THE NMR NEWSLETTER

No. 445

October 1995

continued on inside back cover

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	Frequency Range	Resolution	Switching Time[1]	Phase-Continuous Switching[2]	Rack-Mount Cabinet Dim.[3]	Remote-Control Interface	Price Example[4]
PTS 040	.1-40 MHz	optional .1 Hz to 100 KHz	1-20μs	optional	5¼"H×19"W	BCD (std) or GPIB (opt)	$5,330.00 (1 Hz resol., OCXO freq. std.)
PTS 120	90-120 MHz	optional .1 Hz to 100 KHz	1-20μs	optional	5¼"H×19"W	BCD (std) or GPIB (opt)	$5,330.00 (1 Hz resol., OCXO freq. std.)
PTS 160	.1-160 MHz	optional .1 Hz to 100 KHz	1-20μs	optional	5¼"H×19"W	BCD (std) or GPIB (opt)	$6,495.00 (1 Hz resol., OCXO freq. std.)
PTS 250	1-250 MHz	optional .1 Hz to 100 KHz	1-20μs	optional	5¼"H×19"W	BCD (std) or GPIB (opt)	$7,440.00 (1 Hz resol., OCXO freq. std.)
Type 1 **PTS 310** Type 2	.1-310 MHz	1 Hz	1-20μs	standard	3½"H×19"W	BCD (std) or GPIB (opt)	1 Hz resol., OCXO: $6,425.00 1 Hz resol., OCXO: $5,850.00
PTS 500	1-500 MHz	optional .1 Hz to 100 KHz	1-20μs	optional	5¼"H×19"W	BCD (std) or GPIB (opt)	$8,720.00 (1 Hz resol., OCXO freq. std.)
PTS 620	1-620 MHz	optional .1 Hz to 100 KHz	1-20μs	optional	5¼"H×19"W	BCD (std) or GPIB (opt)	$9,625.00 (1 Hz resol., OCXO freq. std.)
PTS 1000	0.1-1000 MHz	optional .1 Hz to 100 KHz	5-10μs	optional	5¼"H×19"W	BCD (std) or GPIB (opt)	$11,830.00 (1 Hz resol., OCXO freq. std.)
PTS 3200	1-3200 MHz	1 Hz	1-20μs	optional	5¼"H×19"W	BCD (std) or GPIB (opt)	$14,850.00 (1 Hz resol., OCXO freq. std.)
PTS x10	user specified 10 MHz decade	1 Hz	1-5μs	standard	3½"H×19"W	BCD (std) or GPIB (opt)	$3,000.00 (1 Hz resol., OCXO freq. std.)
PTS D310	two channels .1-310 MHz	.1 Hz	1-20μs	standard	5¼"H×19"W	BCD (std) or GPIB (opt)	$8,560.00 (.1 Hz resol., OCXO freq. std.)
PTS D620	two channels 1-620 MHz	.1 Hz/.2 Hz	1-20 μs	standard	5¼"H×19"W	BCD (std) or GPIB (opt)	$13,240.00 (.1 Hz/.2 Hz resol., OCXO freq. std.)

1 Switching Time is dependent on digit (decade) switched; see detailed instrument specifications.

2 For applicable digits, see detailed instrument specifications.

3 Bench cabinets are 17" wide.

4 Prices are U.S. only and include Manual and Remote (BCD) Control; PTS 3200 Digital Front Panel.

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FORTHCOMING NMR MEETINGS

Western Biotech Conference, San Diego, CA, **October 18 - 21, 1995**; Contact: Western Biotech Conf. Registr'n., c/o Tom Lobl, Tanabe Research, 4540 Towne Centre Court, San Diego, CA 92121; Tel. (619) 622-7035; Fax: (619) 622-7080; E-mail: tjlobl@cerf.net.

37th ENC (Experimental NMR Conference), Asilomar Conference Center, Pacific Grove, California, **March 17 - 22, 1996**; Contact: ENC, 1201 Don Diego Avenue, Santa Fe, NM 87501; (505) 989-4573; Fax: (505) 989-1073.

NMR Symposium at the 38th Rocky Mountain Conference on Analytical Chemistry, Denver, Colorado, **July 22-25, 1996**; Contact: Dr. Joel R. Garbow, Monsanto Company, 700 Chesterfield Parkway North, St. Louis, MO 63198; (314) 537-6004; Fax: (314) 537-6806; e-mail: jrgarb@snc.monsanto.com; See Newsletter 445, 48.

XVIIth International Conference on Magnetic Resonance in Biological Systems, Keystone, Colorado, **August 18 - 23, 1996**; Contact: ICMRBS, 1201 Don Diego Avenue, Santa Fe, NM 87501; (505) 989-4735; Fax: (505) 989-1073.

38th ENC (Experimental NMR Conference), Orlando, FL, **March 23 - 27, 1997***[sic]*; Contact: ENC, 1201 Don Diego Avenue, Santa Fe, NM 87501; (505) 989-4573; Fax: (505) 989-1073.

Additional listings of meetings, etc., are invited.

Amazing offer - see page 8!!

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

Tue Aug 8 13:59:17 EDT 1995
(received 8/14/95)

Dr. Barry Shapiro
The NMR Newsletter
966 Elsinor Court
Palo Alto, CA 94303

Dear Barry,

Reliquefied Helium, Anybody?

It occurred to me that some of the Newsletter readers might possibly be interested in our experiences with recovering and reliquefying our own He over the past dozen years. This all started when we first put our 600 into operation in 1979. At that time we were using (and still are using) about 200 l. of He/week, which we bought from the usual suppliers, at about $5.00/l. -- that comes to $52000/yr, which is not inconsiderable. We decided to try recovering and reliquefying He, and bought a used system from a fly-by-night used He liquefier salesman. The liquefier had been in use at the Physics Department of Michigan State University, and was reported to be in good operating condition, though well used. The compressor and liquefier had been in use, according to the odometer about 9000 hours. If I translate this into usage of an automobile, I get a car that has gone about 300,000 miles.

Buying this thing, getting it here, and installing it cost about $80,000. Since 1982, it has, in fact continued to function, and as we got to know it better, has actually improved considerably in efficiency. The first difficulties were to find and rectify the leaks in the system, which were initially serious -- we were losing He at such a rate that we had to order in as much He as we were making. That situation has been rectified to the point where we lose less than 1% a day of our inventory. Actually leaks work two ways -- they let He out, and they let air in, which makes the He unusable. Currently, this is not a problem.

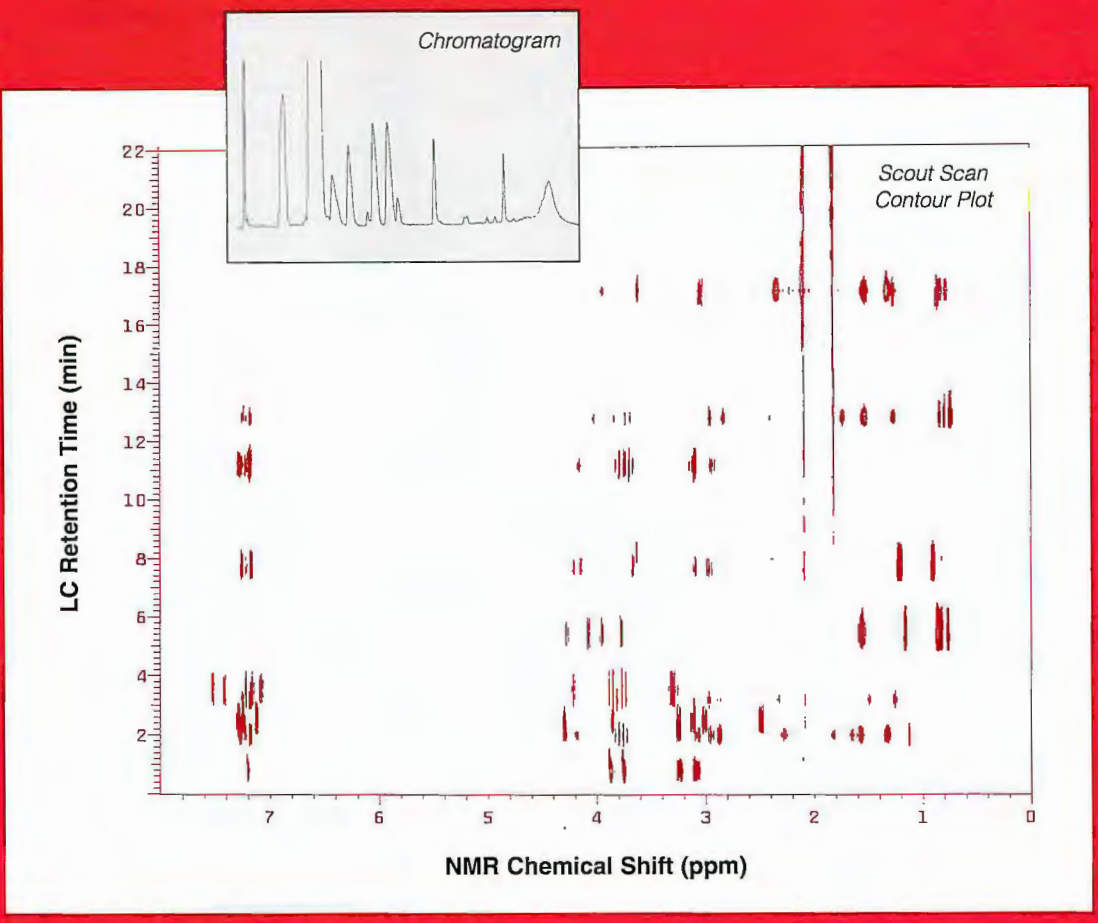
We also gradually found out how to adjust the valves on the expansion engine to maximize the liquefaction rate, which is now better than four liters/hour. Thus we can satisfy our own requirements by running two days/week. We have installed recovery lines to some of the other labs in the building, and run an extra day to reliquefy He for them, for which we charge them $2.00/l. They are happy to get this for less than what it costs from an external supplier. When our inventory of He gets low from the 1% losses, we ask them to buy a 100 l. can from outside; thus the losses are kept at bay, and we get our own He essentially for free.

Well, almost free. We do have to repair the liquefier from time to time. At various times over the last ten years, the purifier sprang a leak, and had to be replaced, a bearing on the expansion engine froze up and broke a piston rod in the expansion engine, and the compressor which supplies the He at 250 psi to the liquefier failed spectacularly, with the high pressure piston jammed sideways in the cylinder. So far we have gained a lot of experience in engine repair, as well as grease under the fingernails, but estimate the actual cost of repairs and replacement parts as about $1000/yr., which is certainly a lot less then $52,000. In addition, as Joe likes to say, the liquefier has proven to be an inexaustible source of entertainment and education. How many people can tell you that 1 lb. of liquid He is 3.63 l. , that 1 l. of liquid He corresponds to 26.3 cu. ft. of gas at room temperature, or that 1 lb of activated charcoal at 77 K absorbs 4 cu.ft. of air before the air content of the He stream rises to 0.1%?

What's the bottom line? We think if your lab uses 10,000 l. or more of liquid He per year, and if you have a certain masochistic streak, it's probably worthwhile for you to consider liquefying your own.

Joe Dadok Aksel Bothner-By

LC-NMR: Separations and Structures



On-flow 500 MHz ^1H LC-NMR data of a peptide mixture using Scout Scan and WET solvent suppression during an LC gradient. The sample was a 100 µl injection of ~1 mg/mL peptides separated using a CH_3CN:D_2O solvent gradient (5% CH_3CN to 50% in 22 min).

Combining the superior separation capabilities of HPLC with the exceptional structure elucidation capabilities of NMR, Varian's exciting new LC-NMR accessory is the most powerful and versatile tool for examining the chemical structures of complex mixtures.

Recent advances in the areas of NMR sensitivity, multiple solvent suppression, modular design, and the control of hardware on-the-fly have made the use of LC-NMR truly practical for solving difficult research problems. As drug and other product development cycles decrease, laboratories worldwide are taking advantage of new technologies which directly impact time-to-market. This innovative hyphenated technique eliminates the need for time-consuming sample isolation and purification steps, thereby streamlining research efforts and liberating resources.

Available for all UNITY NMR spectrometers including the new UNITY*INOVA*, the LC-NMR accessory includes a flow NMR probe, a complete LC system (pump, variable wavelength UV detector, PC, column, etc.), a peak sense module, an interface between the LC and NMR systems, and a comprehensive LC-NMR software package.

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Features	Benefits
• Multiple NMR flow cell volumes	Optimizes chromatographic resolution and S/N for flow and fraction collection methods
• Peak sense module	Automatically positions analyte in NMR probe coil region based on UV or other HPLC detector signal
• Multiple HPLC detectors	Variety of detectors ensures that most chemical species can trigger stop-flow NMR acquisitions
• Modularity of analyte collector	True off-line fraction collection capability provides optimal utilization of NMR spectrometer
• Flexibility of analyte collector	Generic loop collector module provides compatibility with other LC equipment
• Scout Scan™	Perfectly adjusted, on-the-fly solvent suppression for on-flow and stop-flow LC gradient applications
• WET solvent suppression	Combination of selective excitation with pulsed field gradients optimizes multiple peak solvent suppression
• ^{13}C sideband suppression	Efficient removal of ^{13}C sidebands of non-deuterated solvent peaks provides a cleaner NMR spectrum for analyte identification
• NMR "time slicing" of chromatogram	Facilitates identification of individual components from unresolved or coeluting species
• Compatible with non-deuterated organic solvents	Excellent solvent suppression removes the requirement for costly deuterated solvents such as CD_3CN and CD_3OH
• Compatible with non-volitile buffers	Easily allows study of buffered systems which are very difficult for LC-MS
• Powerful & flexible VNMR software	Variety of processing and display macros allows facile examination of LC-NMR data
• Control of LC injections from NMR console	Advanced interface allows operator to initiate LC-NMR experiment from NMR console
• Hypertext, linked on-line LC-NMR manual	Dramatically reduces the learning curve for LC-NMR operation at the spectrometer

LC-NMR Applications

- Drug metabolism
- Drug impurity assays
- Coeluting compounds
- Fermentation studies
- Clinical chemistry
- Peptide synthesis
- Purity of bulk materials
- Synthetic chemistry monitoring
- Lability and degradation studies
- Isomeric ratios for synthetic reactions
- Extent of polymerization
- Functionalization of synthetic polymers
- Chemical waste analysis
- Reaction kinetics
- Molecular complexation
- Monitoring of chemical reactions on-line
- Quality control
- Transition state monitoring
- Tautomerism monitoring

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UMEÅ UNIVERSITET
Avdelningen för organisk kemi
Professor Ulf Edlund

UMEÅ UNIVERSITY
Department of Organic Chemistry
Professor Ulf Edlund

(received 8/28/95)

1995-08-22

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

Dear Barry,

Pressure induced transformation of European footballs into American ones.

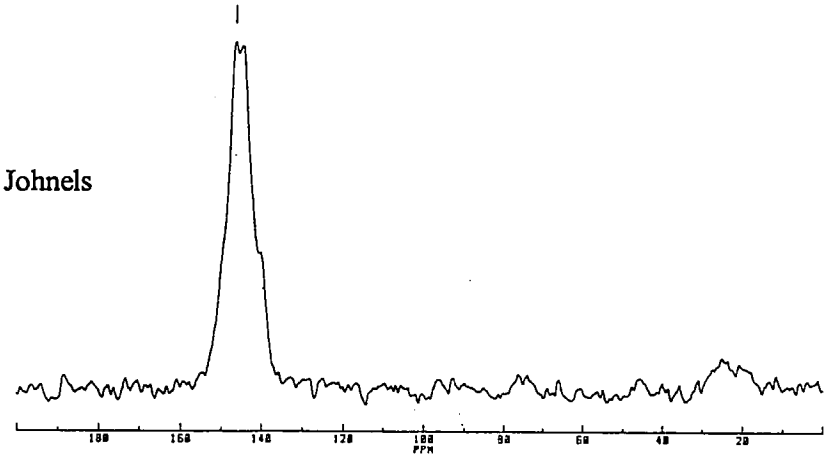
Together with people from the solid state department we have pressure-treated C_{60} at high temperature under carefully controlled conditions. After 2 hours the sample was cooled down and whereafter the pressure lowered to zero during 30 min.

