

**THE**  
***NMR***  
**NEWSLETTER**

**No. 447**  
**December 1995**

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Type 2							1 Hz resol., OCXO: \$5,850.00
PTS 500	1-500 MHz	optional .1 Hz to 100 KHz	1-20 $\mu$ s	optional	5¼"H×19"W	BCD (std) or GPIO (opt)	\$8,720.00 (1 Hz resol., OCXO freq. std.)
PTS 620	1-620 MHz	optional .1 Hz to 100 KHz	1-20 $\mu$ s	optional	5¼"H×19"W	BCD (std) or GPIO (opt)	\$9,625.00 (1 Hz resol., OCXO freq. std.)
PTS 1000	0.1-1000 MHz	optional .1 Hz to 100 KHz	5-10 $\mu$ s	optional	5¼"H×19"W	BCD (std) or GPIO (opt)	\$11,830.00 (1 Hz resol., OCXO freq. std.)
PTS 3200	1-3200 MHz	1 Hz	1-20 $\mu$ s	optional	5¼"H×19"W	BCD (std) or GPIO (opt)	\$14,850.00 (1 Hz resol., OCXO freq. std.)
PTS x10	user specified 10 MHz decade	1 Hz	1-5 $\mu$ s	standard	3½"H×19"W	BCD (std) or GPIO (opt)	\$3,000.00 (1 Hz resol., OCXO freq. std.)
PTS D310	two channels .1-310 MHz	.1 Hz	1-20 $\mu$ s	standard	5¼"H×19"W	BCD (std) or GPIO (opt)	\$8,560.00 (.1 Hz resol., OCXO freq. std.)
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1 Switching Time is dependent on digit (decade) switched; see detailed instrument specifications.

2 For applicable digits, see detailed instrument specifications.

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#### FORTHCOMING NMR MEETINGS

**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.

**Society of Magnetic Resonance, Fourth Scientific Meeting and Exhibition**, New York, NY, **April 27 - May 3, 1996**; Contact: SMR Office, 2118 Milvia St., Suite 201, Berkeley, CA 94704; (510) 841-1899; Fax: (541) 841-2340. E-mail: info@smr.org. Future meetings: 1997, April 12-18, Vancouver, BC, Canada; 1998, April 18-24, Sydney, Australia; 1999, Philadelphia, PA; 2000, Denver, CO.

**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**; Contact: ENC, 1201 Don Diego Avenue, Santa Fe, NM 87501; (505) 989-4573; Fax: (505) 989-1073.

Additional listings of meetings, etc., are invited.

# Mobil Research and Development Corporation

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October 10, 1995  
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The NMR Newsletter  
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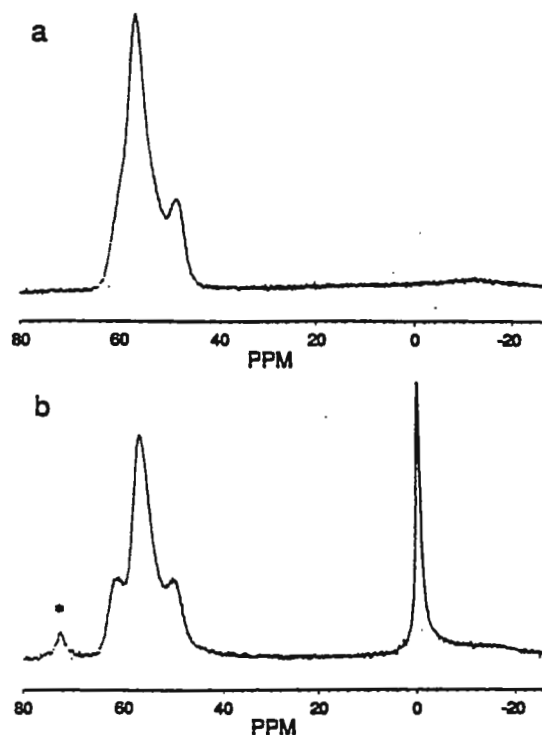
## *<sup>27</sup>Al MAS NMR AT 17.6 T*

Theory tells us and experience has shown that high-resolution solid-state NMR spectra of many inorganic materials can be obtained at high magnetic fields on conventional high-resolution NMR spectrometers by employing magic angle spinning alone. In fact, this recognition of the magnetic field dependence on the NMR spectra of important nuclei such as <sup>27</sup>Al was probably the most important factor in the purchase of our Bruker AM-500 NMR (11.7T) spectrometer over a decade ago. At that time the highest available stable magnetic field available for use with commercial NMR instruments was 11.7T (500MHz for <sup>1</sup>H). Since that time significant technological advances resulted in the commercialization of systems with 14.1T magnets (600MHz for <sup>1</sup>H), and more recently with 17.6T magnets (750MHz for <sup>1</sup>H). As part of our continuing effort to evaluate the potential impact of new NMR technology on our characterization programs, Dr. Stefan Steuernagel of Bruker Instruments graciously agreed to record <sup>27</sup>Al MAS NMR data on a representative group of samples on a 17.6T NMR instrument. In this contribution an example of these very high field NMR data are presented.

The 17.6T <sup>27</sup>Al MAS NMR spectra of zeolite MCM-22 before and after calcination to remove the organic directing agent are shown in the accompanying figure. These data show that there are at least three tetrahedral (T<sub>d</sub>) resonances in the spectrum of MCM-22. The <sup>27</sup>Al MAS NMR spectra of framework aluminosilicates usually consist of one resonance in the 54-68 ppm region of the spectrum that reflects the average environment of an Al atom in the tetrahedral framework. The observation of more than one T<sub>d</sub> resonance for a zeolite is rare. Zeolites omega and ZSM-18 are the only reported cases of zeolites that exhibit two T<sub>d</sub> resonances. Multifield MAS and DOR NMR experiments show that there are no significant second order quadrupolar interactions present and that the multiple T<sub>d</sub> Al resonances are due to distinct T<sub>d</sub> sites in the framework of MCM-22. A discussion of how these unique spectral features relate to the proposed structure of MCM-22 and the nature of the calcination induced changes will be published in the near future.

Best regards,

*Gordon Kennedy*  
Gordon J. Kennedy



<sup>27</sup>Al MAS NMR spectra of MCM-22 before (a) and after (b) calcination. \* denotes sideband from O<sub>h</sub> peak at 0.0 ppm.



*Re*  
The NMR evolution advances...



# XWIN-NMR™ Software:

On-line help is just a click away!



Bruker's new NMR software package, *XWIN-NMR™*, comes complete with an **on-line** manual. *XWIN-NMR™* uses the Frame-Viewer utility to display the manual. Additionally, the table of contents and a keyword index for the manual are organized as hypertext for fast display of a desired item. Now "help" is just a click away!





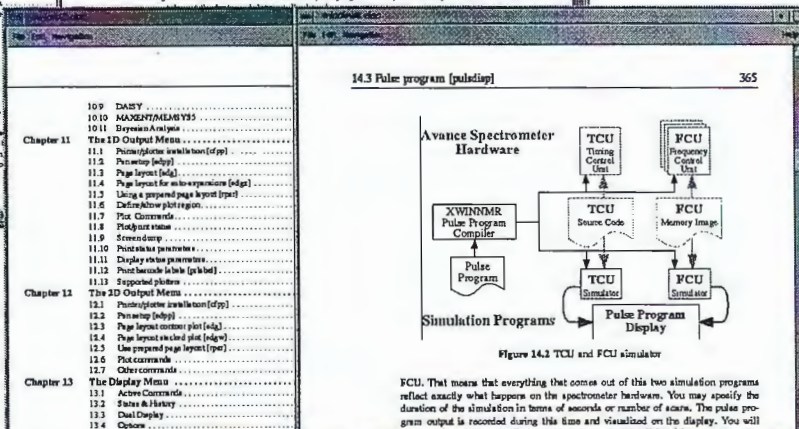
# ...The NMR evolution advances

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## Chapter 3 2D Data Processing Tutorial

This chapter presents 2D data processing based on the data set *exm02d*. The data set is stored in the directory *data\exm02d\exm02d*. In order to have full access permissions to it, you should have a user profile installed on your system, and be logged in as *guest*. Start XWIN-NMR by typing *exm02d* at the prompt. The *-r* option ensures that everything is cleaned up before starting the program, even if the last session was terminated by some problem. The program will start without displaying a data set. Instead, the XWIN-NMR logo will be shown in the data area. In later sections, you can start XWIN-NMR without specifying the *-r* option, and you will immedi-

Here are some pages from the on-line manual, showing the Table of Contents that was used to find the page.



FCU. That means that everything that comes out of this two simulation programs reflect exactly what happens on the spectrometer hardware. You may specify the duration of the simulation in terms of seconds or number of scans. The pulse program output is recorded during this time and visualized on the display. You will find some fine details of a timing sequence, some of immediate delay, within pulse

Top: Shows the tutorial help available with XWIN-NMR™ for 2D processing.

Middle: Explains functionality of the pulse program display software.

Bottom: Explains the fundamentals of writing and using AU programs.

For more information on XWIN-NMR™ software and our other new products, contact your local sales representative.

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## Sandia National Laboratories

Albuquerque, New Mexico 87185-1407

LOCKHEED MARTIN

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

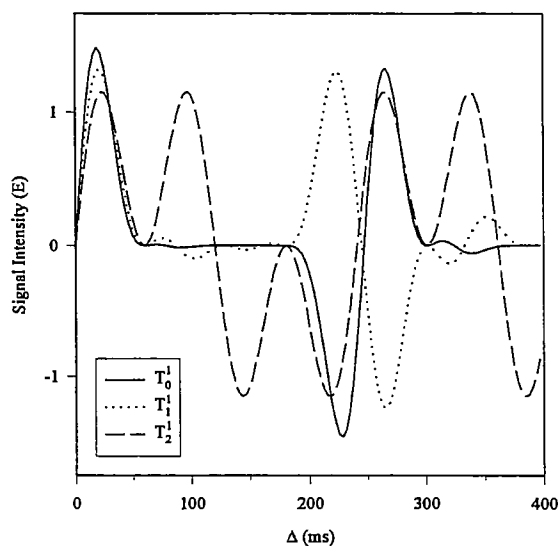
25 October, 1995  
(received 10/30/95)

### Revisiting the INEPT Pulse Sequence and $^{29}\text{Si}$ NMR Identification of Initial Condensation Products in Organoalkoxysilanes<sup>1</sup>

Dear Barry,

A continuing research area at Sandia National Labs is the development and characterization of new materials produced from organically modified alkoxy silanes. Using  $^{29}\text{Si}$  NMR the kinetics of polymerization in these sol-gel materials can be investigated. In recent studies of methyltrimethoxysilane (MTMS) sol-gel systems the assignment of  $^{29}\text{Si}$  NMR resonances for the hydrolysis and initial condensation products were complicated by an unusual small spectral dispersion ( $\sim 0.5$  ppm). This is in contrast to previous studies of tetraalkoxysilane systems where  $^{29}\text{Si}$  resonances assignments are typically based on a substantial downfield shift with increasing hydroxyl number. To aid in assignments for the MTMS systems, the INEPT sequence has been utilized to distinguish various silicon environments, correcting assignments based solely on chemical shift arguments. The INEPT sequence not only proves useful for spectral editing but affords polarization transfer to silicon, a nucleus plagued by long relaxation times and a negative NOE. For MTMS and the resulting hydrolysis and condensation products there are between 12 and 3 protons coupled to each silicon. In addition, there are two different heteronuclear couplings,  $J(\text{Si},\text{H}) = 8.3$  Hz and  $J(\text{Si},\text{H}) = 3.9$  Hz for the methyl and methoxy groups respectively, giving rise to a complicated response for the polarization

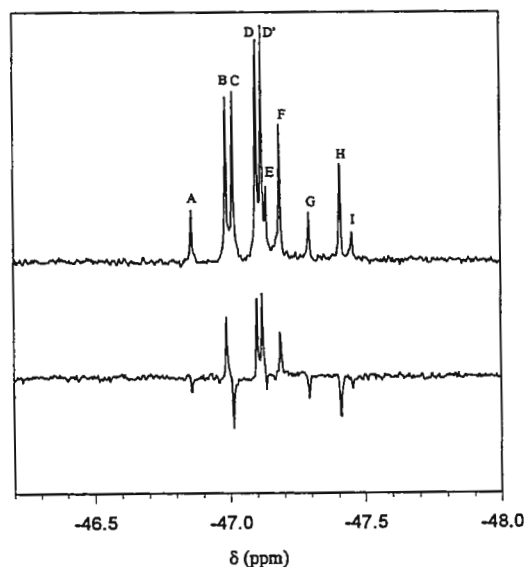
transfer. The protons for attached hydroxyl groups are in rapid exchange and reveal no coupling to the silicon, allowing the degree of hydrolysis to be investigated using the INEPT sequence. For example, the response of these  $\text{IS}_3\text{S}_m$  ( $m = 0, 3, 6$ ) spin systems (as expected in the condensation products) during an INEPT experiment as a function of the refocusing delay  $\Delta$  is shown in Fig. 1. For MTMS the silicon types are denoted as  $\text{T}_m^n$  where  $m$  represents the number of hydroxyl groups and  $n$  the number of siloxane bonds in the unit of  $\text{CH}_3\text{Si}(\text{O}_{0.5})_n(\text{OH})_m(\text{OCH}_3)_{3-m-n}$ .



