

TEXAS A&M UNIVERSITY

NMR

NEWSLETTER

No. 351

December 1987

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FORTHCOMING NMR MEETINGS

- 35th Annual Conference of the Western Spectroscopy Association, January 20-22, 1988; Pacific Grove, California; contact Heather Lafferty, WSA Secretary, P. O. Box 10640, Eugene, OR 97477.
- 39th Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, February 22-26, 1988; New Orleans, Louisiana; contact Mr. J. P. Auses, Exposition Chairman, Pittsburgh Conference, 12 Federal Drive, Suite 322, Pittsburgh, PA 15235.
- NMR-88 - Feb. 14-18, 1988; Thredbo Alpine Hotel, Thredbo, N.S.W. Australia. Chairman: Dr. L.R. Brown, The Australian National University. For further information, contact: Leslie Harland, Research School of Chemistry, The Australia National University, Canberra, A.C.T. 2601, Australia. Telex: AA62172. Facsimile: (61-62)-49-7817. Telephone: (61-62)-49-2863. See Newsletter 349, 20. Fritz Haber International Workshop on Modern Techniques in Magnetic Resonance, Dec. 13-17, 1987; Weizman Institute of Science, Rehovot, Israel; See page 7 of Newsletter #341, February 1987, for additional information.
- BRSG Meeting on Magnetic Resonance Spectroscopy of Colloidal Systems, April 5-7, 1988; Bristol, UK; contact Dr. N. Bodcn, Dept. of Physical Chemistry, The University, Leeds LS2 9JT, U.K.
- Magnetic Resonance in Colloid and Interface Science, April 6-8, 1988; Bristol, UK; contact Dr. T. Cosgrove, School of Chemistry, University of Bristol, Cantock's Close, Bristol BS8 1TS, U.K.
- 29th ENC (Experimental NMR Conference), Apr. 17-21, 1988; Rochester, New York; Chairman: Professor Stanley J. Opella, Dept. of Chemistry, University of Pennsylvania, Philadelphia, Pennsylvania 19104, (215) 898-6459. For information, contact Professor Edward O. Stejskal, ENC Secretary, Dept. of Chemistry - Box 8204, North Carolina State University, Raleigh, North Carolina 27695-8204; telephone (919) 737-2998.
- 9th EENC (European Experimental NMR Conference), May 16-20, 1988; Bad Aussee, Austria; For further information, write Professor H. Sterk, Karl-Franzens-Universität Graz, Institut fuer Organische Chemie, Heinrichstrasse 28, A-8010 Graz, Austria. See Newsletter 348, 15.
- 2nd European Congress on NMR in Medicine and Biology, June 23-25, 1988; Berlin, West Germany; contact Prof. R. Felix, Dept. of Radiology Charlottenburg University Hospital, Spandauer Damm 130, D-1000 Berlin 19, West Germany.
- XIII Intl. Conference on Magnetic Resonance in Biological Systems, Aug. 14-19, 1988; Madison, Wisconsin. See Newsletter 349, 60.

Additional listings of meetings, etc., are invited.

All Newsletter Correspondence
Should be Addressed to:Dr. Bernard L. Shapiro
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto, California 94303 U.S.A.
(415) 493-5971

DEADLINE DATES

No. 353 (February) -----	22 January 1988
No. 354 (March) -----	19 February 1988
No. 355 (April) -----	18 March 1988
No. 356 (May) -----	22 April 1988

Fachbereich 6
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Geb. MG

Datum

16.10.87

(received 10/26/87)

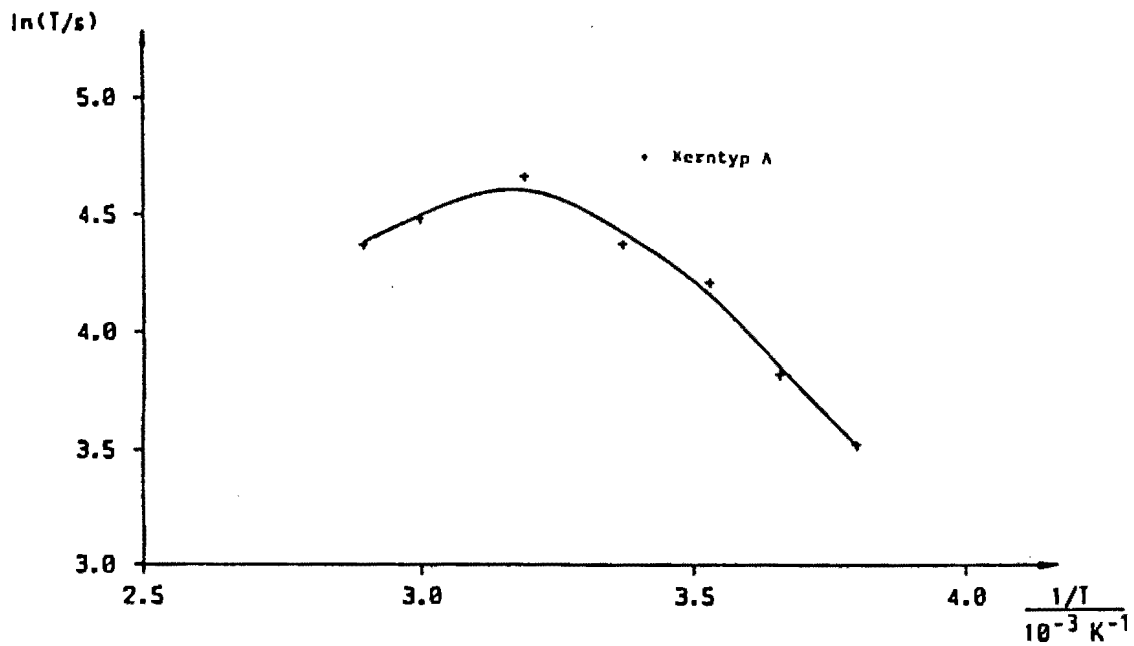
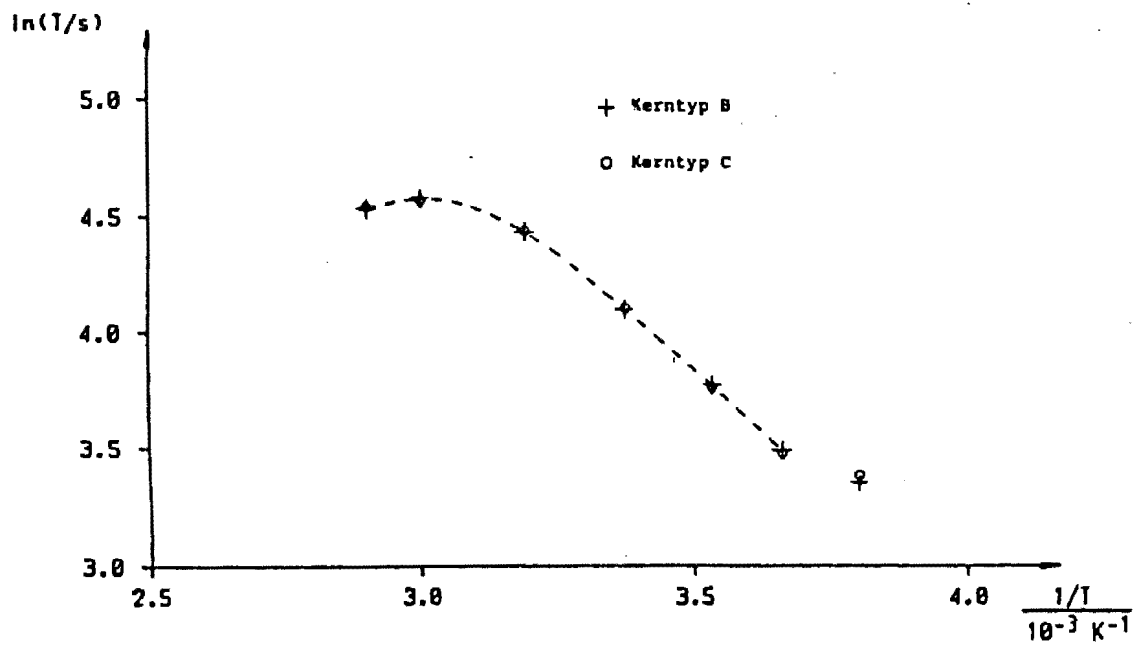
^{29}Si -RELAXATION IN $(\text{CH}_3\text{C}_6\text{H}_5\text{SiO})_3$

Dear Barry,

we have investigated the ^{29}Si -relaxation of the two isomers of $(\text{CH}_3\text{C}_6\text{H}_5\text{SiO})_3$ using the inversion-recovery-pulse-sequence. The silicons type A of the cis-isomer give a single resonance at -20.42 ppm in C_6D_6 , whereas the trans-isomer gives two signals at -20.38 ppm and -20.41 ppm with an intensity ratio of 2:1 for the silicon nuclei of the B and C type.

The T_1 -measurements were performed at two different field-strengths (7T and 9T) in order to separate the share of the chemical shift anisotropy. We found a contribution of about 26% at 9 Tesla and 17% at 7 Tesla for all of the three types of silicons. Thus the CSA-mechanism may have a significant share on ^{29}Si -relaxation.

The temperature dependence of T_1 for both isomers is shown in figures 1 and 2. The T_1 -time for the cis-isomer shows a maximum at 316 K. At this temperature the SR-contribution is equal to the sum of DD- and CSA-contribution. The maximum for the trans-isomer shows a shift of about 17 K to higher temperatures.



Yours sincerely

Robert Kosfeld
Robert Kosfeld

Christa Kreuzburg
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October 20, 1987
 (received 10/26/87)

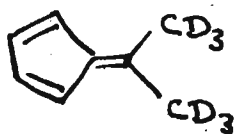
Dr. Bernard L. Shapiro
 TAMU NMR Newsletter
 966 Elsinore Court
 Palo Alto, California 94303

Dear Barry,

Since I suspect I will shortly be receiving one of your (in)famous pink warning slips, I thought I would try for once to beat you to the punch:

FULVENES AND DEUTERIUM - SHIFTLESS AND SHIFTY

In the course of a study of electronic effects in fulvenes, we prepared 6,6-bis-(trideuteriomethyl)-fulvene (1) to see whether we could observe long-range deuterium-



induced ^{13}C isotope shifts like those (ca. 0.1-0.2 ppm to lower field) found in the isoconjugate dimethylbenzyl cation.¹ (We didn't.) However, our initial attempts to condense acetone- d_6 with cyclopentadiene in methanol using pyrrolidine repeatedly led to loss of the deuterium labels. We later established that all ten of the protons of dimethylfulvene can be exchanged by a sequence involving (a) deprotonation of the 6-methyl group, (b) reprotonation of the vinylcyclopentadienide anion to give a mixture of (kinetically-preferred) ring-deuterated vinylcyclopentadienes and (methyl-deuterated) dimethylfulvenes,² and (c) subsequent equilibration to the thermodynamically-favored (randomly-labelled) dimethylfulvene. Deuterium NMR was invaluable, since it allowed us to (a) detect small amounts of deuterated product in the presence of large amount of undeuterated material,

and (b) assign structures to monodeuterated products by the multiplet structures in proton-coupled spectra.

Most of our preparations of 1 were also contaminated with several percent of mono and di ring-deuterated material, with the consequent two- and three-bond deuterium isotope shifts leading to the appearance of minor absorptions upfield of the fulvene ring carbon lines. We were able to extract the two-bond shifts by assuming additivity of isotope shifts, and to establish that the isotope shift across an $C(sp^2)-C(sp^2)$ single bond is smaller than across a double bond. We determined the two-bond isotope shifts in cyclohexene-1-d and 3 cyclohexa-1,3-diene-2-d, combined them with literature data³ for a number of other hydrocarbon π -systems, and found quite a respectable linear correlation between the isotope shifts (Δ) and carbon-carbon bond length:

$$R_{CC} = 1.847 \times 10^{-3} (\Delta)^2 + 1.594$$

(average deviation between calculated and observed bond lengths = 0.01 Å), suggesting that isotope shifts may be a fairly painless and reliable way to quantitatively assess carbon-carbon π -bond lengths in solution. We're now trying to see if this empirical relation is applicable to carbocations as well.

Best wishes,



Dennis J. Sardella
Professor of Chemistry



Alyaa Emad El-din

¹D.A. Forsyth, P. Lucas and R.M. Burk, J. Am. Chem. Soc. **104**, 240 (1982).

²J. Hine and D.B. Knight, J. Org. Chem., **35**, 3946 (1970).

³We chose only shifts which were measured relative to an internal undeuterated standard.



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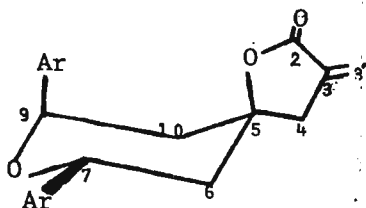
November 9, 1987
(received 11/12/87)

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

Title: Immobile 2,6-Diaryl-substituted Spiro Lactones

Dear Barry:

Your "ultimatum" letter arrived, although we had not received a reminder, and this is our contribution. We have been interested in substituted α -methylene- γ -butyrolactone systems for some time.¹ We have had occasion to prepare lactone I below and have noted a curious result. Intuitively, one would expect the two examples of I to be dynamic molecules undergoing rapid ring reversal as has been true for similar simple spiro lactones.¹ The ¹³C NMR analysis of both members



Ia: Ar = C₆H₅

Ib: Ar = 2-H₃C-O-C₆H₄

[Solvent: DCCl₃; chemical shifts in ppm from TMS]

has revealed individual signals for each carbon and thus we are forced to tentatively conclude that there exists a difference of significance in terms of the compounds having an axial C-O bond versus a C-CH₂ bond at C(5). Only in the formation of extremely biased systems (such as the cis-2,6-diaryl-substituted relatives of I) has this been observed in this family of spiro systems. If some one has an explanation, we would welcome hearing from them.

