TEXAS A&M UNIVERSITY

NMR NEWSLETTER

No. 392

May 1991

Continued on page 2

A monthly collection of informal private letters from Laboratories of NMR. Information contained herein is solely for the use of the reader. Quotation is *not* permitted, except by direct arrangement with the author of the letter, and the material quoted *must* be referred to as a "Private Communication". Reference to the TAMU NMR Newsletter by name in the open literature is strictly forbidden.

These restrictions apply equally to both the actual Newsletter participant-recipients and to all others who are allowed open access to the Newsletter issues. Strict adherence to this policy is considered essential to the successful continuation of the Newsletter as an informal medium of exchange of NMR information.

TABLE 1 DEUTERATED SOLVENTS

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D-122							
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D-28	Chloroform-d	$CDCl_3$	99.8%	1.50	-64	62	0.740 (20)
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FORTHCOMING NMR MEETINGS

Eleventh Delaware NMR Symposium, Univ. of Delaware, Newark, DE, **June 4, 1991** See Newsletter 390, 35.

Tenth International Meeting on NMR Spectroscopy, St. Andrews, Scotland, **July 8-12, 1991**; Contact: Dr. John F. Gibson, Secretary (Scientific), The Royal Society of Chemistry, Burlington House, London W1V 0BN, England; See Newsletter 387, 69.

33rd Rocky Mountain Conference on Analytical Chemistry, Denver, CO, **July 28 - August 2, 1991**; For information on the NMR Symposia, contact Dr. H. Eckert, Dept. of Chemistry, Univ. of California at Santa Barbara, Goleta, CA 93106, (805) 893-8163; For general information, contact: P. Sulik, Conference Chair, Rocky Mountain Instrumental Laboratories, 456 S. Link Lane, Fort Collins, CO 80524, (303) 530-1169.

Gordon Research Conference on Magnetic Resonance, Brewster Academy, Wolfeboro, NH, **July 15-19, 1991**; Chairman: R. Griffin; Information from Dr. A. M. Cruickshank, Gordon Research Center, Univ. of Rhode Island, Kingston, RI 02881-0801; Tel.: (401) 783-4011 or -3372; FAX (401) 783-7644.

Tenth Annual Scientific Meeting and Exhibition, Society of Magnetic Resonance in Medicine, San Francisco, **August 10-16, 1991**; Contact: S.M.R.M., 1918 University Ave., Suite 3C, Berkeley, CA 94704; (415) 841-1899, FAX: (415) 841-2340; See Newsletter 391, 55.

Two-Dimensional NMR Spectroscopy (ACS Short Course), New York, NY, **August 23 - 25, 1991**; See Newsletter 392, 33.

International Conference on NMR Microscopy, Heidelberg, Germany, **September 16 - 19, 1991**; See Newsletter 385, 28.

1991 Joint Meeting FACSS/Pacific Conference, Anaheim, California, **October 6-11, 1991**; NMR/EPR Program Section Chairman: Prof. Cecil R. Dybowski, Chemistry Dept., Univ. of Delaware, Newark, DE 19716. Contact: FACSS, P.O. Box 278, Manhattan, KS 66502-0003.

Eighth Australian NMR Conference, Lorne, Victoria, Australia, **February 2-6, 1992**; See Newsletter 391, 38.

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.

Additional listings of meetings, etc., are invited.

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All Newsletter Correspondence

Should Be Addressed To:

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

(415) 493-5971

DEADLINE DATES

No. 394 (July)---------- 21 June 1991
No. 395 (August)---------- 19 July 1991
No. 396 (September)-------- 16 August 1991
No. 397 (October) --------6 September 1991

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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

To come straight to the point, the overall finances of the Newsletter are not as healthy as one might wish. 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.

The number of Subscriptions to the Newsletter continues to increase, indicating that this publication still serves a useful purpose. Unfortunately, income from Sponsorships and Advertising has fallen off significantly, and I do not yet see 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 urgently required and earnestly solicited.

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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 (with an orderly evolution of the technical scope and the mailing list), the finances are very problematical. Not only is revenue down, but expenses - most notably, mailing costs - continue to increase significantly. I am not 'crying wolf', but am trying to alert all who are interested in the Newsletter to the fiscal realities.

Other ideas about how to alleviate this very real budget crunch will be gratefully received. I will continue to run the Newsletter - with the on-going cooperation of Texas A&M University - in the most no-frills manner possible.

Notice re 1991-92 Invoices and Subscription Rates

Subscription renewal invoices for the October 1991 - September 1992 year will be mailed out at the end of June. If you ought to receive such an invoice, and do not have it in your hands by July 10, please call or write me promptly. *Payment* of these invoices *must be received* by me *no later than September 5, 1991* 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 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.

Because the subscription rate was increased last year, I cannot bring myself to raise the rates again for this year, despite the poor situation of the Newsletter finances. I am most reluctant to raise subscription rates to the point where cost to the Newsletter subscriber/participants becomes an inhibitory factor. Accordingly, the subscription rate for the October 1991 - September 1992 year will remain at US$150.00 for the twelve monthly issues, postpaid. Personal or academic subscriptions will continue to be offered at a 50% discount, US$75.00.

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 May 1991

Carleton University
Ottawa, Canada K1S 5B6

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

April 11/91
(received 4/17/91)

Title: Guest and Host Motions in the Solid 18-Crown-6.2ClCH$_2$CN Complex

Recently PDF André Rodrigue has succeeded in isolating the complex of 2 chloroacetonitriles with 18-Crown-6 ether. The solid phase (Bruker CXP-180 at NRC Ottawa) 45.3 MHz ^{13}C CPMAS spectra show two regions of broadening for the crown ether carbons. At high temperature (310K), a dipolar washout mechanism is operative, leading to broadening when molecular motion has a correlation time approximately equal to the inverse of the decoupling field. Application of the Waugh-Fedin approximation (1) gives an activation energy for 18-C-6 reorientation in the solid complex of ca. 48 kJ/mol. At lower temperatures (240K), broadening occurs when the motional correlation time is equal to the inverse of the chemical shift difference for the 18-C-6 carbons in the low temperature limit (ca.70Hz).

Interestingly, the CH$_2$'s of the guest chloroacetonitrile molecules also undergo dipolar washout, but at a considerably lower temperature (270K). The activation energy for reorientation of these "guest" molecules in the solid complex is, via similar metods to those described above, calculated to be ca. 42 kJ/mol.

1. J.S. Waugh and E.I.Fedin. Sov.Phys. Solid State (Eng. Trans) 4,1633 (1963).

G.W. Buchanan
Professor of Chemistry

P.S. We are eagerly awaiting the arrival of our new Bruker AMX-400 in the late summer!

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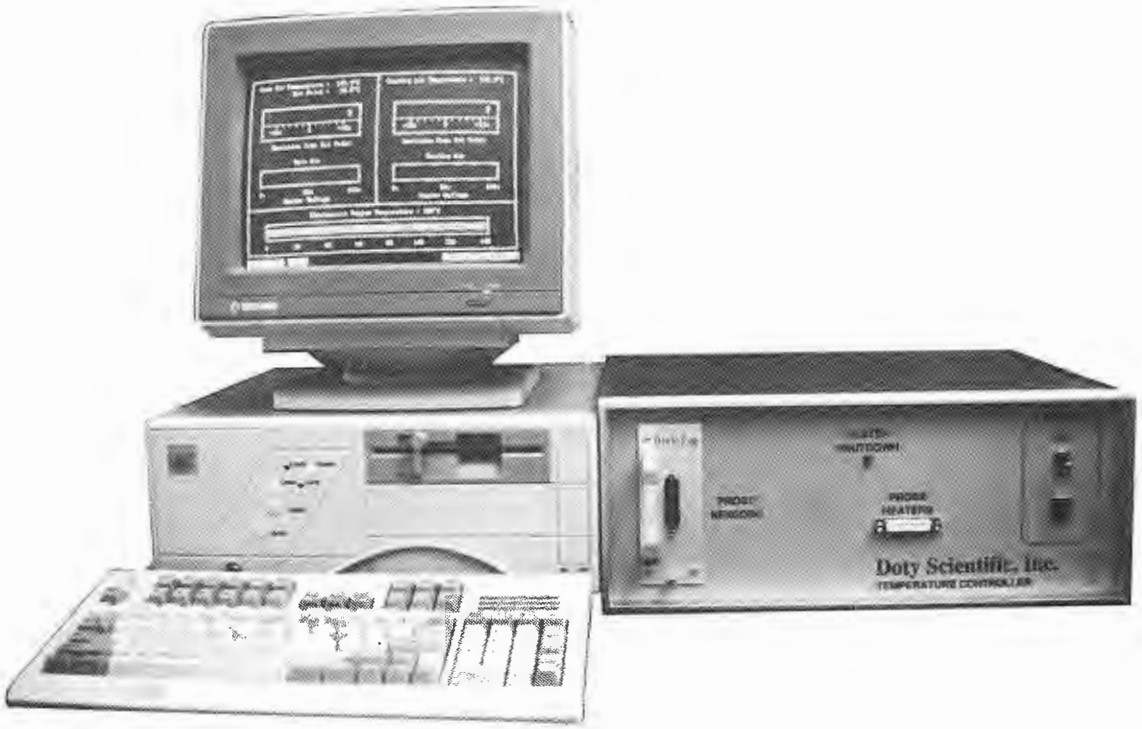

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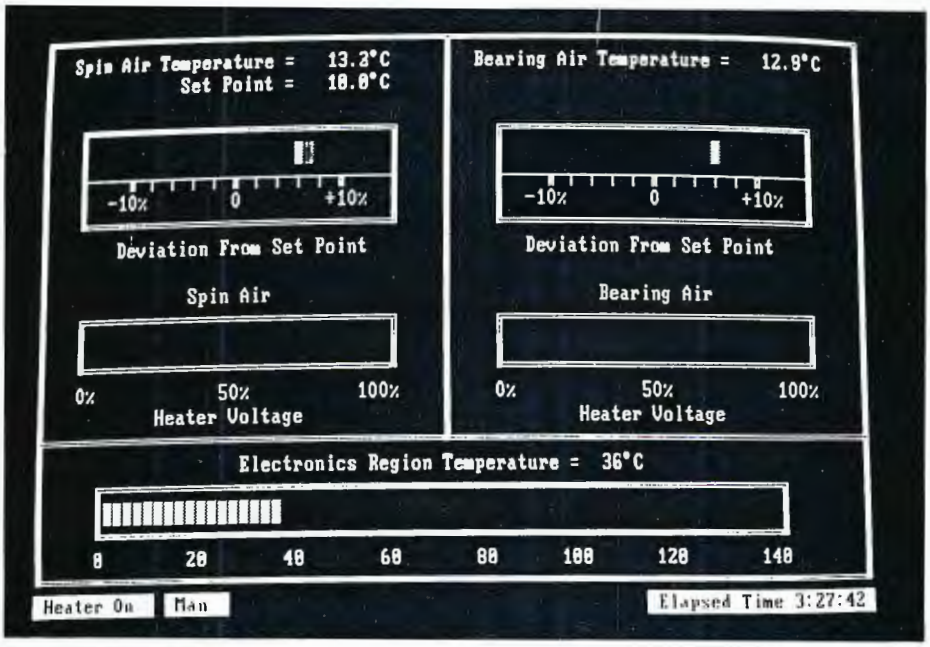

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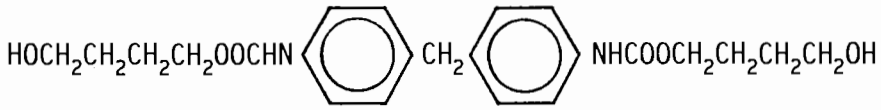
3/20/91 (received 3/23/91)

UNUSUAL CPMAS SPECTRA OF URETHANE HARDSEGMENTS

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

Dear Dr. Shapiro,

As part of an effort to better understand structure-property correlations in urethane elastomers[1-2], we synthesized the hardsegment compound (1) shown below by reacting methylenediphenyl diisocyanate (MDI) with excess butanediol in dimethylformamide (DMF).



(1)

When (1) is reacted with more MDI and a long chain polyether (the soft segment) a typical urethane elastomer is formed.

During the project, we found that IR and x-ray spectra of the product (1) showed differences depending upon the manner in which it was crystallized from the DMF. Generally, the product crystals were formed by adding water and methanol to the DMF-product reaction mixture. The crystals were then resuspended in water to remove any remaining DMF. It was noticed that two types of crystals were obtained from the water suspension. The first was a faintly yellow, granular solid which settled quickly. The second was a white solid which settled much more slowly. It was also possible to separate good product by precipitating all solids from the DMF solution by addition of water and then extracting with methanol. X-ray diffraction patterns of these products (referred to as types 1,2 and 3 respectively) indicated that type 2 was more crystalline than type 1, however type 1 was not completely amorphous. The CPMAS spectra of these solid products are shown in figure 1. The Dixon sequence was used to suppress spinning sidebands. Type 3 is clearly a mixture of types 1 and 2. However, the origin of the shifts between types 1 and 2 is still a mystery. The underlying molecular reason for these shifts may have some relevance to the structure of urethane polymers. Many of our CPMAS spectra of MDI and butanediol containing urethane polymers have an anomalously broad peak at 120 ppm corresponding to the 2,2′ carbon of the aromatic ring (see figure 4 of reference 2). Perhaps one of your readers will be inspired to determine the cause of these shifts.

