TEXAS A&M UNIVERSITY

NMR NEWSLETTER

No. 380

May 1990

TABLE 1 DEUTERATED SOLVENTS

Cat. No.	Description	Formula	Min. Isotopic Purity (%D)	Density (g/ml)	MP (°C)	BP (°C)	$-\chi_v \times 10^6$ @ (°C)
D-11	Acetic Acid-d_4	CH_3COOD	99.5%	1.1	17	118	0.551 (32)
D-120	Acetic Acid-d_4 + TFA		99.5%				
D-13	Acetone-d_6	CD_3COCD_3	99.8%	0.84	-94	57	0.460 (20)
D-121	Aceonte-d_6 + 1% TMS		99.8%				
D-129							
D-14							543 (20)
D-21							0.611
D-122							
D-130							
D-28	Chloroform-d	$CDCl_3$	99.8%	1.50	-64	62	0.740 (20)
D-31	Chloroform-d + 1% TMS		99.8%				

VARIAN

BOX/500 SHEETS

Cat. No.	Paper Size	Instrument Model	Type
WCV-100 (S-100A)	11" X 26"	HA-100, HA-100D	Gridded-Two Color
WCV-60 (A-60C)	11" X 26"	A-60A, A-60D	Gridded-Two Color
WCV-XL (XL-100)	11" X 26"	XL-100 (-12 and FT)	Gridded-Two Color
WCV-XLFT-100	11" X 26"	XL-100 (-12 and FT)	Gridded-Two Color
WCV-XL-200	11" X 26"	XL-200, XL-300, XL-400	Gridded-Two Color
WCV-20 (CFT-20)	11" X 16 3/4"	CFT-20, FT-80, FT-80A	Gridded-Two Color
WC-11[illegible]	X 17"	All Models [illegible] 16 1/2" or 16	Blank

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Continued on page 59

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COLLEGE OF CHEMISTRY NMR Facility

BERKELEY, CALIFORNIA 94720

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

March 26, 1990
(received 3/29/90)

Dear Barry, HP-Laser Plotter/Printer On Bruker X32

We added an HP Laserjet II to a Bruker X32 data system for both plotting and printing. Adding plotter capability is relatively straightforward. Adding a "Plotter in a Cartridge" (Pacific Data Products, San Diego CA) provides HPGL emulation. (This cartridge has been used on other data systems as described by several TAMU contributors.) The wiring of the printer is according to the HP specification:

Laser Jet		X32
2	----------	2
3	----------	3
7	----------	5
20	----------	6 + 8.

The following options must be set up on the printer control panel:

Auto page eject: ENABLED. Eject time out: NO TIME OUT. The printer port options need to be configured: Baud : 9600. Word : 8N1. Select Xon/Xoff. : The default setting is ok for all the other options.

In uxnmr, the cfpp feature is used to add an hp7455a plotter to whichever RS-232 port is available (e.g. 05).

It is possible to use the "plotter" as a printer as well. UNIX requires that a "printer interface" be added and the regular UNIX printer installation procedure must be gone through. The printer interface is a text file containing commands. For operation as a printer, the printer is sent commands to un-load the plotter cartridge, print the text submitted and reload the cartridge. This results in a substantial delay when switching to text mode. On the other hand, if many pages are to be printed, this may still be quicker than a dot matrix printer in NLQ mode (certainly better looking and quieter).

Since the printer interface is too long to reproduce here, anyone interested can send us E-mail at nmr@garnet.berkeley.edu. We will deliver the file by return mail.

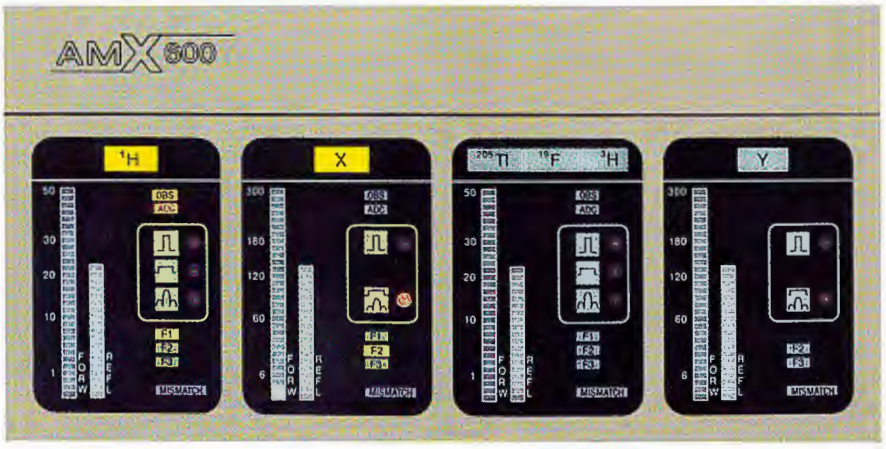
Sincereley,

Rudi Nunlist

Rich Mazzarisi

Introducing the AMX. It's the most flexible high performance NMR spectrometer yet. It comes with: 451 MHz IF. All-new receiver. New HP-pulse/CW-linear RF transmitters. Unique, computer-controlled Router.* Precise pulse-shaping capability. 32-bit UNIX™-based system computer. 25 MIPS array processor. Ethernet™, including TCP/ IP. And more.

Let us show you.



* The new AMX full routing system with PIN diodes allows micro-second switching of the frequency paths and RF transmitters on the fly. Ask your nearest Bruker representative for more details on the new AMX Series.

Bruker Instruments, Inc.
Manning Park, Billerica, MA 01821
In Europe: Bruker Analytische Messtechnik GmbH, Silberstreifen D-7512 Rheinstetten 4, W. Germany

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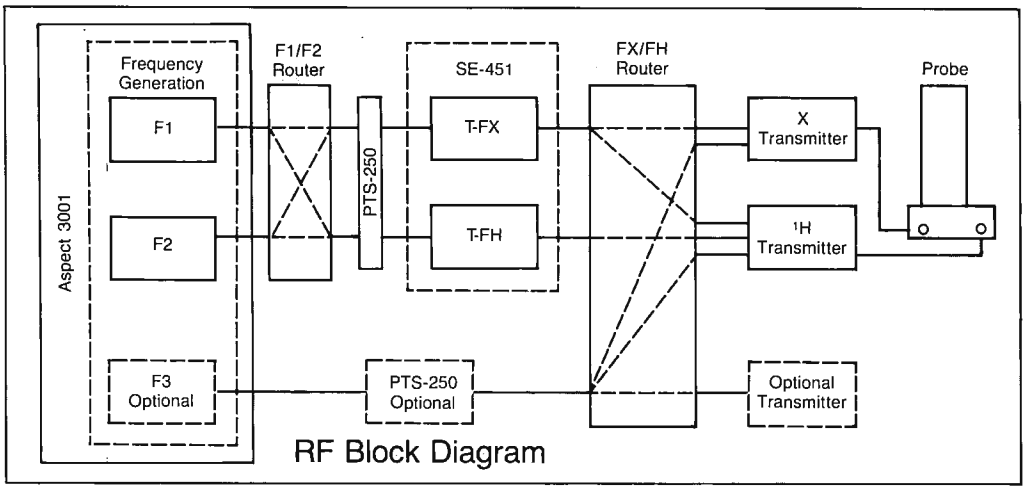
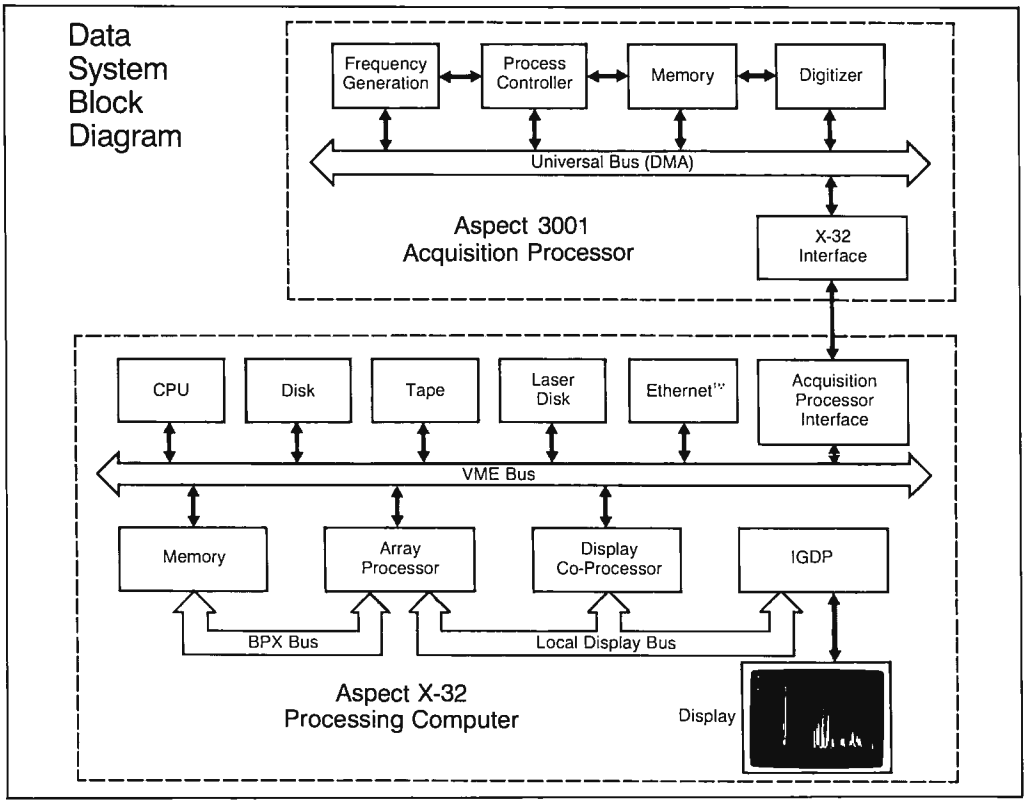
AMX Series Block Diagrams



RF Block Diagram

Multi-channel RF design. The unique full routing system uses PIN diodes to allow microsecond switching of the frequency paths and RF transmitters.



Digital Section. Integration of 32-Bit Aspect X-32 UNIX computer into console together with proven Aspect 3001 acquisition processor. The X-32 is equipped with unique 3-bus structure for optimum NMR data transfer, processing and display.

For more information about the AMX simply call or write:

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(608) 264-5407
E-Mail RALPHJ@VMS.MACC.WISC.EDU
April 16, 1990
(received 4/20/90)

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

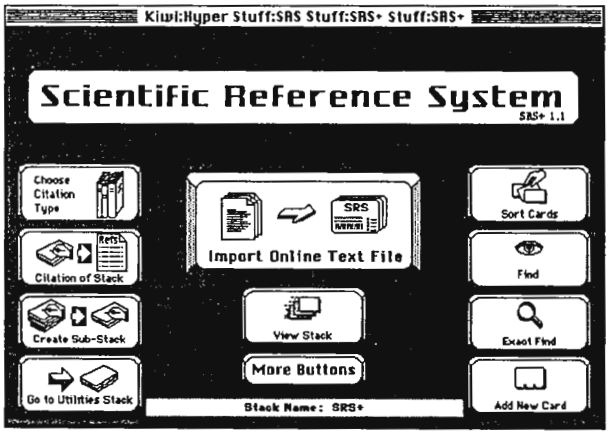
Dear Dr Shapiro,

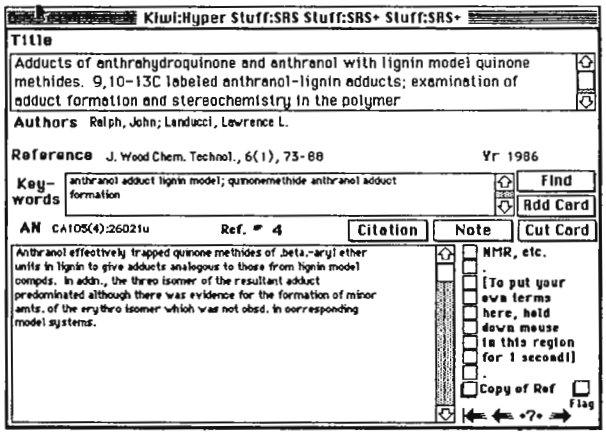
HyperCard Applications

Over the past couple of years we have been having a lot of productive fun with HyperCard,™ the software from Apple Computer that is everything from a Hypertext environment to a 'software erector set' (Bill Atkinson). Right from its introduction it was clear that here was an excellent tool for writing little software projects and applications for which we previously lacked the time or the motivation to develop applications in traditional languages.

I wish to just briefly describe two systems that we are working with, one that is complete and is actually on the market now (so I will try not to hype that too much) and one which is useful already but is awaiting further development and a great deal more NMR data.

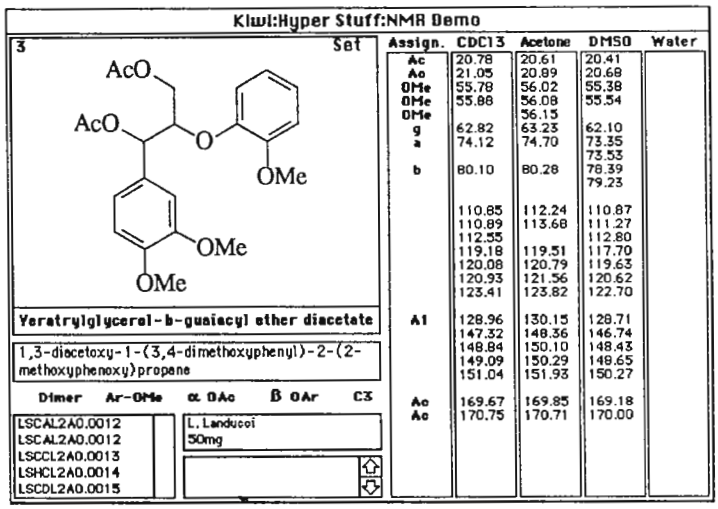
The first application is a reference system for journal references. I was motivated to write this because I could find nothing on the market at the time that would do what I wanted. I still don't think there is anything which is as flexible and as easy as this system is. It also arose from the realization that I and researchers like me were far too lazy (or too busy) to go typing millions of





references into a database. Consequently, the system should import from standard databases. Since I was using STN CAS-Online, that was the first essential. Now, all you do is go online as usual and search for all the references you need. Then you simply import them into HyperCard with the push of a single button. All the garbage is stripped out, and the references have a typical card-like format. But of course it is far more flexible than that. You can very quickly find any reference by searching for hit words on any card, you can create your own notes about the paper or even include the entire paper's text into the card if you wish, you can sort by various criteria, and, very importantly, you can create citations of single references or of the whole stack in any of 100's of journal formats - if you are using a journal that is not in the extensive list you can rapidly create the format in a pictorial way. This software has undergone tremendous improvement since its shareware days, imports from a variety of commercial databases, and is now marketed by Trinity Software, (603) 726-4641, for a rather reasonable price.

The second is less complete but of perhaps more interest to NMR people. It is a simple database of the kind that many of you have perhaps created (or thought about creating) using database or spreadsheet software, but it has a number of nice features that are not apparent from the figure of a sample card (Hyper-literate people will know to try clicking all over the place to see if logical things happen). Firstly you will note that the structure of the compound is on the card (essential!!). Clever trickery is used to create the illusion of intelligence regarding the structure. With NMR data, it is always nice to compare compounds with similar structural features when trying to make assignments (unless you have the luxury of carrying out the full complement of 1D and 2D experiments to unambiguously make complete assignments).



We have two ways of doing that in this stack. Firstly, simply clicking on a portion of the molecule will take you to the next molecule in the stack with that same feature. You can then flip backward and forward comparing the data. Alternatively, you can sort the whole stack by those criteria. This is done in our stack either by selecting the criteria from a menu or, again, using the power of the Mac and HyperCard, by simply clicking (with the option key held down) on the structural features of interest. This groups all those compounds at the front of the stack for your examination. HyperCard programmers may think that we are compelled to make thousands of buttons, all replicated on each card, for all the individual structures' information, but again we have used a nice trick that allows us to store structural information in simple (hidden) text fields and have only one set of background buttons. When you click on a part of a structure, the system looks up where the buttons should be, places them, and then re-clicks on the same spot which carries out that appropriate button's function. All this is invisible to the user who cannot tell that he didn't simply click on a button that was already in place on the structure.

This stack (complete with all the relevant NMR spectra) is being developed for lignin model compounds in a joint venture between our lab and the U.S. Forest Products Laboratory, and we hope to have it out by a 1993 ACS Symposium on NMR in Plant Chemistry. With the exciting announcement at the recent ENC of Bruker's NMR processing package running on the Mac, we hope to include real spectra as well as re-created spectra, perhaps on CD or CD-ROM, with the database.

Sincerely

John Ralph

P.S. I still have funding for further PhD students who are interested in plant chemistry with a high NMR component. Those interested in doing some good synthetic chemistry, 'playing' with NMR, 'playing' with MacIntosh computers, and think they are tough enough for the Wisconsin winters, should contact me directly.

Fremont Magnetic Resonance
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(415)623-0851 FAX

^{31}P / ^{13}C / ^{1}H *QE* Probe

One port tuned for ^{1}H observation.

One port simultaneously tuned for both ^{31}P and ^{13}C observation.

Allows the QE console to observe ^{1}H, ^{31}P and ^{13}C spectra without tuning or switching the probe in any way.

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NMR Instrument Upgrades

Broadband Decoupler

- Broadband RF Circuits (5-500MHz).
- Accepts any Synthesizer Input (0dBm).
- Fast BiLevel RF Power Switching (< 1 usec).
- MLev & Waltz Modulation Schemes.
- 0.5 Watt Output to external RF Power Amplifier.
- Excellent On/Off isolation.
- 90°/180° Phase Shifts (Sub 90° available)
- High Performance / Low Cost

The FMR Broadband Decoupler provides an ideal auxiliary transmitter for polarization transfer and 2DFT experiments. Its high performance features (rapid solid state switching between two levels, MLEV and WALTZ modulation schemes) along with its low cost make the Broadband Decoupler an ideal, flexible and powerful addition for expanding a spectrometer's capabilities. The FMR decoupler can be used with existing power amplifiers or frequency sources and power amplifiers are available as a total package.

The decoupler is of modular construction. Individual modules or a complete package can be purchased. The modules are useful for updating existing instruments. FMR will provide the modules and assistance for the do-it-yourselfer.

- Digital 90° and 180° Phase Shifter Module (Operates at 10 MHz).
- Mixer / Gate / Attenuator Module (Fast BiLevel Operation).
- MLev / Waltz Modulator Module.
- Filters for one frequency.
- Chassis and Power Supplies.

NT Patch Cables

Patch cables which allow immediate installation and removal of the Broadband Decoupler to an NT spectrometer are available. The cables plug into the existing connector of the NT 293 and the existing console cable plugs into the patch cable. The standard configuration for these cables is available as well as custom configurations.

Bruker AM Interface.

A splitter-mixer-amplifier module is available to allow the O1 synthesizer output to be split and used as a frequency source for the decoupler as well as to continue generating the spectrometer observation and receiver frequencies. This process allows the use of the Bruker pulse sequence phase expressions to be used for Broadband Decoupler phase shifts as well as the Decoupler's internal phase shifts and modulation capabilities.

Probes

FMR provides probe services designed to give the NMR marketplace the capability for repairing and upgrading existing NMR probes as well as obtaining new and unique NMR probes. Existing probes can be upgraded at less than half the cost of a new probe. An existing unused 20mm probe, for example, can be converted to a 10mm Broadband probe. A 5mm ^{1}H Probe can be reworked to improve sensitivity in water solutions or water saturation.

Probe Services

- Fast and cost effective probe repairs. If you don't have backup probes, the time spent getting even the most simple probe problem repaired can be agonizingly long. FMR tries to turn every probe around as fast as possible, *and* we are always trying to improve our turn-around time.
- Do you have a probe lying around the laboratory which hasn't been used in years? If so, why not upgrade it? This can be done for a lot less than a new probe. Existing or broken probes can be repaired and upgraded to different insert sizes and nuclei. The following areas are prime reasons to upgrade an existing probe:
  - New technology to give better performance. Some older probes can be upgraded to give more than a 100 % improvement in sensitivity.
  - Repair or upgrade an older unused probe to serve as a backup for existing used probes.
  - Upgrade an unused probe to be a ^{1}H biological (salt) probe. This will give shorter 90° pulses, better sensitivity and better water saturation performance in ionic water solutions.
  - Upgrade an unused or broken probe to be able to do new experiments. For example FMR can upgrade an older, little used probe to be a ^{1}H {^{31}P-^{15}N} probe for today's powerful inverse 2DFT experiments.

Water Saturation Modifications

Many ^{1}H probes used for water saturation experiments have coil lead pickup (see Instrumentation Note 17). FMR can modify these probes to shield the coil's leads and improve the water saturation performance. This problem is easy to detect and solve.

Call us if you have questions.

National Institute of Neuroscience

National Center of Neurology and Psychiatry

4-1-1 Ogawahigashi-cho
Kodaira, Tokyo, 187 Japan
Phone:0423-41-2711
Fax: 0423-42-7521

February 24, 1990
(received 3/20/90)

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

2 Tesla Whole-Body MR Spectrometer at NCNP

Dear Dr. Shapiro,

NMR suffers from the inherent low sensitivity because of dealing with low energy. However, it should be recognized that this makes it possible for NMR to provide a non-invasive and non-destructive method that is essential for in vivo NMR(MRI and MRS). One way to improve this sensitivity problem is to increase the magnetic field strength. Therefore, there has been the desire to use higher magnetic fields even in in vivo NMR. At present, whole-body magnets at fields of up to 4 Tesla are available even though excellent images(MRI) may already be obtained at fields below 1.5 Tesla. However, at higher fields(higher frequencies) there may be problems related to the RF effects, motion artifacts, chemical shifts, tissue susceptibility effects etc. especially in MRI. In fact, at 4 Tesla the degraded proton images of the human head and body are observed due to the RF effects(H. Bomsdorf et. al. NMR in Biomed. 1, 151 (1988)). In clinical applications, image qualities should not be sacrificed for any reasons. In this meaning 4 Tesla is clearly above the optimum.

