TEXAS A&M UNIVERSITY

NMR NEWSLETTER

No. 405

June 1992

TABLE 1 DEUTERATED SOLVENTS

Cat. No.	Description	Formula	Min. Isotopic Purity	Density (g/cc)	MP (°C)	BP (°C)	$-\chi_v$ X 10^6 @ (°C)
D-11	Acetone-d_4	CD_3OD	[illegible]	1.1[illegible]	[illegible]	[illegible]	0.551 (32)
D-120	Acetone-d_4 + 1% TMS	[illegible]	[illegible]				
D-13	Acetone-d_6	CD_3COCD_3	99.8%	0.8[illegible]	[illegible]	[illegible]	0.460 (20)
D-121	Aceonte-d_6 + 1% TMS		99.8%				
D-129							
D-14							543 (20)
D-21							0.611
D-122							
D-130							
D-28	Choloroform-d	$CDCl_3$	99.8%	1.50	-64	62	0.740 (20)
D-31	Chloroform-d + 1% TMS		99.8%				

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VARIAN

BOX/500 SHEETS

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

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FORTHCOMING NMR MEETINGS

Gordon Research Conference: Magnetic Resonance in Biology and Medicine, Tilton, NH, **July 13 - 17, 1992**; Contact: Dr. A. M. Cruickshank, Gordon Research Center. University of Rhode Isalnd, Kingston, RI, 02881-0801; (401) 783-4011/3372; FAX: (401) 783-7644.

ISMAR 92 (The XIth Meeting of the International Society for Magnetic Resonance), Vancouver, B.C., Canada, **July 19 - 24, 1992**; Chairman: C. Fyfe. Contact: ISMAR 92, Dept. of Chemistry, University of British Columbia, Vancouver, BC, Canada V6T 1Z1. Tel: (604) 822-2293; FAX: (604) 822-2847; EMAIL: ismar@unixg.ubc.ca; BITNET: ismar@ubcmtsg.bitnet.

Science Innovation '92, New Techniques and Instruments in Biomedical Research (sponsored by the AAAS), San Francisco, **July 21-25, 1992**; Workshops on biomedical imaging. chemical and structural NMR; Plenary session on NMR on Fri., July 24. Contact: Science Innovation '92, P.O. Box 630285, Baltimore, MD 21263; fax (202) 289-4021.

34th Rocky Mountain Conference on Analytical Chemistry, Denver, Colorado, **August 8-14, 1992**; Contact: M. C. Goldberg, P.O. Box 25046 MS 424, Lakewood, CO 80225; (303)236-4728.

Eleventh Annual Scientific Meeting and Exhibition, Society of Magnetic Resonance in Medicine, Berlin, Germany, **August 8-14, 1992**; Contact: S.M.R.M., 1918 University Ave., Suite 3C, Berkeley, CA 94704; (415) 841-1899, FAX: (415) 841-2340.

XV International Conference on Magnetic Resonance in Biological Systems, Jerusalem, Israel, **August 16 - 21, 1992**; Contact: Prof. Gil Navon, XV ICMRBS, P. O. Box 50006, Tel Aviv 61500, Israel.; Tel. (972-3) 5174571, Fax: (972-3) 655674/660325.

MRI in the Applied Sciences, Duke University, Durham, North Carolina, **October 25-28, 1992**; Contact: Society of Magnetic Resonmance in Medicine, 1918 University Ave., Suite 3C, Berkeley, CA 94704; (510) 841-1899; FAX: (510) 841-2340.

High Resolution NMR Spectroscopy (a residential school), University of Sheffield, England, **April 1993**[*sic*]; Organizer: Dr. B. E. Mann (Sheffield); For information, contact Ms. L. Hart, The Royal Society of Chemistry, Burlington House, Piccadilly, London W1V 0BN, England; Tel.: 071-437-8656.

Additional listings of meetings, etc., are invited.

Please see notices on page 49.

TAMU NMR Newsletter

Editor/Publisher: Bernard L. Shapiro

Address all correspondence to: 966 Elsinore Court, Palo Alto, CA 94303, U.S.A. (415) 493-5971

Newsletter Finances

The overall finances of the Newsletter remain precarious. Even more than in the past, your help is needed so that the continuation of the Newsletter is assured. As some of you may not know, the Newsletter is completely self-sufficient financially, as it has been for well over twenty years - all costs are paid from funds raised by Subscriptions, Sponsorships, and Advertising.

Unfortunately, income from Sponsorships and Advertising has declined significantly during the past year, and there are no signs of a reversal of this trend. Since these two sources account for approximately two-thirds of the total Newsletter revenues, it is clear that something must be done. The help of the Subscribers, Sponsors, and Advertisers is earnestly solicited.

Subcribers whose organizations can be induced to become **Sponsors** are urged to actively pursue this possibility. A Sponsorship donation - minimum contribution of $500/yr. - entitles the donor to at least three Newsletter copies each month, and a listing as a Sponsor if so desired.

More Advertisers are quite urgently required. If you can encourage more frequent advertising by our current advertisers and/or recruit new advertisers, please do so. Advertising in the Newsletter is a well-focussed means of reaching a highly targetted readership. Our ad rates are being raised a bit, but will remain very modest by standards in the field. Please note the excellent level of involvement by our core of loyal, every-month advertisers, and let them know that their support is appreciated. Without them and our other frequent advertisers, the Newsletter would cease to exist. Your help here will surely be effective.

The Newsletter's existence has always been predicated on its self-regulating nature, both insofar as the technical content *and* its financing are concerned. While the former aspect is in good shape , the finances are very problematical - in fact, the problem of fiscal viability in tending toward the critical stage. I am not 'crying wolf', but am trying to alert all who are interested in the Newsletter to the fiscal realities. Increased funding via more Advertising and Sponsorships is absolutely necessary.

Notice re 1992-93 Invoices and Subscription Rates

Subscription renewal invoices for the October 1992 - September 1993 year will be mailed out at the beginning of July. If you ought to receive such an invoice, and do not have it in your hands by July 15, please call or write me promptly. *Payment* of these invoices *must be received* by me *no later than September 10, 1992* to ensure uninterrupted mailing of the Newsletter issues. Please do not delay execution of any necessary paperwork!

Also, please be sure that the instructions on the invoice are followed precisely. In particular, *overseas subscribers* should be careful to see that their name and the invoice number appear on the payment (or, better, that the extra invoice copy which is provided is returned to me with the payment check or money order). Anonymous checks, while otherwise useful, cannot always be credited to the correct account.

The subscription rate for the October 1992 - September 1993 year has been set at US$170.00 for the twelve monthly issues, postpaid. Personal or academic subscriptions will continue to be offered at a 50% discount, at US$85.00. The inexorable rise in costs has necessitated this small increase in the subscription fee.

The new invoices contain entries for *optional* surcharges for First Class or Air Mail Printed Matter mailing. Please adjust the amount you pay accordingly (I trust no one will choose to pay both surcharges.).

Thank you for your understanding and cooperation.

B. L. Shapiro
1 June 1992

ARX – Automated Research Excellence

Performance Data

Summary of Specifications for common probeheads					
ARX		300	400	500	All
Probehead	Sample				
Resolution Test					
All 5 mm ^{1}H 5+10 mm ^{13}C	ODCB/C_6H_6	0.2	0.2	0.2	
Lineshape Test					**SSB %**
All 5 mm ^{1}H	10% $CHCl_3$	6/12	7/15	7/15	<1
All 5 (10) mm ^{13}C	80% C_6H_6	3/7 (3/7)	3/7 (3/8)	4/8 (4/8)	<0.5 (<1)
Sensitivity Test					
5 mm ^{1}H Selective	0.1% EB	175	250	450	<10
5 mm ^{1}H Inverse Detection	0.1% EB	135	190	350	<15
5 mm ^{1}H Dual, QNP, VSP	0.1% EB	100	140	200	<15
5/10 mm ^{13}C QNP, Dual	ASTM	100/320	160/450	180/600	<15/<20
5/10 mm ^{13}C QNP, Dual	10% EB	70/200	100/300	150/400	
5/10 mm ^{13}C VSP multinuc.	ASTM	100/320	160/450	180/600	<15/<20
5/10 mm ^{13}C VSP multinuc.	10% EB	70/200	100/260	150/320	
5/10 mm ^{15}N VSP multinuc.	90% Form.	10/35	15/55	20/70	<25/<30

EB = ethylbenzene (for ^{13}C with ^{1}H-dec.);
ASTM = 60% C_6D_6 in dioxane
Form. = formamide (^{1}H-dec. without NOE)
lineshape: ^{1}H = $CHCl_3$ linewidth at ht. of ^{13}C-satellites/at 20^{6} this level
^{13}C = C_6H_6 linewidth at 0.55% / 0.11 level (^{1}H-dec.)
SSB = Spinning sidebands measured with 8 transients
QNP: 5 or 10 mm ^{1}H, ^{31}P, ^{13}C, ^{15}N
VSP: 5 mm ^{15}N – ^{13}P; 10 mm ^{109}Ag – ^{31}P.

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Telefon 031 65 43 11

Dr. Bernard Shapiro
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto. CA 943030, USA

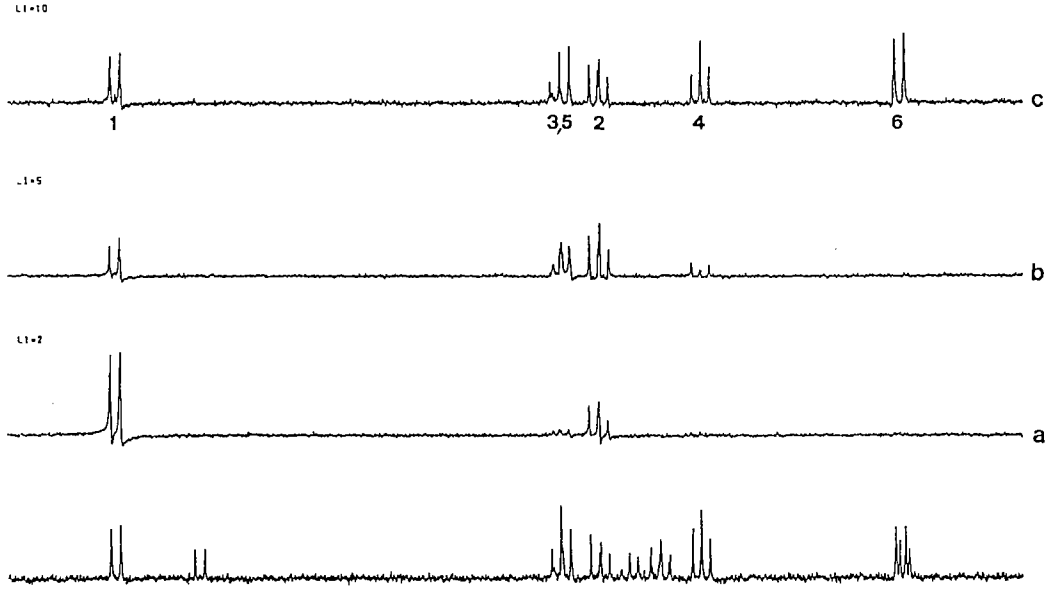
April, 24th 1992
(received 5/15/92)

1D $^{13}C/^{13}C$ TOCSY SPECTROSCOPY

Dear Dr. Shapiro

Carbon-carbon coupling interactions have widely been used in the past to obtain connectivity (usually from ^{1}J CC) and stereochemical (usually from ^{n}J CC) information for molecules of unknown structure. In the course of our attempts to improve the inherent low sensitivity of the selective 1D INADEQUATE sequence - best suited in cases where both connectivity information and precise carbon-carbon coupling constants are of interest - by using selective (shaped) carbon pulses and more efficient accumulation schemes, we have also looked to alternative experiments.

Therefore we modified the popular 1D $^{1}H/^{1}H$ TOCSY experiment to study and measure $^{13}C/^{13}C$ cross-polarization effects. On our BRUKER AM 400 we used a selective excitation unit (SEU) to selectively perturb a selected carbon resonance. A MLEV-17 sequence was applied to the BFX5 carbon transmitter for spin lock and a final z-filter was used. As a test sample we used 99% ^{13}C enriched glucose, the ^{13}C spectrum of which is shown below (bottom trace). It proves the presence of both epimeric forms (α and β). The upper traces demonstrate the effect of cross-polarization after selective perturbation of the anomeric carbon of the β-epimer for three different spin-lock mixing times: a) 7.3 ms, b) 18.1 ms and c) 36.2 ms. The top trace shows the thereby selected carbon spectrum of the β-epimer of glucose.



Yours sincerely
Dr. C. Müller
Dr. P. Bigler

405-6 **ROSKILDE UNIVERSITETSCENTER**
POSTBOX 260, DK-4000 ROSKILDE
Associate professor Poul Erik Hansen, Institute of Life Sciences and Chemistry

Prof. B.L.Shapiro
966 Elsinore Court
Palo Alto, CA 94303
U.S.A.

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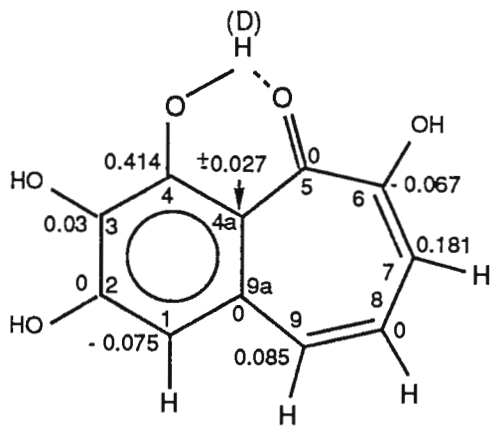
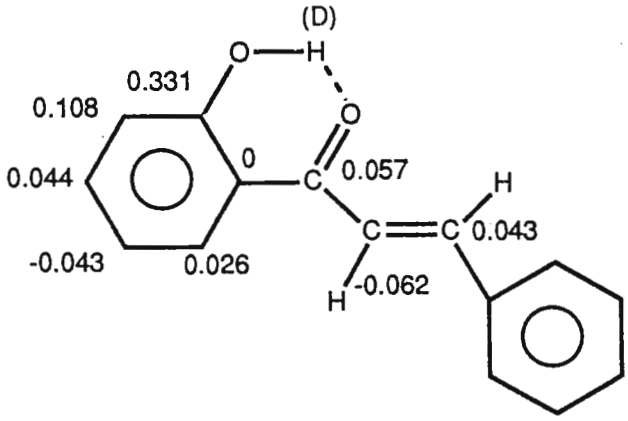
April 29 1992 (received 5/7/92)

"Long-range Deuterium Isotope Effects on ^{13}C Chemical Shifts"

Dear Professor Shapiro

In our studies of deuterium isotope effects on ^{13}C chemical shifts it has become important to understand the mechanism of long-range effects. A comparison of effects in o-hydroxypropiophenone(I), o-hydroxybenzophenone(II), purpurogalline(III) and in o-hydroxychalcone(IV) reveal clearly that the effects are poorly transmitted through single bonds and in aromatic systems.





I shows no isotope effects to the β-carbon and II shows an isotope effects to C-1', but not to the remaining carbons of the other aromatic ring. On the other hand, long-range effects are easily transmitted over as many as seven or eight bonds in compounds with alternating bonds such as III or IV. This picture fits nicely with Cynthia Jamesons recent theoretical predictions.

Yours sincerely

Poul Erik Hansen

Doty Scientific, Inc.

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Why are we the only company that always quotes pulse power requirements?

A recent comparison between a Doty 7 mm CPMAS probe (that had been in the field for three years) and an "equivalent" new probe from the company that "delivers" showed our probe to have nearly twice the sensitivity of their probe. But that didn't surprise us, we understand that sensitivity is inversely proportional to the square root of power. More is not better.

What about frictional heating?

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It seems like everyone wants to sell MAS probes now, and everyone is quick to recite trademarks and buzz-words. One competitor likes to talk about a "spinning advantage", but their spinning speeds don't come close to ours, and their turbine design simply does not allow extended VT operation. Another competitor says "COAX electronics" is superior, but they fail to mention that (1) their decoupling efficiency is less than half of ours, (2) the coax dielectric adds lots of wide-line carbon background, (3) the coax inductance degrades filling factor and transient response, and (4) their added capacitance reduces high-end tuning range.

Just remember, sensitivity is proportional to the square root of efficiency.