The MAS spectra at two fields showed clearly a polymeric phase but the shift spread is much reduced relative what is earlier reported for material from pressure or photoinduced polymerizations. We observed much longer T_1's than for pristine C_{60} and also as expected significant sidebands at high field. The frequencies covered are similar to the domain for C_{70}. No sign of any sp^3 carbons around 67 ppm or so, not even after enormous delay times. The dissociation of the obtained material starts again at about 200° C, again a different from the phototransformed stuff (around 120°C). Raman spectra are also deviating from those earlier reported.

So what could the interaction be like? The absence of any sp^3 signals (but T_1 could be extremely long) and the very minor shift spread are against normal covalent bonds between the spheres. Of course we are eager to figure this out!

Best regards

Ulf Edlund and Dan Johnels



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TEL. (802) 656-2220
FAX: (802) 862-8229

Easy to Use (Really!) Automated Chemical Shift Matching

Dear Barry,

August 10, 1995
(received 8/31/95)

Prior to now I have been able to procure a copy of the NMR Newsletter to read from those around me. However, given the new location, I suppose it is time to make a contribution and "pull my weight in the traces", so to speak.

Currently our research is at the end stage of sequential assignments of a 112 amino acid domain of a protein involved in the cancer signaling pathway. This is work begun, and continuing with, Drs. Mary Senior, Yu-Sen Wang, and Anne Frederick at the Schering Plough Research Institute.

As everyone knows, the bookkeeping and chemical shift matching requirements inherent in analyzing multiple 3D data sets such as HNCA, HN(CO)CA, CBCA(CO)NH, HNCACB, etc., can be challenging. Many researchers write their own software to handle this situation. For the present anyway, I have chosen to speed up this process by using the search and match capabilities supplied in the NMRCompass ™ software developed by Molecular Simulations Inc.

As a basic example, all the possibilities of matches between a particular sequential Cα chemical shift found in the HN(CO)CA and the corresponding intra Cα chemical shift of the HNCA must be found. This process must be completed for all the Cα carbons in the protein. The software in question does this quite efficiently for the entire protein within a minute or two on an SGI Indigo II. I have found an easy way to do this is to take the HNCA data set, perform an automatic peak pick, followed by removal of non-relevant peaks. The HN(CO)CA data set is used to verify which peaks are sequential as opposed to intra, but is not used in the majority of cases to determine the exact chemical shifts of the sequential peaks. Since in the majority of cases, the HNCA contains both the intra and sequential peaks, both resonance shifts are determined from this single data set. This helps to eliminate chemical shift discrepancies between the two data sets. The HNCA peak list is then separated into two peak lists: hnca.seq.pks and hnca.intra.pks, and these two peak lists are searched against each other using the following criteria file:

- Peaks Tables Selected:
- Index Table name Index Table name
- 1. hnca.intra.pks 2. hnca.seq.pks
- Target table index: 2

Define Source and Target Frequency Axes for Matching.
Use table and peaks index to define matching criteria.

Target Table: Source Table(s):

☐ X(PPM):	table index:		◯ X(PPM) ◯ Y(PPM) ◯ Z(PPM)	
	peaks index:		tolerance(PPM):	
☒ Y(PPM):	table index:	1	◯ X(PPM) ◉ Y(PPM) ◯ Z(PPM)	
	peaks index:	1	tolerance(PPM):	0.1
☐ Z(PPM):	table index:		◯ X(PPM) ◯ Y(PPM) ◯ Z(PPM)	
	peaks index:		tolerance(PPM):	

☐ Optional Criteria

End Next Previous Cancel

In our HNCA data set, the y dimension corresponds to Cα, and so we want to search the Cα (y) dimension of the source table (hnca.intra.pks) against the Cα (y) dimension of the target table (hnca.seq.pks). We will soon extend these criteria to include the Cβ frequencies from the 3D HNCACB and CBCA(CO)NH data sets by changing the peak index number from 1 to 2.

The only caveat to this type of search is the usual one: "Garbage In Equals Garbage Out". If the peak lists are not carefully edited to remove spurious picked peaks the number of possibilities of matches increases dramatically. This results in a hung computer which grinds endlessly away until the process is terminated by the user.

Barbara A. Lyons

SmithKline Beecham
Pharmaceuticals

September 18, 1995
(received 9/21/95)

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

Auto- MRI

Dear Barry,

In order to increase the throughput of *ex-vivo* microscopy experiments on our vertical bore 9.4 T system, we needed to automate the data collection process. Given the success of the 'auto sample-changer' in the applications of high resolution NMR, we approached Bruker with the idea of developing a sample changer for micro-imaging experiments. Of course, their first reaction was '*we were crazy*'. However, after repeated conversations with them, we finally convinced Bruker of the utility of a sample changer for micro-imaging applications.

The two major requirements to build such a sample changer turned out to be; i) the need to handle large (up to 30 mm) diameter sample tubes and ii) the need to maintain the orientation of the sample as it is inserted into the magnet. After several discussions with colleagues at Bruker about the design, a prototype sample changer was constructed and delivered to us. We would particularly like to acknowledge the co-operation of Drs. Burum and Rindlisbacher of Bruker in this project.

The sample changer can hold a maximum of seven samples (diameters ranging from 5 to 30 mm) and sits on the top of the magnet dewar. It can easily be removed when not in use. It is controlled by a simple pneumatic system which is activated by software commands from the console. The samples are mounted in special spinners with two grooves diametrically opposite to each other, which fit over two rails in a tube placed in the bore of the magnet. This maintains the orientation of the sample as it is inserted into the magnet.

709 Swedeland Road, PO Box 1539, King of Prussia, PA 19406-0939. Telephone (215) 270 4800. Fax (215) 751 3400.

The sample changer has been in use in our laboratory for a few months now and has performed as expected so far. Now, we can really work 24 hours a day, 7 days a week (provided our house air supply does not shut off). The availability of a sample changer for micro-imaging could have a significant impact on throughput, not only for biological applications but also for applications in materials imaging.

Sincerely,

Rasesh D. Kapadia

Susanta K. Sarkar

Back Issues, Anyone?

Are your missing any back issues of the Newsletter? Are any of your copies battered and bruised? Do you need some copies for Aunt Martha's birthday present, cousin Johnny's graduation, or a St. Swithin's Day grab bag?

A few copies of many back issues of are available, including most of those of the past three or four years. If you are interested, please contact us soon, before the need to reduce the amount of inventory overcomes our tendency to save all pieces of paper.

For the issues of September 1993 and before: Prices for copies ordered prepaid without an invoice (checks payable on U.S. banks only, svp): 1-5 issues, $5.00 each; 6 or more issues, $4.00 each. All prices include postage for printed matter surface mail shipping. For air mail printed matter shipping, inquire. Please call about issues since September 1993, or if you must have an invoice. (All orders from Scotland are hand-carried without charge except for a transportation fee - inquire.)

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Barry & Lee Shapiro

The NMR ~~Re~~evolution advances...

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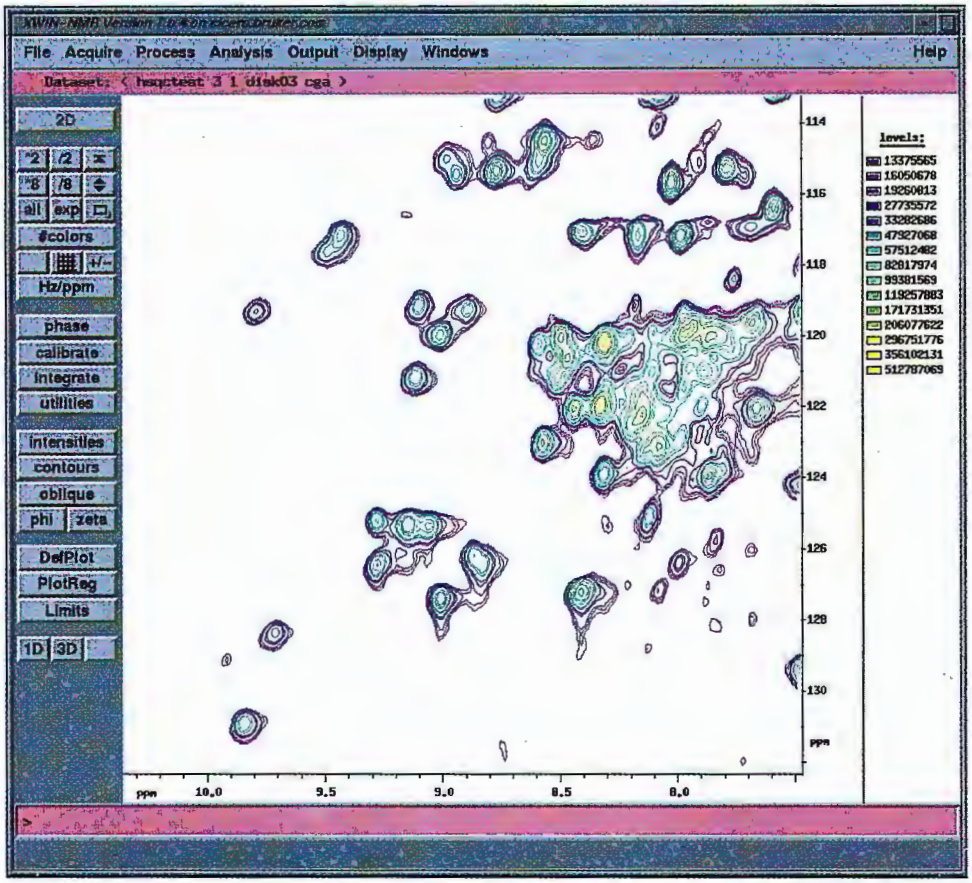
XWIN-NMR™ Software



Bruker introduces *XWIN-NMR™* software with a completely new Graphical User Interface (GUI) and screen layout for the acquisition and processing of NMR data on the *AVANCE* as well as Silicon Graphics based AMX, ARX and ASX spectrometers. ***The result of the new interface is a logical layout for easy navigation and intuitive operation.***

Moreover, the new GUI is immediately familiar and comfortable to users of other WINDOWS-based software packages, as *XWIN-NMR* uses industry standard software conventions. Even the "file" pull-down menu is in its usual location.

XWIN-NMR is derived from UXNMR, Bruker's previous software package, which is known as a very powerful, feature-rich NMR software package. While still maintaining these features, *XWIN-NMR* simplifies their operation. "Tear-off " menus and other routines allow users to create their own customized environments.

BRUKER

...The NMR Revolution advances

Some of the new features and advantages of *XWIN-NMR* are summarized below. Future application notes will elaborate on these and other features.

New features	*Advantages*
• Standard software conventions	➤ intuitive and familiar
• Logical, hierarchical design	➤ easy to use
• Comprehensive functionality	➤ most powerful NMR applications software
• Graphical pulse program simulator for *AVANCE* series spectrometers	➤ 12.5 nsec resolution pulse program display
• On-line hypertext manual	➤ modern documentation
• Menu based Quick NMR package	➤ easy to use
• Plot Editor	➤ easy to use
• Software BSMS control	➤ lock/shim control from any networked workstation
• Shaped Pulse Editor	➤ easy to use

Please contact your local representative to hear more about *XWIN-NMR* and our other new products. We look forward to hearing from you.

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Telephone: (905) 525-9140
FAXMAIL (905) 522-2509

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

13 September, 1995
(received 9/19/95)

New DRX Console for a Bruker AM 500

Dear Barry,

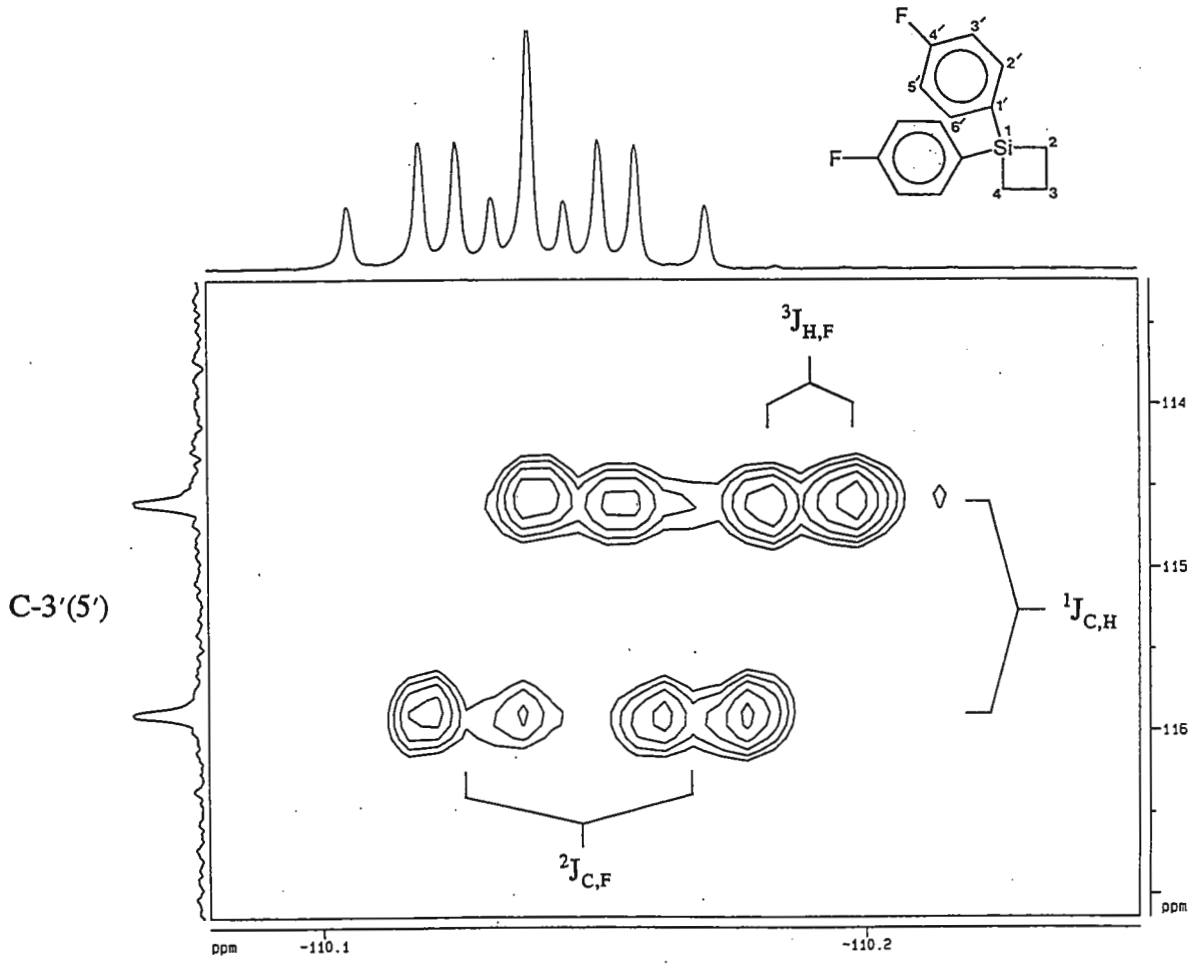
We recently took possession of a new console for our 10 year old AM 500 and were immediately impressed by the increase in sensitivity (approx. 30%) with our existing probes. When our new Bruker probes arrived, it was pleasing to find that they easily met "new" system specifications with our existing 10 year old Bruker magnet and a further similar increase in sensitivity was observed. The proton coil on all of the probes is readily tunable to ^{19}F which has added a new dimension to our capabilities.