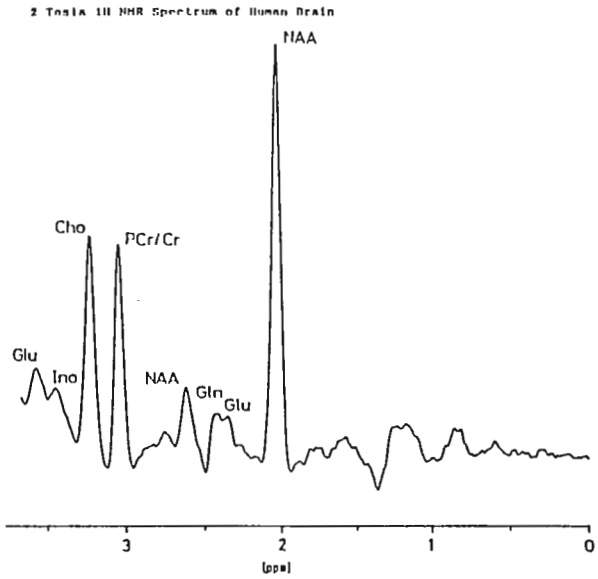
We have recently increased the static magnetic field strength of our Philips Gyroscan 1.5 Tesla whole-body MR spectrometer to 2 Tesla. Image qualities of 2 Tesla head and body coils were first checked on phantoms(including conducting samples) using the Philips 1.5 Tesla standard installation procedures. In the pulse sequences tested(MS, 2D, 3D of SE and FFE), 40-100% improvements in S/N were found compared to those at 1.5 Tesla(The theoretically expected gain is 33-65% depending on sample loads). The extra improved S/N may be partly due to the optimized RF chain at 2 Tesla. Non-uniformities of RF field were about the same as those at 1.5 Tesla. Furthermore, the 2 Tesla proton images of the human head and body were, in general, of good quality with the improvements in S/N and appeared to be better or at least comparable to those made at 1.5 Tesla. A "quadrupole" intensity artifact was only enhanced in the proton density images of transversal section of the human head and body, which heavily depended on patient loads. We expect that the

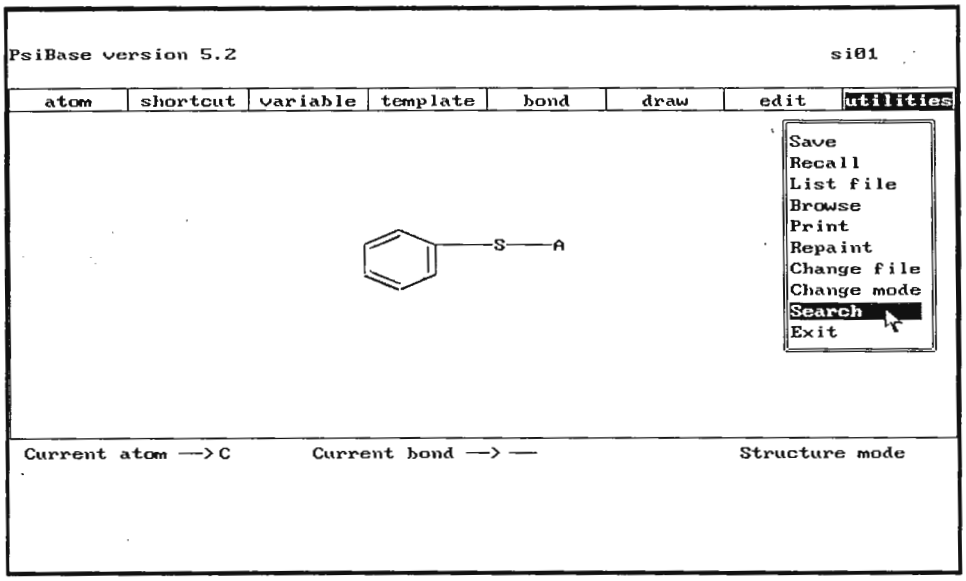
artifact could be reduced or eliminated using a quadrature coil. Increasing the magnetic field strength is more favorable for <u>in vivo</u> NMR spectroscopy(MRS). In particular, the better spectral resolution at 2 Tesla would make water suppression in proton MRS easier. Figure shows a 2 Tesla proton MRS spectrum of human brain(NAA; N-Acetyl Aspartate, Glu; Glutamate, Gln; Glutamine, Cr; Creatine, PCr; Phosphocreatine, Cho; Choline, Ino; Inositol). The spectrum of VOI 27 cc and echo time 100 ms was accumulated only for 2 minutes using a standard head and neck imaging coil. The water suppression factor of 1/500 to 1/2000 was easily obtained depending on echo times. However, it should be noted that at 2 Tesla many metabolite signals such as glutamate become a strongly coupling spin system resulting in disappearance of the signals at longer echo times due to the J-modulation if spin echo is included in the pulse sequence. Considering the increased RF effects at higher fields, we think that 2 Tesla may still be below the optimum for MRS but is a reasonable field strength for clinical applications of MRI and MRS if both are required.

This work was performed in collaboration with Philips Medical Systems of Japan. Part of the experiments described here was performed in collaboration with Dr. Peter R. Luyten and Dr. Jan A. den Hollander of Philips Medical Systems of the Netherlands. The 2 Tesla research project at NCNP is supported by Science and Technology Agency Grant(T.O.) and Grant-in-Aid for Developmental Scientific Research from the Ministry of Education, Science and Culture(T.O.).

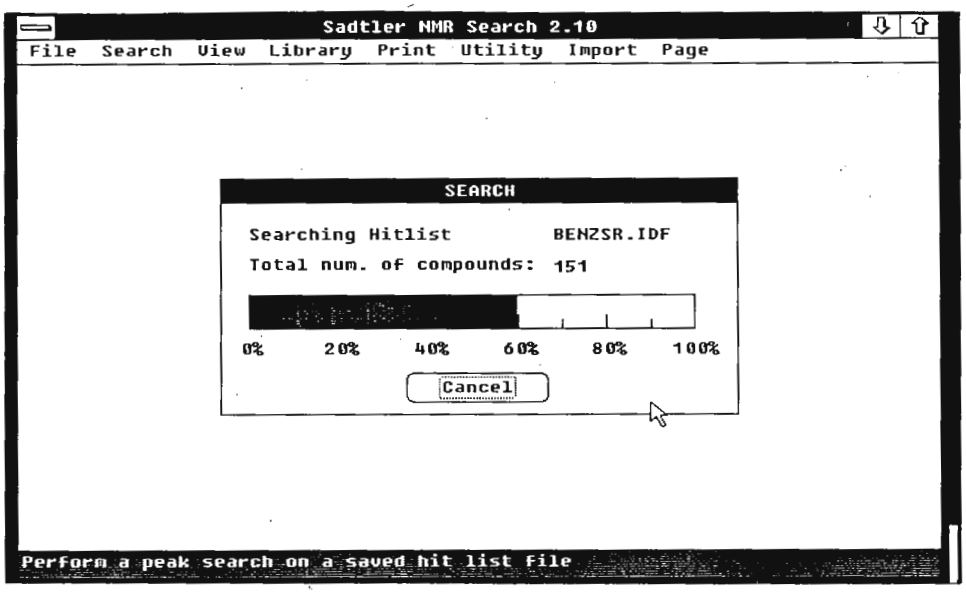
Sincerely,

Takashi Ogino Toshio Yano Susumu Masuda Motoo Nagayama

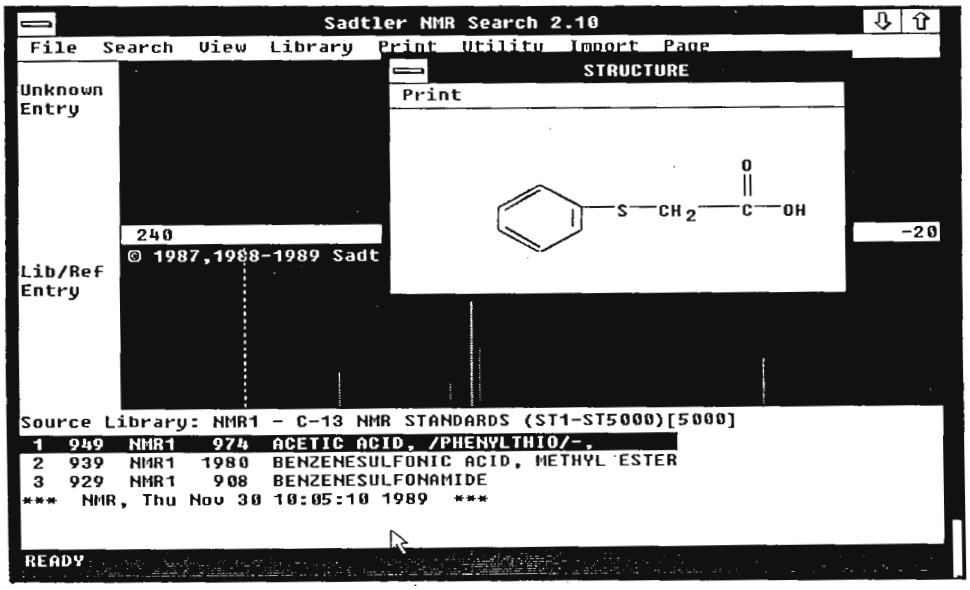




Step 1: Search for all thiophenol derivatives.



Step 2: Search for those derivatives that match your spectrum.



Your search is over!

When faced with a tough analytical problem . . .

Q. How do you find all the thiophenol derivatives matching YOUR CARBON SPECTRUM?

A. ✓ **Sadtler Molecular Substructure Search Software**

✓ Sadtler ^{13}C NMR Libraries and Search Software

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Michigan Technological University

Houghton, Michigan 49931

College of Sciences and Arts
Department of Physics
906/487-2086

March 26, 1990
(received 3/29/90)

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

Dear Dr. Shapiro:

The use of finite fast fourier transforms to approximate an infinite continuous transform has difficulties we are all familiar with. These difficulties are often treated in approximate ways which lead to a loss of information or of efficiency. For example, when acquiring free induction decay data, the signal must decay to zero significantly before the end of the acquisition to avoid the use of either i) adding zeros to the end which becomes equivalent to apodization with a square wave or ii) apodization with a decaying function (such as an exponential or gaussian) which broadens the line. Hence the fraction of data points which are actually used is less than the number available in the hardware.

The effect is due to aliasing but in the reverse sense than it is normally presented. If you recall that the raw data is (essentially) the fourier transform of the spectrum then if the raw data extends to the end of its array (e.g. to high "frequencies") then it has suffered from inadequate "sampling" in the spectrum.

We have implemented and are pleased by an improved fast fourier transform in our software which is far less sensitive to these problems. It is outlined by Froeyen and Hellemans in the Reviews of Scientific Instruments 56, 2325 (1985). I have not heard that such an algorithm is standard in modern NMR spectrometers and it certainly would not be present in older models.

The execution time on the computer which controls our equipment (IBM PS2-80) is double that of a simple fast fourier transform for typical data.

Sincerely,

Bryan Suits

Bryan H. Suits
Department of Physics

The University of Akron

Department of Chemistry (216) 375-7372
Buchtel College of Arts and Sciences
Akron, OH 44325-3601

March 15, 1990
(received 3/26/90)

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

Dear Dr. Shapiro:

We are exploring the use of Principal Component Analysis (PCA) for improving the quality of 2D-NMR spectra. This technique is a well established tool for interpretation of other forms of spectrometric and chromatographic data. It is proving useful for separating artifacts from real signals in 2D-NMR spectra as well.

The method work by interatively calculating a signal component which accounts for the largest degree of variance in the dataset. Each successive principal component is calculated and subtracted from the data. For COSY-type spectra (i.e. those with diagonal symmetry); ridges, baseline offset and roll, and tl noise are the first components removed, followed by diagonal signals. The attached figure shows an example of the change in a typical COSY spectrum.

The technique has been tested on several hundred COSY, NOESY, and ROESY spectra. Unlike some methods of digital signal processing, this method does not attempt to fit the data to some preconceived motion of what should be present. We have never observed signals which were not present in the original dataset. If the data is overprocessed real signals are removed; however, this is not usually a problem since assignments are not made based on the absence of data. Processing times are generally comparable to the FT times for the data, permitting treatment of the entire spectrum.

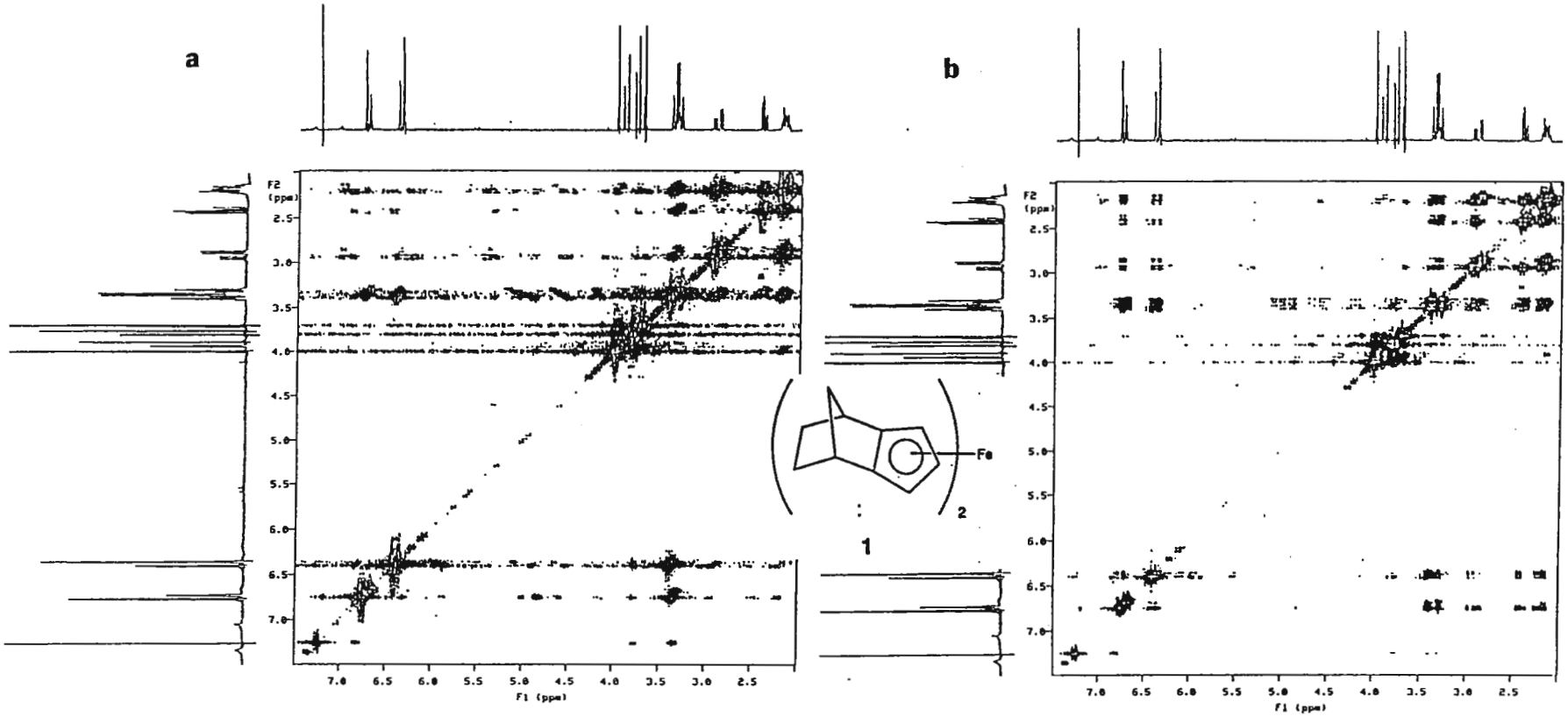
Our current version of the program will only work with data files from Varian's Sun based VNMR software. We are considering methods of distributing this program; if you are interested in receiving a copy, mail me a note and we will add your address to a mailing list.

Sincerely,

James K. Hardy
Associate Professor

Peter L. Rinaldi
Associate Professor
Director of Molecular Spect. Lab

Figure. COSY spectra of compound 1: a) before PCA; b) after PCA.



Accurate Determination of ^{1}H Chemical Shifts of Strongly Coupled ^{1}H Resonances by ^{1}H { ^{13}C } HMQC-COSY

C. Abeygunawardana and C. Allen Bush
Department of Chemistry and Biochemistry, Univ. of Maryland
Baltimore County Campus
Baltimore, Md. 21228
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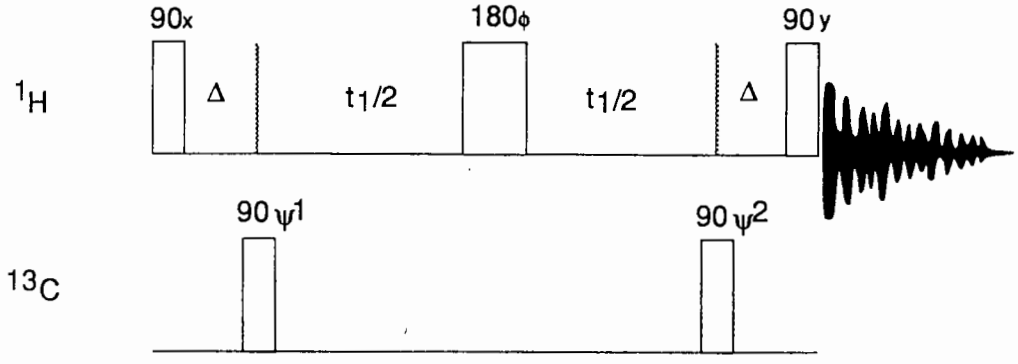
(received 3/20/90)

The chain of vicinally coupled ^{1}H spins extending from the anomeric proton around the ring in carbohydrates provides a convenient scheme for ^{1}H assignments by spin correlation (COSY). Unfortunately, the similarity of the chemical shifts of the methine protons in carbohydrates results in the common occurrence of strongly coupled ^{1}H resonances, preventing successful execution of this scheme. A similar problem may arise in the assignment of the side-chain proton resonances of peptides and proteins, a matter of growing importance in the determination of the complete conformations of small proteins from NOESY data. Two-dimensional HoHaHa spectroscopy (a.k.a. TOCSY) is valuable in this case since the cross peaks of strongly coupled resonances can be far from the diagonal. This method can be used to detect the existence of strong coupling but does not always provide accurate chemical shifts for the strongly coupled protons. In heteronuclear 1-bond correlation of ^{13}C with ^{1}H resonances by the HMQC method, the strong coupling of the protons is absent due to large 1-bond J_{CH} and the low natural abundance of ^{13}C. Since the sugar ring detected in the experiment has a single ^{13}C, the ^{1}H resonance of the proton bonded to it is well separated from the resonances of the neighboring protons which are bonded to ^{12}C. It is possible to combine the large value of $^1J_{CH}$ to remove strong coupling with the $^3J_{HH}$ connectivity to get accurate assignments within a single heteronuclear NMR experiment.

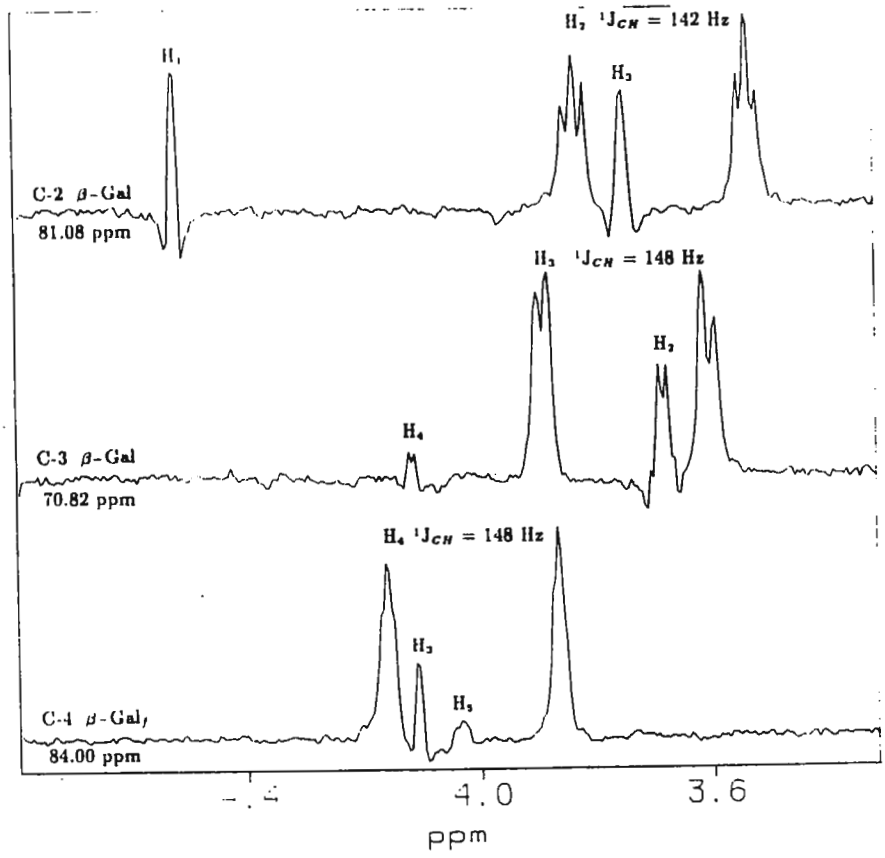
Gronenborn et al (1989) introduced HMQC-COSY for ^{15}N labeled proteins. We have adapted this method to ^{13}C with the single change that we do not decouple at the heteronucleus frequency. The pulse sequence used on the GN-500 spectrometer is:



In this experiment we see crosspeaks at each ^{13}C frequency (F_1 dimension) which relate a carbon atom to the ^1H resonance of a proton connected by one bond which is split by the large $^1J_{CH}$ coupling as well as a cross peak to the proton vicinally ($^3J_{HH}$) coupled to it, which is not split by CH coupling. Since the two resonances are well separated in the ^1H dimension, the chemical shifts of both may be accurately assigned. In case there are three ^1H resonances all strongly coupled, a situation not uncommon in sugars, one must consider cross sections at three different ^{13}C frequencies to get the accurate chemical shifts of all three protons. It will not escape the attention of the alert spectroscopist that this technique is related to the 3-dimensional COSY-HMQC method in which COSY planes are separated by the ^{13}C chemical shifts of directly bonded atoms. The method described here is less demanding of data storage and processing time.

The figure shows an example from the spectrum of a bacterial polysaccharide from *Streptococcus sanguis* C-104 having five sugars and one alditol phosphate in the repeating subunit. In the cross section at the ^{13}C frequency of C-2 of a β-galactopyranoside, the H-2 peak is split by $^1J_{CH}$ =142 Hz and peaks are also detected for the H-1 and H-3 resonances. Likewise the cross section at the ^{13}C frequency of C-3 of the same residue shows the H-3 resonance split as well as the H-2 resonance and a signal at H-4 with intensity reduced due to the small $^3J_{H3-H4}$. A cross section at the C-4 resonance of a β-galactofuranoside the assignment of which has been especially difficult shows a split signal assigned to H-4 as well as resonances assigned to H-3 and H-5 which are not split.

reference: A.M. Gronenborn, A. Bax, P.T. Wingfield and G.M. Clore (1989) FEBS Letters. **243**, 93-98.



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University Chemical Laboratory
Lensfield Road
Cambridge
CB2 1EW

9April 1990
(received 4/19/90)

Prof B L Shapiro
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto CA 94303, USA

Dear Barry

A Reverse Approach to ^{1}H NMR of Microbial Polysaccharides

The proton spectra of complex microbial polysaccharides are a mess. All the interesting signals are squeezed into 2 ppm so they overlap impossibly. In our hands, COSY and TOCSY are not much help, but proton-detected C-H correlated spectra (1) do separate out the signals beautifully (Fig. 1). Of course, this is only useful if you know all the carbon assignments; fortunately we have done carbon-COSY on biosynthetically labelled material so we have complete assignments from a few unambiguous starting points (2). So now we have all the proton assignments too (3) and can interpret the NOESY spectra to get conformations (we hope!).