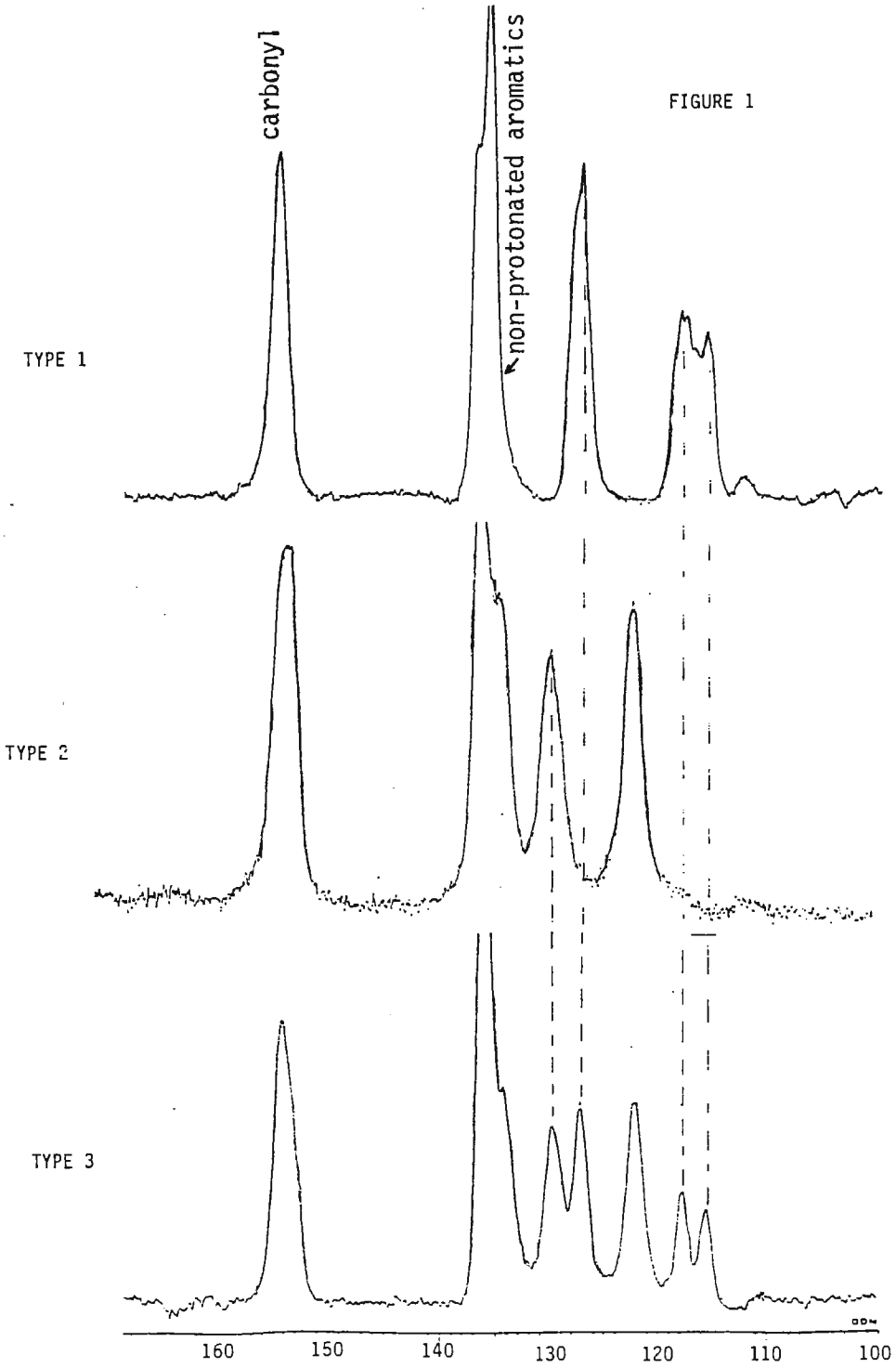
Sincerely,

Michael D. Meadows William L. Howard

[1] Christenson, C.P.; Harthcock, M.A.; Meadows, M.D.; Spell, H.L.; Howard, W.L.; Creswick, M.W.; Guerra, R.E.; Turner R.B. J. Polym. Sci., Polym. Phys. Ed. 1986, 24, 1401.

[2] Meadows, M.D.; Christenson, C.P.; W.L. Howard, W.L.; Harthcock, M.A.; Guerra, R.E.; Turner, R.B. Macromolecules 1990, 23, 2440.

FIGURE 1



Agricultural Products

March 28, 1991
(received 3/30/91)

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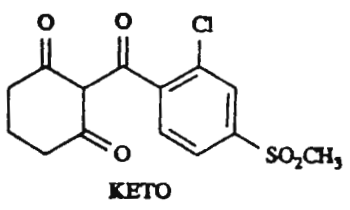
Dr. B. L. Shapiro, Editor
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto, CA 94303

Subject: Keto-Enol Tautomerism of a β-Triketone

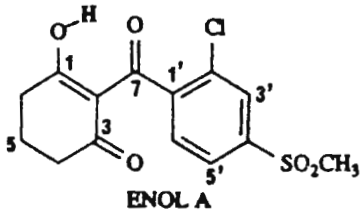
Dear Barry:

A β-triketone compound (I) can exist as many tautomers; among them KETO, ENOL-A, and ENOL-B forms are the three most likely tautomers. We wish to report the results of proton and carbon-13 NMR studies of I.

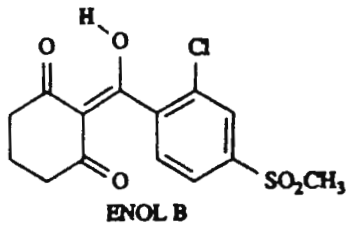
I.



KETO



ENOL A



ENOL B

The proton and carbon-13 NMR spectra were obtained in deuterated chloroform. A proton shift at 16.63 ppm, indicative of an intramolecular H-bonding, suggests that (I) exists in an enol form. The enol form was further confirmed by the observation of six distinct carbon resonances from the cyclohexanedione moiety. To establish the ENOL-A or ENOL-B tautomer, a fully coupled carbon spectrum and an one bond HETCOR spectrum were obtained. The proton and carbon chemical shifts and coupling constants are listed in Table 1. C_1 was shown to couple with OH, H_5, and H_6 by selective decoupling. Similarly, C_3 was shown to couple with H_4 and H_5, and C_7 coupled only to H_6,. Since the OH proton couples to C_1, C_2, and C_6 but not to C_7, we conclude that ENOL-A is the prefered tautomer in solution. These results suggest that the endocyclic double bond is favored in this system.

Sincerely,

Lydia L. Chang

C. K. Tseng

TABLE 1

PROTON AND CARBON-13 NMR SPECTRAL DATA

Atom No.	δ_H	J_{HH}	δ_C	$^1J_{CH}$	$^2J_{CH}$	$^3J_{CH}$
1			197.1		6.5; 4.5[a]	6.5
2			113.7		-	5.3[a,c]
3			193.6		6.3	6.3
4	2.39	6.4	37.4	129.4	4.5	4.5
5	2.00	6.4	18.9	131.7	3.9	3.9
6	2.76	6.4	32.0	129.8	4.5	4.5; 4.5[a]
7			195.7	-	-	4.1
1'			144.0	-	5.3	8.1
2'			130.9	-	4.1	10.0
3'	7.90	1.7	128.1	171.6	-	6.0
4'			141.9	-	**b**	9.4
5'	7.83	8.0; 1.7	125.7	-	**d**	5.8
6'	7.33	8.0	127.7	166.9	**d**	-
CH_3	3.03		44.4	138.6	-	-
OH	16.63					

a coupling to OH

b only line broadening observed

c obtained from the spectrum selectively irradiated H_6 (2.76 ppm)

d not observed

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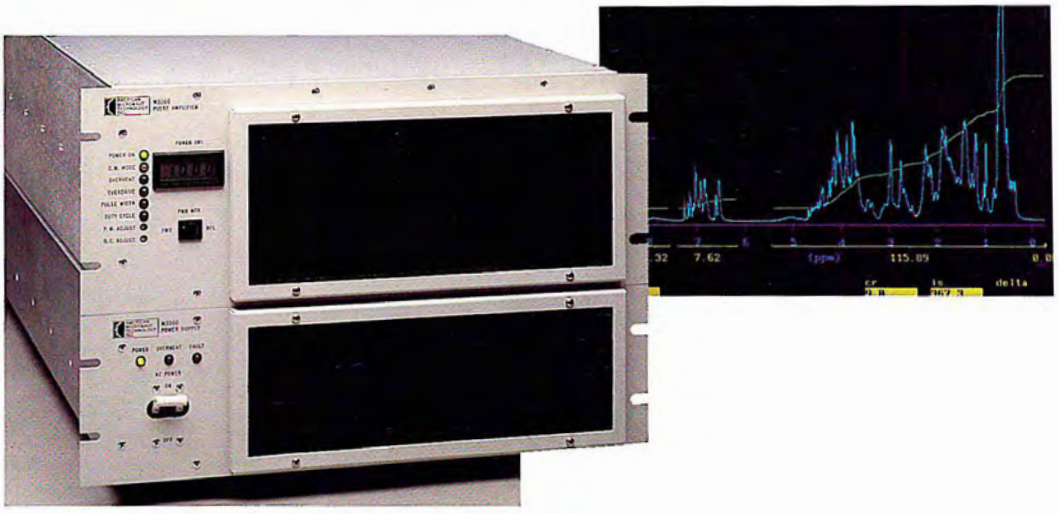

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System Monitors	1. Thermal 2. DC power supply fault 3. Over duty cycle 4. Over pulse width
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Professor Bernard L. Shapiro, Editor/Publisher
Texas A&M University NMR Newsletter
966 Elsinore Court
Palo Alto, California 94303

March 18, 1991
(received 3/19/91)

The Miracle of the Sphere of Lorentz

Dear Barry:

We have gone on and on in print about the wondrous NMR manifestations of the sphere of Lorentz (SOL).[1] However, although chimeric apparitions have been reported by true believers in Ottawa[2] and Bordeaux[3], for years we were never able to actually see a SOL for ourselves. We were beginning to lose faith and think that perhaps this was because it is a hypothetical construct. However, I am very pleased to report that we have finally been blessed with a vision here in Stony Brook[4].

The figure presents two attempts to capture our impressions of this on paper. It is a modification of parts of Figure 2 of an important contribution to the consideration of the effects of bulk magnetic susceptibility (BMS) in biological NMR[5]. The top case is that of a spherical compartment of volume susceptibility χ_i submersed in a medium whose susceptibility, χ_e, is greater (or less negative): $\chi_i < \chi_e < 0$. One can tell this because the density of flux lines (the direction of $\mathbf{B}_0$ is horizontal) is greater outside than inside the sphere. The density seen would be proportional to the **B** value sensed by a nucleus in the absence of local (on the molecular scale) susceptibility cancellation. However, one must consider SOLs (dashed circles) in order to determine the **B** values actually sensed by real nuclei.

The SOL is an imaginary insulating vacuum cavity surrounding each nucleus. It is large on the scale of the nucleus but small on the scale of the sample (and thus exaggerated in the figure). In the top case of the figure, the flux lines inside a SOL inside the compartment are compressed to exactly the same density present inside a SOL on the outside sufficiently far from the compartment. In fact, to a good approximation, the value of **B** actually sensed by a nucleus inside a spherical compartment depends only on χ_e and is completely independent of χ_i.[1] Thus, if a spherical compartment containing any medium is suspended in a vacuum ($\chi_e = 0$), there is no BMS effect for a nucleus inside. The top of the figure also shows that while l_i, the internal inhomogeneous term[1], is zero, l_e is nonzero (the flux lines are not parallel) for regions near the compartment. The inhomogeneities pictured outside the SOLs are, like the spheres themselves, imaginary.

The bottom case of the figure represents a cross-section of an infinitely long cylindrical compartment, oriented perpendicular to $\mathbf{B}_0$, and immersed in a medium where, now, $\chi_e < \chi_i < 0$. In this situation, the flux lines are more dense inside the compartment. However, it is quite remarkable that a SOL inside the compartment actually compresses the flux lines to an extent smaller than one at an infinite distance outside the compartment so that a real nucleus inside the compartment senses a flux density *less* than one sufficiently far away on the outside[1]. This figure also shows that l_i is zero while l_e is nonzero near the compartmental surface. Were this same infinite cylinder oriented parallel to $\mathbf{B}_0$, both l_e and l_i would be zero[1]. However, in this latter case, though SOLs on both the inside and the outside still compress the flux lines by the same extents as in the bottom of the figure, the change in the inside compartmental demagnetizing field consequent to the reorientation causes a nucleus on the inside to sense a susceptibility greater (less negative) than that sensed at an infinite distance on the outside[1]. A number of examples of the critical importance of these local susceptibility effects in real situations have been presented in other places[1,6,7]. The figure shown here is from another paper which is in press[4].

We trust that this miraculous revelation will temporarily stanch the flow of orange notices to our account.

