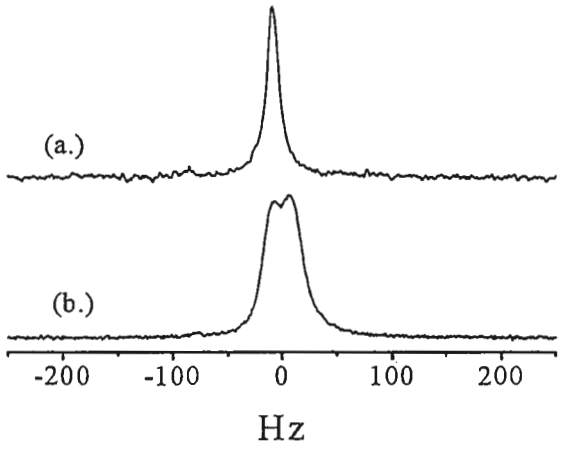

Figure 2 ^{2}H NMR spectra of (a.) Benzene-d_6 and (b.) Chloroform-d swollen into a natural rubber double network without applied stress.

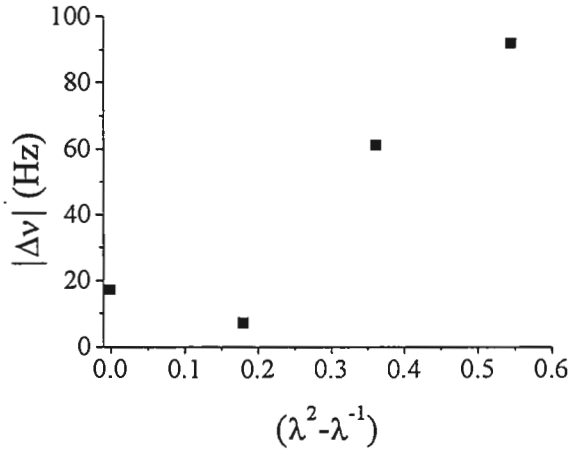

Figure 1 2H quadrupolar splittiing of the chloroform-d in a double network as a function of strain. (λ=extension ratio)

Sincerely,

Judith B. Cain
jbcain@ccf.nrl.navy.mil

Joel B. Miller
miller5@ccf.nrl.navy.mil

ZENECA

ZENECA Ag Products
1200 S. 47th Street
Richmond, CA 94804-0024
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Dr. B. L. Shapiro
The NMR Newsletter
966 Elsinore Court
Palo Alto, CA. 94303

Jan 26, 1999
(received 2/5/99)

LC/NMR of metabolites at 1 microgram level.

Dear Barry:

We recently acquired an HPLC accessory for our Varian Unity+ 500 NMR spectrometer. Because microgram quantities of materials are notoriously hard to purify for NMR analysis, we were anxious to utilize this new hyphenated technique to see how well it would work with metabolites. So, we borrowed a INUS βRAM from the metabolism department and hooked it up in place of the usual UV detector. The scintillation cell was a 15 μL silanized lithium glass solid cell. The LC probe is normally connected to the outlet of the UV detector with about 6 ft of 0.005" diameter PEEK tubing. However, INUS warns against using this small diameter tubing on the outlet of the βRAM due to the chance of bursting the cell if a particle of solid scintillant exits the cell and lodges in the tubing. They recommend 0.040" diameter tubing instead. However, we could not use tubing that large without suffering undo sample dilution in the long transfer line to the probe.

After some experimentation with alternate setups (such as splitting the flow between NMR probe and detector), we decided to retain the existing configuration and the 0.005" PEEK transfer line. We purchased a solid cell with a fairly high (1000 psi) burst pressure and installed a 2.0 micron filter at the outlet of the solid cell. In addition, just upstream of the βRAM, a 500 psi relief valve was teed into the inlet to the detector. The outlet of the valve leads to a small bottle to catch any sample.

The effort has been well worth while. Figure 1 shows the NMR spectrum of a unknown metabolite we analyzed. The metabolite is a glucoside. Figure 1a shows the NMR spectrum (from δ2.3 - 4.2) which was acquired in a 3 mm NMR tube on ~1 μgram of "purified" sample. The metabolite signals are completely obscured by the impurities. Figure 1b shows the same metabolite sample acquired by LC-NMR using a 150X2.1 mm C-18 column, 94/6 D_2O/CH_3CN mobile phase and flow rate of 0.2 ml/min. Although the

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5 mm	535-PP-7	500	7 in	0.015"	0.0005"	0.00025"	15.15	+100	12.85
5 mm	535-PP-8	500	8 in	0.015"	0.0005"	0.00025"	16.65	+100	14.15
5 mm	528-PP-7	400	7 in	0.015"	0.0010"	0.0005"	10.50	+100	9.25
5 mm	528-PP-8	400	8 in	0.015"	0.0010"	0.0005"	11.95	+100	10.15
5 mm	507-PP-7	360	7 in	0.015"	0.0020"	0.0010"	6.95	+100	5.90
5 mm	507-PP-8	360	8 in	0.015"	0.0020"	0.0010"	7.75	+100	6.60
5 mm	506-PP-7	100	7 in	0.015"	0.0020"	0.0020"	5.40	+100	4.80
5 mm	506-PP-8	100	8 in	0.015"	0.0020"	0.0020"	6.45	+100	5.45
5 mm	506-IM-7 New!	100	7 in	0.015"	0.0020"	0.0020"	3.85	+100	3.45
5 mm	506-IM-8 New!	100	8 in	0.015"	0.0020"	0.0020"	4.05	+100	3.65
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3 mm	327-PP-7	400	7 in	0.012"	0.0010"	0.0010"	10.20	+100	9.20
3 mm	327-PP-8	400	8 in	0.012"	0.0010"	0.0010"	11.65	+100	10.55
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5 mm	535-JY-7	500	7"	0.015"	0.0005"	0.00025"	79.85	+10	71.90
5 mm	535-JY-8	500	8"	0.015"	0.0005"	0.00025"	79.85	+10	71.90
5 mm	528-JY-7	400	7"	0.015"	0.0010"	0.0005"	75.70	+10	68.10
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spectrum is noisy, the glucose moiety is clearly observable. The anomeric proton is underneath the D_2O signal. This was confirmed with a TOCSY experiment.

