

THE
NMR
NEWSLETTER

No. 454
July 1996

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FORTHCOMING NMR MEETINGS

- 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.
- 42nd International Conference on Analytical Sciences and Spectroscopy**, London, Ontario, Canada, **Aug. 10-13, 1996**; Chair: M. Stillman, Dept. of Chemistry, University of Western Ontario, London, ON, Canada N6A 5B7; (519) 661-3821; Fax: (519) 661-3022; E-mail: 42info@uwo.ca.
- 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. See Newsletter 452, 59.
- Missouri Magnetic Resonance Symposium (MMRS) and FACSS Meeting**, Kansas City, MO, **Sept. 29 - Oct. 4, 1996**; Contact: (MMRS) Frank D. Blum, Dept. of Chemistry, Univ. of Missouri-Rolla, Rolla, MO 65409-0010; 573-341-4451 fblum@umr.edu. (FACSS) 198 Thomas Johnson Dr., S-2, Frederick, MD 21702-4317.
- 38th ENC (Experimental NMR Conference)**, Orlando, FL, **March 23 - 27, 1997**; Contact: ENC, 1201 Don Diego Avenue, Santa Fe, NM 87501; (505) 989-4573; Fax: (505) 989-1073.
- 4th International Conference on Magnetic Resonance Microscopy "Heidelberg Conference in Albuquerque"**, **Sept. 21-15, 1997**; Contact: E. Fukushima, The Lovelace Institutes, 2425 Ridgcrest Drive SE, Albuquerque, NM 87108-5127; (505) 262-7155; Fax: (505) 262-7043. See Newsletter 449, 37.

Additional listings of meetings, etc., are invited.

May 28, 1996
(received 6/3/96)

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

Short title: NMR on the WWW
Biomedical Magnetic Resonance Laboratory
University of Illinois
1307 West Park Street
Urbana, IL 61801

Dear Barry:

How would you like to be in the lab 8766.15 hours during each sidereal year? No? How about your students? And what if "in the lab" could include sleeping, overlooking a Hawaiian beach, or reclining in your own living room with your favorite beverage, music and companion (human, canine, or other)?

We have recently (starting in January of this year) developed a scheme for operating one of our NMR imaging spectrometers, a Surrey Medical Imaging Systems/Magnex horizontal bore 4.0T/31cm instrument, over the World Wide Web, using a standard Net browser on a desk-top or notebook computer. Essentially all of the variables that one might adjust when at the physical console can be changed from the Web interface, which makes the whole process so simple and intuitive that even a child (perhaps only a child) can do it well. In a multi-institutional K-12 twenty-one day experiment with a developing chicken egg last month, kindergarten kids tended to make the best images.

We have been planning research applications in addition to organizing educational outreach programs. NIH administrators have operated our machine from their office computers in Bethesda, and one or the other of us have done so from a hotel room overlooking Hanelei Bay on the north coast of Kauai, from a hotel ballroom in Seoul, Korea, and from nearer and more prosaic locations. Collaborative and service activities can be enhanced by the availability of this mode of operation, which will be implemented on our other NMR/MRI instruments and eventually on many others in laboratories elsewhere, bringing into existence the "World Wide Lab". Instead of loading up this Newsletter with all of the details, we recommend to you the following Web site: (<http://bmrl.med.uiuc.edu:8080>) and those linked to it.

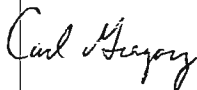
Sincerely,



Paul C. Lauterbur



Joan Dawson



Carl Gregory

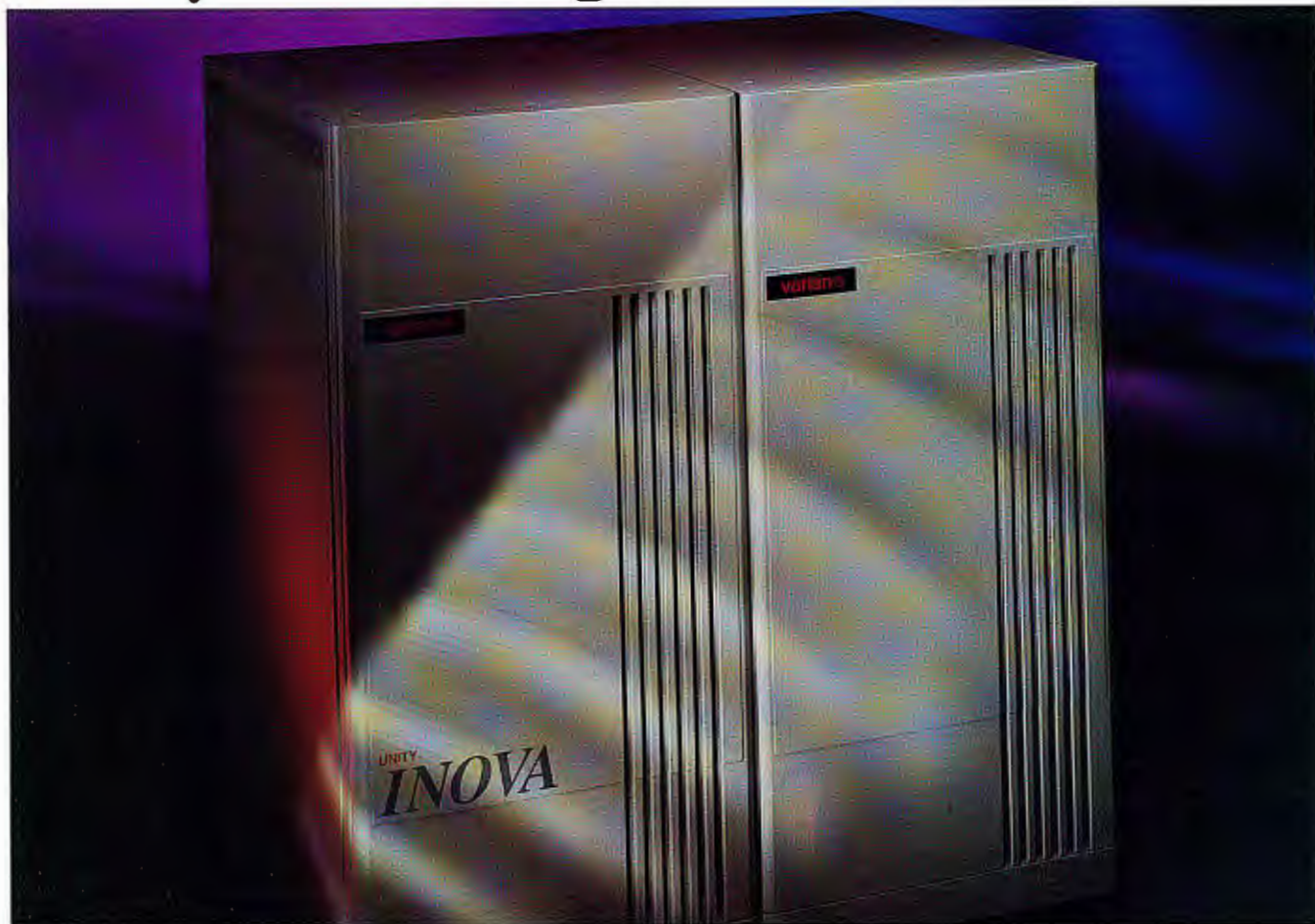


Doug Morris



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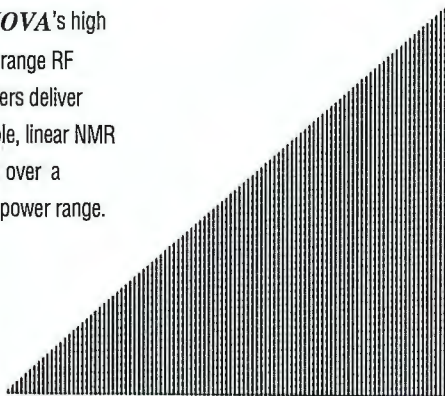
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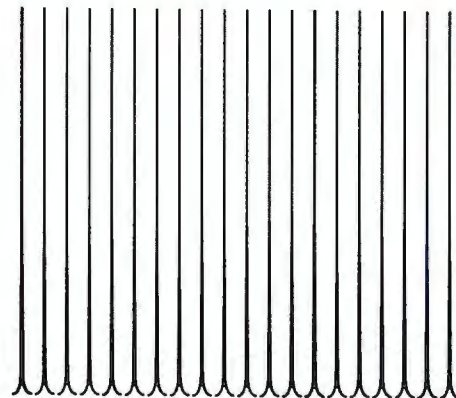
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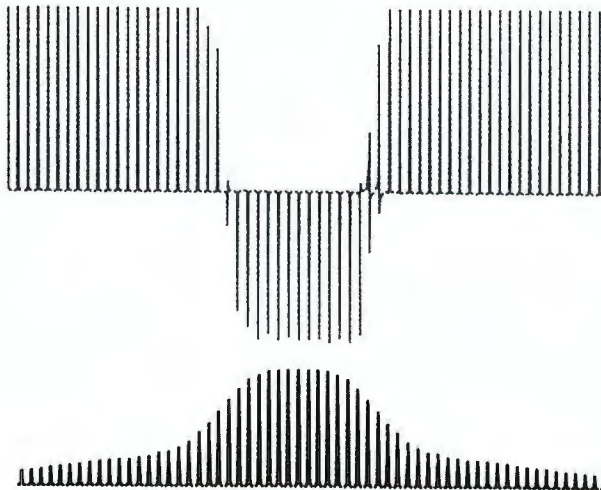
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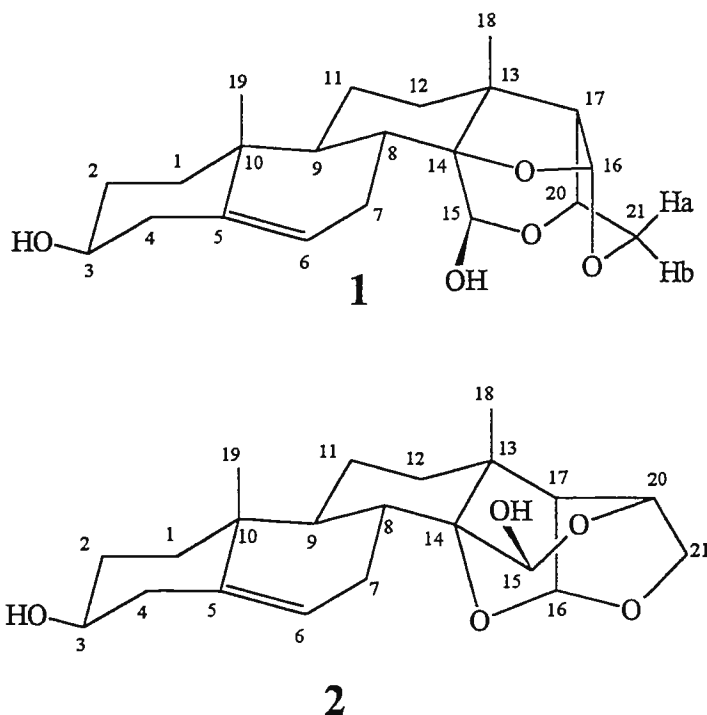
19th June 1996

(received 6/24/96)