	C(2)	C(3)	C(4)	C(5)	C(6)	C(7)	C(9)	C(10)	C(3')	Ar-C	OCH ₃
Ia:	168.8	134.5	40.1	80.5	41.4	70.4	71.7	39.6	122.5	8 signals	
Ib:	169.5	135.2	40.6	81.6	41.4	67.1	68.3	39.9	122.2	12 signals	55.1, 55.2

¹O'Donnell, D. J.; et. al. *J. Org. Chem.* 1978, 43, 4259-4265.
Pantaleo, N. S.; et. al. *J. Org. Chem.* 1981, 46, 4284-4290.

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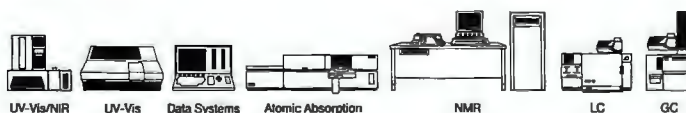
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Texas A&M University NMR Newsletter - Book Reviews

Book Review Editor:

William B. Smith, Texas Christian University, Fort Worth, Texas.**"Modern NMR Spectroscopy - A Guide for Chemists"**

by

Jeremy K. M. Sanders and Brian K. HunterOxford University Press, 200 Madison Avenue, New York, NY 10016;
1987; 308 pages; cloth \$59.95, paper \$35.00.

This book fulfills the promise of its title admirably, by providing practical information for chemists on choosing, performing, and interpreting modern NMR experiments. The major types of experiments are discussed with respect to the information they can provide, the ease with which they can be carried out, and their tolerance for instrument and operator errors. The approach is largely descriptive, with a liberal use of examples and diagrams to illustrate the non-mathematical theoretical presentation.

Many of the key chapters include summary sections covering the chemical consequences of the theories just discussed. The authors have recognized that NMR spectroscopy is not a linear subject, and come back in later chapters to topics covered earlier, for purposes of emphasis and comparison. The book does not cover all of the ever increasing types of NMR experiments, but concentrates on those which are likely to be the most reliable, available, and useful. At the same time, the basic principles of the various 1-D and 2-D NMR experiments are presented, so that as newer techniques are developed, the reader will be in a position to understand them.

The book is limited to FT NMR, with the first chapter covering the simple one-pulse experiment. Instrumental and theoretical limitations of the information available are emphasized. The next three chapters cover decoupling, multi-pulse sequences, and the basic two-dimensional techniques. Experimental considerations are covered in each case, and the analogy between the one- and two-dimensional approaches is stressed.

Chapters 5, 6, and 7 describe NMR experiments that can be used for structure and spectral assignments by determining the connectivity of nuclei through bonds, space, and chemical exchange, respectively. In addition to specific techniques, a general discussion of strategy and tactics at the beginning of Chapter 5 will prove especially helpful to the first-time user of modern NMR methods. Chapter 8 concerns editing, and includes decoupling, solvent and broad line suppression, and subspectral analysis.

After a chapter on the NMR of solids, the final chapter covers a case history of the complete NMR assignment for sucrose octaacetate. This provides an excellent illustration of choosing the most informative and efficient techniques to solve a problem. Especially helpful for the novice is the use of typical, rather than idealized, COSY and NOE-difference spectra, which manifest the ubiquitous artifacts that are seldom seen in the primary literature. This will give guidance and confidence to those who may be inclined to ignore correlations which look like fly-specks and worry about negative or multiphasic NOE-difference peaks.

In summary, this is an excellent book for the practising chemist who uses NMR. It is well produced, and appears to be error free, except for the obligatory typo on page 294, referring to H_2 , instead of H_3 . While there are some inevitable similarities to Derome's book, recently reviewed in these Newsletter pages, the two works complement each other sufficiently to warrant having access to both.

Manfred G. Reinecke
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V/Réf.

N/Réf.

Lausanne, le

29.10.1987

(received 11/9/87)

Separation of Overlapping Lines with Very Different Linewidths

Dear Prof. Shapiro,

to study the exchange of 2,6-dicarboxy-4-hydroxypyridine ($=H_3L$) on La^{3+} in aqueous solution we recorded the ^{139}La -NMR spectrum of a 0.18 m La^{3+} and 0.09 m H_3L solution in H_2O . Even using a high-field instrument (9.395 T) the resonance lines of free and bound metal overlap markedly. A deconvolution with a computer fails because of the large difference in linewidths (the relatively symmetric aquo-complex of La^{3+} gives a small line of about 300 Hz, whereas the linewidth of the strongly asymmetric $La(HL)^+$ is more than 14'000 Hz (Fig. 1). To be able to measure the linewidth of bound La -ions correctly we remembered a well known technique:

We set up an inversion-recovery sequence as used for T_1 -measurement. If one sets the delay time between 180 and 90 degree pulses equal to $\ln(2) \cdot T_1$ no signal will be detected. Because the T_1 values of our two lines are very different, the spin of the bound ^{139}La has already recovered when the z-magnetization of the free one is just zero. The resulting spectrum shows only one line corresponding to the bound ^{139}La (Fig. 2). The small negative residual signal indicates that the delay time chosen was slightly to short.

The spectra shown were recorded at 1.5° C. The exchange is blocked at this temperature and the linewidths give a direct measurement of the quadrupolar relaxation.



Dr. Gabor Laurenczy

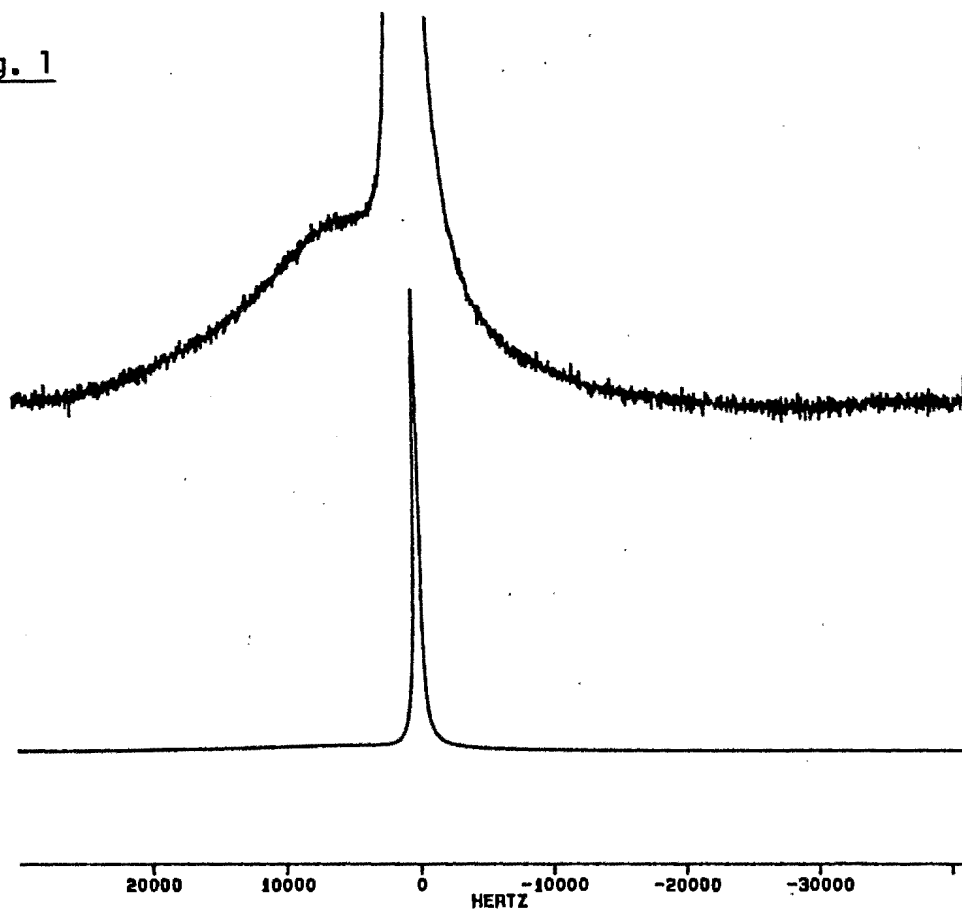


Dr. Lothar Helm

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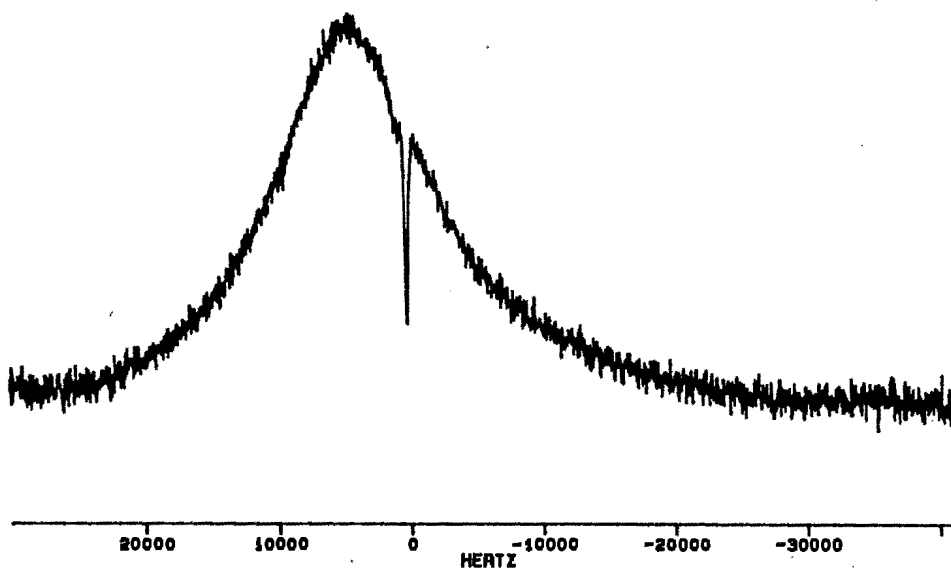
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Fig. 1



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Fig. 2





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Professor B. L. Shapiro,
 966 Elsinore Court,
 Palo Alto, California, 94303,
 USA

23 October, 1987
 (received 10/29/87)

TITLE : NMR and X-ray Structure Correlations of Bismuth Xanthates

Dear Prof. Shapiro,

I think we missed your pink "ultimatum" notice! I hope this contribution will reinstate us on your records.

In our last letter we were able to relate a correlation between the solid state NMR spectrum and the X-ray crystal structure of the methyl xanthate of bismuth. We have extended this study to the ethyl and isopropyl xanthates, one of which, $\text{Bi}(\text{S}_2\text{COEt})_3$, is crystallographically intractable.

The $\text{Bi}(\text{S}_2\text{COMe})_3$ structure contains a *pseudo* mirror plane (not crystallographically imposed) which gives rise to 3 methyl signals and 2 thiocarbonyl signals in the NMR spectrum (Structure I). The $\text{Bi}(\text{S}_2\text{COiPr})_3$ structure however is polymeric, due to the bonding of one S to a neighbouring Bi atom. The Bi and the ligand containing the sulphur forming the polymeric link, define a mirror plane (Structure II) which creates 3 pairs of methyl carbons giving rise to 3 signals in the spectrum. Two resonances in the ratio 2:1 are observed for the methine carbons consistent with the structure, and the S_2CO resonance consists of a sharp and a broad resonance possibly from 2 different environments.

The NMR spectrum of $\text{Bi}(\text{S}_2\text{COEt})_3$ shows 5 sharp resonances for the methyls in the approximate ratio 1:1:2:1:1 and broad resonances for the methylene and S_2CO carbons. We deduce that $\text{Bi}(\text{S}_2\text{COEt})_3$ exists as 2 polymorphs with each of the structures described above. Structure I would be expected to give rise to 3 methyl signals, 2 or 3 methylene signals and 2 S_2CO signals, while Structure II (with its mirror plane) would give rise to 2 methyl peaks (in ratio 2:1) and 2 methylene peaks (also in ratio 2:1).

The observed spectrum is consistent with this, particularly in the methyl region of the spectrum ; the other areas show some overlapping of signals.

All spectra were run on a Bruker CXP300 in standard CP/MAS experiments. This example illustrates the usefulness of a straightforward solid state NMR experiment in solving an otherwise difficult problem.