This strategy, developed by my graduate student David Jones, (Fig . 2) uses two different patterns of carbon labelling: the COSY used 100% labelled glucose (all six positions) to get lots of coupled carbon pairs, heavily diluted with natural abundance material for economy. The reverse correlation used the same overall labelling level, but in the form of 14%-uniformly labelled sugar so that there are intense carbon-13 satellites to aid detection but few C-C pairs to create confusion.

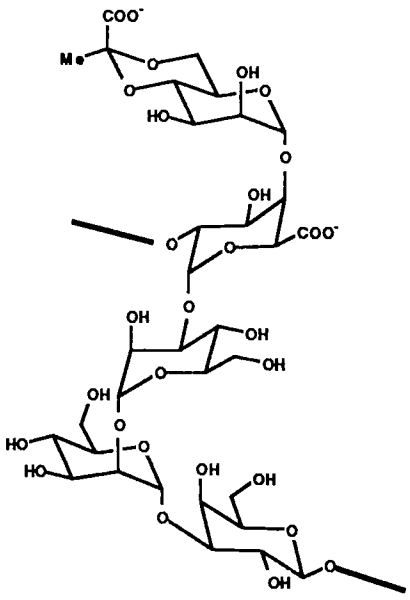
With best wishes,

Jeremy

(1) *Magn. Reson. Chem,* 1988, **26**, 867-875.

(2) *JCS Chem. Commun.*, 1989, 167-169.

(3) *Carbohydrate Research*, in press.



Klebsiella K3 Serotype Polysaccharide

Fig. 1 400 MHz ^{1}H-detected CH correlation spectrum of native *Klebsiella* K3 polysaccharide

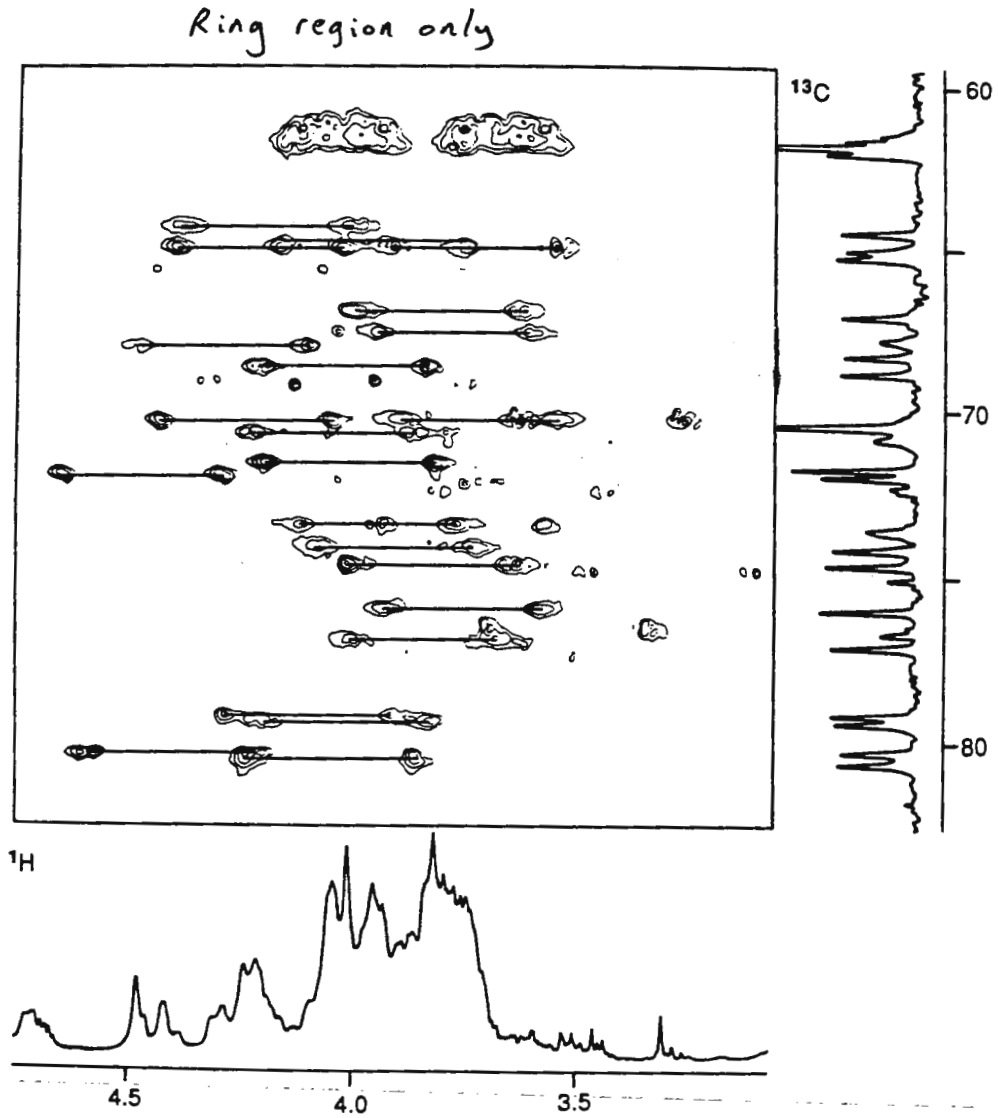
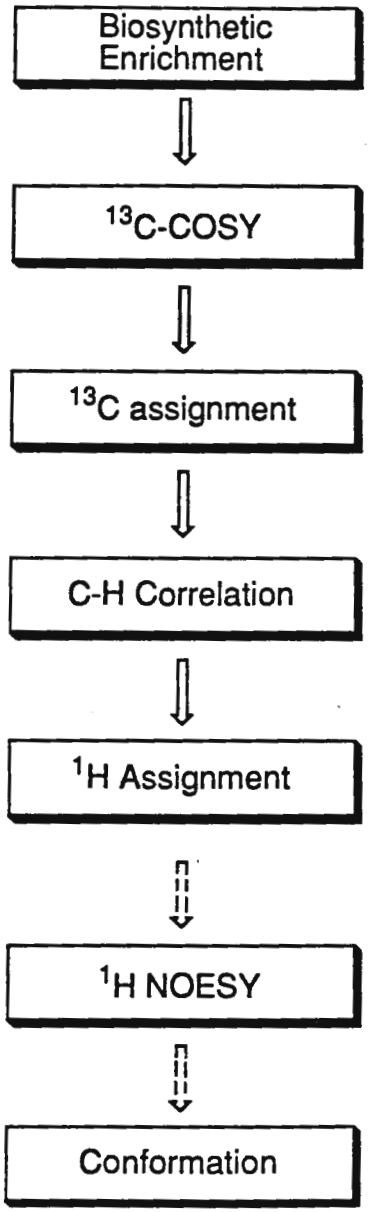


Fig. 2 'Reverse' strategy



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UNOCAL 76

April 19, 1990 AR 90-48
(received 4/20/90)

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

Dear Professor Shapiro:

CHOICE OF LONG-RANGE ^{13}C-^{1}H CORRELATION EXPERIMENT

Long-range ^{13}C-^{1}H chemical-shift 2D NMR spectroscopy has attracted considerable interest in the recent years. It not only provides indirect information about carbon-carbon connectivities (including across heteroatoms) but also helps assign quaternary carbon resonances.

An almost bewildering number of papers have, however, appeared in the literature that describe modifications to the original COLOC and the standard heteronuclear chemical-shift correlation experiments. Most of these modifications involve using various combinations of BIRD and TANGO pulses resulting in pulse sequences that often contain two or more delays that must be carefully optimized. A timely review of this subject has appeared (G. E. Martin and A. S. Zekter, Magn. Reson. Chem., 26, 631, (1988)).

We recently characterized an unusual organolutetium complex by NMR. This novel complex gives 21 ^{13}C resonances consistent with the presence of 21 non-equivalent carbons making up the two ligands about the metal center. The corresponding ^{1}H NMR data also reflects the complete asymmetry of the room temperature conformation adopted by the complex in benzene-d6 solution, Figure 1. In the process of complete assignment of all the ^{1}H and ^{13}C resonances, we had the best results with the long-range ^{13}C-^{1}H correlation pulse sequence described by Krishnamurthy and Nunlist (J. Mag. Res., 80, 280,(1988)). This sequence eliminates the need for the additional "refocusing" delays thereby avoiding the danger of accidentally missing cross peaks, Figure 2. The range of coupling constants present in this system clearly frustrates the optimum choice of such delays present in other pulse sequence modifications. The decrease in signal-to-noise (by a factor of 2) is hardly a "sacrifice" when one also considers the time needed for such optimization.

Please credit this to the account of Dr. P.S. Iyer.

Yours Hastily,

Matthew B. Zielinski

Pradeep S. Iyer

cc: G. C. Graham
J. W. Jost

Figure 1. 2D ^{13}C-^{1}H HETERONUCLEAR CORRELETION (XHCORR) SPECTRUM OF COMPLEX IN BENZENE-D_6.

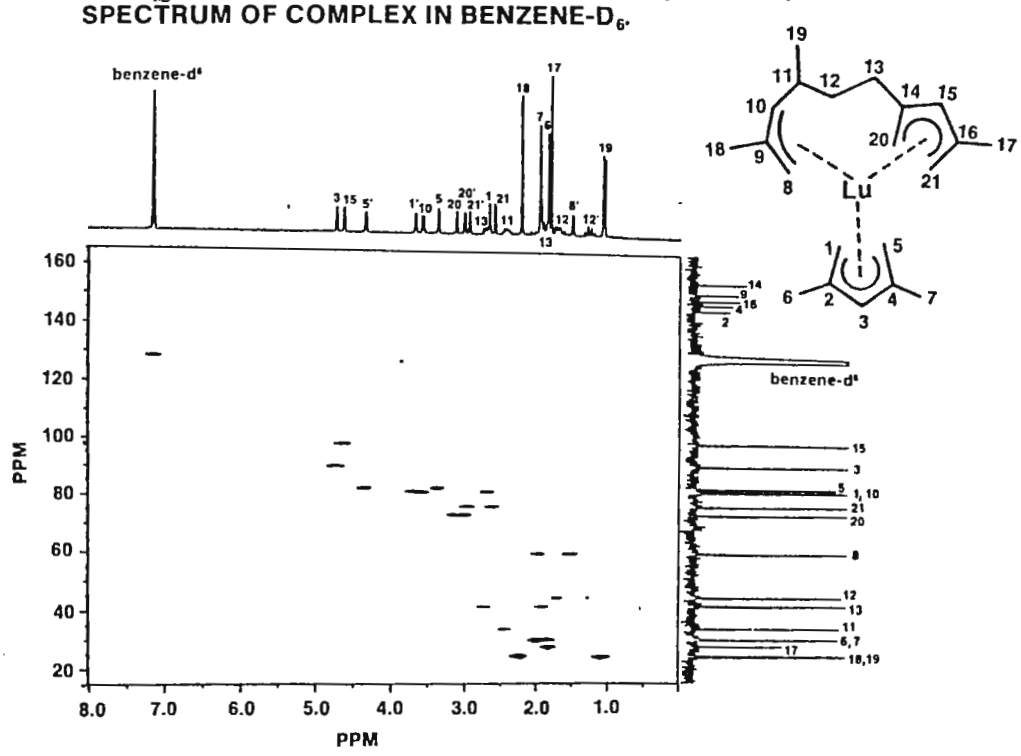
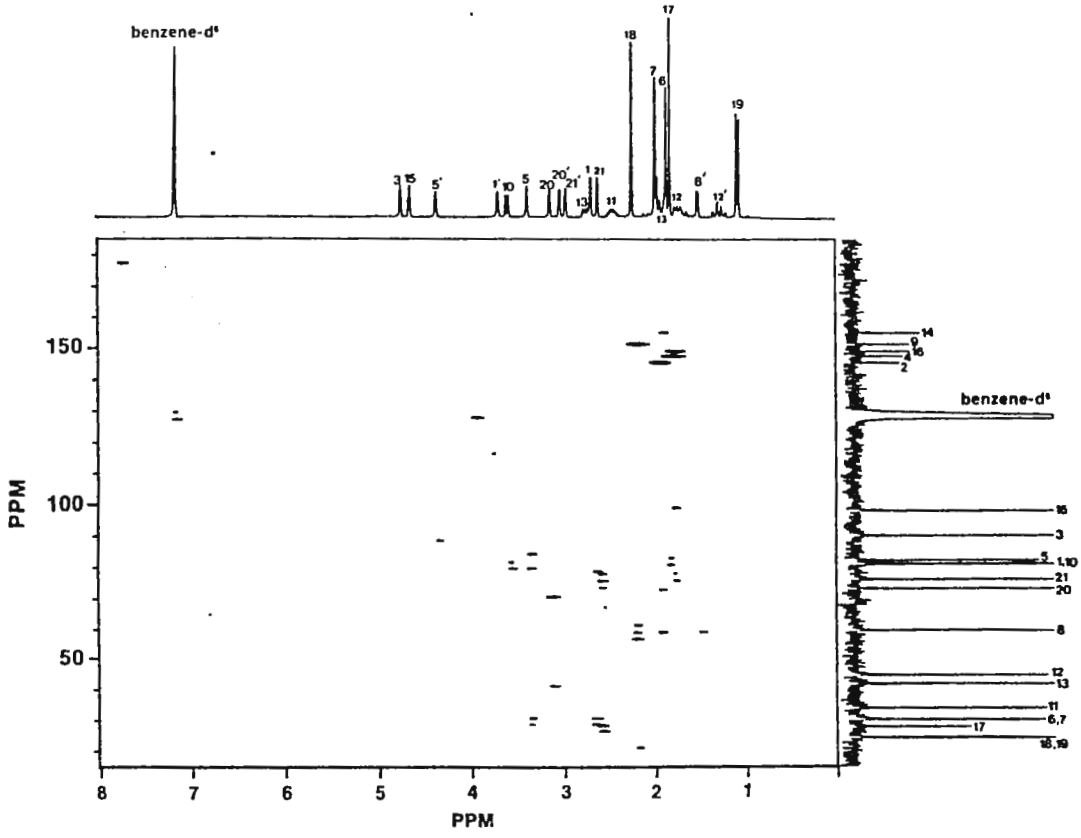


Figure 2. 2D ^{13}C - ^{1}H LONG RANGE HETERONUCLEAR CORRELATION SPECTRUM



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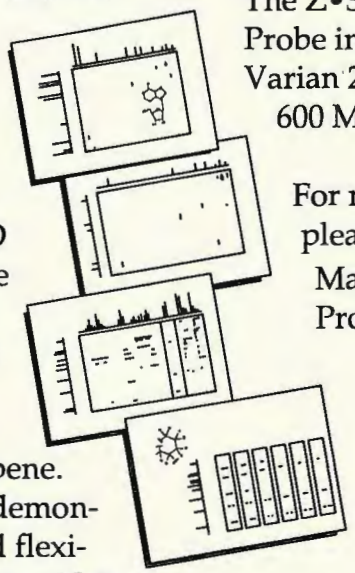
The Z•Spec family of ID probes can make routine work of your samples. Shown on the next page are HMQC $^{1}H/^{13}C$ correlations of Adenosine and a Marine Sesquiterpene. These 500MHz spectra demonstrate the sensitivity and flexibility of Z•Spec ID probes. Note the full chemical shift detection in F1 which is facilitated by the X channel PW90 of 10μ sec for ^{13}C.



TECHNICAL:

The Z•Spec Indirect Detection Probe interfaces directly to any Varian 200, 300, 400, 500 or 600 MHz spectrometer.

For more information, please contact Toby Zens, Manager of the Z•Spec Products Group.

NCC

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837 Arnold Drive, Suite 600, Martinez, CA 94553
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Z·SPEC Indirect Detection Spectra

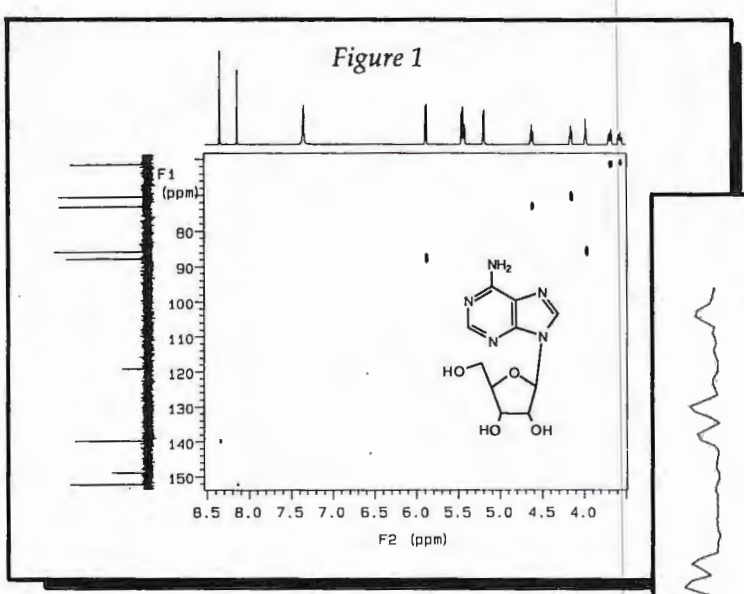
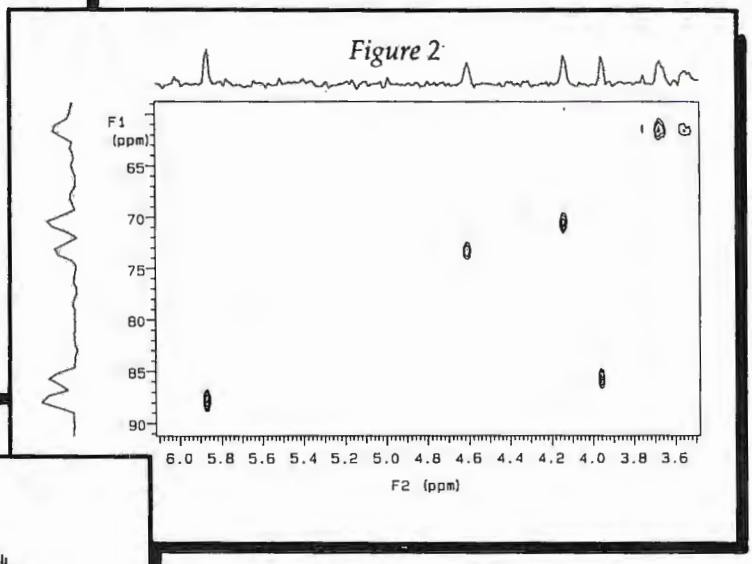


Figure 1: Z•Spec ID probe, HMQC experiment run on a 1 mg/ml $DMSOd_6$ solution of Adenosine. Full chemical shift region, PW90=10 μ sec ^{13}C, 8 transients/FID, total time 19 minutes.

Figure 2: HMQC Ribose region Adenosine, 125 μg/ml. Total acquisition time 2.2 hours, nt = 224.



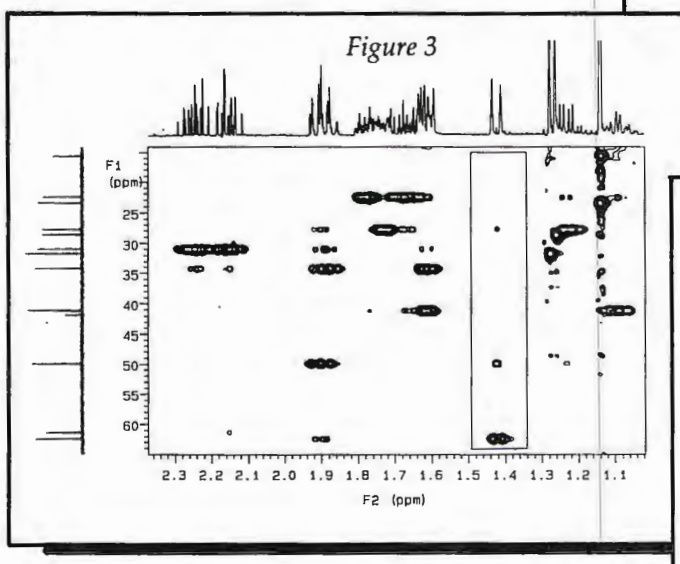


Figure 3: HMQC/TOCSY experiment run on a 8 mg/ml solution of a Marine Sesquiterpene decomposition product. Total acquisition time 2 hours, 10 minutes.

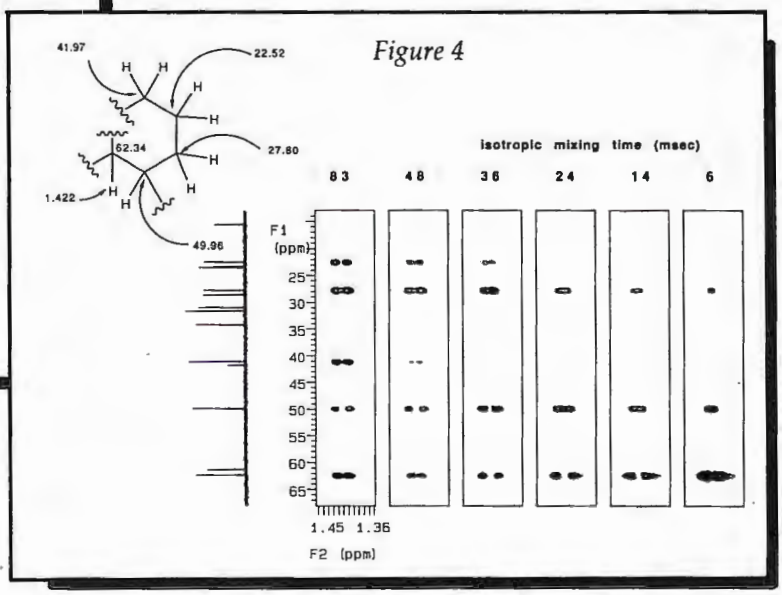


Figure 4: Expansions at the 1.4 ppm proton resonance showing the propagation of magnitization to the carbons with increasing mixing time. Note the excellent F1 phase characteristics throughout.

NALORAC CRYOGENICS CORPORATION
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The University of Akron

Department of Chemistry (216) 375-7372
Buchtel College of Arts and Sciences
Akron, OH 44325-3601

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

April 12, 1990
(received 4/16/90)

Dear Barry:

In our laboratory we have been doing a lot of exciting work with 3D-NMR and principal component analysis for digital filtration of 2D-NMR data. This work would not have been possible without the extensive use of the modern computer hardware and software facilities which we have put together in our laboratory. TAMUNMR readers might be interested in a similar network to facilitate data processing and make more instrument time available.

Our network, based on Ethernet, is outlined in the Figure. The NMR instrumentation is all located in one laboratory, but the computer workstations are dispersed in three locations within our building. With this network and a very basic knowledge of Unix, it is possible to access all the data and hardware on the network from any location. As experiments are completed on the instruments, it is possible to pull the data off to a computer on the other side of the building for processing. Users on any system can spool output to a Postscript printer, an HP Laserjet printer and an HP plotter at one location in the instrument laboratory rather than having to purchase $10,000 worth of output devices for each system.