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Unser Zeichen:

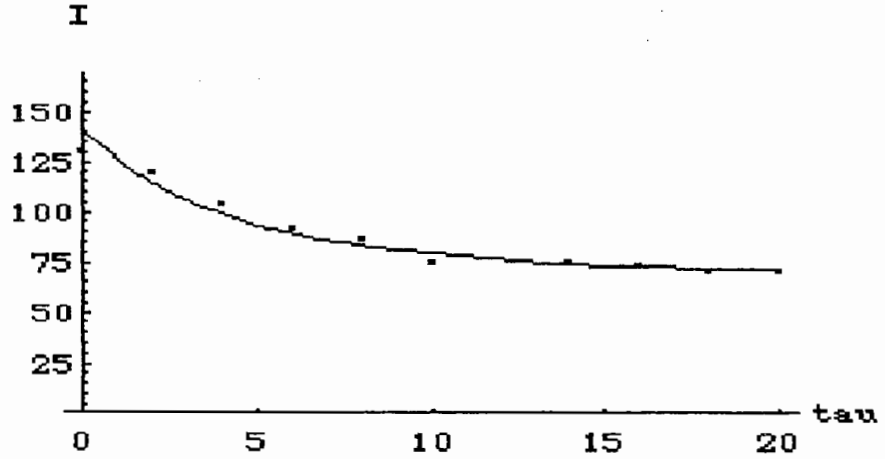
Dr. Bernhard Shapiro
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto, CA 94303

(received 5/13/92)

Synchronous Nutation the better way to measure cross relaxation .

Dear Dr. Shapiro :

Recently G. Bodenhausen introduced an ingenious and outstanding method - the so called synchronous nutation experiment[1] - to measure cross relaxation effects between two spins. The benefit of the new technique is thereby due to the fact that this type of measurements is undisturbed by the entire surrounding of the spins.
As we are since a long time interested in measuring intermolecular cross relaxation effects - K. Lendi at the University of Zürich has formulated a detailed description of this dipolar relaxation effect - we tried this new type of experiment. For a solution of 0.5 % acetone in $CDCl_3$ which contained 0.8% of TMS the synchronous nutation experiment between TMS and acetone is shown below. The observed decay stemming from the auto relaxation ρ as well as the cross relaxation σ is fitted and leads to σ.



As far as we can see ,this powerfull tool should enable us together with K Lendi to have further progress in the description of intermolecular cross relaxation effects.

Yours sincerely

H.Sterk G.Zieger R.Konrat

[1] I.Burghardt, R.Konrat, S.Vincent and G.Bodenhausen, Angew.Chem.(Int.Ed.) in press

Lawrence Livermore National Laboratory

Dr. B. L. Shapiro
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto, Ca 94303

May 11, 1992
(received 5/13/92)

WIDELINE PROTON NMR STUDIES OF ZEOLITIC TUFF ROCK SAMPLES FROM NEVADA TEST SITE.

Dear Barry:

We are continuing our study of water in zeolitic tuff rock samples from Nevada. Our interest is in quantitatively determining the amount of non-pore and pore water in these samples. I expected that the two types of water could be distinguished by linewidth considerations. In this type of rock, non-pore water should exhibit a Gaussian shaped line of 10-30 kHz, whereas pore water would be more like adsorbed water with a linewidth of 1-2 kHz. We have examined a couple of hundred samples from Nevada and in all cases, the dominant observation is that of a single Gaussian line with a FWHM varying between 15 and 20 kHz. At most, pore water, indicated by a small "blip" superimposed on the top of broad Gaussian was a percent or less of the total water. We are interested in comparing our nmr results with neutron-logging data which measures total proton content down-hole. Our nmr results, for total water content, are approx. 1/3 of the neuton-logging values. One of our main concerns is that by bringing the rocks to the laboratory we have lost any pore water that might be present. Attempts to "mimic" pore water by saturating the rocks with water have failed to reveal a "pore water" signal. Instead we observe an exchanged narrowed signal that is still appreciably broader than 1-2 kHz. This is in contrast to saturation experiments with sand where we do observe a 1-2 kHz signal. Pumping gently on the rocks, however, does yield a narrow component superimposed on the broad Gaussian. This signal is slowly unstable, reverting back to the broad Gaussian.

The principal water binding agent in these rock samples is the zeolite clinoptilolite. Its water binding capacity is large, 15% by weight or greater. The proton lineshape of clinoptilolite is Gaussian, ca. 20kHz FWHM at 15% saturation. We believe that the zeolite absorbs any pore water and results in our observing only broadlines. As an aside, we examined the proton linewidth of clino. as a function of water content and dehydration temperature. Temperature studies to 700°C, where the zeolite is heated overnight at a given temperature and then measured at room temperature, reveal an increase in linewidth followed by a decrease and then a further increase -- indicative of three types of water molecules. Further studies using ^{29}Si and ^{27}Al MAS and CPMAS are in progress to examine the effect of temperature and water content on the zeolitic structure.

We have also been the recipients of a Chemagnetics CMX 300. Our Tritium Facility was closed unexpectedly last summer and this new spectrometer was on order. Fortunately, I was able to convence management that we could make good use of it. We are still in the learning stage, but hope to do some interesting experiments with the CRAMPS probe.

Sincerely, Ray

Raymond L. Ward

What's the difference between the new CMX spectrometer and the competition?

We took the complicated and made it simple.

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- Choice of SUN or Silicon Graphics computer
- X-Windows™ software on the spectrometer
- High-speed communications for "real time" display

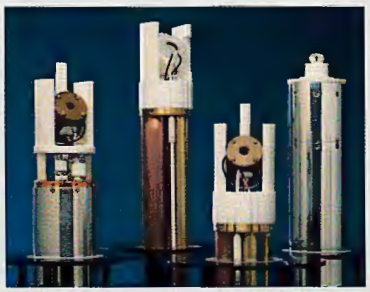
Advanced Console Design
- Full *channel modularity*
- Full channel expandability
- Easy customer access

RF Design
- High IF design provides clean RF
- Ultra fast amplitude, phase, and frequency control
- Direct digital synthesis

Digital Design
- High-speed 100 MHz pulse programmer on each channel
- Complete pulse program flexibility
- Multiple expansion capability

Solids Probes System
- PENCIL™ spinning system for high stability MAS
- Constant spinning speed from – 150°C to + 250°C
- COAX design affords superior tuning and RF capability for multiple resonance experiments
- High-stability, microprocessor-controlled, speed controller

Ergonomics
- Superior comfort, functionality and increased work space for optimum productivity



Chemagnetics

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Dedicated to Design Excellence

Chemagnetics Workshop Set For August 7 & 8

Dear NMR Colleagues:

I'd like to invite you to an NMR workshop that you won't want to miss -- especially if you're attending the Rocky Mountain Conference. Each year, following the conference in Denver, Chemagnetics invites a number of NMR scientists to give seminars on advanced topics and organizes a 2-day opportunity for spectroscopists to visit and get up-to-date on the latest in solid-state NMR. This year's workshop will be held August 7 and 8, 1992 in Fort Collins, Colorado.

We are privileged to have luminaries from both academia and industry. Below is a list of seminars that will be presented on Friday. Saturday will feature hands-on demonstrations of advanced solids techniques, including DOR, fast MAS, and liquids microimaging.

Speakers and topics for the Friday session:

• **Dr. Brad Chmelka,** UC - Santa Barbara	Practical Applications of 2-D exchange NMR in Polymers
• **Dr. Jim Haw,** Texas A&M University	Recent Developments in MAS NMR
• **Dr. Dave Duff,** Raychem Corporation	Triple Resonance Experiments on Protonated and Fluorinated Polymers
• **Dr. Robert Botto,** Argonne National Laboratories	NMR Imaging: A Chemical Microscope for the Analysis of Heterogeneous Materials
• **Dr. Lucio Frydman,** UC - Berkeley	NMR Imaging of Flow
• **Dr. Robert Tycko,** AT&T Bell Labs	New Approaches to Structure Determination in the Solid State
• **Dr. Ann McDermott,** Columbia University	Applications of Rotational Resonance in Enzyme-active Sites
• **Dr. Hellmut Eckert,** UC - Santa Barbara	Heteronuclear X-Y Double Resonance NMR Experiments in Solid Inorganic Materials
• **Dr. Gerald S. Harbison,** SUNY	Solid-State NMR of Highly-Oriented DNA Fibers and Drugs Bound to DNA

If you would like to attend, I urge you to register today. Registrations are limited to 60 people on the first day and 15 for day two. There is no fee to attend the seminar, but registrants are responsible for their own transportation from the Denver airport to Fort Collins (approx. $13), for the hotel, and meals. To help defray your costs, rooms at Chemagnetics' discount rate of $34 have been booked at the nearby Ramada Inn. Please call to register today at 1-800-4-OTSUKA and ask for Jan Anderson.

I look forward to seeing you.

Robert A. Wind

Robert Wind
Director of Research and Development

Postdoctoral Position

The Research and Development Department at Chemagnetics has a postdoctoral opening for a Ph.D. NMR spectroscopist. Experience with solids and NMR and/or MRI techniques and hardware is required. The work involves the development and evaluation of novel solid-state techniques in biosystems. The successful candidate must be interested in working in a problem-solving environment and be able to work as a member of a team.

Interested candidates should send their resume to: D. Hasler, Chemagnetics, Inc., 2555 Midpoint Drive, Fort Collins, CO 80525. For further information, contact Robert Wind (303) 484-0428. EOE

BAYERISCHES FORSCHUNGSINSTITUT
FÜR EXPERIMENTELLE GEOCHEMIE UND GEOPHYSIK
UNIVERSITÄT BAYREUTH

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

Bayreuth, May 5, 1992
(received 5/11/92)

Cross polarisation from ^{19}F to ^{113}Cd

Dear Dr. Shapiro,

recently, we were able to show that cross polarisation from ^{19}F to e.g. ^{29}Si (in combination with other high-resolution solid-state NMR techniques) is a very powerful tool for the investigation of fluorine-doped silicate and aluminosilicate glasses [1,2]. Such fluorine-doped silicate systems are important both in the geo-sciences and in materials research.
Somewhat less relevant in geo-sciences but still an important inorganic class of compounds are cadmium-containing glasses. Again, the simultaneous presence of cadmium and fluorine in these systems is fairly common. This prompted our attempts to increase our "collection" of useful, tested $^{19}F \rightarrow$ X-nucleus CP MAS combinations, now including the combination $^{19}F/^{113}Cd$. The most suitable compound for finding this on-resonance Hartmann-Hahn condition seems to be CdF_2. The $^{19}F \rightarrow {}^{113}Cd$ CP MAS spectrum of CdF_2 is shown in figure 1, together with the ^{19}F-coupled MAS ^{113}Cd single pulse spectrum.
This clearly shows that the investigation of inorganic Cd-F glasses (and of crystalline compounds) of relevant composition by means of $^{19}F \rightarrow {}^{113}Cd$ CP MAS methods should be a promising approach.

Best regards from Bayreuth,

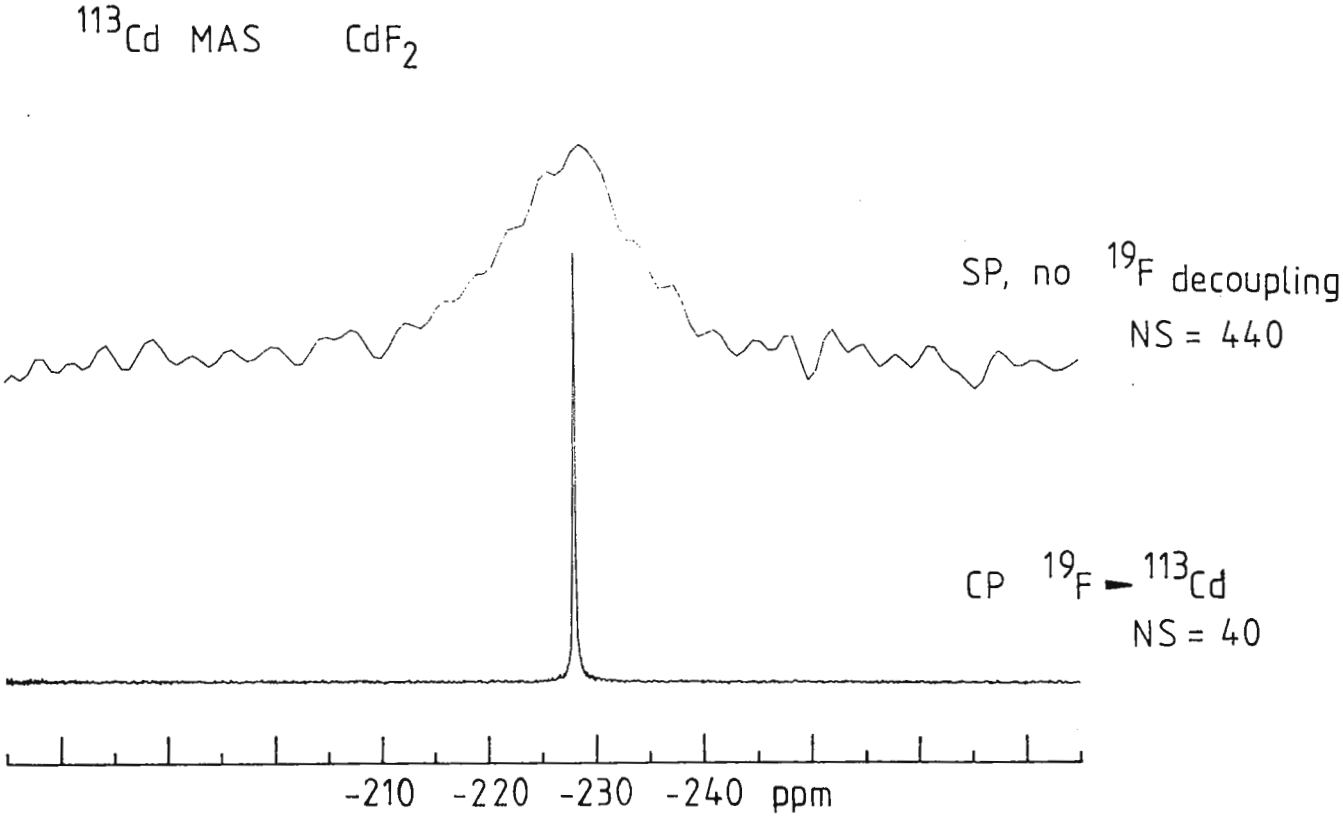
Angelika Sebald

Dr. Angelika Sebald

[1] A. Sebald, L. Merwin, T. Schaller and W. Knöller; J. Magn. Reson. 96, 159 (1992)

[2] T. Schaller, D. B. Dingwell, H. Keppler, W. Knöller, L. Merwin and A. Sebald; Geochim. Cosmochim. Acta 56, 701 (1992)

Fig. 1 ^{113}Cd single pulse MAS (top) and $^{19}F \rightarrow {}^{113}Cd$ CP MAS spectra of CdF_2

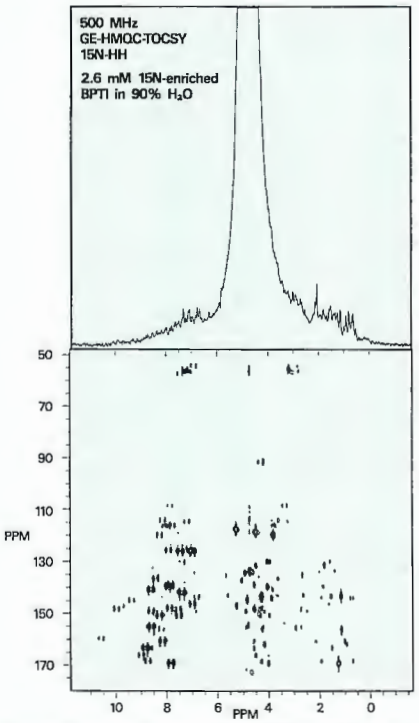


Gradient Enhanced Spectroscopy: a new, practical answer

By Frank Huang, PhD and Paul Calderon, MS

Making Gradient Enhanced Spectroscopy (GES) a viable method for high resolution spectroscopy has long been of interest to researchers. The obvious benefits in speed and information content were too often overshadowed by the drawbacks of signal loss and distortion.

Technology developed at GE NMR Instruments has overcome these challenges. The S-17 Gradient Enhanced Spectroscopy Accessory with integrated inverse probehead makes GES practical for a broad range of applications in 1D, 2D, 3D and 4D experiments.