We are quite excited about this technique and hope to expand its use into other problems.

Sincerely yours,

D. J. Bowler L. L. Chang B. C. Onisko

don.bowler@agna.zeneca.com lydia.chang@agna.zeneca.com

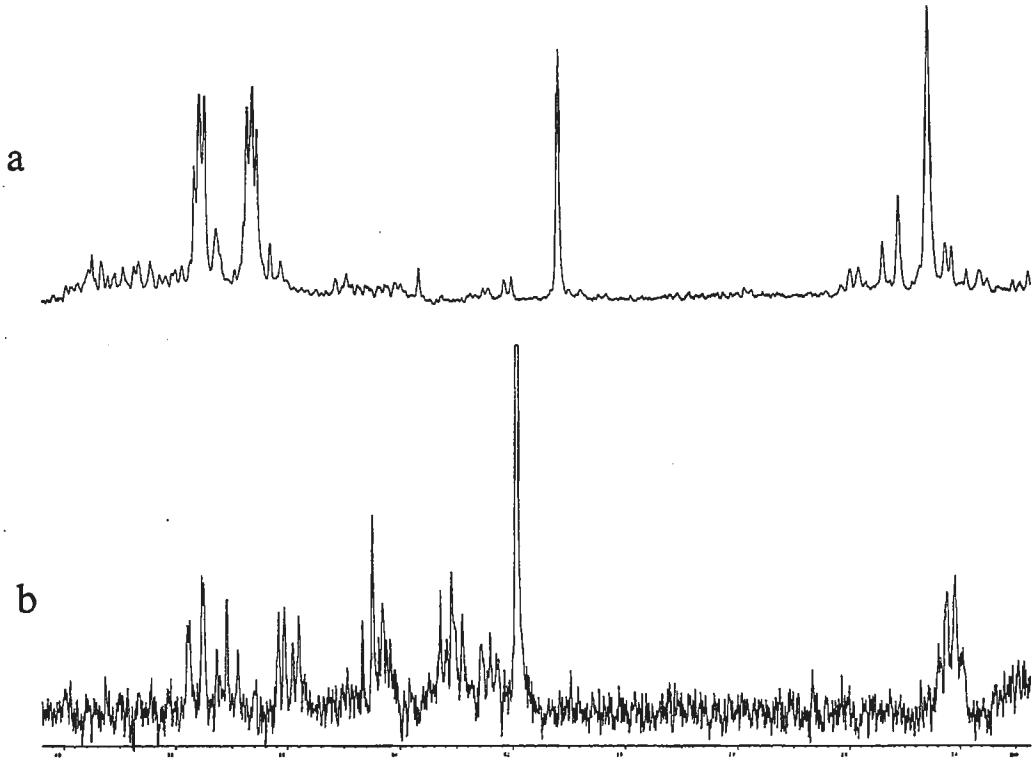

Figure 1. NMR spectrum of ~1 microgram of unknown metabolite. a)Spectrum acquired in D_2O using 3 mm NMR tube. b)Spectrum acquired in 94/6 D_2O/CH_3CN using stop-flow LC/NMR. Total acquisition time was about 2 hours.

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Timothy Saarinen

Timothy Saarinen, Ph.D.
Vice President, Sales & Marketing

CENTRAL RESEARCH & DEVELOPMENT
Experimental Station
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Wilmington, Delaware 19880-0328

Applications of PFG NMR Diffusion Measurements

(received 2/22/99)
February 16, 1999

Dear Barry,

We have recently been applying pulsed-field gradient (PFG) NMR to the measurement of self-diffusion coefficients of small molecules in a variety of polymer systems. Our initial efforts in this area were greatly aided by a collaboration with Prof. Cynthia Larive and Dr. Dimuthu Jayawickrama of the Department of Chemistry, University of Kansas, Lawrence KS. We find that the BPPLED pulse sequence (1) with a spoiler pulse in at least the LED period generally gives the most satisfactory results.

Our applications so far have fallen into one of two principal categories — those in which the specific value of the diffusion coefficient is of interest, and those in which a variation in the diffusion coefficient is used as a vehicle for separating components of complex mixtures. In the former case, the diffusion coefficient of a small molecule in a polymer melt may be of particular value to engineers involved in modeling a polymer process. These results are usually obtained at elevated temperature where, for materials of low or moderate viscosity, convection effects can increase the apparent measured diffusion coefficient, and for this reason we use the DSTE convection correction pulse sequence of Jerschow and Muller (2).