**Figure 1.** Theoretical signal intensity for the initial condensation products of MTMS following the INEPT pulse sequence as a function of the refocusing time  $\Delta$ , for  $J(\text{Si},\text{H}) = 8.3$  Hz and  $J(\text{Si},\text{H}) = 3.9$  Hz. The lines (—),  $\text{T}_0^1$ , (.....)  $\text{T}_1^1$  and (---)  $\text{T}_2^1$  correspond to  $\text{IS}_3\text{S}_6$ ,  $\text{IS}_3\text{S}_3$ ,  $\text{IS}_3\text{S}_0$  spin systems, respectively.

<sup>1</sup> This work is supported by the US Department of Energy under Contract DE-AC04-94AL85000.

For the initial condensation products of MTMS there are three different silicon types present,  $T_0^I$ ,  $T_1^I$  and  $T_2^I$ . With nearest neighbor effects 9 distinct resonances are expected for the  $T^I$ - $T^I$  condensation products. Inspection of Fig. 1 reveal the  $T_1^I$  silicon species is easily distinguished from the  $T_0^I$  and  $T_2^I$  species using the INEPT experiment with a refocusing delay of  $\Delta = 220$  ms. The  $T_2^I$  resonance can be distinguished by noting that this is the only signal at  $\Delta = 90$  and 150 ms. By combining these experiments identification of the various silicon types becomes possible. As an example consider the  $T^I$  condensation products for a 2.24 M solution of MTMS in MeOH, with a 1.5 molar equivalent of  $H_2O$  ( $R=1.5$ ) as shown in Fig. 2. These INEPT experiments were performed at 9.4 Tesla using a 5 mm broadband probe. Only the signals G,H,I are present at  $\Delta = 90$  and 150 ms (not shown) allowing the assignment of these as  $T_2^I$  resonances. Experiments performed at  $\Delta = 20$  and 220 ms easily allows the assignment of  $T_1^I$  species (Fig. 2). Using these results, plus the correlation of signal intensities into pairs, all of the condensation products are easily identified. It is interesting to note the chiral  $T_1^I$  silicons give rise to two distinct resonances in the condensed species  $T_1^I - T_1^I$ , denoted as resonances D and D'. These initial results are very encouraging, and provide an additional tool for the investigation of silicon containing systems.



**Figure 2.**  $^{29}\text{Si}$  NMR INEPT spectra of the  $T^I$  condensation products after 60 minutes for a 2.24 M MTMS/MeOH solution with  $R=1.5$   $H_2O$  added. Spectra are for refocusing delays of  $\Delta = 20$  ms (top) and 220 ms (bottom). The resonances can be assigned to;

$A = \underline{T_0^I} - \underline{T_0^I}$ ,  $B = \underline{T_1^I} - \underline{T_0^I}$ ,  $C = \underline{T_1^I} - \underline{T_0^I}$ ,  $D$  and  $D' = \underline{T_1^I} - \underline{T_1^I}$ ,  $E = \underline{T_0^I} - \underline{T_2^I}$ ,  $F = \underline{T_1^I} - \underline{T_2^I}$ ,  $G = \underline{T_0^I} - \underline{T_2^I}$ ,  $H = \underline{T_1^I} - \underline{T_2^I}$ , and  $I = \underline{T_2^I} - \underline{T_2^I}$ , where the underscore designates the silicon of interest.

Cheers,

*Todd Alam*

Todd Alam

*Roger Assink*

Roger Assink





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November 18, 1995  
(received 11/24/95)

### Molecular Dynamics of Sodium n-Alkylbenzenesulfonate Adsorbed onto Alumina

Dear Barry:

We have been utilizing deuterium NMR to investigate the molecular dynamics of surfactants adsorbed onto alumina particles in aqueous solution. A prototype surfactant, isomerically pure sodium n-decylbenzenesulfonate-d<sub>4</sub>, was synthesized in our laboratory, and g-alumina (D=10 nm, BET specific surface area=79 m<sup>2</sup>/g) was purchased from Johnson Matthey Electronics Co. Ltd. Four surfactant-adsorbed alumina samples with different coverages were prepared at pH 4, 40 °C and transferred to 1.9 mm  $\phi$  8 mm glass tubes after sedimentation. A Doty wide-line probe was used with the quadrupolar echo sequence at 61.395 MHz (400 MHz) on a Varian VXR/S-200/400. All the spectra were referenced to D<sub>2</sub>O-H<sub>2</sub>O (1:1 volume)

The adsorption isotherm of the surfactant onto oppositely charged alumina particles is shown in the log equilibrium concentration — log adsorption amount plots. The four discrete regions labelled as Region 1, 2, 3, and 4 are typical characteristics of ionic surfactant adsorption onto mineral oxides, which represents different adsorption states of surfactant molecules on the surfaces.

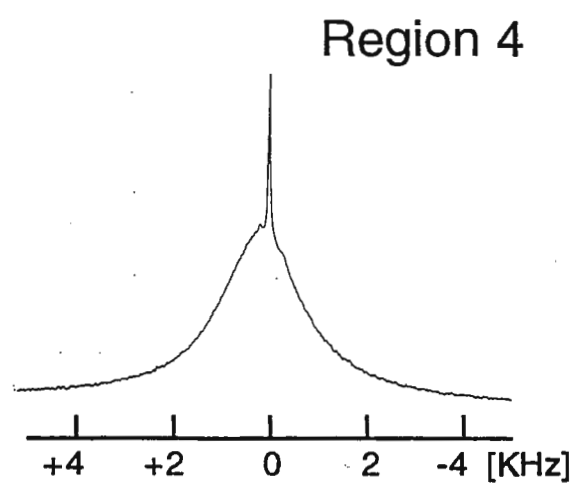
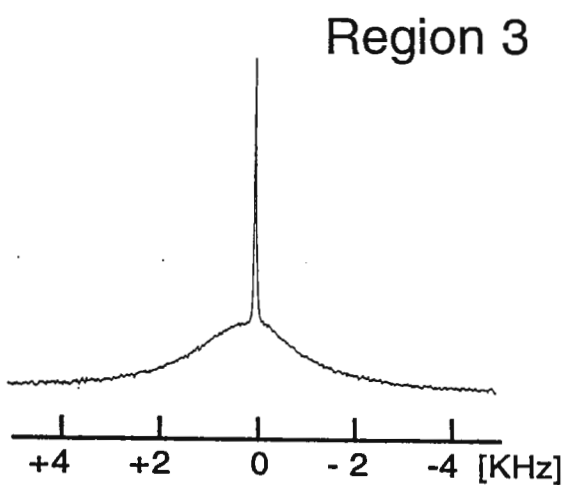
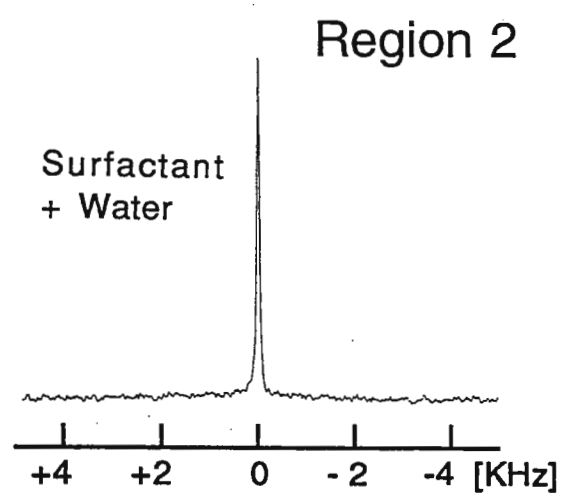
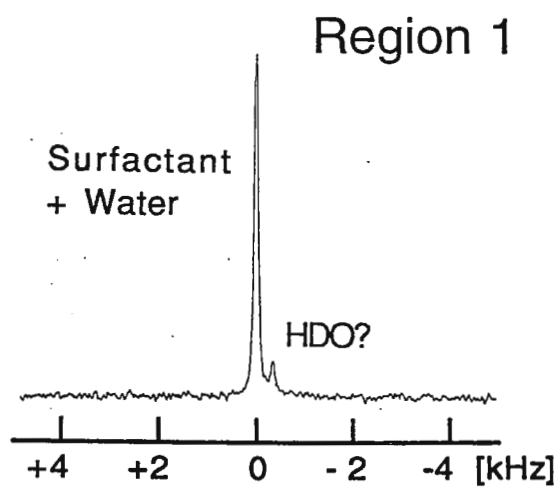
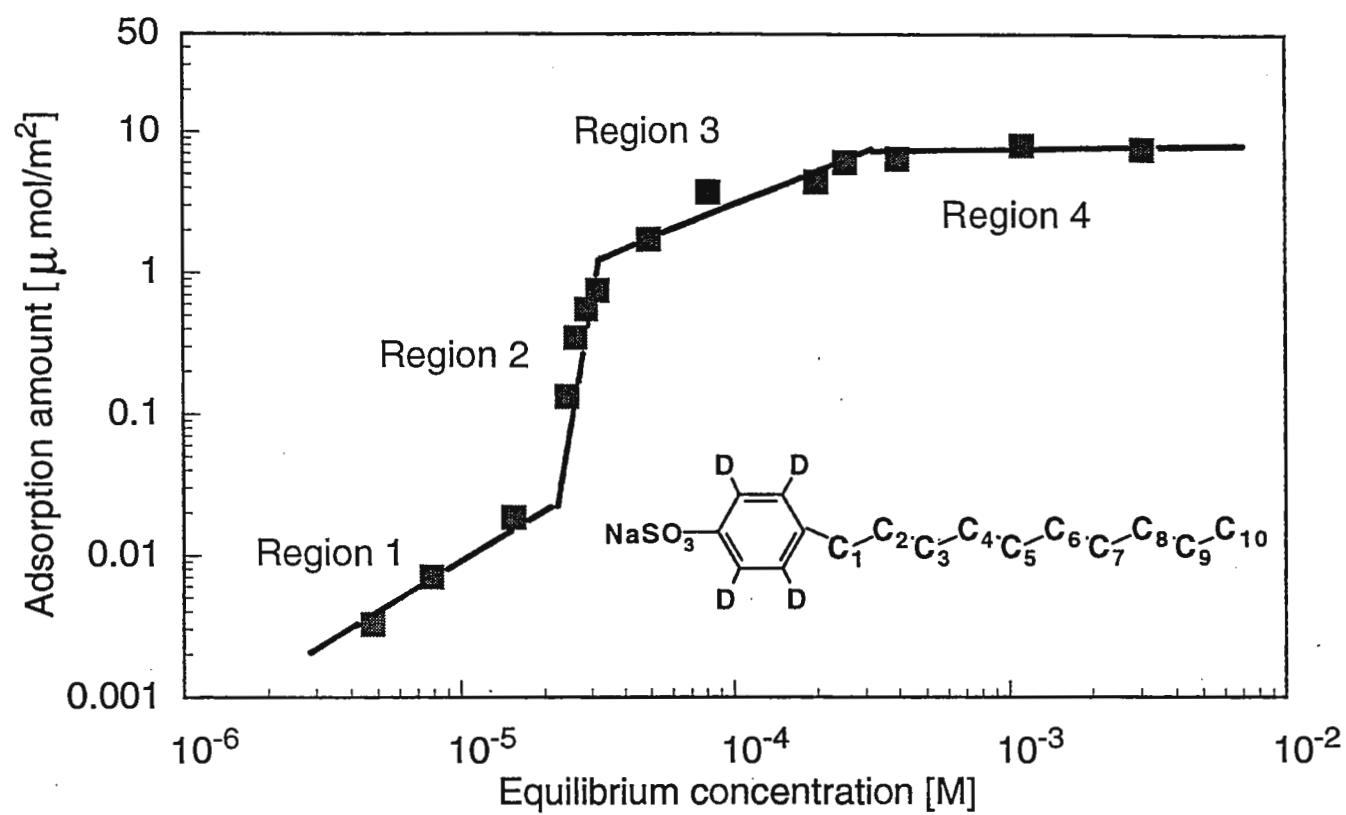
Based on the isotherm, deuterium NMR spectra in each region were obtained to elucidate the adsorption mechanisms. Region 1 and 2 gave relatively narrow single resonances reflecting the rapid molecular motion of the surfactant on the pertinent NMR time scale. This is attributable to fast exchange between the surfactant molecules on the surface and the monomers in the solution phase. Another possible reason for this is that the molecules may diffuse around a particle during one pulse sequence since the lateral diffusion of surfactant molecules on the surface may be quite fast. (The small resonance on Region 1 (-0.36 kHz) is tentatively identified to be that from a small number of water molecules in pores on the alumina surface or interstices between the agglomerates) In these dilute regimes, the surfactants probably form monolayers in which the surfactant molecules are rapidly reorienting with the polar groups more or less close to the alumina surface. Quantitative analysis confirms that the narrow resonances is significantly broadened at the end of Region 2, and regains the original intensity at the onset of Region 4. The Region 3 spectrum acquires another noticeably broad resonance overlapping the narrow resonances from the surfactant and HDO. This phenomenon implies the possible formation of an outer layer (i.e. bilayer formation) where the alkyl chains orient inwards and the polar groups outwards. In Region 4 where saturation adsorption is established, further growth of broadened portion is observable correlating with an increase in total amount adsorbed.