Gradient Enhanced Spectroscopy technique applied to an HMQC-TOCSY experiment demonstrates excellent water suppression without the need for presaturation or selective excitation.

Research Implications

- Speed without phase distortion or signal loss.
- Practical for 1D, 2D, 3D and 4D experiments.
- Accommodates proton and heteronuclear GES techniques.

A better design

The use of a three-axis, actively-shielded gradient set allows GE to overcome the inherent drawbacks of previous GES technology —most notably, phase distortion and signal loss:

Active shielding. Eddy current effects are the major source of phase distortion in GES spectra. Active shielding prevents interaction of strong gradients with magnet and shim components—the source of eddy current effects.

Fast, strong gradients. Short gradient pulses of 35 G/cm minimize signal loss during pulse sequences.

Applications advantages

With its integral inverse probehead, the S-17 Accessory can accommodate proton as well as heteronuclear GES techniques.

Gradient fields of 35 G/cm are able to suppress water in aqueous samples and to improve performance in heteronuclear experiments. Other applications advantages:

- Eliminates phase cycle requirements and subtraction error.
- Reduces T1 noise.
- Reduces collection times for 2D, 3D and 4D data sets.
- Provides lineshape independent water suppression in multiple quantum coherence selection experiments.
- Provides lineshape independent water suppression via diffusion differences for large molecular weight samples.
- Improves water suppression in experiments using selective time reversal RF pulses.
- Separates cross-correlation and exchange phenomena in NOESY experiments.
- Distinguishes chemicals inside the cell from those outside in whole cell applications.

For additional information on the S-17 GES Accessory, write to GE NMR Instruments, 255 Fourier Ave., Fremont, CA 94539. Or call toll free:

1-800-543-5934

GE NMR Instruments

Gradient-Enhanced ^{15}N HMQC

The pulse sequence and the coherence pathway diagram for a ^{15}N GE-HMQC are shown in *Fig. 1*. The pulse sequence was a standard ^{13}C GE-HMQC experiment (1) with different gradient amplitudes to account for the difference between the gyromagnetic ratios of ^{13}C and ^{15}N. The 90° proton pulse creates transverse magnetization which evolves into an anti-phase state with respect to J(NH) coupling at the end of the period Δ (where $\Delta = 1/(2J_{NH})$). The antiphase components are converted into heteronuclear zero- and double-quantum coherence by the ^{15}N 90° pulse and the multiple quantum coherences are allowed to evolve during t_1. The 180° ^1H pulse in the center of the evolution period serves to eliminate the ^1H chemical shift evolution, yielding pure ^{15}N chemical shifts along that axis. The zero- and double-quantum signals are then *coherence-order labeled* by the gradient pulses G1 and G2. After conversion into antiphase proton magnetization by the last ^{15}N 90° pulse, the desired components are refocused by the gradient G3 and detected. The application of a gradient pulse results in a phase factor being applied to the magnetization which is dependent upon gradient strength, duration, the distance from the gradient isocenter, the gyromagnetic ratios of the coupled nuclei, and the desired coherence order. The relative amplitudes of the labeling and refocusing gradient pulses will determine the selection of a specific coherence pathway and are calculated to suppress magnetization components arising from the solvent and other protons not coupled to ^{15}N spins.

The fundamental principle of coherence selection using gradients is that for a pathway to be detected, the cumulative phase factor during the acquisition must be zero:

$$G_1P_i + G_2p'_2 + G_3p'_3 = 0. \quad [1]$$

The subscripts denote steps in the pulse sequence where p′ defines a composite coherence order for the heteronuclear case which includes the gyromagnetic ratios of the coupled nuclei:

$$p' = p^1H + (\gamma^{15}N/\gamma^1H)p^{15}N \quad [2]$$

and p^1H and $p^{15}N$ are the coherence orders for the ^1H and ^{15}N spins respectively.

In the coherence pathway diagram, the relevant values of p' are given to the left and the relative gradient areas (gradient strength x duration) are given next to each gradient pulse. The following pathway (shown in *Fig. 1*):

$$H(+1) \rightarrow H(+1)N(0) \rightarrow H(+1)N(+1) \rightarrow H(-1)N(+1) \rightarrow H(-1)N(0)$$

is detected using a 5:5:1 ratio of gradient areas, since according to Equation 2:

$$5(1.1) + 5(-0.9) + 1(-1.0) = 0 \quad [3]$$

where the numbers in the parentheses refer to the composite coherence orders. Using these relative gradient areas, protons not coupled with ^{15}N spins may pass through an alternate pathway:

$$H(+1) \rightarrow H(+1) \rightarrow H(+1) \rightarrow H(-1) \rightarrow H(-1)$$

which results in a net phase factor:

$$5(1.0) - 5(-1.0) + 1(-1.0) = -1 \quad [4]$$

Thus, signals from this pathway remain defocused during the acquisition.

A 2D ^{15}N GE-HMQC spectrum of ^{15}N enriched BPTI is shown in *Fig. 2*. The spectrum was collected using a 5 mm inverse probe on an Omega™ PSG 500 spectrometer equipped with an S-17 gradient accessory. Half-sinusoid-shaped gradient pulses were applied simultaneously along the X, Y and Z axes with a maximum gradient strength of ≈20 Gauss/cm and a duration of 3.5 ms. A matrix size of 2048 × 128 resulted in 3.5 Hz resolution in the ω_2 dimension and 10 Hz in the ω_1 dimension. No decoupling was applied.

Gradient-enhanced experiments provide a viable alternative to traditional phase-cycling methods for the selection of coherence pathways. In cases where the sensitivity is adequate, gradient selection can substantially reduce the collection time in multi-dimensional experiments. The ^{15}N GE-HMQC data presented here has none of the t_1-noise from cancellation artifacts usually present in phase-cycled versions of the HMQC experiment. In addition, since the suppression of the single-quantum signals is done prior to acquisition, the receiver gain may be increased, which results in a substantial increase in signal-to-noise. For these reasons, gradient pulses should be the method of choice for coherence selection in HMQC experiments.

Reference

1. R.E. Hurd and B.K. John, *J. Magn. Reson.* **91,** 648 (1991).

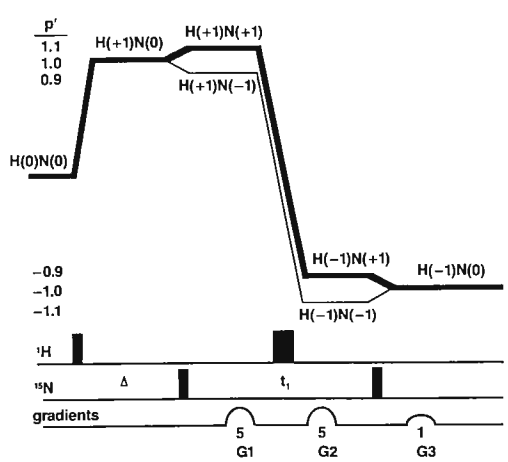


Fig. 1

Pulse Sequence and the coherence pathway diagram for a ^{15}N GE-HMQC experiment.

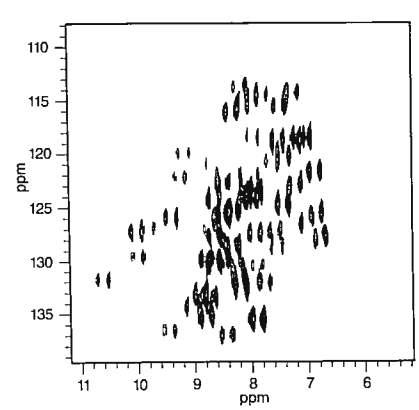


Fig. 2

A 2D GE-HMQC spectrum of ^{15}N enriched BPTI. The sample was 2.6mM in 90% H_2O. The data collection time was 2.6 hours.

Weyerhaeuser

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Dr. Bernard Shapiro
TAMU NMR Newsletter
966 Elsinore Court
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May 8, 1992
(received 5/11/92)

SUBSTITUENT POSITIONS IN GLUCOSE METHYL ETHERS

Dear Barry:

In our on-going work on lightly methylated cellulosic pulps, we thought that normal CP/MAS spectra could reveal some information about the positions of methyl substitution in anhydroglucose (AHG) rings in cellulose. Previous investigators attacked this by acetylating the unsubstituted positions in methyl celluloses and studying the ^{13}C NMR spectra in DMSO or $CDCl_3$ solution (1). This is fine, but we prefer to obtain data directly on intact solid samples.

The chemical shift of an AHG carbon should change by about + 9 ppm when its hydroxyl becomes methoxyl, while its immediate neighbors in the ring ought to experience slight upfield shifts. As a means of verifying these shifts and seeing what underlying subtleties might be involved, we methylated the model compound β-methyl-D-glucoside *via* heterogeneous reaction with diazomethane in ether (9 days, 4° C). The product was taken up in D_2O and its ^{13}C and APT spectra were taken on a QE-300 at Pacific Lutheran University.

About 15 % of the material contained methyl ethers other than the glycosidic methyl group. These appeared to be about equally distributed among the 6, 3 and 2 positions, with a smaller amount at the 4 position. Each etherified ring carbon exhibited three or more signals within a 0.5-ppm range, due to additional methyl substitution at other positions in the same ring. The main assignments and chemical shift effects listed below will assist us in the CP/MAS work. Note the constant β effect of methyl substitution and the small γ effect at carbons next to the substitution site.

^{13}C NMR Chemical Shifts and Substituent Effects in Methylated β-Methyl-D-glucoside.

Unreacted		Directly Substituted			Remote Subst'n. (Neighbor)		
Carbon	Shift	Carbon	Shift	Δδ (β-Me)	Carbon	Shift	Δδ (γ-Me)
1	104.0				1(2M)	104.0	≤ 0.1
3	76.7	3M	86.1	9.4			
5	76.6				5(4M)	76.0	- 0.6
2	73.9	2M	83.4	9.5	2(3M)	73.3	- 0.6
4	70.5	4M	80.2	9.7	4(3M)	69.9	- 0.6
6	61.6	6M	71.9	10.3			
1-OMe	58.0	6-OMe	58				
		3-OMe	59.5				
		2,4-OMe	60.8				

Note: M indicates position has methyl ether. 2(3M), for example, denotes carbon 2 unsubstituted with methyl ether at carbon 3.

Best regards,

Larry Amos

Larry Amos

1. Y. Tezuka, K. Imai, M. Oshima and T. Chiba, *Macromols.*, **1987**, *20*, 2413.

405-18

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Strasbourg, le ??! May 29, 1992
(received 5/4/92)

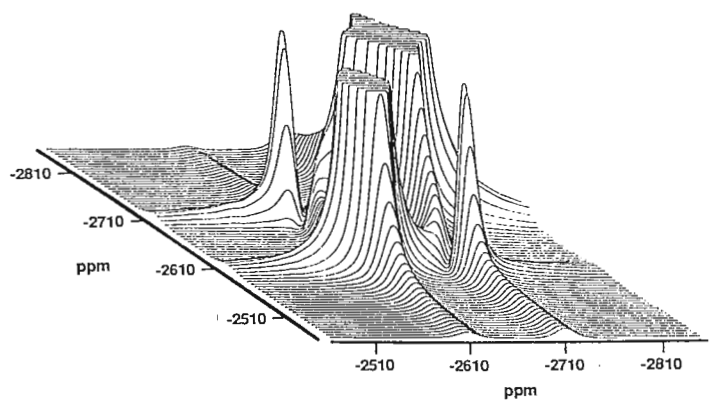
Prof. B. SHAPIRO
966 Elsinore Court
PALO ALTO CA 94303
U.S.A.

Help for 2D Spectra Simulations

Dear Barry,

We have observed for the first time a COSY spectra between cobalt atoms in a cluster. A stack plot is shown below. We have several proofs that the cross peaks are not artefacts and that we have effectively a coupling constant between our cobalts. This coupling constant is now measured using published methods which have been seriously improved.

When we observed for the first time this two dimensional spectrum, we have try to simulate this 2D experiments. Our programs were unable to handle such a spin system.



We have contacted some scientists who have made interesting approaches such as a spin 1/2 coupled to a cobalt which reflect some features but to our knowledge we have not found a program able to compute homonuclear 2D Spectra for three spins 7/2 (A_2B or A_2X case) and which takes into account the relaxation processes which involve T_1Q, $T_{1\ CSA}$, and T_1/T_2 of the second kind.

As simulation of other 2D experiments are also of interest for us. This is the reason why we are searching for such programs. Not a trivial case!!

Yours sincerely,

Pierre GRANGER

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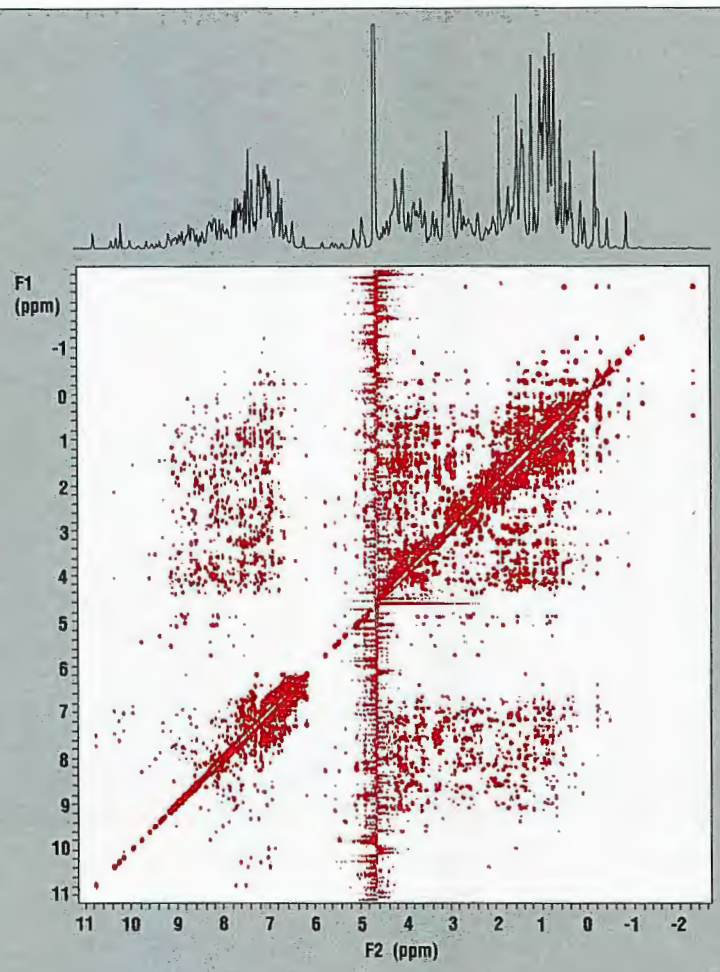

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May 4, 1992
(received 5/6/92)

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

Re: Improvements in MARDIGRAS to Obtain Accurate Distances from 2D NOE Spectra

Dear Barry:

For the past several years, we have spent considerable effort to develop and utilize complete relaxation matrix analysis to obtain ever more accurate distances from proton homonuclear 2D NOE spectra.[1,2] Our effort in recent years has centered on the MARDIGRAS algorithm.[3] We have supplied copies of the programs to several labs, but improvements are continually being made, so it may be worthwhile to obtain updated versions. A big change was made some months ago which has had an important impact.

Although MARDIGRAS utilizes an initial guess for a molecular structure, good distances can still be obtained with a very poor initial model.[3] Indeed a similar program termed MIDGE has recently been described;[4] it was demonstrated that arbitrary initial intensities can be utilized, yielding good distances. However, work with MARDIGRAS has shown that the accuracy of the distances obtained increases with (a) fraction of experimental cross-peaks observed, (b) improved signal-to-noise, and (c) accuracy of initial model structure.[3,5] MARDIGRAS also estimates upper and lower bounds for the calculated distances. The distances and bounds can be utilized in distance geometry and restrained molecular dynamics calculations. Use of the complete relaxation matrix methodology permits longer mixing times to be employed, with consequently larger intensities for weak cross-peaks and the possibility of measuring more distances and longer distances (up to 6 Å).