The new ^{19}F capabilities are allowing us to examine the use of ^{19}F decoupling, polarization transfer and ^{19}F detected 2-D inverse correlation experiments in both organic and inorganic compounds. Two examples of ^{19}F detected HMBC experiments are shown on the following page. Contour plot (a) shows the results of a ^{19}F - ^{13}C HMBC experiment on a fluorine substituted 1,1-diphenylsilacyclobutane in $CDCl_3$. The fixed delay in the pulse sequence was optimized for a $^{2}J_{C,F}$ = 20.1 Hz. The fully coupled spectrum displays not only $^{1}J_{C,H}$ and $^{2}J_{C,F}$ but also $^{3}J_{H,F}$ for the proton directly bonded to C-3'(5'). The F1 ^{13}C spectrum was obtained with ^{19}F decoupling. Figure (b) shows the results of a ^{19}F - ^{15}N HMBC experiment acquired with the use of pulsed field gradients on a neat sample of perfluoropyridazine. The pulse sequence delays were adjusted for $^{2}J_{N,F}$ = 52.0 Hz. The correlation of the fluorine signal at -94.8 ppm with the nitrogen resonance at -53.0 ppm confirmed the assignment of the C-3(6) fluorines. The F1 ^{15}N spectrum was obtained with the INEPT pulse sequence using ^{19}F polarization transfer via the 2-bond coupling interaction. We plan to apply these types of experiments to other nuclei.

Alex D. Bain
(Professor, Chemistry)

Donald W. Hughes
(NMR Applications Specialist)

Brian G. Sayer
(Manager NMR Facility)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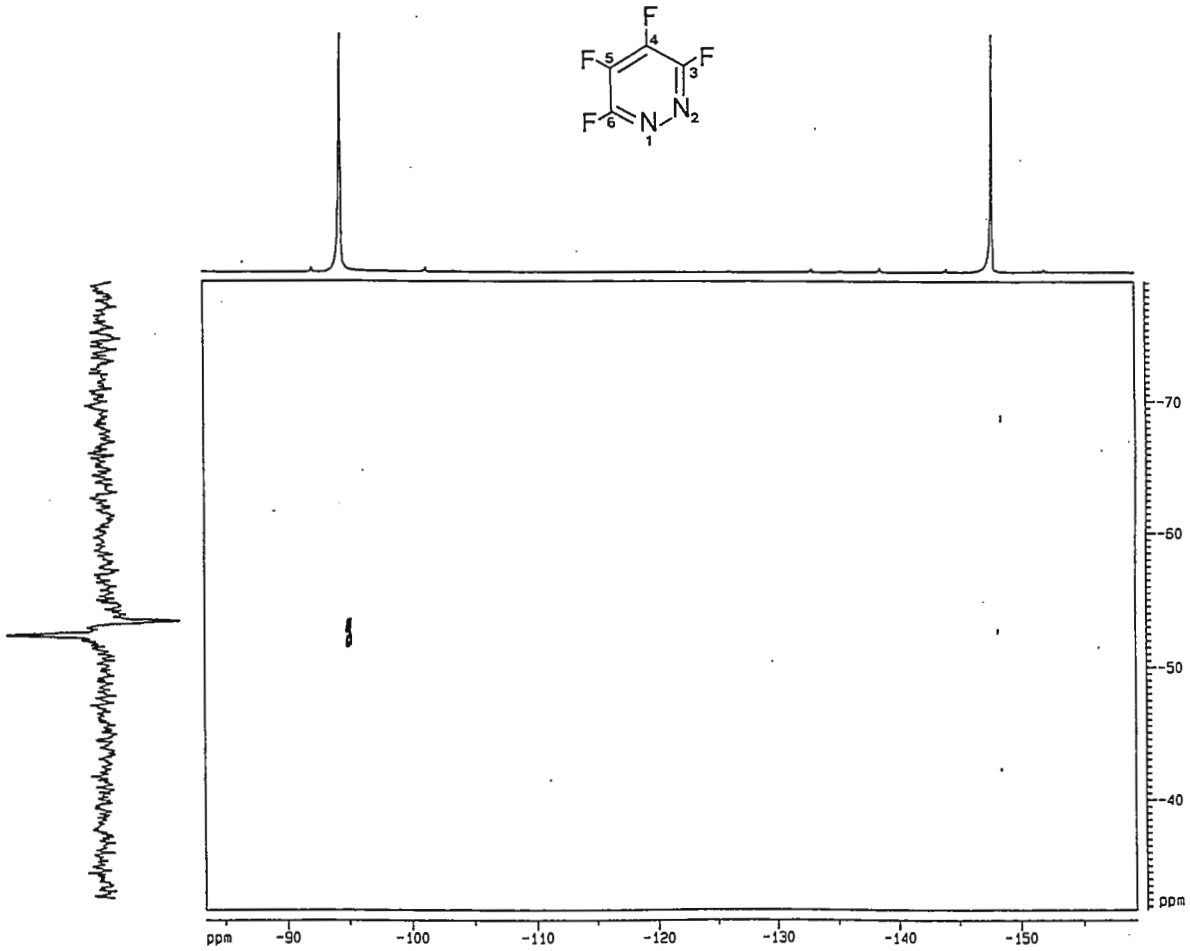
(a)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(b)

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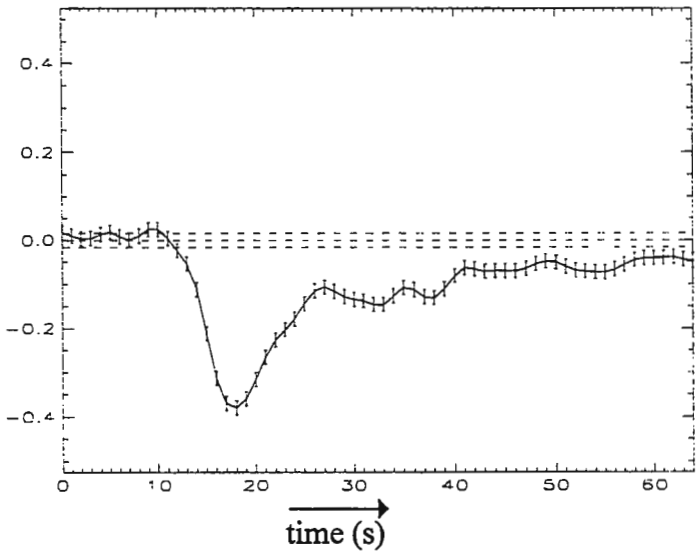
Prof. B.L. Shapiro
Publisher NMR Newsletter
966 Elsinore Court
Palo Alto, CA 94303

Your reference and date	Our reference	Office telephone	Date
		31 15 786041	13-Sep-95.
Subject		Sub-division	(received 9/19/95)

Dear Dr. Prof. Shapiro,

IMAGING OF PERFUSION BY PROTON MAGNETIC RESONANCE

Monitoring the dynamic passage of an injected Gd-DTPA bolus by NMR imaging methods can be used to determine the blood volume and perfusion rate. As the maximum intensity changes due to the bolus passage take place after about 10 seconds in normal rat brain, a good quantification of the perfusion requires a time resolution of a few hundred milliseconds. Two kinds of sequences with a resolution of 90x90, field of view 3x3 cm, were incorporated on our 6.3 T animal system: 1) a T_2^* weighted FLASH imaging sequence, T_R=12 ms, T_E=7 ms, and a total acquisition time per image of 1s; 2) a multi-shot echo planar imaging (EPI) sequence with 5 to 10 echo's per shot. A typical EPI sequence uses 6 echo's per shot and 15 shots, requiring 265 ms per image. For bolus tracking 60 images with a delay of 5 ms between two images are used. In the figure this is demonstrated for transversal images of a healthy rat head; from left to right; reference, image during a bolus passage, the two corresponding difference images. The time series of the bolus passage is determined from the difference images in the indicated area. These FLASH and EPI sequences allow a good quantification of the bolus passage and a determination of blood volume and flow.



Sincerely yours,

W.M.M.J. Bovée T.H. Rozijn

Position Available

The Laboratory of Developmental Neuropsychology at the San Francisco VAMC is seeking to fill the position of **Assistant/Associate Specialist** whose job it will be to manage all aspects of brain MRI analysis in the lab and share scientific responsibility for this effort with the Director, Dr. George Fein. In addition, the individual will be responsible for the support of collaborative brain MR analysis in other laboratories on and off campus. Currently funded projects include subject and control groups in the evaluation of Alzheimer's disease, HIV disease, alcohol and drug abuse, schizophrenia, and subcortical ischemia dementia. **Tasks attendant to this position include:**

1. Efficient data management.
2. Review of MRI literature pertinent to the target groups.
3. Participation in planning of MRI acquisition and analysis protocols.
4. Creating and testing protocols for the anatomical segmentation of MRI's; determination of inter- and intra-operator reliability.
5. Working with programmers on software optimization needed to accomplish MRI analysis.
6. Liaison with MRI technologists for quality control of studies.
7. The use of custom computer software to analyze volume and structure of the MRI studies.
8. Statistical analysis of the MRI segmentation results with Dr. Fein.
9. Participation in manuscript and grant writing (at first and co-author levels) and dissemination of research methods and results at professional meetings and conferences.
10. Recruitment, training, and supervision of other lab personnel involved in brain MRI analysis.

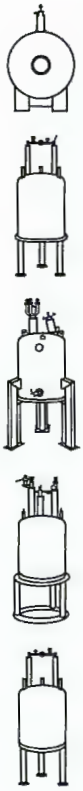
Applicant requirements include:

1. Master's degree or above in a related field.
2. Excellent grasp of neuroanatomy and MRI.
3. Excellent analytical skills.
4. Computer skills that focus on image analysis.
5. Familiarity with statistics and data analysis.
6. Excellent writing skills.
7. Excellent planning, organizational, supervisory, and teaching skills. All applications should be addressed to:

David Norman, MD, c/o Linda Seabright, VAMC (116R), 4150 Clement Street, San Francisco, CA 94121.

Deadline for receipt of applications is: November 5, 1995.

Beware of imitations



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750	51	15	60	3.8
600	51	10	120	3.4
500	51	10	150	3.2
400	54	8	365	2.8
360	54	8	365	2.8
300	54	3	365	2.8
270	54	2.7	365	2.8
200	54	2	365	2.8
100	54	1	365	2.8
500	89	15	120	3.4
400	89	10	180	2.8
360	89	10	365	2.8
300	89	3	365	2.8
270	89	2.7	365	2.8
200	89	2	365	2.8
100	110	1	119	2.8

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NMR UNIT
September 15, 1995
(received 9/20/95)
Dr. Bernard L. Shapiro
The NMR Newsletter
966 Elsinore Court
Palo Alto, CA 94303

NMR MICROIMAGING OF MOUSE TAIL

Dear Barry:

As part of our general interest in connective tissue and changes with disease and aging, we are looking at collagen in mouse tail tendon. Figure 1 (next page) shows a microimage (31.25 μm pixel resolution) of a mouse tail. One can clearly see the tendon fibrils as regions of low intensity. Preliminary studies have shown that the muscle region of interest has a spin-spin relaxation adequately described by a monoexponential. For the tendon, a biexponential better characterizes spin-spin relaxation of H_2O, with two distinct water sites, one with a short T_2 and another with a long T_2. The exact T_2 values obtained with a standard spin-echo imaging sequence underestimate the actual relaxation times due to diffusion attenuation of the signal, especially at longer echo times (TEs). Nonetheless, they do indicate multiexponential spin-spin relaxation for T_2 of H_2O in tendon fibrils, presumably due to multicompartmentalization of H_2O. We are exploring different approaches to obtaining accurate T_2 values. Our goal is to obtain information on water mobility and binding, and on macromolecular structure and dynamics at the water-tendon interface in normal, aging, and diseased states *in vivo*.

Sincerely,

Fernando Commodari, Ph.D.
IRTA Fellow

Richard G. S. Spencer, M.D., Ph.D.
Chief, NMR Unit

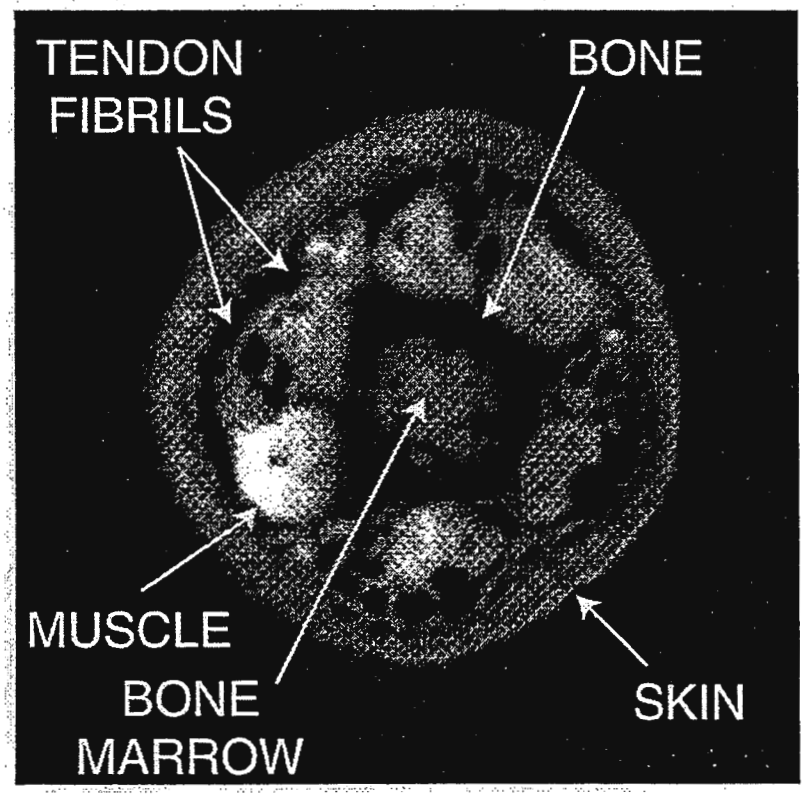

Figure 1. NMR microimage of an excised mouse tail obtained at 400 MHz using a standard 2D spin-echo sequence with 2 scans per phase encode increment, an acquisition FOV of 8mm x 8mm, a 256 x 256 MTX, a slice thickness of 2mm, TE = 8 msec, TR = 1.5 sec and a pixel resolution of 31.25 μm. The maximum vertical and horizontal radial distances for the tail are, respectively, 3.2mm and 3.3 mm.

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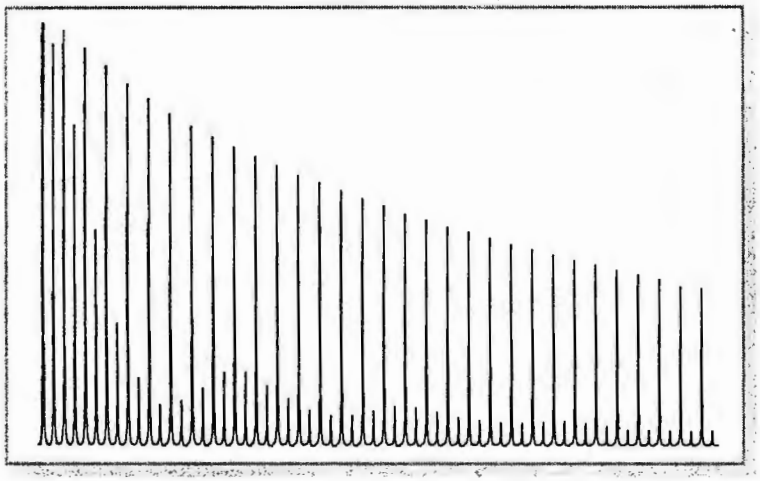
- •Obtain minimum frequency separation between the X and Y channels when needed.



$^{13}C/^{15}N$ REDOR with ^{1}H decoupling, obtained on [2-^{13}C,^{15}N]-glycine.

^{1}H decoupling field, stable RF and stable spinning speed are all critical for REDOR experiments.