In the case of mixture analysis, it may not be necessary or even desirable to determine numerically the diffusion coefficient. In this case, we use PFG-NMR as a spectral editing tool based on the differential diffusional properties of the components of the mixture. Despite the very evident simplicity of this approach, the utility is still rather great. This approach is illustrated in Figure 1 for a mixture of 3 common polymer anti-oxidants (3). At higher gradient amplitudes, the resonances of the faster diffusing components have been eliminated, and one observes a clean spectrum of the highest molecular weight component. When this same technique is applied to samples containing polymer, a clean spectrum of the polymer may be obtained without interference from the low molecular weight additives. Further details and applications may be found in reference 3.

The mathematical techniques that are currently used to resolve information in a DOSY spectrum function best when the overlap in the NMR dimension is limited to 2 or 3 components. The more overlapping resonances that are present, the more difficult the analysis becomes. In order to apply this method to more complex mixtures, we are exploring DECRA (4) and other multivariate analysis methods with Prof. Steve Brown's group at the University of Delaware. Our thanks go to Brian Antalek for helpful advice in this area.

(1) D. Wu, A. Chen and C. S. Johnson, Jr., *J. Magn. Reson. A* **115**, 260 (1995).
(2) A. Jerschow and N. Muller, *J. Magn. Reson.* **125**, 372 (1997).
(3) D. A. Jayawickrama, C. K. Larive, E. F. McCord and D. C. Roe, *Magn. Reson. Chem.* **36**, 755 (1998).
(4) W. Windig and B. Antalek, *Chemometrics and Intelligent Laboratory Systems*, **37**, 241 (1997).

Sincere best wishes.

Chris

Betsy

Jeff

D. Christopher Roe
DuPont CR&D

Elizabeth F. McCord
DuPont CR&D

Jeffrey C. Molloy
U. Delaware

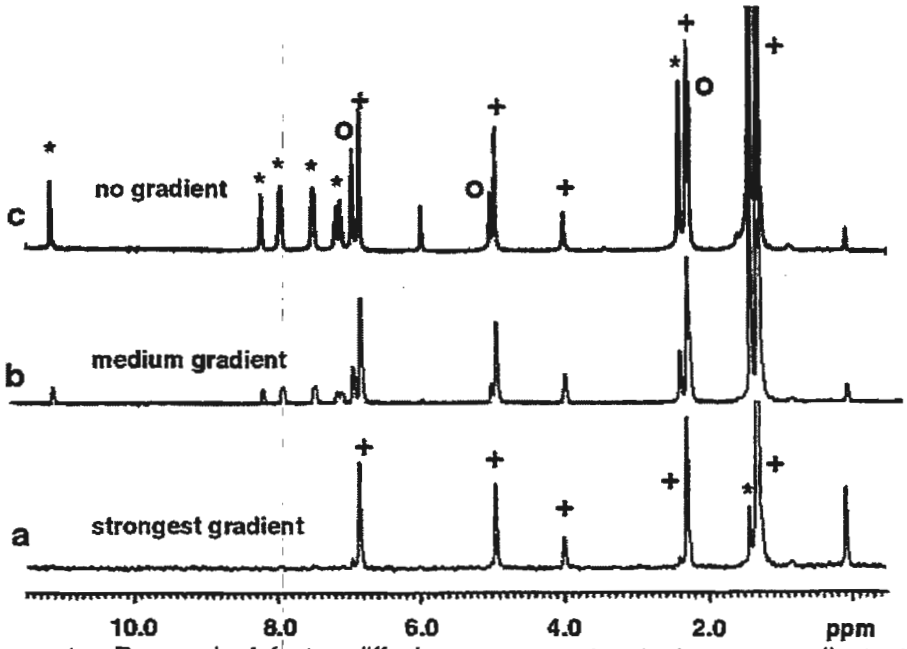

Figure 1. Removal of faster diffusing components at stronger gradients to reveal highest molecular weight component.

UNIVERSITY OF
FLORIDA

Department of Chemistry

PO Box 117200
Gainesville, FL 32611-7200

February 1, 1999
(received 2/8/99)

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

New Instrumentation; More Fluorine NMR

Dear Barry:

Recently our laboratory had the good fortune to acquire new Varian consoles and probes for two of our spectrometers. One system is an INOVA console for our 500 MHz magnet, along with a triple resonance probe and pulsed field gradients. The other is a Mercury-VX, also with pulsed field gradient, for our old wide-bore 300 MHz Oxford magnet. The sensitivities of these two systems are quite gratifying. To be impartial, our department has added a Bruker solids spectrometer with a 400 MHz wide-bore magnet and a variety of probes.

Particularly beneficial in our continuing study of fluorinated molecules is the high resolution which we are able consistently to obtain on the Varian instruments. We have also found the Varian macro 'resolv' to be very helpful in optimizing information obtainable from the spectra. This macro applies a negative exponential function and a gaussian function with the parameters calculated according to the acquisition conditions, and its use saves much time over attempts to optimize the parameters by trial and error. However, if the signal-to-noise is poor, this macro tends to wipe out the resonance multiplets.

One of the interesting generalizations we have found for coupling in long-chain fluorocarbons concerns the splitting pattern for a terminal methyl group. This signal is a larger triplet (J approximately 10 Hz) of smaller triplets (J about 3 Hz). The large triplet arises from four-bond coupling to the second CF_2 group, just as expected, but the smaller splitting arises, not from anticipated three-bond coupling, but from five-bond coupling to the third CF_2 group in the chain. This is indicated both by COSY spectra and by the collapse of the small triplet to a doublet if one of the fluorines in the third CF_2 is replaced by another group.