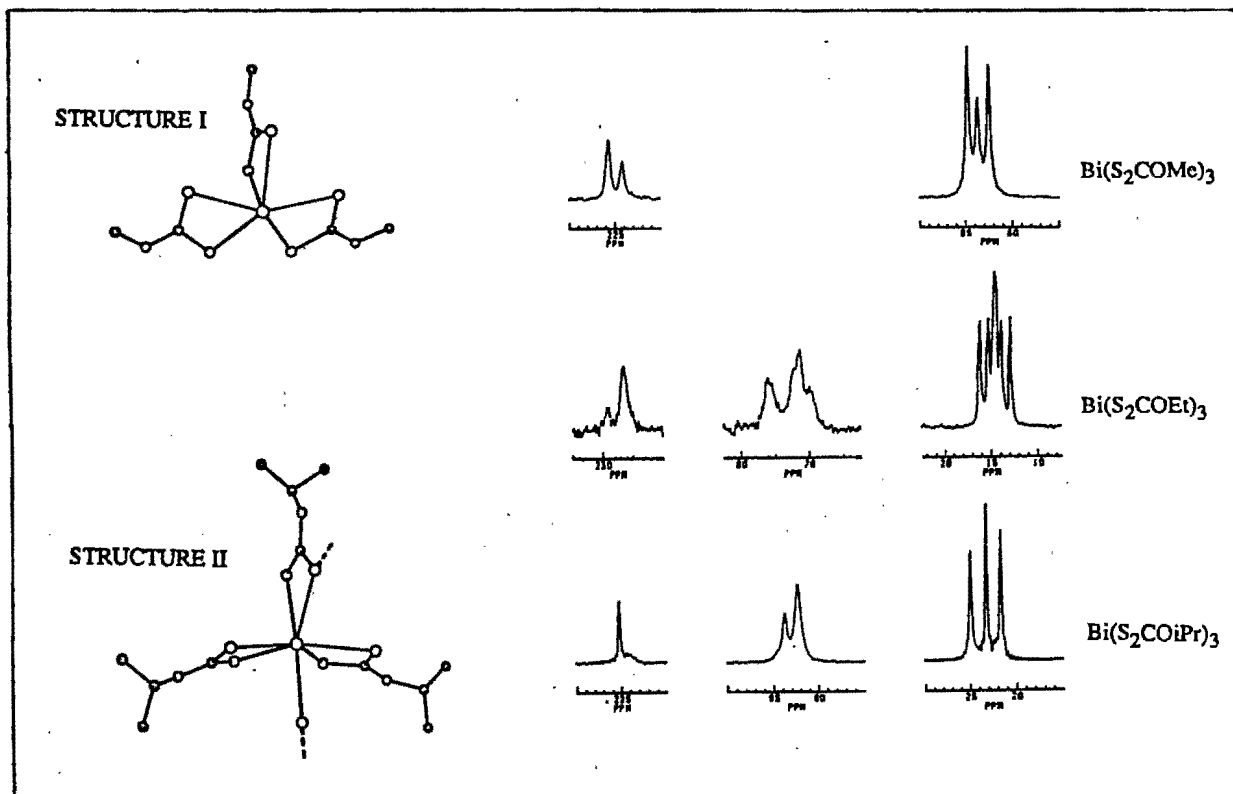
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Phone 614-292-3446

17 November, 1987

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

MRI Postdoctoral Position

Dear Barry,

A postdoctoral position at Ohio State University's Magnetic Resonance Imaging Center is available immediately. Salary will be competitive and determined by the candidate's qualifications. The Ohio State University is an affirmative action, equal opportunity employer.

The successful candidate will have regular access to OSU's General Electric Signa 1.5 tesla whole-body imager in our Radiology Department, and to Bruker AM-500 and MSL-300 instruments at the Campus Chemical Instrument Center. We are particularly interested in the development and applications of proton chemical shift imaging, and seek a candidate with prior experience in one or more of the following areas: protein NMR, in vivo NMR, and two-dimensional techniques.

Interested candidates should submit a vita and graduate transcript, and have three letters of recommendation sent to:

Professor Alan G. Marshall
Department of Chemistry
The Ohio State University
120 W. 18th Avenue
Columbus, OH 43210

Alan G. Marshall

Alan G. Marshall
Professor, Chemistry and Biochemistry



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DEPARTMENT OF CHEMISTRY
TAMPA, FLORIDA 33620813:974-2144
SUNCOM: 574-2144October 26, 1987
(received 10/30/87)Dr. B. L. Shapiro
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto, CA 943038087th Second Thoughts.

Dear Barry:

I was semi-astounded and fully delighted to learn of your move from the Gulf Coast to the Golf Coast. I hope you are happy settling in to your new location.

During the past few years, I have been writing computer programs of some use in NMR and have reported these from time to time in this newsletter. During the past couple of years, there have been some quite significant advances in the compilers available for use with the BASIC language and this language has undergone a complete metamorphosis into a very powerful, highly structured, programming tool. Of particular import are the development of the Quick BASIC (Microsoft) and Turbo Basic (Borland) compilers. Both of these allow the use of IEEE numbers and, more significantly, the incorporation of the power available with the 8087 (Intel) family of math co-processors.

Recently, I have been revising some of my old programs to use the Quick BASIC compiler and to incorporate the power of the 8087 math co-processor. The programs which are being revised at the moment are:

- 1) LISA--a program for handling multiple fast-exchange equilibria in NMR.
- 2) FFTUTOR--a program for generating mathematical functions and then generating and displaying their Fourier transforms.
- 3) NMR--a spectrum simulation for from 1 to 7 spins which calculates all frequencies and line intensities (up to 3003 lines) and plots the spectra if desired.
- 4) MOLPIX--a program for generating and displaying molecular structures of up to 500 atoms (and easily expandable for newer computers using an

EGA instead of a CGA).

These four programs profit greatly from the capability of complete integration of a math coprocessor into the calculations.

Final revisions of the above programs will be finished shortly. Anyone wishing to have copies of these is welcome to have them provided I am sent a DS-DD density disk for each program desired (except that two are needed for MOLPIX) and a self-address disk mailer is included.

Sincerely yours,

Milton D. Johnston, Jr.

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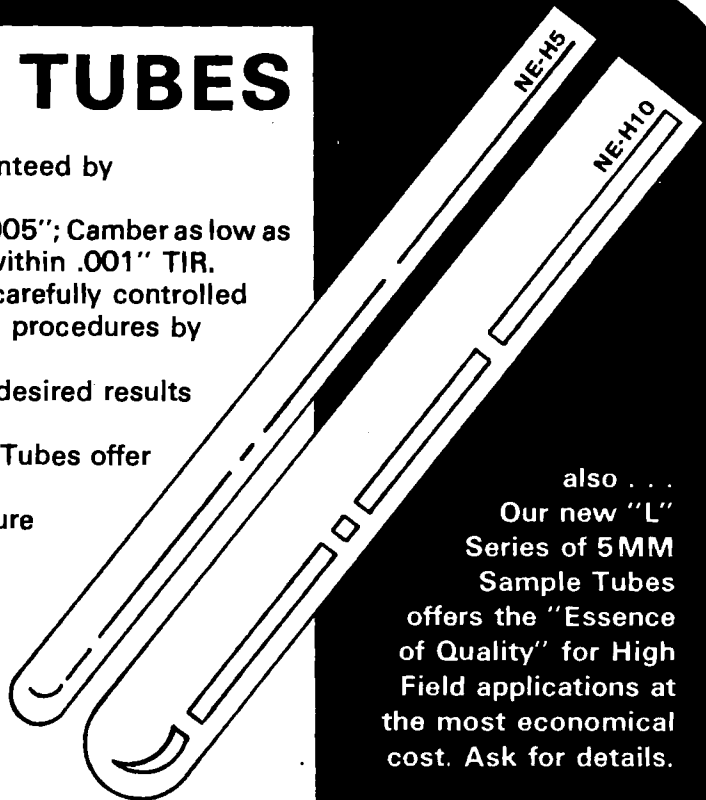
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October 4, 1987
(received 10/30/87)

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

HOHAHA and ROESY Experiments on a General Electric GN-500 Spectrometer

Dear Dr. Shapiro:

We have recently employed 2D homonuclear Hartmann-Hahn (HOHAHA) and rotating frame NOE (ROESY) experiments to aid in the resonance assignments and structural determinations of small peptides. These experiments were performed on our General Electric GN-500 spectrometer, with the pulse sequences supplied by the GE NMR Applications Department. To perform these experiments, a strong spin-lock pulse that produces a homogeneous effective rf field is required. This high-power output can be generated by a high-resolution (0.1 Hz) decoupler, an optional accessory on the GN-500 system. While we were awaiting the delivery of this decoupler, we used the broadband decoupler amplifier as the observe transmitter¹ to obtain HOHAHA data for angiotensin II, an octapeptide, in D₂O and in H₂O.

Figure 1 shows an unsymmetrized, phase-sensitive HOHAHA spectrum of 25 mM angiotensin II in 90% H₂O solution. A 23.5 μ sec 90° pulse was generated, with 3 watts of rf power, by the decoupler amplifier. A MLEV-17 mixing sequence, with 5 msec trim pulses, was employed². A 1-1 pulse was applied to suppress the water signal. Connectivities from the amide protons of val-3, tyr-4 and phe-8 to their respective side chain protons are shown as solid lines in Figure 1.

We tried but were not able to obtain satisfactory ROESY³ data using the decoupler amplifier as the observe transmitter, mainly because we were limited to a single power level for both the 90° and spin lock pulses. Therefore, the high-resolution decoupler is necessary for performing the ROESY experiment on the GN-500 system, especially in H₂O solution.

Figure 2 shows a phase-sensitive ROESY spectrum of angiotensin II in D₂O, acquired with a high-resolution decoupler. Full power from the decoupler was used to generate a 90° pulse of 15.5 μ sec, and an intermediate power level was used to generate a 2.5 KHz rf field spin-lock pulse. In the ROESY contour plot, the intense diagonal signals do not appear since they are in opposite phase with the NOE cross peaks. This allows the observation of cross peaks near the diagonal. Analysis of the inter- and intra-residue NOEs in the ROESY spectrum has allowed us to obtain some structural information on this octapeptide. For illustration, several of the observed NOE cross peaks are labeled in Figure 2.

Please credit this contribution to Fouad Ezra's account.

Sincerely,

THE PROCTER & GAMBLE COMPANY
Research and Development Department

Susannie C. Lee *Anne F. Russell*
Susannie C. Lee Anne F. Russell

¹ suggested and demonstrated by J. Dallas, Applications, General Electric NMR.

² Bax, A. and Davis, D. G., J. Magn. Reson. **65**, 355-360 (1985).

³ Bax, A. and Davis, D. G., J. Magn. Reson. **63**, 207-213 (1985).

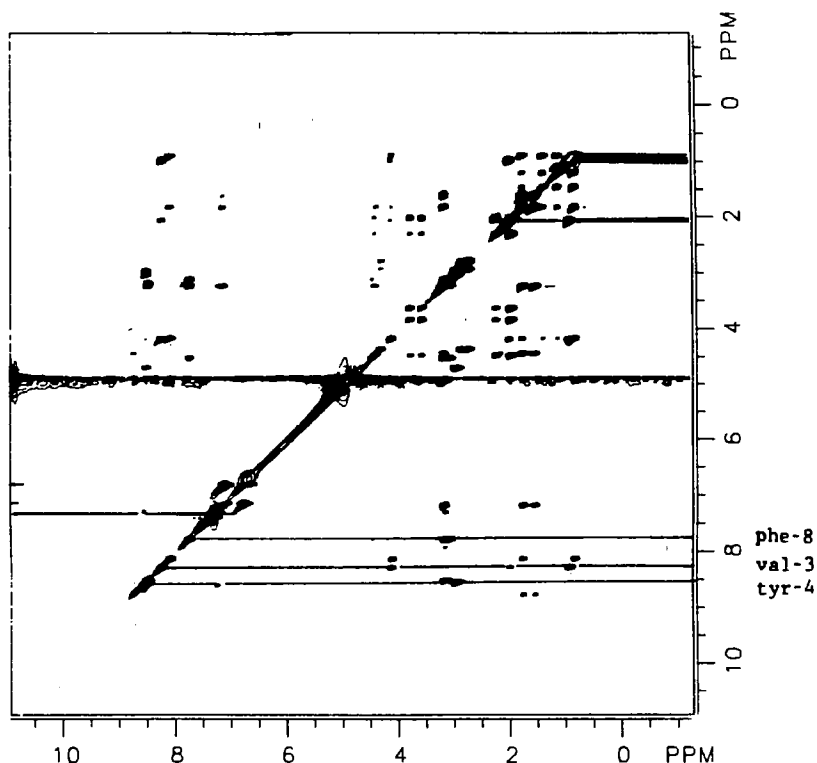


Figure 1. Contour plot of the HOHAHA spectrum of 25 mM of angiotensin II (asp-arg-val-tyr-ile-his-pro-phe) in 90% $H_2O/10\% D_2O$, recorded at 500.12 MHz and at room temperature. Phase sensitive acquisition was obtained by using the TPPI method. A mixing time of 55 msec, consisting of 29 MLEV-17 repetitions (45 msec) and two trim pulses of 5 msec (10 msec), were used. Connectivities from the amide protons of val-3, tyr-4 and phe-8 to the corresponding side chain protons are illustrated as solid lines. The cross peaks from the amide to the α proton of tyr-4 and of phe-8 are not visible in this contour map but can be easily identified in the cross sections.

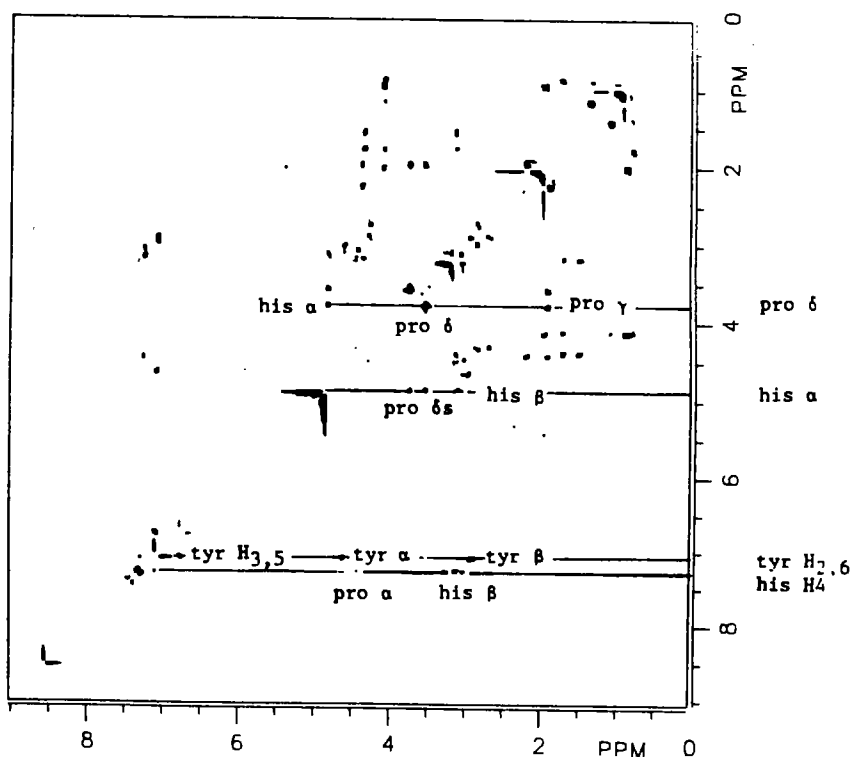


Figure 2. Contour plot of the ROESY spectrum of 25 mM of angiotensin II in D_2O at room temperature. Phase sensitive acquisition was obtained by the States, Haberkorn and Ruben method. A spin-lock pulse of 200 msec was used. Examples of some of the observed NOE cross peaks are labeled and shown as solid lines.