Each computer system contains its own 300 Mbyte disk for the basic Unix file system, Unix utilities, kernel, and virtual memory. All of the systems use Sun's NFS to mount ca. 2 Gbytes of disk storage for users' files so that they can log in on any system and see the same home directory. This eliminates the need to move and duplicate large 2D and 3D files on different workstations. All the systems access 60 Mbyte 1/4" tape and 600 Mbyte read/write (reusable) optical disk drives for backup and data storage.

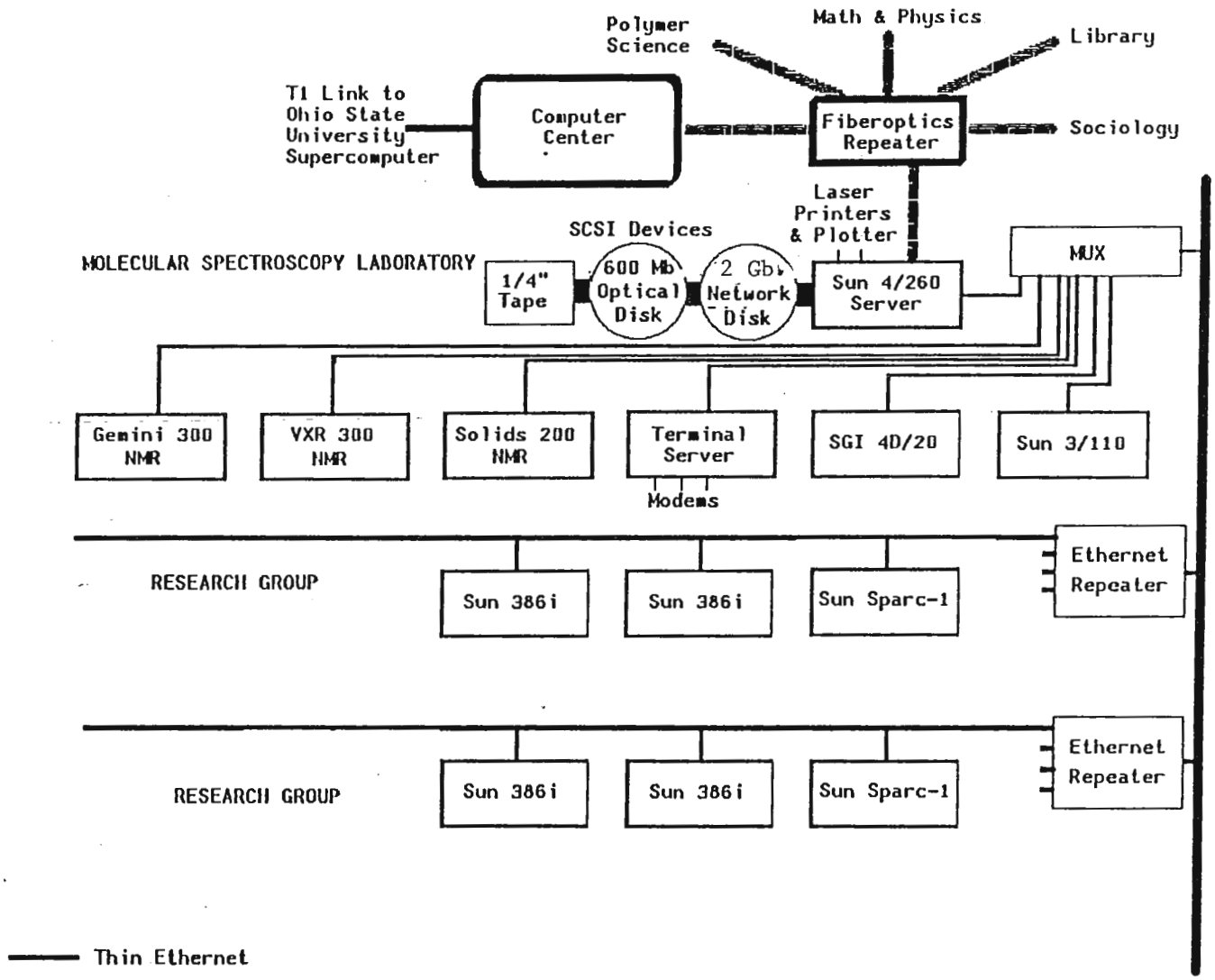
With this hardware configuration it is possible to access all of the hardware and software resources on the network simultaneously from the console of a single workstation. We can utilize desktop publishing software to write papers in one window; in a second set of windows we startup an NMR program to process data and produce a plot to an output file which can be inserted within our manuscript without ever touching a piece of paper; and in a third window we use statistical analysis software to generate tables and graphs of data.

After investing the time to setup the core of the network it has been possible to add new workstations which have complete access to all the facilities desired in less than an hour. The system has tremendously enhanced our research capabilities, and has allowed us to at least double the data output of our NMR spectrometers through the use of offline processing. The price of workstations with capabilities similar to our most powerful Sparcstations is now comparable to the price of PC's 3-4 years ago (i.e. $3-6k). As the number of these workstations increase, less expensive software will make systems like ours available on every chemist's desktop.

Best regards,

Peter L. Rinaldi, Associate Professor

Schematic of the Department of Chemistry computer network and its link to the University of Akron campus and the outside world.



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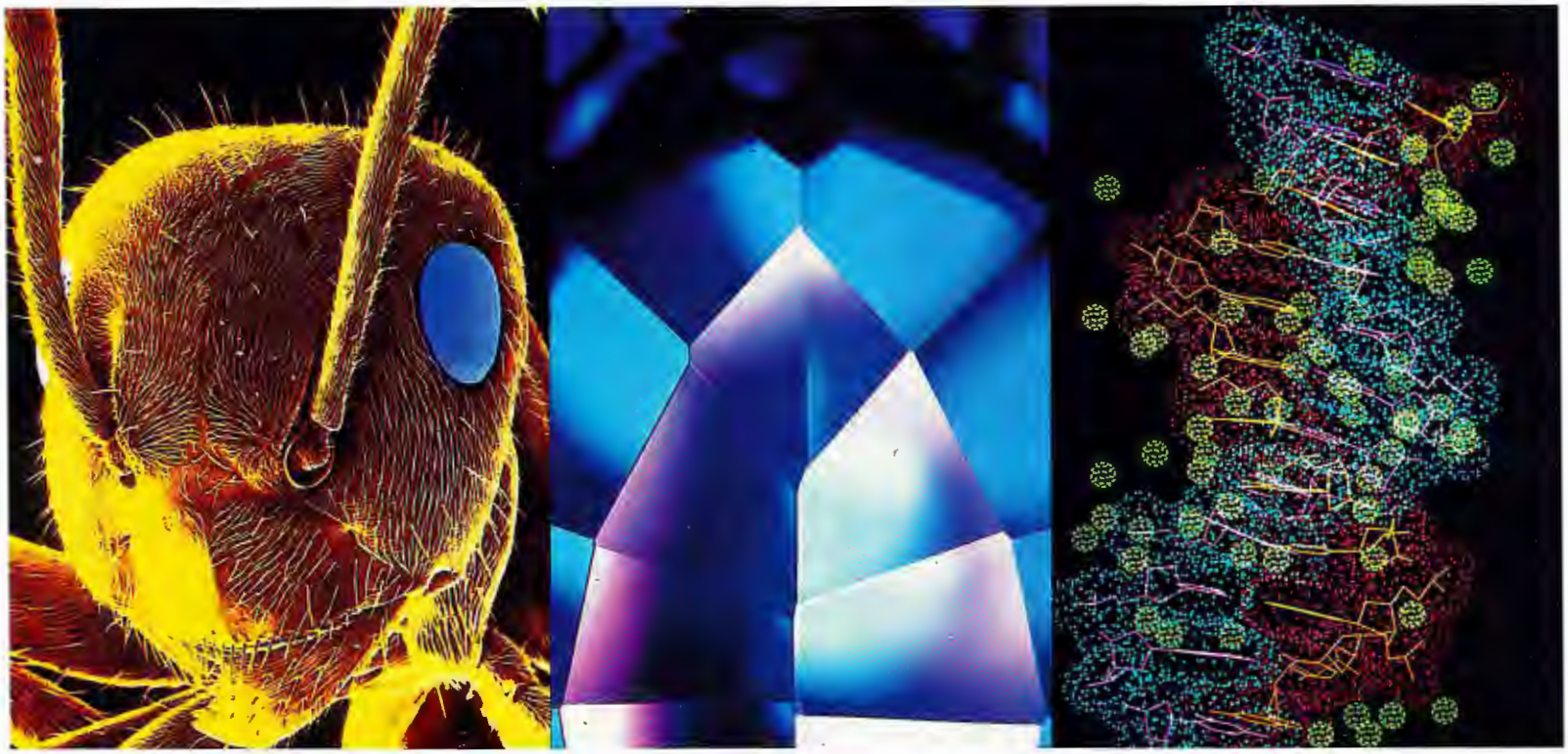

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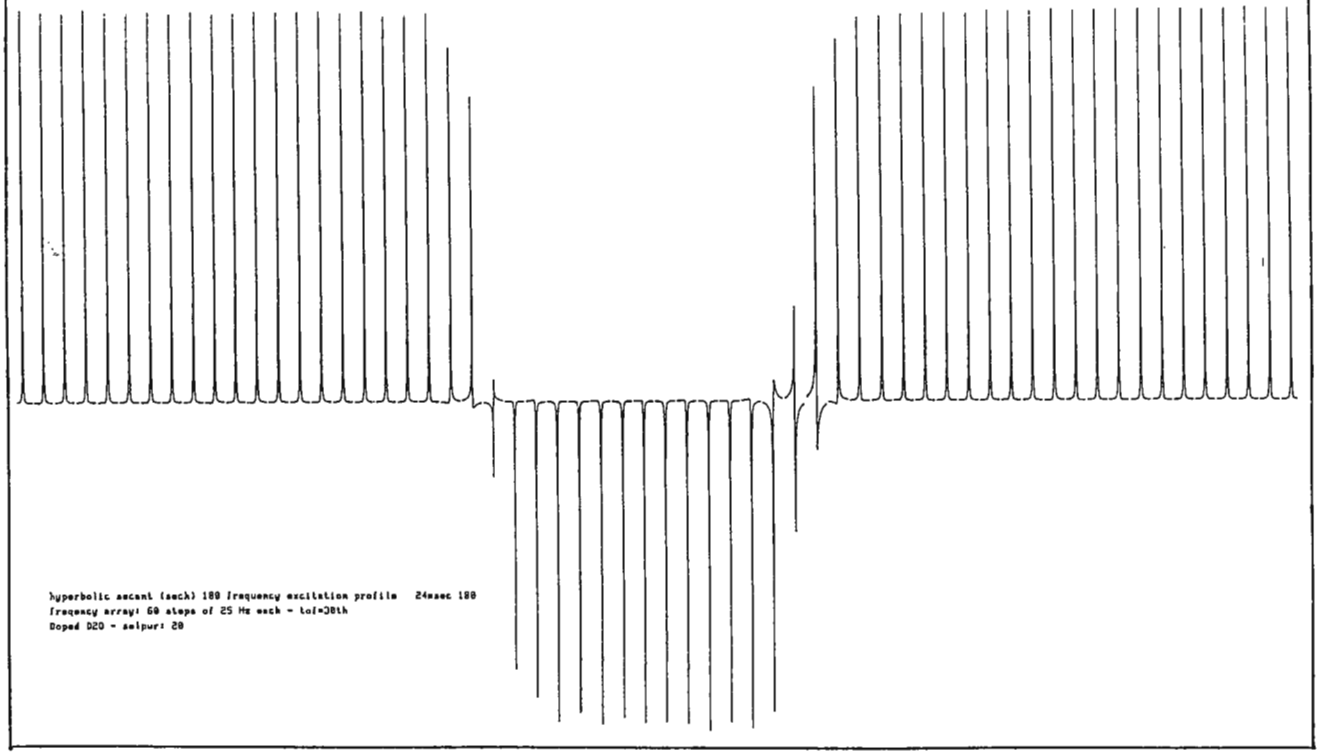
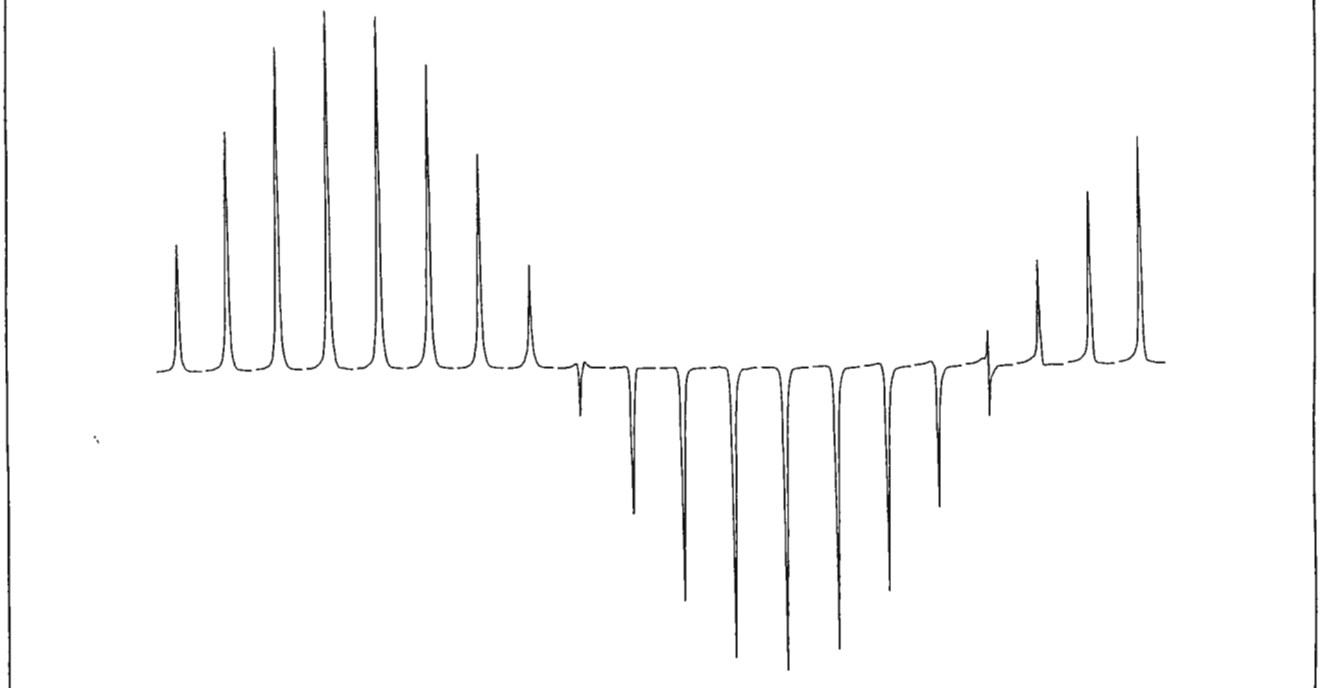
Use of the hyperbolic secant places strong demands on the pulse modulation hardware. UNITY's Waveform Generator performs both amplitude and phase modulation, and in particular, uses the 0.5 degree phase increments necessary for this pulse excitation. In this example for doped water, the offset dependence is explored in 25 Hz steps for a semi-selective inversion(24 ms). Note the flatness of the inversion, the excellent phase characteristics and its specificity.



SHAPED PULSES: BROADBAND EXCITATION

Hard "square" pulses have been the building block of high resolution FT NMR since its inception, however, hardware limitations may have been the driving factor. With the UNITY waveform generator, shaped hard pulses are a real possibility; these may offer advantages for broadband excitation. Shown below is a 32-step gaussian pulse width array. Each spectrum represents an increment of 6.4 usec in pulse width using a generic gaussian pattern to drive the waveform generator. This represents a 27 usec 90-degree pulse width, and a minimum excitation pulse width of only 6.4 usec! (Using doped D2O on a UNITY 500 with a 5mm indirect probe.)



KARL-FRANZENS-UNIVERSITÄT GRAZ
Institut für Organische Chemie

A-8010 Graz, March 8, 1990
Heinrichstraße 28
Tel. (0316) 380 5321

(received 3/21/90)

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

A simple gradient driver for diffusion measurements with pulsed gradients on a Gemini 200

Dear Dr. Shapiro,

Our recent change of equipment made it necessary to construct a gradient driver for a Varian Gemini 200 system. Until now our measurements of self diffusion coefficients were performed with a XL-200 using a blown up homospoil pulse to generate the gradient pulses. Unfortunately, the Gemini system does not provide such homospoil pulses.

The gradient driver we use now consists only of an optocoupler driving a power transistor, current delimitation is done with power resistors (fig.1), connected to a standard 5V/2A power supply. This driver is similar to those presented in refs.1,2.

The pulses used to switch the gradient driver are taken from the Gemini's "spare line", which can be found at plug P3, pin 3 on the U-Pulse Controller board. This "spare line" is provided for computer control of external devices, and can be set to high or low during a pulse sequence.

The signal from the spare line is very poor due to a large 40MHz noise and a low signal level. Therefore we use a preamplifier with a simple RC-low pass filter to produce pulses strong enough to switch the optocoupler of the gradient driver (fig.2).

As the spare line of our U-Pulse Controller is high during acquisition, we use inverse signals to switch the preamp: The spare line is set to high at the start of the experiment, it goes down to low for switching the gradient pulses and is high during acquisition. The transistor behind the operational amplifier reinverts the signal, so that switching of the gradient driver occurs at high level. (Note that we use the inverting input of the operational amplifier because of better performance).

The gradient coil is of anti-Helmholtz type and consists of a couple of oppositely wound coils, each one consisting of

40 windings of 0.6mm Copper-wire, with an outer diameter of 43mm and a distance of 17mm. This coil is fixed on a plastic body and connected to a 5mm ^{1}H-probe, the decoupler coil and a piece of the outer probe shield removed.

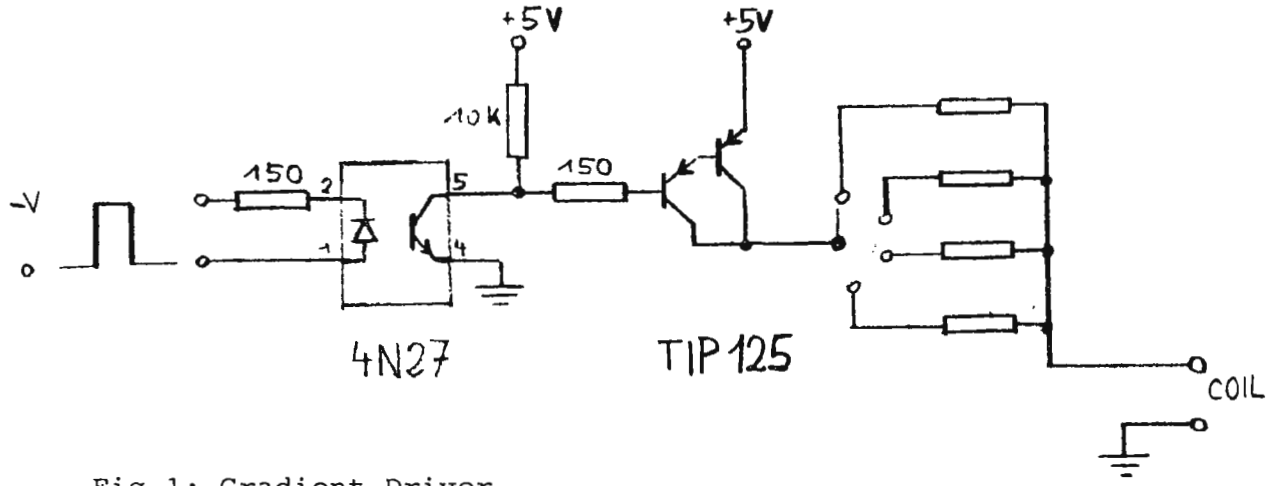


Fig.1: Gradient Driver

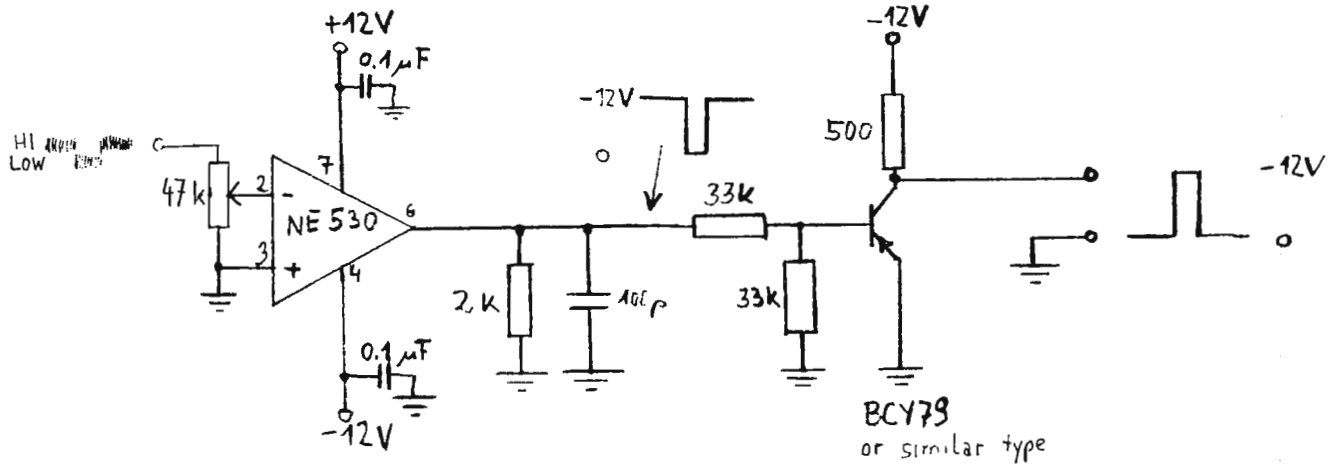


Fig.2: Pre-Amplifier

This arrangement allows excellent measurements of self diffusion with the Gemini. The gradients are rather strong. The gradient driver's power resistors (27 to 9Ω) lead to gradient strengths of 2 to 5.5*10^{-4}Tm^{-1}, with no affection of the system- or magnet stability. We are content with these gradient strength, although the gradient driver would be able to produce even more powerful gradients.