Best regards,

Charlie

Charles S. Springer, Jr.
Professor

Yan Xu

Yan Xu, Postdoctoral Associate
Department of Pharmaceutical Chemistry
University of California at San Francisco

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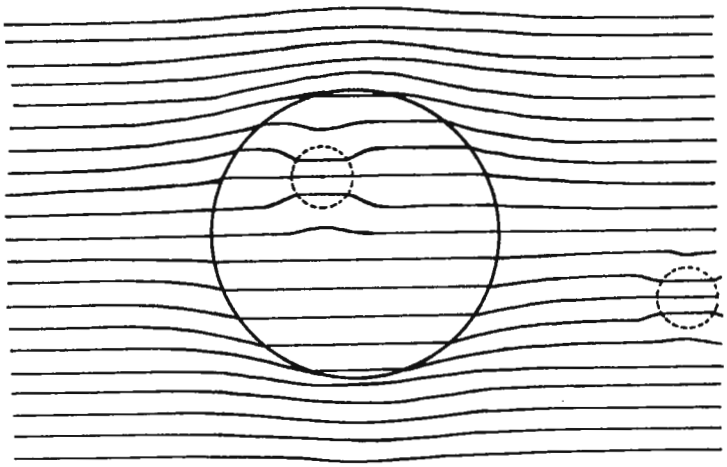

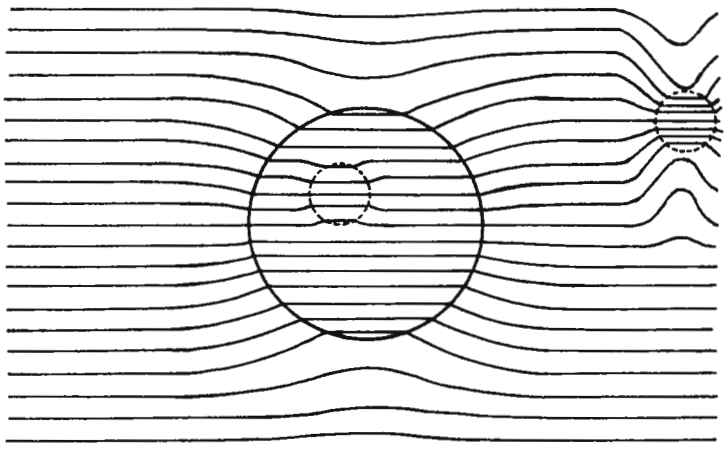

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Dr. B. L. Shapiro, Editor
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto, CA 94303
USA.

March 26, 1991
(received 4/13/91)

The Observation of Myocardial Infarct by ^{23}Na NMR Spectroscopy

Dear Dr. Shapiro

In the past few years we have been concerned with the preservation of hearts for transplantation. Since at present the preservation of a heart is limited to 4-6 hours, Israel cannot exchange hearts through the European transplantation program.

In addition to ^{31}P NMR spectroscopy, we have been looking with ^{23}Na NMR for a possible role of intracellular Na^+ accumulation in the failure of heart preservation. At low temperatures, C. S. Springer's shift reagent Dy(TTHA) gives a very good separation between the unshifted intracellular Na^+ (in), the shifted extracellular Na^+ (ex) and the residual Na^+ in the surrounding mannitol solution (out) (see Fig. 1a).

One of the rat hearts gave a very peculiar spectrum, containing a shoulder on the intracellular peak (marked by arrow in Fig. 1b). We noticed that this heart had a spontaneous infarct and did not recover upon reperfusion. Indeed the observation was confirmed by controlled experiments with induced infarcts. Such an experiment with coronary artery ligation is shown in Fig. 1b.

Our interpretation is that in the infarcted region the disturbed membranal permeability allows the penetration of shift reagent into cells, shifting the intracellular Na^+.

Peculiar NMR observations should not be always thrown away to the waist basket. Sometimes they may turn out to be quite interesting.

Sincerely,

Nadir Askenasy Gil Navon

6426212
קרית האוניברסיטה, רמת אביב, תל אביב 69 978 טל. 03-5450751, פקסימיליה 972-3-[redacted]
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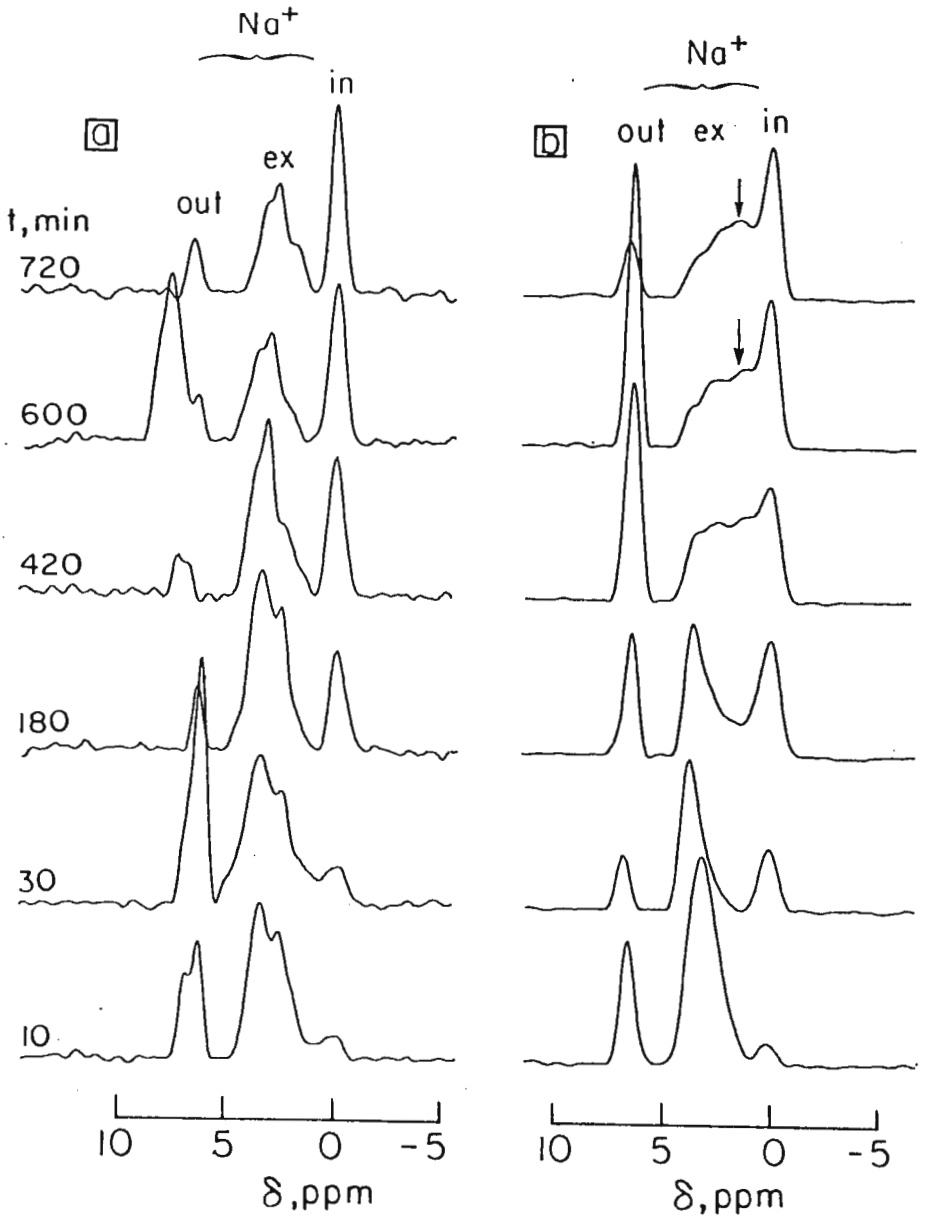

Fig. 1: ^{23}Na NMR spectra of rat hearts stored in Krebs Henseleite-Dy(TTHA) solution at 4°C, bathed in mannitol-Dy(TTHA) during the NMR measurements. a) control. b) coronary artery ligation.

Equipment Available: Bruker AC-F 200 NMR spectrometer (process controller version); variable temperature accessories, VSP probe, ultra low loss magnet; solids accessories including double air bearing multinuclear probe. All items in excellent working condition. Phone Dave at (403) 463-1602 or (403) 462-2798 for details.

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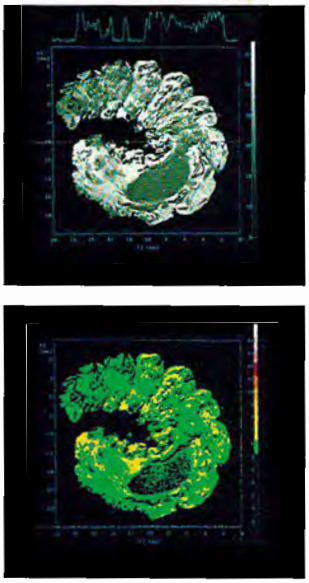

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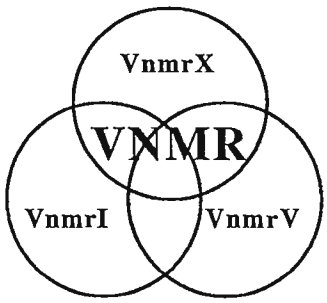

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PENNSTATE

Department of Chemistry
College of Science

152 Davey Laboratory
The Pennsylvania State University
University Park, PA 16802

(received 3/25/91)

Powder Lineshape
Spin Gymnastics for I=3/2

Dear Barry,

We observed that frequency domain simulations of quadrupolar powder lineshapes obtained with the Hare software algorithm QNS did not match our experimental ^{7}Li quadrupole echo spectra. We now know that the main reason is extremely long T_1's for ^{7}Li in solid state samples (hours or days!), so that we were observing only fast-relaxing "amorphous" ^{7}Li. Nevertheless, we wrote a Fortran program which simulates FID's obtained for I=3/2 nuclei in static powders. The advantage is, of course, that we can more accurately approximate the conditions of the quadrupole echo experiment.

With our simulator, up to 16 pulse or delay intervals may be applied prior to "acquisition". In addition, the pulses may be phase cycled. The secular quadrupolar Hamiltonian in the rotating frame is active during delays, and both it and the radio frequency Hamiltonian are active during pulses. Chemical shift effects are neglected. Required parameters are the rf field strength (ω_1) the quadrupole coupling constant (ω_Q), the asymmetry parameter (η), the durations of all pulses and delays, the phase cycle for each pulse, the sweep width (i.e. dwell time), the number of complex data points to acquire, and the number of increments in theta and phi for the powder lineshape. The FID's are processed with Hare software.

It was reassuring to find that for hard pulses, the FID's obtained by our simulator immediately after the pulse yielded exactly the same spectrum as the QNS simulation for Hare software. Of course, in real experiments one must wait for ringdown, so quadrupole echo experiments are used. Figure 1 shows the result of an idealized (but impossible to achieve experimentally) single pulse experiment where the FID is acquired immediately after the pulse. $(\omega_Q/2\pi)$ = 80 kHz, η = 0.5, $(\omega_1/2\pi)$ = 58.82 kHz, 512 complex data points, 128 increments for both theta and phi. The data was Gaussian broadened by 6000 Hz prior to the Fourier transformation. Figure 2 shows the result obtained for the same parameters with a quadrupole echo experiment (90_x - τ_1 - $90_{\pm y}$ - τ_2 - acquire) with τ_1 = 30 usec, τ_2 = 27 usec, the data left shifted to the echo maximum, and 6000 Hz of Gaussian broadening. The satellite transitions are greatly enhanced relative to the single pulse experiment. Figure 3 shows what happens to the quadrupole echo experiment with incomplete phase cycling (e.g. 90_x - τ_1 - 90_{+y} - τ_2 - acquire). Because there is a non-zero imaginary component to the FID, there is an asymmetry in the

resulting spectrum which can not be phase corrected. In addition, we have found that the $90_x - \tau_1 - 90_{\pm y} - \tau_2$ sequence gives about 8-10% more intense spectra (i.e. echo maxima) than the $90_x - \tau_1 - 45_{\pm y} - \tau_2$ sequence.

Sincerely,

Alan Benesi

Lloyd Jackman

Deniz Cizmeciyan

Figure 1

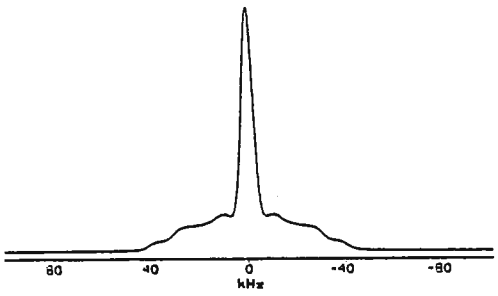


Figure 2

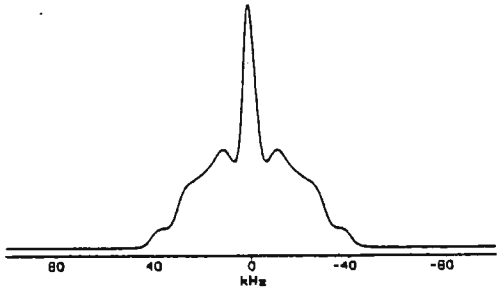
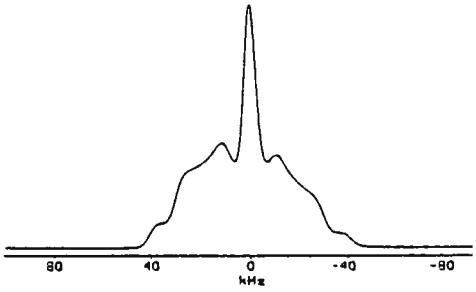


Figure 3



POSTDOCTORAL POSITION, STRUCTURAL BIOLOGY/NMR.