Snake-Bite Medicine

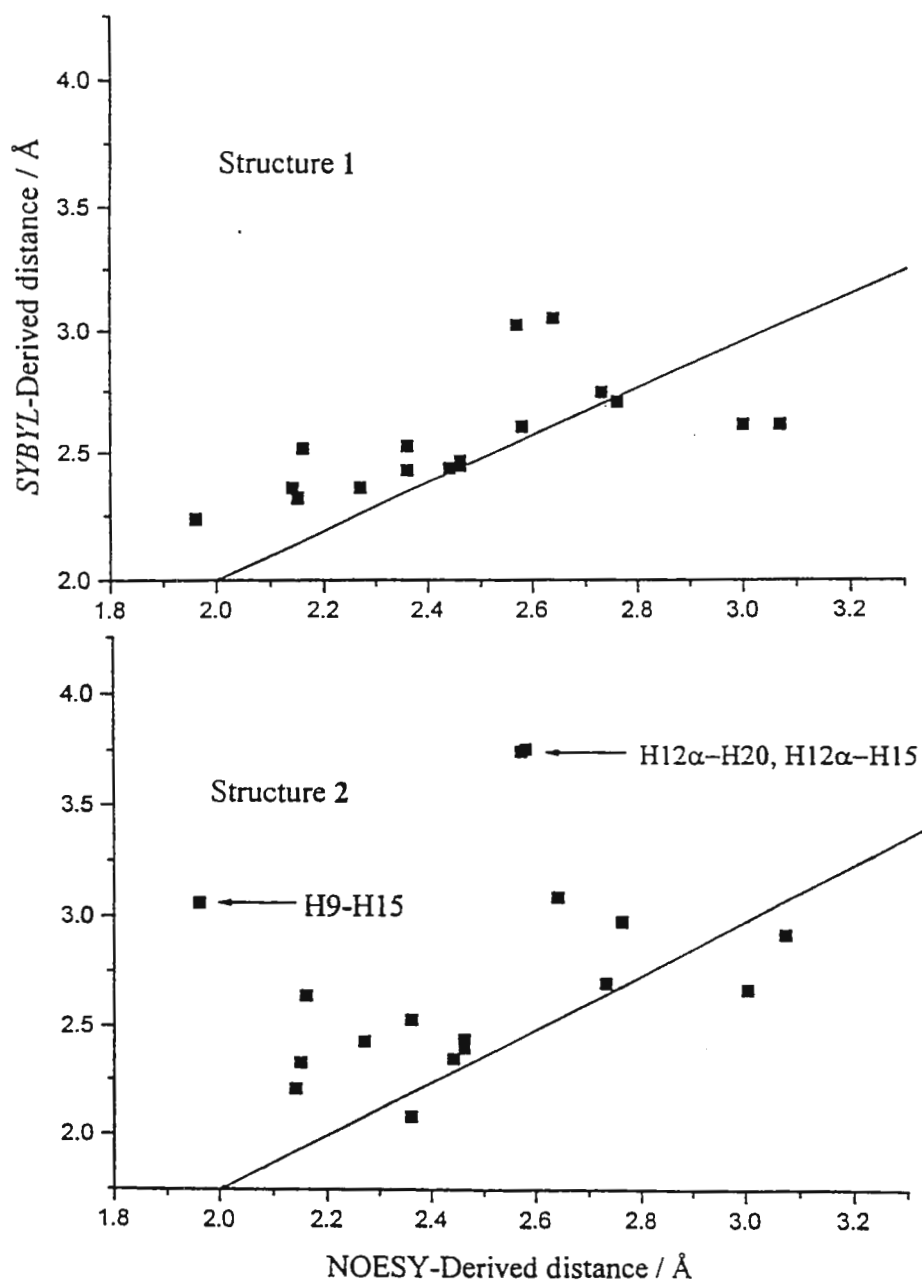
Dear Barry,

We have been working for some time¹ to determine the structure of a pure compound (*velutinol A*) isolated from the rhizomes of the native Brazilian plant *Mandevilla velutina*. Extracts of this plant are used in folk medicine, and taken orally are particularly effective against the effects of the certain snake-bites - especially those of *Bothrops jararaca*, a variety of lancehead responsible for most reported snake-bites in Brazil. Our preliminary studies using the now normal ¹H and ¹³C methods pointed to one of two possible structures:



The selection of 1 as the correct structure was made on the basis of quantitative interproton n.O.e. data. A series of NOESY spectra, analysed by the initial slope method and calibrated against a methylene pair distance, provided the experimental

distances. Energy-minimised *SYBYL*-generated structures of **1** and **2** provided calculated interproton distances, and the correlations between experimental and calculated distances for the two structures are shown below.



The straight lines indicate an ideal 1:1 correspondance. For **1** all points have < 0.5 Å difference between experimental and calculated distances, while for **2** there are three severe 'violations'. Clearly structure **1** wins.

G.E. Hawkes
G.E. Hawkes

E. Bento
E. Bento

¹ R.A. Yunes, M.G. Pizzolatti, A.E.G. Sant'Ana, G.E. Hawkes and J.B. Calixto, *Phytochemical Analysis*, 1993, **4**, 76; E.S. Bento, J.B. Calixto, G.E. Hawkes, M.G. Pizzolatti, A.E.G. Sant'Ana and R.A. Yunes, *J. Chem. Soc. Perkin Trans. 2*, in press.

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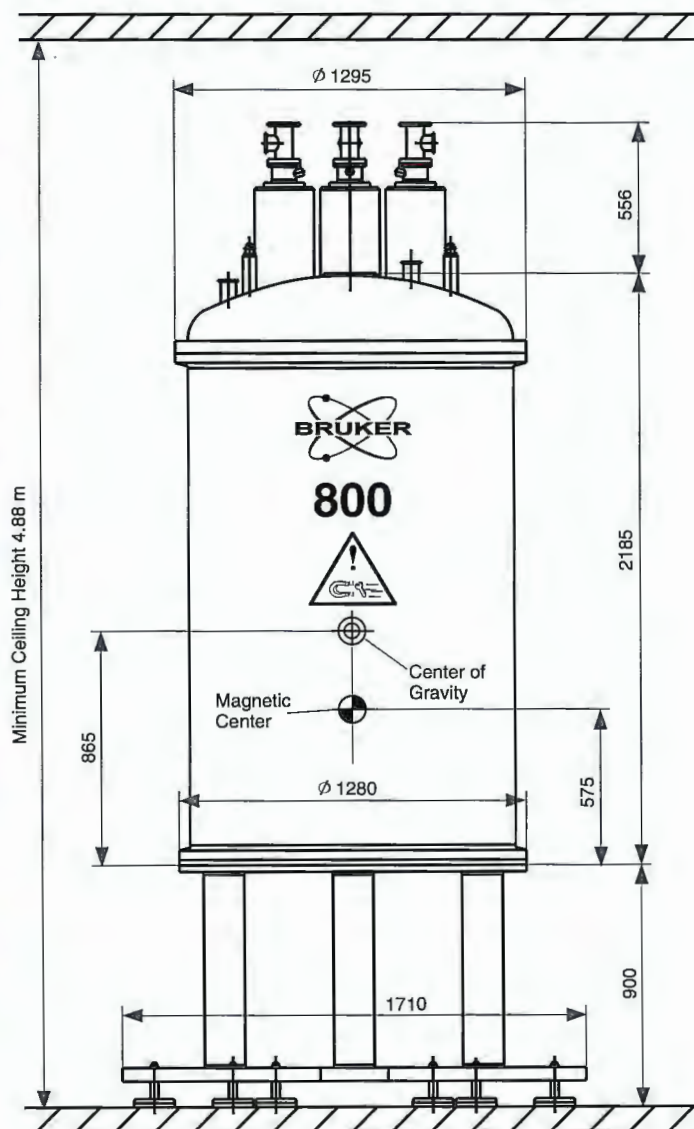
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NMR Frequency	800 MHz
Field Drift	< 8 Hz/hr
Superconducting Shims	z, z ² , x, y, xz, yz, xy, x ² -y ²
Resolution at 50% 1% CHCl ₃ 5 mm spinning	< 0.45 Hz
Lineshape 1% CHCl ₃ 5 mm non-spinning	
	at 0.55% < 6 Hz *
	at 0.11% < 12 Hz *
Spinning Sidebands	< 1%
5 G Line from the Magnetic Center	
- radially	6.1 m
- axially	7.6 m

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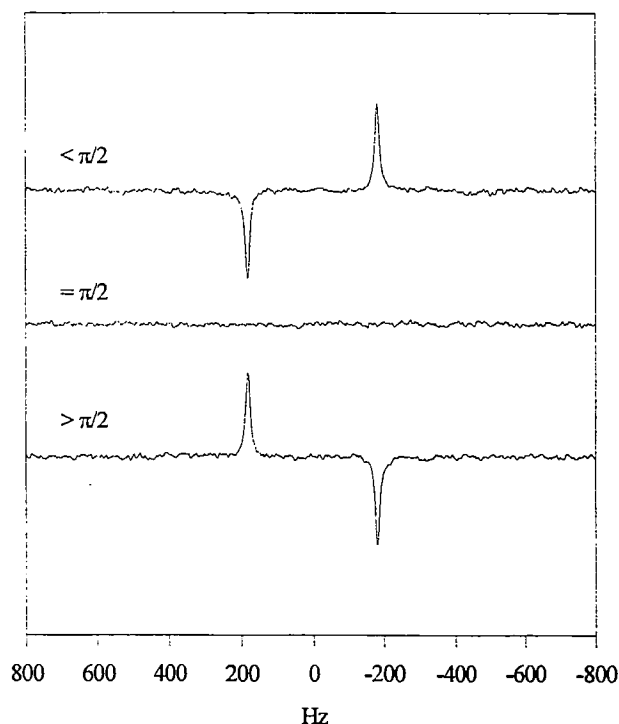
Standard Sample for Calibration of ^1H Decoupler Channel in ^{29}Si Polarization Transfer Experiments*

Dear Dr. Shapiro,

(received 6/28/96)

The utilization of the INEPT and DEPT polarization transfer experiments for ^{29}Si NMR characterization of organically modified alkoxysilane sol-gel materials is becoming more routine at Sandia National Laboratories. These methods are attractive because they afford improved ^{29}Si sensitivity and spectral editing capability. Calibration the ^1H decoupler pulse lengths are crucial for optimal performance of the INEPT and DEPT sequences. For a simple estimate of the decoupler pulse length, the null in the observed X signal following the $\pi/2(\text{X}) - \tau - \pi/2(^1\text{H})$ sequence can be used [1]. Benzene is typically used as a standard calibration sample for ^{13}C observations. We have looked for suitable standard samples that would provide similar results while investigating ^{29}Si systems, and have found that trichlorosilane, Cl_3SiH , provides a convenient sample for decoupler calibration as shown in Figure 1. At pulse lengths shorter than or greater than the decoupler $\pi/2$ pulse the expected anti-phase doublet is observed, while at a $\pi/2$ pulse a null signal is observed. The interpulse delay is optimized to the heteronuclear coupling, $J(\text{Si,H}) = 364 \text{ Hz}$, by using $\tau = (2J)^{-1} = 1.37 \text{ ms}$. A 5 mm neat Cl_3SiH sample provided a S/N of approximately 50:1 on our AMX400 using a 5 mm broadband probe. A recycle delay of 10 - 15 s was sufficient to prevent signal saturation. A draw back for using Cl_3SiH as a standard is the inhalation hazards associated with this compound (readers are encouraged to consult the MSDS), but by using sealed samples these hazards are minimized.

Figure 1. ^{29}Si NMR spectra of Cl_3SiH during ^1H decoupler pulse length calibration. Spectra obtained using neat Cl_3SiH at 79.49 MHz on a Bruker AMX400 using 1 scan and a 10 s recycle delay.



[1] D. M. Thomas et al. *J. Magn. Reson.* **42**, 298-306 (1981).