Yours sincerely

(Michael Geringer) (Heinz Sterk)

1.) J.E.Tanner, Rev.Sci.Instr.36, 1086 (1965)
2.) P. Stilbs, Progr.NMR Spectrosc.19, 1 (1987)

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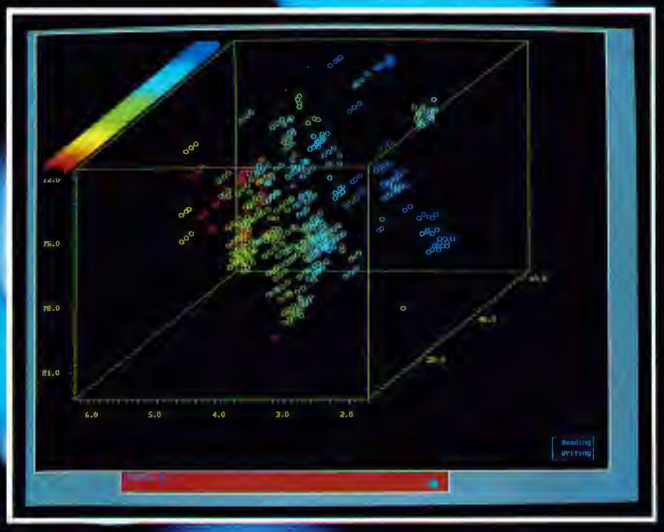

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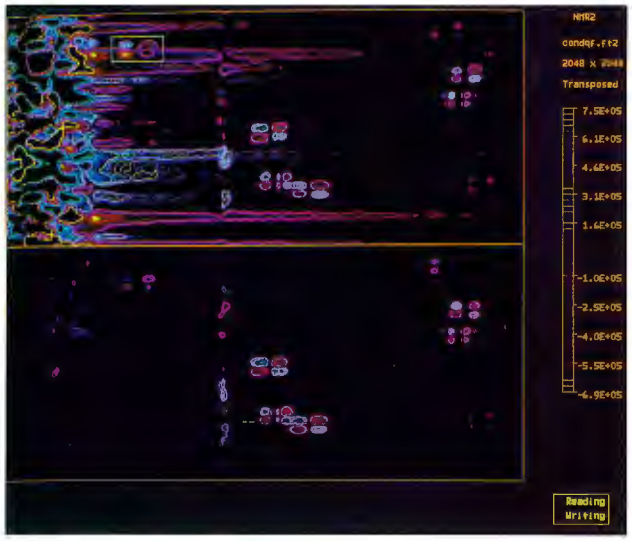

Solvent signal obscures the H-alpha/HN region in the conventionally processed conotoxin spectrum (above); NMR2's solvent filtering method can efficiently suppress solvent signal while retaining spectral peaks (below), leading to an easier and more complete analysis.

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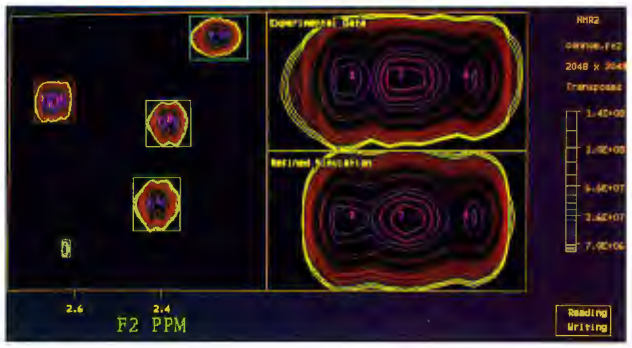
- Real Time Tools for the examination of 1D slices for a 2D data set.
- Flexible User Interface based on the Industry Wide X-window Network Graphics Standard.

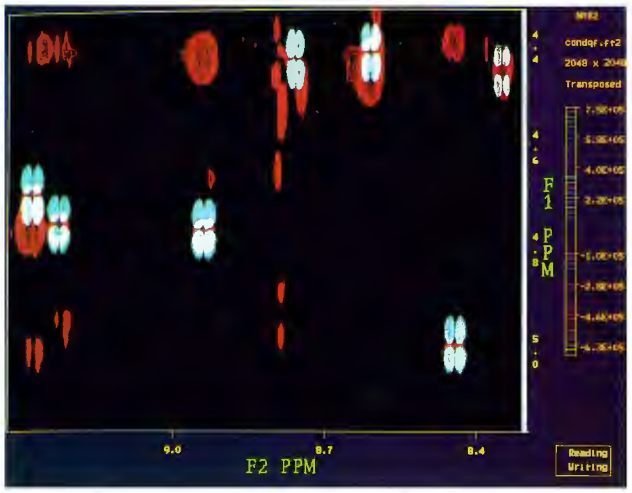
Spectral features can be measured accurately even in situations of severe overlap, using advanced non-linear 2D surface fitting, to produce reliable NOE volumes or coupling constants. Cluster analysis is used to identify each overlap region for fast and effective automated analysis of one or several spectra.



Analysis Capabilities

- Automatic Multidimensional Peak Picking.
- Flexible Peak Table Editing.
- 2D surface fitting for more accurate determination of peak position and volume information, even in highly overlapped regions.
- Automated Clustering of Peaks.
- Macro-oriented Command Language gives the user more flexibility and control of data processing and display.

NMR2 offers flexible methods for comparative spectroscopy, including algebraic combination of spectra, as well as graphical overlays. Shown here is an overlay of corresponding regions from the DQF-COSY (blue) and NOE (red) spectra of conotoxin; peak overlap between the two spectra is shown in white, clearly distinguishing inter- and intra-residue interactions for easier spectral assignment.



Cover Inset *Top: a view of NMR2/MODEL, in an analysis session with a DQF-COSY spectrum of conotoxin. Bottom: the results of a three-dimensional peak pick on a proton-carbon-carbon spectrum.*

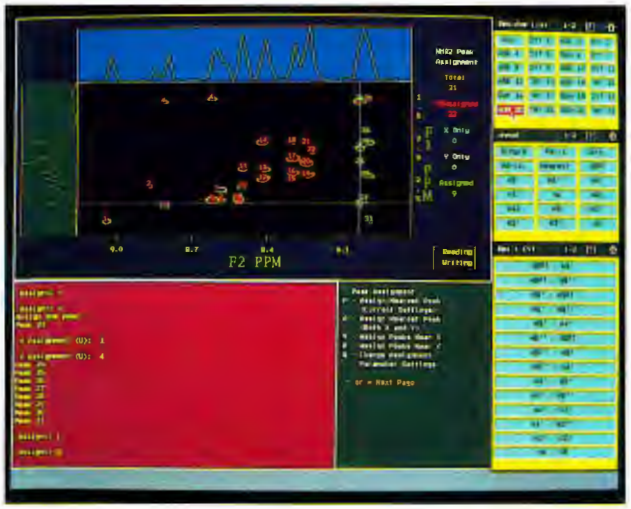
NMRgraf™

NMR2/MODEL™

Assign NMR Data, Extract Structure Information, and Confirm Molecular Structures.

Interactive Tools for Spectral Assignment of 2D Data

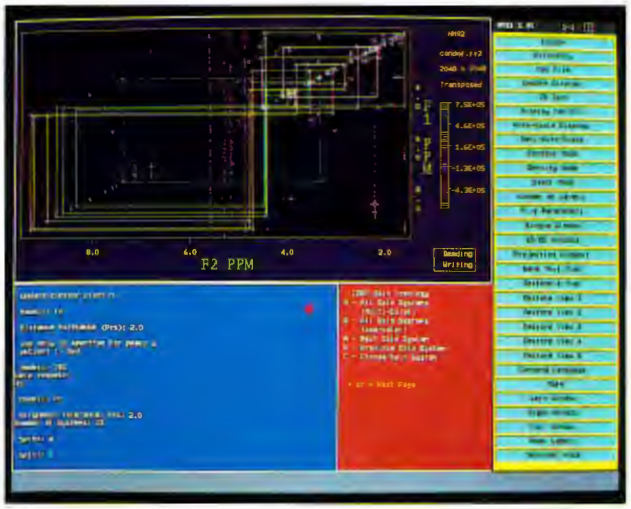
- NMR2/MODEL creates a user database of 2D spectral peaks with associated atom assignments.
- Automated cross peak cluster analysis for consolidating spin-spin multiplets.
- Computer-assisted generation of NOE walks and COSY connectivities.



NMR2/MODEL's general-purpose assignment modules take advantage of molecular structure file information to generate atom lists targeted specifically to the spectrum at hand. In this example, the assignment lists for this DNA NOE spectral region show only those proton pairs likely to give crosspeaks.

Automated Intramolecular Distance Determination

- Distances determined from either NOESY cross-peak volumes or multi-spectra NOE build-up curves.
- Distance and Coupling Constant information is written to a constraint file that can be read directly into NMRgraf or other supported molecular modeling packages.



Many forms of assisted spectral analysis are available, including NOE-walk generation and recognition of COSY AX multiplets and spin connectivities. Shown here is the result of an automated COSY connectivity analysis, with each spin-system outlined in a different color.

Spectral Simulations

- NOESY or DQF-COSY spectra may be simulated from a NMRgraf molecular structure file, or other supported formats including the Brookhaven Protein Data Base format.
- NOESY spectra are simulated using complete relaxation matrix calculations.

Molecular distances can be calculated automatically from single spectra, or from NOE volume-evolution curves, as shown here. Build-up curves can be modeled with splines, exponentials, or user-defined functions.

NMRgraf™ NMR2/MODEL™ NMR2™

Powerful Software for Molecular Structure Determination and Simulation Designed for the NMR Researcher.

Model Building

- Rapid construction of virtually any small organic or biological macromolecule.
- Specialized builders for proteins, lipids, carbohydrates, DNA/RNA, and solvents.
- Molecular structures can also be read in from a database.
- Flexible NMR data entry via data file, keyboard, or "point-n-click" graphics screen.

Prediction Methodologies

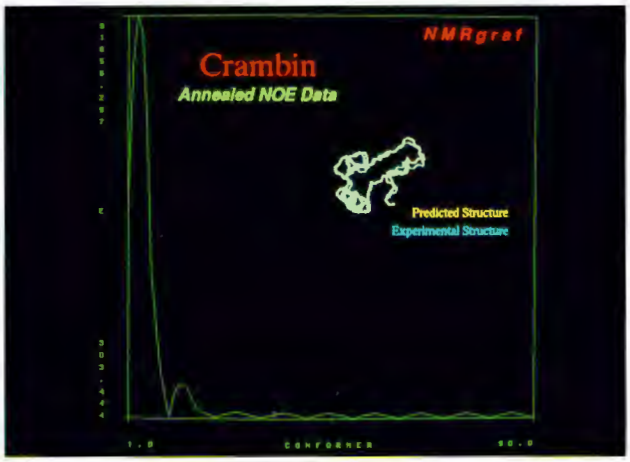
- Multiple pre-defined strategies for predicting structures.
- Full molecular mechanics energy expression incorporating NOE and J-coupling data.
- Highly customizable potential functions supported for creation of novel prediction methodologies.
- Special treatments for disulfide linkages, cis-peptides, D-amino acids, pro-chiral centers, phenyl rings and methyl groups.

Extensive Analysis Capabilities

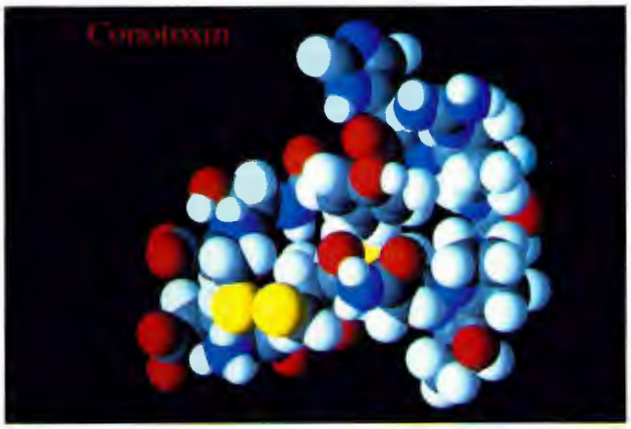
- Graphical display of NOE and J-coupling restraints, energy and force analysis, strained structures, and geometries.
- Statistical Reports on fitting; restraint and distance errors; Φ, Ψ, and Ω dihedral angle analysis; force and energy analysis; and molecular geometries.
- Potential NOE's and J-couplings, automatically generated from candidate structures, can be passed back to NMR2/Model for further analysis.

Interactive and Flexible User Interface

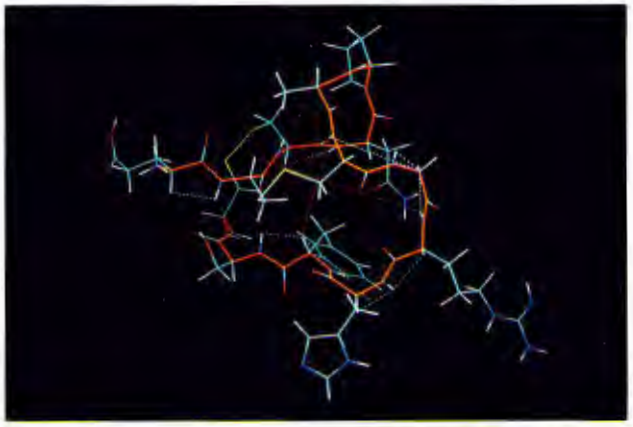
- Menu-driven ("point-n-click") interface for casual and novice users.
- Simple sequential process flow for major operations.
- Macro support for developing advanced applications.
- Complete integration of graphics, prediction, and analysis capabilities ensures data integrity.
- Direct data input from NMR2/MODEL, or any software that generates PDB-compatible structure files.
- Direct interface to BIOGRAF and POLYGRAF molecular modeling programs.



Display of energy vs. conformation number for the automated prediction of the 3-D structure of crambin using NOE data. Note that the convergence of the energy vs. conversion number indicates a plausible 3-D structure of crambin has been found.



CPK rendering of candidate conotoxin structure.



Full 3-D line rendering of candidate structure (shown mid-optimization process) showing satisfied (blue dotted lines) and unsatisfied (red dotted lines) NOE distance restraints. Main chain shown in solid red.

NMR2™ NMR2/MODEL™ NMRgraf™

Coordinated Software for 3D Structure Determination.

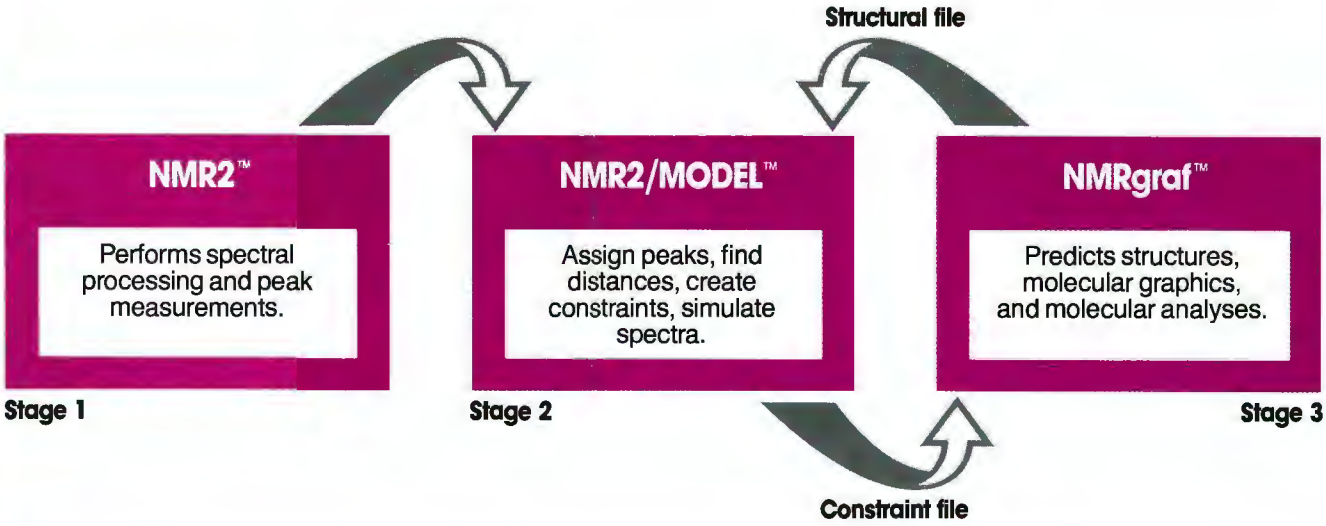
The NMR2™ System joins with NMRgraf™, bringing to scientists a new approach for determination of 3-dimensional molecular structures from NMR spectral data and molecular modeling.

New Methods Research, Inc., world leader in advanced software for NMR data processing and analysis, and BioDesign, Inc., developer of BioGraf™, Polygraf™ and NMRgraf™, present a powerful new approach to NMR-molecular modeling:

- Full automated processing and quantitation of 2D (3D, 4D, . . .) NMR data.
- NMR2/MODEL™ has a suite of data analysis and spectral assignment/data handling tools that eliminate manual tracking of NOESY cross peak information.
- Precise initial distance constraints are automatically generated.
- NMRgraf provides a comprehensive environment for using this information to generate structures *and evaluate* overall and local structural details.
- NMRgraf and NMR2/MODEL work together to help you validate calculated structures, by energy minimization, dynamics calculations, and using NOESY spin simulations (complete relaxation matrix approach).

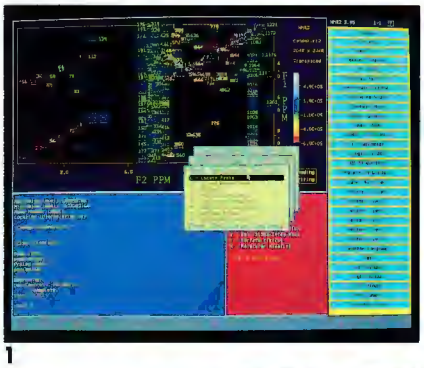
Top-level menu of NMRgraf showing full 3-D line rendering of candidate structure (shown mid-optimization process) with main chain in solid blue.

NMR2, NMR2/MODEL and NMRgraf Let's see how they work together . . .

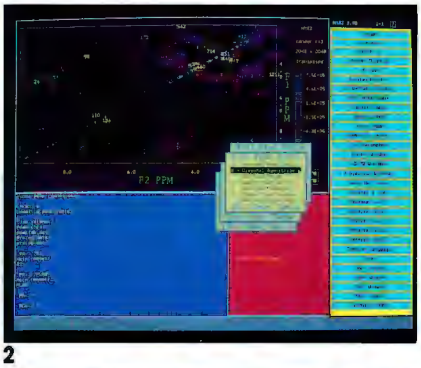


A diagram indicating the coordination of NMR2, NMR2/MODEL and NMRgraf for 3D molecular structure determination.

Views of NMR2, NMR2/MODEL and NMRgraf.



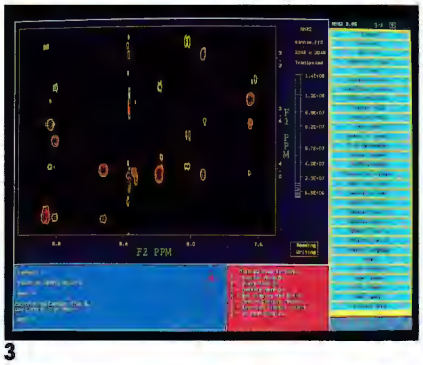
1



2

1. *In the first stage of spectral analysis, NMR2 automatically locates and characterizes all spectral peaks. Shown here is the result of a peak pick, with each peak labeled with its index number; the label colors indicate peak intensity.*

2. *Many operations are available for refining the peak table data. Shown here is the result of peak table symmetrization, which removes noise ridge entries from the peak table without the loss of peak structure information.*



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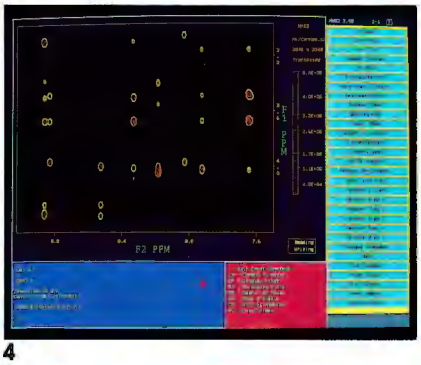
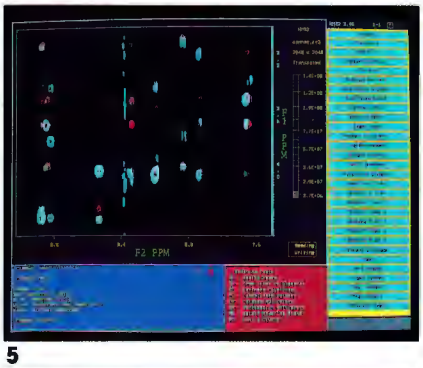


4

3. *This NH/H-alpha region of the conotoxin NOE spectrum is the subject of the NOE back-calculation shown at right.*

4. *A back-calculated NOE simulation of the conotoxin data shown at left. The simulation is based on a relaxation matrix calculation which provides for variable correlation times, to account for molecular dynamics.*

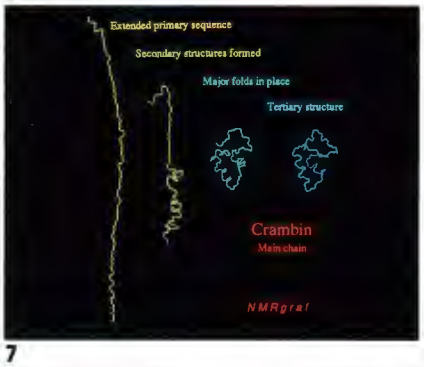


5

6

5. *Comparison of experimental and simulated NOE data provides a powerful tool for confirmation of a proposed molecular model. Shown here is an overlay of the simulated and measured conotoxin spectra; the differences provide valuable information for the continued refinement of the structure and dynamics of the molecule.*

6. *NMR2/MODEL's DQF-COSY simulations can be used to assist assignment procedures and facilitate interpretation of complicated coupling structures. Shown here is the aromatic region of a tryptophan spectrum (top), with a corresponding COSY simulation.*



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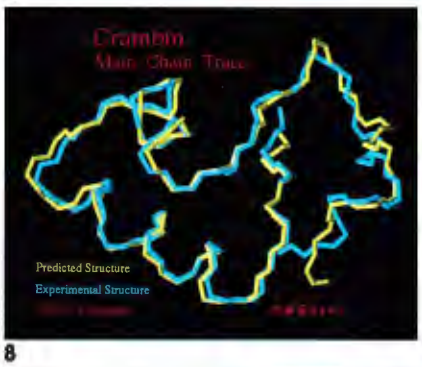

8

7. *Snapshots of the main chain of crambin as it is folded from its primary sequence into its proper tertiary structure by NMRgraf. The trajectory utilized a proprietary scheme, NOE Annealing, that incorporates both experimental NMR data and molecular modeling techniques to predict complex molecular structures.*

8. *Comparison of the predicted structure of crambin (main chain atoms only) with the native structure obtained from X-ray crystallography. The NOE data set of approximately 600 interatomic distances was generated from the experimental structure. The RMS differences for the main chain atoms is 0.9 angstroms.*

NMR spectral data and parameters from all manufacturers are converted into a common open file format.

Supported platforms: NMR2, NMR2/MODEL and NMRgraf can operate on a single computer, or the processing can be distributed across a UNIX, VMS or heterogeneous computer network. NMRi and BioDesign provide turnkey solutions in many configurations, with user-transparent formatting of data and sharing of computational resources.