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NMR Laboratory - University of Mons - 7000 Mons - BELGIUM

April 9, 1991
(received 4/19/91)

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

IMPLEMENTATION AND OPERATION OF AN INTEGRATED AND COMPREHENSIVE RELAXOMETRIC DATA BANK.

Dear Dr. Shapiro,

Relaxometry (i.e. the measurement of nuclear relaxation parameters) can easily produce, when performed with appropriate, specialized instrumentation, several hundred of data points in one single day. Depending on the machine and the operator, data are normally stored on different media, in a variety of formats and at different locations. For a whole group dedicated to research in this area, storage and retrieval of data can thus very soon become a problem, specially when results from different projects (e.g. relaxometry of tissues and of contrast media) are to be combined. The same problem will arise when guests of the laboratory are given access to the data or produce data by themselves. In consequence, we have decided to build up a microcomputer-based relaxometry data bank into all current and previous data are stored and accessible to all members of the group.

The application has been written in Clipper and uses DBASE-organized files. When required, as with data produced by the APL software of the IBM Research Relaxometer, the files are reformatted. This software runs on PC-XT, PC-AT or PS-2 equipped with a hard disk, a floppy disk and not less than 640K RAM. The application is aimed to perform the management of data like T1s and T2s collected over a wide range of frequencies on various instrumentations, to include informations like comments about samples nature, to allow specific retrieval of informations and their display. Today the data bank contains around 20,000 data points concerning 2,000 samples. Retrieval of the data is performed by a routine using combinations of logical operators as well as several levels of parenthesis. In addition to this pure storage-retrieval of the data, the program combines modules for manual or automatic fitting of the Nuclear Magnetic Relaxation Dispersion (NMRD) profiles . A

straightforward extension of this option will be the calculation of natural or induced image contrast. The program architecture also allows transfer of the selected data to standard packages like 123 (LOTUS) and GRAPHER.

Robert N. MULLER

Yves VAN HABERBEKE

P.S. : Our coworkers (Didier DECLERCQ, Patrick VALLET, Fabian GIBERTO, Bernard DAMINET, Helmut W. FISCHER and Frédéric MATON) have had a major role in this work which also took benefit of a collaborative action with Prof. Peter A. RINCK of the MR Center of TRONDHEIM (NORWAY).

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Our research group is equipped with an upgraded BRUKER AM 400 with a separate data station. The position could become available as early as November 1991. Interested persons should apply to: PD Dr. P. Bigler Institute of Organic Chemistry, University of Berne, Freiestrasse 3, CH-3012 Bern.

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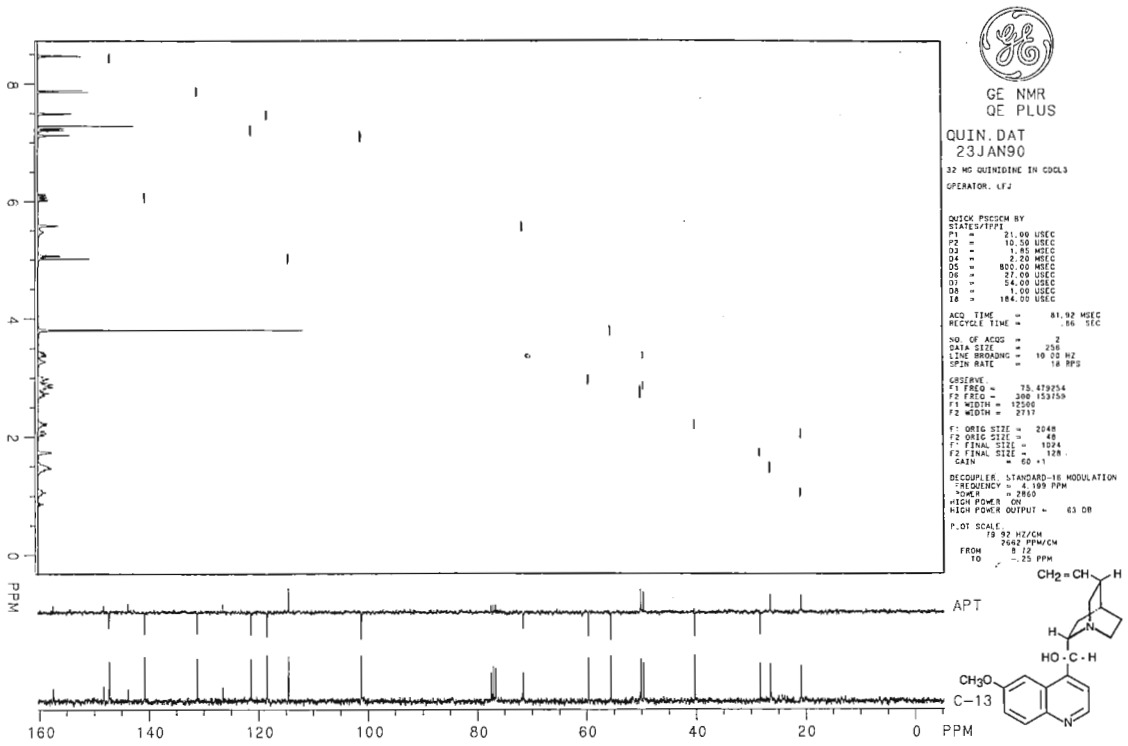

An automated run using MACRO mode operation on a sample of 32 mg of quinidine in 0.5 ml chloroform-d (0.20M). Data were obtained using the 5 mm broadband probe. ^{1}H, ^{13}C, APT and phase sensitive 2D data were collected, processed and plotted—including the 2D contour—in only 8.2 min.

WORCESTER POLYTECHNIC INSTITUTE

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April 1, 1991
(received 4/8/91)

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

Anisotropic Diffusion NMR Imaging

Dear Dr. Shapiro,

We have developed a novel anisotropic diffusion imaging NMR method, which allows two images weighted with directional diffusion to be obtained in a single imaging experiment. The method is based on a four-pulse RF sequence, which excites both SE and STE signals, combined with two pairs of diffusion-sensitive gradient pulses. The SE signal is used to monitor relatively rapid diffusion in one direction; whereas the STE signal is used to monitor the more restricted diffusion in the second direction. The diffusional attenuation of the SE and STE is controlled independently by separate adjustment of each pair of the diffusion-sensitive gradient pulses.

The pulse sequence used is shown in Fig. 1 and is denoted by θ_x-τ_1-90°_x-τ_2-180°_y-τ_2-(SE)-τ_3-90°_x-τ_1-(STE). At t=0, a θ (usually <90°) pulse is applied, which brings a fraction of the initial magnetization onto the xy plane. At time τ_1, this transverse component will have a phase shift. A 90° pulse is then applied at $t=\tau_1$, which nutates the residual z-magnetization onto the xy plane. This 90° pulse also divides the transverse component existing prior to the pulse into two parts, one of which is brought back to the z direction and the other one remains in xy plane. At this point, there two transverse components, one of which has been phase shifted during the τ_1 period and the other unshifted. The phase-shifted component is unwanted and can be removed. The unshifted component dephases following the second RF pulse and is rephased by the 180° pulse applied at $t=\tau_1+\tau_2$ so that a SE occurs at $t=\tau_1+2\tau_2$. Throughout the SE formation, the phase memory of the z-magnetization is preserved. The z-magnetization is refocused by the last 90° pulse and a STE is formed at τ_1 later. The first, second and fourth RF pulses have the same phase while the 180° pulse is phase shifted by 90°.

To probe directional molecular diffusion, two pairs of diffusion-sensitive field-gradient pulses are introduced into the RF pulse sequence, one of which is placed in the τ_1 intervals and the other, in the τ_2 intervals. The amplitudes of the SE and STE signals can be shown to be, respectively,

$$S_{SE} = \cos\theta \exp[-2\tau_2/T_2 - \gamma^2 g_1^2 D_1 \delta_1^2 (\Delta_1 - \delta_1/3)], \quad [1]$$

and

$$S_{STE} = (1/2)\sin\theta \exp[-TM/T_1 - 2\tau_1/T_2 - \gamma^2 g_2^2 D_2 \delta_2^2 (\Delta_2 - \delta_2/3)], \quad [2]$$

where γ is gyromagnetic ratio, D_1 and D_2 are diffusion coefficients along the two respective directions and T_1 and T_2 are spin-lattice and spin-spin relaxation times, respectively. The two pairs of gradients can be either parallel or orthogonal to each other, depending on a particular application; δ, Δ and g denote the duration, spacing and the amplitude of the gradient pulses, respectively (Fig .1), with the subscripts indicating different gradient-pulse pairs.

For imaging, the first and second pulses are slice-selective, and the other pulses remain nonselective. The field gradient, g_x, is applied in the τ_1 and τ_2 intervals to phase encode

spatial information in x-direction of the SE and STE. The echoes are recorded in the presence of a second field gradient, g_y, in separate data collection windows. The SE and STE images can be obtained via the standard 2-D Fourier transformation.

The pulse sequence illustrated in Fig. 1 not only induces the desired SE and STE, but also leads to other unwanted echoes and free induction decay signals. The strong diffusion-sensitive gradient pulses coincidentally help remove all the unwanted transverse responses except a spin echo appearing in the second acquisition window, which interferes with the STE. The unwanted spin echo can be efficiently removed via a two-step RF phase cycle.

Quantitative confirmation of this method was made by obtaining the diffusion coefficient (*D* value) for doped water at 18°±0.5°C. All experiments were carried out in a GE CSI-II 2.0T/45cm imaging spectrometer operating at 85.5 MHz for protons, equipped with self-shielded gradient coils capable of producing maximum field strength of ±20 G/cm. The SE and STE signals from the whole water sample were recorded. The z-gradient pulses inserted in the τ_2 intervals were applied with δ=10 ms, Δ=50 ms and the amplitude incremented from 1.2 to 12 G/cm, in 1.2 G/cm steps. For the x-gradient pulses placed in the τ_1 intervals, δ=10 ms, Δ =350 ms, and the amplitude was incremented from 0.4 to 4 G/cm in 0.4 G/cm steps. For these measurements, TR =8 sec, τ_1=20 μs, and τ_2=50 μs. Figure 2 shows two semilog plots of the peak heights against the square of gradient strength. The solid lines are linear fits to the two sets of data points. The *D* value was calculated from the slopes of the straight lines, yielding $(1.83\pm0.01)\times10^{-5}$ cm²/s from the SE and $(1.82\pm0.01)\times10^{-5}$ cm²/s from the STE.

The imaging method is currently used to obtain NMR images of the rat brain *in vivo*. Two diffusion-sensitive images can be obtained with the gradient pulses g_1 along the direction in which the restricted diffusion of tissue water is dominant since large Δ_1 can be set without considerable signal loss. We hope that this method will be useful for diffusion measurements for the molecules diffusing relatively free in one direction while being impeded by barriers in another direction in systems where the T_1 of the diffusing substance is greater than T_2.

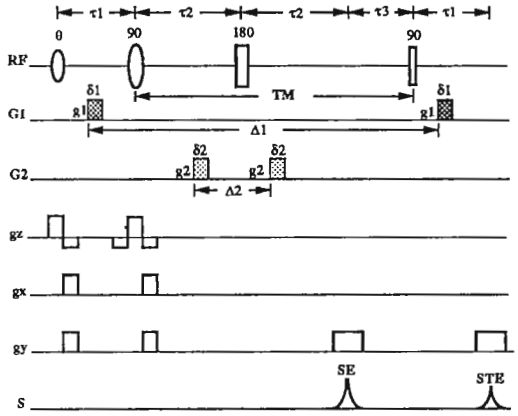


Fig. 1. A pulse sequence for anisotropic diffusion imaging.

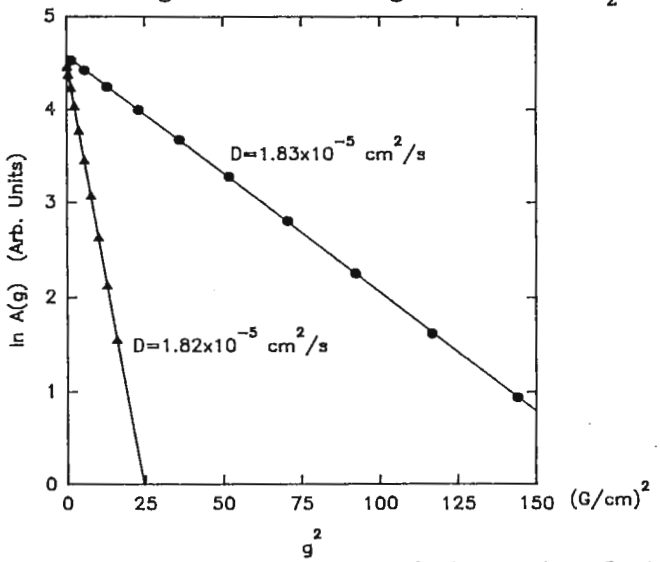


Fig. 2. The semilog plots of the SE (circles) and STE (triangles) vs the square of g_1 or g_2.