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Ottawa, Canada
K1A 0R6

1987-11-13
(received 11/23/87)

Dr. B.L. Shapiro
966 Elsinor Court
Palo Alto, California
U.S.A. 94303

Dear Barry:

¹²⁹Xe resonances in Block Copolymers

This is a follow-up on the pioneering work of Stengle and Williamson on Xenon dissolved in polymers.¹ They found different chemical shifts for xenon dissolved in different regions of amorphous linear low density polyethylene. We have studied polystyrene, polyisoprene and block copolymer polystyrene-polyisoprene systems. The line width variation, shown in the accompanying table, may be explained by diffusion between the two different phases in those block copolymers where phase separation occurs. Assuming that the excess line width is caused by lifetime broadening, one can calculate a diffusion co-efficient of $3 \times 10^{-7} \text{ cm}^2 \text{ s}^{-1}$ in the polyisoprene phase, compared with $5 \times 10^{-7} \text{ cm}^2 \text{ s}^{-1}$ obtained by a completely different method, for natural rubber.

Sample	Molecular Weight	Width Hz
CDCl ₃		3
Polystyrene	10,000	3,300
PS	280,000	2,300
Polyisoprene	2,000	26
PI	251,000	150
PS-PI	4,800 - 5,000	360
PS-PI	21,000 - 11,000	6,700 - 1,100
PS-PI	50,000 - 50,000	4,300 - 740
PS-PI	750,000 - 750,000	3,000 - 620

1. Macromolecules 20, 1428 (1987).

Yours truly,

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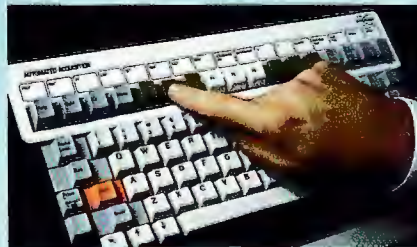
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18 November, 1987
(received 11/23/87)

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

**Beating the Nyquist Limit by Means of
Interleaved Alternated Delay Sampling**

Dear Barry,

According to the Nyquist theorem, the highest signal frequency which can be represented without foldover (aliasing) in a Fourier transform frequency-domain discrete spectrum is one-half of the time-domain sampling frequency.

However, we can "beat" the Nyquist limit by interleaved addition (Figure 1) of two digitized time-domain transient signals, one of which is delayed by one-half of one sampling period (i.e., half of one cycle of the time-domain sampling frequency) with respect to the other, to yield a time-domain discrete waveform which is indistinguishable from a single waveform produced by sampling at twice the original sampling rate. If the sampling period is divided into three or more equal parts, with interleaved addition of three or more correspondingly delayed transients, the same method can further increase the upper frequency limit. We have experimentally demonstrated up to a factor of 4 increase in sampling rate by addition of four successively delayed transients, and applied the method to extension of the bandwidth of Fourier transform ion cyclotron resonance mass spectra.¹

The method depends upon: (a) identical magnitudes of the two interleaved transients, and (b) delay of precisely one-half of the ADC dwell time between the two transients. Violation of either constraint will produce unwanted "image" peaks, located at frequencies obtained by reflecting the true spectrum about its bandwidth midpoint. However, in practice we have found that the necessary timing accuracy is not difficult, and any difference in magnitude between the two transients can be avoided by normalizing them to a common magnitude before interleaving. Image peak magnitude can thus be reduced to well below 1% that of the true peak.

It may be worth noting that this experiment differs from the "Alternating Delay Acquisition" experiment² in which two transients differing by half a cycle of the solvent frequency are added without interleaving. The authors thank Richard N. Moore for introducing us to the interleaving idea.

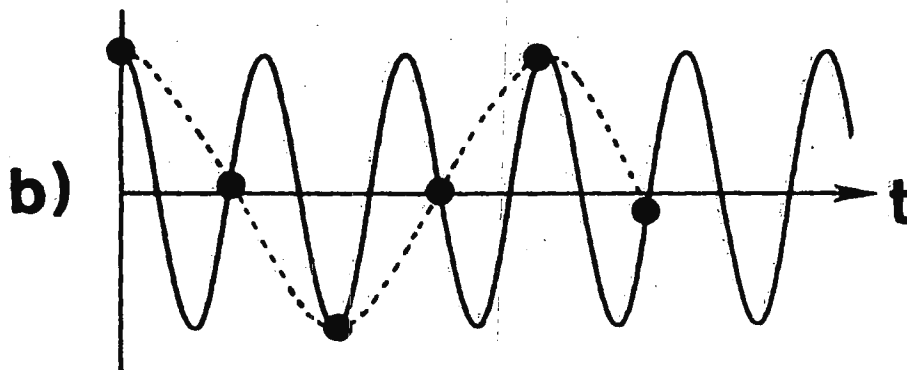
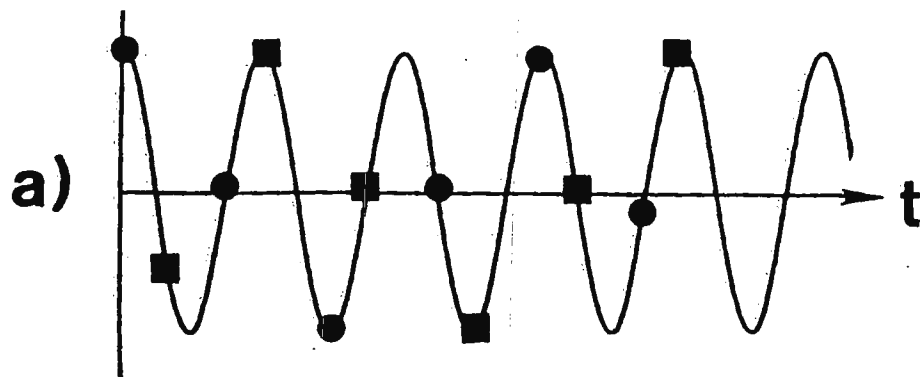
1. Verdun, F. R.; Ricca, T. L.; Marshall, A. G.; Appl. Spectrosc. (Feb 1988 issue)
2. Roth, K.; Kimber, B.G.; Feeney, J. 1980, J. Magn. Reson. 41, 302-309.

Best regards,

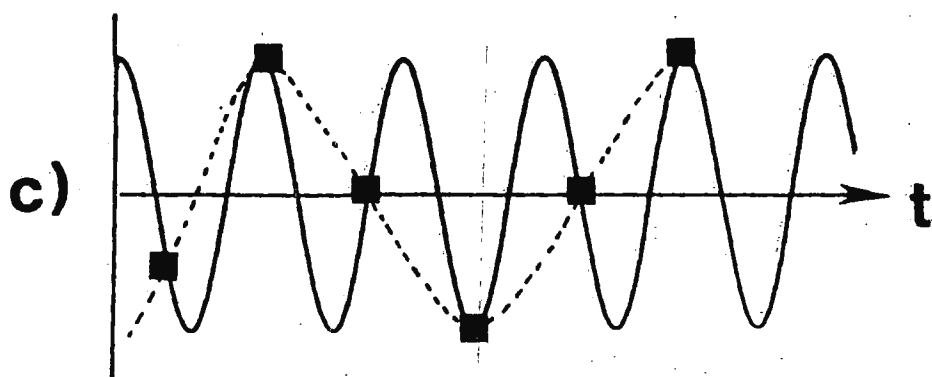
Francis R. Verdun

Tom L. Ricca

Alan G. Marshall



+



●■: sampled data



Dalhousie University

Department of Chemistry
Halifax, Nova Scotia
Canada B3H 4J3
(902) 424-3305

November 3, 1987
(received 11/12/87)

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

Dear Barry,

ISOTOPE EFFECTS ON NUCLEAR SHIELDING AND SPIN-SPIN COUPLING CONSTANTS

In order to obtain a better understanding of isotope effects on nuclear shielding constants and spin-spin coupling constants we have measured $2/1\text{H}$ isotope effects in several simple hydrides (1-3). Results for the phosphorous and tin hydrides are summarized below ($A = \text{P-31}$ and Sn-119 respectively).

Molecule	$\Delta A(^{2/1}\text{H})$	$\Delta_p ^1J(A, ^{2/1}\text{H})$
PH_2^-	-2.76 ppm/D	$2.7 \pm 0.1 \text{ Hz}$
PH_3^+	-0.80 (ref.4)	13.4 (ref. 4)
PH_4^+	0.00	-1.7 ± 0.4
SnH_3^-	-3.09	10.5 ± 1.0
SnH_4^+	-0.43	-2.7 ± 0.6
SnH_3^+	-0.05	-11.6 ± 7

The P-31 nmr spectrum of $\text{PH}_{2-n}\text{D}_n^-$ ($n \leq 2$) at 20°C in liquid ammonia is shown in Figure 1.

There are four points that we would like to make:

1. In each series, isotope effects on the chemical shifts appear to be a function of nuclear charge. This is as one would predict on the basis of recent theoretical calculations by Chesnut (5).
2. To a good approximation the isotope shifts were additive. Deviations were in agreement with predictions of Jameson and Osten (6).
3. Primary isotope effects on J are generally small however in the case of the stannyl ion, $(\gamma_H/\gamma_D)|J(\text{Sn},\text{D})| - |J(\text{Sn},\text{H})|$ is 10.5 Hz or about 9.4% of the value of $J(\text{Sn},\text{H})$.

Dr. Bernard L. Shapiro
November 3, 1987
Page 2

4. Primary isotope effects on spin-spin coupling constants are positive in hydrides where the heavy atom has one or two electron lone pairs; otherwise they are negative. These observations may be explained using a model proposed by Jameson and Osten (7).

I am hoping that you have recovered from your recent move and that you are beginning to enjoy your semi-retirement. Best wishes,