All of these programs are supported on the following workstations and computers: Sun, VAX, Silicon Graphics, and Stardent Titan series systems. NMRgraf additionally runs on Alliant and Convex systems. NMRi software additionally runs on IBM RISC System/6000, Stardent GS series, DECstation™ and DECsystem™ computers.

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NORTHWESTERN UNIVERSITY DEPARTMENT OF CHEMISTRY

Joseph B. Lambert
Professor

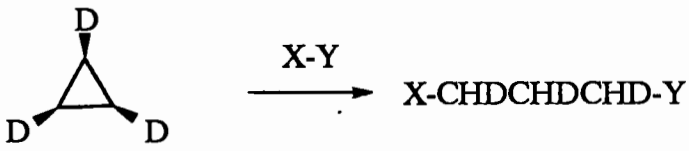
2145 Sheridan Road
Evanston, Illinois 60208-3113
Telephone (708) 491-5437

(received 4/20/90)

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

Dear Barry:

We have found NMR to be indispensable in our analysis of the electrophilic ring opening of cyclopropane. To determine the stereochemistry of both the electrophilic and the nucleophilic steps, we prepared cyclopropane-*cis*-1,2,3-d_3 and allowed it to react with bromine, chlorine, or mercury(II) acetate. For Br_2 and Cl_2, the products would be



meso from double inversion and dl from retention/inversion. For $Hg(OAc)_2$, they would be erythro from double inversion and threo from retention/inversion.

These products can be distinguished by the temperature dependence of the vicinal ^1H-^1H couplings. For all three undeuterated products (see figures on the next page: **a** is X=Y=Br, **b** is X=Y=Cl, **c** is X=BrHg, Y=OAc) the couplings decrease with lower temperature, as expected when the gauche-gauche conformer is stablest. For Br_2 and Cl_2, the couplings also decrease with lower temperature for the deuterated materials (not shown). This is the expected behavior for retention/inversion. In contrast for $Hg(OAc)_2$, the couplings for the deuterated material increase with temperature at both ends of the molecule (see **d**). This behavior is expected for double inversion.

Retention/inversion stereochemistry is characteristic of edge attack by the electrophile, and double inversion is characteristic of end attack to form an unsymmetrical corner adduct. Thus halogen and mercury appear to exhibit different mechanisms.

Sincerely,

Joseph B. Lambert

Erik C. Chelius

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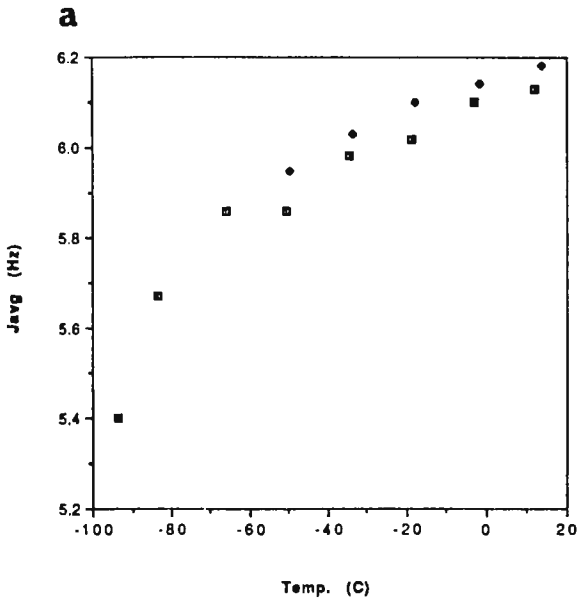
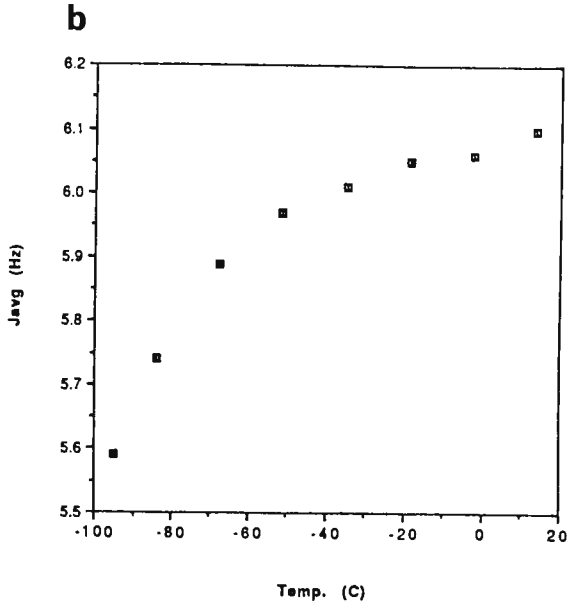
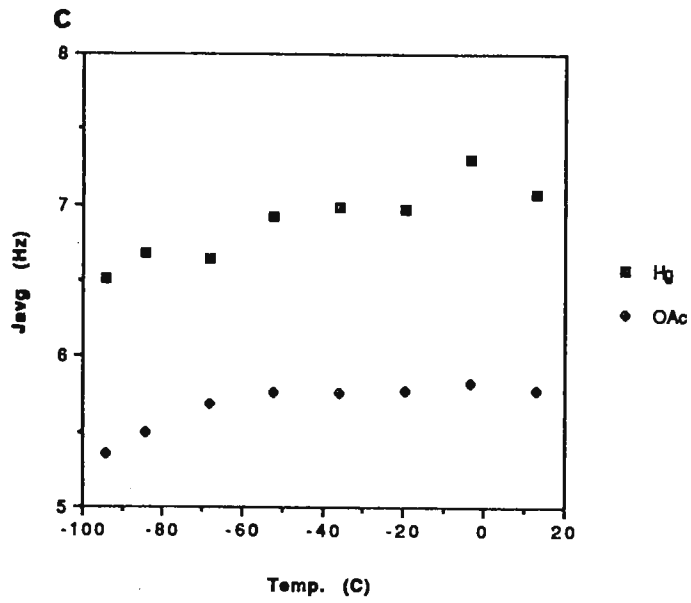
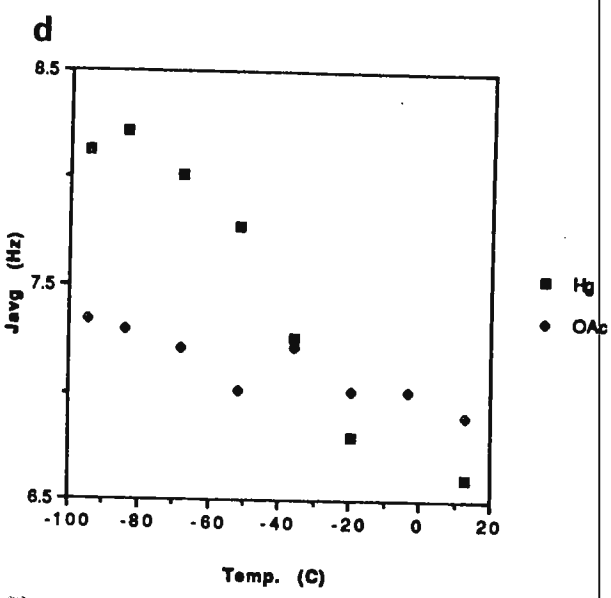

a
Javg (Hz)
Temp. (C)


b
Javg (Hz)
Temp. (C)


c
Javg (Hz)
Temp. (C)
Hg
OAc


d
Javg (Hz)
Temp. (C)
Hg
OAc

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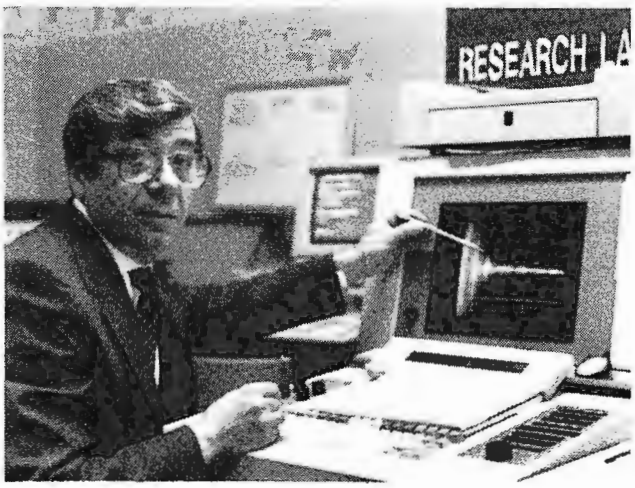

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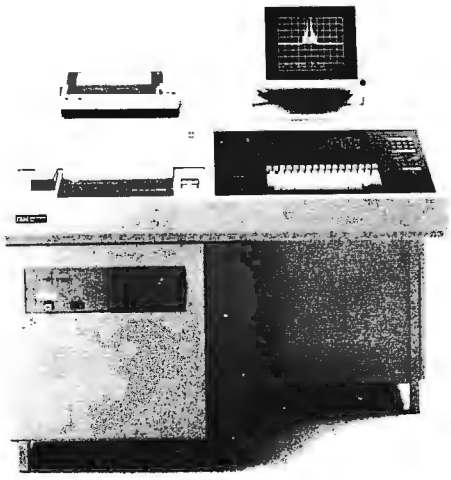
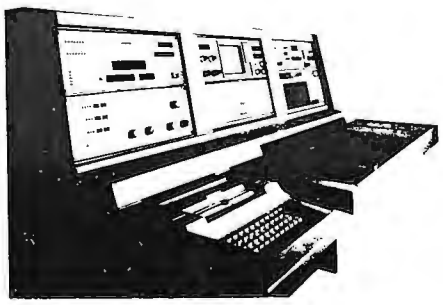
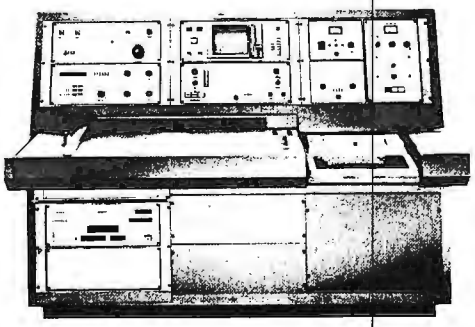


Bruker AC-200



Bruker WM-360



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2600 GA Delft, The Netherlands

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

March 12, 1990
(received 3/21/90)

"Kicking the main magnet coil . . ."

Dear Dr. Shapiro,

Recently a horizontal bore (20 cm) 6.3T Oxford Instruments magnet system has been installed in our laboratory. It has been in operation for about four years at the Philips Research Laboratories in Eindhoven and to us it is a welcome increase in capacity for our localized in vivo spectroscopy program. Since the magnet is equipped with conventional gradient coils (free access 13.5 cm, gradient strength 2G/cm) the first thing we had to do was to readjust all eddy current compensations. The people at Philips developped a compensation method (1) which turned out to work also satisfactory at our site. In their method the eddy current fields are modelled as a number of exponentials, some with the spatial dependence of the switched gradient and others with different spatial dependences. With this method we obtained good results with the transverse gradients by applying the usual preemphasis on the gradient itself as well as a multiexponential decay on a Z_0 coil. In the case of the Z gradient the situation was somewhat different: after applying the correction on the gradient itself a time dependent field shift remained with a maximum of 30 Hz at about 150 ms after switching off the gradient (Fig.1).

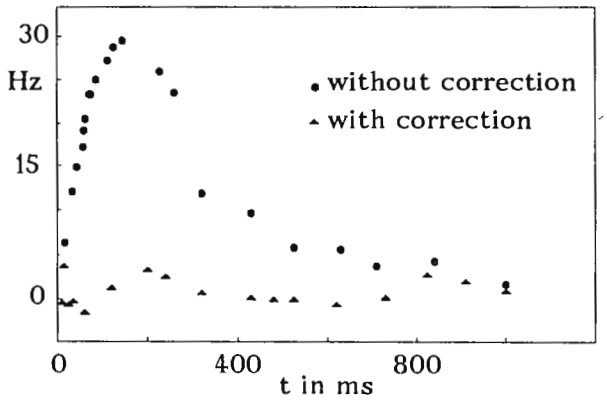


Fig. 1 The frequency shift as a function of time after switching off a Z gradient of 1 G/cm.

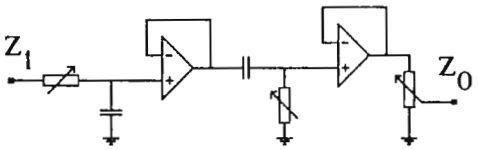


Fig. 2 The compensation circuit to correct the second order $Z_1 \rightarrow Z_0$ respons.

This shift is not negligible compared to the linewidth of 7 Hz in measurements without gradient and compared to the achievable linewidth in in vivo experiments. It appeared that the curve could not be fitted with exponentials to yield a reasonable correction so we looked for another mechanism to understand it. A simple eddy current can be modelled by a circuit with some resistance and inductance yielding a first order frequency response. The observed response (Fig. 1) looks more like a second order effect, not caused by a simple eddy current. We think that it caused by a mechanical vibration of the main magnet coil assembly.

The coupling of the Z gradient with the inhomogeneous part of the main field results in a force (in the z direction) between the gradient coil and the main magnet coil. The gradient coil is mounted rigidly in the room temperature bore but the main magnet coil (horizontal !) is contained in a helium can which is just suspended from two neck tubes in the cryostat and has no further constraints in the z direction . . .

Whatever the real cause of the observed frequency shift may be, it can be easily corrected by a second order compensation circuit, consisting of an integrating and a differentiating part, both with a time constant near 150 ms, as shown in Fig. 2.

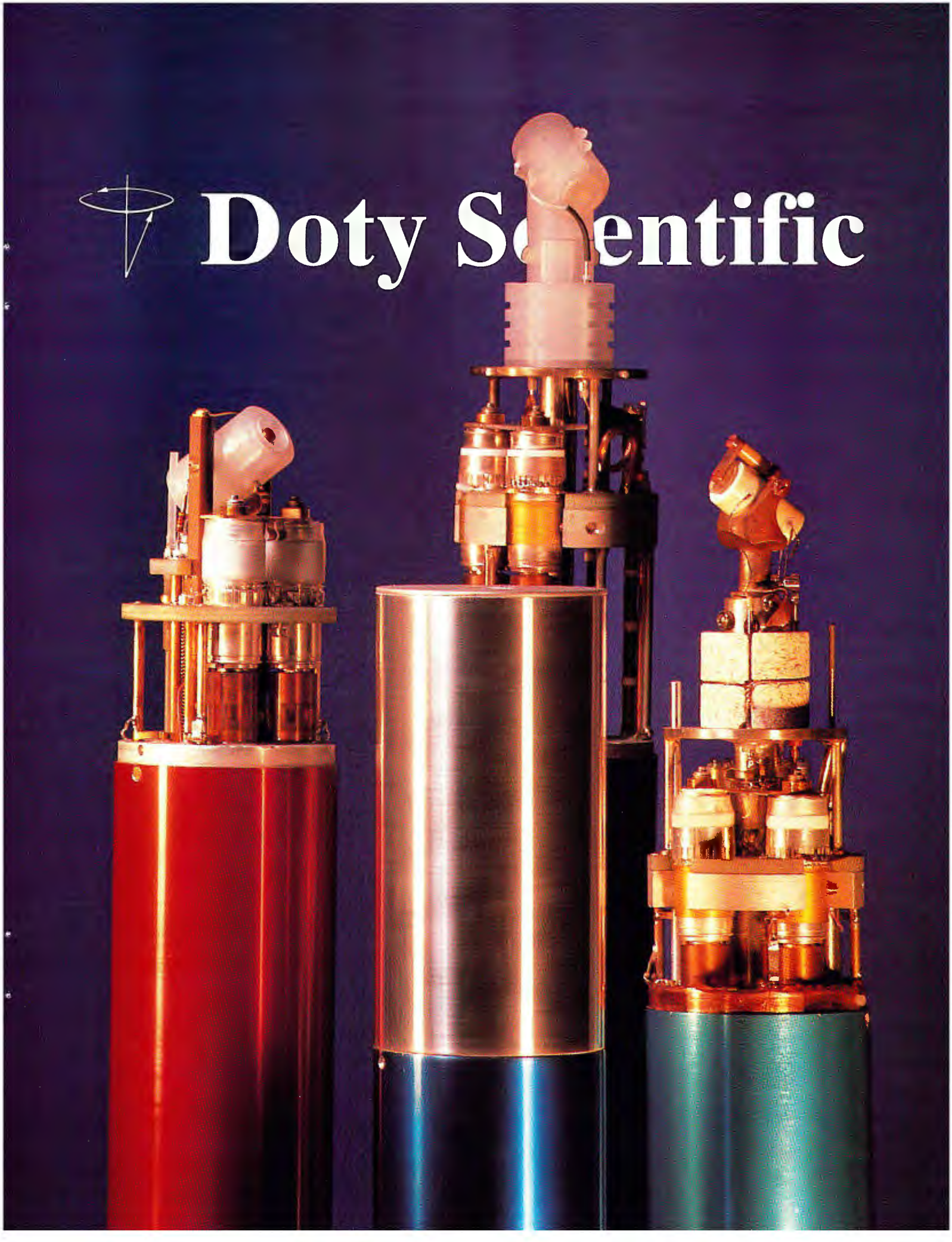
In Fig. 1 the resulting behaviour of the field after these corrections is displayed.

Sincerely yours,

P. van Gelderen, H.M. Borsboom, J.H.N. Creyghton.

1. Van Vaals, J.J., Bergman, A.H., Van Stapele, R.P. , SMRM abstracts 1989, 183.

Please credit this contribution to the account of W.M.M.J. Bovee.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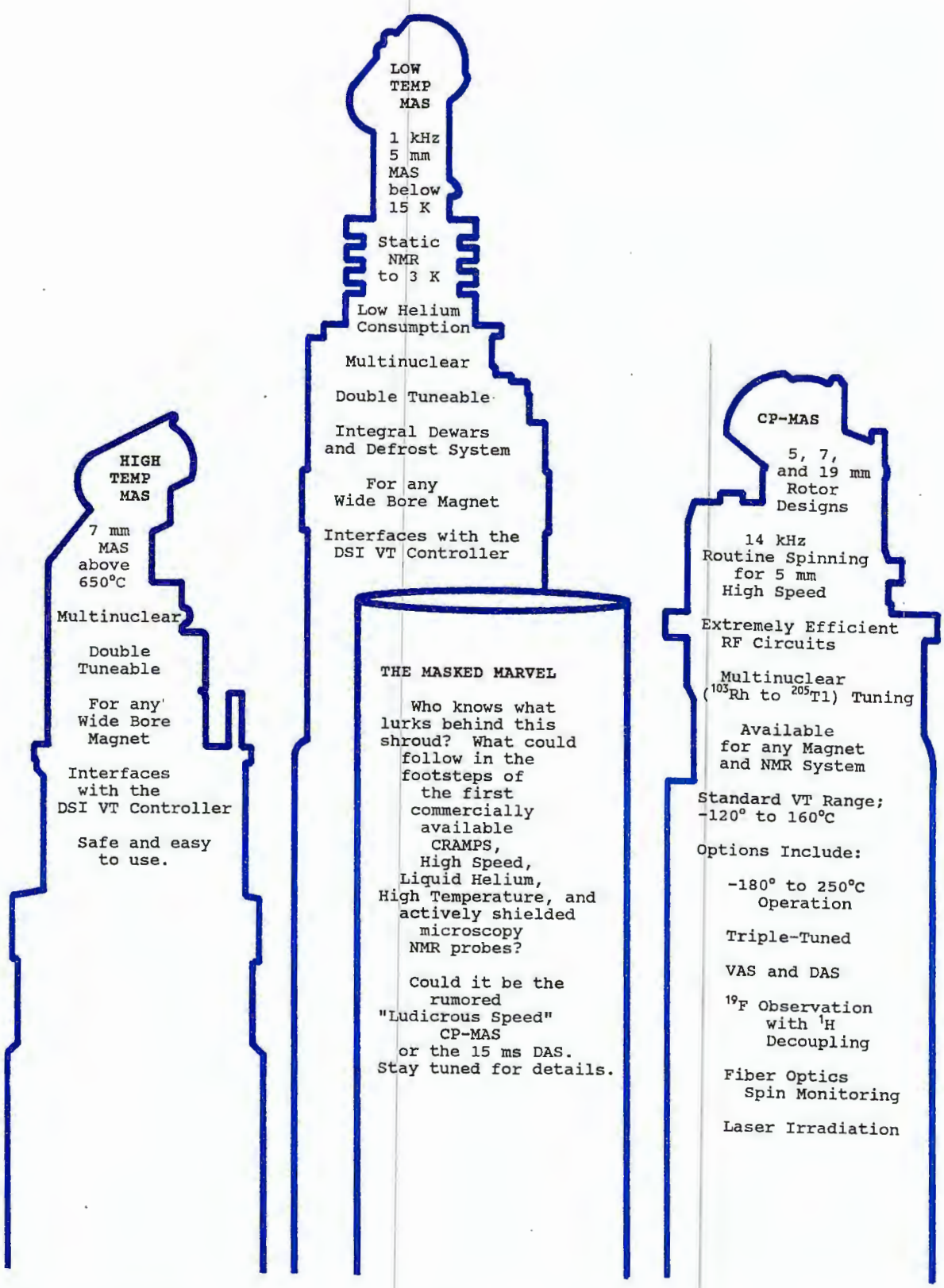

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CHLOROFORM-D "100%"	10x0.5 ml	$ 15.50
DLM-29 (D,99.96%)	10x0.75 ml	$ 22.00
	10 ml	$ 18.00
CYCLOHEXANE-D_{12}	10x1 g	$ 92.00
DLM-17 (D,99.7%)		
DEUTERIUM OXIDE	10x100 g	$375.00
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DLM-11 (D,99.9%) (low paramagnetic, low conductivity)		
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DLM-6 (D,99.96%)		
DEUTERIUM OXIDE "100%"	10x0.7 g	$ 35.00
DLM-8 (D,99.996%)	10x1 g	$ 50.00
	10 g	$ 35.00

1,2-DICHLOROBENZENE-D_4	1 g	$ 20.00
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DLM-25 (D,99.5%)	10x1 g	$155.00
DIMETHYL SULFOXIDE-D_6	10x1 g	$ 11.50
DLM-10 (D,99.9%)	10x10 g	$108.00
DIMETHYL SULFOXIDE-D_6 "100%"	10x0.5 ml	$ 28.00
DLM-34 (D,99.96%)	10x0.75 ml	$ 61.00
1,4-DIOXANE-D_8	10 g	$ 90.00
DLM-28 (D,99%)		
METHANOL-D_4	10x1 g	$ 33.00
DLM-24 (D,99.8%)	10x10 g	$320.00
METHANOL-D_4 "100%"	10x0.5 ml	$ 76.00
DLM-51 (D,99.96%)	10x0.75 ml	$102.00
METHYLENE CHLORIDE-D_2	10x1 g	$ 62.00
DLM-23 (D,99.9%)	5x3 ml	$110.00
	25 g	$125.00
1,1,2,2-TETRACHLOROETHANE-D_2	10 g	$ 36.00
DLM-35 (D,99.6%)	100 g	$270.00
TETRAHYDROFURAN-D_8	5x1 g	$ 75.00
DLM-36 (D,99%+)	10x1 g	$145.00
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DLM-5 (D,99.6%)	10 g	$ 35.00
P-XYLENE-D_{10}	5 g	$ 29.00
DLM-313 (D,98%)		

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DEPARTMENT OF POLYMER SCIENCE

(received 4/21/90)

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

Dear Barry:

The dreaded "pink slip" has reminded us that we haven't communicated any recent experimental results. Hopefully, this communication will remedy the situation.