Recently, we have been examining the fluorine and carbon-13 spectra of a number of oxetanes which were prepared a while back by the students of Paul Tarrant. These compounds have a four-membered ring which includes one oxygen. Initially, we were interested in seeing whether we could tell if the rings are puckered, truly planar, or apparently planar because they are undergoing rapid inversion between two puckered extremes. We do not yet have an answer to this question, which may require extensive additional relaxation time determinations, but we have found some indications of nonplanarity in unsymmetrically substituted molecules, as well as obtaining some interesting NMR correlations.

Most of our oxetanes were made by photochemical addition of hexafluoroacetone to an olefin, and therefore one of the carbons next to oxygen bears two CF_3 groups. The substituents on the other two carbons include various combinations of hydrogen, fluorine, chlorine, and CF_3. A chlorine has the effect of deshielding fluorines on the adjoining position, just as in open-chain chlorofluorocarbons, but does not have much effect on the CF_3 shifts. In some of the oxetanes, there is cross-ring F-F coupling between a CF_3 and an F, but this is not always observed. This cross-ring coupling appears, then, to be a possible indication of nonplanarity.

Typical molecules are those made by the addition of CF_2=CHCl to hexafluoroacetone, which may occur in either of two directions. In the oxetane (I) with CF_2 next to $C(CF_3)_2$, the two-bond F-F coupling in 207 Hz, whereas in the oxetane (II) with the CF_2 group next to the ring oxygen, the value is only 88 Hz. Whether this difference results from different types of ring puckering produced by the location of the chlorine atom or whether it reflects some participation in bonding by the orbitals of the unshared electrons on the oxygen remains an open question. In either I or II, there is a substantial difference between the two values of the two-bond coupling from the C in the CHCl group to the CF_2 fluorines. In I, the values are 23 and 35 Hz, and in II they are 26 and 36, indicating that there is no great effect from attachment to oxygen.

Our work in this area is continuing with relaxation measurements as well as interpretation of many of the spectra by non-first-order analysis and by selective decoupling for simplification of the specta.

With best wishes,

Wallace

Wallace S. Brey

THE UNIVERSITY OF TEXAS
SOUTHWESTERN MEDICAL CENTER
AT DALLAS

Department of Radiology
The Mary Nell and Ralph B. Rogers
Magnetic Resonance Center

Southwestern Medical School
Southwestern Graduate School
of Biomedical Sciences
Southwestern Allied Health Sciences School

February 17, 1999
(received 2/22/99)

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

Early Days of NMR in the Southwest

Dear Barry,

Several years ago, a friend agreed to chair a session on the history of chemistry in the Southwest at the American Chemical Society's national meeting in Dallas in 1998 and he invited me to give a paper on the rise of NMR in the area. Since I knew many of the early participants and had contacts who could put me in touch with the others, I accepted his invitation. After interviewing and corresponding with many people, I prepared a preliminary account entitled "Early Days of NMR in the Southwest." Excerpts of it were presented orally at the American Chemical Society meeting. Since the number of people who attend any given session of such meetings is small, I thought that a more complete account might be of interest to readers of The NMR Newsletter. This history does not attempt to detail the technical development of NMR in the area; instead, it describes the path of development, the general types of instrumentation, and the people involved.

This is the first of three projected contributions on the subject. Because I had no response from several inquiries, I solicit corrections, additions, comments, etc., to enable me to prepare a more complete and correct account.

When I entered graduate school in chemistry at the University of Illinois in the fall of 1952 and first heard of NMR, much of the early history had already taken place. However, while pursuing my thesis research under Herb Gutowsky in the mid-1950's, I had peripheral contact with some of the early NMR players of the Southwest. I will mention these in the appropriate places. Also, I recall two visits by Varian people while I was at Illinois. First, Sigurd and Russell Varian visited with Gutowsky in an attempt to induce him to define the chemical shift unit size and algebraic sign with respect to a reference. In those early days of high resolution NMR spectroscopy, several different definitions were in use. Also, physicists and chemists used different conventions. As background for the second visit, I mention that I had constructed the first pulse NMR machine in a university chemistry department in the country and Gutowsky then presented many talks aimed at promoting pulse NMR in chemistry research. Around 1955 or 1956, several people from Varian again came to Urbana and took Gutowsky and me to lunch with the apparent purpose of assessing the level of interest in a commercial pulse NMR machine. Evidently, they decided there was insufficient interest to warrant marketing pulse NMR. (This incident is related to a later section of

this account.) Many years later, with the advent of Fourier Transform NMR spectroscopy, the situation changed drastically.

INTRODUCTION

Soon after the first detection in 1945 at Stanford University and Harvard University of NMR signals in condensed matter, scientists in the Southwest proceeded to develop the technique both as a fundamental research method and as an analytical tool. These two goals were synergistic and scientists in petroleum industry research laboratories as well as in academic institutions and government laboratories eagerly explored the potential of this new method to investigate solids, liquids and gases. Indeed, the first commercial NMR machines (wideline, high-resolution, and pulse, manufactured by Varian) and the first working Bruker pulse NMR machine in the U.S. were installed in Texas. Because of the widespread interest in NMR, this paper concentrates on several laboratories that were pioneers in this area of the country.