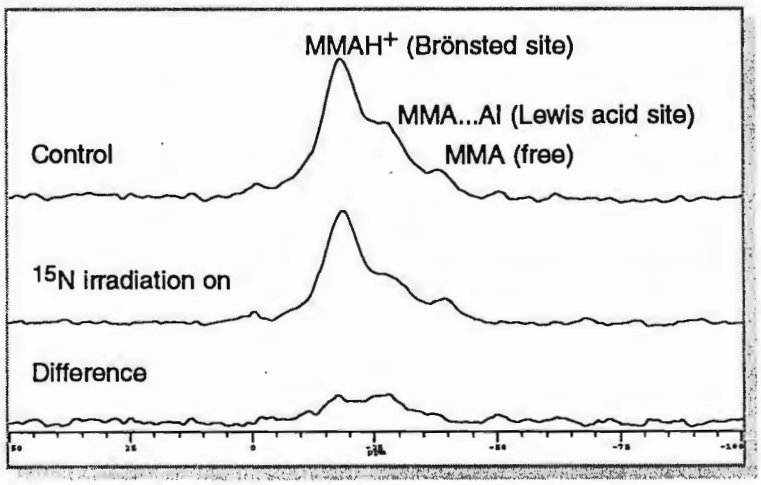
Control experiments (1st, 3rd, etc. peaks) demonstrate high decoupling powers. Significant signal remains after 64 rotor periods, as seen in the next to last peak.

$^{27}Al/^{15}N$ TRAPDOR with ^{1}H decoupling, of monomethyl amine (MMA) on a zeolite surface, obtained at -140°C to freeze amine motion on the zeolite surface.

Stability in probe tuning and spinning speed must be maintained at -140°C in order to obtain TRAPDOR data.

The TRAPDOR technique is similar to REDOR in that distance information is obtained through dipolar couplings.

data courtesy of C. Grey, SUNY, Stony Brook.



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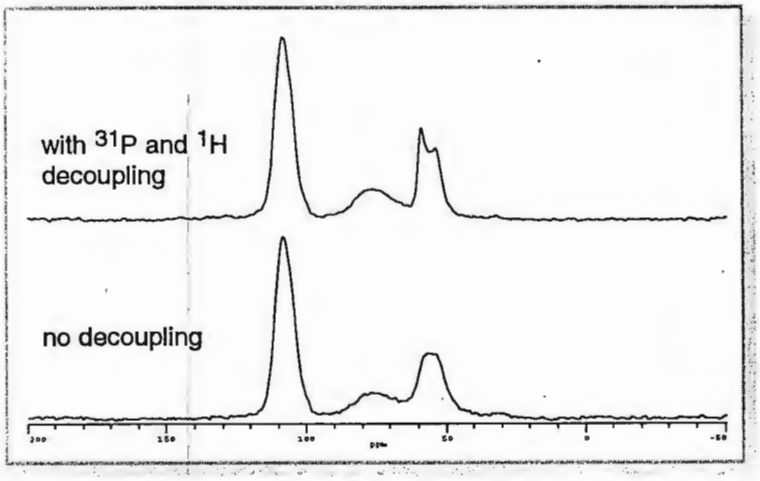
Chemagnetics Triple Resonance Technology



^{27}Al Observation with X/H decoupling, of $AlPO_4$-H2.

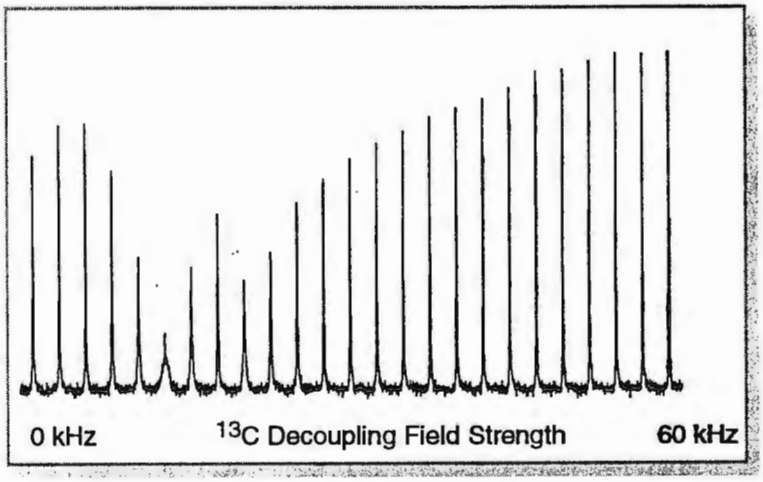
Decoupling of the ^{31}P and ^{1}H nuclei provides enhanced resolution in the ^{27}Al spectrum.

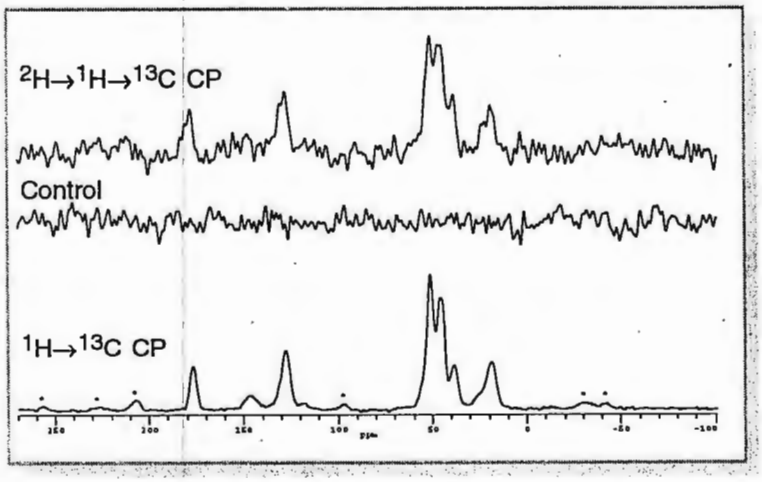
The signal-to-noise does not degrade with addition of ^{1}H and ^{31}P decoupling. This is the result of good X/Y channel isolation in the probe and filtering between the probe and receiver.

^{15}N Double-Cross Spectra of [2-^{13}C,^{15}N]-glycine. Cross polarization is performed in the direction $^{1}H \rightarrow {}^{13}C \rightarrow {}^{15}N$.

As the ^{13}C decoupling field increases from left to right, the noise level remains the same. Signal-to-noise is best with a sufficient level of ^{13}C decoupling.

Minima in peak intensities correspond to ^{13}C decoupling fields equal to and at twice the spinning frequency.





^{13}C CP and Double-Cross Spectra of d_8-PS/PMMA copolymer.

$^{1}H \rightarrow {}^{13}C$ CP shows peaks from both PS and PMMA components of the copolymer, indicating intimate mixing of the two materials. The $^{2}H \rightarrow {}^{1}H \rightarrow {}^{13}C$ double-cross spectrum demonstrates ^{2}H polarization transfer to ^{13}C via ^{1}H's. The Control Experiment, with ^{2}H CP power off, shows that all double-cross signal originated from ^{2}H.

*spinning sidebands. Sample/idea courtesy of N. Zumbulyadis, Eastman Kodak.

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Dr. B.L. Shapiro, Publisher
The NMR Newsletter
966 Elsinore Court
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19 September 1995
(received 9/22/95)

Xenon Gas Diffusion in Polystyrene

Dear Barry:

We have devised a method for measuring diffusion constants of sorbed gas in polymers using polymer microspheres. The method involves measuring chemical shift exchange between the sorbed and free gas.

^{129}Xe is an ideal example as there is a large chemical shift between the polymer sorbed and free xenon gas. The exchange is monitored using the classic Forsen/Hoffman selective saturation experiment when the decay of the sorbed signal is monitored as a function of saturation time of the free gas signal. Diffusion is described mathematically as diffusion out of a sphere.

The deficiency of the Xe magnetization at point r from the center of the polymer sphere at time t is $M_0 - M_z(r,t)$ and the time dependence of the magnetization can be written as:

$$\partial M_z(r,t)/\partial t = D\nabla^2 M_z(r,t) + [M_0 - M_z(r,t)]/T_1$$

Solutions of this equation are available and the decay of magnetization can be fitted to the appropriate solution. A typical fit is shown in the diagram, and measurements as a function of temperature yield an Activation Energy of 36kJ/mole for the method has applicability if uniform microspheres can be obtained and if a reasonable chemical shift dispersion exists between sorbed and free gas

POSITION AVAILABLE.

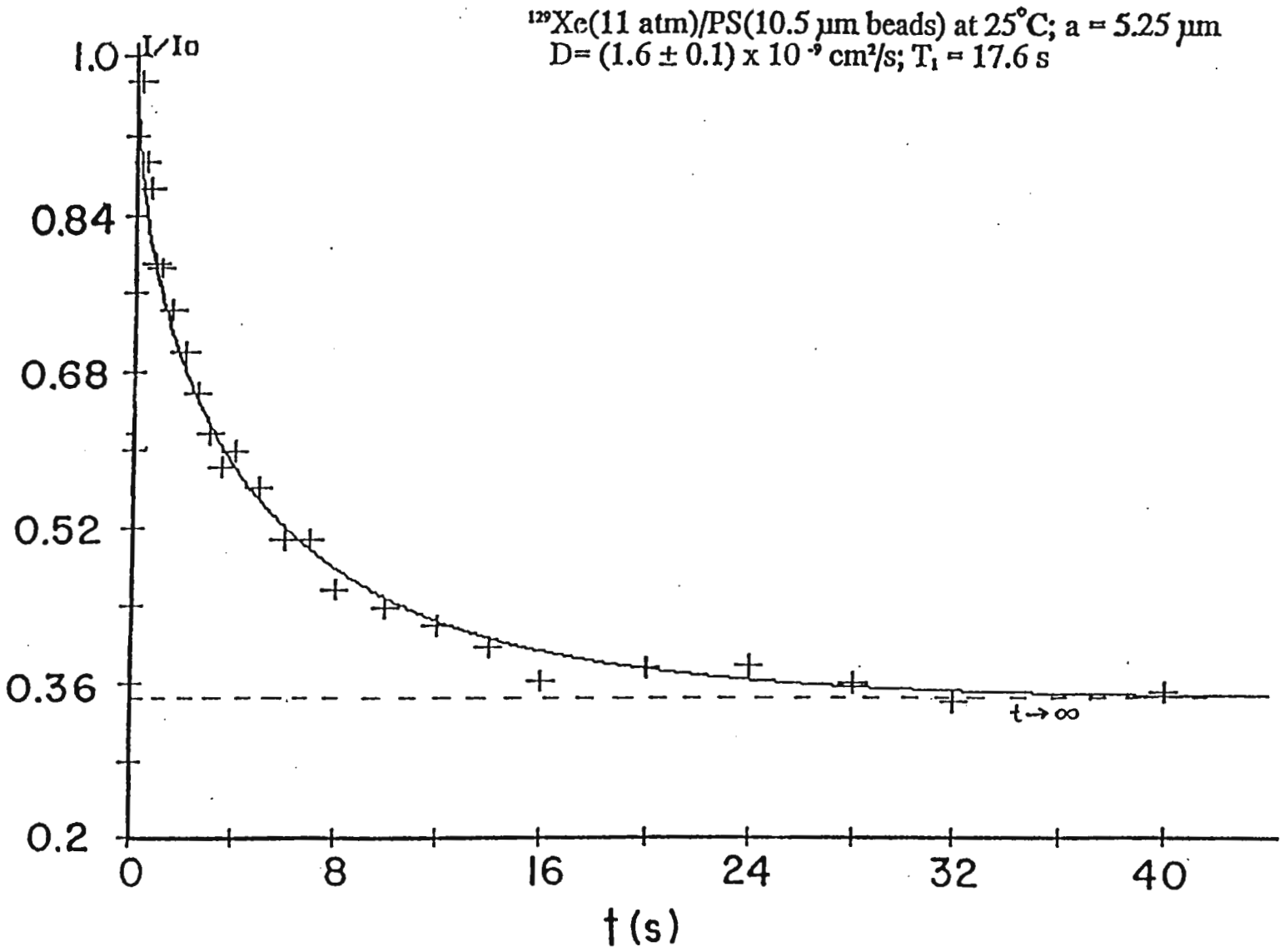
A Post-Doctoral Fellowship is available at Clark University for the study of Solid State NMR of Polymers. The Fellowship is for two years funded by NSF with a salary of $26,000. per annum.

Experience in solid state NMR techniques including 1 and 2D Lineshape analysis is desirable. The study involves computer simulation of motional models to analyze NMR data. The use of a variety of solid NMR techniques including wide line, narrow line and spin diffusion will be employed. Some knowledge of polymer systems is also desirable.

Sincerely,

Paul T. Inglefield
Professor & Director NMR Facility

PTI/ecb


^{129}Xe(11 atm)/PS(10.5 μm beads) at 25°C; a = 5.25 μm
D= (1.6 ± 0.1) x 10 $^{-9}$ cm²/s; T_1 = 17.6 s
I/Io
1.0
0.84
0.68
0.52
0.36
0.2
t→∞
0
8
16
24
32
40
t (s)

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Pulse width	20 ms	20 ms
Duty cycle	Up to 10%	Up to 10%
Amplitude droop	5% to 20 ms typ.	5% to 20 ms typ.
Harmonics	Second: -25 dBc max. Third: -24 dBc max.	
Phase change/output power	10° to rated power, typ.	
Phase error overpulse	4° to 20 ms duration, typ.	
Output noise (blanked)	< 10 dB over thermal	
Blanking delay	< 1 µ s on/off, TTL signal	
Blanking duty cycle	Up to 100%	
Protection	1. Infinite VSWR at rated power 2. Input overdrive 3. Over duty cycle/pulse width 4. Over temperature	

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System monitors	1. Forward/Reflected RF power 2. Over pulse width/duty cycle	3. DC power supply fault	4. Thermal fault
Front panel controls	1. AC power	2. Forward/Reflected power	
AC line voltage	208/230 VAC, 10%, 1Ø, 47-63 Hz		

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Department of Neurology

Professor B.L. Shapiro,
TAMU NMR Newsletter,
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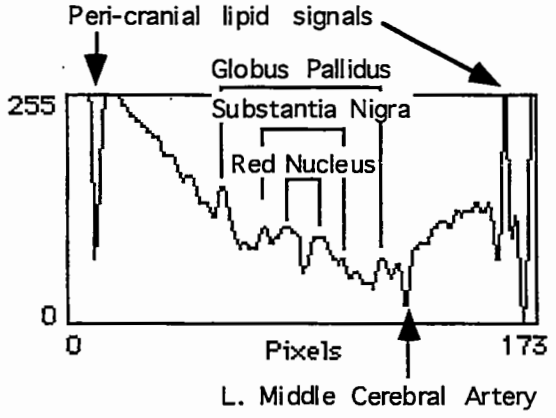
September 13, 1995
(received 9/18/95)

MORE FIELD MAPPING/ POST-DOCTORAL POSITION AVAILABLE

Dear Professor Shapiro,

Thank you for your recent fluorescent ultimatum. Our previous contribution to THE NEWSLETTER (#435, p. 25) was concerned with the use of phase-angle reconstruction of gradient-echo MR images, for the purpose of measuring variations in the static magnetic field $\mathbf{B}_0$. Such information may be useful for automatic shimming routines, or for frequency and lineshape corrections in spectroscopic imaging data.

Applying this technique to the human brain lead to a number of observations, only some of which were expected. First, it is well known that the phase angle will depend on both chemical shift and $\mathbf{B}_0$. While the proton NMR spectrum of brain is almost 100% water (cerebral lipids have very short T_2's under normal circumstances), phase angle imaging proved very sensitive at detecting small mobile lipid signals, for instance found in *lipomas*, or in the bone marrow of the *falx cerebri*. Second, flow in the presence of a field gradient induces a phase shift, and it was often possible to visualize major vessels (such as the *middle cerebral artery*) in phase images, just as in phase-contrast angiography. Finally, bulk susceptibility shifts resulting from paramagnetic species also induce phase shifts. Such species include both exogenous contrast agents such as GdDTPA or indigenous materials such as iron. One such example is shown below, which is a trace extracted from a phase image, passing through various structures within the brain.