One of the basic focuses of our research is using ^{15}N solid-state CP/MAS to investigate the molecular structure and motions of nylons and other polyamides. One of the major aspects of our work involves the use of isotopic labelling to circumvent low S/N problems. This is particularly necessary in performing ^{15}N T_1 experiments.

Recently, we have begun the study of ^{15}N labeled nylon 12. Table 1 gives a brief summary of a nylon 12 film treated three different ways. First, the film is heated above its melting point and quenched in liquid nitrogen. Then the film was annealed at a temperature just below its melting point for ~30 hours. Finally, the film was dissolved and precipitated from a phenol/ethanol solution. As expected, the quenched sample shows only two peaks, one for the γ crystal form at 89.3 ppm and the other for the amorphous form at 87.0 ppm. The annealed sample shows, in addition to the γ crystal and amorphous peaks, another peak at 90.3 ppm, which may correspond to an unidentified crystal form. The sample precipitated from a phenol/ethanol solution shows peaks for both the α and γ crystal forms as well as the amorphous form. The ^{15}N T_1 values basically follow expected trends, i.e. the more crystalline materials having longer T_1. However, for the quenched sample the γ crystal T_1 is shorter that the amorphous T_1. One explanation for this anomaly is that for the quenched sample, the peak at 87.0 ppm corresponds to a more ordered, interfacial region which upon annealing goes into the crystalline domains. Thus, for the annealed samples, the peak at 87 ppm corresponds a "true" amorphous region, while for the quenched and reprecipitated samples this peak corresponds to both the amorphous and interfacial regions. Similar behavior has been observed for polyethylene (Kitamaru, Horii, and Murayama, *Macromolecules*, **1986**, *19*, 636) and nylon 6 (Powell and Mathias, *J. Am. Chem. Soc.*, **1990**, *112*, 669).

Table 1. T_{1N} Values for Nylon 12[a]

Chemical[b] Shift (PPM)	90.3	89.3	87.0	84.2
quenched		107.6 / 2.8	130.5 / 3.7	
γ(annealed)	319.3 / 10.7	313.8 / 20.2	40.6 / 4.2	
$\alpha + \gamma$		159.7 / 9.1	107.8 / 1.4	322.9 / 2.2

[a] T_{1N} values were determined using two component SIMFIT fits; the value given after the / refers to the faster relaxing component, which usually comprised ~30% of the peak.

[b] ^{15}N chemical shifts were referenced to glycine (=0 ppm).

Sincerely,

C. Greg Johnson

W. L. Jarrett

Lon J. Mathias

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POSTDOCTORAL POSITION

I currently have a postdoctoral position open in my laboratory. Projects include development of new methods for rapid collection of 3D-NMR data, development of new methods for digital filtration of 2D-NMR data, development of new 2D- and 3D-NMR methods for characterizing high molecular weight molecules in solution, and development of 2D-NMR techniques to study substrate binding. We are applying these methods to the characterization of new synthetic polymers and the investigation of substrate binding to biomolecules. Equipment available includes Gemini-300, VXR-300 and XL-400 widebore liquids and 200 MHz widebore solids spectrometers. These instruments are linked via an Ethernet network to ca. 9 Sun and Silicon Graphics workstations for offline data processing and molecular modeling.

If you are interested in this position contact me and send a CV and 3 reference letters.

Peter Rinaldi
Department of Chemistry
The University of Akron
Akron, OH 44325-3601
216-375-5990

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BOX 498, G.P.O., ADELAIDE, SOUTH AUSTRALIA 5001

POSITION AVAILABLE

Research Officer Grade 3/Grade 4

The NMR Facility of the Chemistry Departments consists of three Bruker instruments, an AC300P, CXP300 and WP80 and is primarily used for research undertaken by academic staff. The Departments are seeking a research officer to manage and control the research support programs of the facility. The incumbent would also be expected to act as a consultant to other users both within and outside the University as required, and to collaborate in the undertaking of research projects and the development of the facility.

It is anticipated that the successful applicant would have had several years experience in the field of nuclear magnetic resonance. Broad experience in the area of 2D NMR techniques is essential and some experience in solid state NMR would be an advantage. Further information may be obtained from the Chairman, Department of Physical and Inorganic Chemistry, by FAX on 618 2236771.

The salary will be in the range $A37,426 - $A46,954 depending on the grade of appointment. Applications, in duplicate, quoting reference number 4370/1765 and giving details of educational qualifications, should reach the Senior Assistant Registrar (Personnel), The Registry, University of Adelaide, GPO Box 498, Adelaide, South Australia, 5001, no later than 30 May, 1990.

NMR spectroscopist to be in charge of spectroscopic facility containing a XL-300 advanced spectrometer, a GEMINI-300 spectrometer, a VG-7070E mass spectrometer, and a FTS-15/80 IR spectrometer. Familiarity with mass spectrometry and/or computer-aided drug design desirable.

Send Resume to:

Dr. Michael Garst
Vice Present of Chemical Sciences
Allergan Inc.
2525 Dupont Drive
Irvine, CA 92715

NMR SPECTROSCOPIST

The University of Minnesota Medical School is inviting applicants for a tenure track or tenured faculty position to be filled by a scientist specializing in the study of biological macromolecules by high resolution NMR spectroscopy. This position, which can be at the level of Assistant, Associate, or Full Professor, will be available in the Fall of 1990. We seek a faculty member with three years minimum experience who will interact with and complement the existing structure groups in several programs, such as protein x-ray crystallography, electron microscopy and EPR. Presently, three NMR spectrometers (250, 360 and 500 MHz) are available, and additional high resolution instruments are planned.

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This position will be affiliated with the newly formed Institute of Biomedical Engineering. Teaching duties will be associated primarily with the Department of Biochemistry in the Medical School.

Interested candidates should send, by May 15, 1990, a curriculum vitae, documenting qualifications, professional accomplishments and future research plans, together with names and addresses of three references to:

H.P.C. Hogenkamp
Professor and Head
Department of Biochemistry
4-225 Millard Hall
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O1    7900.000
SI      32768
TD      32768
SW    5000.000
HZ/PT     .305

PW        5.0
RD        0.0
AQ      3.277
RG         32
NS         72
TE        297

FW       6300
O2    7925.000
DP    10H PO

LB        0.0
GB        0.0
CX      36.00
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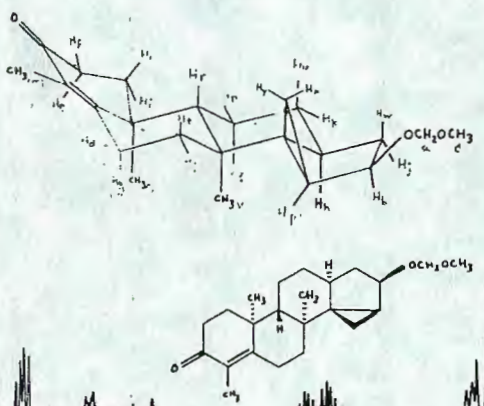

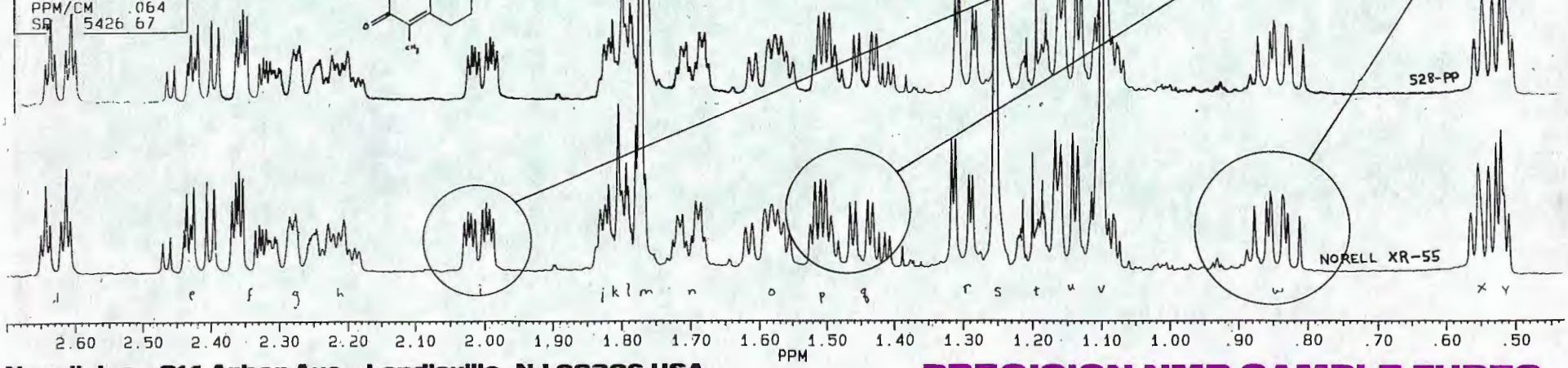

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camber ± 0.03mm (0.0015 in.);
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add $0.08 per cm ($0.20/in).

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camber ± .013mm (0.0005 in.).
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1-5 tubes....$9.50 ea.
6-25.....$9.25 ea.
26 +.....$9.00 ea.

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March 20, 1990
(received 3/22/90)

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

TANGO Dancing or BIRD WALTZING

Dear Barry,

I recently faced a long range correlation problem in the 1H and 13C NMR analysis of the cyanogenic diglucoside, D-amygdalin (I). After completing 1H assignments and correlations to the direct carbons, long range correlations were desirable to locate the oligosaccharide rings in sequence.

Because response intensity in long range transfer is modulated by one-bond coupling (1), long range responses often vary in result. Several experimental schemes have been put forth (2-5). The direct response is often still visible in long range experiments. Sometimes, the direct response remains more intense than the desired long range response (5).

Fig. 1 shows a long range experiment optimized for 6.25Hz based on the TANGO operator (4) at a contour level just above noise. A strong long range response is seen corresponding to the 2J from the methine proton to the cyano carbon. Direct responses persist from aromatic, CH, A1, B1 and other sugar resonances. A second experiment optimized at 10 Hz using the TANGO operator gave even fewer responses.

Fig. 2 shows a modified long range experiment which uses a BIRD operator to remove the modulations of the response intensity by the one-bond J coupling (5). The experiment was again optimized for 6.25 Hz responses. The 2J from the CH to the CN and the direct CH to CH responses are again seen. Additional long range responses to the A1 anomeric carbon, the aromatic quarternary and the aromatic ortho carbon signals are now observed. The 2J response at the A1 anomeric carbon and the absence of response from the CH to B1 specifically places the A ring adjacent to the aryl-cyano moiety. A reciprocal response is seen from the A1 proton to the methine carbon. The A6 proton to B1 carbon response and the reciprocal B1 proton to A6 carbon response specifically verify the B1 to A6 glycosidic linkage. Additional long range responses are seen within the aromatic and carbohydrate rings. Except for the single CH direct correlation, no other direct responses are seen testifying to the efficacious removal of one-bond modulations by the BIRD operator.

It's not always this nice, of course! But in the present case at least it appears that two TANGO dances do not "deliver the goods" as well as one WALTZ with a BIRD.

Sincerely,

Tony

Anthony A. Ribeiro

1. K.V. Schenker & W. Von Phillipsborn J Mag Res. 61 294 (1985).
2. W.F. Reynolds et al Can J Chem 62 2421 (1984).
3. M. Levitt et al Chem. Phys. Lett. 94 540 (1983).
4. S. Wimperis & R. Freeman J. Mag. Res. 58 348 (1984).
5. A. Zekster et al Mag. Res. Chem. 25 752 (1987).

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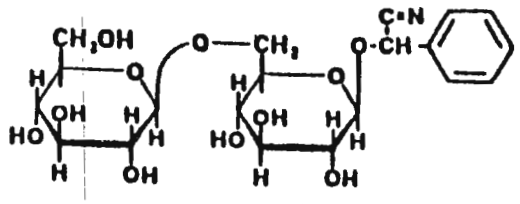

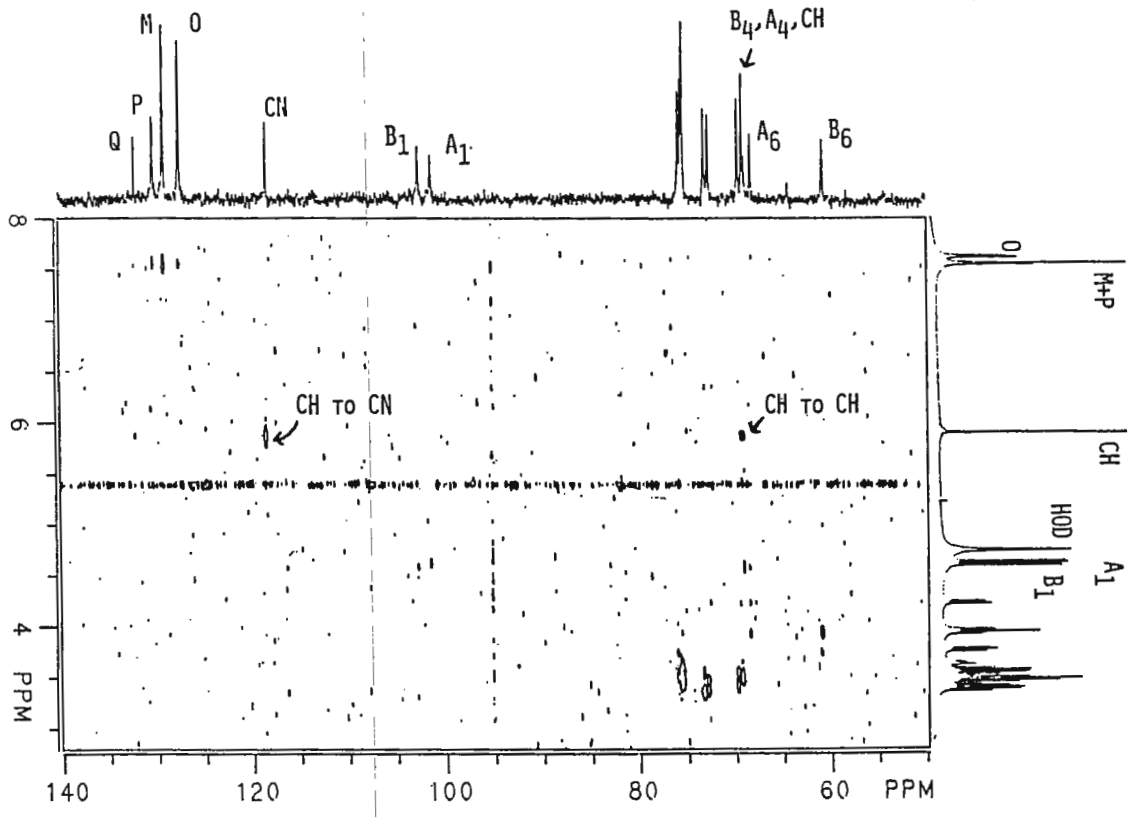

Fig. 1
Long-range
Correlation
using
TANGO
operator
optimized
for 6.25 Hz.

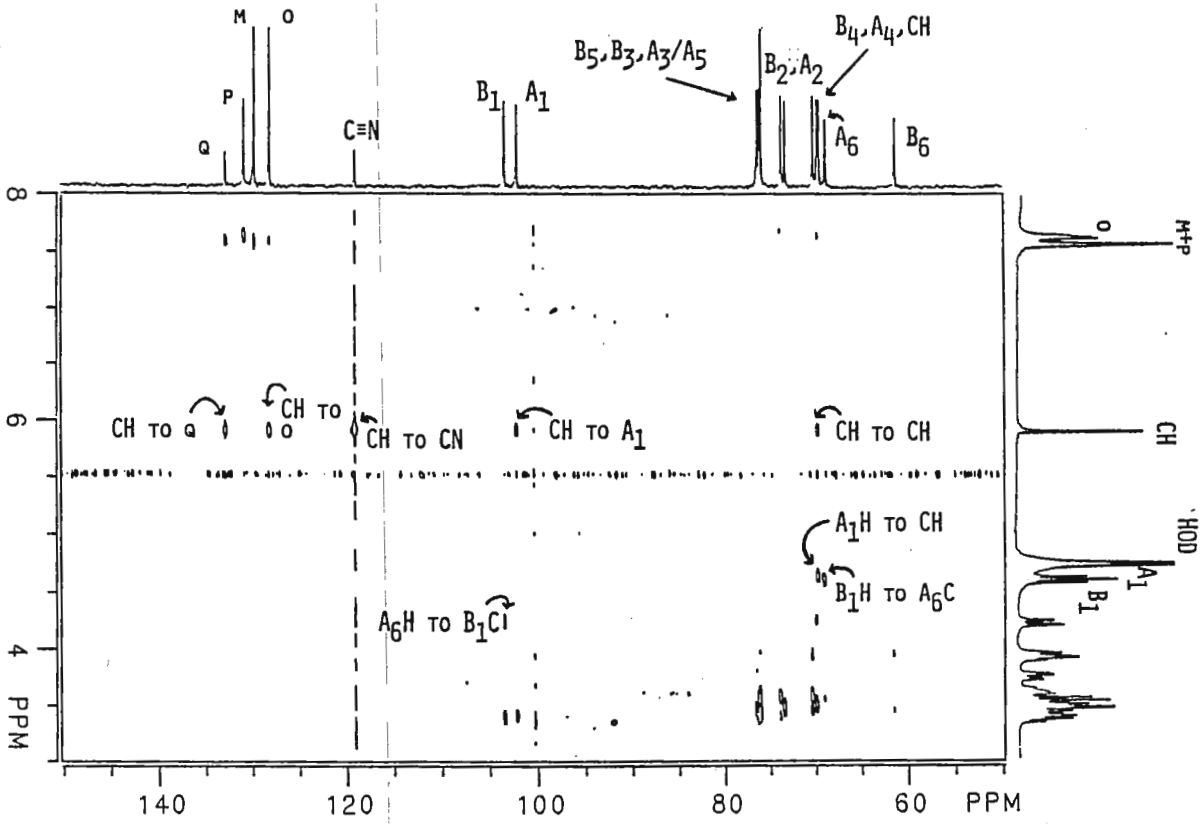

Fig. 2
Long-range
correlation
using
BIRD
operator
optimized
for 6.25 Hz.

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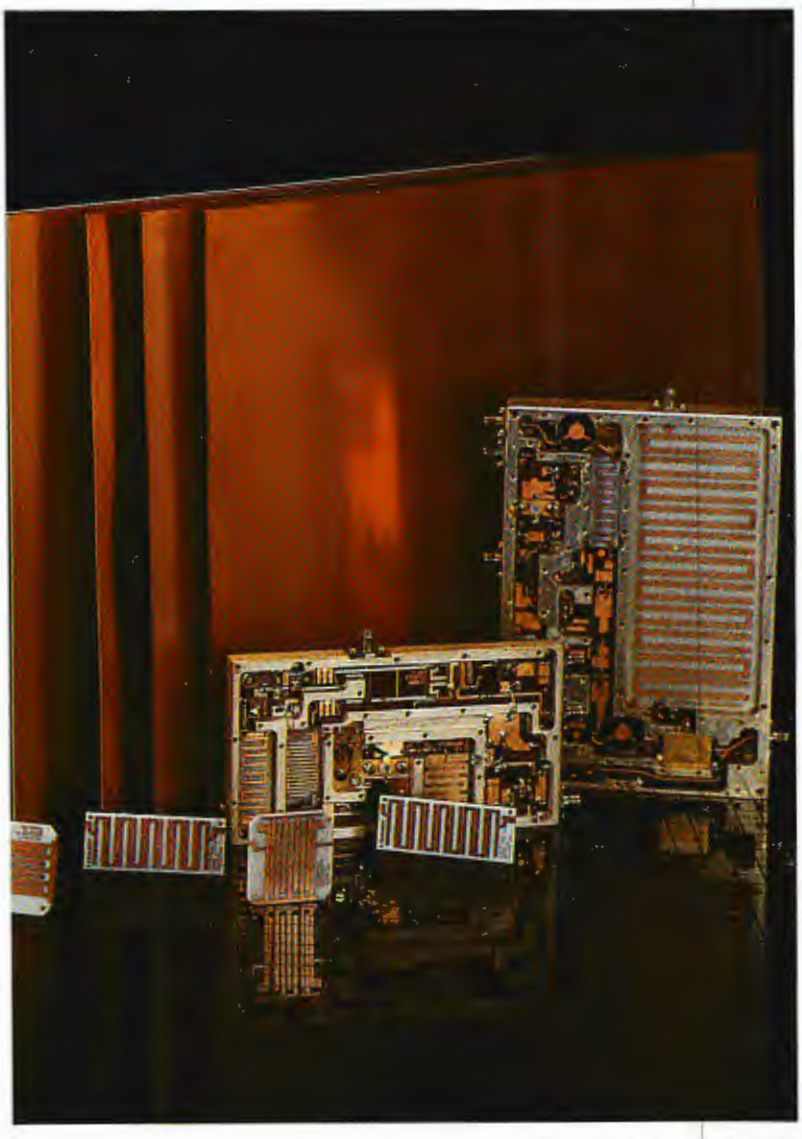