Best regards,

K. Nagashima

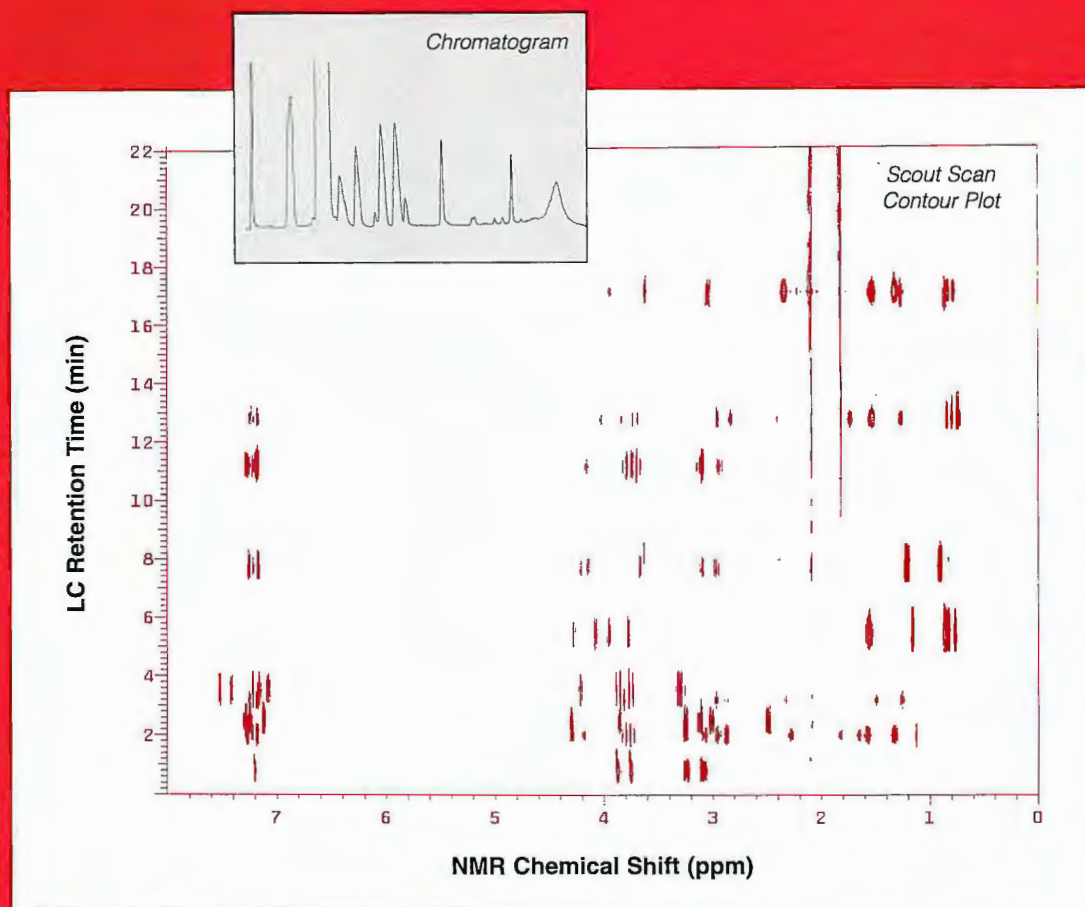
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Frank D. Blum  
Curators' Professor of Chemistry  
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# LC-NMR: Separations and Structures



*On-flow 500 MHz  $^1\text{H}$  LC-NMR data of a peptide mixture using Scout Scan and WET solvent suppression during an LC gradient. The sample was a 100  $\mu\text{L}$  injection of  $\sim 1\text{ mg/mL}$  peptides separated using a  $\text{CH}_3\text{CN}:\text{D}_2\text{O}$  solvent gradient (5%  $\text{CH}_3\text{CN}$  to 50% in 22 min).*

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 Berkeley, California 94720-1462  
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NMR with electronically conductive samples

Dear Barry:

November 16, 1995

(received 11/21/95)

One exciting area of research currently being pursued in our lab is the study of adsorbates on electrocatalyst surfaces. A necessary constraint in electrochemical systems is a sufficiently high electrical conductivity to prevent ohmic losses. It has been shown that such a constraint results in coupling of the sample to the coil of an NMR probe<sup>1-3</sup>. Since it is our desire to perform *in-situ* experiments, we have been forced to develop a better understanding of the physical interactions of a solenoid and an electronically conducting sample.

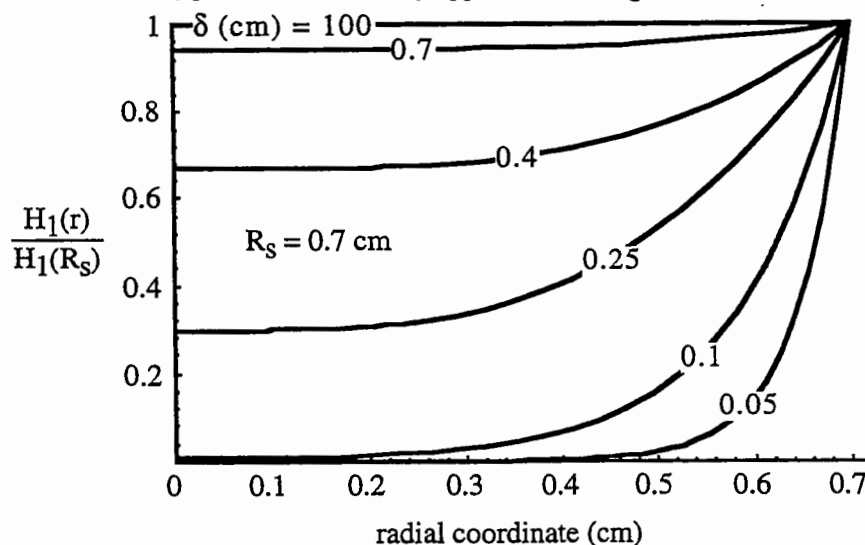
In an ideal NMR experiment the *rf* field will be constant everywhere in the sample. As conduction electrons become mobile, however, a loss mechanism is provided via Ohm's law; the applied field is then expected to vary spatially within the sample. An analytical expression for the field distribution can be derived for the case of a 1-D cylinder using Maxwell's equations. Combining Faraday's and Ampere's laws in MKSA units, and separating spatial and time variables, results in a general second-order differential equation for the *rf* field  $H_1$ :

$$\nabla^2 H_1 = (i\omega\mu\sigma - \mu\epsilon\omega^2)H_1, \quad (1)$$

where  $\sigma$  is the conductivity,  $\mu$  and  $\epsilon$  are the sample permeability and permittivity, respectively. For a sample of appreciable conductivity we can neglect the displacement currents described by the real term on the right side of equation (1), leaving a special form of Bessel's equation. We apply boundary conditions that  $H_1(0)$  is finite and  $H_1(R_s)$  is simply equal to the field in an equivalent coil in the absence of the conductor. The solution for the applied  $H_1$  field in the sample is given by

$$\frac{H_1(r)}{H_1(R_s)} = \frac{J_0\left(\frac{\sqrt{2}}{\delta} r e^{-i\frac{\pi}{4}}\right)}{J_0\left(\frac{\sqrt{2}}{\delta} R_s e^{-i\frac{\pi}{4}}\right)} \quad (2)$$

where  $J_0$  is a Bessel function of order zero, and  $\delta = \left(\frac{2}{\omega\mu\sigma}\right)^{\frac{1}{2}}$  is a characteristic length termed the skin depth. The effect of sample conductivity on *rf* penetration is readily apparent in the figure shown below.



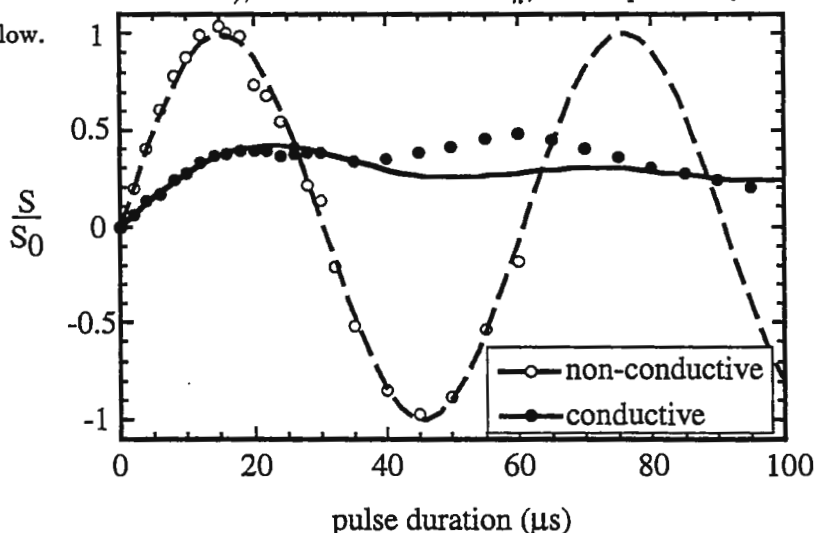
One would expect this distribution of applied fields to affect the nutation behavior of a group of spins distributed throughout a conducting matrix. In order to confirm this we compared results of nutation experiments with conductive and non-conductive samples. Measurements were performed on two samples in our home built 270 MHz spectrometer equipped with a  $^{13}\text{C}$  probe designed to minimize coupling of the sample to the coil. A conductive sample was prepared by soaking  $1\text{-}^{13}\text{C}$ -enriched acetic acid into a 14mm diameter porous graphite plug of known conductivity. The other sample consisted of only  $^{13}\text{C}$ -enriched methanol. Transmitter power was set for  $15\mu\text{s}$   $\frac{\pi}{2}$ -pulses for the methanol sample and amplitudes of the resulting Lorentzian lines from both samples were recorded as a function of pulse length.

In order to fit the data, we recognize that the contribution from an individual nuclear spin in the sample following application of an  $rf$  pulse of magnitude  $H_1$  is related to the magnitude of the component of its magnetic moment in the  $x$ - $y$  plane (with the static  $B_0$  field aligned along the  $z$  axis). A summation over the entire sample yields an expression for the detected signal,  $S$ :

$$\frac{S}{S_0} = \frac{\int_0^{R_s} \sin(\gamma H_1(r) t_p) dV}{\int_0^{R_s} dV} = \frac{2}{R_s^2} \int_0^{R_s} r \sin\left(\omega_n^* t_p \frac{H_1(r)}{H_1(R_s)}\right) dr \quad (3)$$

where  $\omega_n^* = \gamma H_1(R_s) = \frac{2\pi}{t_p(\frac{\pi}{2})}$  and  $\frac{H_1(r)}{H_1(R_s)}$  is given by (2). Numerical techniques were used to fit the data using  $\delta = 0.09\text{cm}$  (based on a measured value of  $\sigma$ ), calculated values for  $\omega_n^*$ , and amplitude  $S_0$  as an adjustable parameter.

The result is shown below.



This simple calculation shows quantitatively how the electrical conductivity of an NMR sample couples inductively to the coil, resulting in attenuation of the applied  $rf$  as the field penetrates the conductor. This spatial variation of the field can cause severe distortion of the nutation function, rendering precise manipulation of sample magnetization difficult, or perhaps impossible. For applications requiring high conductivity, the situation can be improved by investigating methods of sample preparation that minimize the length scales over which eddy currents can propagate<sup>4</sup>.

Jeffrey A. Reimer

Best regards to all,

Mark S. Yahnke

p.s. Please contribute this to the Raychem account.

1. Bloembergen, N. *J. Appl. Phys.* **23** (1952) 1383.
2. Hoult, D. I. and P. C. Lauterbur. *J. Mag. Res.* **34** (1979) 425.
3. Gadian, D. G. and F. N. H. Robinson. *J. Mag. Res.* **34** (1979) 449.
4. Kugel, H. *J. Mag. Res.* **91** (1991) 179.



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November 17, 1995 (received 11/20/95)

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### Decoupler Problem or New Decoupler Test?

Dear Barry:

As Heinz Sterk pointed out in the November newsletter, "Sometimes ... things turn out to be much more complicated than expected at the first glance.". We would like to report some of the problems and solutions encountered recently while setting up a simple experiment.