Several resonances in the 2D NOE spectrum are usually not resolved. Many of these manifest averaging either due to molecular motions or peak overlap; methyl protons, methylene protons, and pseudosymmetric protons in aromatic rings are typical examples. The usual practice is to adopt the pseudoatom approach: the distance from a particular proton to the geometric center of the averaging group is estimated, and a correction factor is added (typically 1-2 Å for pseudosymmetric aromatic protons, 0.3-1.5 Å for methyls, and 0.3-1 Å for methylenes) to the upper bound distances. Inclusion or neglect of averaging effects explicitly in evaluating the distance will influence the particular value of the distance derived from 2D NOE intensities as well as choice of distance bounds.

Simulating 2D NOE spectra using a model structure, even with local motions, is not too difficult, but extracting geometric average distances from cross-relaxation rates in the presence of internal motions is arduous. Commonly, internal motions will either be much faster or much slower than the overall molecular tumbling, leading to $\langle r^{-3} \rangle$ averaging or $\langle r^{-6} \rangle$ averaging, respectively, and knowledge of the exact rate of internal motion is not required; but the orientation of the rotational axis relative to the interproton vectors, which is generally not known, affects spectral densities and consequently cross-relaxation rates. An iterative fitting routine will find the best value of the distance to the geometric center consistent with the limited orientation information.[6] This procedure has been implemented with MARDIGRAS, permitting choice of different motional models which depend on how much is known about the structure. Inclusion of internal motion or overlap averaging was found to improve the accuracy of distances calculated between the geometric centers of methyl protons, methylene protons, and pseudosymmetric aromatic protons, and single protons. This is demonstrated by results listed in the following Table. Utilizing a simulated data set (with realistic random noise and peak overlap) for the protein BPTI, distances are calculated via MARDIGRAS either with or without consideration of internal motions.[6] Of course, it goes without saying that the relaxation matrix approach will nearly always give a more accurate distance than the usual two-spin approximation; but inclusion of motional effects will yield further improvements. In addition, MARDIGRAS calculates upper and lower distance bounds for

any of the averaging models chosen, using worst case geometries; so bounds in DG and rMD calculations can be selected in a logical fashion.

Sincerely yours,

He Liu

Thomas L. James

1. Keepers, J. W. & James, T. L. *J. Magn. Reson.* **57**, 404-426 (1984).
2. James, T. L. *Curr. Opin. Struct. Biol.* **1**, 1042-1053 (1991).
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4. Madrid, M., Llinas, E. & Llinas, M. *J. Magn. Reson.* **93**, 329-346 (1991).
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6. Liu, H., Thomas, P. D. & James, T. L. *J. Magn. Reson.*, in press (1992).

Table. Comparison of a random sampling of geometric average distances (Å), obtained with and without consideration of internal motions, using a simulated 2D NOE spectrum of BPTI.[a]

proton interaction	experimental intensity	model structure distance	calculated distance isotropic motion only	calculated distance plus internal motions
H – methyl				
ile-19 H – ile-19 MD1	0.01137	3.81	3.53	3.81
cys-5 HB2 – leu-6 MD2	0.00285	4.70	4.34	4.90
H – aromatic				
phe-45 HZ – phe-4 RE	0.08902	4.07	2.22	4.07
tyr-10 H – phe-33 RD	0.00194	5.06	4.51	4.79
H – methylene				
glu-7 H – leu-6 QB	0.00741	3.81	3.55	3.82
phe-45 H – tyr-21 QB	0.00846	3.90	3.44	3.79
methyl – methyl				
ala-16 MB – ile-18 MD1	0.00782	4.11	3.57	4.30
leu-6 MD1 – leu-6 MD2	0.02561	3.16	2.91	3.26
methyl – aromatic				
leu-6 MD2 – tyr-23 RE	0.00411	5.90	3.91	6.02
ala-25 MB – tyr-23 RD	0.00575	5.60	3.72	5.47
methylene – aromatic				
tyr-21 QB – tyr-21 RE	0.00332	4.52	5.17	4.97
gly-56 QA – tyr-23 RE	0.01645	4.64	3.04	4.68
methylene – methyl				
gly-56 QA – met-52 ME	0.01698	3.53	3.10	3.62
leu-6 QB – leu-6 MD2	0.01780	3.40	3.14	3.55

[a] The simulated spectrum was generated for a mixing time of 160 ms using CORMA assuming the real structure is the crystal structure of BPTI, an overall tumbling time of 2.0 ns and internal motion or overlap averaging of methyls, methylenes, and symmetric aromatic protons. The subset of 611 cross-peak intensities used for input to MARDIGRAS corresponds to those observed experimentally. MARDIGRAS calculations utilized the crystal structure as the initial model structure, so only the effect of neglecting internal motions could be examined.

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Professor B. L. SHAPIRO
966 Elsinor Court
Palo Alto
California 94303 - Etats Unis

Dear Dr Shapiro

Theix, 1992 may 4
(received 5/9/92)

ASSESSMENT OF WATER DISTRIBUTION BY NMR MICRO-IMAGING IN GELATINE DURING DRYING

The relationship between effective water diffusivity (D_{eff}) and water content in solids can be calculated from the evolution of the water content profiles during drying. Shrinking, especially marked in biological materials, must be taken into account in the calculations. An experiment was designed to promote unidirectional drying - water migration and shrinking - in gelatine samples. NMR micro-imaging was used to record 2D-images or 1D-projections of gelatine water distribution in the samples. This method was chosen because it is non-destructive and provides high resolution.

Just one side of the parallelepipedic samples (OZ = 3 cm, OY = 1 cm, OX = 1.5 cm) underwent drying. The air was blown in the Z direction, and parallel to the plan ZOY, at 30°C and about 1 m/s for 2 hours. NMR experiments were recorded on a Bruker AM400 spectrometer equiped with the AM imaging accessory. A 25 mm saddle-shape coil was used. Images and projections of a 2 mm slice thickness were obtained at 10 min intervals during 2 hours of drying with a standard spin echo sequence. The slice orientation was perpendicular to the air stream and to the direction of the static magnetic field. Spin-lattice and spin-spin relaxation times dependence of water content of gel were previously determined in order to optimize imaging parameters. The image matrix was 32 x 32 pixels, giving a pixel size of 780 µm and an acquisition time of 5 min. The projections were accumulated into 1K, resulting in a resolution of 24 µm between two points and an acquisition time of 10 s.

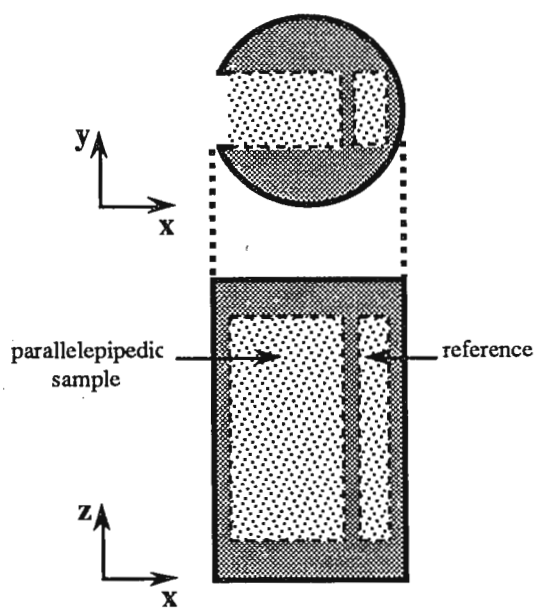
Image evolution during drying of gel having an initial water content of 75% wet basis was recorded. The image projection was split into different parts according to the OY direction. Each part had the same behaviour proving that both shrinking and water migration occured mainly in the X direction.

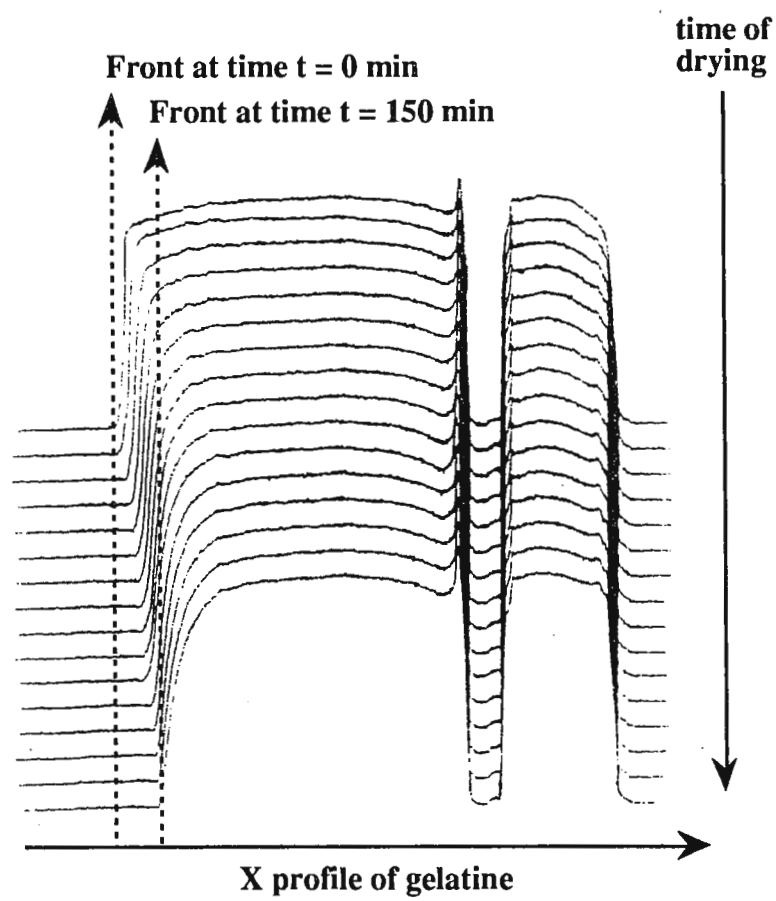
Projections in the X direction were obtained for three initial water content : 85, 75 and 65%. A stacked plot of these projections versus drying time is displayed for a water content of 75%. The water content profiles were calculated from the projections by assuming the signal intensity proportional to the volumetric water content. The shrinkage of the samples was measured accurately : the thickness varied from 15 mm to less than 14 mm after 2 hours of drying. The water content gradients were very steep in the first 3 mm below the surface while the deepest part ($\Delta X = 7$ mm) opposite to the dried face was unaffected by drying.

The experimental procedure set up in this study with the model material (gelatine) will be applied to determine D_{eff} in various biological materials such as foodstuffs.

Sincerely yours

L. FOUCAT J.D. DAUDIN J.P. RENOU





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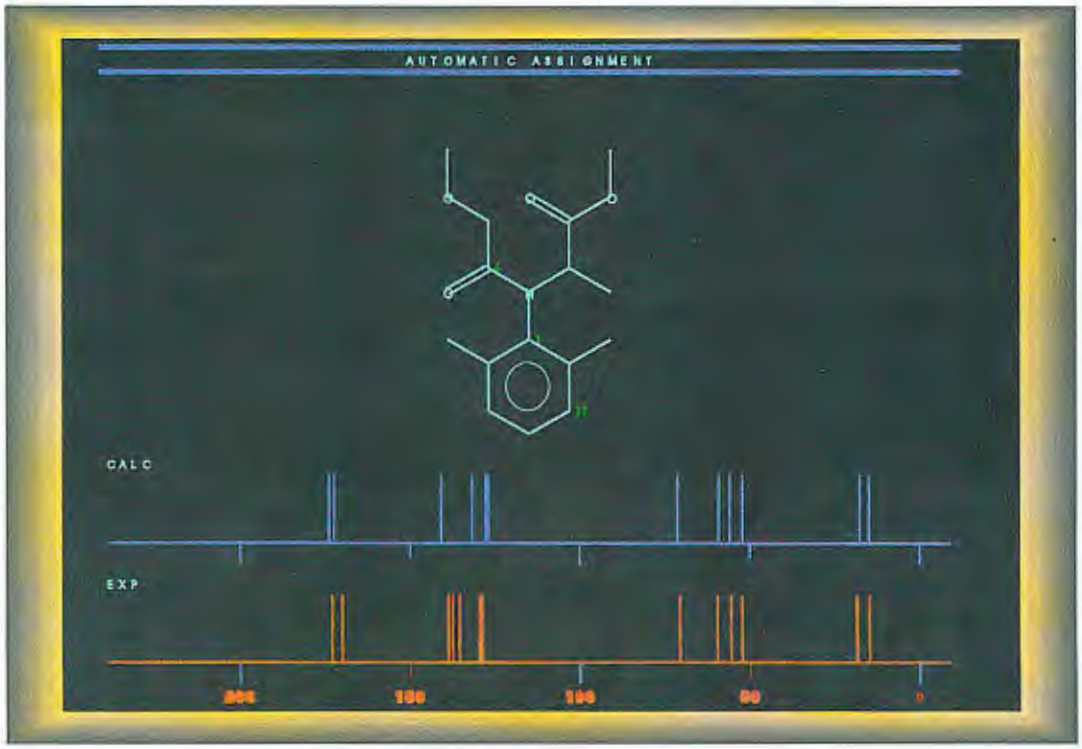

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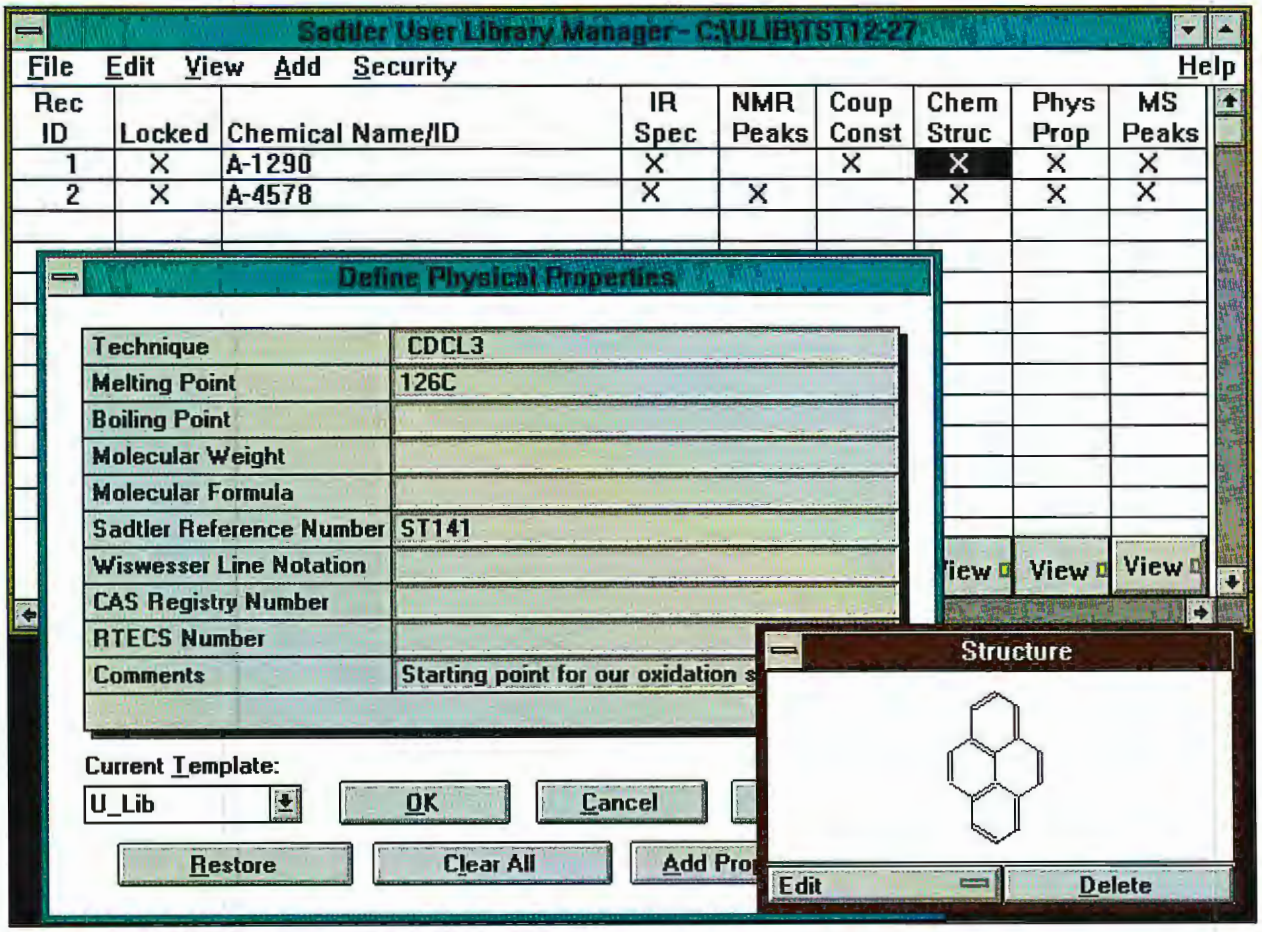

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CONFORMATIONAL ANALYSIS AND ABSOLUTE CONFIGURATION OF FOUR NATURAL PERYLENEQUINONE PIGMENTS

Milano, May,19th 1992

(received 5/24/92)

Dear Barry,

Perylenequinones form a group of chemically interesting and biologically active pigments, some of which are phytopathogens. They present a photodynamic activity and the interest in these compounds is increasing, because of their possible application in the phototherapy of cancer.