Sincerely,

T. Alam

Todd M. Alam¹

* This work is supported by the US Department of Energy under Contract DE-AC04-94AL85000

¹ Properties of Organic Materials Department, Sandia National Laboratories, Albuquerque, NM 87185-1407

KARL-FRANZENS-UNIVERSITÄT GRAZ
Institut für Organische Chemie

Dr. Heinz Sterk

A-8010 Graz, 14.5.1995
Heinrichstraße 28
Tel. (0316) 380 DW. 5321 bzw. 5320

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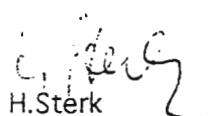
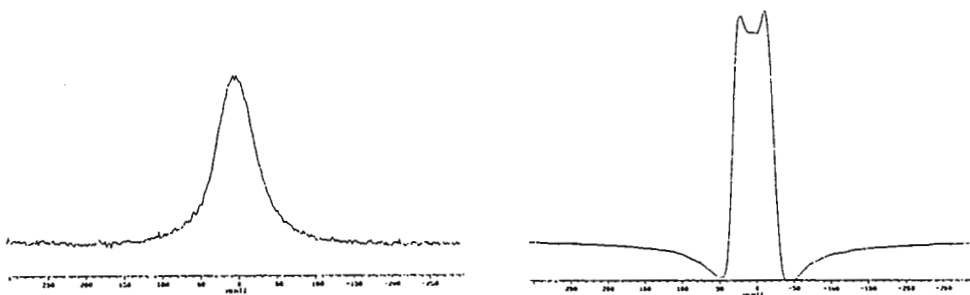
(received 6/10/96)

Dr. Bernhard Shapiro
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto, CA 94303**Gradients on an old AM 360**

Dear Dr. Shapiro :

In our laboratory we are used to a good brave old work horse called AM 360. During the periods of years we taught our horse to do inverse spectra, later on selective pulses, and with the beginning of this year it learned to perform z-gradient pulses, using the Bruker designed ARX-Z-Gradient WFM as well as the BGU Gradient unit. We don't know if either Bruker or, which is more likely, we have improved that much during the last years, as far as the handling of the spectrometer is concerned, but nothing we ever changed in our spectrometer came that fast and without any problems to run as the z-gradients did. From "Watergate" to GOESY our old mare runs now in full career. So if anyone owns a 8 to 9 year old AM, an investment like z-gradients is really worth while. To give some examples, the measured excitation profiles of a 270° selective Gaussian pulse in chloroform (left) and an E-Burp in water (right), with some radiation damping, are shown beneath.

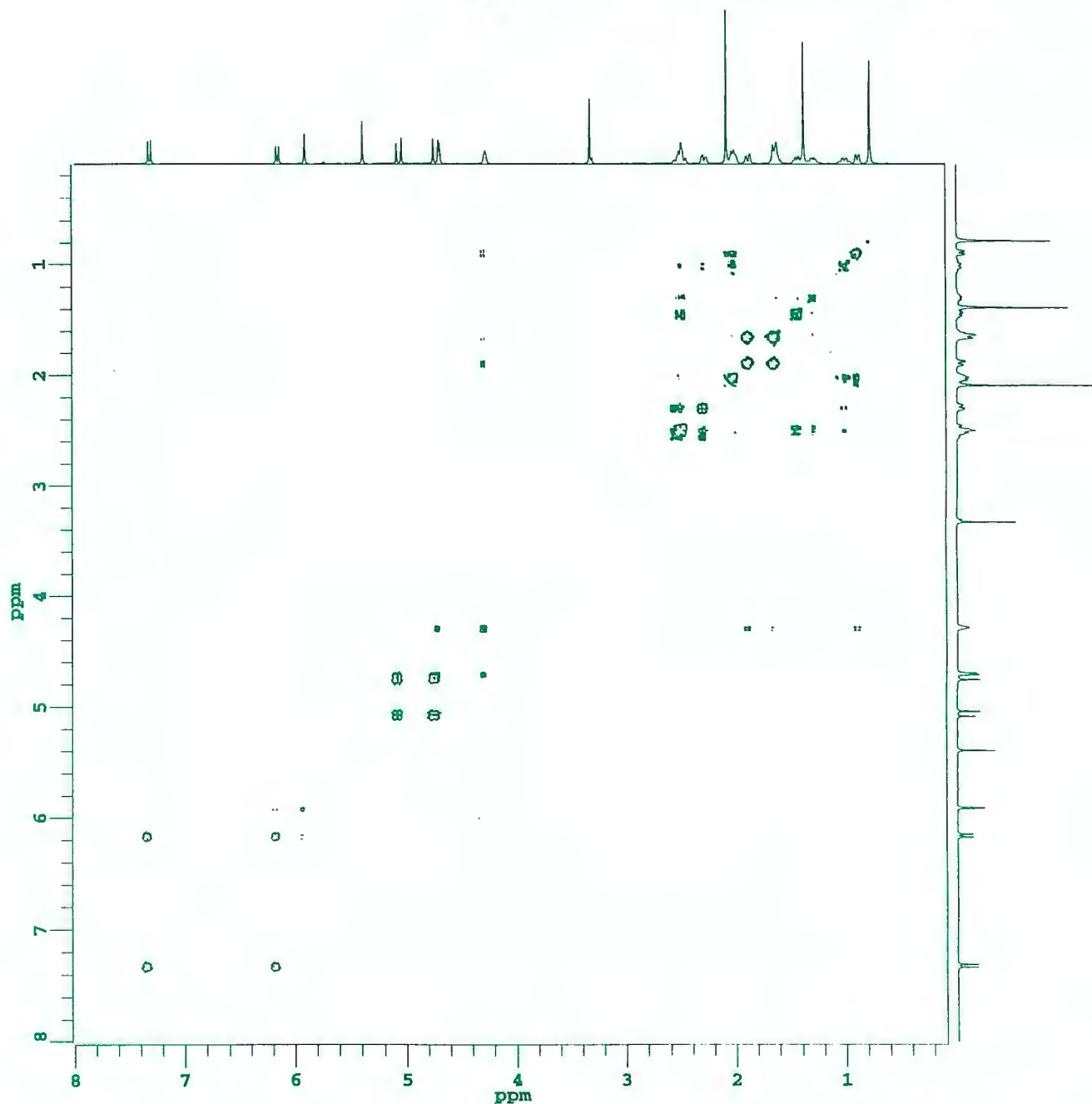
Yours sincerely


K. Zangger
H. Sterk



Chemagnetics

Otsuka Electronics USA Inc.



This spectrum was acquired as 1024 rows of 1024 complex points each.

**^1H - ^1H PHASE-SENSITIVE PFG DOUBLE QUANTUM
FILTERED COSY OF
PREDNISOLONE 21-ACETATE**

**Shown here is the ^1H - ^1H PFG DQFCOSY
spectrum of prednisolone-acetate in
DMSO- d_6 collected on the
*Chemagnetics*TM 400 MHz CMX Infinity
Spectrometer.**

- Excellent signal-to-noise and resolution are obtained in this spectrum. Only two scans per row were necessary for the signal-to-noise seen here.
- SpinsightTM software features multiple viewports which can contain acquisition data and processed data of multiple dimensions. Parameters can be exchanged between the viewports for easy experiment setup.
- Using pulsed field gradients dramatically reduces experiment time.
- Compared to the traditional phase-sensitive COSY experiment, the double quantum filtration version suppresses single quantum coherences.
- This filtration simplifies the spectrum along the diagonal thereby allowing for more straightforward structural analyses. Because we are observing higher order coherences, greater sensitivity is necessary to detect these weak couplings



Chemagnetics

Otsuka Electronics USA Inc.

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To
Dr. Bernhard L. Shapiro
The NMR Newsletter
966 Elsinore Court
Palo Alto CA 94303
USA

(received 6/20/96)

Vienna, 29.5.1996

Suppression of two signals with 3-9-19 WATERGATE

Dear Dr. Shapiro,

I would like to report an application for solvent suppression in the field of carbohydrate NMR. We did separations on a high performance anion exchange chromatography system (HPAEC) collecting fractions from an analytical column. The resulting samples, very low in concentration, additionally contain acetate derived from the potassium hydroxide / potassium acetate elution gradient of the HPAEC system. In order to avoid losing too much material by tedious desalting techniques these samples should be investigated directly by NMR just after freeze drying and redissolving them in D₂O.

Of course there are a lot of techniques available to suppress both the solvent and the undesired acetate signal. I choose the off-resonance version of the WATERGATE sequence with the tailored 3-9-19 puls train as described by V. Sklenar et. al. (J. Magn. Reson., Series A 102, 241, 1993). In my opinion this is the easiest applicable method for obtaining the best results. There is only one hardpulse to calibrate, the carrier is set in the middle between the two signals to suppress, the delay in the puls train $\tau = 1/\Delta\nu$ of the two signals to suppress. Then the phase-order {0°, 180°, 0°, 0°, 180°, 0°} delivers zero net excitation at the solvent signals and the second gradient will not rephase them. In Fig. 1 the proton spectrum of a pentasaccharide is shown. The ¹³C satellites of the acetate are attenuated by the 3-9-19 puls train, ¹³C decoupling can be applied if desired. Of course also anomeric protons in vicinity to the HDO signal are reduced in their intensity. But in the corresponding 2D experiments the necessary crosspeaks can all be seen undisturbed at the ω_1 frequency, e.g. Fig. 3 shows a HOHAHA (MLEV-17 spinlock) spectrum of the same sample. The trace in Fig. 2 gives a row of the 2D at ω_1 of an attenuated anomeric signal. The complete monosaccharide can be seen without any disturbing artefacts at the frequency of the HDO and the acetate protons.

Sincerely yours,

Dr. Kählig Hanspeter

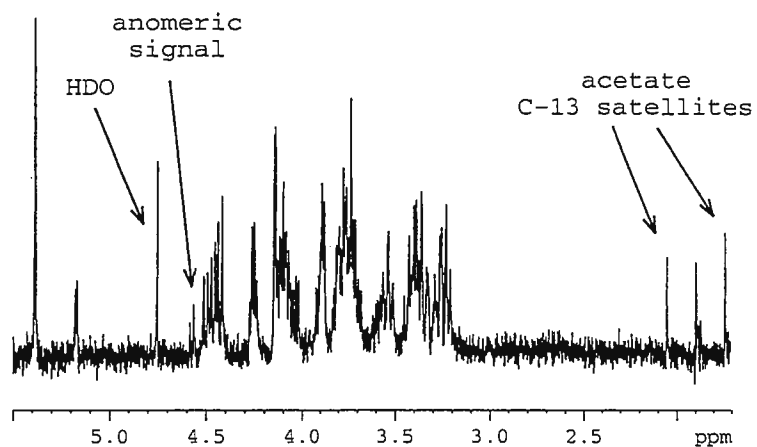


Fig. 1:

H-1, 400 MHz
3-9-19 WATERGATE
(8 gauss/cm, 1 ms)

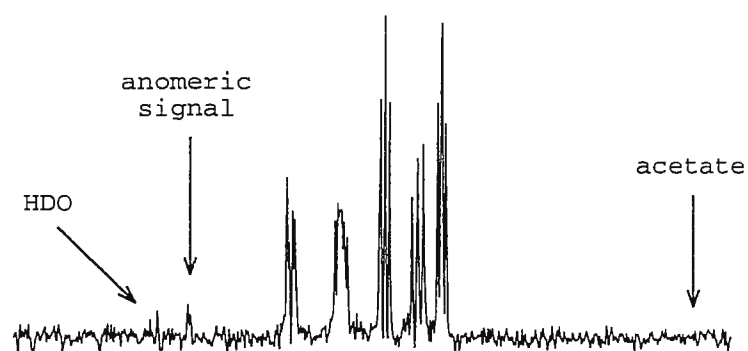


Fig. 2:

TOCSY row at a)

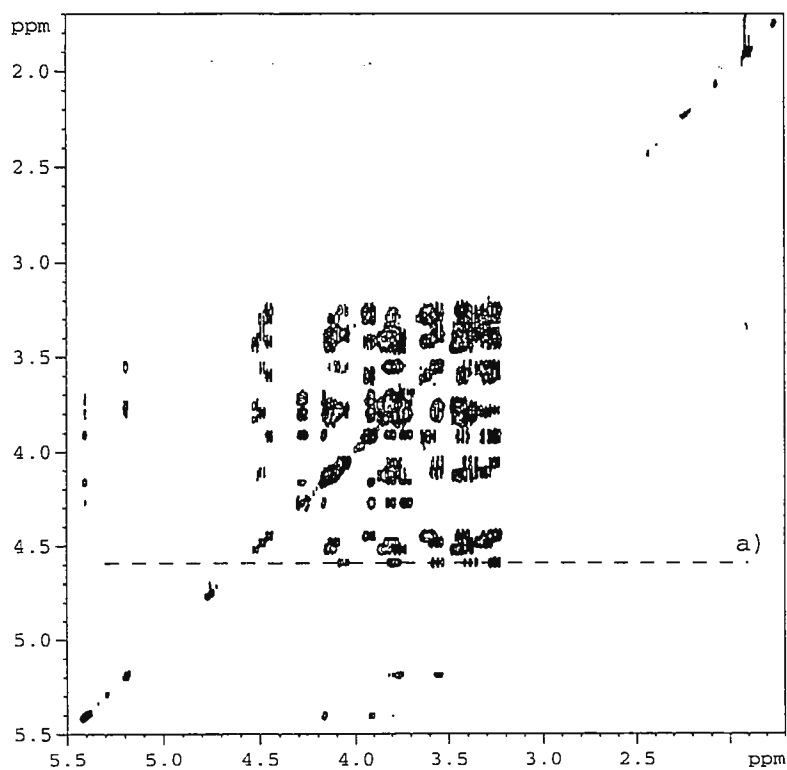


Fig. 3:

HOHAHA, 400 MHz
MLEV-17 spinlock
3-9-19 WATERGATE
(8 gauss/cm, 1 ms)

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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
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200	89	2	365	2.8
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Dr. B.L. Shapiro, Publisher
The NMR Newsletter
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(received 5/28/96)

Your reference and date

Our reference

Office telephone

Date

31 15 786041

21-May-96.