On another subject, I have an immediate opening in my lab for a post-doctoral fellow to develop imaging and spectroscopy techniques primarily for human brain studies. Equipment available includes 1.5 and 3.0 Tesla whole-body MR scanners and a 7.0 Tesla, 20 cm horizontal bore system. Interested parties can contact me either via email or at the above address,

Sincerely Yours,

Peter Barker.

Peter B. Barker. EMAIL: barker@neurnis.neuro.hfh.edu

The NMR Newsletter - Book Reviews

Book Review Editor: **William B. Smith**, Texas Christian University, Fort Worth, TX 76129

" Biomolecular NMR Spectroscopy "

by

Jeremy N. S. Evans

Oxford University Press, New York, 1995;
ISBN 0 19 854767 6 (Hbk), 0 19 854766 (Pbk);
439 pages + index; Paperback, $49.95.

This textbook is directed to those students of biochemistry, biophysics and allied fields who require a background in the application of NMR to the solution of problems in molecular structure and properties. An indication of the content is given by the chapter headings and the number of pages devoted to each:

Part I: Theory. 1, Introduction: 53 pages; 2, Methods of spectral assignment - multidimensional NMR: 60 pages; 3, Obtaining NMR structures: 27 pages.

Part II: Proteins. 4, Proteins: 61 pages; 5, Protein folding: 18 pages.

Part III: Enzymes. 6, Enzyme function: 31 pages; 7, Acyl and phosphoryl transfer enzymes: 39 pages; 8, Other enzymes: 30 pages.

Part IV: Nucleic Acids and Carbohydrates. 9, Nucleic acids and carbohydrates: 52 pages.

Part V: Membranes. 10, Membranes and membrane proteins: 21 pages.

Appendices. There are four appendices: 1, Examples of product operator calculations; the COSY pulse sequence; the REDOR pulse sequence. 2, Useful NMR data on amino acid residues. 3, Useful NMR data on oligonucleotide residues. 4, Useful NMR data on common solvents.

Index. A six-page subject index is provided, but no author index.

In the sections starting with Part II, the techniques described in the earlier parts of the text are applied, and numerous interesting examples with complete literature references are given. Persons wishing to know the state of the art will find these sections to be valuable. There are references to a variety of computer programs for molecular modeling and solutions to structure problems.

The introductory parts of the text are best described as hurried. If this book is used as a classroom text, the instructor will find plenty of places where details and illustrative examples can be added. Chapter 2 covers all of 2-D, 3-D and 4-D spectra in 60 pages. I question the value of introducing product operators in two pages and then applying them to a variety of 2-D experiments at a high level. Better introductions can be found in the Handbook by Freeman or the Dictionary by Homans (both reviewed here previously). The majority of potential readers will be NMR users; not developers of new pulse sequences. Verbal descriptions of these experiments a la Derome or Saunders and Hunter would better serve their purposes.

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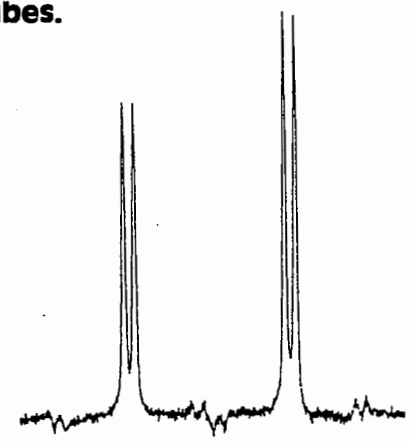
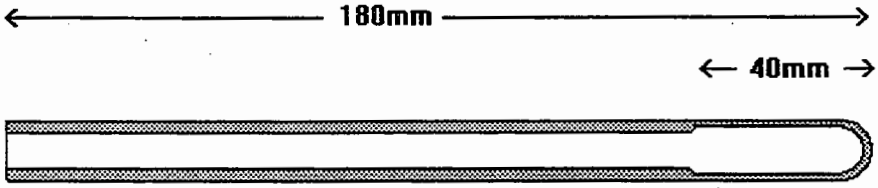
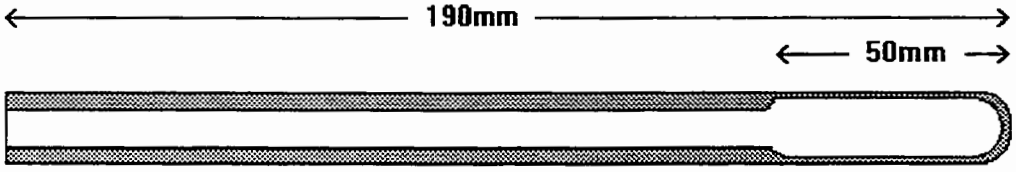


Fig.1a Fig.1b

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



PST-001 and PST-002



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

O.D. (mm)	Product Number	Wall (mm)	Concen-tricity/Camber (μ)	OD (mm)	ID (mm)	Price Each 1-99	Price Each 100+
5	PST-001	0.21	20/8	4.96 + 0.00 - 0.01	4.54 ± 0.01	$15.00	$13.50
	PST-002	0.21	40/15	4.96 + 0.00 - 0.01	4.54 ± 0.01	$13.00	$12.00
8	ST8-001	0.25	40/8	8.00 + 0.00 - 0.01	7.52 ± 0.01	$31.00	$28.00
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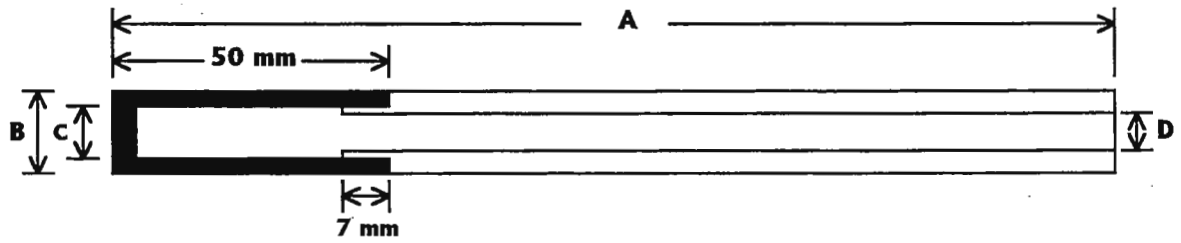

	A Length (mm)	B OD (mm)	C ID (mm)	D OD (mm)	Camber (μ)
Si-005	180	4.965 + 0 − 0.005	4.0 ± 0.1	2.5	± 0.02
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Professor B. L. Shapiro
The NMR Newsletter
966 Elsinore Court
Palo Alto, CA 94303

Sepertember 1, 1995
(received 9/18/95)

Using Spin-Echo Water Suppression in HMQC for Correlating ^{1}H-^{15}N chemical shifts in Proteins

Dear Professor Shapiro:

We have been using a Spin-Echo water suppression pulse sequence in HMQC experiments(1) for correlating ^{1}H and ^{15}N chemical shifts in proteins. This method has the advantage of preventing saturation transfer of water protons with amide protons when water suppression is achieved by presaturation. Setting up such an experiment is straightforward. This pulse sequence not only provides very good water suppression, but also gives great sensitivity. We would like to share the pulse sequence, which was written on Bruker DMX system, with our colleagues. Figures 1a and 1b show the HMQC spectra of a stromelysin-inhibitor complex with presaturation and Spin-Echo water suppression, respectively. As shown in Figures 1a and 1b, several ^{1}H-^{15}N cross peaks disappear in the presaturation HMQC spectrum due to fast exchange of these amide protons with water, yet these peaks are present in the Spin-Echo water suppression HMQC spectrum (marked with *).

As a cautionary note, the spectral width of amide protons that can be covered with this Spin-Echo pulse sequence is relatively narrow, hence peaks outside the spectral width range will not appear. For example, peaks (marked with Δ) located at the border of the amide region in Figure 1a are not observed in the Spin-Echo water suppression HMQC spectrum (Figure 1b) due to narrow coverage of the spectral width. These peaks can be recovered by moving the excitation center downfield.

1. Vladimir Sklenar and Ad Bax, J. Magn.Reson. 74, 469-479 (1987).

Sincerely Yours,

Xiaolu Zhang Yu-Chin Li Nina C. Gonnella

```
;SE-HMQC
;2D HMQC with Spin-Echo water suppression

#include <Avance.incl>
1 ze
2 d11 do:f2
3 d1 pl1
  p1 ph1
  d6
  p1 ph2
  d2 pl2:f2
  p3:f2 ph3
  d0
  p1 ph5
  d7
  p1 ph6
  d0
  p3:f2 ph4
  d13
  d2 pl12:f2
  go=2 ph31 cpd2:f2
  d11 do:f2 wr #0 if #0 id0 ip3 zd
  lo to 3 times td1
exit
ph1=0
ph2=2
ph3=0 0 0 0 2 2 2 2
ph4=0
ph5=0 1 2 3
ph6=2 3 0 1
ph31=0 2 0 2 2 0 2 0

;p1: 90° 1H pulse
;p3: 90° 15N pulse
;pl1: power level for 1H pulse
;pl2: power level for 15N pulse
;pl12: power level for 15N CPD decoupling
;d0: incremented delay, 3 usec
;d6=1/4*offset, d7=1/2*offset
;d2=1/4JH-N,
;d11=30 msec
;in0: 1/(4 * SW(X)) = (1/2) DW(X)
;nd0: 4
;ns: 8*n
;MC2: TPPI
;cpd2: decoupling sequence defined by cpdprg2
```

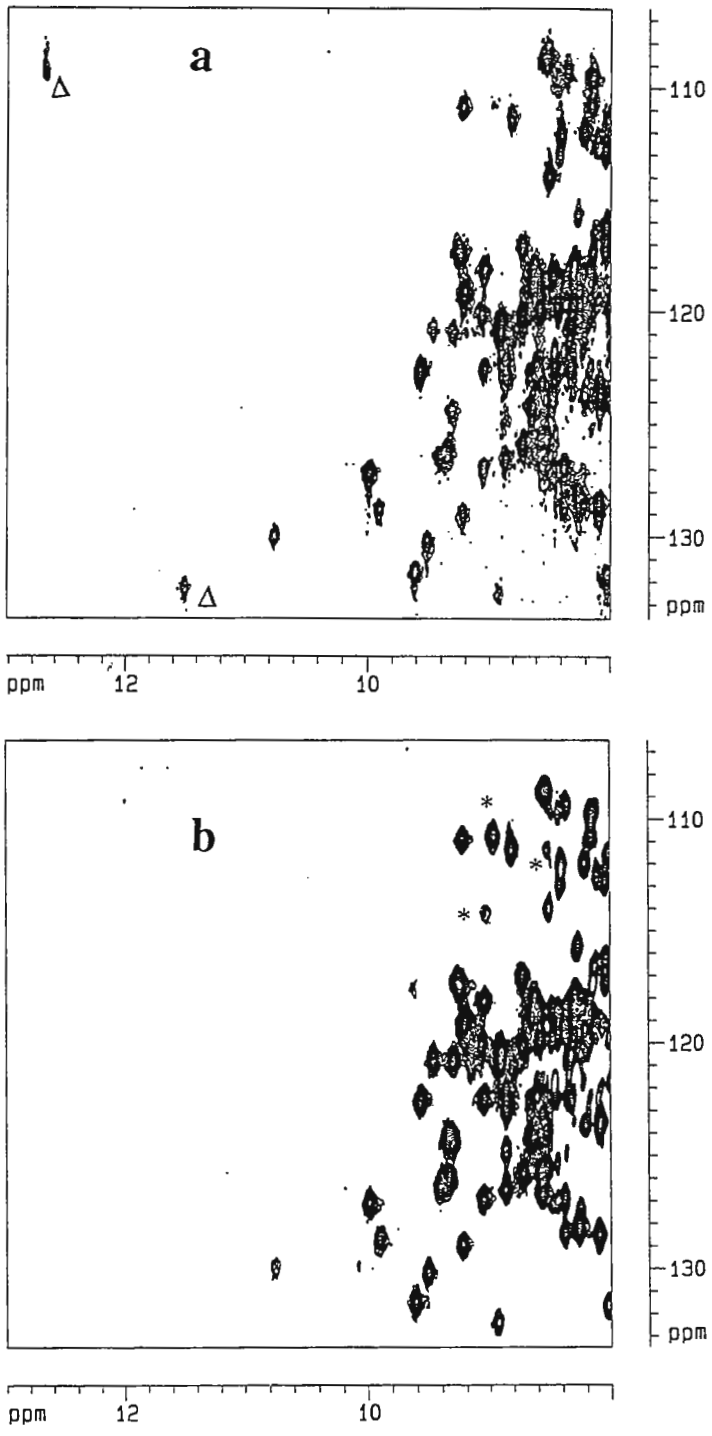

Figure 1. a) ^{1}H-^{15}N HMQC spectrum of stromelysin-inhibitor complex using presaturation for water suppression. b) ^{1}H-^{15}N HMQC spectrum of stromelysin-inhibitor complex with Spin-Echo water suppression. The excitation center was 3.2 ppm from the water peak.

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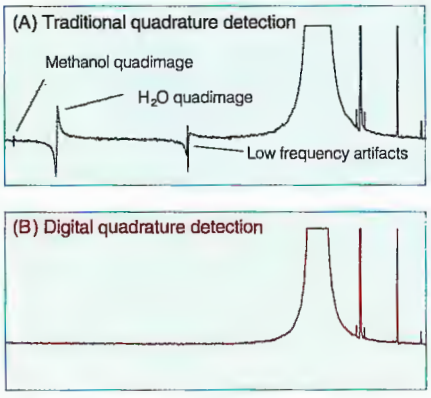

Bruker standard dynamic range sample, obtained with (B) and without (A) DQD

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Bruker Instruments, Inc.

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Billerica, MA 01821
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Fax: (508) 667-3954

August 28, 1995
(received 8/31/95)

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

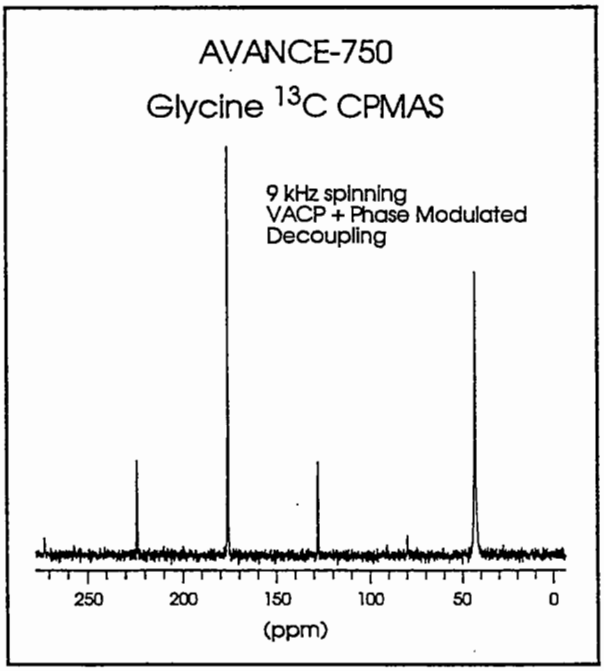
Solid State CPMAS results at 750 MHz

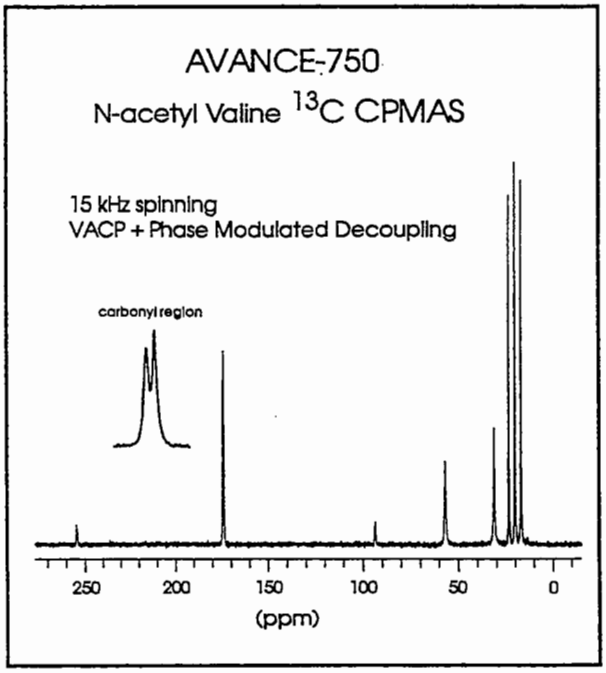
Dear Barry:

750 MHz NMR of biological molecules in solution has become almost commonplace, but CPMAS data above 500 MHz are still rare. This is probably due in large part to the difficulties associated with designing a CPMAS probe that will operate efficiently at high frequencies.