Sincerely,

Limin Li and Chris Sotak

CLARK UNIVERSITY
950 Main Street Worcester Massachusetts 01610-1477

Department of Chemistry

Telephone (508) 793-7116
FAX (508) 793-8861

April 11, 1991
(received 4/19/91)

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

Spin Diffusion and Molecular Dynamics in Solid State 2D Experiments

Dear Barry:

In solid state 2D exchange spectroscopy off-diagonal intensity can be produced by both spin diffusion and molecular reorientations in the appropriate timescale of the mix time. Depending on one's interest in either structure or dynamics it is desirable to eliminate the appropriate one of these from the 2D pattern. Our interest is primarily solid state dynamics and so identification and elimination of spin diffusion contributions to off-diagonal intensity is desirable. The extensive use of deuteron spectroscopy for dynamic studies in this regard avoids the problem since spin diffusion is slow even in fully deuterated materials due to the small dipolar coupling between deuterons. However, in the case of spin 1/2 nuclei such as ^{13}C or ^{31}P, where spin diffusion can be appreciable, we have found that simple methods can be empolyed to conveniently identify and eliminate spin diffusion contributions and retain the information content of the 2D pattern with respect to reorientation dynamics.

In the 2D exchange experiment the primary source of spin diffusion is between resonant nuclei of different orientations in H_o. In contrast to dynamics the off-diagonal intensity due to this spin diffusion will be temperature independent in the ms to sec timescale unless the solid mobility is such that the dipolar coupling is significantly reduced. This allows for the identification of spin diffusion. The ^{13}C pattern in in fig. 1a and b for a polymer blend, one component of which has a labeled ^{13}C site, is an example. The suppression of spin diffusion is easily accomplished by either:

(a) isotopic dilution of the observed nucleus

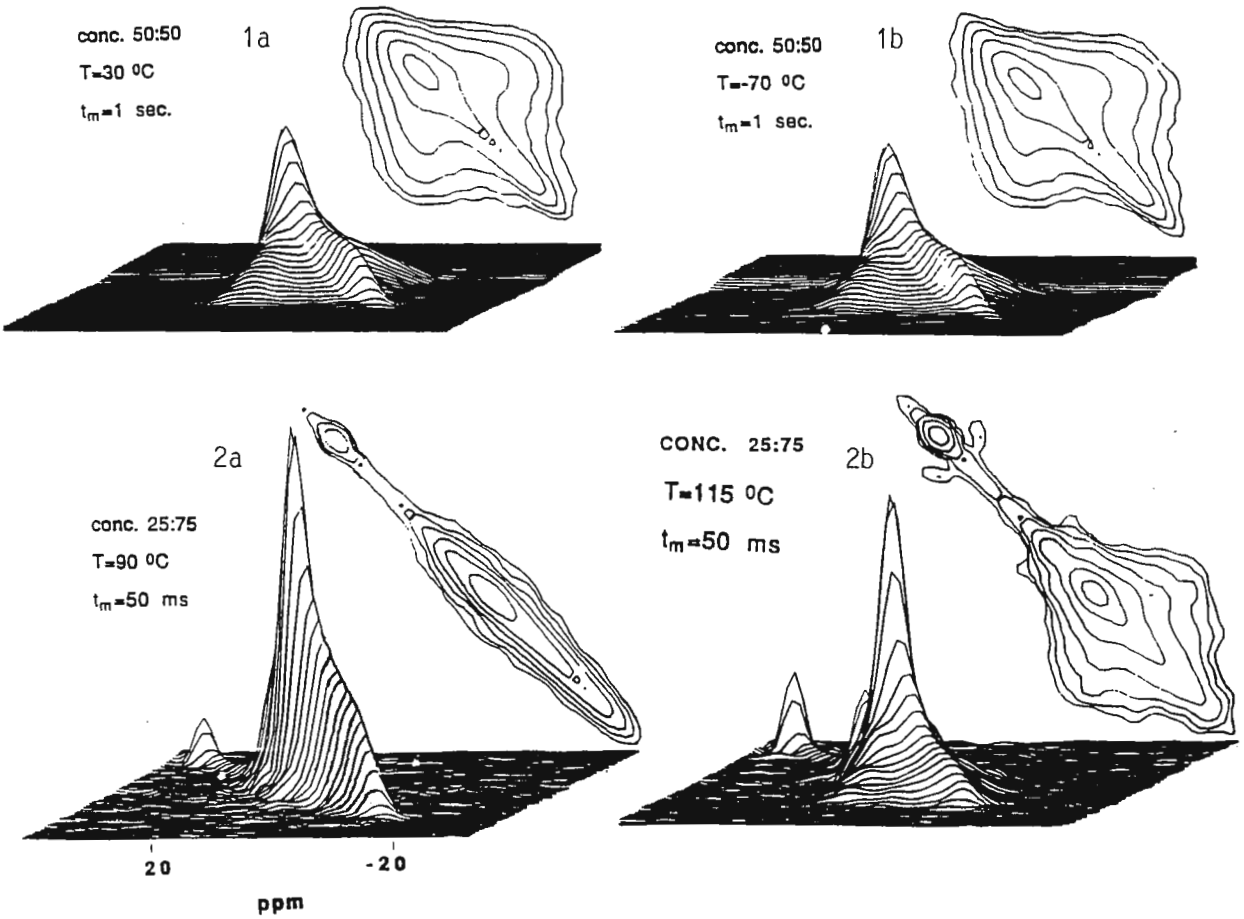
(b) selection of a mix time range fast of the spin diffusion rate

(c) selection of a temperature and mix time regime appropriate to the dynamics and not spin diffusion

Fig. 2a shows the effect of dilution and mix time, and 2b the effect of temperature showing the onset of molecular dynamics. In the systems we have been interested in either one of the above considerations or a suitable combination has been convenient with respect to eliminating spin diffusion effects from the data.

Sincerely,

Paul T. Inglefield



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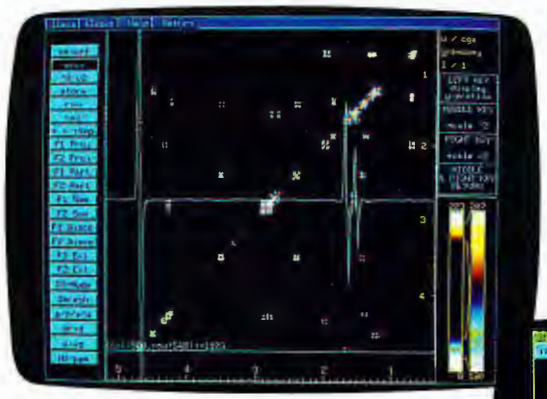

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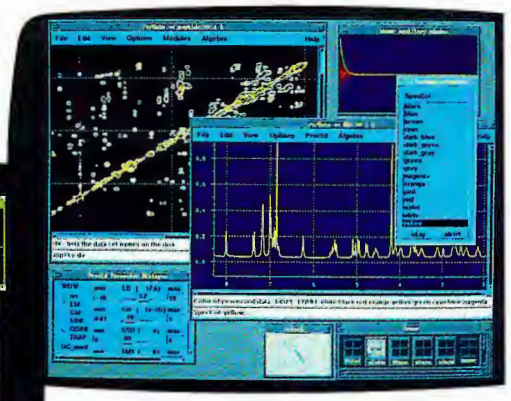

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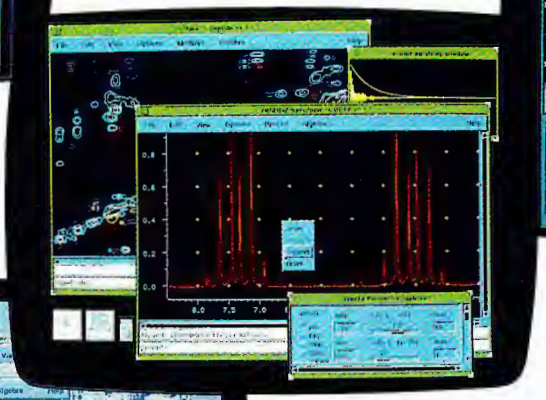
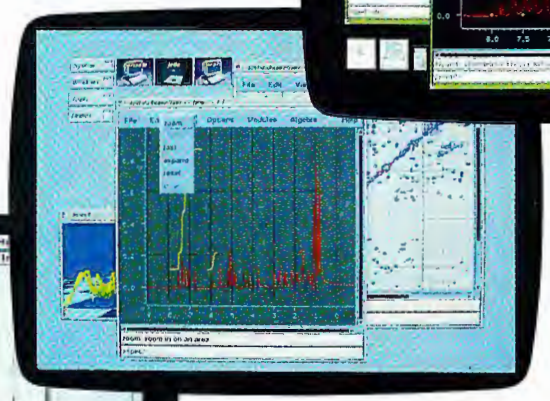
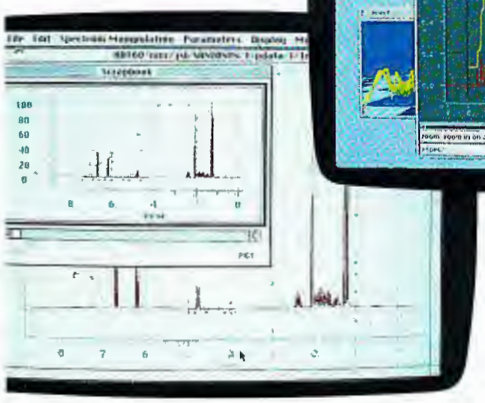


UXNMR on your SUN SPARCstation.

UXNMR on your SGI/IRIS.



WINNMR on your Mac II or PC/PS system.



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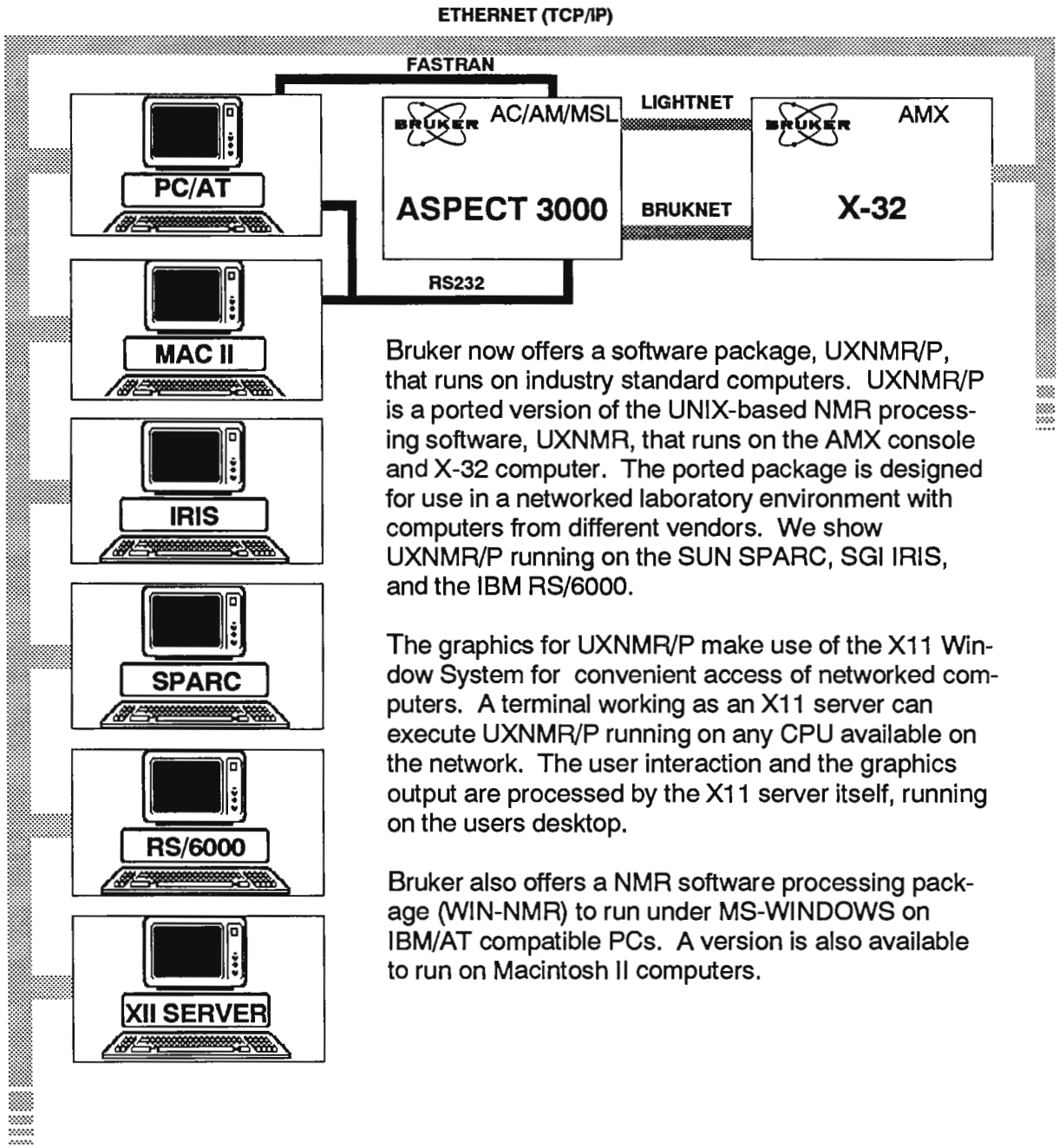

Bruker now offers a software package, UXNMR/P, that runs on industry standard computers. UXNMR/P is a ported version of the UNIX-based NMR processing software, UXNMR, that runs on the AMX console and X-32 computer. The ported package is designed for use in a networked laboratory environment with computers from different vendors. We show UXNMR/P running on the SUN SPARC, SGI IRIS, and the IBM RS/6000.

The graphics for UXNMR/P make use of the X11 Window System for convenient access of networked computers. A terminal working as an X11 server can execute UXNMR/P running on any CPU available on the network. The user interaction and the graphics output are processed by the X11 server itself, running on the users desktop.