Subject

Sub-division

Dear Prof. Shapiro,

A Non-invasive Perfusion experiment

Recently some non-invasive perfusion imaging techniques have been presented in which both magnetic labeling and signal detection take place within the same slice (1-3). Here a method is described in which in-flowing spins with equilibrium magnetization change the in-slice partially saturated magnetization. The difference between a slice selective and a non-slice selective experiment is a measure for the flow of the spins.

In a saturation period the detection slice is partially saturated by N slice selective pulses and crusher gradients. These N pulses are repeated with a repetition time T_R (fig 1). RF-spoiling is used to eliminate the transverse magnetization. During this saturation period the longitudinal magnetization reaches a steady state value. Due to perfusion equilibrium magnetization enters the detection slice and changes this steady state. An imaging sequence following the saturation period measures the new magnetization in the detection slice (perfusion sensitive image). Next the same is done with N non-slice selective pulses in the saturation period (control image). Now the in-flowing spins do not alter the magnetization in the detection slice. The difference between the perfusion sensitive and the control image is a measure for the flow of the spins. To increase the in-flow effect an additional in-flow waiting time T_i is added before imaging.

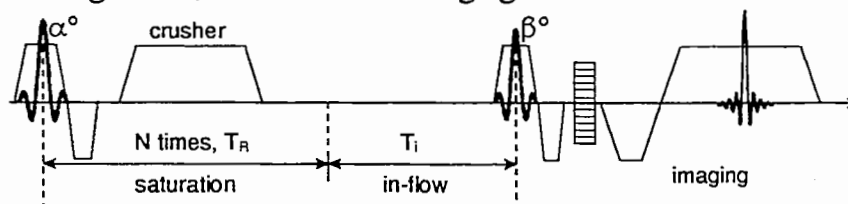


Figure 1.
A perfusion experiment containing a saturation and an imaging period.

Experiments on a flow phantom were performed on a 6.3T/20 cm bore home built spectrometer. The parameters for the saturation period were $N=10$, $\alpha=90^\circ$ and $T_R=0.1$ s. Images were obtained as a function of T_i . Laminar flow with a maximum flow velocity of $v_{max}=0.04$ cm/s was induced in the phantom. The laminar flow profile was clearly seen in the difference image. Maximum SNR is obtained with an in-flow time $T_i \approx T_1$. Increasing v_{max} a

factor 1.5 to 0.06 cm/s also increased the signal intensity about the same factor. This shows that in principle relative quantification of blood flow is possible.

The results show that this method is sensitive to small flow values. The SNR of this steady state experiment, compared to the inversion recovery based techniques (1-3) is a factor 2 lower. However these techniques suffer from signal decrease due to saturation effects if the experiment is repeated too fast. Therefore the SNR loss can be less than a factor 2, because in the above introduced perfusion experiment there is no need for any waiting time between two perfusion sensitive images. This also results in an increased time resolution.

References: 1. Kim SG et al. [1995] *MRM* 34: 293-301. 2. Kwong KK et al. [1995] *MRM* 34: 878-887. 3. Schwarzbauer C et al. [1996] *MRM* 35: 540-546

Sincerely yours,

Ole T. Karlsen

Wim M.M.J. Bovée

POSTDOCTORAL POSITION

We are looking for a post-doctoral fellow to study the structural biology of membrane-bound peptides and proteins (1-3) in the Department of Chemistry and Biophysics Research Division at the University of Michigan, Ann Arbor, Michigan, USA. We are particularly interested in identifying a candidate with expertise on peptide synthesis through an automated solid-phase peptide synthesizer, HPLC, molecular biology of membrane proteins, multi-dimensional high resolution solution NMR of macromolecules, EPR, CD, molecular dynamics, FTIR, computation, and solid-state NMR. The position is available in the fall of 1996. The department has 200, 300, 360, & 500 MHz NMR spectrometers, FTIR, CD, spectrophotometers, peptide synthesizer, HPLC, EPR (Q band), Raman, several Silicon Graphics computers, and facilities for molecular biology work (including gene cloning, protein expression, isolation and purification). Solid-state NMR 400 MHz spectrometer will be installed soon. Application including CV, a list of publications, and two reference letters should be sent to the following address.

A. Ramamoorthy
Department of Chemistry
University of Pennsylvania
Philadelphia, 19104.

e.mail: rams@chestnut.chem.upenn.edu
Tel: (215)898-8301
Fax: (215)573-3899

Related research papers:

1. A. Ramamoorthy, F. M. Marassi, M. Zasloff, and S. J. Opella, *J. Biomol. NMR.*, **6**, 329-334 (1995).
 2. S. J. Opella, Y. Kim, and P. McDonnell, *Meth. in Enzymol.*, **239**, 536-560 (1994).
 3. S. O. Smith and O. B. Peersen, *Ann. Rev. Biophys. Biomol. Str.*, **21**, 25-47 (1992).
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Dr B L Shapiro
The NMR Newsletter
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DUNCAN M. SMITH
Molecular Structure Group
e-mail: duncan@amgen.com

May 28, 1996
(received 6/1/96)

Automatic Conversion of AVANCE™ Data to NMRPipe

Dear Barry,

Many people use off-line NMR data processing to take advantage of features not available from the manufacturers' software or simply to leave the spectrometers acquiring data. One such package is NMRPipe (1) which is routinely used in our laboratory. Like most other processing packages, the raw NMR data must first be converted to the required format before it can be processed. In NMRPipe this involves writing a small shell script in which information such as the number of points, spectral width and carrier etc. must be provided for each dimension. This process is fairly labor-intensive, provides significant opportunity for error and, most importantly, has in effect already been carried out by the spectrometer. On Bruker AVANCE™ spectrometers this information is stored in *acqu*, *acqu2s* and *acqu3s* files.

I have written a small suite of shell scripts running on SGI's which extract the relevant parameters from the *acqu* files and create the conversion script for 1D, 2D and 3D AVANCE™ data. The script could be extended to include 4D data, and would need to be modified for AMX data as some of the parameter names differ. The parent script is called "drx2pipe", and the whole process takes only a few seconds once the pathname of the data has been entered. This significantly reduces the possibility for error as the script extracts the values of the parameters actually used in the experiment (caveat: as long as the information in the *acqu* file is correct!). This has several other advantages:

- it enables the user to check on the progress of multidimensional experiments as only the number of acquired FID's appears in the conversion script. This makes the monitoring of the progress of longer multidimensional experiments trivial.
- additional information is recorded in the conversion script header, including the instrument name, sample temperature, pulse program and number of scans per FID.

- further information can be included as the script searches for the string "; NMRPipe" in the pulse program, and so the author can add information for the user such as phasing in the indirect dimension(s) in the comments section.

Below is an example output for an HNCO experiment:

fid directory does not exist, creating it ...

Fetching information from acqu files ...

Creating convert.com file ...

```
#!/bin/csh -f
# convert.com
# File created by drx2pipe on Thu May 9 08:25:39 PDT 1996
# Data recorded at 298K on drx500
# Thu May 9 08:20:28 1996
# File: /u/data/username/nmr/mydata/12/acqus
# using pulse program hncowseags3d with 8 scans per FID
# Phases for 13C & 15N = -90,180 (13C zero order approx.)
#
setenv NMRTXT /directoryname/nmrtxt
bruk2pipe -in /machinename/u/data/username/nmr/mydata/12/ser -DMX -decim 24
-noswap \
-xN      1024   -yN      60   -zN      128   \
-xT      512   -yT      30   -zT      64    \
-xMODE   Complex -yMODE   Complex -zMODE   Complex \
-xSW     8012.821 -ySW     1216.545 -zSW     1635.056 \
-xOBS    500.130000 -yOBS    50.677748 -zOBS    125.757453 \
-xCAR     4.702   -yCAR     118.000 -zCAR     176.000 \
-xLAB     1H     -yLAB     15N     -zLAB     13C   \
-ndim      3    -aq2D      States \
| nmrPipe -fn MAC -macro $NMRTXT/ranceY2.M -noRd -noWr \
| pipe2xyz -out fid/hnco%03d.fid -verb -ov -nofs
```

Checking available disk space ... OK

Start 3D data conversion now? y/n [y]

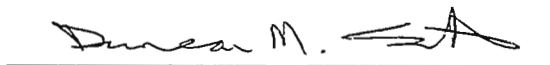
Hitting "return" then copies and converts the data into a directory called "fid" within the current directory. Bruker users will note that I have changed the basic spectrometer frequency from the default value for ^{13}C to give closer chemical shift referencing in that dimension. Note also that the script interprets the pulse program name, stripping off predefined pairs of letters to make a guess at a sensible output filename. If the user wants a different name, he/she answers "n" and then edits the script as desired. In addition, the string "ea" in the pulse program name is used to call the macro "ranceY2.M" to convert the data in a manner analogous to Bruker's "Echo/Anti-echo" for gradient sensitivity enhancement.

For those interested in using "drx2pipe" simply download it from Bruker's ftp server. Proceed as follows:

```
ftp bruker.com
login as "anonymous"
provide your e-mail address as password
cd pub/nmr/download/external/xwin2pipe
get the 5 files in that directory
the README file gives installation instructions
```

There are a few things which need to be customized to suit your needs such as directory names and allowed instrument names. No guarantee is made that these scripts will work on every system!

Good luck!



Duncan M Smith

1. "NMRPipe - A Multidimensional Spectral Processing System Based on Unix Pipes", F Delaglio et al, J Biomol NMR 6 #3, pp277-93 (1995)



Prof. Dr. Bernhard Blümich

Tel.: 0241/80 6420

Fax: 0241/8888 185

e-mail: bluemich@rwth-aachen.de

Datum: 12.06.96

RWTH, Makromolekulare Chemie, SB Chemie

Worringer Weg 1, D-52056 Aachen

Dr. B. L. Shapiro

The NMR Newsletter

966 Elsinore Court

Palo Alto, Ca 94303, USA

(received 6/19/96)

re.: The NMR MOUSE

Dear Barry,

Apart from a few exceptions the size of the object in NMR is restricted to fit the geometry of the magnet bore. Homogeneous magnetic fields are acquired at high cost to obtain spectroscopic resolution for chemical analysis. On the other hand, spatial resolution is obtained by the use of inhomogeneous magnetic fields, which most often involve constant field gradients for linear variation of the magnetic field with space. In fact the predominant asset of NMR imaging versus other image forming techniques, the tremendous range of parameters generation of image contrast, can often be exploited in inhomogeneous magnetic fields as well. Thus NMR with spatial resolution can be done with magnets, which provide inhomogeneous fields in the first place.