The elsinochromes, like other members of the series, present a strong optical activity, mainly due to the fixed non coplanarity of the extended chromophore, which assumes a helical shape, and also to the presence of asymmetric carbon atoms, i.e. C-13, C-14, C-16 and C-17.

The sign of the axial chirality was established as M(R) by comparison of the CD spectra with that of cercosporin, a natural perylenequinone, the structure of which has been determined by X-ray analysis[1].

The absolute stereochemistry of all the other centers was determined through the conformational analysis of the alicyclic ring and of the side-chains, by using H,H and C,H coupling constants and some NOE experiments.

The equatorial-equatorial preferred orientation of the benzylic protons followed from the very low value (1.5 Hz) of $J(H_{13},H_{16})$ in agreement with a dihedral angle of 80°. This established the absolute configuration of C-15 and C-16 as S for the four compounds. The configuration of the asymmetric centers in the side-chains was established by using the vicinal H,H and C,H coupling constants, i.e. $J(H_{16},H_{17})$, $J(C_{12},H_{17})$, $J(C_{13},H_{17})$ and $J(C_{18},H_{16})$ with the appropriate limiting J values[2]. The populations relative to the three staggered conformers were then obtained by solving the appropriate linear equation $[A][X] = [J_{exp}]$ with the least squares method for each of the two epimers 17R and 17S. The coefficient matrix [A] assumes the following values:

$$[A] = \begin{vmatrix} J^{\alpha}_{H16,H17} & J^{\beta}_{H16,H17} & J^{\gamma}_{H16,H17} \\ J^{\alpha}_{C12,H17} & J^{\beta}_{C12,H17} & J^{\gamma}_{C12,H17} \\ J^{\alpha}_{C13,H17} & J^{\beta}_{C13,H17} & J^{\gamma}_{C13,H17} \\ J^{\alpha}_{C18,H16} & J^{\beta}_{C18,H16} & J^{\gamma}_{C18,H16} \end{vmatrix} \quad [A]_{17R} = \begin{vmatrix} 3.2 & 11.4 & 2.0 \\ 1.1 & 1.1 & 8.0 \\ 8.0 & 1.1 & 1.1 \\ 7.9 & 2.1 & 1.1 \end{vmatrix} \quad [A]_{17S} = \begin{vmatrix} 2.0 & 11.4 & 3.2 \\ 1.1 & 1.1 & 8.0 \\ 8.0 & 1.1 & 1.1 \\ 1.1 & 2.1 & 7.9 \end{vmatrix}$$

The consistency of model and experimental coupling constants can be judged by the fact that the sum of the derived populations is very close to one. In the case of **2**, the values of $J(C_{12},H_{17})$ and $J(C_{18},H_{16})$ allow to distinguish between the 17R and 17S configuration, the best agreement between experimental and calculated Js occurring for 17R.

Therefore, if **2** has the 17R configuration, the epimer **1** must be 17S. The other pair of elsinochromes, **3** and **4**, follows the same line of reasoning.

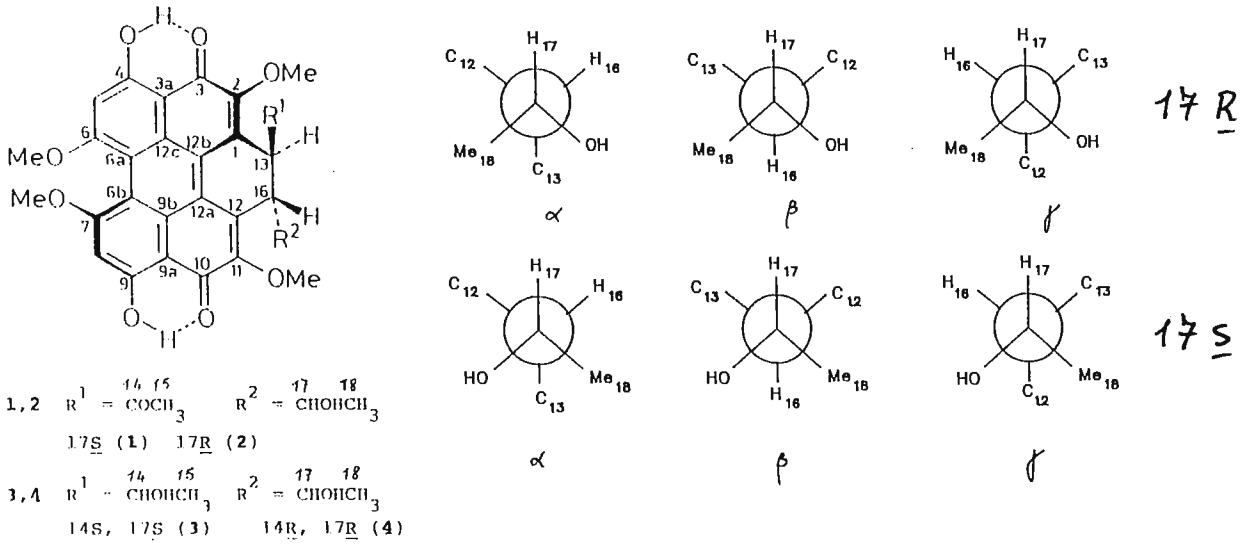
The cross-relaxation rates reported in Table 2 were obtained from 1D-NOESY experiments (irradiation of H-18), by measuring the rate of build-up of NOE values with respect to the mixing time. A τ_{eff} of 0.8 x 10^{-10} s was obtained,as the interproton distance $\langle r^{-6}_{18,17}\rangle$ is known (2.6 Å). The τ_{eff} was used to calculate the σ values between H-18, H-13 and H1-6 in model geometries for both the epimers 17R and 17S, taking also into account the populations of the three main conformers (Table 2).

TABLE 1 - EXPERIMENTAL AND CALCULATED COUPLING CONSTANTS FOR ELSINOCHROMES (1-4).

	1		2		3		4	
	J_{exp}	J(17S)	J_{exp}	J(17R)	J_{exp}	J(17S)	J_{exp}	J(17R)
$J(H_{16},H_{17})$	8.3	8.33	7.0	6.98	8.0	8.03	7.6	7.56
$J(C_{12},H_{17})$	1.6	1.92	1.9	1.90	1.5	1.79	1.8	1.81
$J(C_{13},H_{17})$	3.0	2.99	4.0	3.78	3.4	3.39	4.0	3.66
$J(C_{18},H_{16})$	2.9	2.56	4.0	4.23	2.7	2.40	3.8	4.16

TABLE 2 - CROSS-RELAXATION RATES $\sigma(s^{-1})$ FOR 17R AND 17S STEREOISOMERS.

	Elsinochrome B_1 (2)			Elsinochrome B_2 (3)		
	σ^{exp}	σ^{calc}_{17R}	σ^{calc}_{17S}	σ^{exp}	σ^{calc}_{17R}	σ^{calc}_{17S}
H18,H13	0.014±0.001	0.053	0.015	0.051±0.002	0.035	0.015
H18,H16	0.043±0.001	0.026	0.017	0.026±0.004	0.020	0.024
H18,H17	0.072±0.010	0.070	0.070	0.068±0.008	0.070	0.070



Thus the 17R epimer, already identified with **2** on the basis of the Js, exhibits the larger cross-relaxation rate between H-13 and H-18, in agreement with the calculated value. On the contrary the 17S epimer (**1**) shows the smaller cross-relaxation rate, together with the smaller calculated value.

[1] U. Weiss, L. Merlini, G. Nasini, Prog.Chem.Org.Nat.Subst. 52, 1 (1987).
[2] M. Barfield, J. Am. Chem. Soc., 102, 1 (1980); M. Barfield et al., ibid., 102, 7 (1980); C.A.G. Haasnoot, F.A.A.M. De Leeuw, C. Altona, Tetrahedron, 36, 2783 (1980).

Yours sincerely

Rosanna Mondelli Enzio Ragg Lucio Merlini

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Switching: 1-20μs

Output: +3 to +13dBm; 50ohm
Spurious Outputs: −70dBc
Phase Noise: −63dBc (0.5Hz-15KHz)

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Switching: 1-20μs

Output: +3 to +13dBm; 50ohm
Spurious Outputs: −70dBc
Phase Noise: −63dBc (0.5Hz-15KHz)

Freq. St'd: OCXO, TCXO, Ext.
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PTS 1000

Range: 0.1-1000 MHz
Resolution: 0.1Hz-100KHz (opt)
Switching: 5-10μs

Output: +3 to +13dBm; 50ohm
Spurious Outputs: −70dBc (0.1-500 MHz), −65dBc (500-1000 MHz)
Phase Noise: −60dBc (0.5Hz−15KHz)

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Initial Results with a Nalorac Micro Indirect Detection Probe

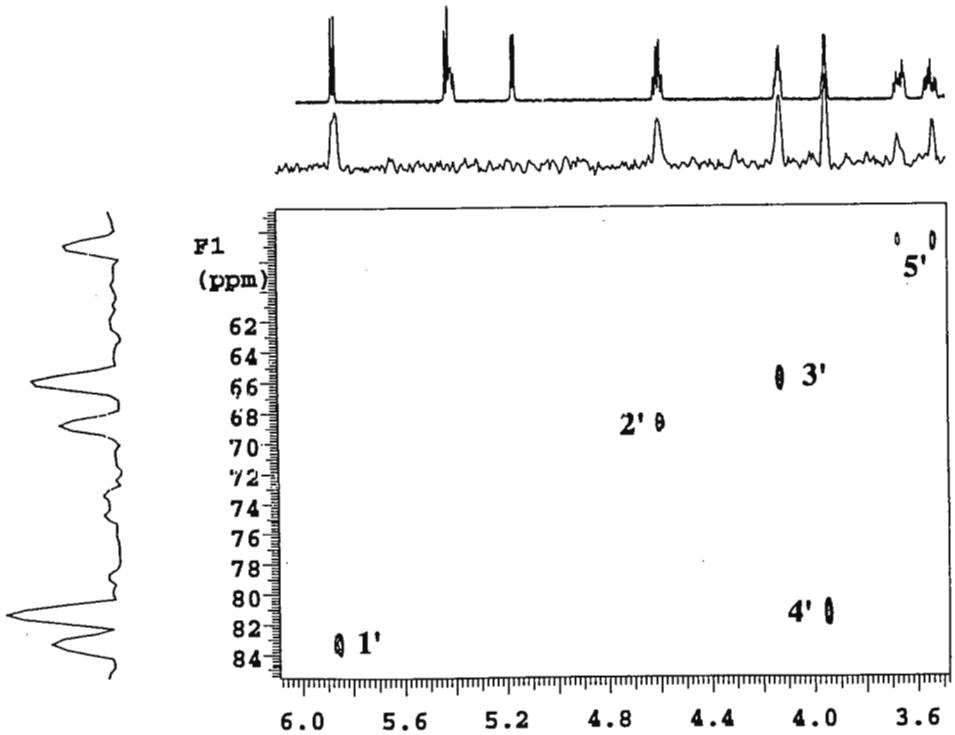
19 May 1992 (received 5/21/92)

Dear Barry,

Toby Zens of Nalorac Cryogenics Corporation dropped by last month with an exciting piece of new equipment : a MID500 Micro Indirect Detection probe for our Varian Unity 500. We were rather skeptical about how much we would gain in performance with small samples over our triple resonance probe, which gives better than 500:1 signal to noise on 0.1% ETB. Our initial results, which were obtained with a 100 μg sample of adenosine in 45 minutes, were quite encouraging! The data plotted below were acquired in 32 transients, for 24 hypercomplex pairs, across 5380 Hz in F_1. The acquisition time was 149 msec, garp ^{13}C decoupling was employed with a B_2 field of 3600 Hz. We find that the ^{1}H 90°'s can be 5 μs, while the ^{13}C 90° tip is about 11 μs.

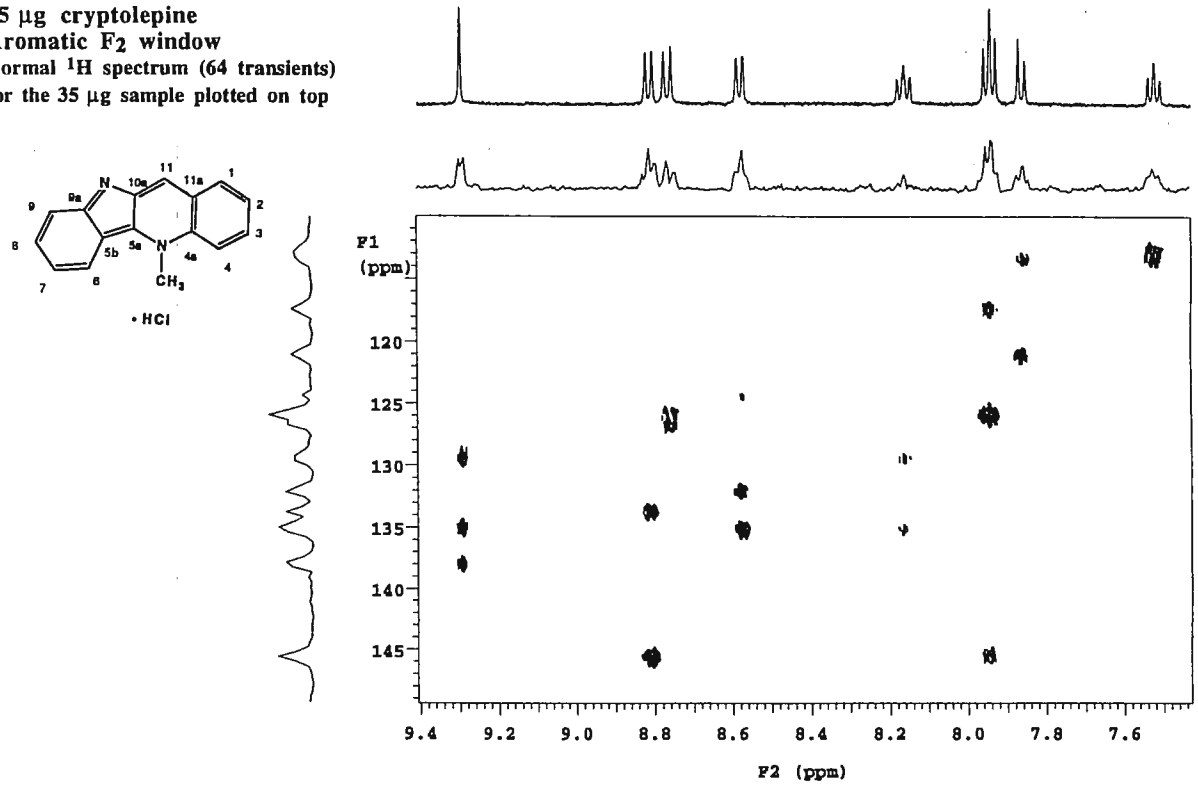
At the very top of the spectra plotted below you will find the results of 32 transients in a normal ^{1}H spectrum for this 100 μg sample. The spectrum was acquired without spinning; the lineshape and resolution are quite acceptable. Projections through the 2D data matrix are also plotted.

Inspection of the projections revealed that we had plenty of signal to noise. Careful comparisons of relatively large samples (150 μg) in this micro probe, with the 5mm probe described above, indicate about a 4 fold advantage in time for equivalent data with the micro probe. This time advantage extends even more as the sample gets smaller.



We are presently "pushing the envelope" with this new beast with respect to net sample size. Depending upon your point of view, another more exciting and generally useful avenue will be to pursue the relative improvements of such experiments as HMBC on ~1/4 mg samples. At present, we can say that so far excellent results have been obtained on several problem samples in this size range with about 1 - 2 hours of instrument time. The digital resolution required in F_1 is the deciding factor; 16 transients are generally sufficient. The smallest sample on which we have acquired HMBC data so far has been a 35 μg sample of cryptolepine HCL. The figure plotted below was obtained in 22 hours. Truly, a revolution has occurred !