Bruker also offers a NMR software processing package (WIN-NMR) to run under MS-WINDOWS on IBM/AT compatible PCs. A version is also available to run on Macintosh II computers.

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April 5, 1991
(received 4/8/91)

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

Dynamics of *trp*-repressor molecules from ^{15}N reverse experiments

Dear Dr. Shapiro,

We are studying the molecular dynamics of the *trp*-repressor from *E. coli* (a 25000 kD dimer). In each monomer there are six helices denoted as A, B, C, D, E and F. Helices A, B, C and F constitute the hydrophobic core of the molecule while D and E are the DNA-binding region. The structure of *trp*-repressor depends on whether it is L-trp bound (holorepressor) or L-trp free (aporepressor). Our studies focused on both forms of *trp*-repressor and involved proton detected ^{15}N-^{1}H correlated spectroscopy of uniformly ^{15}N labeled samples. In order to investigate the exchange of NH protons to deuterons we monitored the changes in intensities of individual lines in 2D correlation spectra after adding D_2O to the sample. The results show that protons from the core of the *trp*-repressor molecules are replaced by deuterons typically after many hours while those from the DNA-binding region undergo a very rapid exchange. Usually their signals cannot be observed even in the first 1D exchange spectrum recorded in ~20 min after adding D_2O. The exchange times exhibit a strong temperature dependence. Moreover, the exchange times for holorepressor in the core of the molecule are approximately three times longer than the corresponding values for aporepressor. The exchange times for residues from the D and E helices are of the order of milliseconds and were determined from the ^{15}N relaxation spectra. Fig. 1 and Fig. 2. show the plots of exchange times vs. residue number for aporepressor at 308 and 318°K.

Sincerely,

Jerzy Czaplicki

(Credit to the account of Oleg Jardetzky)

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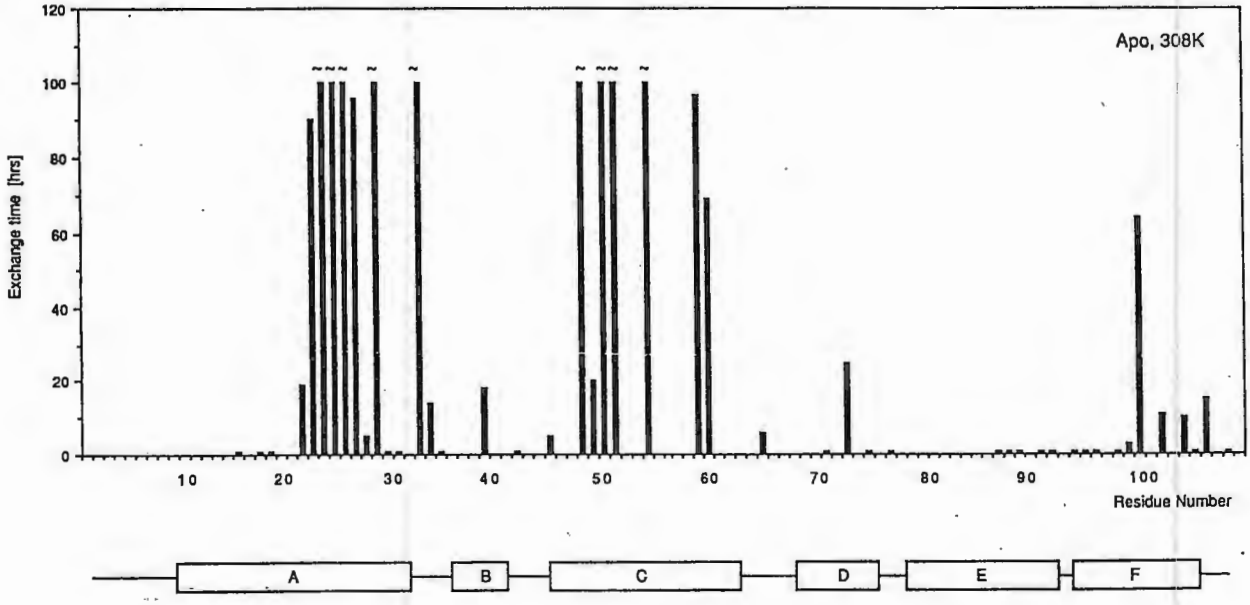

Fig.1. Exchange times for aporepressor at 308K as a function of the residue number. "~" denotes times longer than 100 hrs that could not be measured accurately.

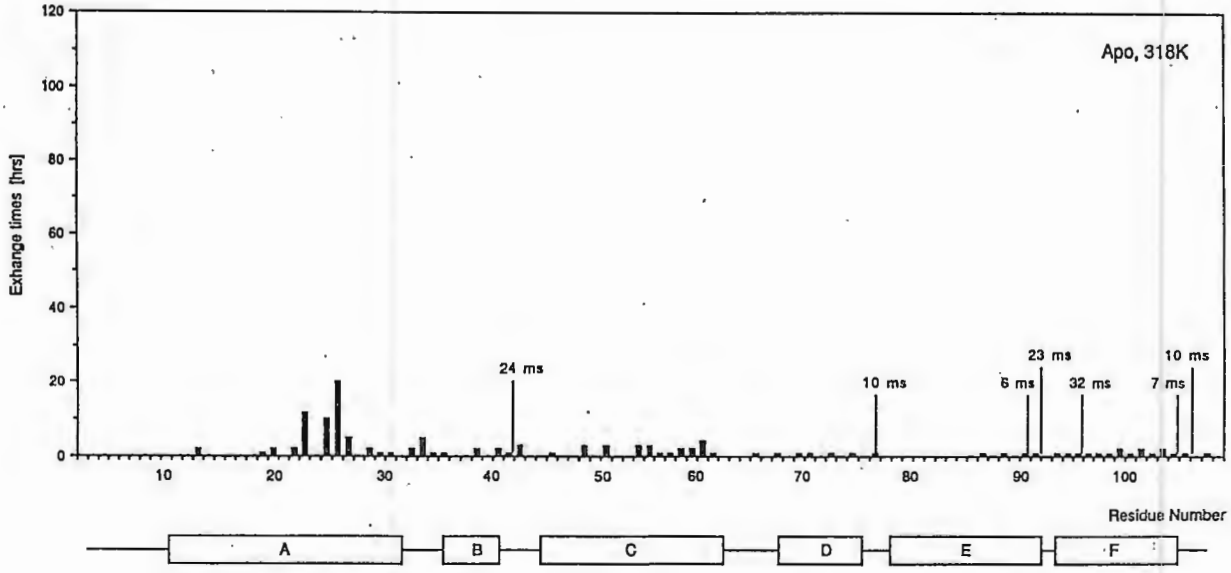

Fig.2. As in Fig.1, but for 318K. The exchange times in the millisecond time range were determined from ^{15}N relaxation spectra.

TWO-DIMENSIONAL NMR SPECTROSCOPY

Friday-Sunday, August 23-25, 1991
In Conjunction with the 202nd ACS National Meeting, New York, NY

Who Should Attend

Chemists who are familiar with structural organic chemistry and have a basic understanding of 1-D proton NMR. No prior knowledge of 2-D NMR is assumed.

Key Topics You'll Learn About

- Principles and basic concepts of 2-D NMR
- Galibration and selection of experimental parameters
- Implementation of new pulse sequences
- Strategy for selection of 1-D and 2-D NMR techniques
- The most useful types of 2-D experiments
- The most efficient methods of assigning chemical shifts and extracting both homo- and hetero-coupling constants
- Ways to set up and optimize 2-D experiments
- What is on the horizon for NMR instruments and techniques

Seven Key Ways You'll Benefit from This Course

1. Be able to read and understand current literature which now makes extensive use of 2-D NMR experiments
2. Learn how to select the best 1-D or 2-D NMR experiments to solve specific molecular structure problems
3. Gain special insight to decisions on digital resolution and on the use of weighing functions in both 1-D and 2-D experiments
4. Learn four criteria for distinguishing "real signals" from noise in carbon-carbon double quantum experiments
5. Understand how to deal with specific problems caused by improper calibrations or improperly selected NMR parameters
6. Be exposed to different philosophies and attitudes in solving chemical structure problems using 2-D techniques
7. Understand how to communicate with your NMR people if you do not analyze your own samples

About the Instructors

LeRoy F. Johnson, Manager, Analytical R&D, GE NMR Instruments, is the author or co-author of more than 80 papers dealing with a wide variety of applications of NMR spectroscopy, as well as the the co-author of the book, Carbon-13 NMR Spectra. He is co-author with Roy Bible of the ACS Audio Course, "Interpretation of NMR Spectra."

Roy H. Bible, Jr., Senior Fellow and Director of Physical Methodology, G. D. Serle & Company, is the author of Interpretation of NMR Spectra: An Empirical Approach and A Guide to the Interpretation of NMR Spectra--A Workbook.

Program Agenda

Check-in will begin at 8:30 a.m. the first day. The course will run from 9:00 a.m. to 5:30 p.m. each day.

Day 1

Review of 1-D Techniques (1H, 13C, Gated and Selective 1H Decoupling, APT, DEPT, and INEPT)
Basic Concepts in FT-NMR (Rotating Frame, Transmitter and Receiver Phase Cycling
Principles of 2-D NMR (Generation of 2-D Data, Types of Plots, the COSY Experiment)
Problem Session (COSY)
Heteronuclear Experiments (Polarization Transfer, the HETCOR Experiment)
Problem Session (HETCOR)
Calibration and Selection of Experimental Parameters (the NOESY Experiment)
Review/Questions & Answers

Day 2
Problem Session (NOESY)
Additional Concepts (Composite Pulses, Broadband Decoupling, Pure Phase 2-D Experiments)
Additional Pulse Sequences (RELAYH, HOMO J and Selective HOMO J, Soft Selective INEPT)
Problem Session (RELAYH, INEPT)
Additional Pulse Sequences (Heteronuclear Long Range J Correlation, Carbon-Carbon Double Quantum)
Problem Session (LRHETCOR, INADEQUATE)
Implementation of New Pulse Sequences
Review/Questions & Answers

Day 3
Problem Session (LRHETCOR, INADEQUATE)
Advanced Techniques (Use of Spin-Locking Fields, The HOHAHA and ROESY Experiments, Inverse Detection Experiments)
Problem Session (HOHAHA, ROESY, RPT)
Case Studies (Determination of Complete Structure and Stereochemistry
Summary (Strategy for Selection of 1- and 2-D Techniques)

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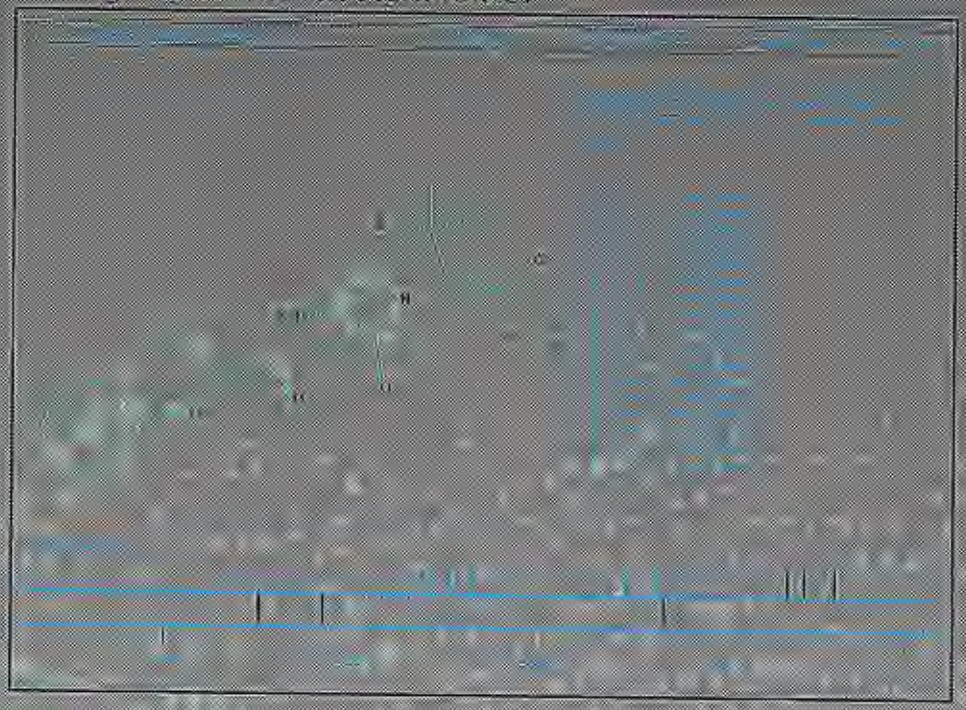

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(received 4/19/91)

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

A Cookbook for Felix on SGI and Sun Workstations

Dear Prof. Shapiro,

What do you do when you have 200 users of your NMR facility and a rapidly changing hardware and software environment for these users? This is an ongoing problem that faces the Molecular Spectroscopy Lab staff here at the University of Illinois. Theoretically, now is the time to anticipate the user's needs, subdivide the experiments and processing packages, and prepare training materials for each function. Training materials are prepared for specific activities and the users can then select the training (or reorientation) handout for the specific task that confronts them. Since both the user's workspace environment and the demands of his/her research change, up-to-date training materials must be efficiently produced and updated.