This idea is by no means new. The oil people looked into this as of the early fifties, Damadian's FONAR technique is based on inhomogeneous fields, and so are the inside-out NMR techniques of Bob Kleinberg, the stray field imaging of Zamoilenko and the molecular diffusion work of Franz Fajara in a dedicated, super-conducting gradient magnet. Stimulated in part by earlier work of Al Garroway, Joel Miller, and Bob Kleinberg we have constructed an NMR surface scanner from permanent magnets and a surface coil. We call it the NMR MOUSE for *MO*bile *U*niversal *S*urface *E*xplorer. Our B_0/B_1 probe fits into one hand. It produces nice echo signals at 17 MHz from about any proton containing object: Frozen food, the human hand, wood, plants, rigid polymers and rubbers. Depending on the frequency, the penetration depth can be adjusted between 0 and 5 mm with the present prototype design. The probe is attached to a single-channel, low-field, PC-based spectrometer console. Present work focusses on technical rubbers, where we could map cross-link density and strain. In an impact-modified polystyrene sheet we could detect a region of stress whitening by changing decay times of the multi-echo decay. We believe, that there is great potential for useful applications of such an NMR MOUSE, but the theory of NMR in inhomogeneous B_0 and B_1 fields is far more tricky than the simple case of homogeneous fields.

In the mean time we have learned, that a similar machine is commercially available for investigations of water in concrete, and that even a patent exists for clinical imaging with such an arrangement in connection with surface gradients. So nothing is really new, but progress nevertheless is incremental.

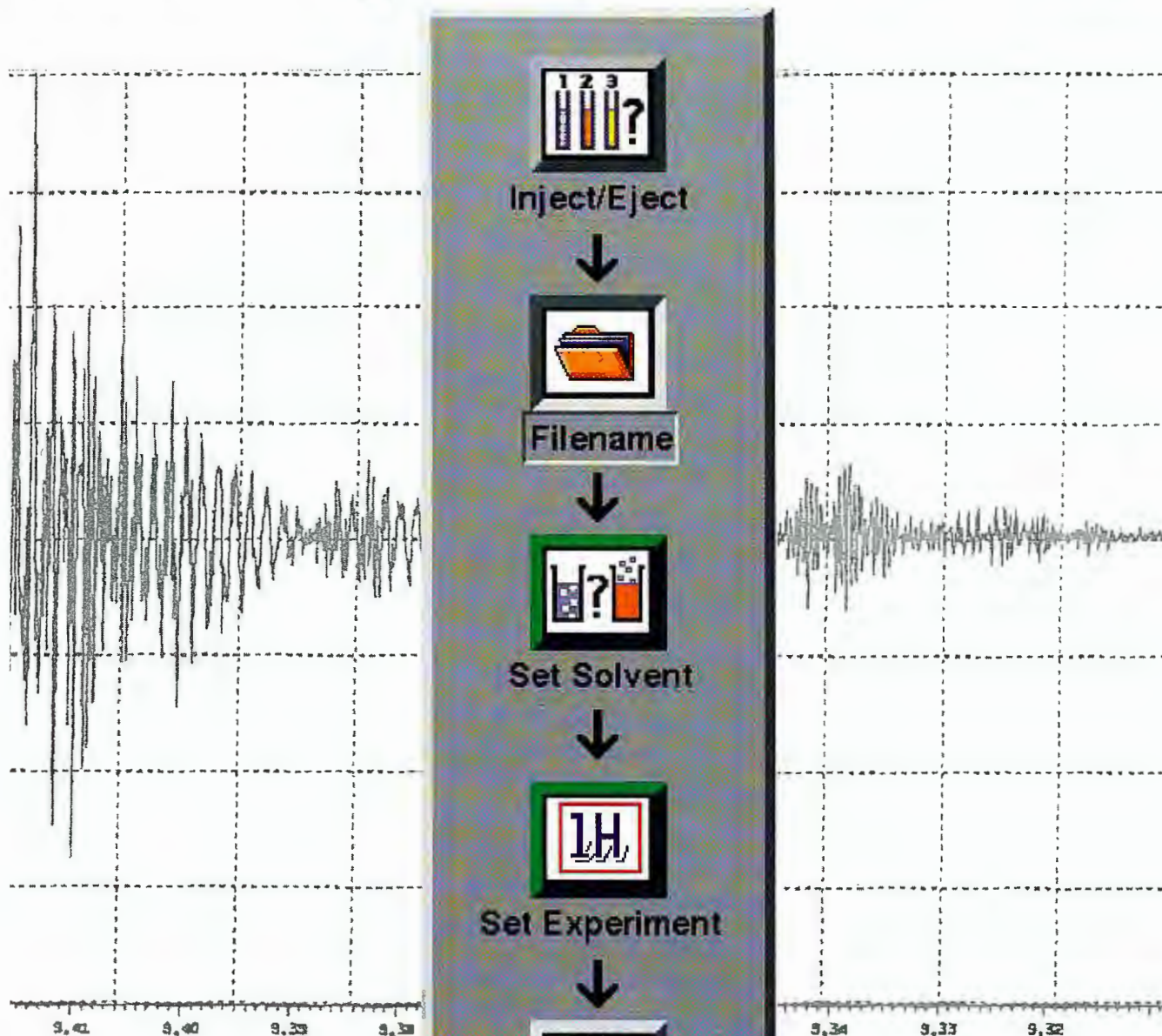
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Bernhard Blümich

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

June 11, 1996
(received 6/13/96)

Local Structural Perturbation of the C5a L43A Mutant

Dear Professor Shapiro:

C5a is a 74 amino acid serum glycoprotein cleaved from the fifth component of the serum complement (C5). Production of this protein has been implicated in a variety of inflammatory diseases including acute respiratory distress syndrome, ischemia and reperfusion injury, rheumatoid arthritis, psoriasis and inflammatory bowel disease. To better understand the relationship between the structure and function of this protein, the 3-dimensional structure of a key C5a mutant (L43A) was determined using NMR spectroscopy. This mutant was of particular interest since the point mutation from leucine to alanine at residue 43 showed a three fold reduction in receptor membrane binding and an eighteen fold reduction in intracellular calcium rise in human neutrophils (agonist potency) relative to C5a.

Comparison of the NMR derived structures of C5a and L43A C5a are shown in figure 1. Overall both structures retain similar global conformations for core residues 1-64 while the C-termini (residues 65-74) remain conformationally undefined. The point mutation at residue 43, however, was found to cause a local structural perturbation in the third loop of the L43A mutant, where Ala 43 is able to form a hydrophobic association with the core of the protein. This interaction is not likely to occur with Leu 43 due to unfavorable steric interactions.

The conformational difference between C5a and the L43A mutant suggests that the local perturbation in the core of the mutant is responsible for the corresponding decrease in receptor binding and biological activity. Hence these structural studies combined with point mutation analyses were instrumental in identifying which structural features are necessary to preserve C5a receptor binding and agonist potency.

Sincerely,

Xiaolu Zhang, Ph.D.

Nina C. Gonnella, Ph.D.

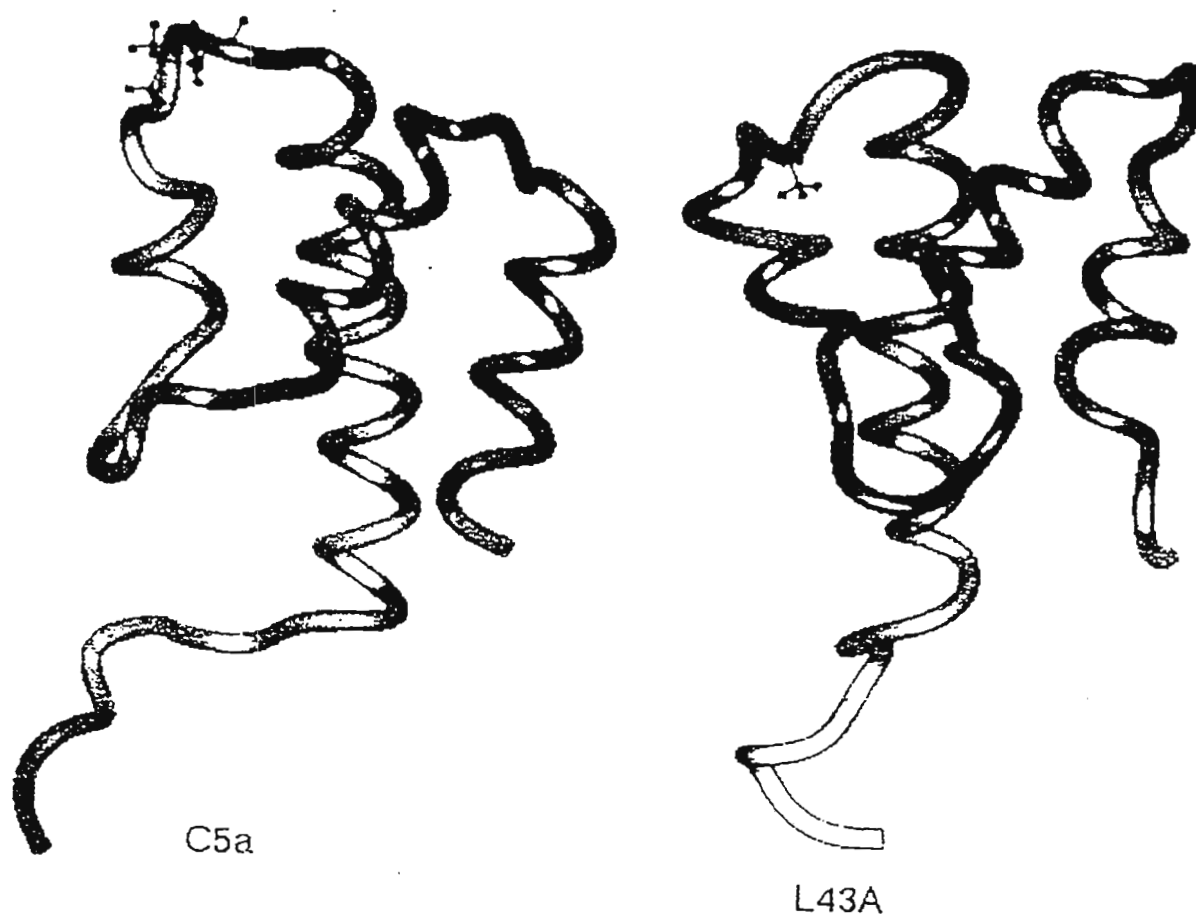


Figure 1. Ribbon structures of C5a and the L43A mutant are shown. Contact residues Leu 43 and Ala 43 are displayed for C5a and L43A mutant respectively. The structures are oriented with the N-terminus in the lower right and an arbitrary conformation of the C-terminus in back.

WORTH A FORTUNE!