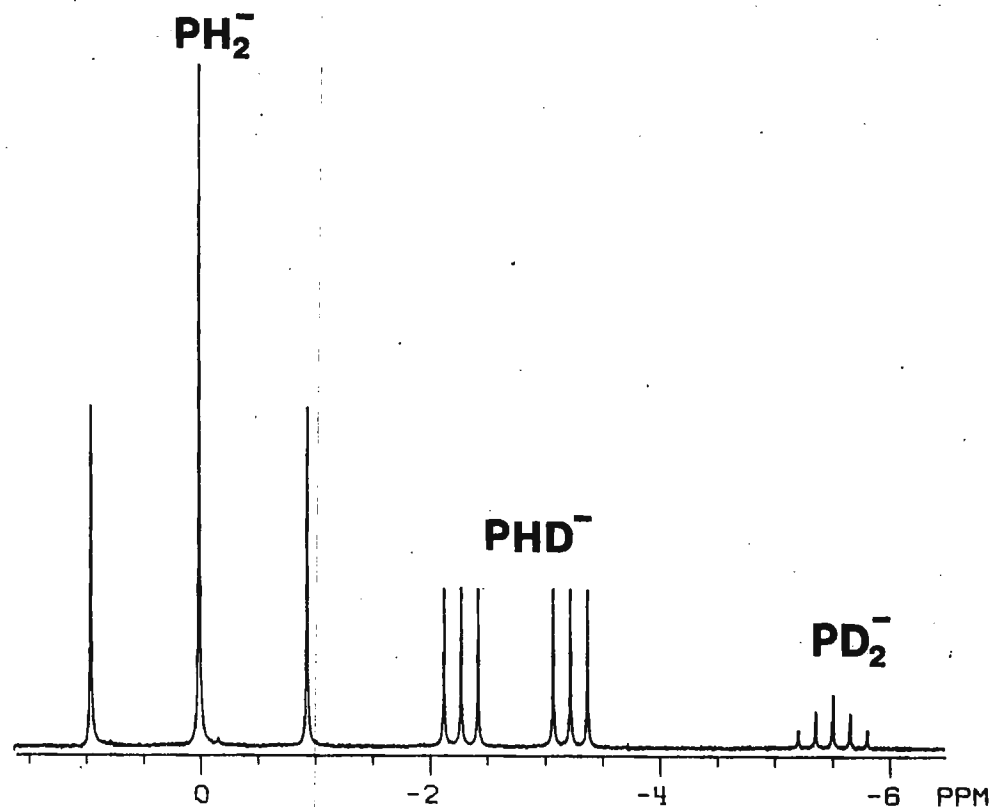
Yours sincerely,

Rod

Rod Wasylishen
Professor of Chemistry

REW/cas

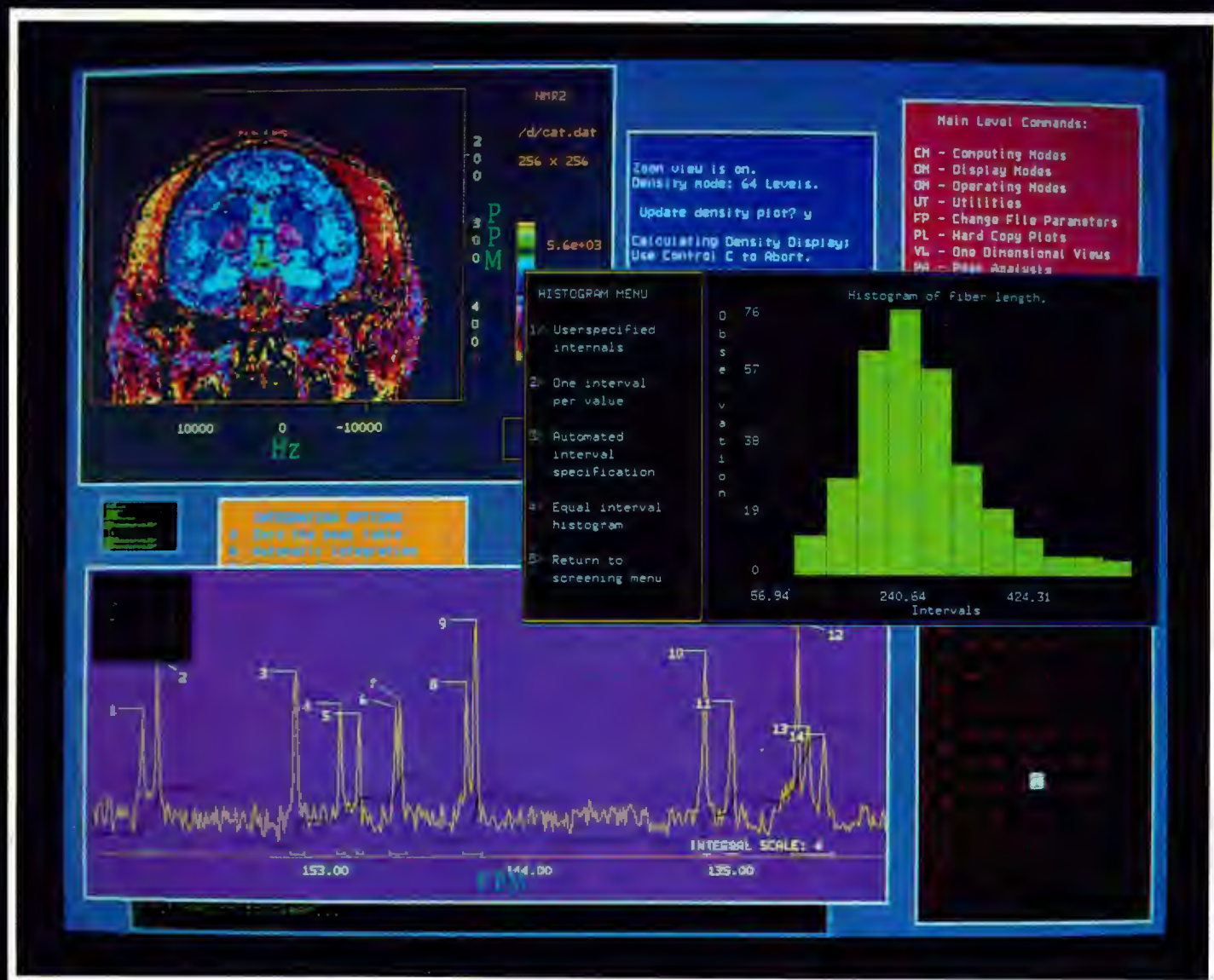
1. K.L. Leighton and R.E. Wasylishen, Can.J.Chem. 65, 1469 (1987).
2. R.E. Wasylishen and J.O. Friedrich, Can.J.Chem. 64, 2238 (1987).
3. R.E. Wasylishen and N. Burford, Can.J.Chem., to be published and J.Chem.Soc., Chem. Commun., to be published.
4. A.K. Jameson and C.J. Jameson, J.Magn.Reson. 32, 455 (1978).
5. D.B. Chestnut, Chem.Phys. 110, 415 (1986).
6. C.J. Jameson and H.J. Osten, J.Chem.Phys. 81, 4293 (1984).
7. C.J. Jameson and H.J. Osten, J.Amer.Chem. Soc. 108, 2497 (1986).



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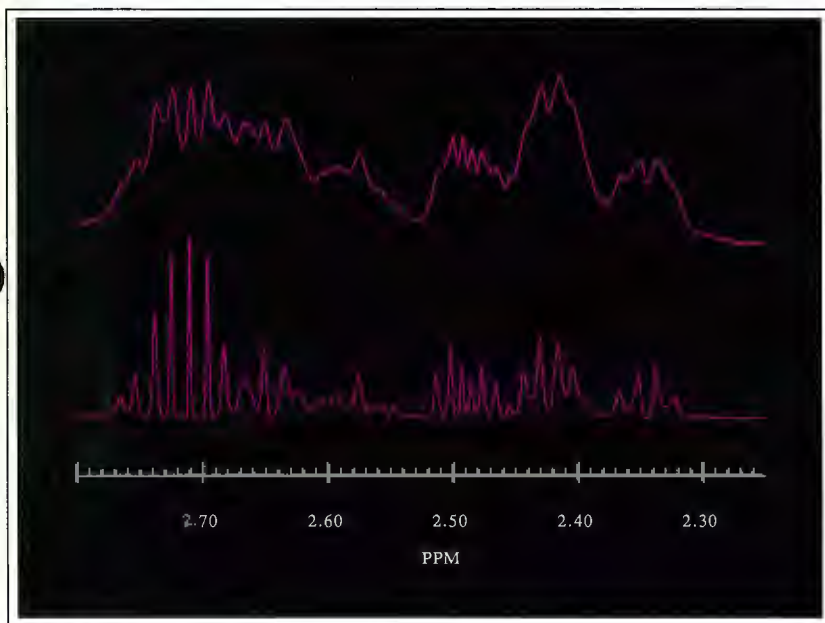


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Image processing methodology for 2-dimensional NMR Spectroscopy: top — Noisy 2D data set; middle — conventional symmetrization; bottom — use of a morphological filter. All contour plots made with levels equally spaced starting at 10% of data maximum.



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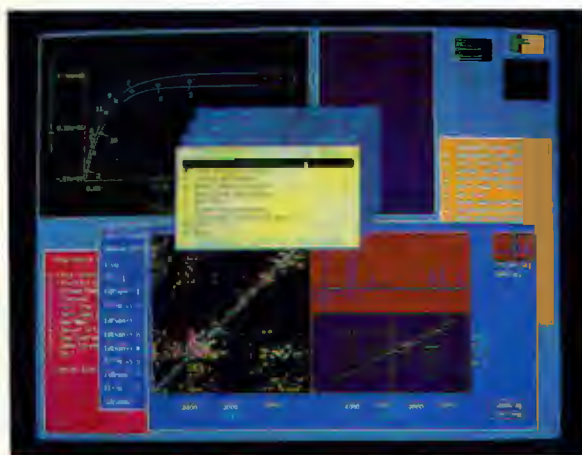
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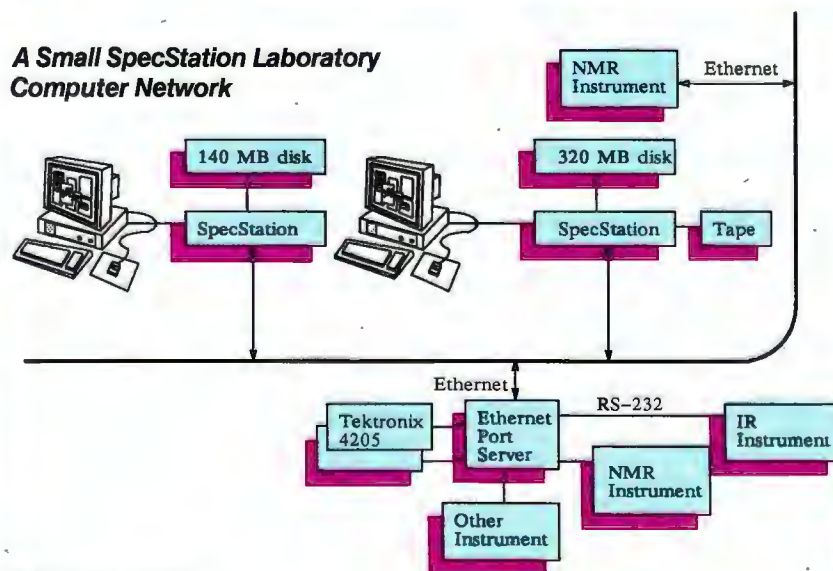
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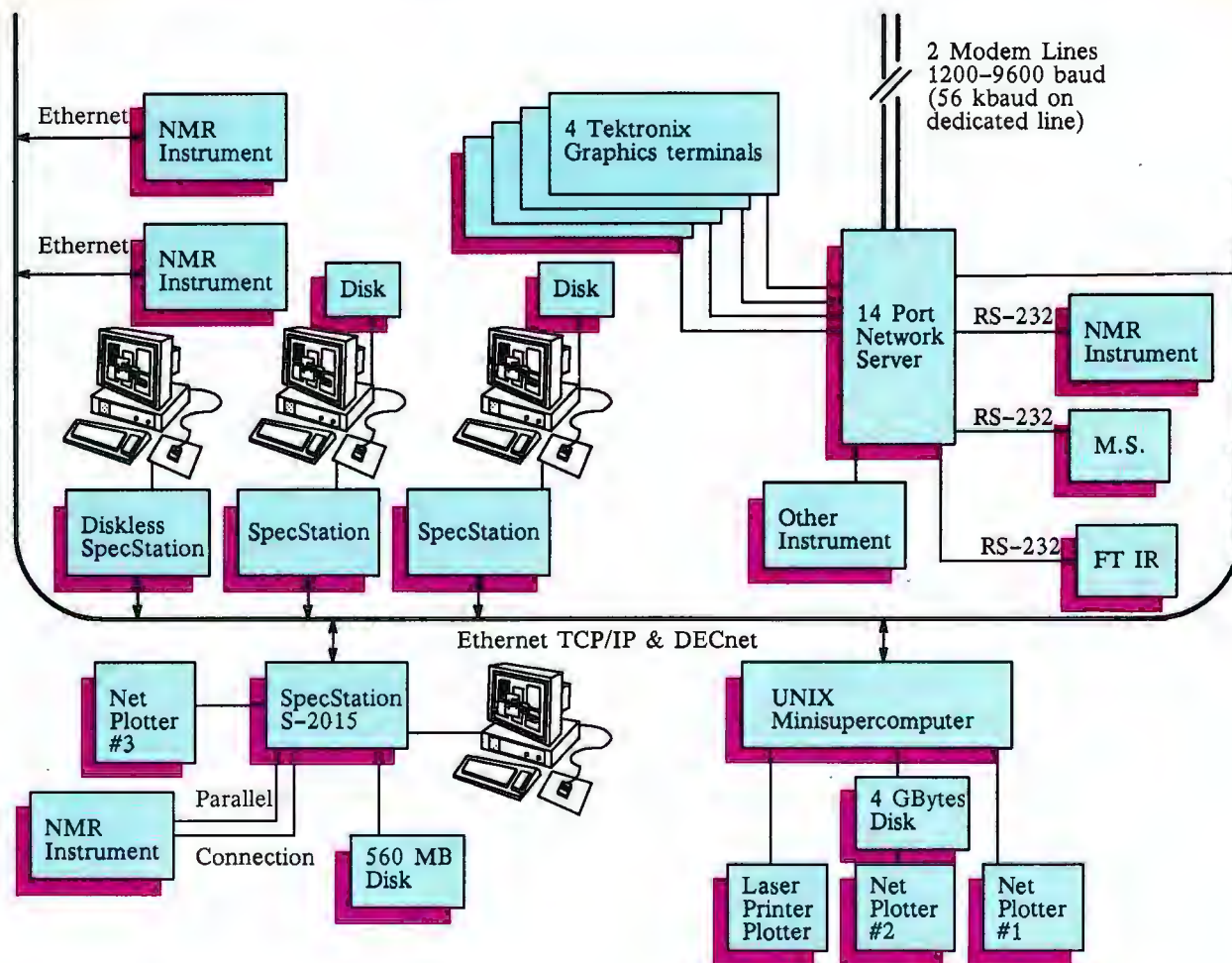


SpecStation screen showing processing of 2D NMR data and, in another window, an automated regression analysis. The 2D NMR graphics is configured by the user into four software windows giving full and zoomed 2D views along with full and expanded views of a single slice from the data set. A pop-up user option menu is shown in the 1D program window.