As an example, the plummeting cost of very powerful graphic workstations has led a proliferation of these computers among our users. The recent improvements in these workstations have lead to increased speed and quantity of NMR data that can be efficiently processed. However, as is often the case, software is the primary limitation. Let's face it, the UNIX operating system of these workstations was designed by hackers and worse, designed for hackers. The two most popular software packages, (Hare Research Inc's "Felix", and NMRi's "NMR1, MNR2") both originated as tools designed for expert users. Both companies have made efforts to simplify their programs' interfaces and concepts; however, we have found that often 10-20 hours of training is required to produce confident users, rather than the one to two hours which is expected to learn a high quality Macintosh program.

Although we have had Hare's programs available for some time in our lab, we are now producing a three-part cookbook that introduces users to felix on the Silicon Graphics IRIS and Sun Sparc workstations. Our hope is to cut the time necessary for our users to become competent at data workup with Felix. This cookbook is accompanyed by a tape of data sets that are used in the tutorials. In addition to introducing the user to the concepts behind Felix, he/she is also provided with procedure templates which can be altered for personal data analysis, as well.

Part one of the cookbook covers (completed):

1) conversion of data to Felix format
2) 1D data processing, includes apodization functions, baseline correction, etc.
3) accumulation of multiple 1D spectra into a matrix for T1IR or temperature studies
4) 2D processing of magnitude COSY and NOESY data sets
5) 2D processing of States-type phase-sensitive COSY and NOESY data sets
6) strategies for phasing and baseline correction of 2D data sets

Part two of the cookbook covers (in testing):

1) more complex 2D data analysis such as PE COSY
2) extraction of coupling constants
3) strategies for assignment based on COSY and NOESY data

Part three of the cookbook covers (in preparation):

1) model building
2) simulation of NOESY spectra
3) refinement strategies

These materials will be made available on a "you send us an appropriate blank tape basis", as the sections are completed. Please contact us in advance regarding media since even this is in a state of transition right now. We hope to move away from 1/4 inch data cartridges to CD and/or DAT. We suggest that you have the appropriate conversion program for your spectrometer data to Felix format and that your users are somewhat familiar with the *vi* editor. We will also provide handouts on "20 common Unix commands" and "A Minimalist's Guide to *vi*".

A similar set of tutorials is also being prepared for the NMRi data analysis software, and we will report on this later. We can also make available a small program which makes it possible to port the processed spectra to a Mac via ethernet where final thesis/publication quality spectra can be produced with a graphics program such as Abobe Illustrator. Please send requests or direct questions to MFL at above address.

Please credit this submission to the account of Dr. Vera V. Mainz. Thank you.

Sincerely,

Dr. M.F. Leopold
leopold@aries.scs.uiuc.edu

Dr. Howard Robinson
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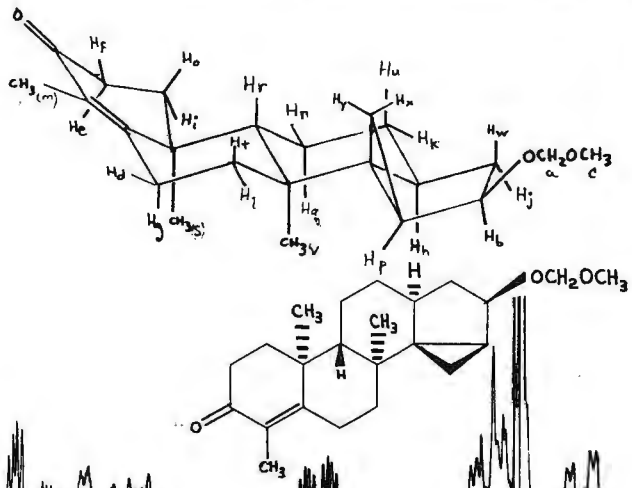

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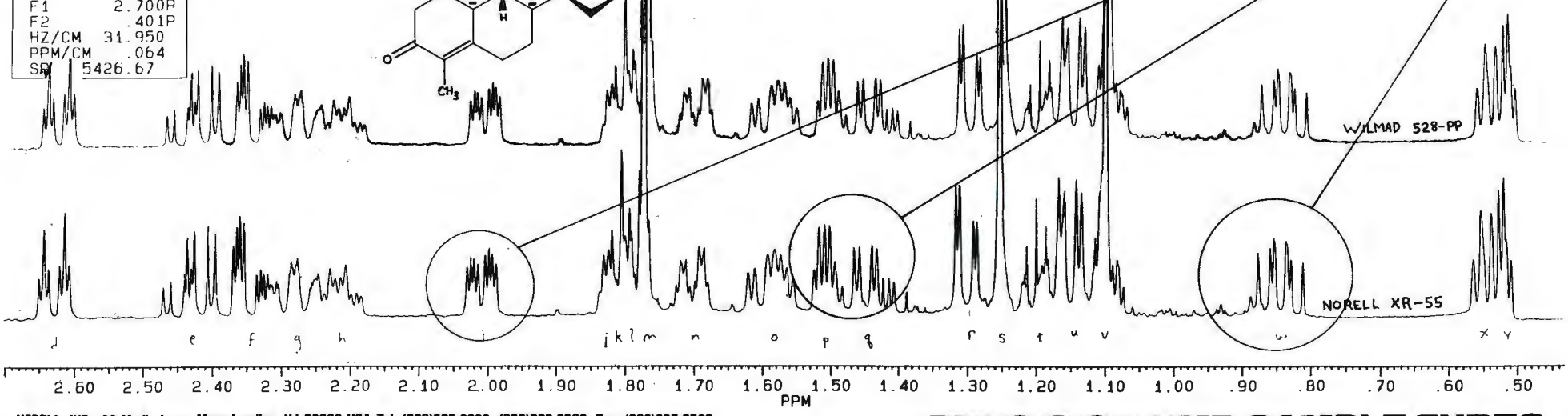

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(received 4/17/91)

Dr. Bernard L. Shapiro,
TAMU NMR Newsletter,
966 Elsinore Court,
PALO ALTO, CA 94303
USA

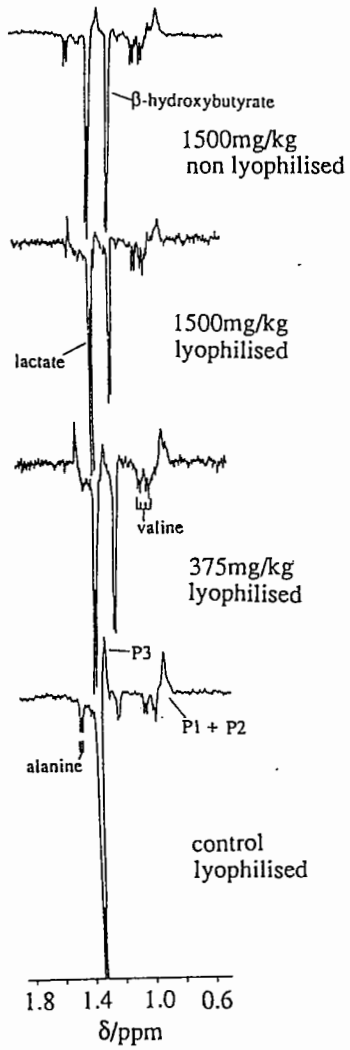
10 April, 1991

Dr. Shapiro,

NMR of plasma: detection of an elevated enzyme activity

As part of an on-going and very fruitful collaboration with Jeremy Nicholson of London University, we have a graduate student, Maria Anthony, who is studying aspects of kidney toxicity using NMR of body fluids.

Usually ^1H NMR spectra of plasma are measured using a simple spin-echo, 90–τ-180–τ-acq, sequence with τ=68ms to cause vicinally coupled doublets to appear inverted. During a series of experiments, Maria noticed that the alanine methyl signal was sometimes an inverted doublet and sometimes it was an upright singlet. In plasma from control animals either untreated or freeze-dried and resuspended into D_2O, a normal inverted doublet was seen. In animals dosed with a particular kidney toxin, the untreated plasma showed an inverted doublet but if this plasma was freeze dried and reconstituted into D_2O, then the inverted doublet gradually over a period of time changed into an upright singlet. This is shown in the attached figure which shows, from the bottom, a control spin echo spectrum, the result of dosing with the toxin at 375mg/kg and 1500mg/kg both from lyophilised plasma reconstituted into D_2O and, at the top, plasma after dosing at 1500mg/kg but not lyophilised.



We believe that, in the plasma from the toxin treated animals, there is an elevated level of alanine aminotransferase (ALT) which catalyses the reaction

alanine + 2-oxoglutarate ↔ pyruvate + glutamate

and hence in D_2O this leads to the deuteration of the α-CH of alanine, the loss of vicinal coupling and the refocussing effect. We are in the process of characterising this phenomenon further because ALT is widely used as a quantitative marker of liver and kidney damage by clinical biochemists and toxicologists. Thus NMR may represent an exceptionally convenient and independent method in this area.

Yours sincerely,

John

DR. J.C. LINDON
Department of Physical Sciences

392-42

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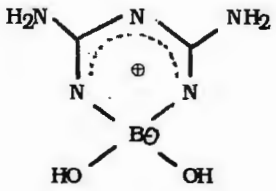
9 April 1991 (received 4/18/91)

Dr Bernard Shapiro
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto CA 94303
UNITED STATES OF AMERICA

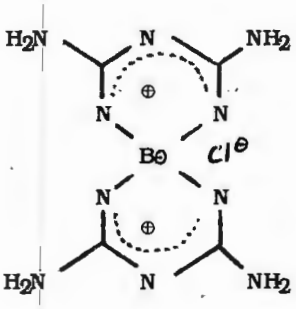
Dear Dr Shapiro

RE: ^{11}B NMR OF BORON NITROGEN HETEROCYCLES

We have recently been honing our synthetic skills with respect to boron-nitrogen heterocycles. Compounds I-VI were synthesised and looked at by solution and solid state NMR. The solid state spectra were recorded on a MSL-400 using a probehead free of boron background (vespel stator) which I had access to while in Germany. Solution spectra were recorded on an AC-200 in D_2O.

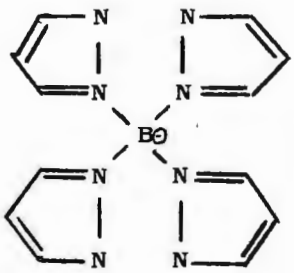


I



II

III II crosslinked with N-methylol groups



IV $K^{\oplus}$ salt
V Ca^{2+} salt
VI $H^{\oplus}$ chelate

Table of ^{11}B Chemical Shifts (reference external $BF_3.OEt_2$)

		I	II	III	IV	V	VI
δ^{11}B (ppm)	solution 64.2 MHz	-1.5	5.6	-	3.3	3.3	-1.9
	solid 128.3 MHz	-	-6.2	-10.6	-	-	-

Of particular note is the change in shift between IV, VI and V due to the chelation of $H^{\oplus}$. There is an observable increase in linewidth from ca. 3 Hz to 50 Hz when chelated with $H^{\oplus}$. Compounds II-VI are stable for several months at pH 3. As yet we are unable to explain the difference between compound II solution and solid state chemical shifts.

Regards.

Roger Meder

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Tübingen, den 18.3.1991
(received 3/23/91)

Physikalisches Institut, Morgenstelle, D-7400 Tübingen

Prof. Dr. Bernhard L. Shapiro
Editor TAMU NMR Newsletter
966 Elsinore Court
Palo Alto, California 94303, U.S.A.

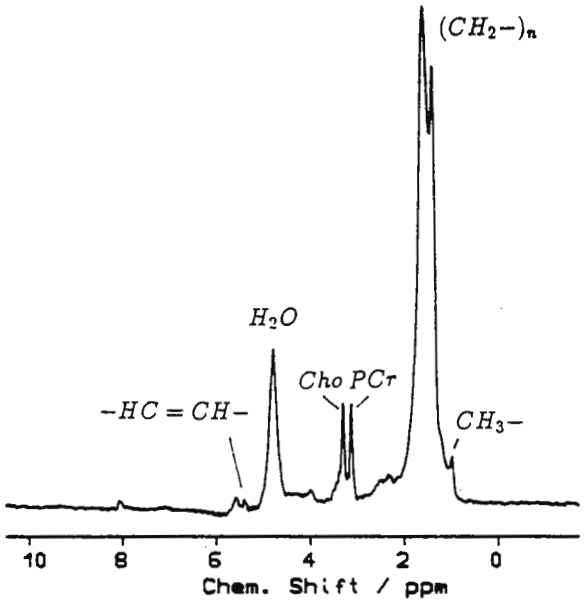
Localized Proton NMR Spectroscopy with a 1.5 T Whole Body Imager

Dear Barry,

we have proceeded with volume selective ^1H spectroscopy with the whole body imager of the University of Tübingen. Modified double spin echo sequences with variable echo times have been implemented and carefully adjusted. Experiments with phantoms showed good localization features and high resolution and stability. In the meantime the sequences have been tested in vivo with volunteers in different organs in cooperation with H. Bongers of the Radiologische Klinik , Universität Tübingen. Patient studies are also running. A typical in vivo spectrum is given below.

Sincerely

(Otto Lutz) (Wulf-Ingo Jung) (Fritz Schick)



Double spin echo in vivo ^1H NMR spectrum of human calf muscle:

Volume: $(13\ mm)^3$
Echo time: 100 ms
Repetition time: 1.5 s
Acquisitions: 256
Measuring time: 6 min.

**Department of Chemistry,
University of Cambridge,
Cambridge, CB2 1EW,
England.**

(received 3/26/91)
19th March, 1991.