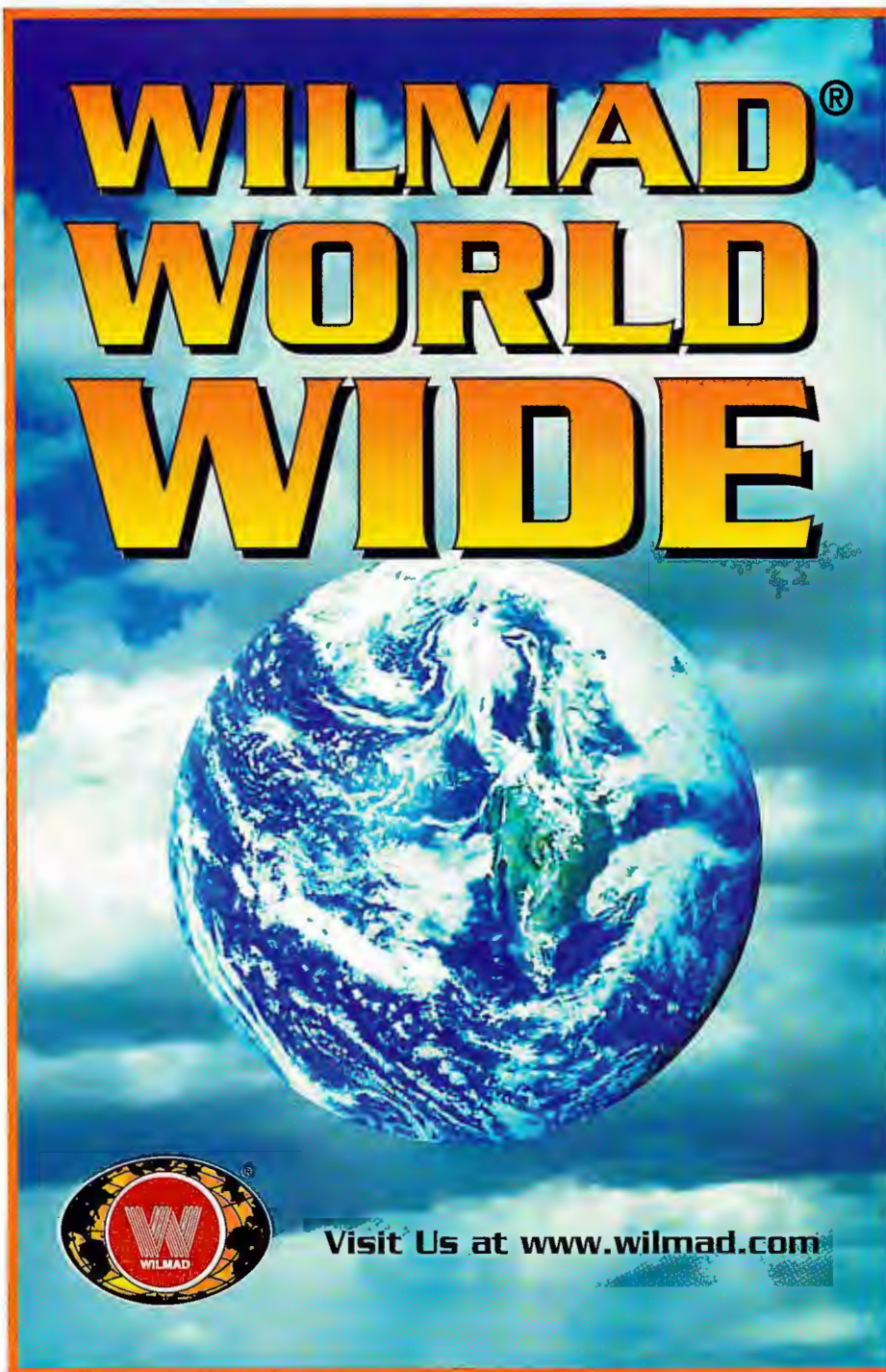
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June 13, 1996
(received 6/17/96)

Interatomic Distances in Solids Via Zero Quantum Coherence.

Dear Barry:

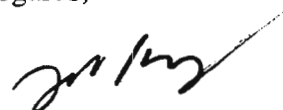
In recent years, our group has used solid state spin diffusion techniques including rotational resonance⁽¹⁾ to measure intermolecular distances in polymeric blends. In order to eliminate the competing effects of intramolecular spin diffusion in these systems, only a single isotopic label was typically introduced. As a consequence, it was extremely difficult to quantify the Zeeman exchange. However, we have found that by combining rotational resonance with 2D Zero Quantum Correlation Spectroscopy we can easily separate the spins which undergo exchange and those which do not (which is virtually impossible in the generic rotational resonance experiment using a single label). Thus, a quantitative determination of intermolecular distances is possible in such systems.

In theory, the cross peak build-up depends upon the excitation delay, τ , in the sequence $\pi/2-\tau-\pi-\tau-\pi/4-t_1-\pi/4-ACQ$ ⁽²⁾, under rotational resonance conditions with standard CPMAS. Further, the cross peak intensity maxima, τ_{max} , is related to the dipolar coupling; from which the carbon-carbon bond distance can be determined from the position of this maximum. To this end, we have investigated the viability of using such a dependence for determining distances in $Zn(^{13}CH_3^{13}COO)_2$ using 2D Zero Quantum Correlation Spectroscopy under rotational resonance conditions. We have followed the cross peak build-up in the zinc acetate system as described and we were able to determine $r_{CC} = 1.61 \text{ \AA} \pm 0.10 \text{ \AA}$ using simulations based on GAMMA⁽³⁾. The ultimate precision depends on fine graining of τ in the region of τ_{max} . The figure shows the carbon-carbon ZQ spectrum of crystalline doubly labeled zinc acetate.

Furthermore, we have applied this type of experiment to polymeric samples and have found that the selectivity of rotational resonance and Zero Quantum Correlation Spectroscopy gives us the potential of determining the distance distribution from a series of 2D measurements where only a single label is present.

1. M. H. Levitt, D. P. Raleigh, F. Creuzet and R. G. Griffin, J. Chem. Phys. 92, 6347 (1990).
2. L. Muller, J. Mag. Res. 59, 326 (1984)
3. S. A. Smith, T. D. Levante, B.H. Meier and R. R. Ernst, J. Mag. Res. 106a, 75 (1994)

Best regards,

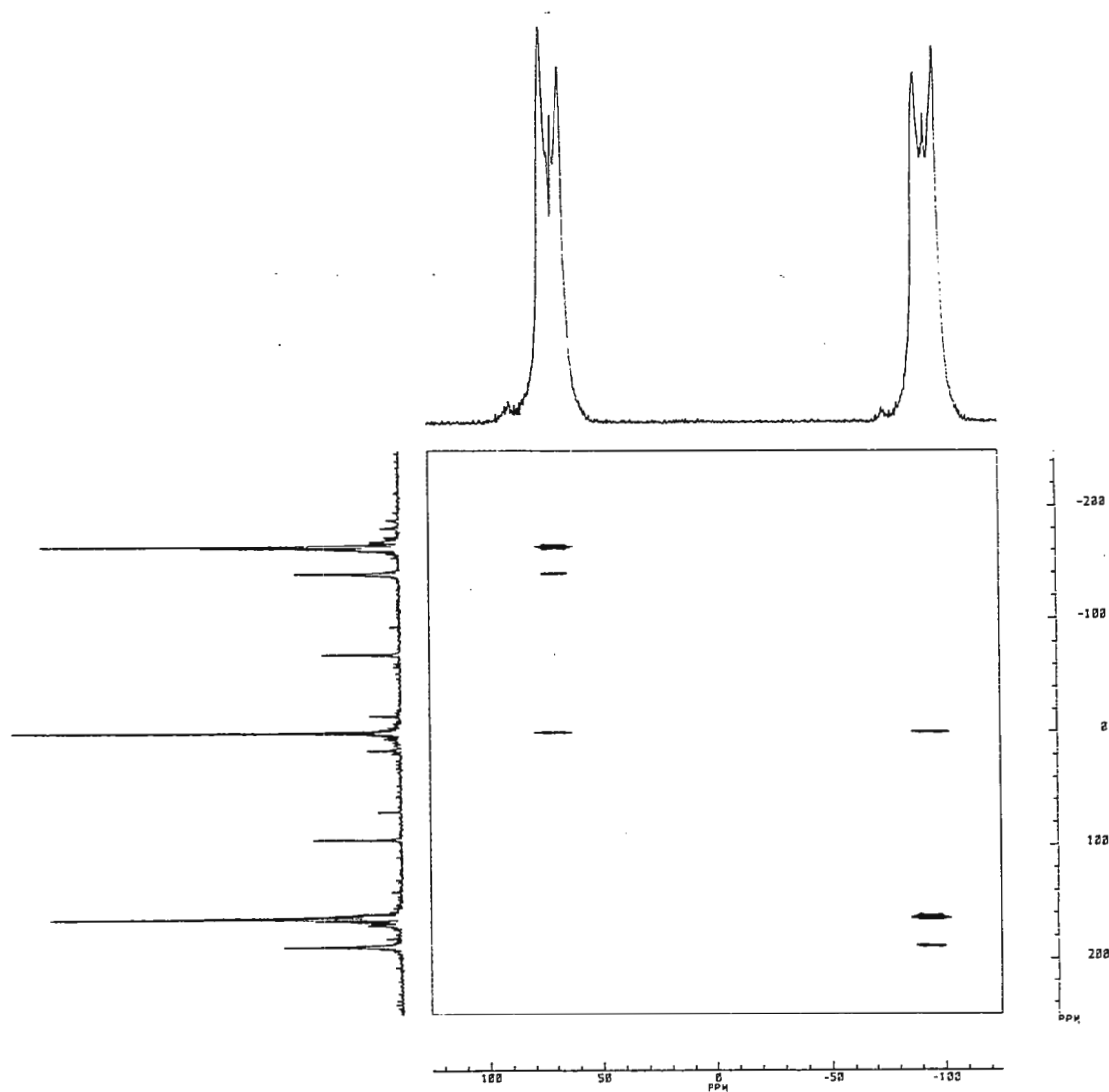


Jeff Koons



Paul Inglefield

Figure: Carbon-carbon zero quantum spectrum of solid $\text{Zn}(\text{}^{13}\text{CH}_3\text{}^{13}\text{COO})_2$. The spectrum is recorded on a Bruker MSL300 spectrometer in a CPMAS probe with $\omega_r = 13,085\text{Hz}$. The total number of scans is 8K with 1024 t_1 values and τ is 1ms.



DOTY MP-II

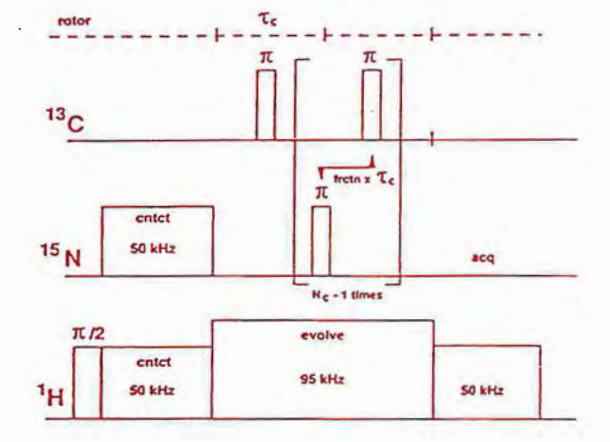
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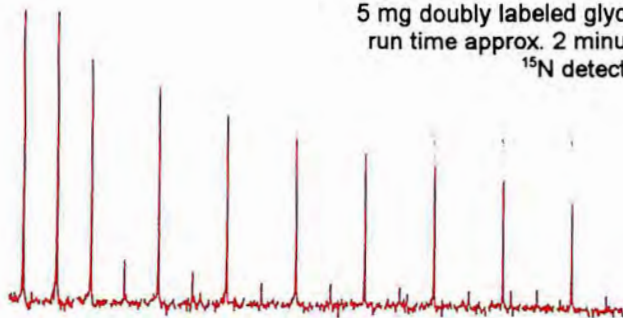


If you've never done solids NMR, you may need some additional hardware. You will need high-power decoupling (60 to 110 kHz, 100 to 300 W), but modern balanced circuits have virtually eliminated the noise and break-through problems that were common with high-power decoupling in the early days. You may need a high-power (200 to 500 W) observe transmitter. State-of-the-art 5-mm Magic Angle Spinning probes routinely spin samples for quadrupolar nuclides at speeds above 18 kHz, but you'll only need to spin your REDOR samples 3 to 6 kHz. If you have a recent software release, you probably already have the necessary pulse sequences. If not, your spectrometer manufacturer will be happy to provide them. And of course, you'll need a triple-resonance solids probe with high ^{15}N sensitivity for your magnet.