Z*SPEC MID500
HMBC - 22 hours
35 μg cryptolepine
Aromatic F_2 window
Normal ^1H spectrum (64 transients) for the 35 μg sample plotted on top



Best Regards,

Ron Crouch, John Shockcor, & Gary Martin

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Indirect Detection ^{1}H Observe, ^{15}N-^{31}P Decouple	ID200	ID250	ID270	ID300	ID360	ID400	ID500	ID600
Triple Resonance Indirect Detection: ^{1}H, (^{13}C, ^{15}N)				IDT300	IDT360	IDT400	IDT500	IDT600
Microsample Indirect Detection				MID300	MID360	MID400	MID500	MID600
Broadband ^{15}N-^{31}P Observe	BB200	BB250	BB270	BB300	BB360	BB400	BB500	BB600
Dual Broadband ^{1}H/^{15}N - ^{31}P Switchable	DB200	DB250	DB270	DB300	DB360	DB400	DB500	DB600
Dual ^{1}H/^{13}C Switchable	D200	D250	D270	D300	D360	D400	D500	D600
^{1}H/^{19}F Observe BB Decoupling of ^{1}H or ^{19}F	H-F200	H-F250	H-F270	H-F300	H-F360	H-F400	H-F500	H-F600
^{1}H/^{19}F Observe	HF200	HF250	HF270	HF300	HF360	HF400	HF500	HF600
Microsample ^{1}H/^{19}F Observe	MHF200	MHF250	MHF270	MHF300	MHF360	MHF400	MHF500	MHF600

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Columbia University *in the city of New York*

Department of Chemistry
Box 555 Havemeyer Hall
New York 10027

Christopher J. Turner
(212) 854 - 2155 Voice
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Monday, May 4, 1992 (received 5/9/92)

Dr. Barry Shapiro, Editor
TAMU NMR Newsletter
966 Elsinore Ct.
Palo Alto, CA 94303

Interleaved Hypercomplex Data Acquisition

Dear Barry:

The major problem in attempting to measure 2-D spectra as fast as possible is a lowering in the quality of the data due to the generation of repetition rate artifacts, sometimes called "Big T_1-noise". If the data are acquired quickly, then the magnetization from one transient can carry over and interfere with the next. In this context, there is a particular problem with the phase-sensitive COSY phase cycle, since it is discontinous when the background phase-cycling needed for absorbtion mode displays is considered.

	First Data-Set		Second Data-Set	
First Pulse	0	2	1	3
Second Pulse	0	0	0	0
Receiver	0	2	0	2

Thus, dummy transients are usually necessary during high speed data-acquisition, in order to avoid interference between adjacent data-sets. However, the idea of adding one dummy transient for every two "real" ones, is horrific, since this would mean wasting a third of the total time.

All we need do to get rid of the dummy transients, is to rearrange the order in which the data are acquired, thus:-

	First Data-Set	Second Data-Set	First Data-Set	Second Data-Set
First Pulse	0	1	2	3
Second Pulse	0	0	0	0
Receiver	0	0	2	2

A detailed account of this work is scheduled to appear in JMR next year.

Best Wishes

Chris

University of Illinois
at Urbana-Champaign

School of Chemical Sciences
1209 West California Street
Urbana, IL 61801

Molecular Spectroscopy Laboratory

May 6, 1992
(received 5/8/92)

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

Soft Pulse in MAS

Dear Berry:

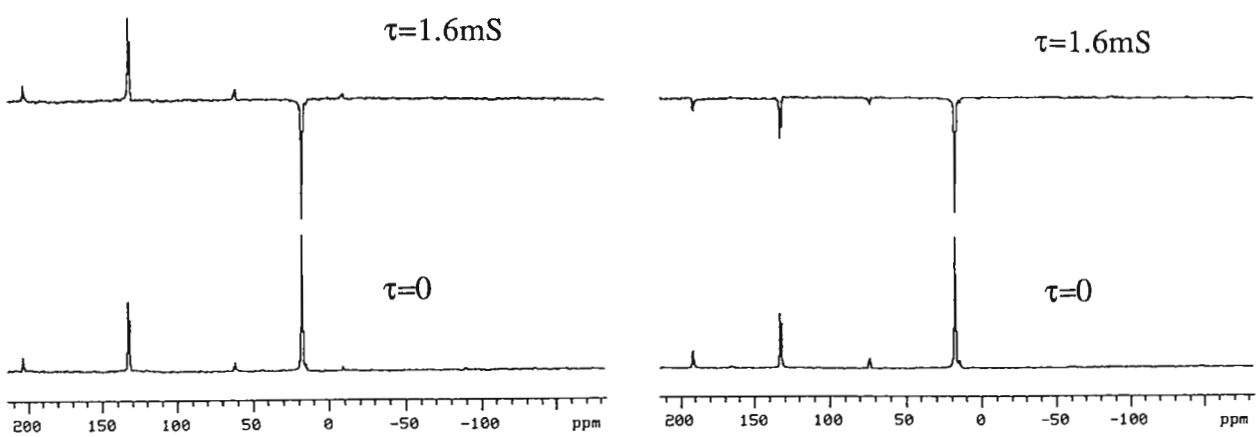
Using soft pulses is the simplest method to selectively excite or invert the spins near the carrier frequency. We show here an interesting phenomena by using a soft pulse on rotating solids. The soft pulse perturbs the spins not only near the carrier but also near the $\pm\omega_r$, $\pm 2\omega_r$ to the carrier frequency (ω_r is the rotor frequency). The effect is caused by the chemical shift modulation due to CSA by the sample rotation. The detail of the theory will be explained in a paper to be published in *J. Magn. Reson.* and it is similar to the theory of rotational resonance of homonuclear coupled pair systems.

This effect is examined by a HMB sample on a 200 MHz spectrometer. After the cross polarization, a soft pulse (rf field strength 1 kHz and duration τ) with 90° phase shift is applied. The carrier is set near the methyl peak, therefore the methyl peak is inverted as shown below, both in (a) and (b). In (b), the chemical shift of the aromatic peak is about $2\omega_r$ from the carrier but not in the case of (a). The match of this condition can be seen from the position of the spinning sidebands from the aromatic peak relative to the carrier. The spectra show that the aromatic peak in (b) is inverted after τ=1.6mS while the same peak in (a) is almost unchanged.

Yours Sincerely

Zhehong Gan

Vera M. Mainz



(a) (b)

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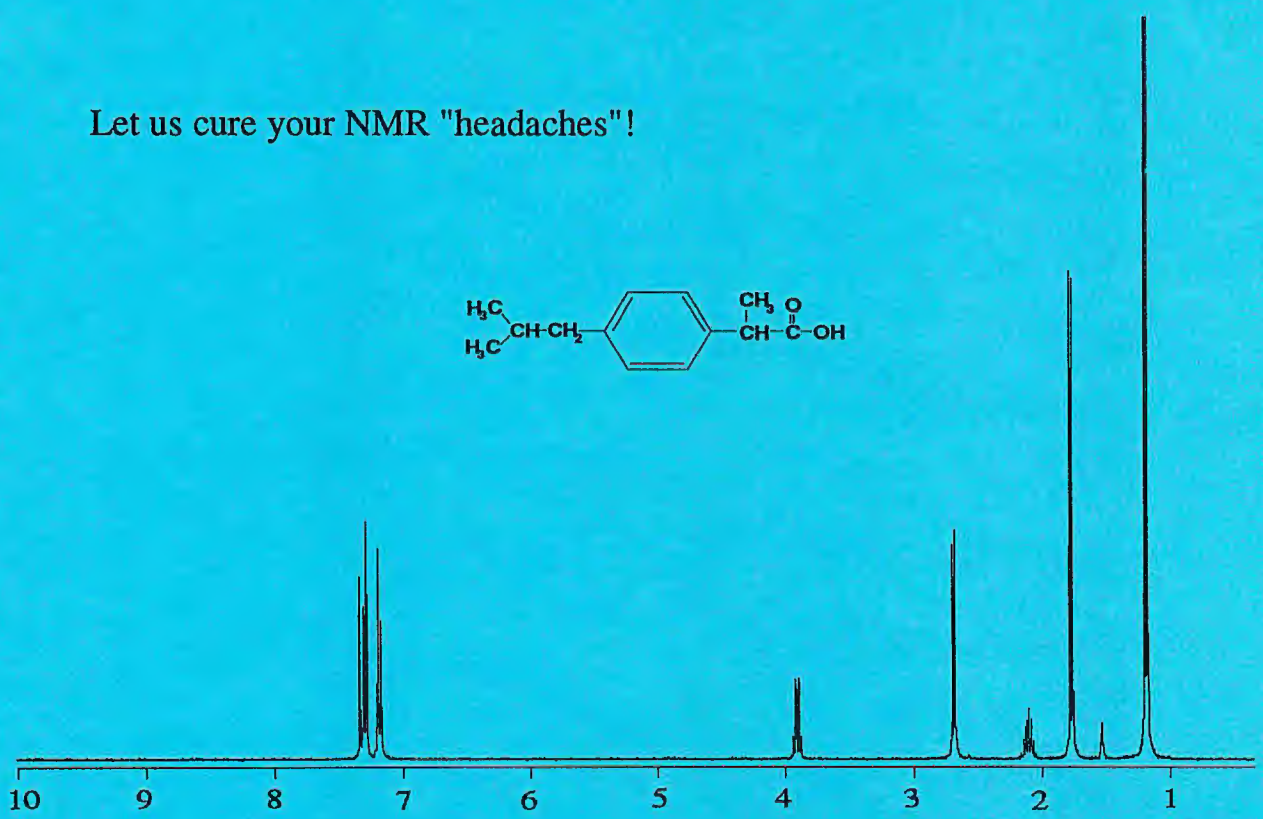

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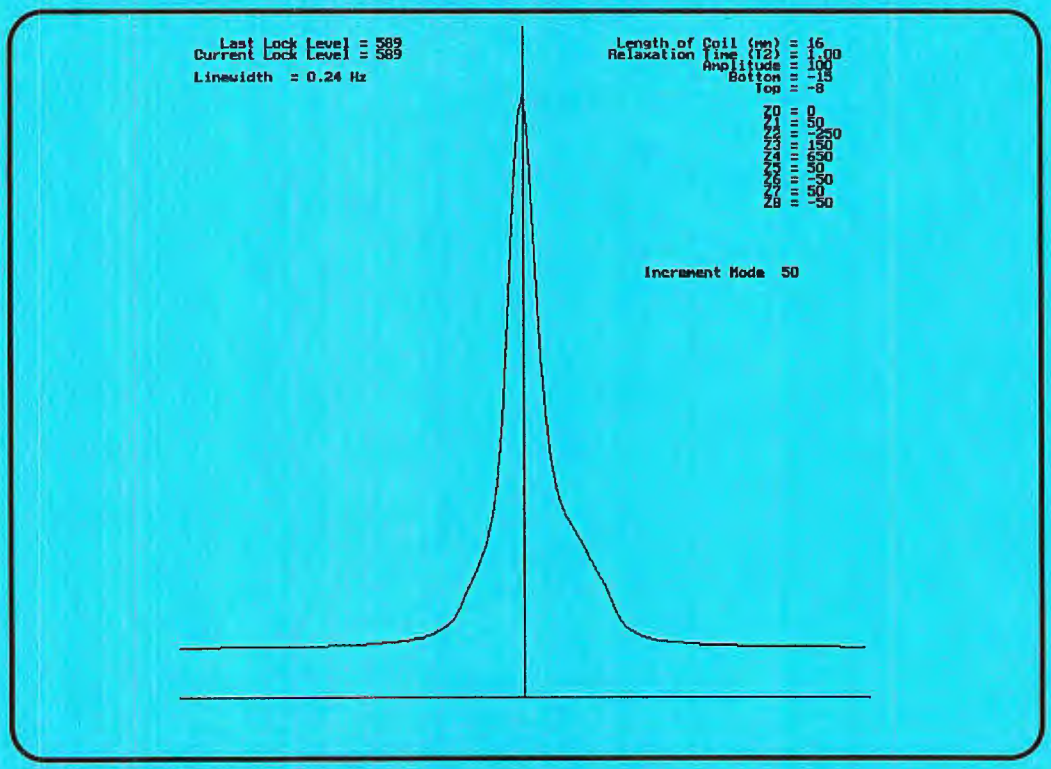

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May 1, 1992 (received 5/22/92)

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

Distributed Magnetic Resonance

— or —

How Many Spectrometers Does It Take to Screw In a Light Bulb?

Dear Barry:

We are in the process of developing echo planar imaging (EPI) on a 2.0 T 18 cm Nalorac magnet which is serviced by a SISCO console. As you know, EPI requires an enormous receiver bandwidth to be effective, and relatively complex signal processing to reconstruct the images. In order to have the best of all possible worlds, we chose to utilize the SISCO console to produce the pulse sequence, and to route the receiver signals to our GE 1.5 T Signa, which is equipped with Hyperscan, GE's trade name for the Advanced NMR Systems EPI accessory. The ANMR accessory has a 2 MHz bandwidth, far greater than that of the SISCO console. Additionally, it processes EPI signals beautifully.

However, it turns out that the Signa receiver cannot handle RF outside its design frequency range, so we were forced to use our homebuilt 6.0 T solid state spectrometer, with a 5 MHz receiver bandwidth, for the demodulation to audio. The audio signals were then routed to the Signa.

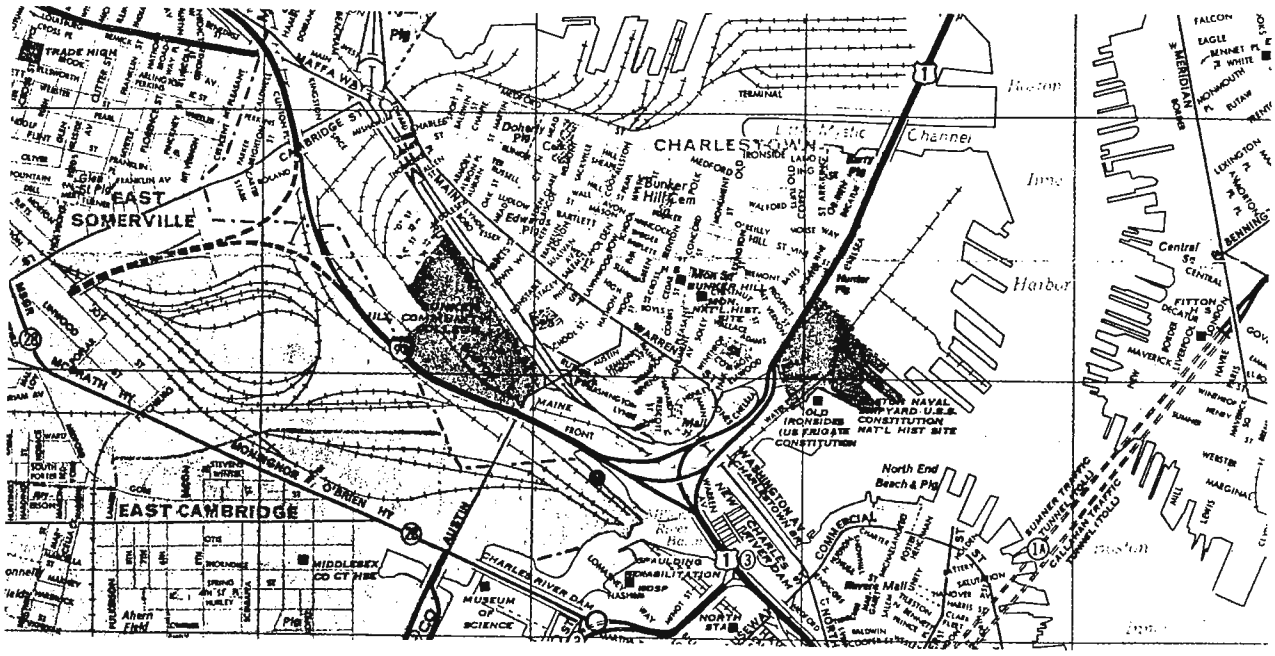
Depending upon how you count it (the GE/ANMR could be considered an NMR within an NMR) we needed three to four machines to obtain one image.