Multiplicity Editing of ^{13}C Spectra Using Heteronuclear Isotropic Mixing.

Dear Dr Shapiro,

Recently it has been demonstrated that pulse sequences borrowed from broad-band heteronuclear decoupling, such as WALTZ and MLEV, can be used successfully for in-phase heteronuclear polarization transfer in liquid state NMR. This until now has been almost exclusively carried out using pulsed techniques based on INEPT and DEPT.

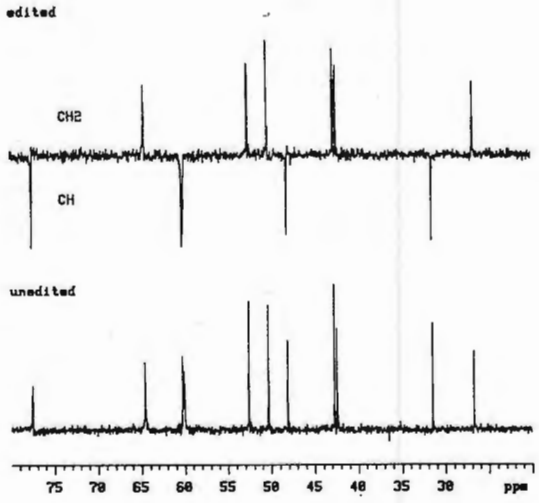
Here we show that heteronuclear isotropic mixing can be used to improve multiplicity editing of ^{13}C spectra. Our sequence achieves separation without complicated post-acquisition manipulations, such that the edited and unedited spectra have equal intensities, neglecting relaxation effects. The pulse sequence and phase cycle we use are shown below.

^1H:	90°(±y)	WALTZ8(x)	-	Δ	-	Ω(x)	- Δ -	Dec.
^{13}C:		WALTZ8(x)				180°(x)		Acq(±y).

Note that only a two-step phase cycle is required. The first 90° pulse on protons followed by the heteronuclear isotropic mixing sequence generates in-phase carbon magnetisation. Multiplicity editing is then achieved during the delay 2Δ; when Ω is set to 180°, chemical shift is refocused but not the heteronuclear coupling and the spectrum is edited. When Ω is set to 0°, the heteronuclear coupling is refocused and the spectrum is not edited. If the delay Δ is chosen to equal 1/(2J), and Ω is set to 180°, the methylene signals have opposite sign to the methyl and methine signals. Any magnetisation which remain because of incomplete refocusing due to variation in J values is anti-phase so will be cancelled by broad-band heteronuclear decoupling during acquisition. Experimental results obtained using a sample of strychnine in $CDCl_3$ are shown below.

The following advantages are gained (a) full intensity 'subspectra' are obtained, thus sensitivity is not reduced through cosine and sine modulations; (b) no post-acquisition manipulation of the spectra is required to achieve separation; c) clean editing is obtained over a range of J values - only the intensity of the signals is affected; and (d) the signals are pure absorption.

Please credit this to the account of Dominique's father.



Tobias Domke, Esq.

Lisa McIntyre.

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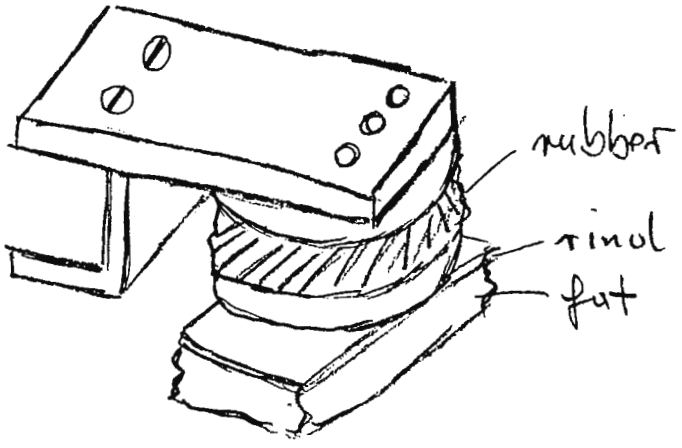
Prof. Dr. B. L. Shapiro
TAMU-NMR Newsletter
966 Elsinore Court
Palo Alto, Cal. 94303
USA

A method no manufacturer will approve of - or high field on bacon

Dear Barry,

To get room for our new Bruker AMX-500 spectrometer we had to move our AC-300 with sample changer about 7 meters. Considering the costs in time and money if we brought the system down to transport the magnet properly, we decided to take the risk and move it on field.
We will document here a method by which this can be done rather safely if one has (i) not a carpeted floor and (ii) if one owns already the standard shock mountings provided by Bruker against floor vibrations.
We released the air from the shock mountings and took them off the magnet's base. Under each of the three shock mountings a strip of about 10 x 20 cm of bacon (the German variety of 100% fat with rind) with ca. 4 cm thickness was placed. With the bacon underneath the shock mountings were put back and air pressure applied to them. Now the magnet could easily be pulled by one person to the desired place, which I did rather slowly and carefully.
By this method the total downtime of the instrument was about 5 hours and the sample changer went into work on the same day the instrument was moved. Besides the delicious smell which hanged around in the nmr lab for several days the method had one more disantvantage: nobody in the department was willing to fund the pound of bacon bought in the local supermarket...

Sincerely yours



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?? July 25, 1989 ??
(received 3/22/91)

Dr. Bernard L. Shapiro
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto, CA 94043

UCSF Sparky: Multi-spectra Assignment Software

Dear Dr. Shapiro:

We report here on the development of software for interactive assignment of multiple spectra. The major design goal is to provide a fully graphical environment that will handle equally well automated and manual interpretation methods. This requires an interface to automatic methods as well as an auditing scheme for manual methods.

Some of the current features of Sparky are:

- Multiple 2D spectra, with multiple views possible in each. Each view can display bitmap or contoured data. Views can be independently sized, scaled, transposed and scrolled. Synchronization between views is possible by coordinated scrolling and cursors. Slices through the data can be shown at the edges of each view.
- Automatic or manual peak selection. Integration of peaks inside a box or ellipse, or by linefitting. Deconvolution of overlapped peaks by linefitting. Multiplets and fine-structure can be combined into single cross-peaks.
- Annotations such as peaks, cross-peaks, labels, lines (showing connectivity) and grids (an alignment aid.) Annotations scale with the spectra (see figure.) Notes attached to annotations can provide further details.
- Other features include: mouse- and menu-driven with keyboard accelerators, dialog boxes for settable parameters, save and restore, printed output to PostScript printers.
- Hardware/software requirements are a Sun workstation and OpenWindows 2.0.

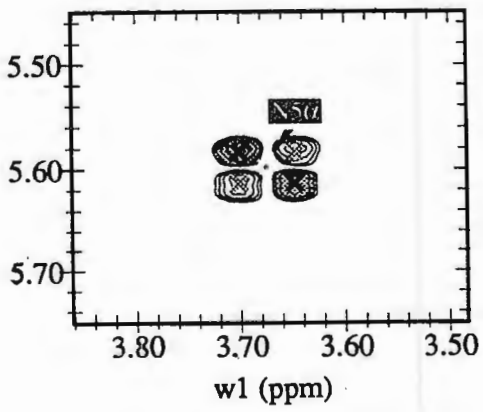
Future directions include:

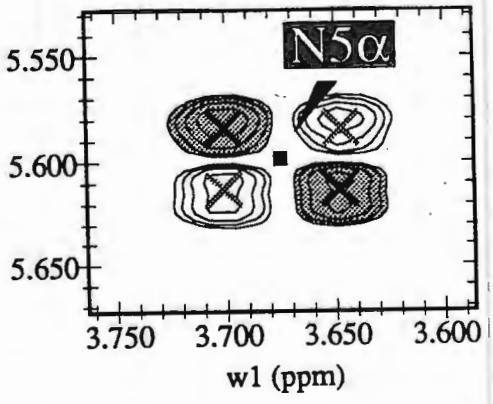
- 3- and higher-dimension support through the use of 2D data planes along arbitrary axes.
- Higher level structure (such as sequences -> atoms -> resonances -> cross-peaks) and data base with browsers.
- Interface to automated methods, with manual and automatic updating of the data base.

Sparky is currently in use at UCSF and will be available in source form for a nominal fee to educational institutions.

Sincerely,

Donald Kneller Tack Kuntz Tom James





Gradient Enhanced Spectroscopy

S-17 GES Accessory

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The S-17 is designed to mount and operate like a standard high resolution probe and will easily interface with an Omega PSG system. Control of the S-17 is accomplished with the Gradient Amplifier accessory which provides shaped gradient control for each axis via the standard pulse sequence language and 16 bit pulse sequence generator boards.

Typical Applications

Implemented GES phase pulse sequences include:

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- GE-NOESY
- GE-TOCSY
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- GE-HMQC-TOCSY 15N-HH

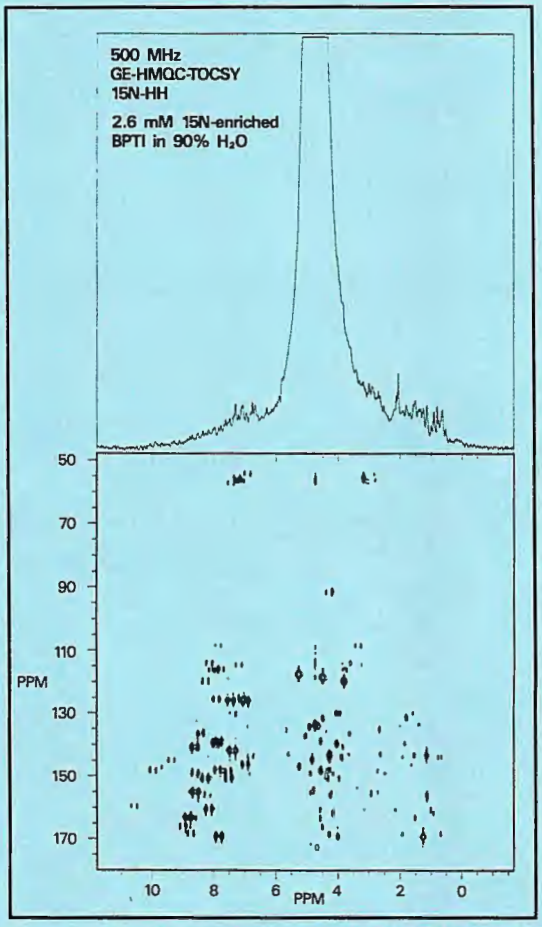

Fig. 1 — Gradient Enhanced HMQC-TOCSY at 500 MHz

Gradients have been applied to HMQOC-TOCSY experiments at 500 MHz and confer many significant advantages to this class of experiments. Foremost of these advantages is the excellent water suppression without need for presaturation or selective excitation. This enables the observation of exchangeable protons and those resonances which are close to or overlapping with the intense water signal. Gradient pulses have proven to be very efficient at suppressing the signal from the ^{15}N bound protons as well. As only the desired signal components are detected in each scan, dynamic range limitations and signal cancellation problems are eliminated.

TOCSY filtered ^{15}N HMQC of 2.6mM BPTI (^{15}N enriched) in 90% H_2O is shown in Fig. 1. The direct ^{15}N-^{1}H correlations and the relayed TOCSY connectivities are observed at the respective ^{15}N chemical shifts. The direct correlations exhibit the J(^{15}N-^{1}H) coupling along the acquisition dimension. The high degree of water suppression allows the observation of the relayed correlations which overlap with the solvent resonance. The data was collected on an Omega PSG 500 equipped with the S-17 Gradient Enhanced Spectroscopy accessory using a 5mm inverse probe.

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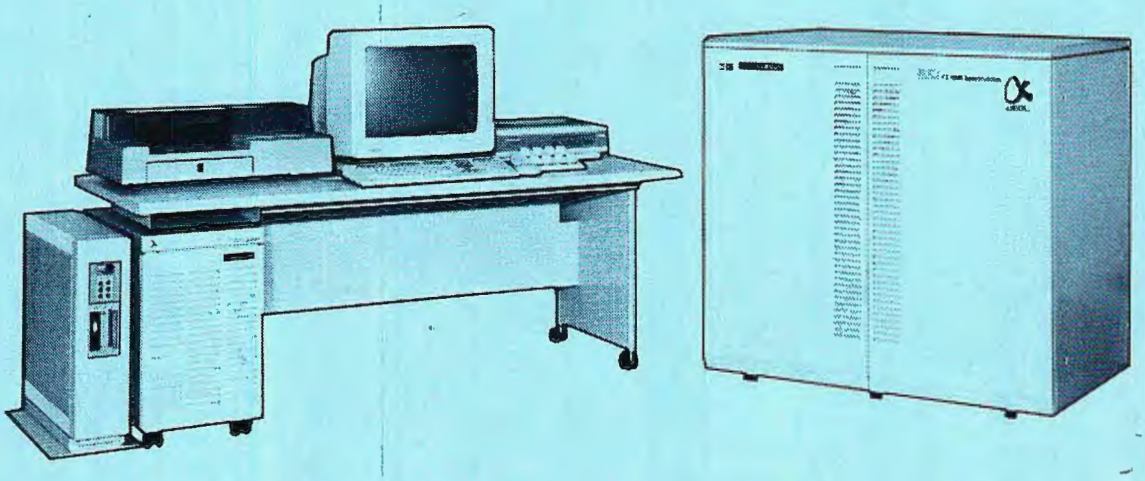

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