The simple experiment is a cross-relaxation or magnetization transfer experiment. (J.Y. Wu & T.M. Eads; *Carbohydrate Polymers* **20** (1993) 51-60 and J.Y. Wu; R.G. Bryant & T.M. Eads; *J. Agric. Food Chem.* **40** (1992) 449-455) for detection of solid-like components in gelatinized starch. The experiment is simply a series of presaturation experiments with the presaturation offset frequency varied between -50kHz and +50kHz. The amplitude of the liquid spectra are recorded to give the cross-relaxation spectrum. The cross-relaxation spectrum contains information about the solid component, specifically mobility information.

We expected the experiment to be somewhat demanding of our equipment (GN300s) due to the use of fairly high power presaturation pulses of 4-5 seconds. The presaturation pulses to give proton precession frequencies of 300-500Hz are greater than 1 watt of rf power. Possibly a problem for the standard probe and certain components. Larger resistors were installed in the rf module and care was taken to be sure to have adequate air flow through the probe body.

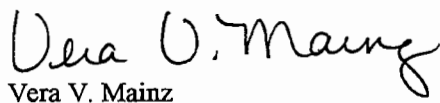
As a test of the experimental setup we looked at the cross-relaxation spectra of water. We had obvious problems when the spectra showed decreased intensities at offset frequencies greater than  $\pm 40$ kHz with a severe dip from +27.5kHz to +37.5kHz. Expected behavior was observed near zero offset; with near zero intensity for offsets within  $\pm 500$ Hz of resonance and constant intensity for offset frequencies between +40kHz and +5kHz and between -5kHz and -20kHz. (see figure) We first questioned whether this was a probe or console problem. Rerunning the experiment with a Chemagnetics MAS probe in place of the liquids probe ruled out a probe related problem due to the fairly high power. Spectra were the same for the two probes.

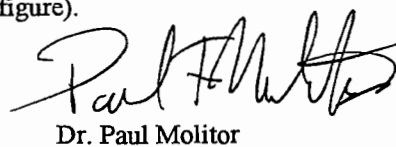
A decrease in power would have been the obvious malfunction. However, the scope trace of the decoupler pulse showed constant rf voltage at all frequencies. Also, a drop in power of the saturation pulse would not be consistent with the spectral evidence. The problem became apparent on closer inspection of the decoupler rf output. As the offset frequency increased the signal became noisy. The transmitter noise having a significant component at the water resonance frequency. This noise component partially saturated the water resonance.

Like many problems the solution was more difficult than characterization. After swapping nearly every logic chip in the rf generation unit we were exactly where we started. We noticed that the scope trace improved with tapping on the chips (last resort type troubleshooting). This led to the speculation that the high-speed chips were not working properly due to poor electrical contact with the sockets. The signal quality improved as each chip was removed and the terminals cleaned. Eventually the unit produced a clean signal at all frequencies.

This was verified by running the cross-relaxation spectrum for water (see figure). Having a correct water spectrum we acquired the now reasonable cross-relaxation spectrum of a starch gel (see figure).

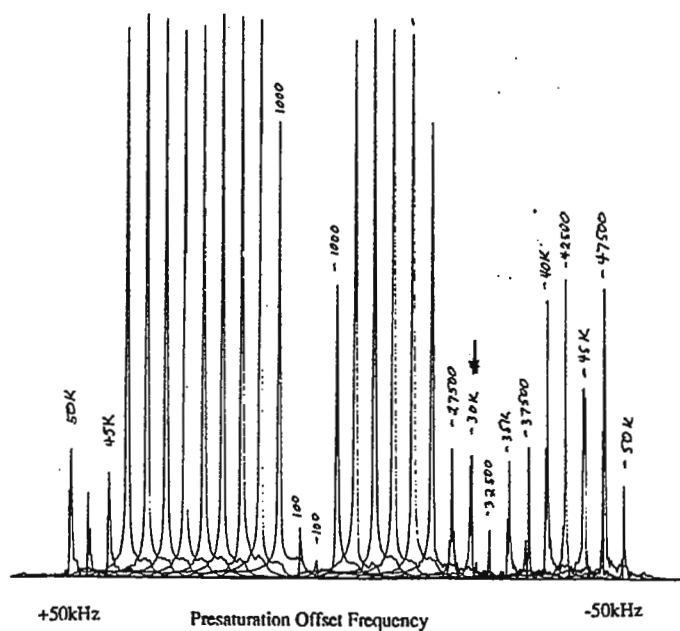
Sincerely,

  
Vera V. Mainz

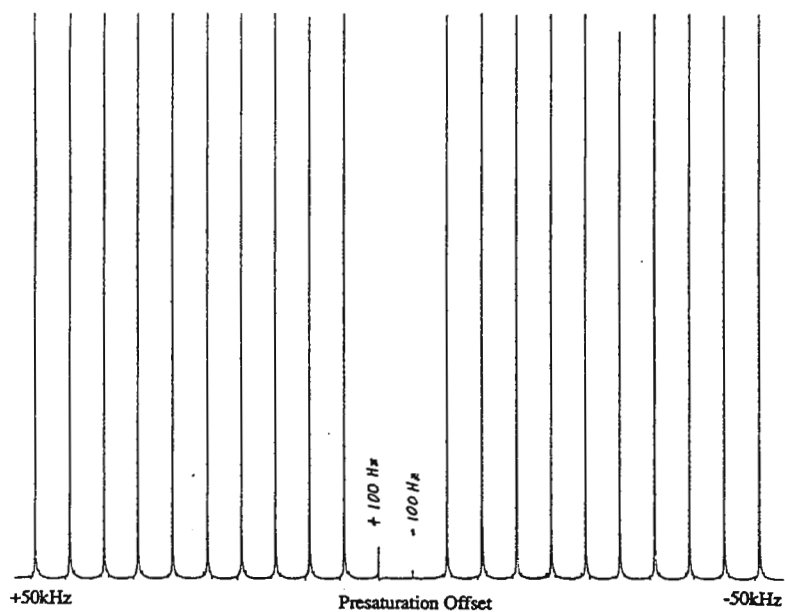
  
Dr. Paul Molitor

Molecular Spectroscopy Laboratory

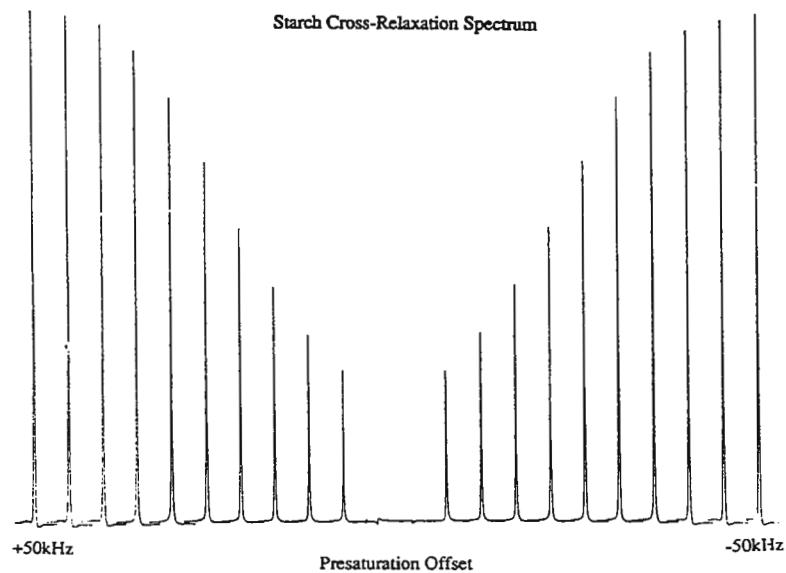




### H<sub>2</sub>O Cross-Relaxation Spectrum



### Starch Cross-Relaxation Spectrum



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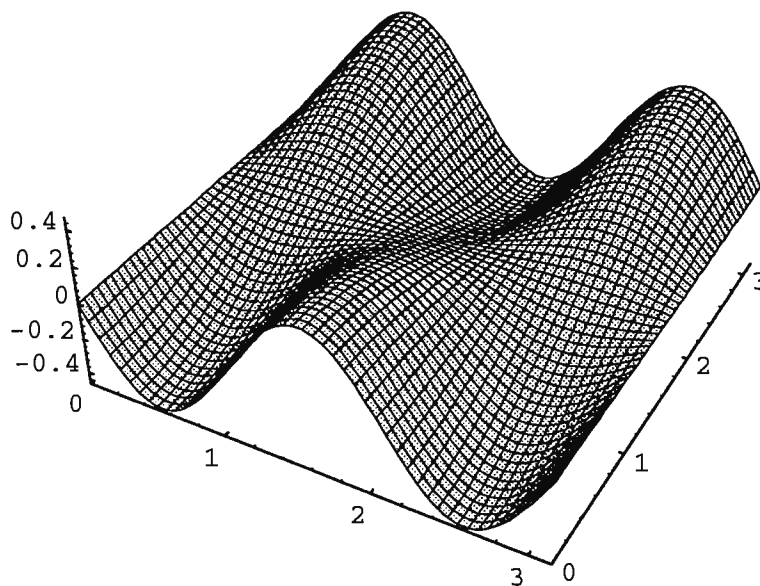
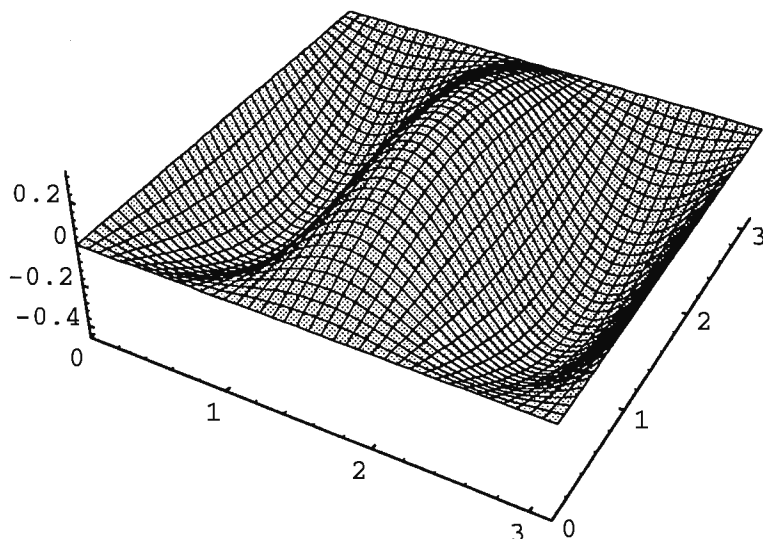
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## DEUTERATED NMR SOLVENTS - HANDY REFERENCE DATA

Compound Mol. Wt.	$d_4^{20}$	m.p. <sup>a</sup>	b.p. <sup>a</sup>	$\delta_H$ (mult)	$J_{HD}$	$\delta_C$ (mult)	$J_{CD}(CF)$
Acetic Acid-d <sub>4</sub> 64.078	1.12	17	118	11.53(1) 2.03(5)	2	178.4(br) 20.0(7)	20
Acetone-d <sub>6</sub> 58.08	0.87	-94	57	2.04(5)	2.2	206.0(13) 29.8(7)	0.9 20
Acetonitrile-d <sub>3</sub> 44.071	0.84	-45	82	1.93(5)	2.5	118.2(br) 1.3(7)	21
Benzene-d <sub>6</sub> 78.11	0.95	5	80	7.15(br)		128.0(3)	24
Chloroform-d 120.384	1.50	-64	62	7.24(1)		77.0(3)	32
Cyclohexane-d <sub>12</sub> 96.236	0.89	6	81	1.38(br)		26.4(5)	19
Deuterium Oxide 20.028	1.11	3.8	101.4	4.63(DSS) 4.67(TSP)			
1,2-Dichloroethane-d <sub>4</sub> 102.985	1.25	-40	84	3.72(br)		43.6(5)	23.5
Diethyl-d <sub>10</sub> Ether 84.185	0.82	-116	35	3.34(m) 1.07(m)		65.3(5) 14.5(7)	21 19
Diglyme-d <sub>14</sub> 148.263	0.95	-68	162	3.49(br) 3.40(br) 3.22(5)	1.5	70.7(5) 70.0(5) 57.7(7)	21 21 21
Dimethylformamide-d <sub>7</sub> 80.138	1.04	-61	153	8.01(br) 2.91(5) 2.74(5)	2 2	162.7(3) 35.2(7) 30.1(7)	30 21 21
Dimethyl-d <sub>6</sub> Sulfoxide 84.170	1.18	18	189	2.49(5)	1.7	39.5(7)	21
p-Dioxane-d <sub>8</sub> 96.156	1.13	12	101	3.53(m)		66.5(5)	22
Ethyl Alcohol-d <sub>6</sub> (anh) 52.106	0.91	<-130	79	5.19(1) 3.55(br) 1.11(m)		56.8(5) 17.2(7)	22 19
Glyme-d <sub>10</sub> 100.184	0.86	-58	83	3.40(m) 3.22(5)	1.6	71.7(5) 57.8(7)	21 21
Hexafluoroacetone Deuterate <sup>b</sup> 198.067	1.71	21		5.26(1)		122.5(4) 92.9(7)	(287) (34.5)
HPMT-d <sub>18</sub> 197.314	1.14	7	106(11)	2.53(2 x 5)	2(9.5)	35.8(7)	21
Methyl Alcohol-d <sub>4</sub> 36.067	0.89	-98	65	4.78(1) 3.30(5)	1.7	49.0(7)	21.5
Methylene Chloride-d <sub>2</sub> 86.945	1.35	-95	40	5.32(3)	1	53.8(5)	27
Nitrobenzene-d <sub>5</sub> 128.143	1.25	6	211	8.11(br) 7.67(br) 7.50(br)		148.6(1) 134.8(3) 129.5(3) 123.5(3)	24.5(p) 25 26
Nitromethane-d <sub>3</sub> 64.059	1.20	-29	101	4.33(5)	2	62.8(7)	22
isoPropyl Alcohol-d <sub>8</sub> 68.146	0.90	-86	83	5.12(1) 3.89(br) 1.10(br)		62.9(3) 24.2(7)	21.5 19
Pyridine-d <sub>5</sub> 84.133	1.05	-42	116	8.71(br) 7.55(br) 7.19(br)		149.9(3) 135.5(3) 123.5(3)	27.5 24.5(g) 25
Tetrahydrofuran-d <sub>8</sub> 80.157	0.99	-109	66	3.58(br) 1.73(br)		67.4(5) 25.3(br)	22 20.5
Toluene-d <sub>8</sub> 100.91	0.94	-95	111	7.09(m) 7.00(br) 6.98(m) 2.09(5)	2.3	137.5(1) 128.9(3) 128.0(3) 125.2(3) 20.4(7)	23 24 24(p) 19
Trifluoroacetic Acid-d <sup>c</sup> 115.030	1.50	-15	72	11.50(1)		164.2(4) 116.6(4)	(44) (283)
2,2,2-Trifluoroethyl Alcohol-d <sub>3</sub> <sup>d</sup> 103.059	1.45	-44	75	5.02(1) 3.88(4 x 3)	2(9)	126.3(4) 61.5(4 x 5)	(277) 22(36)