OVERVIEW

The field of NMR in the early days was cleanly divided between two different types of measurements: NMR spectra and NMR relaxation times. Gutowsky at Illinois pioneered the application of spectral measurements to molecular structure of liquids. Bloembergen at Harvard pioneered in the measurement and theoretical interpretation of NMR relaxation times in terms of molecular dynamics and structure. Those earliest measurements employed the continuous wave (cw) method in which the sample was continuously irradiated by weak radio frequency (rf) energy and the NMR spectrum was recorded while the magnetic field strength or the rf frequency was scanned over a narrow range of values. In 1950, the measurement of relaxation times was greatly facilitated by the development of pulse NMR by Hahn at the University of Illinois physics department. In contrast to cw NMR, the sample is excited by a short, intense burst (pulse) of rf energy and the NMR signal is generated by the sample as it "relaxes" to its equilibrium state. Subsequently, the two different measurements came to be done with different types of electronic instrumentation. Commercial NMR spectrometers were not readily available until the mid 1950's; therefore the earliest experiments were done on home-made instruments.

The early applications of NMR spectra were the determination of molecular structures of organic molecules from hydrogen NMR measurements. Because of these capabilities, petroleum companies in the Southwest, such as Mobil and EXXON, made early use of it to study the molecular structures of petroleum and petroleum fractions. EXXON was particularly aggressive in this regard and extended the technique to carbon-13 NMR spectroscopy right after such measurements were demonstrated. Mobil carried out much in-house instrumental development to improve the quality of hydrogen spectra.

Measurements of relaxation times were early applied to the relaxation in aqueous solutions of paramagnetic metal ions (an outgrowth of the original relaxation measurements of Bloembergen on such samples) and to the effects of surfaces on shortening the hydrogen relaxation times of surface molecules. Early research at Los Alamos and at The University of Texas concentrated on the former and Mobil did pioneering research in the latter area. The research in both areas led to widespread applications at the present time, such as in clinical magnetic resonance imaging and NMR logging of oil wells.

continued on p. 39

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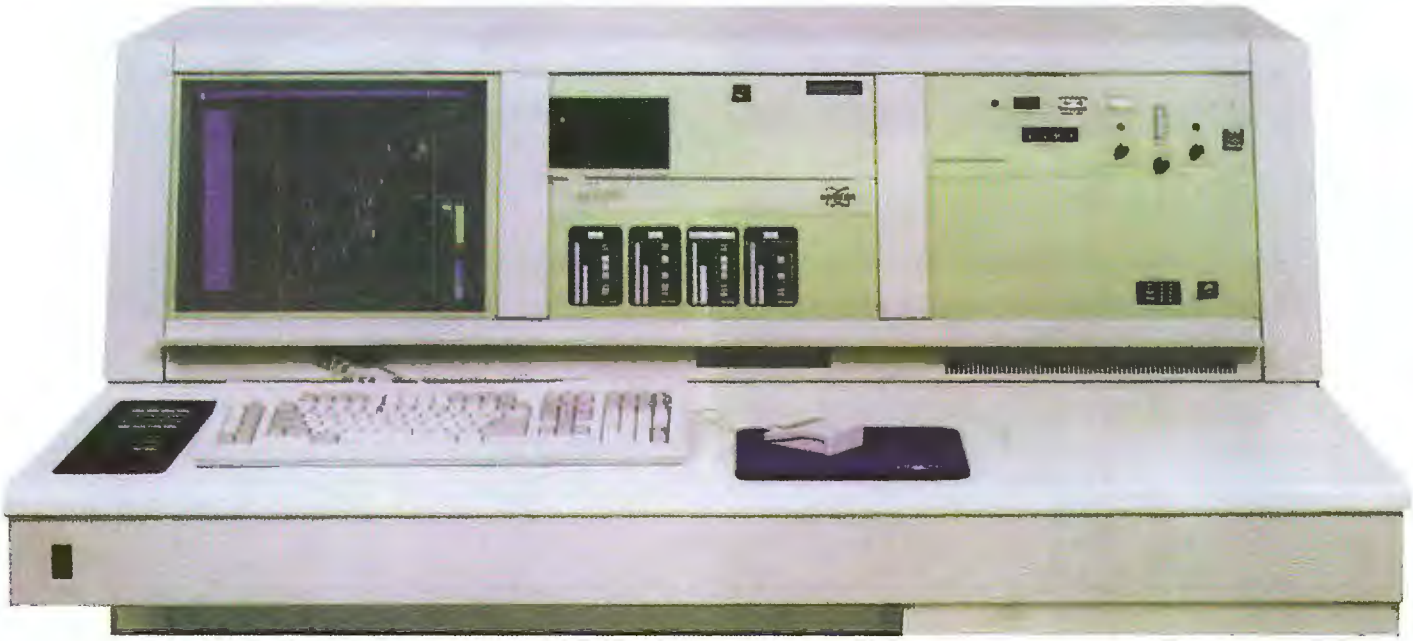

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continued from p. 36

LOS ALAMOS

Probably the earliest NMR experiment in the Southwest was performed at Los Alamos. In 1946, Felix Bloch and Martin Packard brought their NMR equipment from Stanford to Los Alamos. Working with Rod Spence and Al Graves of Los Alamos, they determined the nuclear spin quantum number and magnetic moment of the triton (the tritium nucleus). Perhaps it was easier to do the measurements by taking the equipment to Los Alamos than by taking the tritium to Stanford.