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NMR Data Reduction— The NMR1/NMR2 System

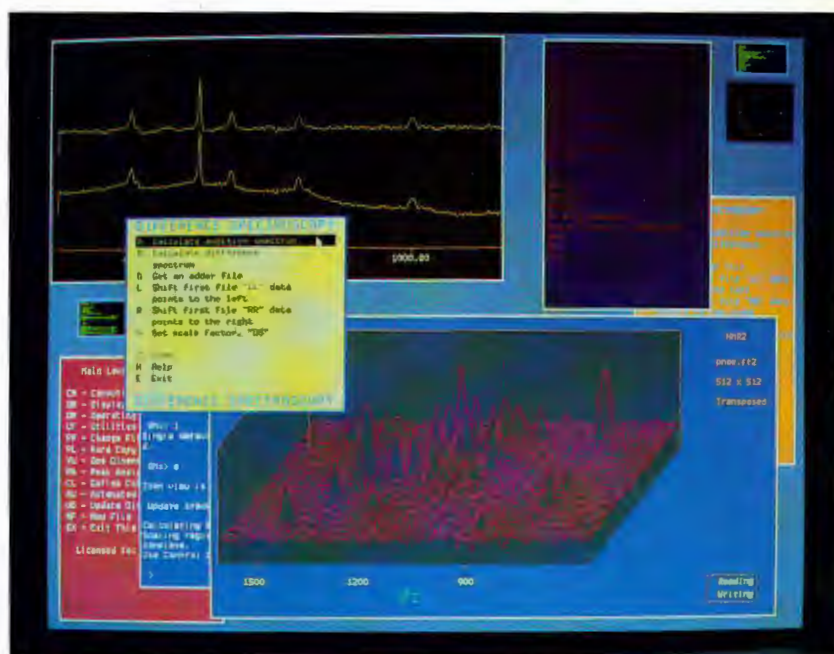
The **NMR1/NMR2** system is the most comprehensive data reduction system available for NMR spectroscopy and imaging. **NMR1** provides basic and advanced processing of 1-dimensional NMR data; **NMR2** uses powerful and efficient algorithms for analysis of 2-dimensional FT NMR spectra and images. *Reliable* network-style data transfers are provided from NMR instrumentation using Ethernet, parallel, or economical RS-232 serial connections. Data from *all* brands of NMR spectrometers are combined into the standard LAB ONE file format to optimize data archiving and laboratory information management. The **NMR1/NMR2** system is available on μ VAX and VAX computers, several UNIX minisupercomputers and on NMRi's SpecStation systems, based on Sun workstations or VAXstations.

NMR1

NMR1 provides automated and intelligent algorithms for processing and quantifying spectral data, incorporating unique features that optimize results from non-ideal data, while minimizing introduction of operator bias. A variety of advanced algorithms are available, including: automatic baseline evaluation and correction, rapid peak analysis, curve fitting of 10 or more overlapping peaks with complex lineshapes and non-linear optimization methods. Complex user-defined protocols with intelligent branching can be created. As with all LAB ONE software, all printouts and plots including laser plots are automatically spooled and proceed concurrently with other processing. Also, several mechanisms are provided to support inter-laboratory processing via standard phone connections.

NMR2 for 2-Dimensional FT NMR Spectroscopy and Imaging.

NMR2 is the most powerful software system in existence for processing 2-dimensional NMR spectra and images. The program includes a broad range of capabilities for processing phase-sensitive 2D spectra and MR images. On SpecStation systems, complete processing of a $1K \times 1K \times 2$ data array (including all I/O) can be performed in under 2 minutes [SpecStation S-2015 with array processor]. Available routines in **NMR2** include: automated peak analysis with 2D peak fitting, MEM, phasing of hypercomplex 2D data in both dimensions after FFT, versatile noise and ridge suppression, automatic local baseline compensation and integration over one or several data sets, and subsequent distance calculation and connectivity analysis (the latter features are incorporated in Release 4.0). **NMR2** provides the user



NMR1 and **NMR2** processing. Powerful baseline conditioning flattens poor baselines from *in vivo* and other spectra. Also shown is a stacked view in one of five **NMR2** window modes.



In the top window, a small region of a 2D dataset is shown with projections of the zoomed spectrum in both dimensions. This region has undergone automatic peak analysis and integration (blue rectangles). The two vertical white lines define a region which is summed along the f_2 dimension. The **NMR1** window shows automatic quantitation by curve fitting of the nine peaks in this thick slice.

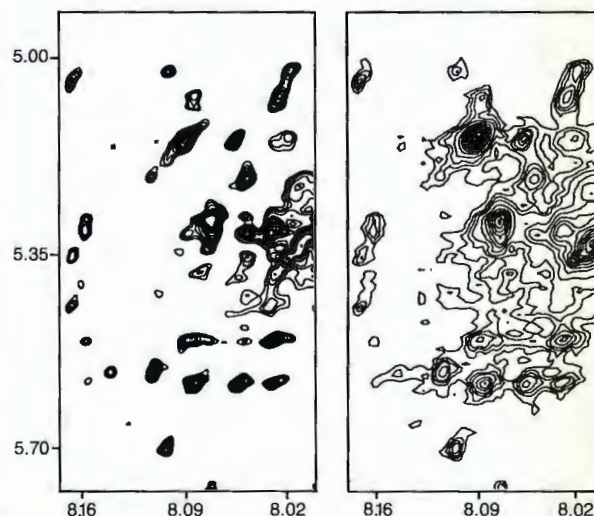
with a rich set of data manipulation tools and the **NMR2** user interface allows users to choose graphics modes for all circumstances. The **HELP** facility in **NMR2** can always provide information specific to the moment, including knowledge of all user- and system-set parameters. The optional **NMR2** image enhancement (**IMAGE**) program provides powerful procedures to improve contrast, structure definition and overall signal-to-noise in images. Algorithms include: histogram modification, contrast stretching, edge and region detection, morphological filtering and maximum entropy reconstruction. Several of the image enhancement procedures are also applicable to, and optimized for, complicated 2D NMR spectra.

Non-Linear Methods; MEM/LPM Spectral Optimization

The **MEM/LPM** spectral optimization program adds efficient maximum entropy and linear prediction non-linear processing methods for signal-to-noise and resolution manipulation of 1-dimensional data; 2-dimensional maximum entropy methods for spectra and images are provided in **NMR2**. Users can obtain results from raw data or perform maximum entropy Fourier spectral deconvolution (MEFSD) on spectra or spectral segments. The latter technique can be used to optimize low signal-to-noise features in spectra having very large spectral lines (e.g., solvent resonances). Unlike conventional deconvolution methods, MEFSD and LP techniques can work at very low spectral signal-to-noise ratios.

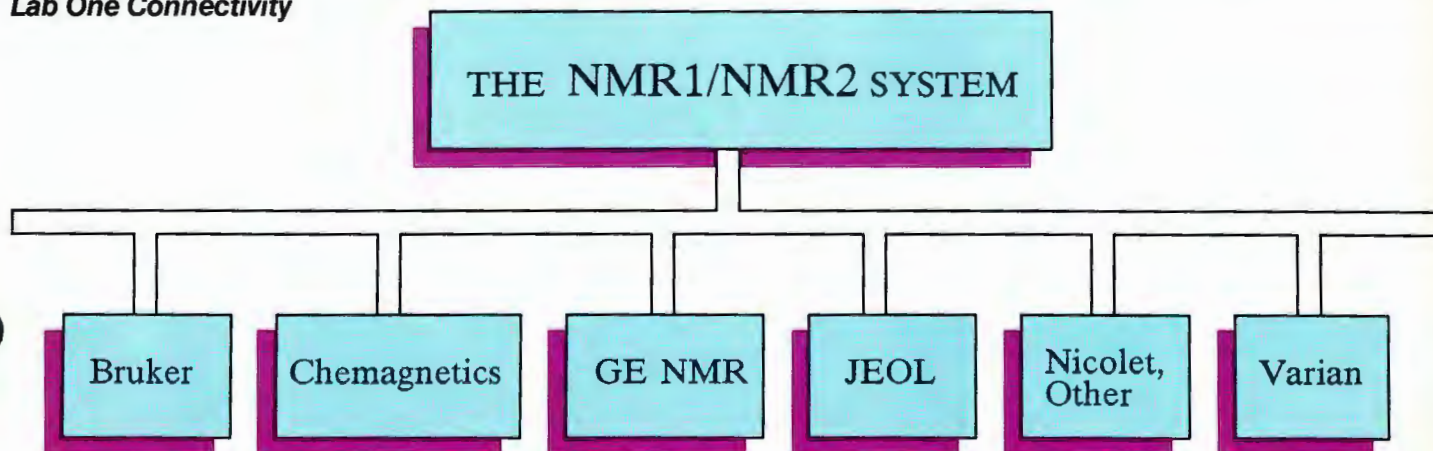


Maximum entropy processing can be used effectively to resolve overlapping spectral features, even when experimental signal-to-noise is low.



Two-dimensional Maximum Entropy Fourier Spectral Deconvolution (MEFSD) of a 25 nucleotide RNA molecule containing a hairpin loop. These expansions show part of the H1'-base region processed (left) with MEFSD and (right) using conventional FFT techniques.

Lab One Connectivity



Infrared and other Optical Spectroscopy

The SpectIR™ System

SpectIR, intelligent IR data processing, is a joint product of NMRI and Bio-Rad, Digilab Division. Thus, Digilab's expertise in FT-IR is combined with NMRI's experience in advanced algorithms, graphics and networking. With the introduction of **SpectIR**, the spectroscopist now has tools to process volumes of data from several instruments on a system with the power to perform intelligent, complex processing.

NMRI provides software to effect transfer of IR data from FT-IR instruments; the *open-standard* LAB ONE header format with JCAMP conversion allows users to interface laboratory instruments not directly supported by NMRI.

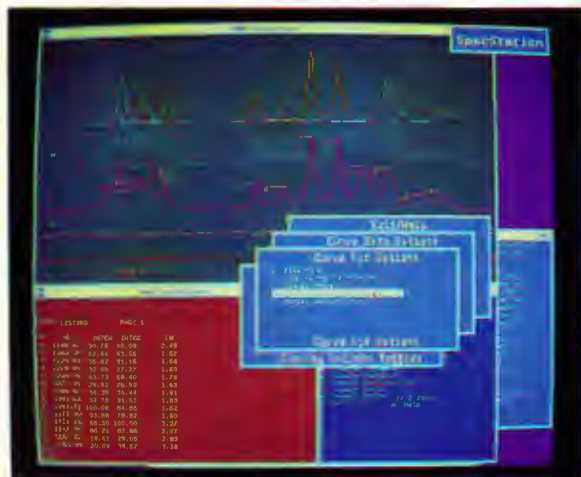
SpectIR is available on VAX computers as well as on NMRI's SpecStation workstation computers and networks. Several users may simultaneously execute **SpectIR** and other LAB ONE software on a single computer. Spectral data may be accessed and processed across SpecStation or other laboratory computer networks. **SpectIR** provides automatic peak location and integral determination. The user can also decompose complex overlapping sets of peaks into their components by means of rapid, *automatic* curve fitting. The components may be approximated by a number of lineshapes, including Gaussians, Lorentzians, and Voigt functions and users may mix lineshapes for different peaks in a region.

SpectIR math routines allow addition and subtraction of spectra, and include several algorithms to allow automatic subtraction of solvents, water, or other interfering species. Operations in the Fourier domain are implemented, allowing advanced apodizations and signal processing.

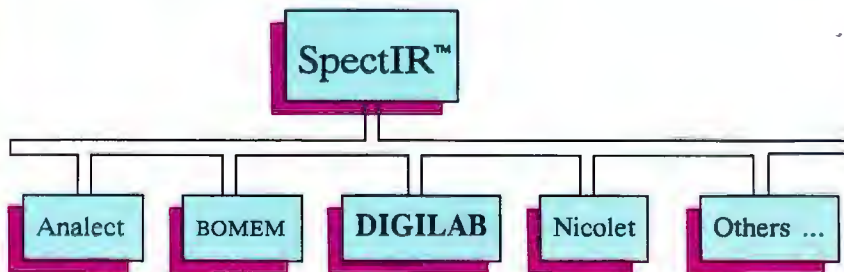
Optional **SpectIR** software includes unique Maximum Entropy processing (MEM-IR), spectral library search and Principal Components Analysis (PCA).



SpectIR displays FT-IR data in transmittance and absorption modes, interchangeably. Here a small region is quantified using rapid and automatic curve-fitting.



SpectIR curve-fitting uses an efficient and powerful Levenberg-Marquardt algorithm. Users may select from a number of lineshape functions, and mix lineshapes in a single region.



Intelligent Analysis of Laboratory Data— ESS™ The Expert Statistical System

A new concept

ESS is the first of a new generation of intelligent statistical systems especially designed for scientists and engineers. ESS allows non-statisticians to carry out advanced statistical analyses—simply, rapidly, reliably and with minimal training.

Artificial Intelligence (AI) technology allows statistical knowledge to be incorporated into the software. ESS contains PROLOG rules, formulated by expert statisticians. For instance, data management, graphics configuration, evaluation of methods applicability and relevance are largely automated. Statistical strategy, included in the knowledge base, allows complex statistical analyses to be conducted automatically by ESS.