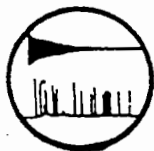
<sup>a</sup>Melting and boiling points (in °C) are those of the corresponding light compound (except for D<sub>2</sub>O) and are intended only to indicate the useful liquid range of the materials. The multiplicity br indicates a broad peak without resolvable fine structure, while m denotes one with fine structure.

<sup>b</sup> $\delta_F(CFCl_3)$  82.6(1)

<sup>c</sup> $\delta_F(CFCl_3)$  76.2(1)

<sup>d</sup> $\delta_F(CDCl_3)$  77.8(5),  $J_{FD}$  1.2





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October 19, 1995  
(received 11/13/95)

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

**From spectral editing to extracting dipolar couplings in partially oriented systems**

Dear Barry,

We were interested in finding a suitable editing sequence for identifying carbon resonances in static liquid crystal samples in their nematic phases. We realised that the dipolar dephasing sequence of Opella and Frey<sup>1</sup> would not work, since in the case of the oriented liquid crystal sample the carbons with attached protons have possibly resolved dipolar couplings with a small line-width and hence do not dephase fast enough. On the other hand, we observed that the cross-depolarisation sequence proposed by Zumbulyadis<sup>2</sup> works well for such samples (Fig.1).

In the course of optimising the depolarisation time for p-methoxy benzylidene p'-n-butylaniline (MBBA), we noticed that the intensities of the carbon peaks undergo an oscillatory change superposed on a monotonic change both during cross-polarisation and cross-depolarisation (Fig.2). These are in fact the transient oscillations in cross polarisation reported by Muller et al. on a single crystal of ferrocene<sup>3</sup>. The oscillation frequencies can be employed to provide the proton-carbon dipolar couplings and the corresponding liquid crystal order parameters<sup>4</sup>. A two dimensional experiment with the pulse sequence shown (Fig.3) has also been tried. The experiment provides the above information in one go and hence is more convenient (Fig.4).

**References**

1. S.J. Opella and M.H.Frey, J. Am. Chem. Soc., 101, 5884 (1979).
2. N. Zumbulyadis, J. Chem. Phys. 86, 1162 (1987).
3. L. Muller, Anil Kumar, A. Baumann and R.R. Ernst, Phys. Rev. Lett., 32, 1402 (1974).
4. R. Pratima and K.V. Ramanathan, J. Magn. Reson. A (In Press).

Regards,

*K.V. Ramanathan*

K.V. Ramanathan

*C.L. Khetrapal*

C.L. Khetrapal



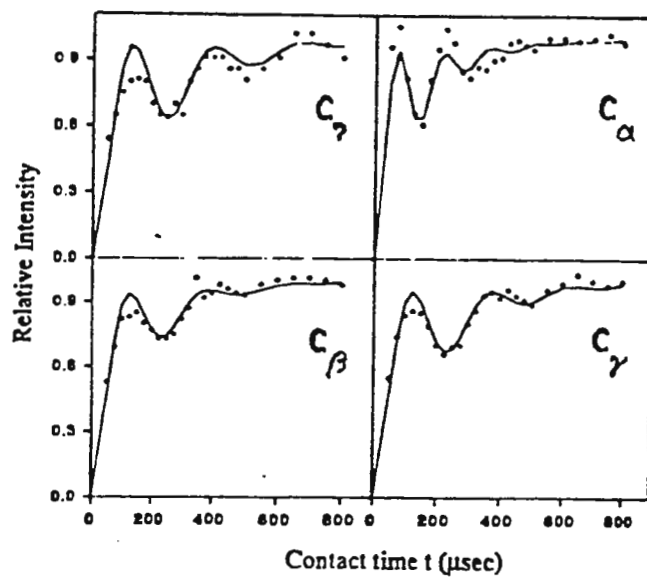
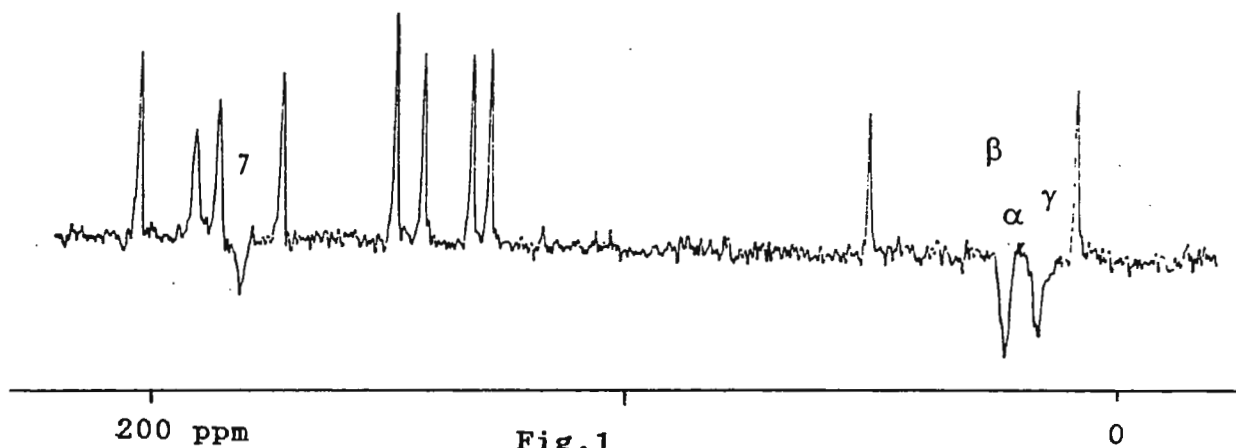
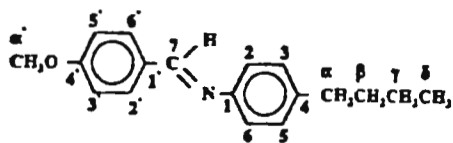


Fig. 2

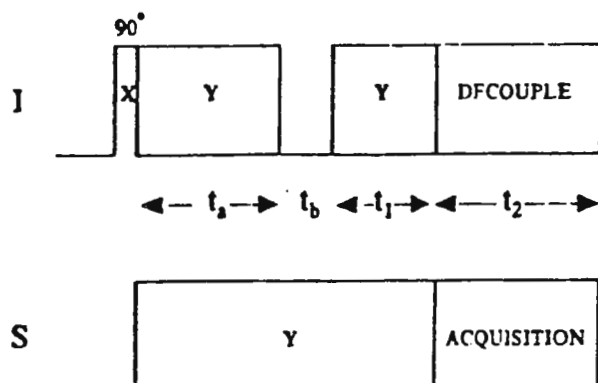


Fig. 3

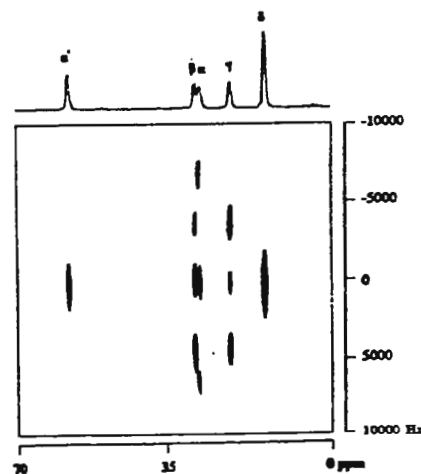
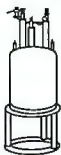
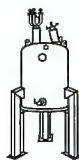
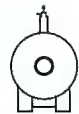


Fig. 4

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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
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November 14, 1995  
(received 11/17/95)

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

re: Multislice  $^1\text{H}$  Magnetic Resonance Spectroscopic Imaging to Study Dementia

Dear Dr. Shapiro,

We have been using  $^1\text{H}$  magnetic resonance spectroscopic imaging (MRSI) in our laboratory for several years to study Alzheimer's disease (AD) [1]. So far, however, data acquisition was constrained to a box-shaped region within the brain, sufficient away from the surface cortex, and without multislice capability. This was necessary to minimize contamination of  $^1\text{H}$  metabolite spectra from intense lipid resonances, primarily from lipids of subcutaneous tissue. Recently, we have implemented a new version of multislice MRSI on our clinical 1.5T MR scanner (VISION<sup>TM</sup>, Siemens) which enables us to observe  $^1\text{H}$  metabolite resonances from the entire brain, including surface cortex, an area which is believed to be affected very early in AD.

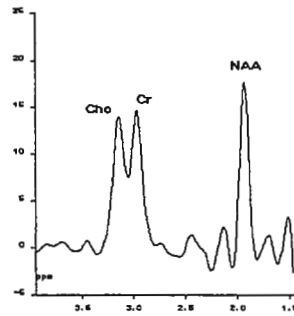
Multislice MRSI has been reported by several groups, but most strategies employ outer volume suppression pulses to reduce the intensity of the lipid signal. Unfortunately, these pulses also saturate spins of the surface grey matter of the brain, making quantitation of  $^1\text{H}$  metabolites difficult. We have developed a technique for multislice  $^1\text{H}$  MRSI which does not utilize outer volume suppression pulses. Lipid removal is accomplished entirely by postprocessing. This technique takes advantage of the method suggested by Hu et. al. [2] and the Papoulis-Gerchberg algorithm for data reconstruction by k-space extrapolation [3]. This method accomplished to reduce the intensity of the lipid signal by about 85%, sufficient to obtain a well resolved resonance line from NAA, as well as from choline (Cho) and creatine (Cr), the two other major peaks in brain spectra.

Using this approach of multislice MRSI, we have been able to acquire  $^1\text{H}$  metabolite MR spectra from the entire brain of AD patients. A metabolite image of NAA through temporal lobes and hippocampi from a 81 years old AD patient is shown below. This image was obtained by line-fitting of the NAA resonance and subsequent integration. Also shown is the proton image (MRI) from the same brain region. To our knowledge, this is the first time that NAA has been observed from the entire region of the temporal lobes of an AD brain, including the lateral temporal association cortices (positions (c) and (d) indicated in the MRI). The four metabolite spectra shown below and selected from temporal association cortices and hippocampi (a,b) demonstrate

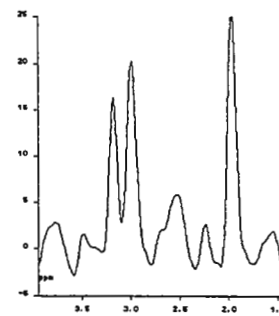
the excellent spectral resolution achieved. Total acquisition time of the experiment was 30 minutes, including the selection of 4 slices (each 1.5cm thick), TR/TE values of 1800/140ms, and 36 x 36 phase-encoding steps yielding a nominal pixel resolution of the NAA image of 8 x 8 mm<sup>2</sup>. We anticipate that multislice <sup>1</sup>H MRSI will be very helpful to determine regional patterns of metabolite changes in AD which eventually might aid the early detection and diagnosis of this devastating disease.