We have also been known to use other, nearly as bizarre, schemes to overcome various shortcomings of a collection of instruments. For example, Greg Moore and Gil Gonzáles have used the Bruker or the Tecmag as an auxiliary spectrometer in association with the Signa for simultaneous multinuclear imaging and spectroscopy (SMIS). On another occasion, the

homebuilt 6.0 T console was used for solids imaging on the 2.0 T magnet before the SISCO console was obtained.

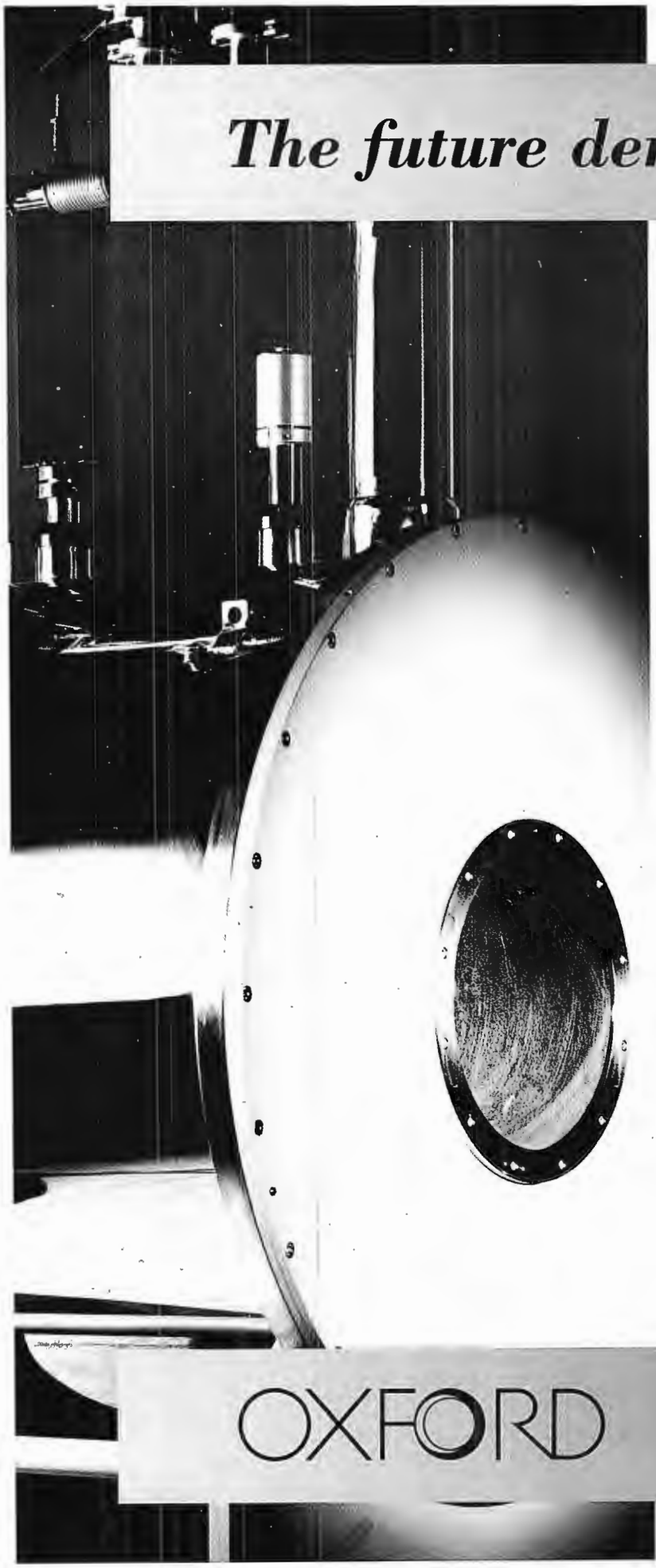
Such efforts suggest the general concept of DMR (distributed magnetic resonance), in which all magnets and spectrometers are part of a network, just like on modern computers, and may share resources. In fact, almost all of our machines are presently linked via masses of coax cable strung in the lab ceiling, enabling any of these hookups to be made fairly readily. For those interested in the details, a diagram of our system appears below. Generally speaking, we find that packet collisions on the network are most frequent around 9:00 am and 5:00 pm, and so we try to avoid these hours. Control and status information is transmitted not on the signal network, but by voice mail (prop open the door and shout).

One may envision various extensions of this concept of DMR. For example, imagine the use of a clinical magnet for tedious multidimensional solid state measurements on samples with long T_1's. We estimate that roughly 1600 narrow bore solid state probes may be placed within the homogeneous volume of the large magnet. With a suitable electronic switching arrangement or multiple massively parallel spectrometers, each probe may be pulsed in rapid succession or even simultaneously, perhaps obtaining the total data set in a time the order of a single FID. Alternatively, completely independent experiments might be run. Now that's throughput! We could probably match the solid state NMR installed capacity of the Western Hemisphere. We are currently seeking industrial partners to help develop this venture.



Best regards,

Robert Weisskoff Leo Garrido James Christensen Chun Zuo Marty Lizak Jerry Ackerman



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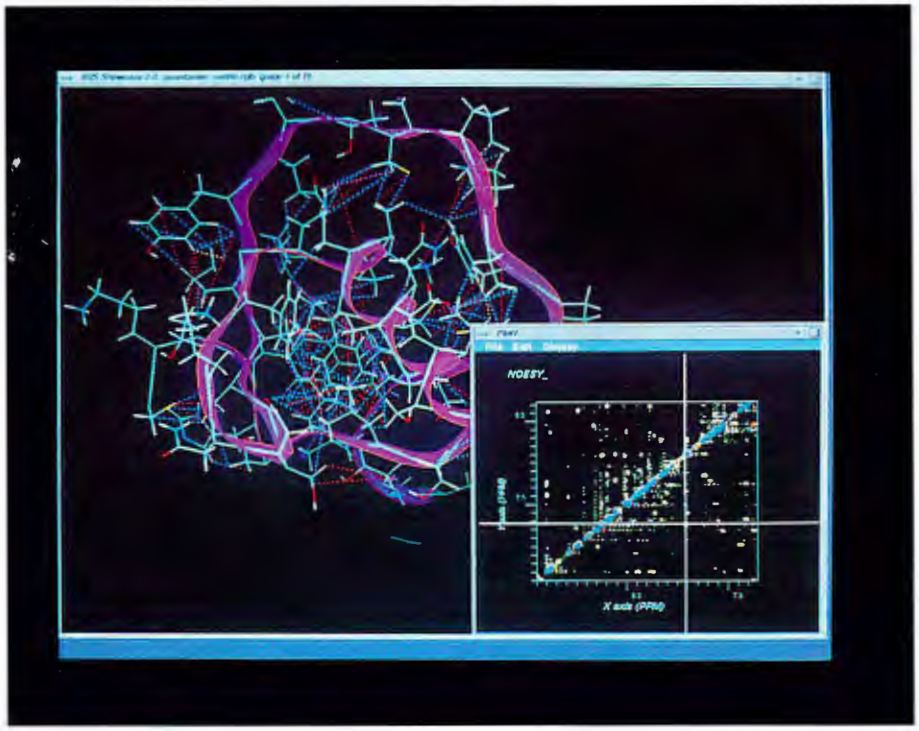
An Integrated System for NMR Structure Determination



Model-assisted NOE assignment through an interactive graphical link between structure and NOE spectrum and a constraint table.

Over the last few years, NMR spectroscopy has gained wide acceptance as an important technique for the determination of solution structure of molecules complementing other methods like X-ray crystallography[1]. The impetus for this has come from significant advances in high-field spectrometers, computers and multi-dimensional homonuclear and heteronuclear spectroscopy with isotopically enriched samples.

The interpretation of NMR data to predict 3-D molecular structures first requires complete assignment of spectra (2D, 3D or 4D) and extraction of the bulk of structural information from NOE data as interproton distances, and scalar coupling data as dihedral angular constraints. These experimental distance and torsional constraints are later used with a combination of distance geometry[2] and restrained dynamics simulations to generate candidate structures that reliably conform with the input data[3].

Spectroscopists now face the challenge of managing a myriad of data during the entire process of structure determination. Molecular Simulations has developed NMR Workbench- an integrated system that combines interactive multi-dimensional spectral analysis and advanced molecular modeling methods to provide a streamlined process of data extraction and structure prediction with increased accuracy and throughput.

Interactive Link Between Spectra and Structure

NMR Workbench provides an unmatched facility to resolve ambiguities, catch assignment errors and identify additional constraints through a model-assisted NOE assignment method that interactively links a NOE spectrum to trial 3-D structures. The trial 3-D structures could be a structure built with Protein Workbench, a protonated X-ray structure of the molecule in study, or a candidate distance geometry/restrained dynamics structure. Potential NOE constraints identified by this method are compared with the NOE spectrum and are automatically included in a constraints database.

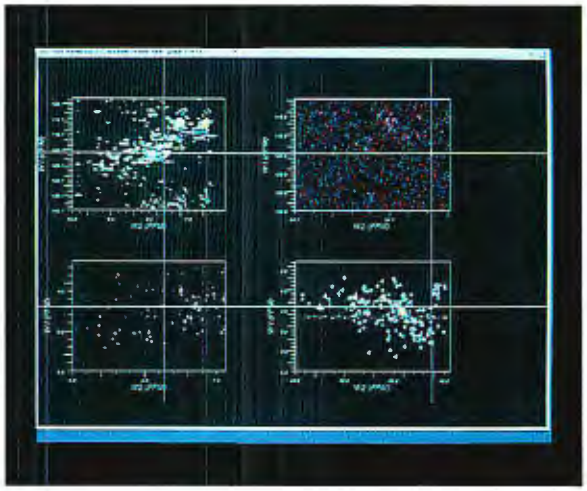
Spectral Database At The Core

At the core of NMR Workbench is a comprehensive spectral database that manages information on the thousands of peaks observed in different NMR experiments. This spectral database can be updated with relevant parameters, such as chemical shifts, spin systems, assignments, volumes, intensities, scalar J-couplings, line-widths and t_1 or t_2 relaxation measurements. This database provides smooth flow of data between different stages of structure determination: spectral assignment, extraction of constraints, generation of 3-D structures and evaluation of resulting structures. A powerful feature of this database is the ability to assign an unknown system based on knowledge drawn from previously determined systems (for example, assigning a mutant protein based on the information available on the wild type).

Computer-Aided Spectral Assignment

Pencils, T-squares and large paper plots have been the state of the art in spectral assignment. Now, NMR Workbench provides a unique and powerful set of interactive graphical tools which substantially accelerates the assignment process. High-resolution color graphic display provides the spectroscopist with an entire spectral data set at the user's fingertips, quickly moving from one spectrum to another, zooming spectral regions for closer inspection, examining 1D slices for peak resolution, inking spin systems for sequential assignment, and annotating assigned peaks.

Special tools rapidly pick spin systems within a 2D spectrum, or between correlated spectra from homonuclear and heteronuclear 2D/nD experiments. Data are interactively recorded in a spectral



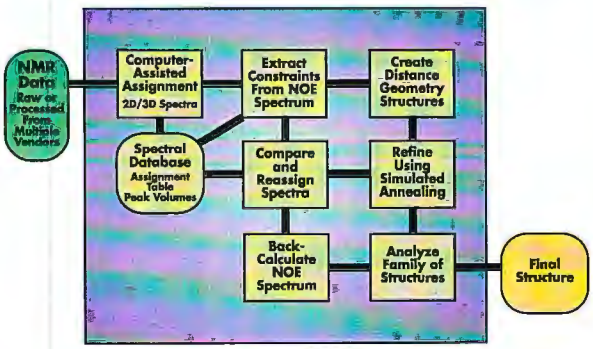
Computer-aided spectral assignment of multiple correlated spectra. An excellent tool for visualizing, tracing and bookkeeping of spin connectivity patterns in spectral databases.

database to provide excellent bookkeeping facilities. Computer-aided spectral assignment[4] reduces the reliance on slow, manual techniques and affords spectroscopists a new power tool for efficiently assigning the spectrum.

Molecular Modeling: No Previous Experience Required

NMR Workbench integrates graphical molecular modeling tools with X-PLOR, the leading software for structure determination. New molecules can be quickly sketched or built from standard peptide, nucleotide, or carbohydrate units. Distance and torsional constraints are easily set up from the Spectral Database. An easy-to-use menu interface to X-PLOR simplifies structure generation through a combination of distance geometry and restrained dynamics simulated annealing methods.[5,6]

Extensive analysis tools are provided for the analysis of resulting structures. An interactive RMSD fitting and measurement tool provides for overlay and comparison of subsets of the molecular system. Cluster analysis on RMSD, torsions, and distances readily identifies different conformational families. The system reports important statistics including pairwise RMSDs for selected structural regions and constraint violations and exports data in ASCII format for further study or import into other programs.



NMR Workbench is a complete integrated system for determining three-dimensional structures from NMR Data.

Validation and Refinement through Back Calculation

After predicting a 3-D structure, NMR Workbench back calculates the NOE spectrum by computing a NOESY relaxation matrix with cross and auto peak intensities using a set of coordinates and experimental parameters such as correlation time, mixing time, and relaxation delay. These intensities are converted into a spectral map and compared with the experimental NOE to resolve ambiguities in assignments and improve the constraints. NMR Workbench also includes options for iterative refinement of predicted structures by minimizing a penalty function defining the differences in back calculation and experimental intensities using X-PLOR/Refine.

Components of NMR Workbench

NMR Workbench consists of three major components: MADNMR, QUANTA/NMR and X-PLOR. MADNMR is used for NMR data processing, analysis, and computer-aided spectral assignment with options to generate experimental distance and torsional constraints for 3-D structure determination. QUANTA/NMR is an integrated system which drives the following components: DISCON/ NOESYSIM, X-PLOR/DG, and X-PLOR/Refine. DISCON/NOESYSIM is used for back calculation of NOE spectra from predicted structures. X-PLOR/DG generates structures with distance geometry and simulated annealing, and X-PLOR/Refine is used for refinement of trial structures with a novel residual relaxation matrix method.

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1. Wüthrich, K., Science 243, 45 (1989)
2. Crippen, G. and T. Havel. Distance Geometry and Molecular Conformation. Research Studies Press: Taunton, Somerset, England. (1988)
3. Clore, G.M. and A. Gronenborn, Science 252, 1390 (1991)
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5. Nilges, M., Clore, G.M., Gronenborn, A.M., FEBS Lett. 239, 317-324 (1988)
6. Nilges, M. and Brunger, A., Private Communication (1992)

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Clemens Anklin (Applications Manager)

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Should Be Addressed To:

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No. 409 (October) -------18 September 1992
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(received 5/2/92)

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

VISUALIZATION OF SOIL REMOVAL BY ^{1}H 1-DIMENSIONAL NMR IMAGING

Dear Barry,

We have been using ^{1}H NMR profiling experiments to measure the efficiency with which nonionic alcohol ethoxylate surfactants lift soils from fabrics. The technique has been recently extended to study paper deinking processes. Alcohol ethoxylates consist of a hydrocarbon chain connected to a run of ethylene oxide units. For example, $C_{12}E_4$ is used to represent a 12-carbon hydrocarbon chain terminated by a run of 4 ethylene oxide units. Traditionally, light microscopy has been used to visualize soil removal in fabrics. However, fundamental research has shown that the optimum temperature for removing nonpolar soils from fabrics using alcohol ethoxylates occurs well above the cloud point of the ethoxylates and near their phase inversion temperature (PIT). Optical techniques are inherently limited by sample turbidity and thus are unable to study alcohol ethoxylate-based systems above their cloud point.

Figure 1 shows the set up used for soil removal studies. D_2O is used in order to minimize spectral interference of water with the oil signal. The 1 cm diameter patch of fabric (cotton, polyester/cotton blend , paper, etc.) is epoxied to the bottom of the vial. Next the patch is soaked with a known weight of oil. The D_2O/surfactant mixture is then added to the vial and the 1-D imaging sequence is started immediately following placement of the sample in the magnet. The system used for this work is a General Electric CSI-2T spectrometer equipped with actively shielded gradients, a 3" diameter bird cage radio frequency coil and a 30 cm bore Oxford horizontal magnet.