The next NMR research done at Los Alamos started about 1953 by Jasper A. Jackson. W. B. Lewis had an electron spin resonance (ESR) program that used a Varian 12-in electromagnet. Jackson used a commercial NMR magnetometer (Pound-Watkins oscillating detector) to map the magnetic field in this magnet. This magnetometer was then modified to obtain NMR signals from various samples and used to obtain low-resolution NMR spectra.

A few years later, in 1956, Henry Taube of the University of Chicago was a summer consultant at Los Alamos. He conceived the idea of using the newly observed NMR signals of O-17 to study ionic hydration in aqueous solution, provided that separate signals could be obtained from oxygen in the water of the first coordination sphere of the ion and from bulk water. This led to the approval of a proposal to apply NMR to inorganic chemistry using the signals from many different isotopes, in addition to O-17. Jackson arranged for the purchase of a Varian model V4200 wide-line high-sensitivity NMR machine. It was installed in 1957 and NMR signals from many different nuclei were studied. However, they were unsuccessful in detecting a separate signal from water in the hydration shell.

Then, in the summer of 1959, Henry Taube made the suggestion to add a paramagnetic specie with short exchange time for its water of hydration to the aqueous solution of nonmagnetic cations. A solution of $Al(ClO_4)_3$ showed only a single oxygen-water NMR signal. After adding a dilute concentration of $Co(ClO_4)_2$, the O-17 signal in bulk water shifted downfield (and broadened), but a residual peak remained at the original bulk water position. The second peak was attributed to tightly bound water in the hydration shell of the Al^{+++} ion. This was the first reported use of a paramagnetic shift reagent. Separate hydration signals from Be^{++} and Ga^{+++} ions in aqueous solution were also detected.

Before the above experiment was performed, the only source of water enriched in O-17 was the Weissman Institute of Science in Israel. The success of these "early" experiments led to building a plant at Los Alamos to produce material enriched in O-17 for the NMR program. After several months of production, an improved method for separation of oxygen isotopes was discovered in Switzerland. This method was implemented at Los Alamos and production of copious quantities of O-17, O-18 and N-15 ensued. Numerous papers were published by the Los Alamos NMR group in the 1960's using the O-17 and N-15 produced by this plant. In 1969 the plant was converted to produce C-13 for biomedical NMR and was renamed the National Stable Isotope Resource. for several years it was the nation's supply of C-13 as well as other stable isotopes (O-17, O-18,N-14, N-15, S-33, etc.).

The NMR instrumentation was later expanded. In the mid 1960's another Los Alamos group became active in NMR. It acquired a Varian HR-60 high resolution spectrometer. This was then converted into another dual-purpose spectrometer and, in 1967, Eiichi Fukushima, who had just finished his Ph.D. at the University of Washington, joined their program. Jackson also built an

instrument large enough to hold a 150-gram rat and, in 1967, made the first NMR observations on a whole, live animal.

At the 1963 Gordon Conference on NMR, Jackson met Dr. Leon O. (Tom) Morgan of the University of Texas at Austin. Because of Morgan's work at the Metallurgical Laboratory of the University of Chicago on the Manhattan Project of World War II, he was known to Los Alamos researchers and Jackson arranged for Morgan to visit in 1964. He became a regular consultant to Los Alamos for many years.

THE UNIVERSITY OF TEXAS AT AUSTIN

NMR in the chemistry department started with the efforts of Tom Morgan. The early research concentrated on NMR relaxation times; high resolution NMR spectroscopy was started later. In graduate school in Berkeley in 1947, he attended a seminar by Bloch which inspired him to follow later developments, reading as much as he could from the early papers of the few then publishing in the fields of his interest.

Morgan came to Texas in September, 1947, fresh from Berkeley with a Ph.D. in what amounted to nuclear chemistry and physics, but nominally, chemistry. His primary interest, starting in 1941, was nuclear chemistry and his initial efforts were to establish a research program in this field. With willing assistance from the Berkeley cyclotron group, they were able to produce some publishable research, but that dried up when cyclotron maintenance and re-building pushed such research back in the schedule.

To fill the gap, they became interested in following details of electrode processes using tracer techniques. Their emphasis was on complex electrode processes, such as those involved in chromium plating, and perchlorate production, which were of interest to the Office of Naval Research (ONR). Their experiences there convinced them that they needed a better way of determining both detailed structures and rate processes in surface interactions.

From the early publications on NMR relaxation of protons in aqueous solutions of paramagnetic ions, it was apparent that NMR might indeed provide the better way. Of special value to them were the papers of Bloch, Hansen, and Packard [Phys. Rev. **70**, 474 (1946)], Bloembergen, Purcell, and Pound [Phys. Rev. **73**, 679 (1948)], Conger and Selwood [J. Chem. Phys. **29**, 383 (1952)], and J. R. Zimmerman (J. Chem. Phys. **21**, 1605 (1953)]. Conversations with Zimmerman, who was then at Mobil Research in Dallas, provided a big stimulus for the Austin group, as did the work of Bernheim, Brown, Gutowsky, and Woessner at the University of Illinois. Equipment to perform pulse NMR relaxation measurements was built in the physics department by Prem Mahendroo, a graduate student from India. The rf pulses were obtained from a gated amplifier used in conjunction with a highly stable rf signal generator. First, they used a permanent magnet of 6,800 gauss field strength with a pole face diameter of six inches. Later, a Varian iron electromagnet with six-inch pole faces was used. Use of this magnet with adjustable magnetic field strength allowed a wide range of NMR resonance frequencies, enabling determination of the frequency dependence of the relaxation times. Modern NMR spectrometers with superconducting magnets do not have this versatility.