600 MHz Narrow Bore, H-C-N REDOR.

5 mg doubly labeled glycine
run time approx. 2 minutes

^{15}N detection



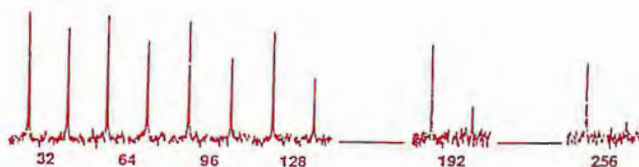
Dr. Dave Rice, Applications Chemist, Varian NMR
Dr. David Doty, Doty Scientific, Inc., Columbia, SC

If you're fighting sensitivity problems in the liquid state because of low solubility and aggregation, or if you simply need to know the solid-state conformation, maybe it's time to look at a powerful, new solid-state technique — REDOR. One merely fits the normalized difference in intensities (with and without the third channel) vs. rotor cycles to a simple function of bond length.^{1, 2} If you've been told triple-resonance solids can't be done in a narrow-bore, take a look at the above 600 MHz ^{15}N spectra taken on a fully multinuclear probe in about two minutes. You're probably already doing triple-resonance hi-resolution experiments, and modern solids probes are just as reliable and easy to use.

300 MHz WB, 95 kHz ^1H during evolution

Sample: WHWLQLKPGOPNLeY

^{13}C π pulses on alternate cycles



CYCLES

Prof. Ruth Stark, CUNY, College of Staten Island

1. T. Gullion and J. Schaefer, "Detection of Weak Heteronuclear Dipolar Coupling by Rotational-Echo Double-Resonance NMR", in *Adv. in Magn. Reson.*, ed. W. S. Warren, Academic Press, San Diego, 1989, Vol. 13, 57-83.
2. J. R. Garbow, M. Breslav, O. Antohi, F. Naider, *Biochemistry* 33, (1994), 10094-10099.

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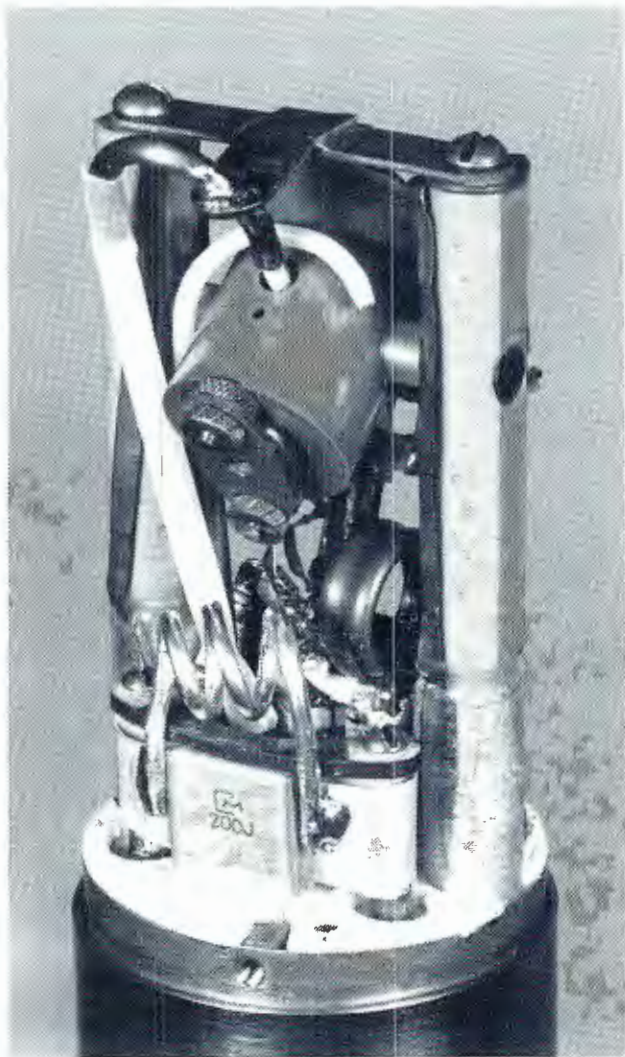
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200 WB	7	90	170	83 / 78	230
200 WB	10	70	260	56 / 50	420
300 NB	5	105	180	78 / 60	180
300 NB	7	71	160	63 / 40	440
300 WB	10	50	210	40 / 40	800
400 NB	5	90	180	68 / 55	260
400 WB	5	100	210	83 / 83	300
400 WB	7	60	160	50 / 45	770
500 NB	5	75	190	57 / 55	400
500 WB	5	90	200	83 / 63	440
600 NB	5	70	210	50 / 45	530
600 WB	5	75	210	55 / 50	570
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PROBE
ANALYTICAL
13 May 1996
(received 5/31/96)

NMR (ALMOST) ON-LINE - "NMR-NIR 2-D" CORRELATIONS

Dear Dr Shapiro

Near Infrared (NIR) spectroscopy is concerned with the 1100-2500 nm region, containing overtones and combinations of fundamental IR vibrational bands. NIR spectra incorporate a lot of chemical structure information, but the spectra look like a squashed blancmange! Band assignments are difficult due to multippeak overlap, and often not even attempted. Carbon-13 NMR spectra, conversely, incorporate a wealth of highly-resolved chemical structure information.

NIR spectrometers are cheap(ish), portable, robust, multiplexible, fast and easy to use. Plants wanting on- or at-line analytical information (viscosity, acid values, water content, etc) love them. NMR spectrometers, conversely, are expensive, immobile, fragile, slow and obtuse. Plants ignore them.

Here, we describe our first efforts to map the exquisite molecular structure data inherent in carbon-13 spectra onto concurrently run NIR spectra of urea-formaldehyde condensates ("NMR-NIR 2D" - signal quantum transfer achieved by me carrying the samples from the NIR to the NMR lab).

Nine samples were taken on the plant every 20 minutes or so from a 20 tonne batch of urea-formaldehyde resin undergoing condensation to a final specification viscosity. NIR was being used ostensibly to monitor only viscosity. The nine quenched samples were transported to our lab and NIR and carbon-13 spectra acquired within a six hour period.

A representative carbon-13 spectrum is shown (see figure over page). Ten distinct methylene carbon types are resolved, including important species such as free formaldehyde (peak 3, as the hydrate, OHCH_2OH) and methylene bridges between two $\text{N,N}'$ -substituted urea nitrogens (peak 8, $\text{NR}-\text{CH}_2-\text{NR}$), believed to be primarily responsible for the viscosity increase as the reaction proceeds. Chemical shifts and assignments are listed in the Table. Each of the ten methylene integrals, expressed as a fraction of the total integral normalised to 1 in each spectrum, was plotted for the nine samples in the time series. One (peak 1, $\text{NRCH}_2\text{O}(\text{CH}_2\text{O})_n\text{CH}_2\text{X}$) was constant throughout, eight increased or decreased but plateaued at the 4th or 5th point, and only one (peak 8, NRCH_2NR) increased steadily throughout the reaction, in concert with viscosity. The kinetic curve for peak 8 is shown. The y-error (area) seems to be $\pm 0.5\%$.

The nine datum points for each NMR peak were then used as constituent values for the corresponding nine NIR spectra. The NMR fractional values were linearly regressed against each of the 1048 2nm channels constituting the visible-NIR spectrum (400-2500 nm) leading to a "best λ " with reasonable to excellent r in 9 out of the 10 cases (see Table and NIR spectrum), the exception being peak 1. In each of the nine viable correlations the "best λ " could be sensibly allied with a vibrational overtone or combination associated with the NMR-derived molecular fragment (see Table).

Thus, the Plant now can potentially monitor not only viscosity, but the fate of nine relevant molecular fragments, by NIR, giving operators a detailed comparison of urea-formaldehyde chemistry from batch to batch, thanks to "remote on-line NMR!".

Please credit this to Jo Weigold's account.

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Telephone (03) 283 6400 Facsimile (03) 283 6408

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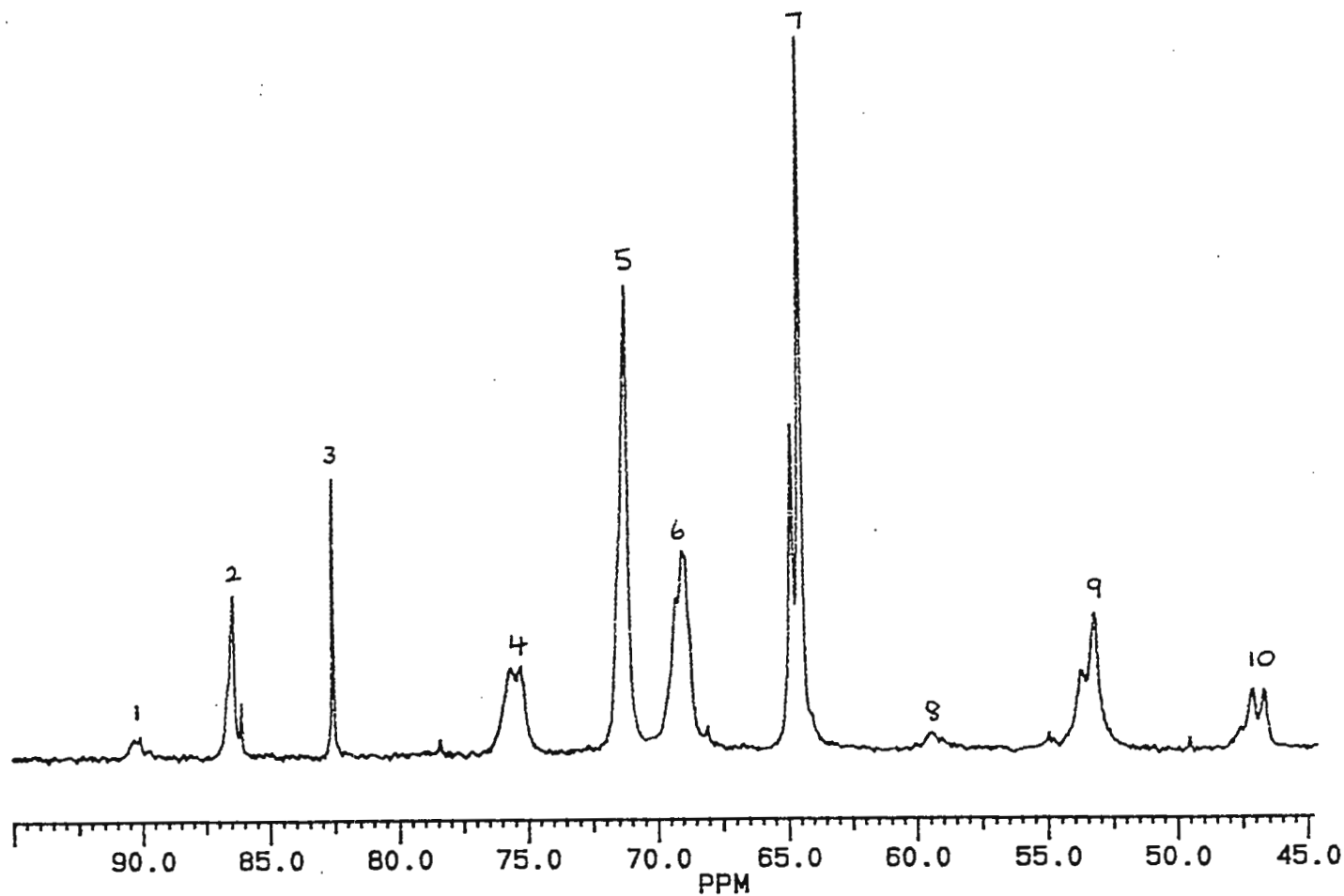
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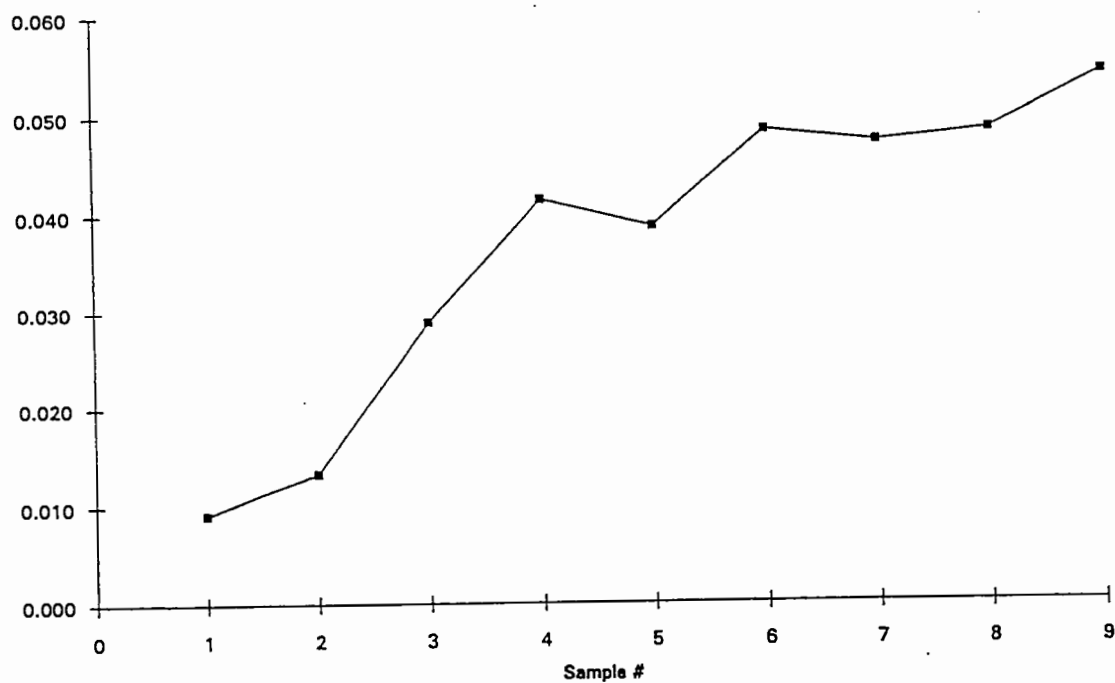
Peak	$\delta^{13}\text{C}(\text{ppm})^a$	Assignment ^b	Best λ (nm)	$ \epsilon $	Possible Vibrational fragments
1	90.0	$\text{NR}-\text{CH}_2\text{O}(\text{CH}_2\text{O})_n\text{CH}_2\text{X}$	(1472) ^c	0.633 ^c	-
2	86.4	$\text{NR}-\text{CH}_2\text{OCH}_2\text{OH}$	1352	0.974	$(\text{OCH}_2)-\text{OH}$ (1st overtone)
3	82.5	HOCH_2OH	2040	0.986	$(\text{OCH}_2)-\text{OH}$ (C-OH combination)
4	75.4	$\text{NR}-\text{CH}_2\text{OCH}_2\text{X}$	2172	0.886	$(\text{N})-\text{CH}_2\text{O}-(\text{R})$ (C/H/O comb.)
5	71.1	$\text{NR}-\text{CH}_2\text{OH}$	1472	0.846	$(\text{N})-\text{CH}_2\text{O}-(\text{R})$ (C/H/O comb.)
6	68.9	$\text{NR}-\text{CH}_2\text{OCH}_2\text{X}$	2284	0.994	$(\text{N})-\text{CH}_2-(\text{O})$ (CH+CH comb.)
7	64.7/64.4	$\text{NR}-\text{CH}_2\text{OH}$	1614	0.996	?
8	59.0	$\text{CONR}-\text{CH}_2-\text{NRCO}$	1476	0.978	$(\text{N})-\text{CH}_2-(\text{O})$ (CH+CH comb., 1st o/tone)
9	53.1	$\text{CONR}-\text{CH}_2-\text{NHCO}$	2390	0.987	$(\text{N})-\text{CH}_2-(\text{N})$ (CH+CH comb.)
10	47.0/46.6	$\text{CONH}-\text{CH}_2-\text{NHCO}$	1968	0.858	CONHR (C=O str., 2nd o/tone)