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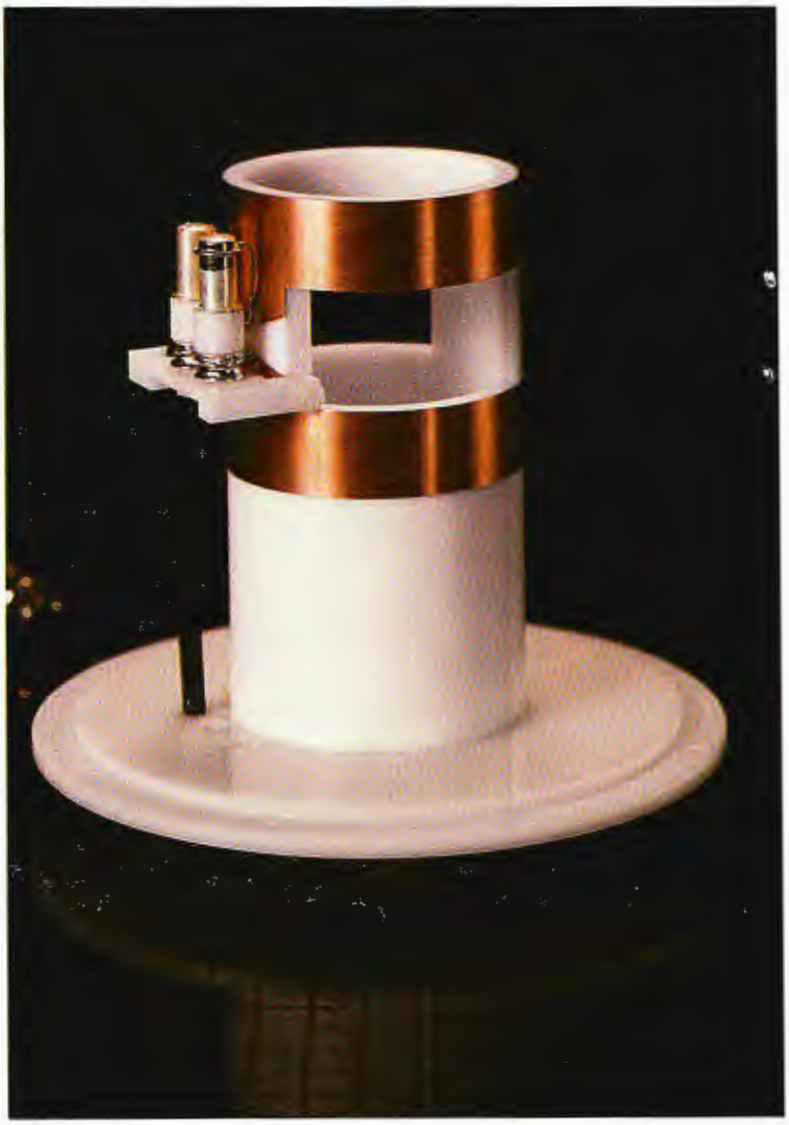

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UNIVERSITY OF UMEÅ
Department of Organic Chemistry

Telephone
46 - 90 - 16 50 00

March 27, 1990 (received 4/4/90)

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

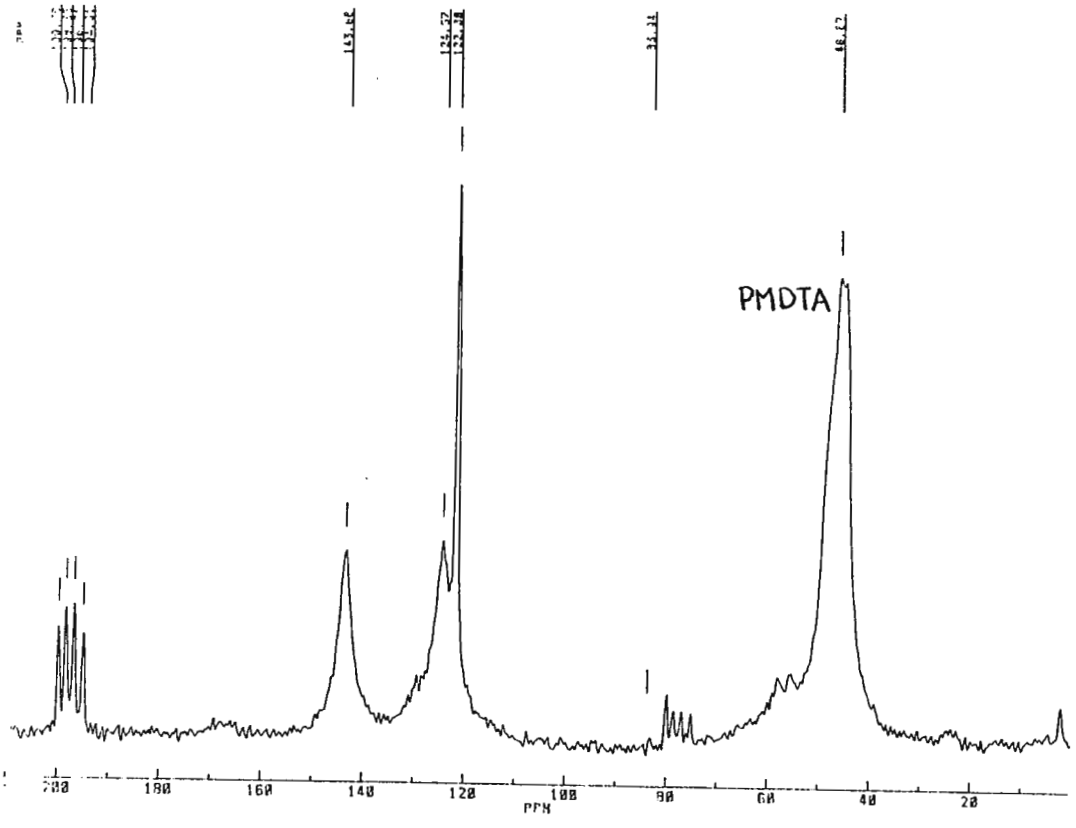
Observation of ^{7}Li-C Couplings in Phenyllithium.

Dear Barry,

We are still very enthusiastic about the possibility to use CP/MAS techniques to obtain structural information of highly reactive organometallic species. In a study of various phenyllithium-6(7) (PhLi) complexes we were able to observe the ^{7}Li-C coupling for the first time in the monomeric PhLi·PMDTA compound. The coupling was close to 40 Hz (see below) which is what could be expected from the 15 Hz obtained in solution from Ph^{6}Li. However, a slight asymmetry in the coupling pattern is noted at 2.3 T , which arises from the mixing of dipolar and scalar coupling. The observed broadening of the ortho and meta positions is due to restricted rotation along the symmetry axis, a broadening absent in the dimers or tetramers. The fact that any self-decoupling due to fast Li relaxation is absent opens interesting possibilities in this area of organolithium chemistry.

Best regards

Ulf Edlund Dan Johnels



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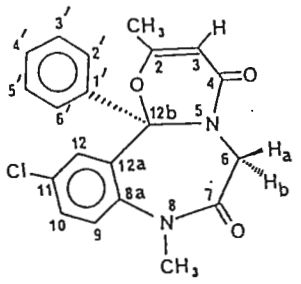
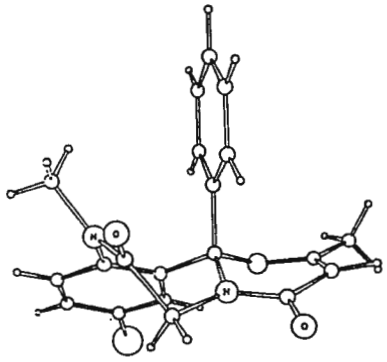
Professor B. L. Shapiro
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto, CA 94303

April 18, 1990
(received 4/18/90)

Dear Barry:

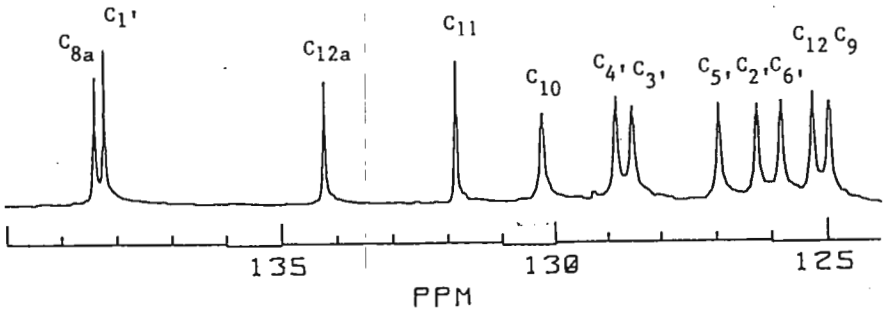
Ketazolam is a 1,4-benzodiazepine derivative containing an unsubstituted phenyl ring. This phenyl ring undergoes hindered rotation about the aryl to tert-alkyl bond even at room temperature and therefore exhibits broad ^1H





phenyl resonances at 200.1 MHz. As the temperature was lowered below the estimated coalescence temperature of 1°C, the broad phenyl ^1H signals became progressively sharper, consistent with an approach to a slow exchange spectrum reflecting two nonequivalent *ortho* protons.

The ^{13}C spectra of ketazolam /$CDCl_3$ solutions show more changes than the ^1H spectra. At room temperature the *meta* carbons, as well as the *ortho* carbons, are coalesced. Below -40°C, separate resonances are observed for all 20 carbons of ketazolam. The broadband decoupled ^{13}C spectrum (50.3 MHz) obtained at -58°C (0.592 M in $CDCl_3$) is shown.



A combination of one-bond and long-range (COLOC) ^{13}C-^1H chemical shift correlation experiments were carried out at low temperatures to make the ^{13}C chemical shift assignments. Empirical energy calculations are in accord with a two-fold barrier to rotation about the $C_{1'}$-C_{12b} bond.

Sincerely,

Lori

Laurine A. LaPlanche

Robert Rothchild
John Jay College of Criminal Justice
The City University of New York

Continued from page 1:

FORTHCOMING NMR MEETINGS

10th European Experimental NMR Conference, **May 28 - June 1, 1990;** Veldhoven, The Netherlands. Contact: M. J. A. de Bie: see Newsletter 376, 26.

Gordon Research Conference: Magnetic Resonance in Biology and Medicine, **July 16-20, 1990;** Tilton School, Tilton, NH. Chairman: R. G. Bryant. Contact: Dr. A. M. Cruickshank, Gordon Research Center, Univ. of Rhode Island, Kingston, RI 02881-0801.

Workshop of Special Topics in Medical Magnetic Resonance, sponsored by the Society of Magnetic Resonance in Medicine and the National Research Council of Canada, **July 23-27, 1990;** Whistler Mountain, BC, Canada. Contact: L. Forget - see Newsletter 374, 46.

Expanding Frontiers in Polypeptide and Protein Structural Research, sponsored by the National Research Council of Canada, **July 23-27, 1990;** Whistler Mountain, BC, Canada. Contact: L. Forget - see Newsletter 374, 46.

Tenth International Biophysics Conference, sponsored by the International Union of Pure and Applied Biophysics and the National Research Council of Canada, **July 29 - August 3, 1990;** Vancouver, BC, Canada. Contact: L. Forget - see Newsletter 374, 46.

Bat-Sheva Workshop on New Developments and Applications in NMR and ESR Spectroscopy, **October 14-24, 1990,** Israel; Contact: Dr. D. Goldfarb, The Weizmann Institute of Science, Rehovot, Israel. See Newsletter 377, 10.

Advanced Tomographic Imaging Methods for the Analysis of Materials, Symposium at the Fall Meeting of the Materials Research Society, Boston, Mass., **Nov. 26 - Dec. 1, 1990;** See Newsletter 378, 57.

Additional listings of meetings, etc., are invited.

All Newsletter Correspondence

Should Be Addressed To:

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

(415) 493-5971

DEADLINE DATES

No. 382 (July) ----------15 June 1990
No. 383 (August) ----------20 July 1990
No. 384 (September) -----17 August 1990
No. 385 (October)----14 September 1990

Notice re 1990-91 Invoices and Change in Subscription Rates

Subscription renewal invoices for the October 1990 - September 1991 year will be mailed out during the last ten days of June. If you ought to receive such an invoice, and do not have it in your hands by July 6, please call or write me promptly. *Payment* of these invoices *must be received* by me *no later than September 5, 1990* to ensure uninterrupted mailing of the Newsletter issues. Please do not delay execution of any necessary paperwork! Also, please be sure that the instructions on the invoice are followed precisely.

When you receive your invoice, you will find that it has become necessary to make a modest increase in the subscription rate. Note that this is the first such increase in three years. I regret that inflationary pressures in general, and a substantial incipient increase in the postage rates in particular, make the subscription rate increase necessary. Advertising rates will also increase modestly, and our loyal Sponsors will be asked to increase their contributions. The surcharge for Air Mail Printed Matter mailing will also need to be incremented.

The new subscription rate, effective 1 October 1990, will be US$150.00 for the twelve monthly issues, postpaid. Personal or academic subscriptions will continue to be offered at a 50% discount, US$75.00.

Thank you for your understanding and cooperation.

B. L. Shapiro
1 May 1990

Mailing Label Adornment: Is Your Dot Red ?

If the mailing label on your envelope of this issue is adorned with a large **red dot** or circle: this decoration means that you will not be mailed any more issues until a technical contribution has been received by me.

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Attention overseas subscribers: If you must use paper which is longer that 11", *please* take care that nothing appears below 10" (25.5 cm) on your pages. It is costly to make reductions. Thank you.

University of Delaware

DEPARTMENT OF CHEMISTRY AND BIOCHEMISTRY
NEWARK, DELAWARE 19716

April 18, 1990
(received 4/23/90)

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

^{2}H NMR Spectra of Trapped Organic Materials

Dear Barry,

^{2}H NMR lineshapes have been shown by a number of authors to be a good probe of molecular motion in organic solids like polymers and in liquid crystals. Recently, for example, Boddenburg and his co-workers have used ^{2}H NMR spectra to study the mobility of benzene in catalysts.

We have been interested in guest-host interactions of relatively large, conjugated, aromatic molecules in various zeolites. As examples, we show ^{2}H NMR spectra of phenanthrene in X zeolites with different counterions. The spectra are generally a quadrupolar powder pattern superimposed on a sharp feature. The relative proportions of these two features are temperature dependent, with the relative amount of the narrow feature at a particular temperature depending on the counterion.

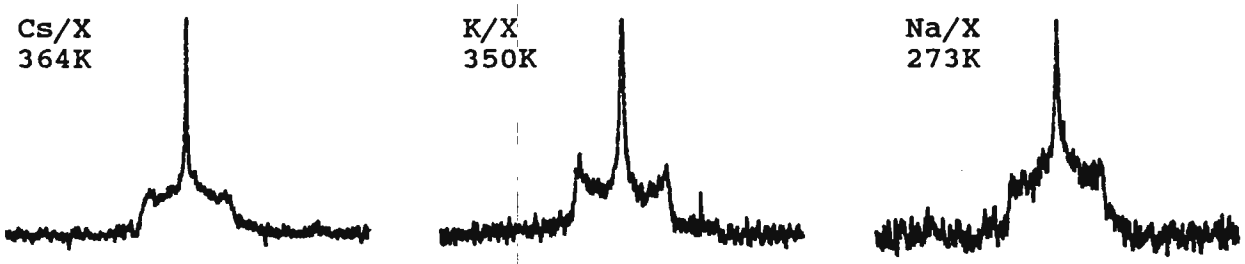
The lineshapes and their temperature dependences do not seem to be consistent with a broad distribution of correlation times, as is sometimes seen for molecules on catalysts. We are continuing to investigate systems of this type to understand the dynamics of these kinds of molecules in modestly constrained environments.

Yours truly,

Cecil

Cecil R. Dybowski

Mark A. Hepp



CSI 2T Applications

Shielded Gradients: Theory and Design

NMR imaging and localized spectroscopy depend on the use of pulsed magnetic field gradients. As these techniques have grown more complex, it has become apparent that eddy currents created in the magnet cryostat and other structures by pulsed gradients have become the chief limitation to many sophisticated applications.

Figure 1a illustrates the design problem for unshielded gradients. Figure 1b illustrates the shielded gradient arrangement. Figures 2 and 3 show the contours of constant flux for an unshielded and shielded Z gradient coil, respectively. This demonstrates that, for the shielded gradients, most of the flux has been kept away from the magnet bore.

The dramatic reduction of eddy currents which can be made over the conventional, unshielded gradients is shown in Figures 4a, b. These graphs show frequency as a function of time following the application of a long, constant amplitude gradient pulse which is suddenly cut off. Soon after cut off, a 90° pulse is applied and the complex FID recorded. The instantaneous frequency is then obtained from the FID and normalized by dividing by the frequency offset at the sample during the gradient pulse.

Figure 4a shows a typical decay of extra magnetic fields in a CSI 2T instrument caused by eddy currents in the conventional, unshielded gradient set with compression.

Figure 4b shows the decay of the uncompensated shielded Z gradient and Figure 4c shows the Z gradient decay with compensation. Note that the time scale for 4b and 4c is five times shorter than that for the unshielded gradients.

Fig. 1a.

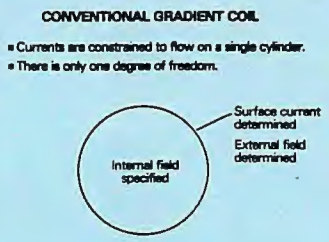


Fig. 1b.

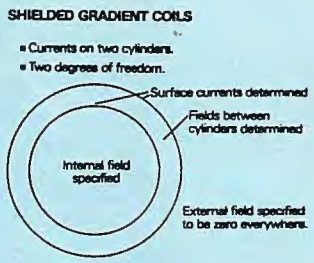


Fig. 1a—Design problem for unshielded gradients. The field inside the winding is specified to be a linear gradient and the current pattern on the cylinder is determined. Fig. 1b—Design arrangement for shielded gradients. The field inside the inner cylinder is specified to be a linear gradient and the field beyond the outer cylinder is specified to be close to zero. The current patterns on both inner and outer cylinders are then determined.

Fig. 2.

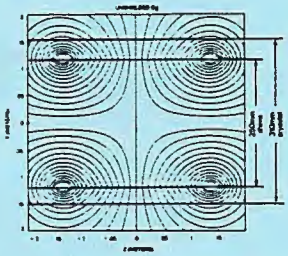
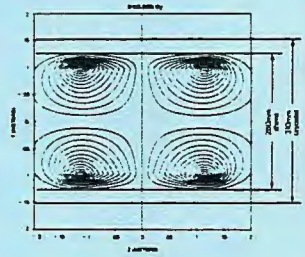


Fig. 3.



Lines of constant flux for Z-gradient. Fig. 2—Unshielded gradient. Note that flux lines extend well beyond the cryostat bore. Fig. 3—Shielded gradient. Flux lines are kept within the outer gradient cylinder.

Fig. 4a.

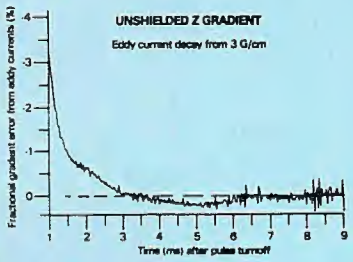


Fig. 4b.

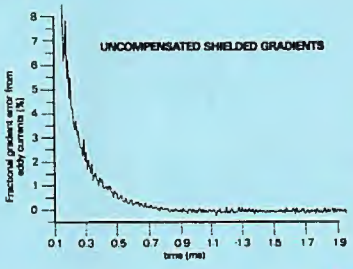
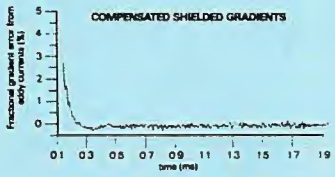


Fig. 4c.



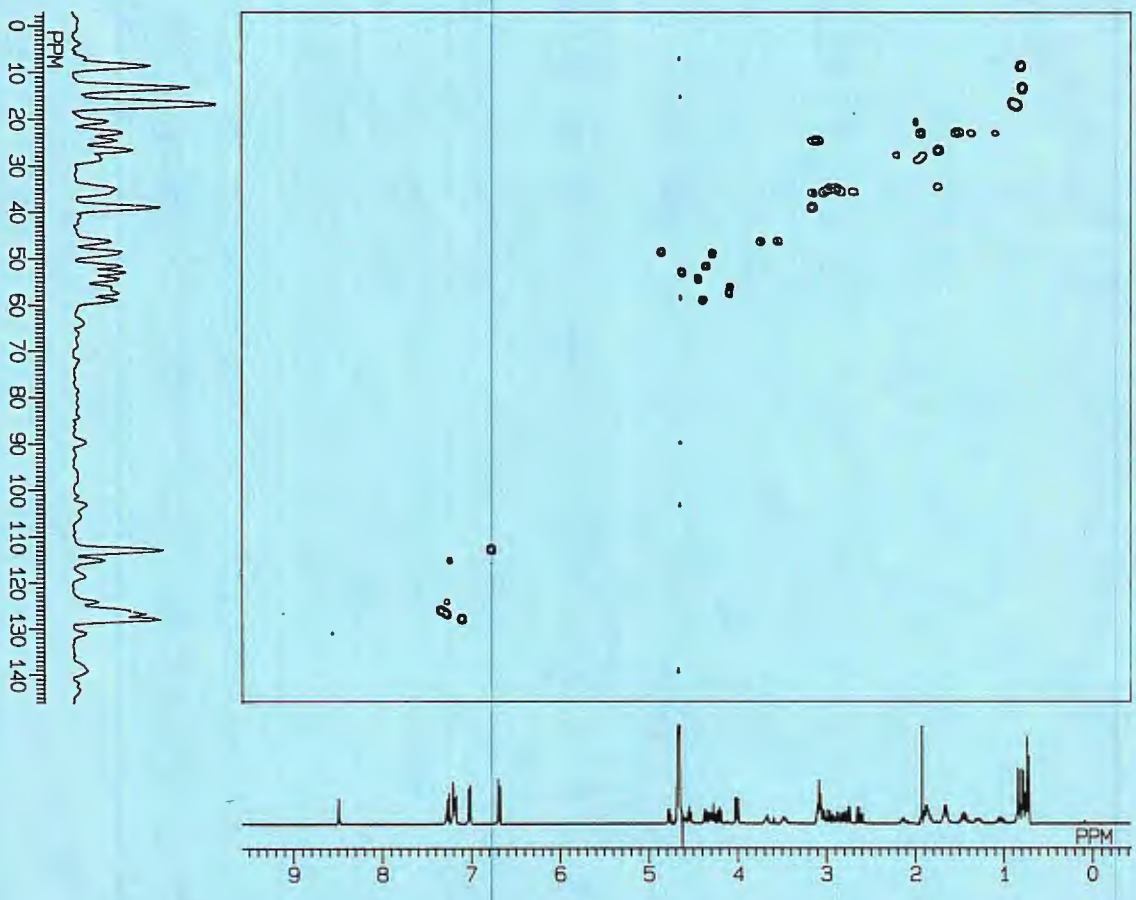
Decay of field following application of square gradient pulse. Fig. 4a—Unshielded gradients. Fig. 4b—Shielded S150 gradient with no waveform compensation. Fig. 4c—Shielded S150 gradient with waveform compensation.

GE NMR Instruments

AFTER THE WORK IS FINISHED...

THE OTHER CPF COMES OUT TO PLAY

When all of the day's production samples have been run and the piles of spectra plowed through, JEOL's CPF can quickly switch operation modes. This same highly automated machine which is used for all of the production work can produce very high quality research data for those non-routine problems that always seem to appear. In many cases all that is necessary to make the change is to log into the research account. This changes the CPF from an automated, limited instrument into a wide-open research-grade spectrometer. With the addition of the appropriate accessories the low cost CPF is capable of running the most sophisticated research type experiments including CP MAS solids. Now that the CPF is available at a field strength of 400 MHz in addition to the very popular 270 MHz, the high field research spectrometer has become very affordable. The above 400-CPF data is a reverse detection 13-C experiment run on Angiotensin-II. The instrument used for this experiment is a standard CPF with the addition of the optionally available reverse detection Broad Band probe. This data clearly shows that the routine need never be the only thing you can do.



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