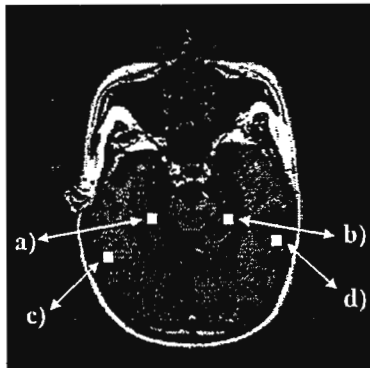
NAA Metabolite Image



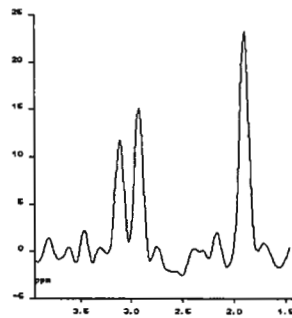
(a) from right



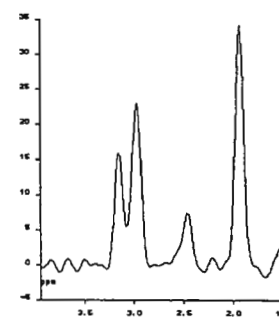
and (b) left hippocampus



MRI



(c) from right



and (d) left temporal association cortex

1. MacKay, S., Ezekiel, F., Di Sciafani, V., Gerson, J., Meyerhoff, D.J., Norman, D., Fein, G., and Weiner, M.W. Combining MRI segmentation and <sup>1</sup>H MR spectroscopic imaging in the study of Alzheimer's disease, subcortical ischemic vascular dementia and elderly controls. *Radiology* (In Press).
2. Hu, M. Patel, and K. Ugurbil. A new strategy for spectroscopic imaging. *J. Magn. Reson. B*, **103**, 30-38 (1994).
3. Haupt, C. I., Schuff, N., Weiner, M. W., Maudsley A. A.: Lipid removal in <sup>1</sup>H spectroscopic imaging by data extrapolation. (submitted)

Sincerely yours

Norbert Schuff

Christine I. Haupt

Michael W. Weiner

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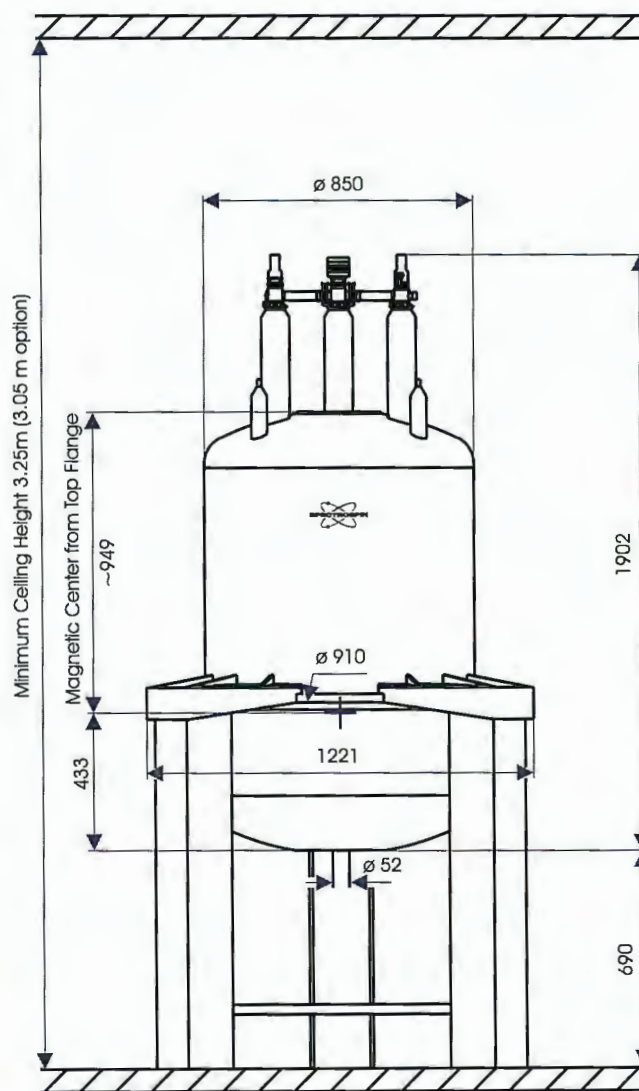
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Field Drift	< 9 Hz/hr
Cryo-shims	z, z <sup>2</sup> , x, y, xz, yz, xy, x <sup>2</sup> -y <sup>2</sup>
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October 4, 1995  
 (received 10/30/95)

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

Dear Dr. Shapiro,

### Non-Selective $T_1$ Relaxation Times Measurements by the Newly Developed IR-TOCSY

Longitudinal relaxation times are usually determined by the classical  $^1\text{H}$  non-selective inversion-recovery (IR) sequence. Yet this sequence is of limited interest in molecules which overcrowded 1D spectra.

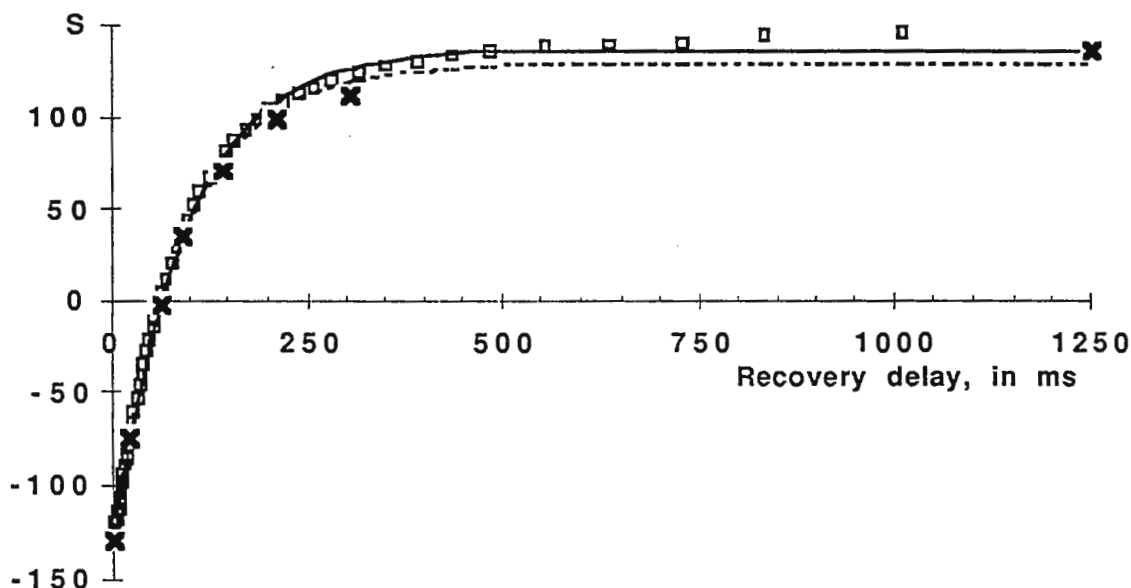
The IR-COSY sequence<sup>1</sup> takes advantage of the better resolution of 2D spectra to measure non-selective  $T_1$  values. This sequence consists of the conventional inversion-recovery followed by a COSY sequence. The recovery delay is incremented in order to obtain a serie of 2D experiments. The intensity of a cross peak between protons H1 along F1 and H2 along F2 is a function of the IR delay and of the non-selective  $T_1$  value of H1 but not of H2<sup>1</sup>.

The IR-TOCSY was built by substituting the COSY sequence by a TOCSY<sup>2</sup> one. The IR-TOCSY combines the advantages of IR-COSY and TOCSY, mainly increased resolution and cross peaks in phased pure absorption mode. Furthermore, in the case of paramagnetic proteins, the broad antiphase correlation peaks often cancel out in a COSY-type experiment.

The applicability of the IR-TOCSY to measure the non-selective  $T_1$  of paramagnetic reduced *Chromatium vinosum* high potential ferredoxin<sup>3</sup> has been demonstrated by comparison with 1D inversion-recovery sequence.

The intensity of 1D signals and 2D correlation peaks was fitted with the function :  $S = A \left( 1 - 2B \exp \left( -\frac{\text{delay}}{T_1} \right) \right)$

The results obtained with the NH of Ile 71 are shown.



The squares and the crosses correspond to the values obtained from 1D IR and IR-TOCSY experiments respectively. The continuous and the dashed lines are the corresponding fits.

Non-selective  $T_1$  values of 94 ms and 92 ms were deduced from the 1D IR and IR-TOCSY experiments respectively. These values were in good agreement for all the protons studied, with less than 10 % error, which is the classical estimated error.

## REFERENCES

- 1 - Arseniev A. S. , Gobol A. G. and Bystrov V. F. (1986) J. Magn. Reson. 70, 427-435
- 2 - Bax A. and Davis D. G. , J. Magn. Reson. 65 (1985) 355-360
- 3 - Gaillard J. , Albrand J.-P., Moulis J.-M. and Wemmer D. E. (1992) Biochemistry 31, 5632-5639

Sincerely yours,

Huber J. G.

Gaillard J.

This work was carried out in collaboration with Dr. J.-M. Moulis, CEA, DBMS, Laboratoire de Métalloprotéines, Grenoble, France

P.S. Please credit this contribution to the account of Dr. Philippe Vottero.



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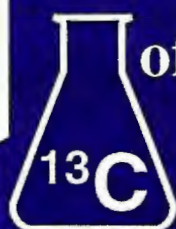
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### Pure-phase selective excitation

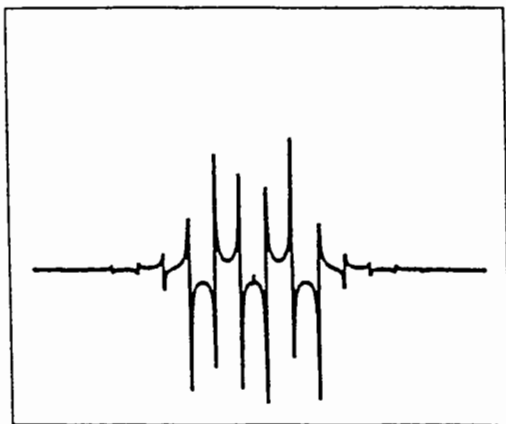
Lausanne, Tuesday, October 31, 1995 (received 11/4/95)

Dear Dr. Shapiro,

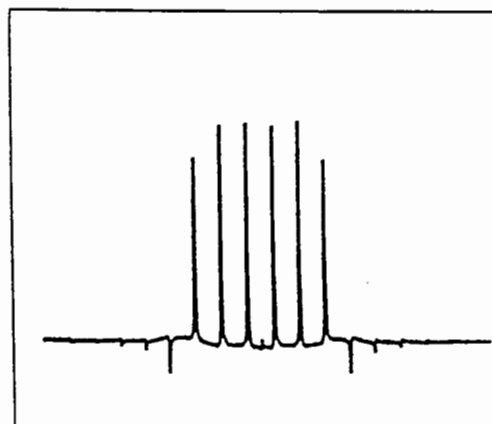
Selective excitation has been used in NMR as a useful tool to achieve accurate measurements in complex systems. Typical advantages of selective methods are: the possibility of limiting the duration of the time domains in a multidimensional experiment; reduced high-dimension experiments can be conceived.; selective removal of unwanted effects (refocussing of spin-diffusion, decoupling).

On the other hand, the technique is not flawless. One of the problems that have to be faced is the linear phase distortion that is introduced in the excitation window as a function of the offset. This is a general effect for symmetric excitation pulses and can be understood in terms of the shift theorem of the Fourier Transform. In fact, the complex FT of a symmetric pulse is a pure real function in the frequency domain only if the origin of the time is set in the middle of the excitation pulse. A shift in the time domain corresponds to a linear phase gradient in the frequency domain. Therefore, the linear phase gradient observed in the NMR experiment is due to the choice of the origin of time (i.e. the beginning of the acquisition) at the end of the pulse. The effect is then ubiquitous but much more dramatic in the case of selective excitation, where the long pulses used in order to achieve the desired window of excited frequencies are comparable in length to the dwell time.

Excitation schemes designed to prevent this drawback have been proposed over the years. They include projection methods, that only keep the magnetization with the desired phase or self-refocussing pulses, that drives the magnetization along specific paths in order to amount to good excitation and no offset dependent phase gradient as an overall effect.



Excitation profile of a sinc-shaped DANTE pulse train



Excitation profile of a sinc-shaped DANTE pulse train with retro-active acquisition



Recently, we proposed an alternative methodology that allows to achieve "pure-phase" excitation using arbitrary symmetric shaped pulses. The scheme consists in altering a "normal" NMR experiment by regressing the origin of time back to the middle of the pulse.

Experimentally this is implemented by "chopping" a regular shaped pulse, in order to create what can be regarded as a shaped DANTE pulse train. The usual requirements for the pulse/delay ratio typical of DANTE are observed. The "windows" in the pulse (i.e. the periods with RF off) can be used to acquire points before the end of the pulse, a special case being the middle of the pulse.

We decided to call this technique *pure-phase achieved by retroactive acquisition in DANTE-implemented selective excitation (PARADISE)*, noticing that it constitutes perhaps the last step on the path of DANTE ...