Their first paper in the field, submitted on September 1, 1955, was a collaboration among A. W. Nolle (Physics at UT), chemistry graduate students Robert Hull and Joyce Murphy, and Tom

Morgan [J. Chem. Phys. **25**, 206 (1956)]. In this they emphasized the importance of relatively non-labile solvation shells or coordination spheres about metallic ions, and of atomic exchange interactions which could lead to averaging. They found no evidence for viscosity control of relaxation rates in those instances. In another short letter they illustrated the possibilities for two proton phases in a complex species having a ligand such as glycerin, with two separate proton species, exchanging independently [J. Chem. Phys. **24**, 906 (1956)]. They then discovered that Zimmerman and F. J. Karal were to present a more general paper on two-phase nuclear systems at an American Physical Society meeting in Houston, TX, on February 24-25, 1956, which were consistent with their observations. To this point, their research was largely supported by the ONR through grants to the University of Texas.

They broadened their efforts to include a variety of paramagnetic ion solutions with differing electronic structures, e.g., d3, d5, d6, d8, d9, f2, f7 [with A. W. Nolle, J. Chem Phys. **26**, 642 (1957); **31**, 365 (1959)] with additional support from the Robert A. Welch Foundation, Houston, TX. That work led to Morgan's collaboration with Nicolaas Bloembergen, in 1960, [J. Chem. Phys. **34**, 842 (1961)] which produced the so-called Solomon, Bloembergen, Morgan theory, widely cited in MRI research and applications as well as in chemistry and physics research publications as the SBM theory.

At about the time his first work was submitted, Morgan approached Herb Gutowsky and W. George Parks on the question of having a Gordon Research Conference on Nuclear Magnetic Resonance. Morgan and Gutowsky had received letters of support from a number of people representing major industrial and academic research centers in the field. In the Southwest, Zimmerman at Mobil and Williams at EXXON strongly supported Morgan in this effort. As a result, the first such Gordon Conference was held June 30 - July 4, 1958 in New Hampton, New Hampshire. It was chaired by Gutowsky and was very successful, having over 100 participants.

High resolution spectrometry was started around 1960 by Ben Shoulders (with Pete Gardner) and John Mahler (with Rowland Pettit) with a Varian DP60; then with an A60 with external lock; and an HA100, in about 1964. Ben Shoulders had experience at Illinois operating 40 MHz and 60 MHz cw instruments as a service for the organic chemists. (This was while I was there, and I used the 40 MHz magnet with my pulse NMR console for some relaxation experiments.) He had further experience as a graduate student at Texas Christian University. Jefferson Davis arrived in the mid sixties, and with continuing Departmental acquisitions, others in organic and biochemical research as well as inorganic and physical chemistry, also became active. Morgan's work trended toward multinuclear spectrometry as more separated isotopes became available.

More in the next installment.