Figures 2 and 3 show vertical and horizontal profiles visualizing the lifting of dodecane from a patch of polyester/cotton blend by a 0.5% solution of $C_{12}E_4$. In this case, 8 scans were averaged using a 5 s recycle delay time and profiles were acquired every 5 minutes. The horizontal profile in Figure 2 shows a steady decrease in signal from the 1 cm patch. At the same time, the profile reveals an increase in signal on either side of the patch indicating that, upon leaving the patch, dodecane distributes itself across the whole 1" diameter of the vial. The vertical profile confirms the lifting process. It is interesting to note that, upon leaving the fabric, the dodecane rises to the top of the water phase forming a thin oil layer as opposed to remaining emulsified throughout the aqueous phase. This "creaming to the top" effect explains the regions of higher intensity observed at the edges of the vial in the

horizontal profile; they are due to the meniscus effect at the oil/water interface.

Figure 4 compares $C_{12}E_4$ and $C_{12}E_3$ solutions in lifting dodecane from a patch of polyester/cotton blend. Note the dramatic difference between the two surfactants. No significant oil removal is detected for $C_{12}E_3$. These results are expected since the PIT for the $C_{12}E_4$ /dodecane system is at the experimental temperature of 23°C, while the PIT for $C_{12}E_3$/dodecane is well below 20°C.

We have implemented this approach to the screening of a wide variety of soil/surfactant/fabric combinations. The soil polarity is varied by mixing nonpolar oils with organic acids or alcohols. Surfactant polarity is varied by changing the length of the ethylene oxide moiety. We plan to expand this capability to include variable temperature and solution agitation schemes in order to better simulate laundering and deinking processes. Please credit this contribution to the account of Dr. L.L. Sterna.

Sincerely,

Pierre Tutunjian

John Ferris

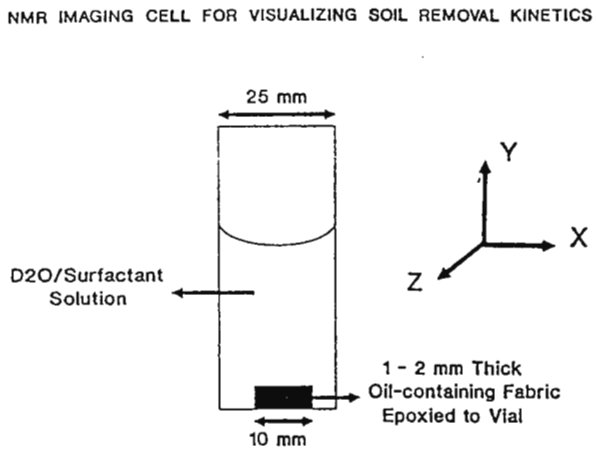


FIGURE 1

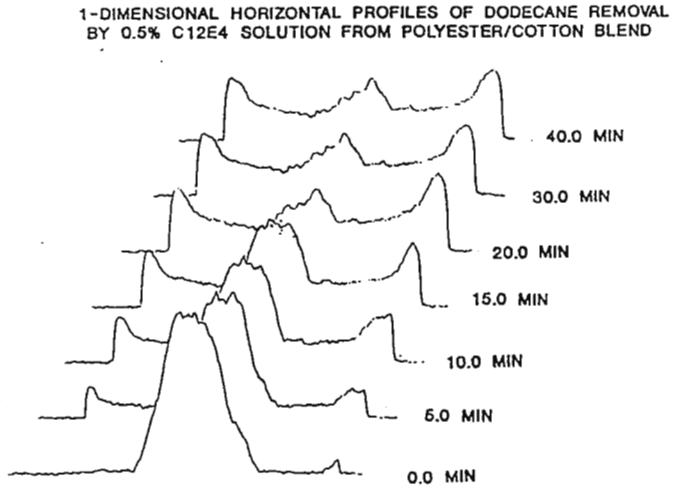


FIGURE 2

FIGURE 3

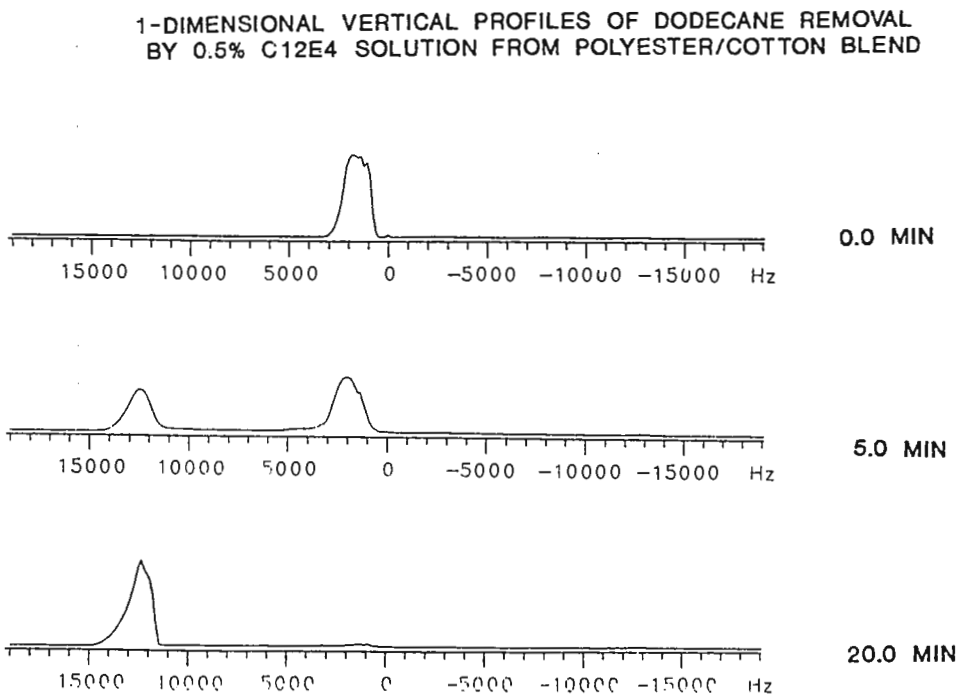
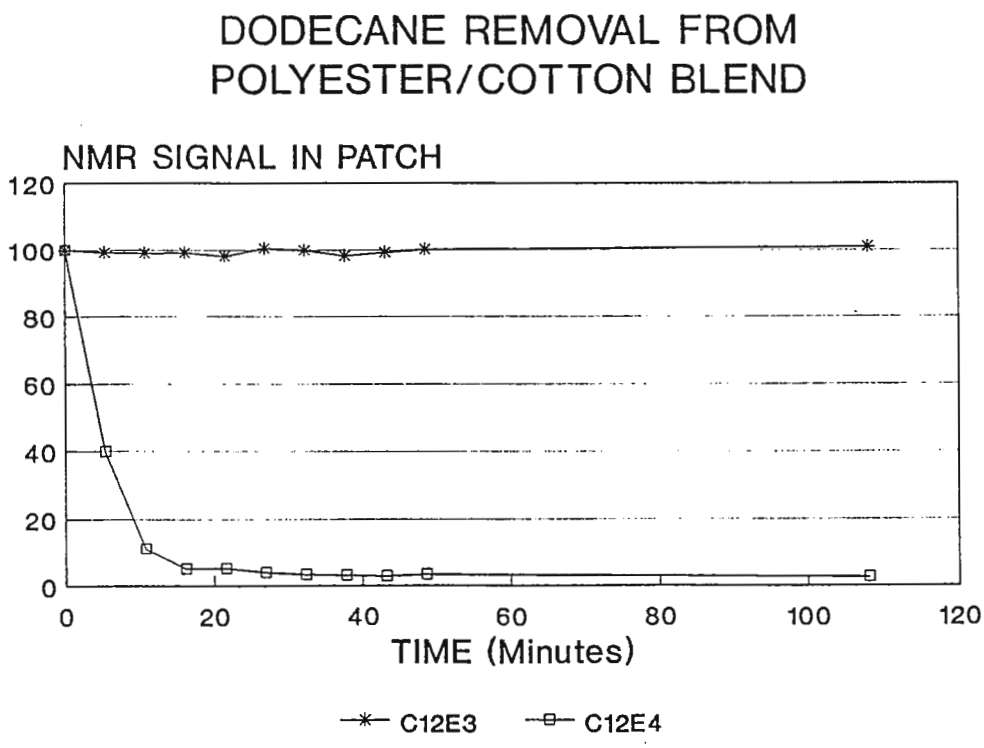


FIGURE 4



Gradient Enhanced Spectroscopy

Phase Sensitive DQF COSY

The selection of multiple quantum coherence with gradients is an effective method for suppressing the water resonance in aqueous solutions and for reducing t_1 noise and other artifacts. A further enhancement is the use of selective water excitation using crafted RF pulses followed by gradient dephasing to attenuate the water signal prior to the coherence selection sequence. This technique has been applied to the phase sensitive gradient enhanced DQF COSY experiment for a 5mM lysozyme sample in 90% H_2O. The data were collected on an Omega PSG 500 equipped with the S-17 Gradient Enhanced Spectroscopy accessory using a 5mm inverse probe.

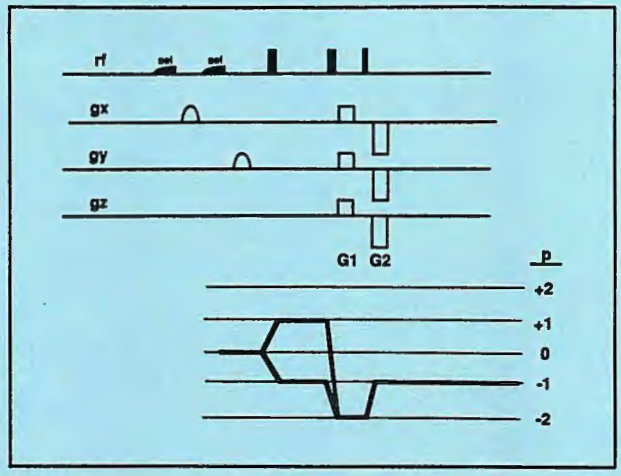


Figure 1

The pulse sequence and the corresponding coherence level diagram for the phase sensitive GE-DQF COSY experiment. The water resonance is selectively excited using a crafted RF pulse followed by a shaped gradient pulse along the X axis which dephases the transverse magnetization. A second selective RF pulse is applied with a dephasing gradient along the Y axis to avoid gradient recalled echoes. This is followed by a phase sensitive GE-DQF COSY sequence.

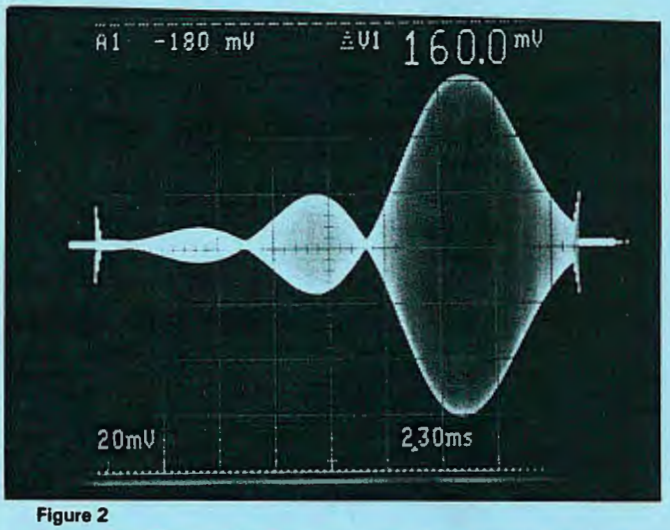


Figure 2

The crafted RF pulse used in this sequence. This pulse was designed using the "hard pulse approximation" method. It is characterized by e flat amplitude response in the selected region and minimal excitation in the out of band region. The duration of the selective RF pulse was set to 20 ms, corresponding to an excitation bandwidth of 175 Hz.

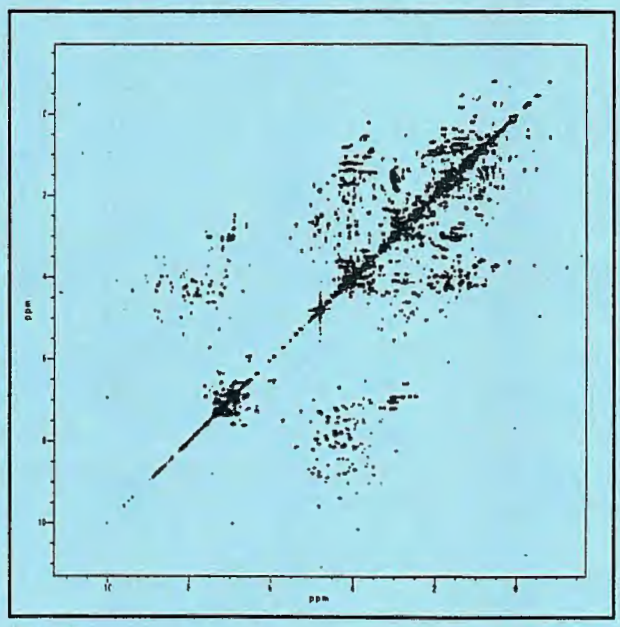


Figure 3

A phase sensitive GE-DQF COSY spectrum of 5 mM lysozyme in 90% H_2O. Thirty-two scans were accumulated for each of the 700 t_1 increments resulting in a total data acquisition time of approximately 9.7 hours. Half-sinusoidal gradient pulses of 20 ms duration with an amplitude of 10 G/cm were used to dephase the excited water signal. Coherence selection was achieved using 2 ms gradient pulses of 17 and 34 G/cm amplitude.

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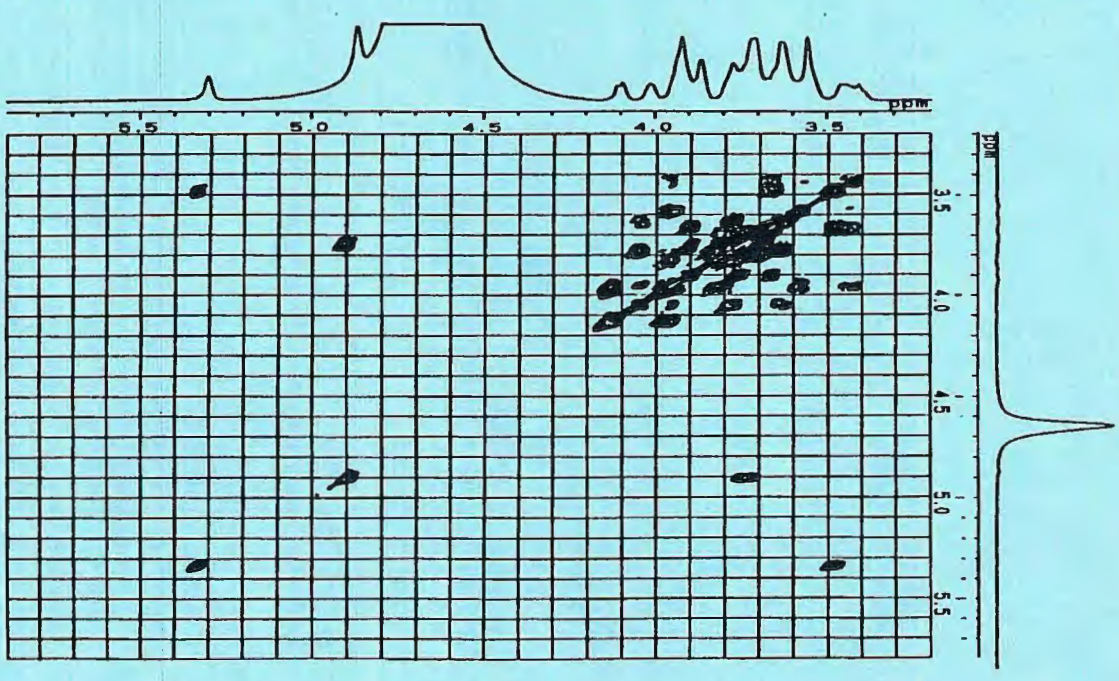

JEOL's gradient probe is tuned to observe ^{1}H with ^{15}N to ^{31}P decoupling and is designed to be used for reverse detection experiments. The above data were collected on an **ALPHA** 500 with the pulsed field gradient accessory. The sample is stachyose in 90% H_2O. By comparing the 1D spectrum plotted at the top with the 2D data, one can see the magnitude of the water suppression afforded by the gradient probe. It is also worthwhile to note that each slice was obtained in one scan and that the double quantum filtering was accomplished with the gradients. This 512 by 512 matrix was obtained in approximately ten minutes.

If you would like to get more information about JEOL's **ALPHA,** please contact us at one of our offices listed below.

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