a) Ref $\text{CH}_3\text{OH} = 49.5$ ppm

b) $\text{R} = \text{CH}_2\text{X}$; $\text{X} = \text{OH}, \text{OCH}_2\text{N}(\text{H},\text{R}), \text{N}(\text{H},\text{R})$ etc. ^{13}C peak multiplicities attributable to variation in these distal substituents.

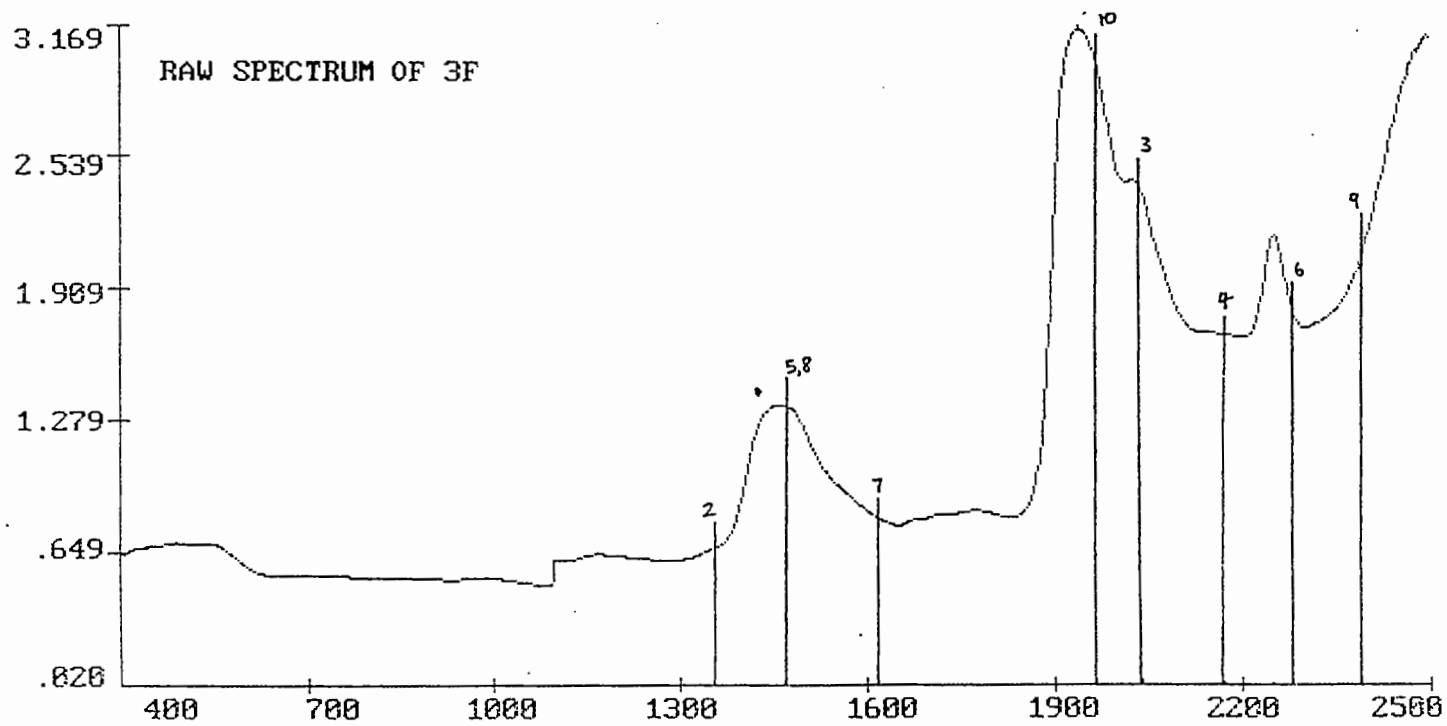
c) Concentration virtually constant.



Fraction $\text{CON}(\text{CH}_2\text{X})\text{CH}_2\text{N}(\text{CH}_2\text{X})\text{CO}$ 

NIR-11

File: UF03NMR Spectrum: 1 Sample: 3F



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B. L. Shapiro
1 July 1996

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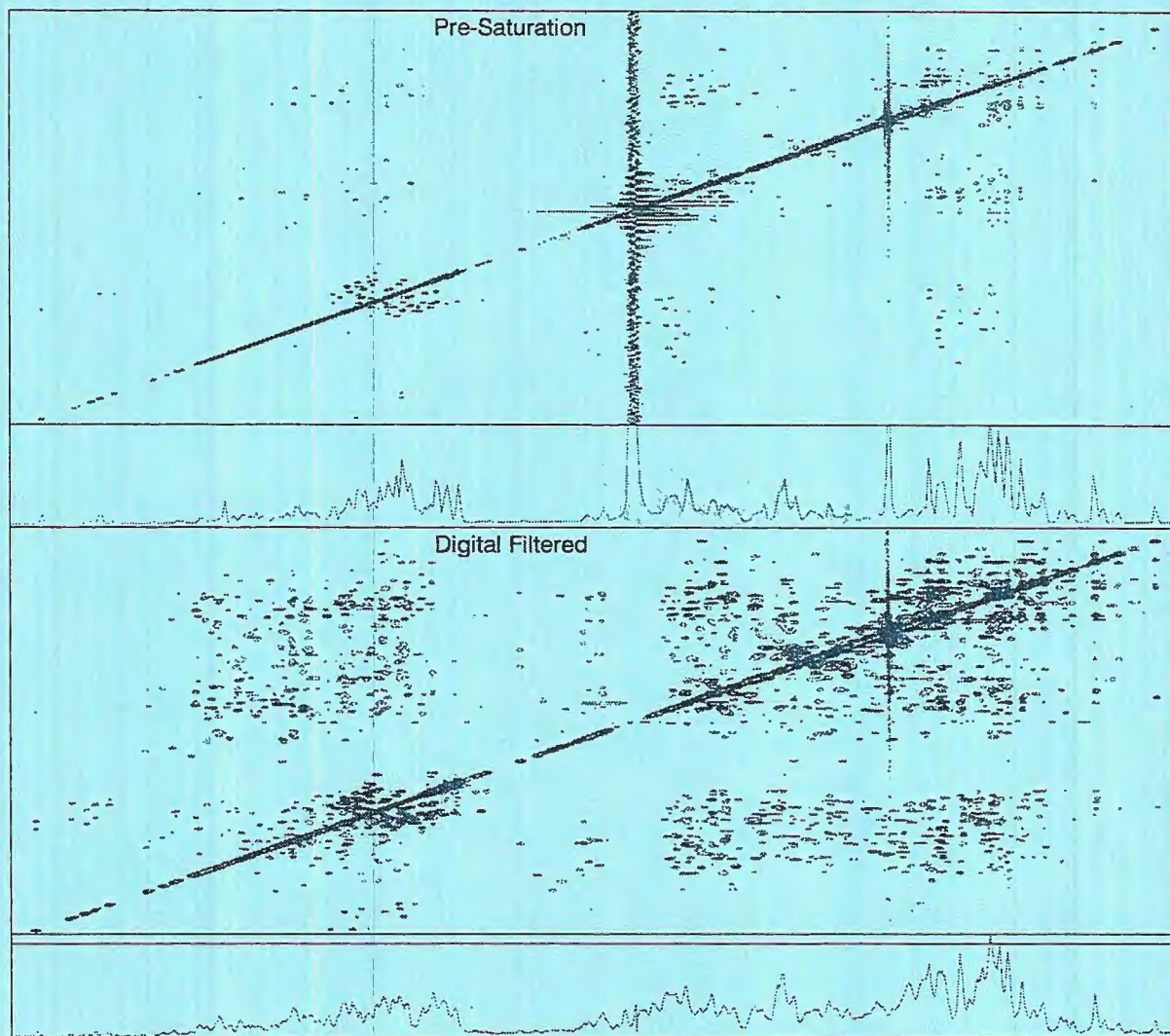


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