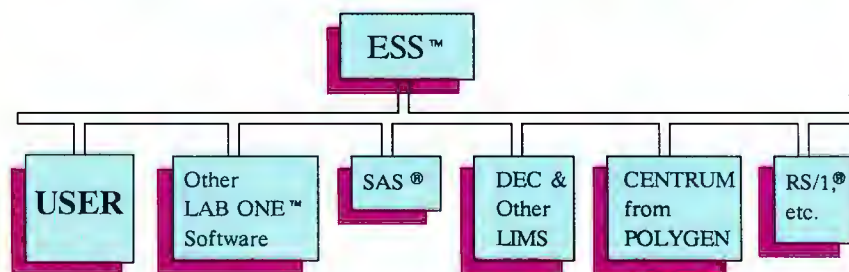
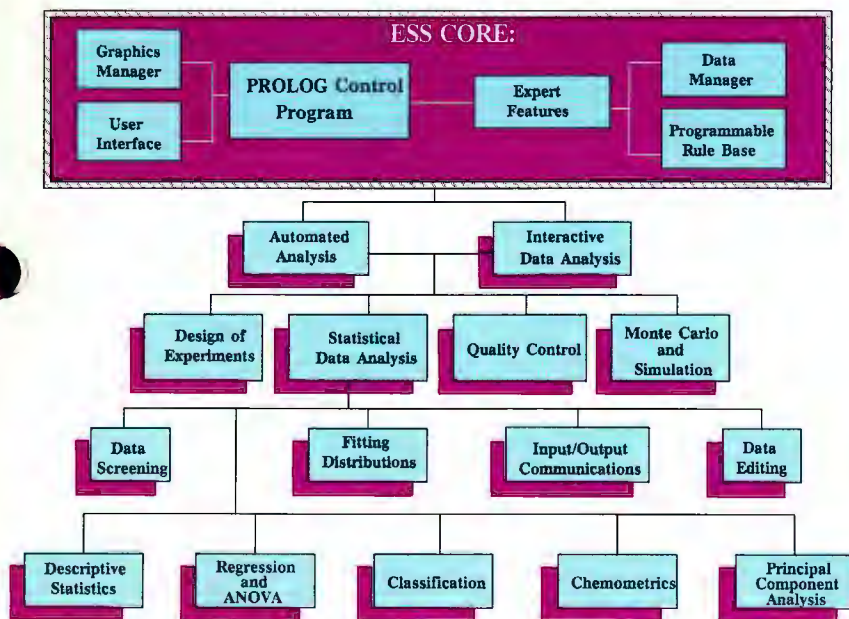
Powerful Statistical Analysis Procedures

ESS provides procedures for common statistical computations as well as advanced methods. Users will also be able to write their own rules to perform complex analyses on a routine basis.

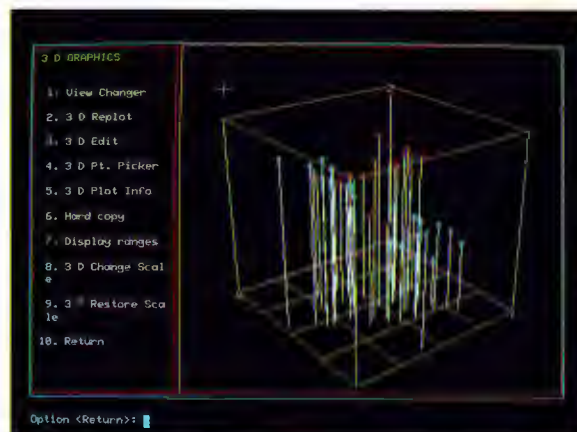
ESS offers intelligent routines for most fields of statistical computing: experimental design, quality control, data visualization and statistical data description, regression analysis, chemometrics, estimation and hypothesis testing, classification, Monte Carlo and simulation, etc. The scope and number of procedures offered is continually growing.

Focus on Data Communication

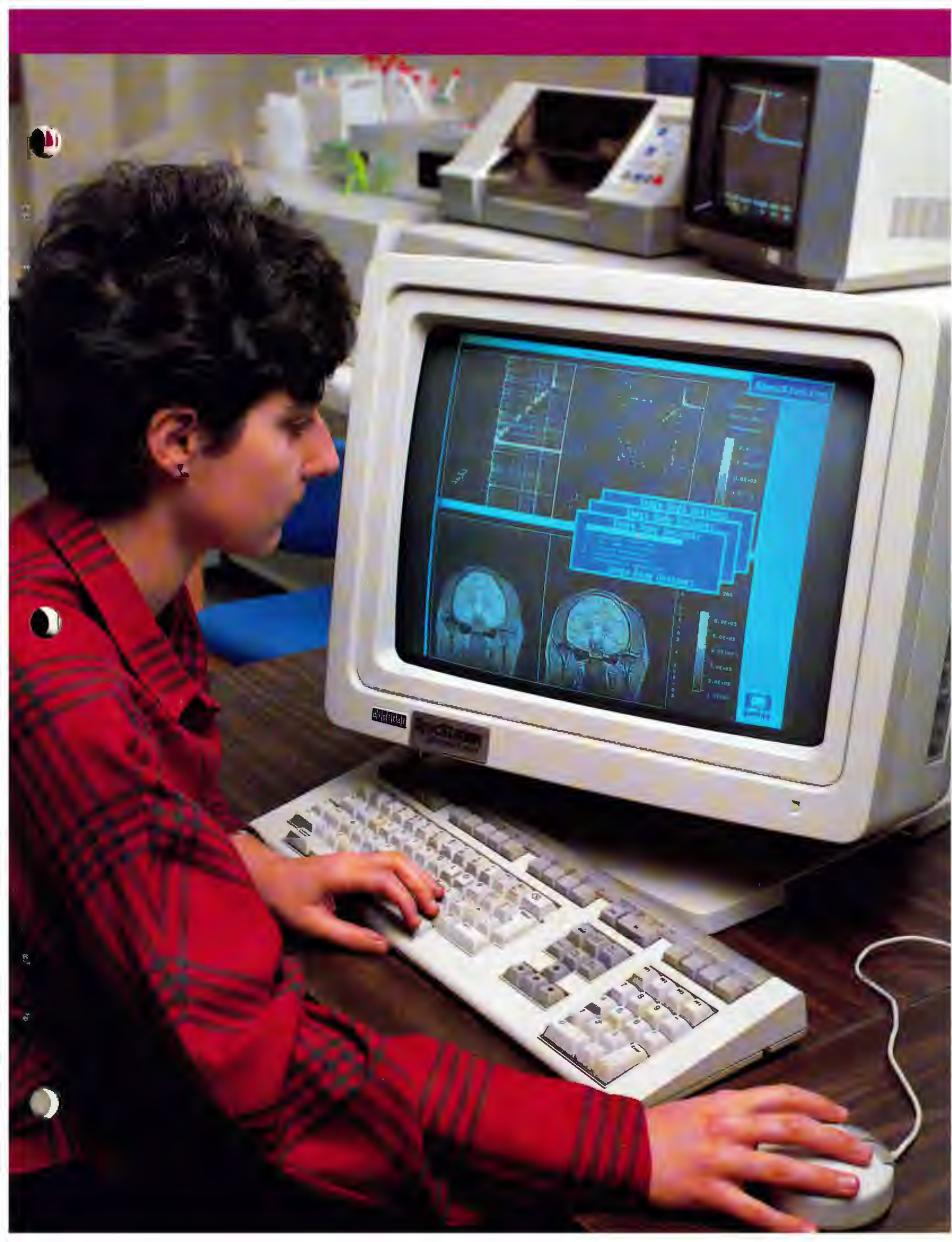
ESS can automatically exchange data with other statistical packages, DEC LIMS, or other LAB ONE applications software. Ordinary text files can also be read into ESS.



Two dimensional scatter-plot with bivariate normal probability contours, generated by ESS default configuration rules.



Three dimensional projection of two classes of experimental observations automatically generated by ESS.



NMRI's Commitment to Excellence

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NMRI

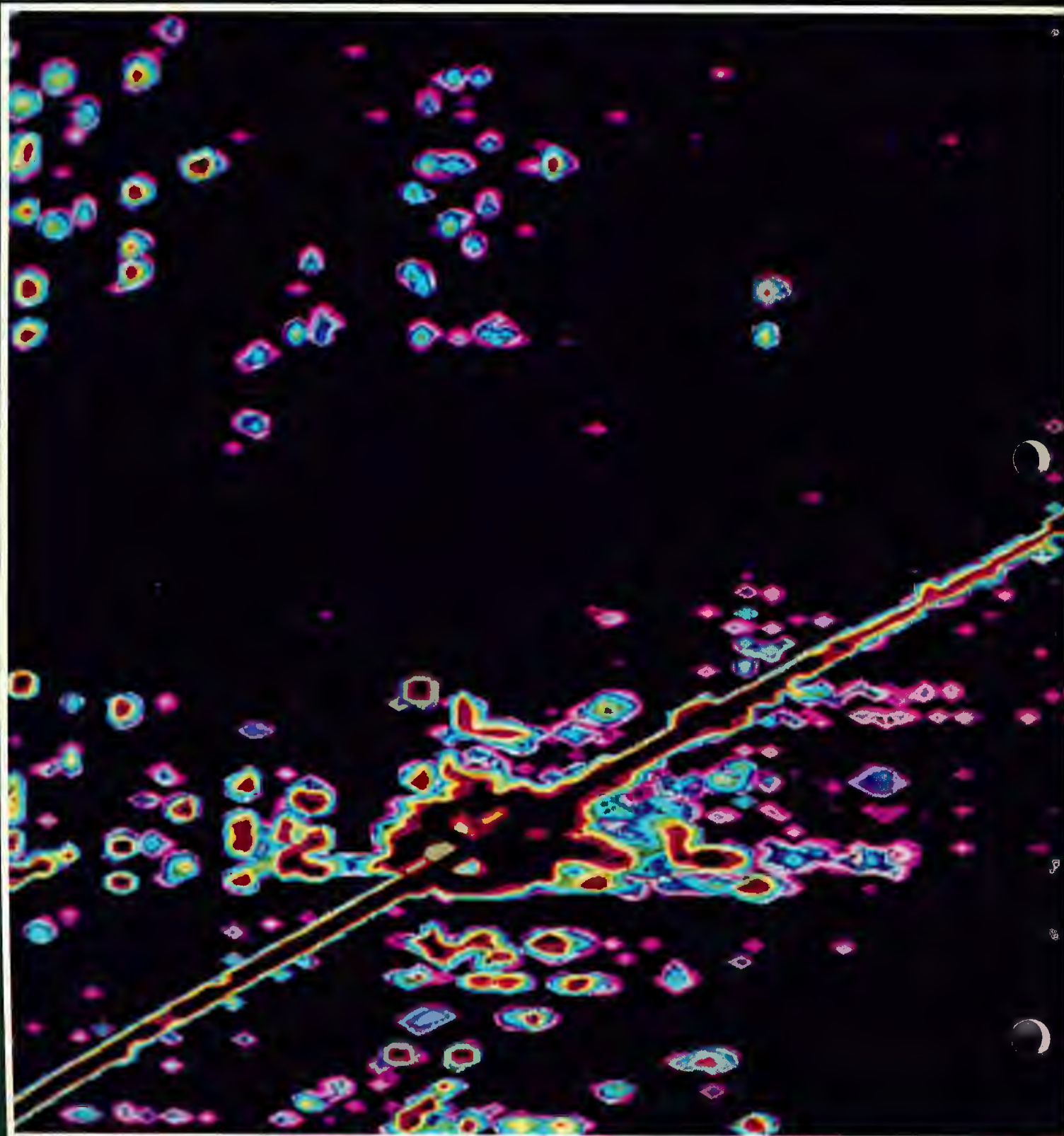
New Methods Research, Inc.

719 East Genesee Street
Syracuse, New York 13210

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DEPARTMENT OF THE NAVY
NAVAL RESEARCH LABORATORY
WASHINGTON, D.C. 20375-5000

IN REPLY REFER TO:

NRL Postdoctoral and Visiting Scientist Positions

The Naval Research Laboratory has two postdoctoral programs: the National Research Council (NRC) associateship and the Office of Naval Technology (ONT) fellowship, administered through the American Society for Engineering Education (ASEE). An immutable requirement is that the applicant be a U.S. citizen.

The NRC appointment is for two years; the stipend is expected to be \$32.5K. There are two selection cycles yearly with deadlines of 15 January and 15 April 1988. Applications are available from:

Associateship Office, GF 424
National Research Council
2101 Constitution Avenue, N.W.
Washington, D.C. 20418
(202) 334-2867

The ONT program is newer and smaller than the NRC program and the research projects are of an applied nature. Appointment is for one year, renewable for a second and possibly third year. The ONT stipends start at \$31K. There are four award cycles with deadlines of 1 January, 1 April, 1 July and 1 October 1988. For applicant's packages, contact:

ASEE
Projects Office
11 Dupont Circle, Suite 200
Washington, DC 20036
(202) 293-7080

The Laboratory has an Intergovernmental Personnel Act (IPA) program for visiting faculty members on sabbatical or leave. There is also an ASEE summer fellow program which brings university faculty to NRL for a 10 week period.

Within the Polymer Diagnostics Section we address a wide range of problems through magnetic resonance. Present efforts and interests include: adsorbed molecules; NMR imaging in solids; mechanical properties of polymers; hydrodynamics of polymers in solution; ESR of energetic materials; NMR and ESR in phospholipids. We have a new joint program involving NMR on archaebacteria with the NRL Molecular Bioengineering Branch: someone with a more biological bent would be very effective.

Please encourage anyone interested to contact one of us directly and informally.

Al Garroway	(202) 767-2323
Henry Resing	-2025
Dale Pace	-3239
Joel Miller	-2337

Or, write to: A. N. Garroway, Head, Polymer Diagnostics Section, Code 6120, Naval Research Laboratory, Washington, DC 20375-5000.



Department of Chemistry

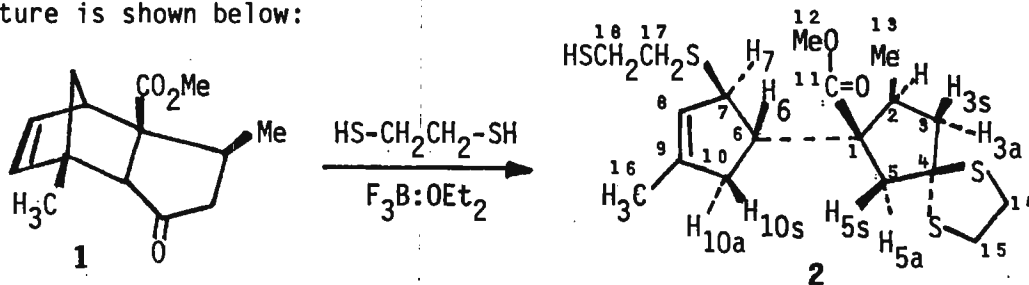
November 10, 1987 (received 11/12/87)

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

Dear Barry:

**ASSIGNMENT OF RESONANCES IN THE PROTON AND CARBON-13 NMR SPECTRA OF A
FRAGMENTATION PRODUCT DERIVED FROM 6-CARBOMETHOXY-1,exo-5-DIMETHYL-
endo-TRICYCLO[5.3.2.0^{2,6}]DEC-8-EN-3-ONE**

Reaction of the title compound (**1**) with ethanedithiol in the presence of boron trifluoride etherate has been found to afford a novel fragmentation product, **2**, whose structure is shown below:



We have assigned the structure of **2** via analysis of its proton and carbon-13 NMR spectra as detailed below.