In our experience, it is nearly impossible to extend the traditional doubly-tuned solenoid design to frequencies above 500 MHz and still obtain acceptable performance. Therefore, we have been working on a novel approach proposed by Dr. David Cory that uses a resonator instead of an rf coil to achieve strong ^{1}H rf fields with low input power.

Below are some preliminary results obtained with our new probe design at the University of Pennsylvania in cooperation with Dr. Stan Opella. In all cases, the sample was packed in a 4mm rotor, and the proton decoupling field strength was approximately 70 kHz, with less than 100 W of power applied. The ^{13}C CPMAS spectrum of glycine was obtained



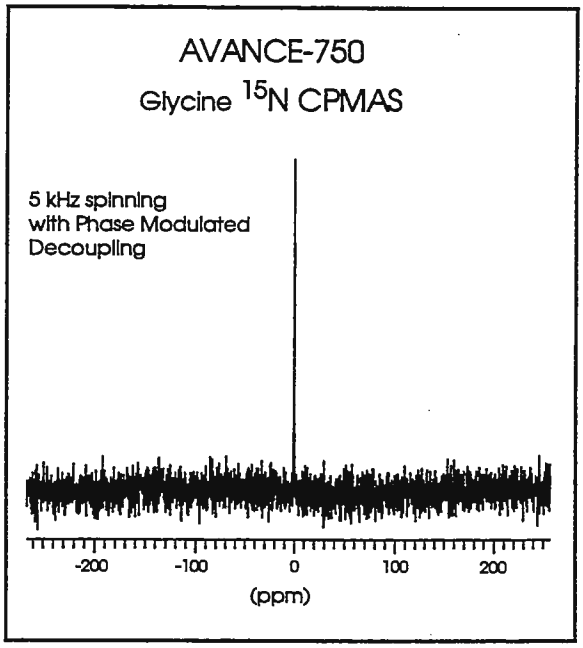


in four scans at 9 kHz spinning speed. To further improve the spectrum, Variable Amplitude Cross Polarization (VACP)[1] was used to enhance the signal intensity, and a phase modulated ^{1}H decoupling scheme[2] was applied during acquisition.

The ^{13}C CPMAS spectrum of N-acetyl valine shown above was obtained in 64 scans at a spinning speed of 15 kHz using the same combination of VACP and phase modulated decoupling. The down field peak in the carbonyl region and the peak near 60 ppm are both strongly coupled to ^{14}N, and are significantly narrowed in comparison to spectra of the same compound obtained at lower fields.

The natural abundance ^{15}N spectrum of glycine at the right was obtained in four scans, and shows an approximately four-fold improvement in sensitivity compared to similar results obtained at 400 MHz.

Although our initial tests have been at 750 MHz, we expect this new probe design to also provide superior results at ^{1}H frequencies of 500 MHz and 600 MHz.



Sincerely,

Douglas P. Burum

David Cory

Martine Ziliox

Martin Rindlisbacher

Werner Maas

Joel Lewandowski

Frank Laukien

[1] O. B. Peersen, X. Wu, I. Kustanovich, and S. O. Smith, *J. Magn. Reson. A*, 1993, **104**, 334

[2] A. E. Bennett, C. M. Rienstra, M. Auger, K. V. Lakshmi, and R. G. Griffin, *J. Chem. Phys* in press.

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0.1% 0.01%	Ethylbenzene TMS in Chloroform-d (min. 99.8%D)	^1H	Sensitivity
0.2 mg/ml 0.1% 1%	Gadolinium Chloride DSS (Sodium 2,2-dimethyl-2-silapentane-5-sulphonate) Water in Deuterium Oxide	^1H	Homogeneity
1%	Chloroform in Acetone-d_6 (min. 99.9%D)	^1H	Line Shape
5%	Chloroform in Acetone-d_6 (min. 99.9%D)	^1H	Line Shape
40%	p-Dioxane in Benzene-d_6 (min. 99.9%D)	^{13}C	Sensitivity/ Resolution
0.05%	Trifluorotoluene in Benzene-d_6 (min. 99.6%D)	^{19}F	Sensitivity
0.0485 Molar	Triphenylphosphate in Chloroform-d (min. 99.8%)	^{31}P	Sensitivity

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ROSKILDE UNIVERSITY

Associate professor Poul Erik Hansen, Department of Life Sciences and Chemistry

e-mail poulerik@mmf.ruc.dk

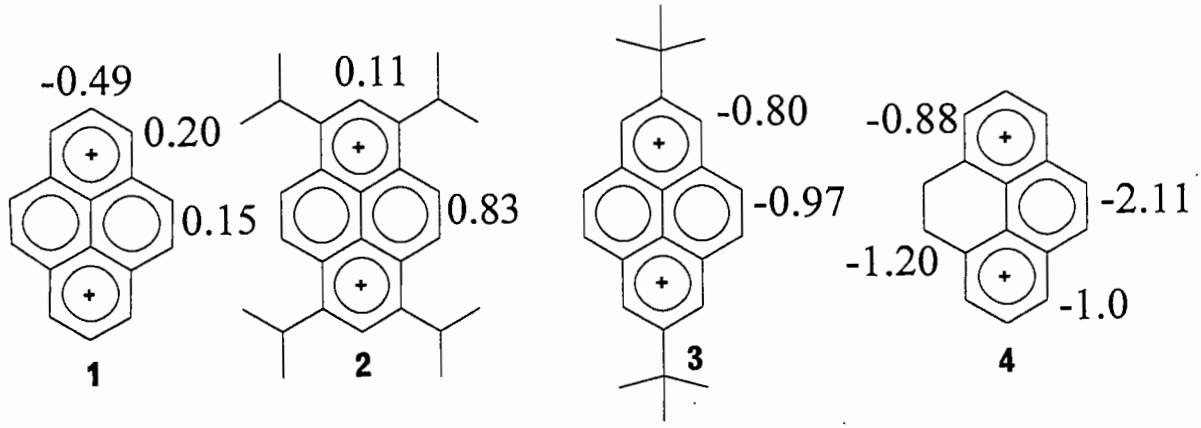
Dr. B. L. Shapiro
The NMR Newsletter
966 Elsinore Court
Palo alto
CA 94303, USA

DATE
August 22 1995
(received 8/29/95)

Dikations. Charge shifts.

Dear Dr. Shapiro

We (Ken K.Laali and undersigned) have for some time investigated the ^{1}H and ^{13}C spectra of carbonium ions of PAH and substituted PAH either as protonated or as dikation species (e.g. pyrene and substituted pyrenes (1 - 3)).



Early studies showed[1] that the total charge induced shift in dikations ranged from 2.3 ppm per electron charge for anthracenes to as much as 14 ppm for perylenes. For the pyrenes we observed apparently substituent (pattern) dependent high field shifts[2] by formation of dikations as seen in the figure. However, the ^{13}C spectra showed more than the expected 160 ppm/e low field shift. Recently, we have investigated **4**, formally a 4,5-dialkyl-substituted phenanthrene. It showed marked high field shifts of the aromatic protons but also of the CH_2 protons of the bridges. In this case no low field shift was found in the ^{13}C spectrum.

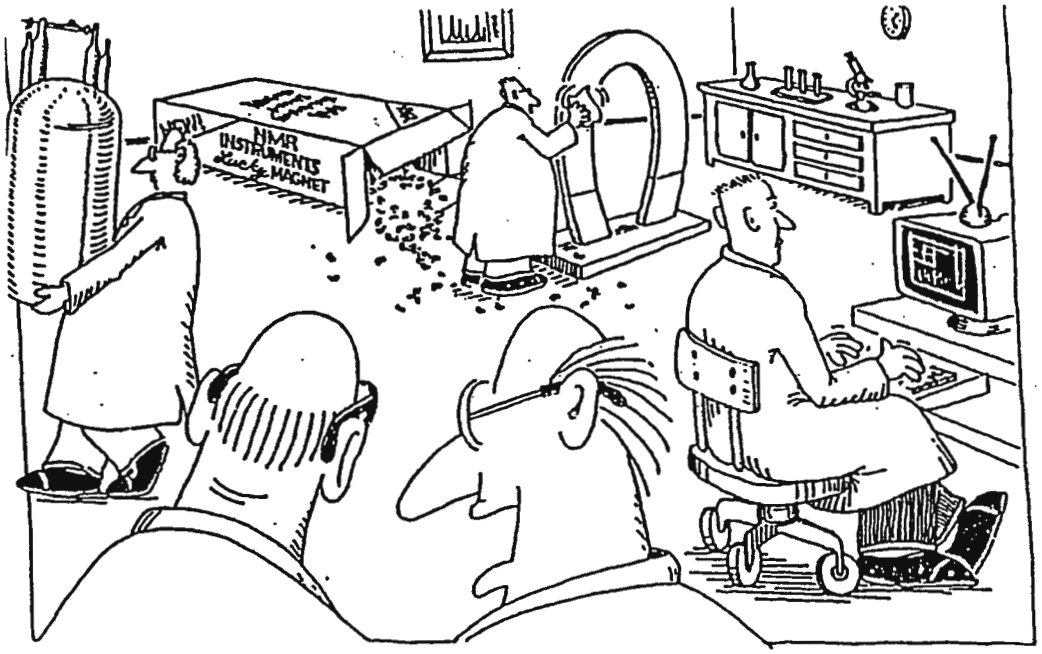
Another interesting feature of the ^{1}H NMR spectra of the carbonium ions is the dependence on radical formation. That radicals change the spectra is not new, but in this

case the formations is "in situ" and is not necessarily evident as linebroadenings are not overwhelming (and the resonances are in most cases broad anyway). This calls for caution as such spectra are less useful both for identification purposes, but also in calculation of the effects of charge.

Yours sincerely

Poul Erik Hansen

[1] D.M.Brouwer and J.A. van Doorn, Rec.Trav.Chim.Pays Bas. 1972 ,91 1110.
[2] K.K.Laali, P.E.Hansen, E.Gelerinter and J.J.Houser, J.Org.Chem. 1993, 58, 4088.



"...I just expected it to be, I don't know, more complex, I guess."

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DLM-41	Acetic Acid-d_4 "100%" (D,99.96%)	4.76	4.3-5.2	10x0.75 ml 5 ml	$63 $35
DLM-710	Ammonium Deuteroxide (D_5,99%) (26% solution in D_2O)	9.25	8.8-9.7	50 g 100 g (of solution)	$100 $177
DLM-1273	Ammonium Chloride (D_4,98%)	9.25	8.8-9.7	5 g 10 g	$65 $85
DLM-1099	Ammonium Bromide (D_4,98%)	9.25	8.8-9.7	5 g	$80
DLM-407	Betaine (D_{11},98%) (Trimethylglycine)	1.83	1.4-2.3	1 g	$515
DLM-286	Formic Acid (D_2,98%) (contains </= 5% D_2O)	3.77	3.3-4.2	5 g	$125
DLM-280	Glycine (D_5,98%)	2.34 9.6	1.9-2.8 9.1-10.1	5 g	$130
DLM-3786	N-2-hydroxyethylpiperazine -N'-2-ethanesulfonic Acid (D_{18}, 98%) (HEPES)	7.55	6.8-8.2	1 g	$600

* pK_a values are for unlabeled compounds. Deuterated compounds may have slightly different pK_a values. Ranges are approximately +/- 0.4-0.5 from pK_a value.

Buffers

Catalog#	Compound	pK_a*(20°)	Range	Size	Price
DLM-3033	Imidazole (D_4,98%)	7.05	6.6-7.5	1 g 5 g	$200 $650
DLM-1361	Sodium Formate (D,99%)	3.77	3.3-4.2	5 g	$145
DLM-831	Succinic Acid (D_6,98%)	4.19 5.48	3.8-4.6 5.0-5.9	5 g	$225
DLM-1842	Tricine (D_8,98%)	8.15	7.6-8.8	0.1 g	$290
DLM-1814	Tris(hydroxymethyl)methylamine (D_{11},98%) (TRIS)	8.3	7.8-8.8	5 g	$495
DLM-3593	Tris(hydroxymethyl)methylamine (D_{11},98%) (TRIS) (1M solution in D_2O)	8.3	7.8-8.8	10 ml (of solution)	$190

* pK_a values are for unlabeled compounds. Deuterated compounds may have slightly different pK_a values. Ranges are approximately +/- 0.4-0.5 from pK_a value.

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DLM-197	Sodium Dodecyl Sulfate (D_{25},98%) (SDS)	1 g	$390
	Protein Stabilizers:		
DLM-2622	DL-1,4-Dithiothreitol (D_{10},98%) (DTT)	0.5 g	$685
DLM-2713	2-Mercaptoethanol (D_6,98%)	0.5 g	$600
	Chelating Agent:		
DLM-414	Ethylenediaminetetraacetic Acid (D_{12},98%) (EDTA)	0.1 g 0.5 g 1 g	$115 $450 $690

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Cornell University

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Department of Chemistry
Baker Laboratory
Ithaca, NY 14853-1301

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(607) 255-4767

September 15, 1995
(received 9/18/95)

Using Noise to Your Advantage ...

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

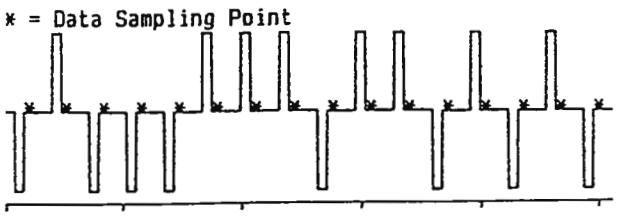
Dear Barry:

Please don't send the dreaded ultimatum! We are trying to comply–it's entirely my fault that your reminder disappeared onto my desk and has only now resurfaced!

In recent months we have been exploring the application of noise spectroscopy, of the kind popularized by a long line of NMR researchers over the past 25 or 30 years (Ernst, Kaiser, Ziessow, Blümich, Hoatson, etc.) to new problems in solid state NMR–and have rediscovered the advantages of an irradiation scheme which uses low power and works in the linear excitation limit of the rf field–i.e. the excitation profile is approximately given by the inverse of the pulse length, and not its amplitude. In brief, we have shown that one can observe spectra of quadrupolar nuclear spin species without distortion in lineshapes and/or intensities independent of the quadrupole coupling constants, and that low-temperature absorption lineshapes can be observed with good lineshape fidelity and over very broad bandwidths with low-power rf capacitors with little fear of arcing in the He vapor atmospheres often required.

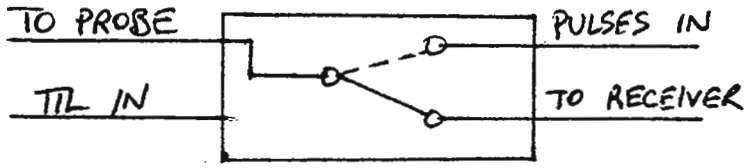
In this note, we'd like to provide some of the experimental details which seemed out of place in a journal article–particularly given that while the technique remains relatively unfamiliar, its implementation today is not that different from that of years ago. The design requirements include the continuous application of weak pulses (typically $\sim 1°$, with rf pulses at the 0-10 dBm level) alternating with delays, until sufficient signal has been accumulated. The pulse separation Δt determines the Nyquist frequency for the experiment, as data is accumulated one point at a time after each of the pulses.