The effect of implementing this particular scheme are shown in the figure for a sinc pulse shape, but other symmetrical shapes may be used according to anyone's right to choose their own PARADISE.

Best regards,

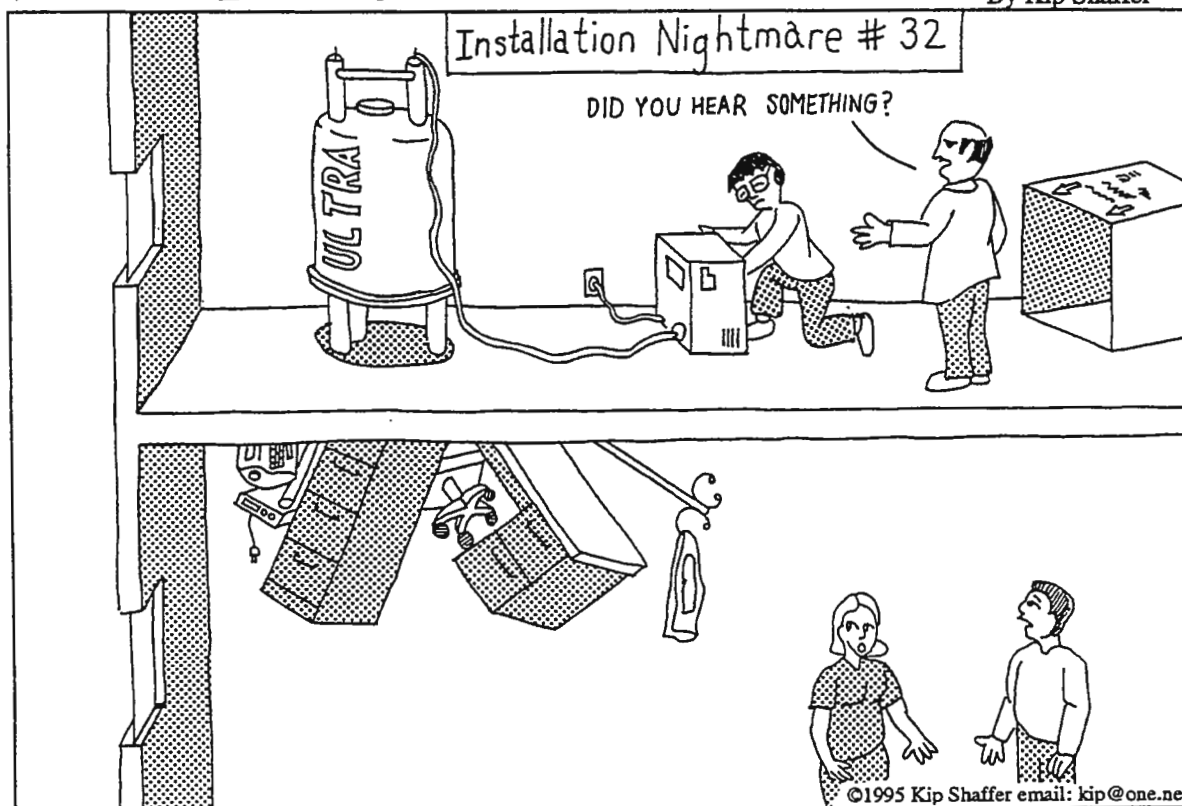
  
Stefano Caldarelli

(1) S. Caldarelli, A. Lesage and L. Emsley, Journ. Magn. Reson. 116,129 (1995)

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Dear Dr Shapiro

Theix, 7 November 1995  
(received 11/20/95)

## THE MAGNETIC FIELD DEPENDENCE OF WATER PROTONS TRANSVERSE RELAXATION IN GELATIN

To characterize water-gelatin interactions, the transverse water proton relaxation rates ( $1/T_2$ ) were measured on water-gelatin solution at two Larmor frequencies (20 and 400 MHz). The CPMG pulse sequence was used with a 2 ms-interpulse spacing. Gelatin concentration was 10% (w/w) and the pH was adjusted to 6. Samples were thermostated at three sol state temperatures.

At 20 MHz,  $1/T_2$  increases with the inverse of absolute temperature while it decreases at 400 MHz. These results can be explained by two dynamic processes. In our condition of long interpulse spacing,  $\frac{1}{T_2} = \frac{P_a}{T_{2a}} + \frac{P_b}{T_{2b}} + \frac{P_b(\Delta\delta\omega)^2}{k_b}$  where  $T_{2i}$  is the relaxation time in the site  $i$ ,  $P_i$  is the population in the site  $i$  and  $1/k_b$  is the lifetime in the site  $b$  and  $(\Delta\delta\omega)$  is the chemical shift difference between the two sites (1). At high frequency,  $1/T_2$  depends on the chemical exchange because  $\frac{(\Delta\delta\omega)^2}{k_b} \gg \frac{1}{T_{2b}}$ , while at 20 MHz,  $(\Delta\delta\omega)^2$  is 400 times less than at 400 MHz and  $\frac{1}{T_{2b}}$  should also decrease. So dipole-dipole interactions are prevailing.

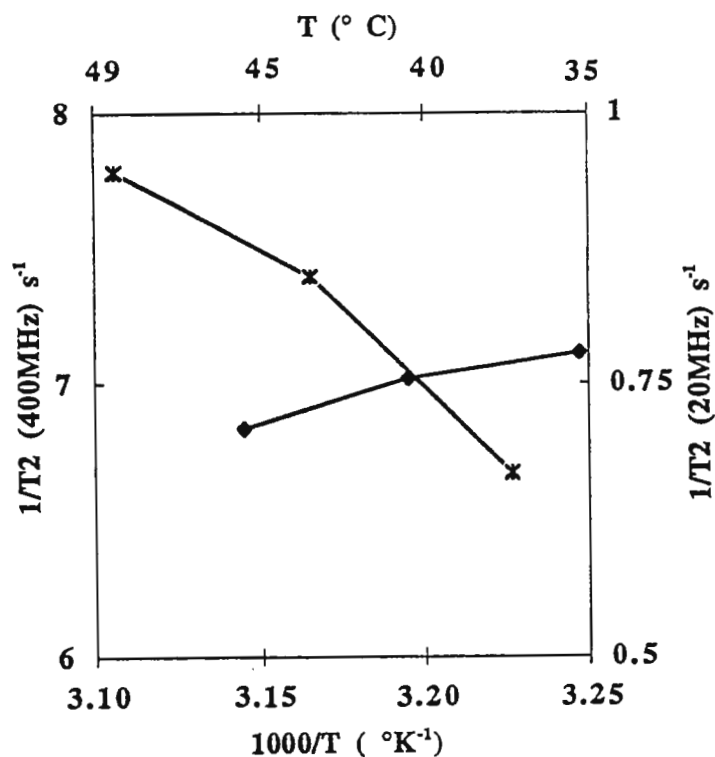
Sincerely yours,

A. TRAORE

L. FOUCAT

J.P. RENOU

(1) Carver J.P. & Richards R.E. (1972) *J. Magn. Reson.* 6; 89.



Transverse water proton relaxation rate  $1/T_2$  ( $s^{-1}$ ) plotted versus reciprocal temperature for gelatin 10% at 20MHz (♦) and 400MHz(\*).

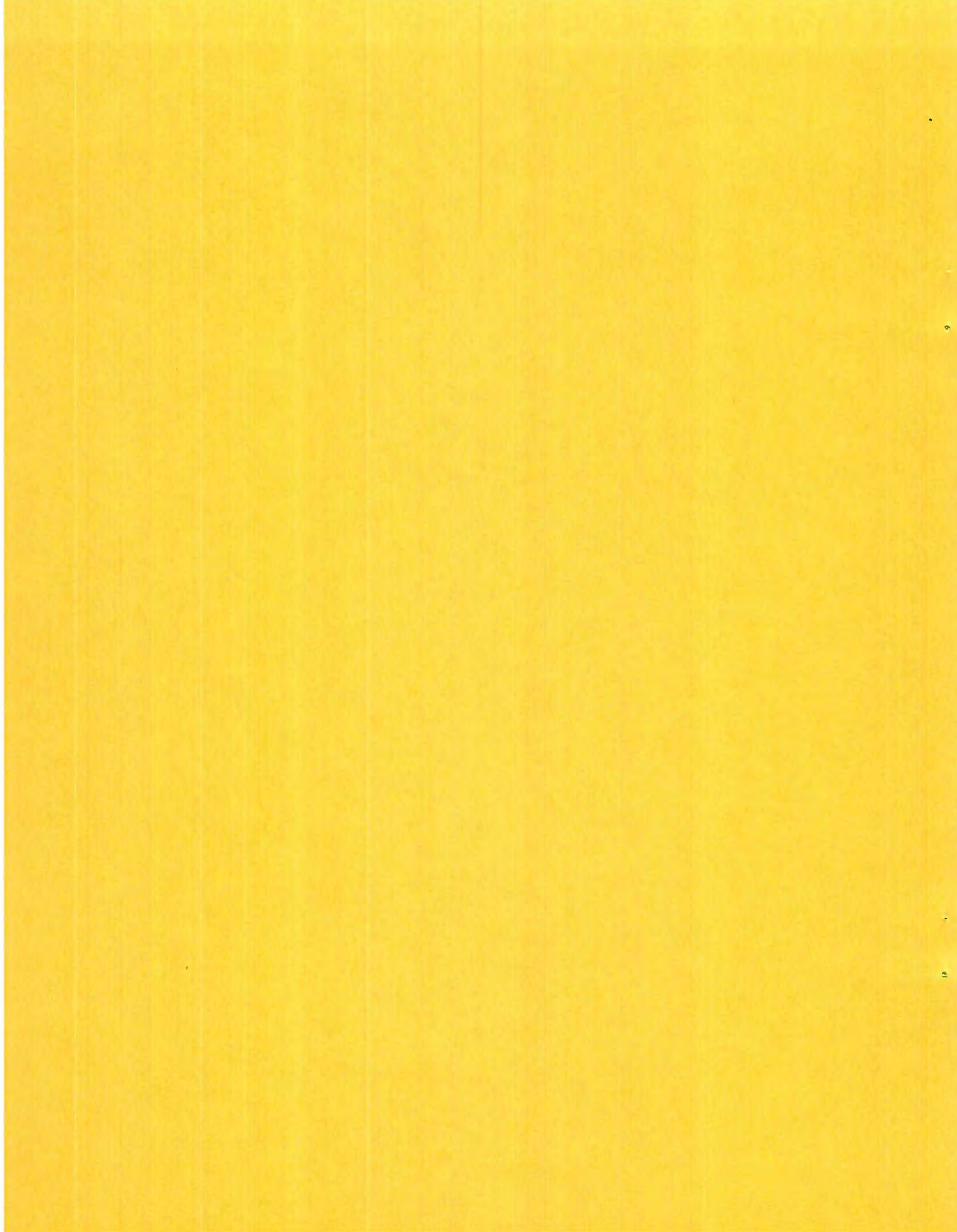
- Continued from page 40.

Table 1:

ATOM	cal_T1(sec)	exp_T1(sec)	ATOM	cal_T1(sec)	exp_T1(sec)
A3H2	6.33	12.3	C2H6	1.80	-
A5H2	8.42	-	T6H6	2.67	2.4
A8H2	6.74	-	C12H6	1.60	-
A10H2	11.64	21.7	T13H6	1.62	1.7
A14H2	10.70	-	T15H6	3.10	-
A17H2	4.52	-	C16H6	2.23	-
A19H2	8.51	-	T20H6	2.43	2.4
G1H8	1.64	-	T4H6	2.15	-
A3H8	2.15	2.3	T9H6	2.50	2.5
A5H8	2.20	2.6	T18H6	2.55	-
G7H8	2.50	2.6	C22H6	2.62	-
A10H8	2.05	2.3	T4M7	2.10	2.6
A14H8	2.36	2.2	T6M7	1.77	2.6
A17H8	2.38	-	T9M7	2.43	2.6
A19H8	2.03	2.6	T13M7	1.72	1.9
G21H8	2.26	-	T15M7	2.51	2.9
A8H8	2.14	2.3	T18M7	2.18	2.7
G11H8	2.70	-	T20M7	1.64	2.4

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## at a glance...

November '95

- Tony, sales ('87 University of Houston) - When Hakeem Alajuwon scored his 20,000<sup>th</sup> career point early this season 1995, it marked the second time a UH alumnus had achieved that milestone in the NBA. The first was Elvin Hayes. Clyde Drexler is also expected to reach that milestone by the end of this season. No other school has more than one alumnus that had achieved that mark.
- John, sales ('88 University of California at Berkeley) - Early preseason AP poll places California Golden Bears at 25<sup>th</sup> in the nation. Both recent former Cal players Jason Kidd of the Mavericks and Lamond Murray of the Clippers are doing well in the NBA with Kidd taking Rookie of the Year honor last year.