Sincerely,

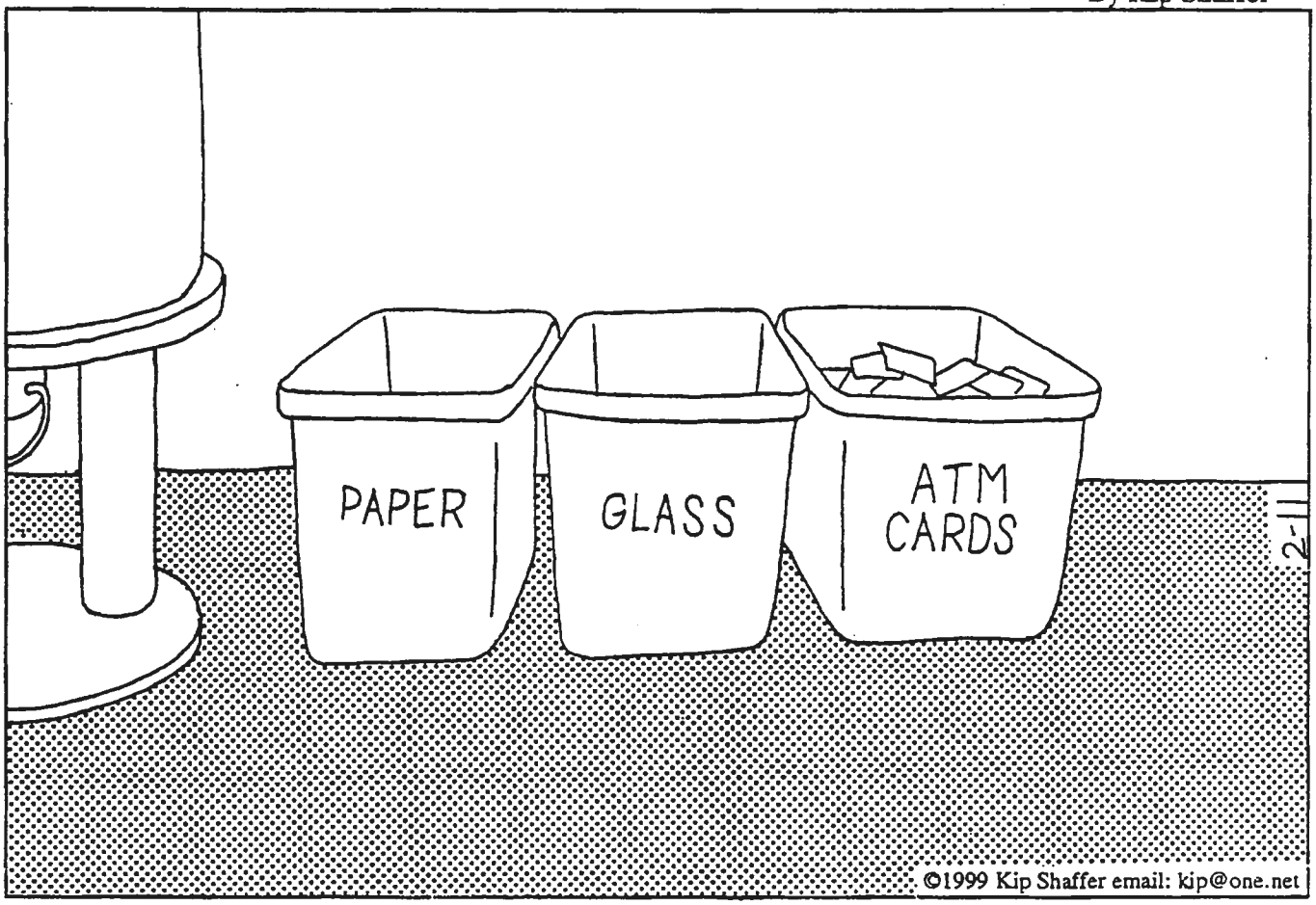
Don

Donald E. Woessner, Ph.D.
Adjunct Assistant Professor of Radiology

dwoess@mednet.swmed.edu

Field of Dreams

By Kip Shaffer



Address all Newsletter correspondence to:

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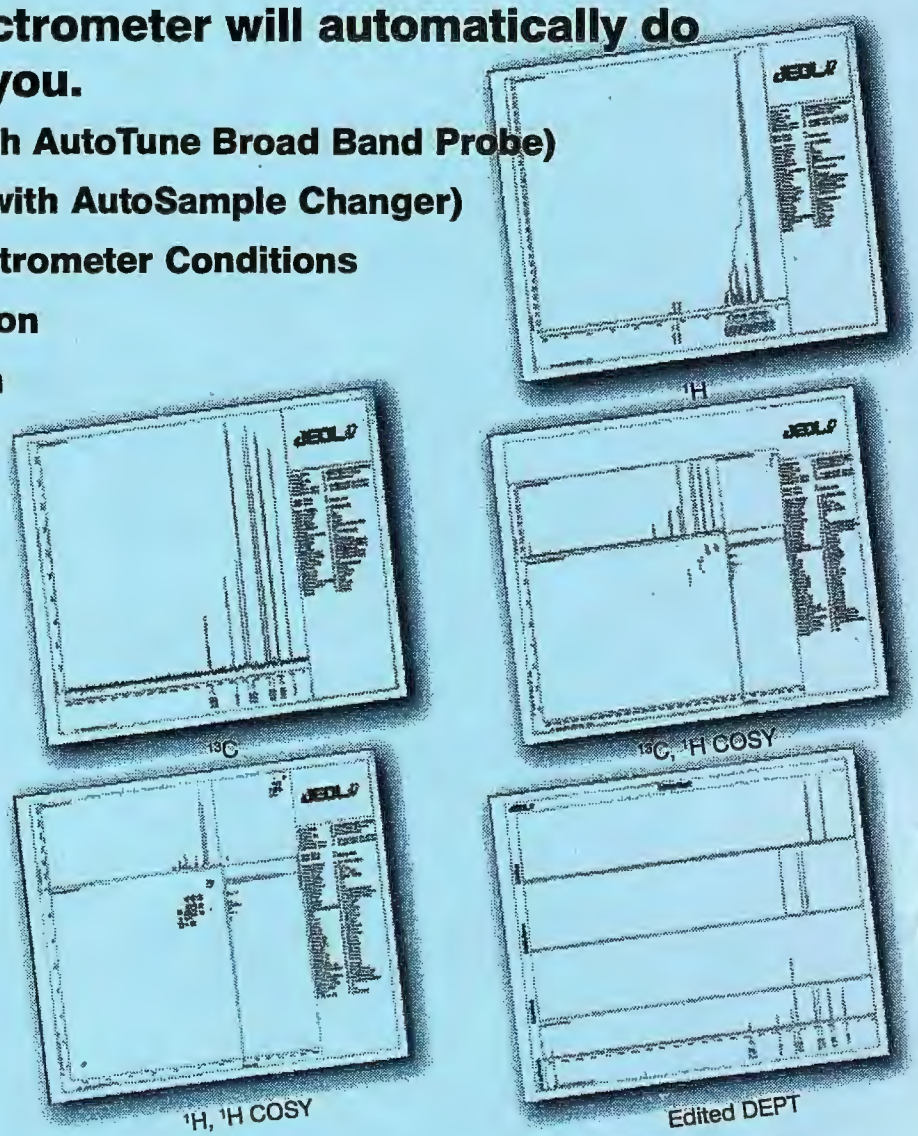

JEOL USA, Inc., 11 Dearborn Road, Peabody, MA 01960
Tel: (508)535-5900 Fax: (508)536-2205
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