Pulse phases are chosen so as to provide a uniform excitation bandwidth over as broad a frequency range as possible. Poorly chosen sequences excite in only very small bandwidths–an example of which is provided by the always popular DANTE sequence of weak pulses of constant phase, which excites only a narrow bandwidth exactly on resonance (and at multiples of the repeat frequency of the cycle). For spectroscopic applications, the uniform excitation over broad spectral widths are generally preferred. Good choices of phase sequences which require only 0-180° phase shifts can be designed by looking to the electrical engineers, who long ago discovered that reasonable approximations to broadband white noise might be found in appropriately designed pseudo-random digital sequences. White noise is characterized by nearly-uniform amplitudes, but random phases in the frequency spectrum of the irradiation sequence. (Two references where these sequences are described are D. Lancaster's *The TTL Cookbook*, chapter 7 (Howard Sams & Co.) or Horowitz and Hill's *The Art of Electronics*, (2nd edition) sections 9.32-9.35 (Cambridge University Press)). These sequences seem to perform perfectly adequately in our hands, and generate uniform excitation bandwidths to frequencies of about $0.25/\tau$, where τ is the pulse length.) An example of an experiment using noise excitation is illustrated below.



The interface between the receive and transmit sections of the spectrometer can be substantially simplified because our pulses are so attenuated in strength. Where one generally worries about duplexing so that the preamp is protected from the pulses, we have replaced the high-power amplifier plus various diode boxes with a

simple SPDT switch, which alternately connects the probe to either the low-power section of our spectrometer (for pulses) or the receiver (for signal detection). As there is not much power in our pulse, standard GaAs switches have both excellent rise/fall times and high isolation. We have used Anzac Model SW-229 switches, which come with an integrated TTL driver; other competing models are likely to be perfectly adequate for the job.



The final change comes from the design of the probe. Many of us are working in very confined spaces, and the need to fit beefier capacitors into narrow spaces at high fields makes probe design awkward. Much of our work involves low temperatures, which adds to the complications. Particularly when He vapor is used as the coolant, as arcing becomes an even more serious problem. Using noise excitation sequences, we have effectively eliminated all the arcing by operating at powers so reduced that even the cheapest variable capacitors we can find are adequate, and we have found that we can cover without distortion reasonably broad bandwidth spectra. An example of the sorts of spectra we can routinely observe is shown in the accompanying figures. The advantages we have found? Better spectral fidelity, less need for more and more rf power, and the experiment is less likely to fry any of our sensitive components.

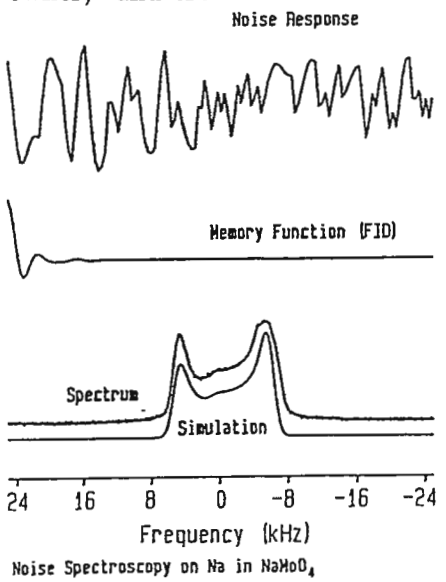
Disadvantages? I'd like to say that there are none, or at least they are relatively minor. First–the data as it arrives off the spectrometer is not your traditional FID. The latter can be recovered by cross-correlation of the pulse sequence with the data, or, it would seem, by multiplication of the FT of the data with the complex conjugate of the FT of the input record. Second–the spectral bandwidth is limited because data collection alternates with pulses, so that the dead time after a (very weak) pulse limits the data collection rate. Third–it is hard to imagine playing the sorts of coherent averaging tricks which are now routine in many applications. Fourth–the pulses really do need to come out of your spectrometer "continuously" for as long as it takes to acquire a good spectrum, as gaps inserted in the averaging to allow the pulse programmer to reset and data to upload reduce the amount of signal acquired per unit time, and introduce systematic noise into the data set. Phase cycling (so as to reduce quadrature artifacts) has the same sort of problem associated with it; we have chosen to interrupt data accumulation for one or two trips through the pulse sequence when shifting the irradiation and receiver phases through the standard sets of 4 phases. Some examples of our spectra are shown below, using rf field strengths of about 100 Hz.

We've had no problem implementing this work on our home-built spectrometer (powered by a TecMag digital front and back end) but I make no claims about the suitability of any other console.

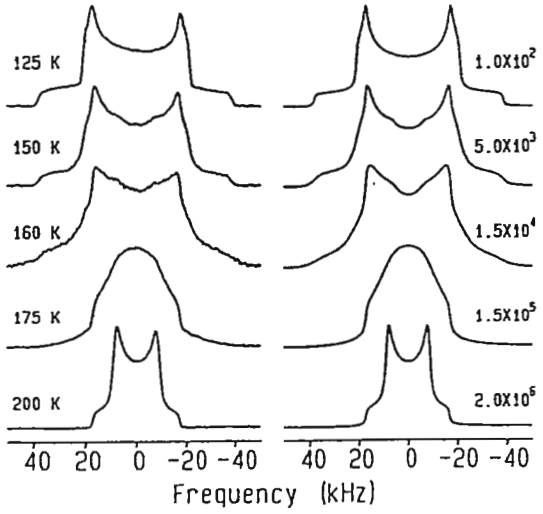
The new director of our NMR facility is Dr. Cathy Lester. Please change our subscription so that she is the "owner," and credit this submission to her account.

Sincerely,

David B. Zax



Noise Spectroscopy on Na in $NaHoO_4$



^{2}D NMR Spectra of Hexamethylbenzene (d-18)
Left: Noise Absorption Spectra
Right: Simulations: Jump Rates as Indicated

Tuning Stations

Model 405NV and 505NV R.F. Sweepers

These units replace the conventional sweeper and oscilloscope tuning stations commonly used to adjust NMR probes and MRI coils.

The oscilloscope has been replaced by an integral liquid crystal display which is unaffected by magnetic fields. The display, bridge, and RF generator have been incorporated into a compact hand held design, with microprocessor control.

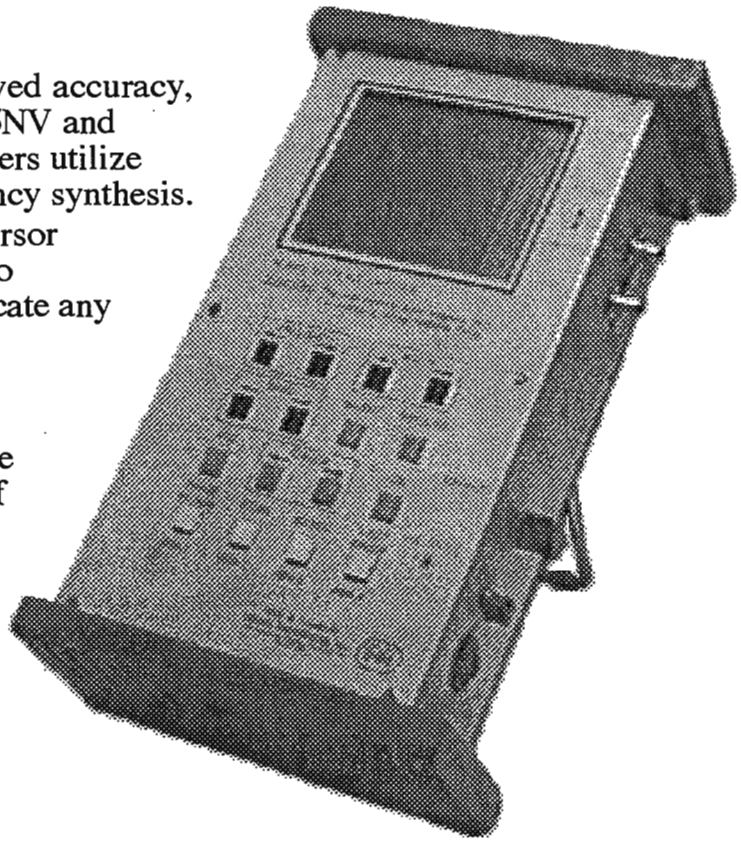
An internal rechargeable battery powers the units for three hours of continuous use. Each unit includes an AC adaptor for bench operation and recharging.

For improved accuracy, the model 405NV and 505NV sweepers utilize digital frequency synthesis. The screen cursor may be used to precisely indicate any frequency, or to center the sweep, thus eliminating the tedious task of counting markers.

Since these sweepers will operate in the fringe field of superconducting magnets, it is now possible to tune MRI coils in situ.

Nonvolatile digital storage of setup parameters and displays gives these units additional flexibility.

To further extend the utility of these units they may be used to test two port devices such as filters, or to characterize the RF field homogeneity of probes and coils.



Features

- ability to operate in the fringe field of superconducting magnets
- portability
- liquid crystal display, with on-screen parameters and cursor, unaffected by magnetic fields
- integral tuning bridge and detector
- digitally synthesized RF section
- microprocessor control for ease of operation
- non-volatile memory for sweeps and setup parameters

Morris Instruments Inc.

1382 McMahon Ave., Gloucester, Ontario, Canada K1T 1C3
(613) 523-7448 pmorris@cyberplus.ca

Morris Instruments Inc.

1382 McMahon Ave., Gloucester, Ontario, Canada K1T 1C3
(613) 523-7448 pmorris@cyberplus.ca

Model 405NV and 505NV R.F. Sweepers

Specifications

- Size: 12.1 in x 7.2 in x 3.5 in
- Weight: 5.5 lbs
- Battery: sealed lead acid 8V 4AH
- Operating time on a full battery charge: 3hrs minimum
- Battery charger: 12V 1Amp

User interface

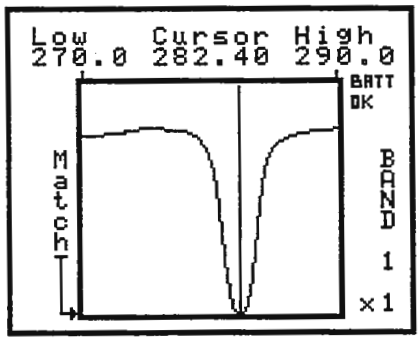
- LCD graphic display of swept frequency response (101 points) plus setup data including high and low frequencies, ADC scale, and cursor frequency
- battery condition continuously monitored and displayed on screen
- UP/DOWN keypad controls for frequency, sweep width, range, and cursor
- keypad controls for display invert, offset calibration, CW mode, ADC scale
- autorepeat on all keypad controls
- switch to set mode for single port or two port devices
- BNC connectors for RF OUTPUT (used only in two port mode), and TUNE/DETECTOR (used as detector input in two port mode or as tuning bridge connection for single port mode)
- continuous R.F. output level control (Nominal 10dB range)
- screen contrast control
- non-volatile memory for user programmable setups and digitized sweeps

R.F.

- impedance 50Ω
- maximum level: nominal 0 dBm ±3dB. The level control allows the output to be reduced by a minimum of 10dB.

Output Range (MHz)

Band	Model 405NV	Model 505NV	Resolution (kHz)
1	165.0 - 405.0	205.0 - 505.0	100.0
2	82.5 - 202.5	102.5 - 252.5	50.0
3	41.25 - 101.25	51.25 - 126.25	25.0
4	20.625 - 50.625	25.625 - 63.125	12.5
5	10.313 - 25.313	12.813 - 31.563	6.25
6	5.156 - 12.656	6.406 - 15.781	3.125
7	2.578 - 6.328	3.203 - 7.891	1.5625



probe matched to 50Ω at 282.40 MHz

Upgrades

- available upgrade from model 405NV to 505NV
- microprocessor firmware in socketed ROM permits firmware updates
- low cost NV upgrade from earlier models 405 and 505

Warranty

- units covered for one full year against defects in manufacture, or failure under normal use

Spin Imaging*)
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The Netherlands

Laboratoire de RMN, CNRS D2057**)
Université Claude Bernard LYON I–CPE
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Dr. B. L. Shapiro
The NMR Newsletter
966 Elsinore Court
Palo Alto, Ca 94303

August 14, 1995
(received 9/11/95)

Effect of a digital audio filter on the NMR signal and its evaluation

Dear Dr. Shapiro,

Thanks to progress in digital circuitry, oversampling and digital filtering with finite impulse response (FIR) filters have recently been introduced in NMR spectroscopy and tomography. This allows efficient suppression of folding of the spectrum from outside the spectral width. In addition, since FIR filters can have strictly linear phase response, phase correction of the spectrum is simplified (*1*).

The mentioned properties strongly improve the quality of the spectrum. On the other hand, the digital filter affects the shape of the FID, as shown in Figure 1. The filter transient response causes reduction and spreading of energy of the original FID signal, also toward negative virtual time. In fact, the filtered FID starts at virtual time $t=-t_g$, where t_g is the group delay time of the filter.

From the viewpoint of DFT processing all data points (including $-t_g \leq t < 0$) must be kept to obtain a spectrum that closely approximates the theoretical one (Fig. 2 a,b). A very large first-order phase correction is needed due to the delay t_g. Alternatively, one can apply a circular shift to the data such that the signal starts approximately at virtual $t=0$, and subsequently perform the usual small first order correction. Of course, any apodisation must take place prior to this circular shift. It is also to be pointed out that a simple left shift (omitting of the signal in $-t_g \leq t < 0$) of the data introduces a baseline distortion of the whole spectrum (Fig. 2 c).

Using a parametric method like HSVD, we have established that the effect of the filter on the time domain model fit parameters is negligible, provided one starts at virtual time $t \geq 0$ (in contrast to DFT!). In addition, the filter introduces extraneous spectral components near ± the cut-off frequency which appeared easily identifiable.

(Supported by EU/Human Capital and Mobility/Networks.)

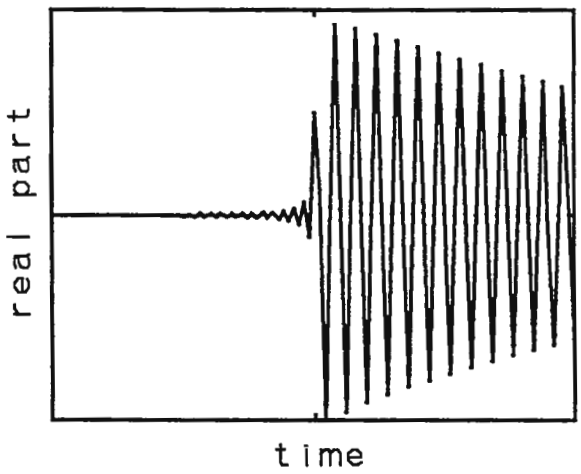

FIG. 1. The beginning of a simulated FID comprising six sinusoids with amplitude ratio *20000:1:2:4:2:1* and equal damping factors. This signal was filtered by a multistage digital decimation FIR filter and an oversampling ratio of *256:1*.

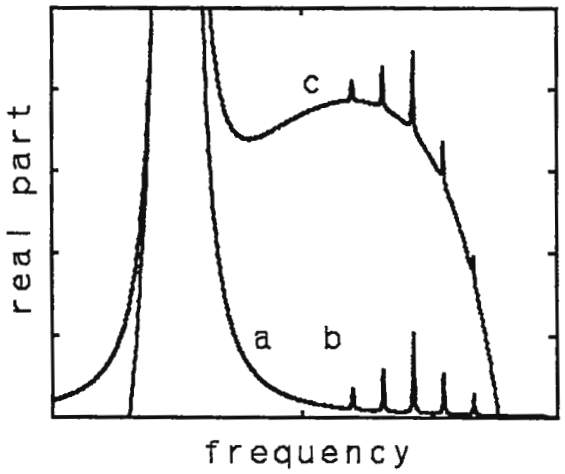

FIG. 2. a) Theoretical spectrum of the simulated signal. b) DFT of the recorded digital FIR filtered signal. c) DFT of the same signal with improper omission of the initial part in $-t_g \leq t < 0$.