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(received 11/24/95)

### Correction for Intensities of Partially Relaxed NOESY Experiments

Dear Barry:

Obtaining interproton distances from NOESY spectra for structure studies is well established. As we have wished to obtain distances and bounds as accurately as possible, we have used much instrument time to generate (nearly) completely relaxed spectra, and we use a complete relaxation matrix approach (MARDIGRAS) to account for spin diffusion in our analysis of the spectral intensities. We have recently developed a way to analyze partially relaxed spectra, as shown below, which can save us much NMR acquisition time without the previous fear of skewing the resulting distances.

The integral of a NOESY cross peak  $I_{ij}$  is proportional to the longitudinal magnetization of spin  $j$ , i.e.,  $M_{zj}(0)$ , recovered before the pulses of next  $t_1$  incrementation. The relaxation delay is recommended as three times the longest  $T_1$  values. For short repetition time where  $M_{zj}(0)$  is smaller than 1, the intensities are scaled:

$$\begin{aligned} I_{ij} &= I_{0ij} M_{zj}(0) & (1) \\ \text{with } I_{0ij} &= [\exp(-Rt_m)]_{ij} & (2) \\ \text{and } M_{zj}(0) &= \sum [I - \exp(-R(rd + aq))]_{jk} & (3) \end{aligned}$$

where  $I_{0ij}$  is the intensity with full relaxation,  $R$  is the relaxation rate matrix,  $rd$  is the relaxation delay and  $aq$  is the acquisition time.

Cross relaxation rates (hence interproton distances) are determined with Eq. (2), which requires fully relaxed NOE intensities. Previous work on obtaining fully relaxed  $I_{0ij}$  from partially relaxed  $I_{ij}$  (M. Kock and C. Griesinger, *Angew. Chem. Int. Ed. Engl.* 1994, **33**, 332) relies on the assumption that the molecule is relatively small and  $T_1$ s of all spins can be measured. In this letter, we describe an approach that uses the ratio of NOE intensities above and below the diagonal to obtain  $I_{0ij}$ .

In general,  $I_{ij}$  does not equal  $I_{ji}$  if spin  $i$  and spin  $j$  relax at different rate. We consider  $M_{zj}(0)$  in Eq. (1) as a scaling factor, and it can be determined by the ratio:

$$M_{zi}(0) / M_{zj}(0) = I_{ij} / I_{ji} \quad (4)$$

We have developed a program that applies the above relation to a partially relaxed NOESY spectrum iteratively to obtain a set of  $M_{zi}(0)$ s that satisfy the following equations:

$$\begin{aligned} I_{0ij} &= I_{ij} / M_{zi}(0) & (5) \\ \text{and } I_{0ji} &= I_{ji} / M_{zj}(0) & (6) \\ \text{with } I_{0ij} &= I_{0ji} & (7) \end{aligned}$$

i.e., assuming the fully relaxed upper- and lower-diagonal intensities are equal. In another words, a partially relaxed NOESY matrix can be symmetrized by  $M_{zi}(0)$ s, which can be obtained by iteratively applying Eq. (4) to the upper- and lower-diagonal pairs of the NOE matrix. Once the  $M_{zi}(0)$ s are known, an estimation for  $T_1$ s is possible.

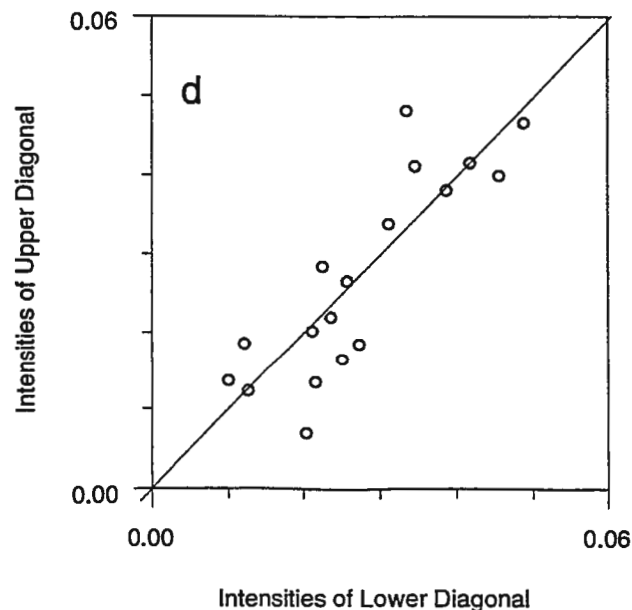
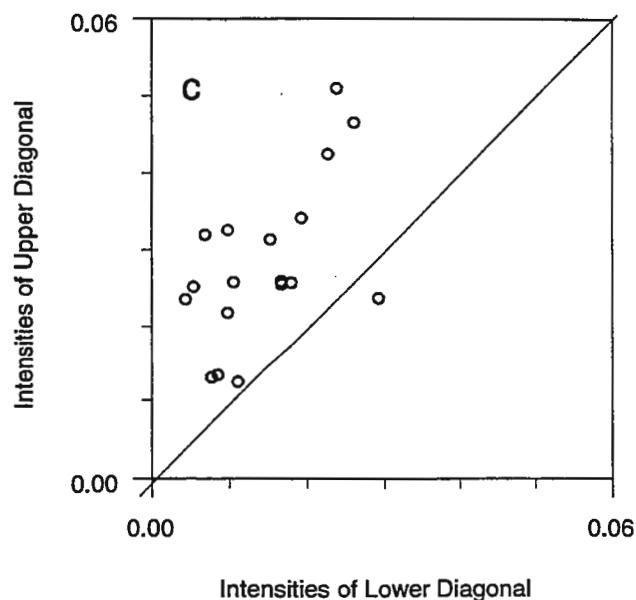
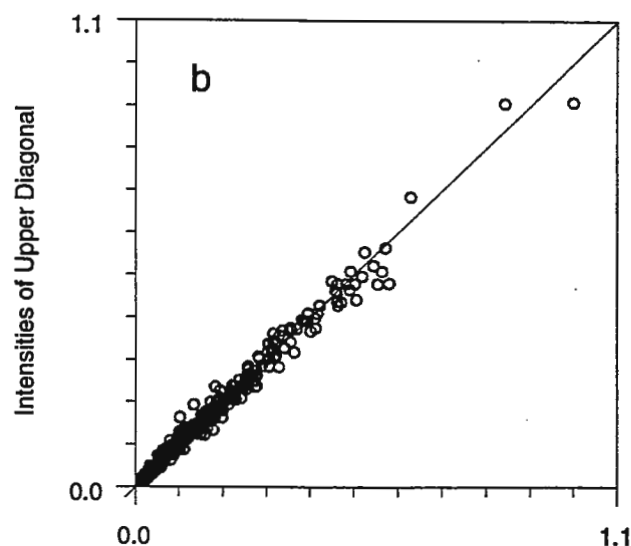
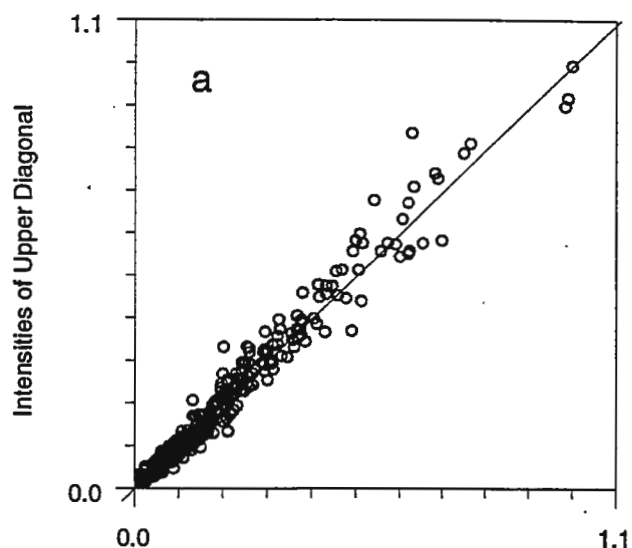
The method has been applied to a 270 ms NOESY spectrum of  $d(\text{GCATATGATAG})_2$  with 2.5 sec repetition. The circles plotted in Figure 1a are the upper diagonal intensities versus the lower diagonal intensities before the correction is applied. Figure 1b shows that after the correction the intensities are more symmetric. In Figure 1c, ADE H2 intensities are plotted. All the upper diagonal intensities (except one) are much stronger than the lower diagonal intensities. Figure 1d shows the intensities after correction.

The ratio of the upper- and lower-diagonal intensities may be affected by three factors: (1) partial relaxation; (2) experimental and integration errors; (3) different resolution for  $\omega_1$  and  $\omega_2$  axes. (1) and (3) yield systematic errors, i.e., peaks of each row are scaled by the same fraction. Point (2) errors are random and will be partially averaged by the summation in the iterations. The challenge to estimate  $T_1$ s is to separate  $M_{zj}(0)$ s from other systematic contributions. We assumed that for this particular data set, the effect of  $M_{zj}(0)$ s dominates the difference between upper and lower diagonal intensities. By assuming that  $M_{zj}(0) \approx 1 - \exp[-(rd + aq)/T_{1j}]$ , we obtained  $T_1$ s for all the protons. Table 1\* compares calculated and measured (by inversion-recovery)  $T_1$ s of H2, H6, H8 protons and THY methyl groups.

Sincerely,

*He Liu, Marco Tonelli, Thomas L. James*  
He Liu, Marco Tonelli, Thomas L. James

\* Continued on page 36



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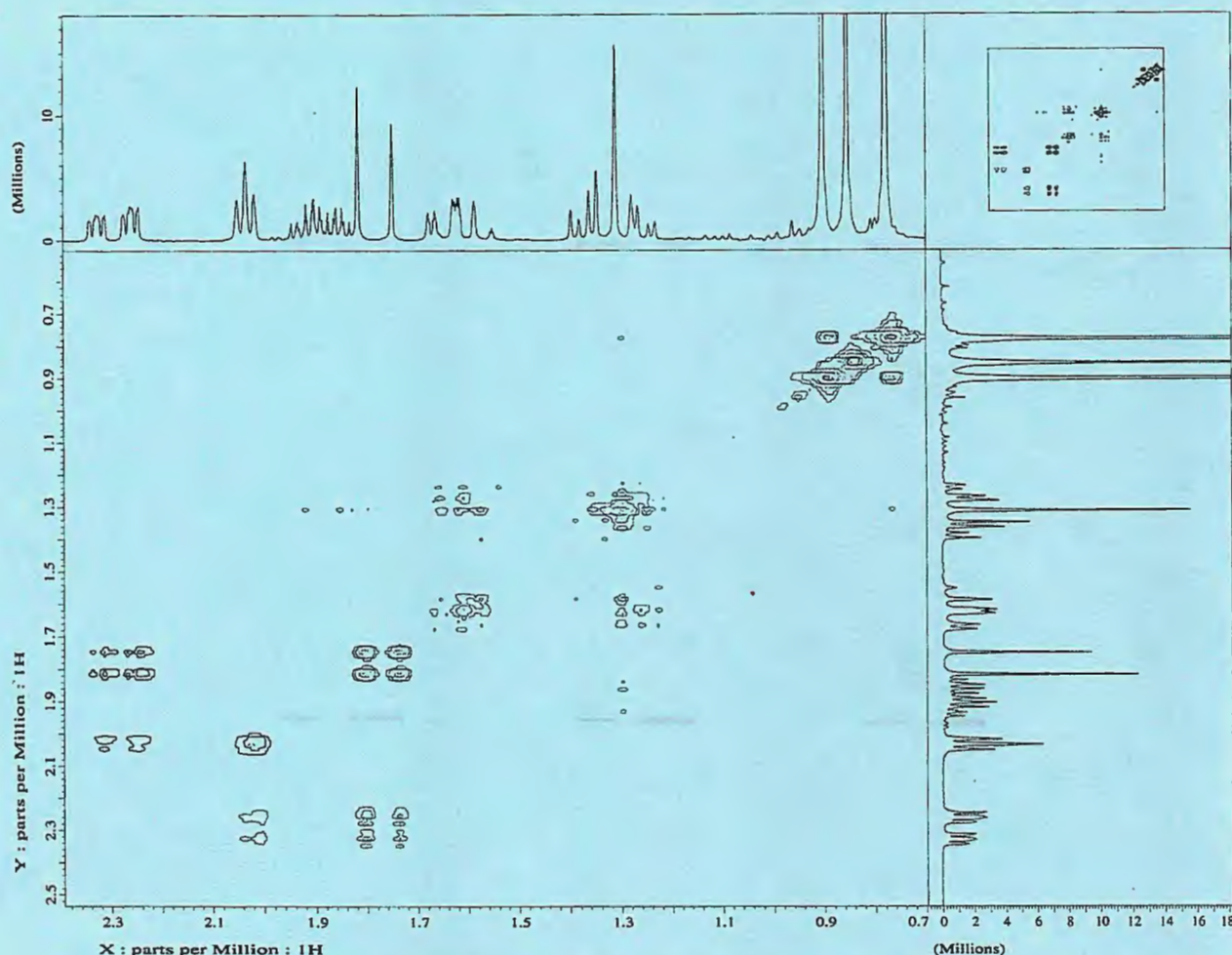
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