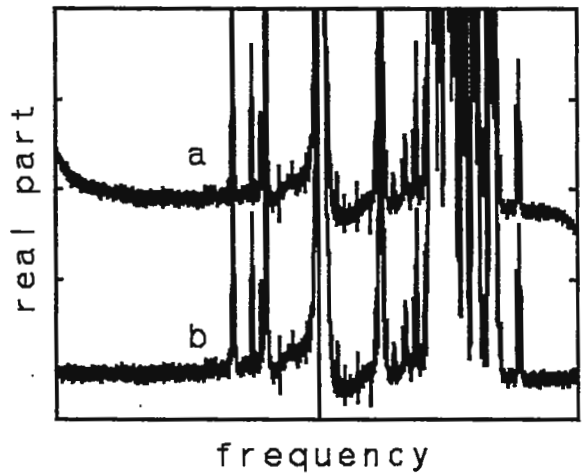

FIG. 3. Experimental *500 MHz* spectrum of melezitose dihydrate obtained with a Bruker DMX 500. a) DFT with improper omission of the initial part $-t_g \leq t < 0$ of the FID. b) DFT with inclusion of the initial part of the FID.

References

1. J. Halámek, V. Vondra, and M. Kasal, "The Elimination of Baseline Distortions Induced by Audio Filters", *J. Magn. Reson.*, A **110**, 194, (1994).

Please credit this to the account of A. Briguet.

Sincerely,

B. Fenet[**)] D. Graveron–Demilly[**)] D. van Ormondt[*)] V. Vondra[*)]

(on leave from Institute of Scientific Instruments, Czech Academy of Sciences, Královopolská 147, 612 64 Brno, Czech Republic)

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Internet: jerry@nmr.mgh.harvard.edu

September 16, 1995

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

Dear Barry:

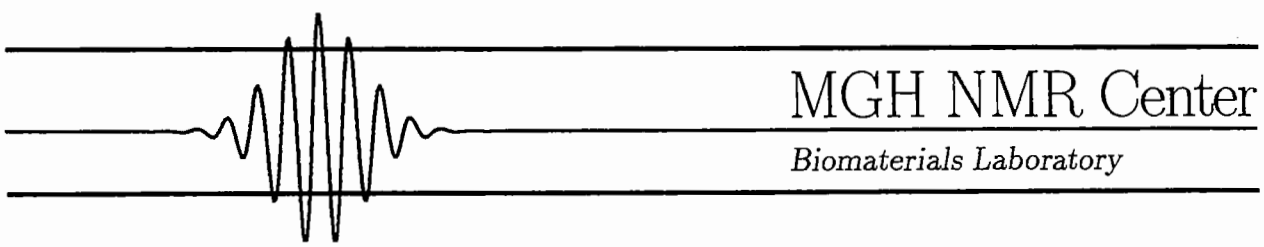
Please insert the following announcement into the next NMR Newsletter:

Postdoctoral Position

In Vivo Solid State Imaging and Spectroscopy of Bone Mineral

We have an immediate opening in the Biomaterials Laboratory of the Massachusetts General Hospital NMR Center for an NIH-funded project involving *in vivo* solid state NMR of bone mineral. Postdoctoral fellows will have Harvard Medical School academic appointments. The MGH NMR Center encompasses over 50 researchers, with backgrounds in chemistry, physics, biology, medicine and engineering, including graduate students from Harvard and MIT. It is the focus of many cutting edge developments in clinical and basic science research. Please reply to Prof. Jerome L. Ackerman, Biomaterials Laboratory NMR Center, Room 2301, Massachusetts General Hospital, 149 13th Street, Charlestown, MA 02129. Phone: 617-726-3083, fax: -7422, email: jerry@nmr.mgh.harvard.edu.

Best regards,



Monsanto

ANALYTICAL SCIENCES CENTER

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September 21, 1995
(received 9/22/95)

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

NMR Symposium at the 38th Rocky Mountain Conference on Analytical Chemistry

Dear Barry:

The NMR Symposium at the 38th Rocky Mountain Conference on Analytical Chemistry will take place Monday - Thursday, July 22 - 25, 1996 at the Hyatt Regency Denver in downtown Denver, Colorado. The program will consist of invited lectures and contributed papers for oral and poster presentations on NMR research in areas such as solid-state NMR, spin dynamics, new techniques, macromolecules, solid-state chemistry, calculations/simulations, environmental science, and geochemistry.

Each year, we dedicate a half-day session of the NMR Symposium to the memory of Professor Robert Vaughan. The 1996 Vaughan Memorial lecturer will be Professor Gary Maciel, Department of Chemistry, Colorado State University.

The NMR Symposium is organized by Joel Garbow - chair, James Yesinowski - co-chair, Lucio Frydman, Bernie Gerstein, Clare Grey, Jeffrey Reimer, Hans Thomann, and Robert Wind.

For further information, I can be contacted at: Monsanto Corporate Research, Monsanto Company, 700 Chesterfield Parkway North, St. Louis MO 63198. Phone: 314-537-6004; Fax: 314-537-6806; Internet: jrgarb@snc.monsanto.com.

Best Regards,

Joel R. Garbow

THE NMR NEWSLETTER

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The NMR Newsletter
966 Elsinore Court
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Fax. (415) 493-1348
E-mail:71441.600@compuserve.com

Policies and Practical Considerations

(Slightly revised September 1995)

The NMR Newsletter (formerly the **TAMU NMR Newsletter**, the **IIT NMR Newsletter**, and originally, the **Mellon Institute NMR Newsletter**), now in its thirty-eighth year of consecutive monthly publication, continues under the same general policies as in the past. All communication with the Newsletter should be sent to the address above.

1. **Policy**:

The NMR Newsletter is a means for the rapid exchange of information among active workers in the field of NMR spectroscopy, as defined broadly, including imaging. As such, the Newsletter serves its purpose best if the participants impart whatever they feel will interest their colleagues, and inquire about whatever matters concern them. Technical contributions should always contain a significant amount of information that has not already been published or that will appear in the formal literature within a few weeks of the appearance in the Newsletter.

Since the subscriber/participant clearly is the best judge of what he or she considers interesting, our first statement of policy is "We print anything." (This is followed by the reservation, "that won't land us in jail or bankruptcy court.") Virtually no editorial functions are performed, although on rare occasions there is the need to classify a contribution as 'not for credit'. I trust that the reasons for this policy are obvious. The Newsletter is not, and will not become, a journal. We merely reproduce and disseminate exactly what is submitted. Foreign participants should not feel obliged to render their contributions in English.

2. **Public Quotation and Referencing:**

Public quotation of Newsletter contents in print or in a formal talk at a meeting, etc., is expressly forbidden, except as follows. Reference to The NMR Newsletter by its present or previous names in the scientific literature is *never* permissible. *In order to quote or use material from the Newsletter, it is necessary, in each individual case, to obtain the prior permission of the responsible author and then to refer to the material quoted as a "Private Communication".* If your copy of the Newsletter is shared with other readers, it is your obligation as the actual recipient of the Newsletter to see that these other readers of your copy are acquainted with, and abide by, these statements of policy.

3. **Participation is the prime requisite for receiving the Newsletter: In order to receive the Newsletter, you must make at least occasional technical contributions to its contents.**

We feel that we have to be quite rigorous in this regard, and the following schedule is in effect: Eight months after your last technical contribution, you will receive a "Reminder" notice. If no technical contribution is then forthcoming, ten months after your previous contribution you will receive an "Ultimatum" notice, and then the next issue will be your last, absent a technical contribution. Subscription fees are not refunded in such cases. If you are dropped from the mailing list, you can be reinstated by submitting a contribution, and you will receive back issues (as available) and forthcoming issues at the rate of nine per contribution.

Frequent contributions are encouraged, but no advance credit can be obtained for them. In cases of joint authorship, only one contributor may be credited. Please indicate to whose account credit should be given. Please note that meeting announcements, as well as "Position Available," "Equipment Wanted" (or "For Sale"), etc., notices are very welcome, but only on a not-for-credit basis, *i.e.*, such items do not substitute for a *bona fide* technical contribution. Similar considerations must occasionally be applied to a few (quasi-)technical items.

4. **Finances**: The Newsletter is wholly self-supporting, and its funding depends on Advertising, Sponsorships, and individual Subscriptions. The **Subscription** fee for the October 1995 - September 1996 year is US$190, with a 50% academic or personal subscription discount. Subscriptions are available for a minimum of the twelve monthly issues which end with a September issue. However, a subscription can be initiated at any time, with the price for more than twelve issues being prorated.

Continued

Corporations are also invited to join the list of **Sponsors** of the Newsletter. Sponsors' names appear in each month's Newsletter, and copies of the Newsletter are provided to all Sponsors. The continuation of the Newsletter depends significantly on the generosity of our Sponsors, most of whom have been loyal supporters of this publication for many years. We will be happy to provide further details to anyone interested.

Another major, indeed most essential, source of funds for the Newsletter is **Advertising**. We earnestly encourage present and potential participants of the Newsletter to seek advertising from their companies. Our rates are very modest. Please inquire for details.

5. **Practical Considerations**:

a) All technical contributions to the Newsletter will be included in the next issue if received on or before the published deadline dates.

b) Please provide short titles of all topics of your contributions, so as to ensure accuracy in the Table of Contents.

c) Contributions should be on the *minimum* (NOTE!!) number of 8.5 x 11" (21 x 27.5 cm) pages, printed on one side only. Contributions may not exceed three pages without prior approval. Each page must have margins of at least 0.5" (1.3cm) on all four sides. Black ink for typing, drawings, etc., is essential. All drawings, figures, etc., should be mounted *in place* on the 8.5 x 11" pages. We are not equipped to handle pieces of paper larger than 8.5 x 11" (21 x 27.5 cm).

Please do not fold, clip, or staple your pages. Protect the condition of your letters from the ravages of the mails by enclosing what you send in a cardboard or plastic folder, etc.

Foreign subscribers are reminded that regardless of the standard paper length you use, *all material* - letterhead, text, figures, addresses printed at the page bottom, *everything - must not exceed 10" (ca. 25.3 cm) from top to bottom.*

Significant savings of Newsletter pages and total space can be made by exercising close control over the formatting and type sizes of the contributions. **Please consider the following:**

i) Try using a smaller type font. The body of this page is printed in 10 point type, which I believe is adequate for most purposes. Even 11 or 12 point type is acceptable if the particular font is not too large. Those who are computerized can also employ non-integral spacing of lines so that sub- and superscripts don't collide with lines below and above. Type smaller than 7 point should not be used.

ii) **PLEASE** avoid excessive margins. *Instruct your secretaries to avoid normal correspondence esthetics or practices, however time-honored or 'standard'!* This page has margins on both sides of 0.6" (*ca.* 1.55 cm), which is very adequate. Margins of the same size at the top and bottom are sufficient also, but don't worry if there is more space at the end of your document, for I can often use such spaces for notices, etc.

Also, please avoid large amounts of unused space at the top of letters. Give thought to the sizes of figures, drawings, etc., and please mount these so as to use the minimum space on the page.

iii) 'Position Available', 'Equipment Wanted', and Similar Notices. These are always welcome, but not for subscription credit. Such notices will appear, however, *only* if received with these necessarily rigid constraints: a) Single spaced; b) both side margins 0.6 - 0.7" (1.5 - 1.7 cm.)- NOT WIDER; c) the minimum total height, please, but definitely no more than 4.5" (11.5 cm.).

iv) **AVOID DOUBLE SPACING LIKE THE BLACK PLAGUE ! ! !** This is extremely wasteful of space.

6. **Suggestions**: They are always welcome.

B. L. Shapiro
September 1995

*Telephone: (415) 493-5971. Please confine telephone calls to 8:00AM-10:00PM, *Pacific Coast Time.*
*Fax: (415) 493-1348 (Do not use for technical contributions which are to appear in the Newsletter, for Fax quality is not adequate.)
*E-mail: 71441.600@compuserve.com.

Table of Contents, continued

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Address all Newsletter correspondence to:

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

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

Deadline Dates

No. 446 (Nov.)	27 Oct. 1995
No. 447 (Dec.)	24 Nov. 1995
No. 448 (Jan.)	22 Dec. 1995
No. 449 (Feb.)	19 Jan.1996
No. 450 (Mar.)	23 Feb. 1996

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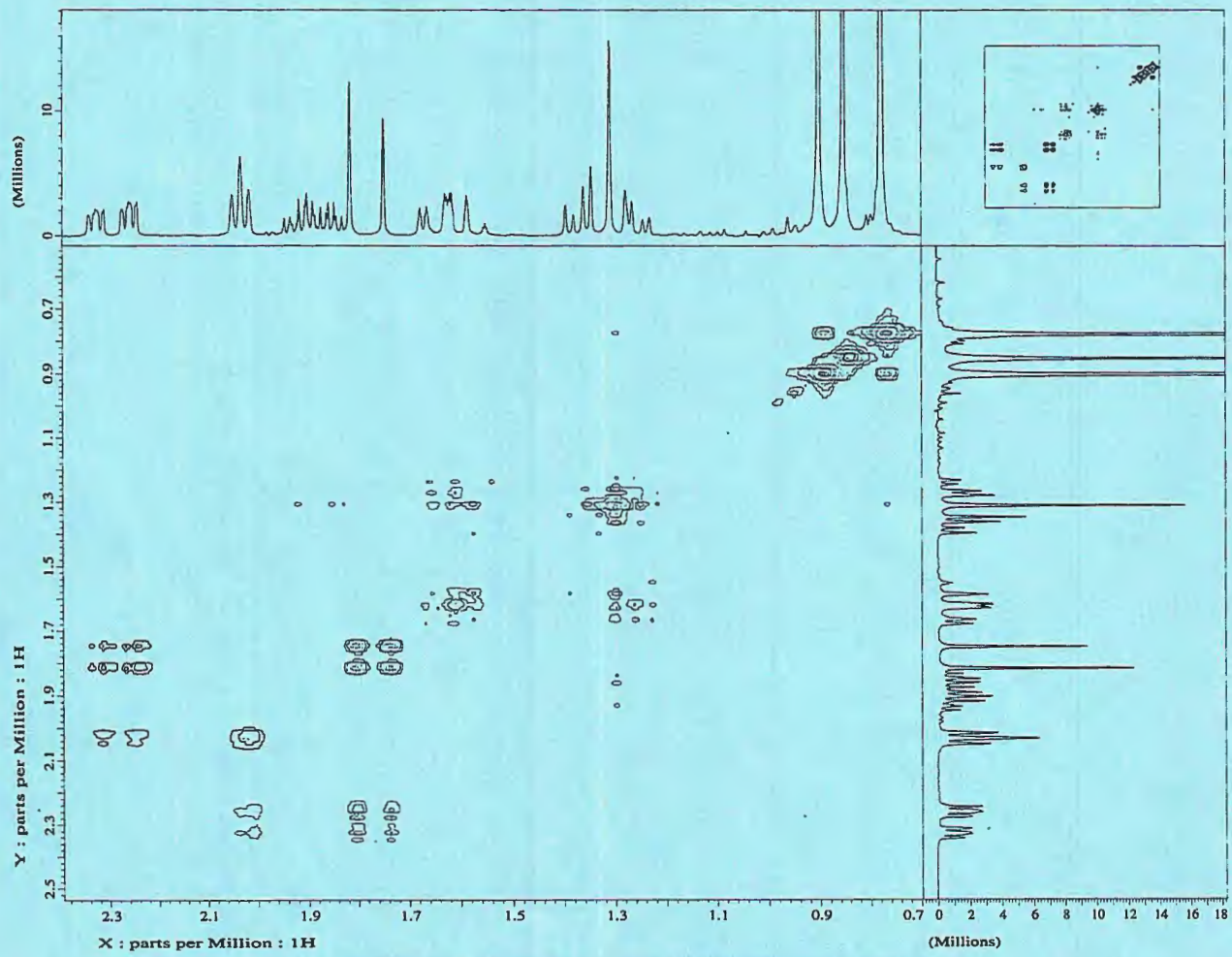

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