The stereochemistries at C(6) and C(7) and the assignments of protons on C(3), C(5) and C(10) were arrived at via analysis of: (i) the magnitudes of proton-proton scalar couplings and application of the Karplus relationship and (ii) the results of proton-proton NOE experiments. Thus, the trans nature of H(6)-H(7) and of H(6)-H(10a) is suggested by the magnitudes of their respective coupling constants (i.e., 4.9 Hz, gleaned via analysis of the resonance at δ 2.62). Analysis of the resonance at δ 1.75 reveals that $J[H(6)-H(10s)] = 10$ Hz, suggesting that these protons are mutually cis. No NOE enhancement was observed between H(6) and H(7), a result which is consistent with the earlier conclusion that these protons are mutually trans.

Irradiation of H(2) results in NOE enhancement of H(3a) (δ 2.2) and of the methyl protons at C(13). However, no corresponding NOE enhancement is observed for H(3s) (δ 1.7). These observations suggest that H(2) and H(3a) are mutually cis, whereas H(2) and H(3s) are mutually trans. Assignment of H(5s) and H(5a) (δ 3.05 and 2.3, respectively) is facilitated by the observation that irradiation of H(2) produces sharpening of the resonance at δ 2.3 but has no effect upon the appearance of the



resonance at δ 3.05. Long-range (four-bond) coupling via a "zigzag" (i.e., " ω ") pathway is anticipated between H(2) and H(5a) but not between H(2) and H(5s). Irradiation of either H(12) or H(13) produced no detectable change in the signals that correspond to either H(5s) or H(5a); hence, we were unable to confirm the assignments of H(5s) and H(5a) via an NOE experiment.

Assignments of C-13 signals that appear in the Table are consistent with the observed spin-lattice relaxation times. In particular, the four quaternary carbons C(1), C(4), C(9) and C(11) all display relatively long T_1 's with C(9) having the shortest T_1 [due, apparently, to the greater number of neighboring protons that can contribute to dipole-dipole relaxation of C(9)]. All of the methine carbons, i.e., C(2), C(6), C(7) and C(8), display spin-lattice relaxation times on the order of ca. 1 second, while ring methylene carbons C(3), C(5) and C(10) all display considerably shorter T_1 values (ca. 0.6 seconds, which is roughly half of that for the methine carbons). This result is consistent with **2** behaving as a relatively rigid structure that undergoes overall isotropic tumbling in solution.

T_1 values for the methylene carbons, C(14) and C(15), in the dithioketal ring are considerably longer than are the corresponding T_1 values for C(3), C(5) and C(10). Apparently, C(14) and C(15) undergo additional internal motion relative to the methylene carbons in the cyclopentene and cyclopentane ring in **2**.

Methyl carbons C(12) and C(16) both display T_1 values that are roughly three times that of methine carbons C(2), C(6), C(7) and C(8); this result suggests that both methyl groups are free rotors. By way of contrast, T_1 of methyl carbon C(13) is considerably shorter than that of methyl carbon C(12) and of methyl carbon C(16); this suggests that rotation of the C(13) methyl group is restricted.

Experimental. Proton and carbon-13 NMR spectra were obtained at 300 MHz and 75 MHz, respectively. Proton NMR experiments were performed at 4000 Hz spectral width, 32 K transform, no delay between acquisitions. Carbon-13 NMR experiments were run with Waltz proton decoupling and typically were performed at 17,500 Hz spectral width, 32 K transform, no delay between acquisitions. Proton-proton NOE experiments were usually one transient with a continuous wave decoupling field with $\gamma H_2 = 112$ Hz. Carbon-13 T_1 values were obtained by using the standard inversion-recovery² pulse sequence with a 60 second delay time between acquisitions. Data were analyzed by using the manufacturer's standard three-parameter fitting routine.

Carbon-13 chemical shift assignments in **2**

Carbon Atom	δC	$T_1(\text{obs})(\text{sec.})$
1 (or 4)	67.68	13.3
2	41.19	1.0
3	52.91	0.6
4 (or 1)	61.20	11.3
5	47.69	0.6
6	47.08	0.9
7	52.12	1.0
8	125.67	1.1
9	142.03	7.0
10	42.46	0.7
11	175.73	12.6
12	51.59	2.8
13	14.41	0.8
14 (or 15)	39.79	1.4
15 (or 14)	40.12	1.3
16	16.51	2.9
17 (or 18)	34.52	1.7
18 (or 17)	25.26	1.9

Proton chemical shift assignments in **2**

Proton	δH	Comments
2	2.55	$J(2-13) = 6.7$ Hz
3a	2.2	
3s	1.7	
5a	2.3	unresolved $^4J(2-5a)$
5s	3.05	$J(5a-5s) = 15.5$ Hz
6	2.90	$J(6-10s) = 10$ Hz; $J(6-10a) = 4.9$ Hz; $J(6-7) = 4.9$ Hz
7	3.82	
8	5.32	br s
10a	2.62	
10s	1.75	
12	3.65	
13	0.95	
14,15	3.30	multiplet
16	1.7	br s
17,18	2.8	multiplet

Sincerely yours,

Alan Marchand *Ruthanne Thomas* *V. Vidyasagar* *Don Ellington*
 Alan Marchand, Ruthanne Thomas, V. Vidyasagar,
 and Don Ellington



SYRACUSE UNIVERSITY

NMR and DATA PROCESSING LABORATORY
GEORGE C. LEVY, DIRECTOR (315) 423-1021

DEPARTMENT OF CHEMISTRY, BOWNE HALL, SYRACUSE UNIVERSITY, SYRACUSE, N.Y. 13244-1200

November 10, 1987
(received 11/12/87)

Professor Bernard L. Shapiro
966 Elsinore Court
Palo Alto, California 94303

Dear Professor Shapiro:

Spectral Quantitation by MEFS

Our laboratory has been evaluating alternative computer processing methods for use in NMR spectroscopy. One method we have been using, Maximum Entropy Fourier Spectral Deconvolution (MEFS)(1), has been showing great promise as a deconvolution tool which preserves quantitative information as well as offering increased apparent S/N and resolution enhancement.

The figure shows the deconvolution result of a ^{13}C spectral region of a steroid. The S/N of the original data was lowered by adding experimental noise. To confirm the accuracy of the integrals, the experiment was repeated on synthetic data, modeled after the steroid data, where integral values for each peak are known. Integral values for the MEFS result were obtained using an algorithm based on Simpson's Rule. Values for the "matched filter" result were obtained by curve fitting using Lorentzian lineshape models(2). This test was repeated in replicate (same spectral region but different synthetic noise) for this and a variety of other cases also modeled after experimental spectra. Some cases included large solvent peaks (100:1 and 1000:1 dynamic ranges) to test the software performance in such cases. Comparison of the relative integral values show that MEFS of spectral regions gives accuracies that are at least as good but more often are better (as much as a factor of 2 or more) than conventional FFT and curve fitting methods in low S/N ($\leq 10:1$) cases(3).

The technique is powerful in that it can select a region of interest from a large data set and create a separate pseudo-FID for the region. This obviates many of the limitations encountered in regular maximum entropy processing: (a) solvent peaks or other large peaks can be excluded from the calculation, avoiding dynamic range limitations on MEM, (b) fewer data points are needed for calculating reconstructions of selected spectral regions,

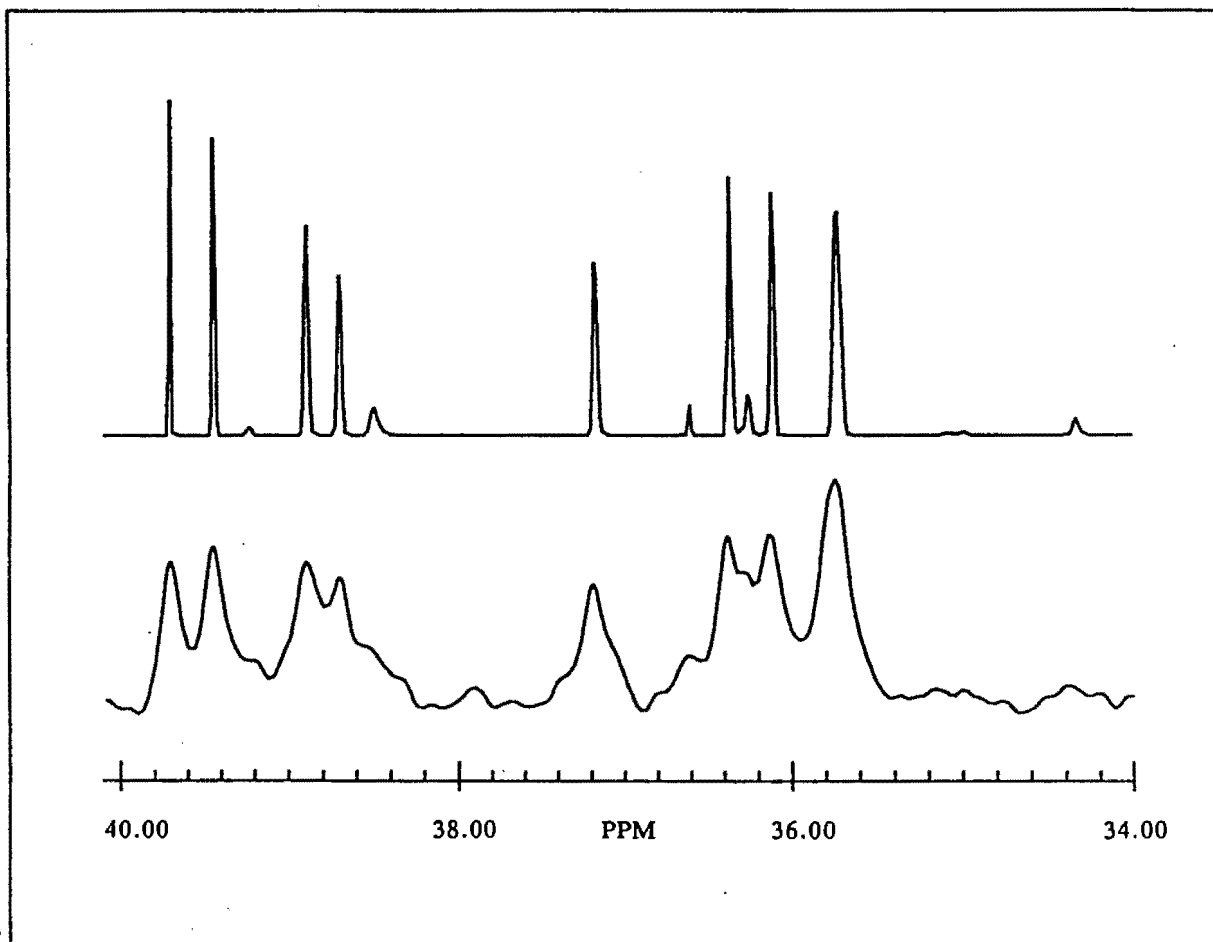
leading to efficient calculations. We would also like to note that this method is a general technique for use on all "spectral" data and not limited to deconvolution of NMR data.

Sincerely,

Anthony R. Mazzeo
Anthony R. Mazzeo

George C. Levy
George C. Levy

1. Ni, F., G.C. Levy, and H.A. Scheraga, J. Magn. Res. 66, 385(1986).
2. Kumar, A., C.H. Sotak, C.L. Dumoulin, and G.C. Levy, Comput. Enhanced Spectros. 1, 107(1983).
3. Mazzeo, A.R., M.A. Delsuc, and G.C. Levy, submitted to Computer Enhanced Spectroscopy 1987.



A ^{13}C spectral region of a steroid is shown. The bottom figure shows the conventional "matched filter" spectrum, the top figure shows the MEFSD reconstruction with enhanced resolution and higher apparent S/N.



TEXAS CHRISTIAN UNIVERSITY

Box 32908

Fort Worth, Texas 76129

817-921-7195

Department of Chemistry

November 12, 1987

(received 11/14/87)

Dear Barry:

After many fruitless trials and a number of consultations with more knowledgeable persons, I had pretty well concluded that the 2-D NOE experiment in either the plain vanilla or more elegant phase sensitive versions (NOESY) just didn't work with small organic molecules in the range of 200-400 MW. True, one did occasionally see examples in the literature which purported to have successfully done the experiment. Often these involved somewhat larger molecules and 500 MHz instruments, and sometimes I just didn't believe the results.

A couple of weeks ago, David Lankin of Varian informed me that he had defined some conditions that should allow us to do the experiment with success on our XL-300. Basically what is required is minimize the t_1 noise by temperature control and not spinning the sample. The preacquisition delay should be 1-3 \times , the longest proton T_1 and the mix time should be about equal to that T_1 . The NOESY software for the XL-300 is based on the procedure of States, Haberkorn and Ruben, J. Magn. Reson. 48, 286 (1982). No special treatment of the mix period is used, but the mix times for small molecules (3s seems a good trial value for both periods) are sufficiently long that COSY correlations as well as zero and multiple quantum coherences have faded away. It is recommended that the number of transients (NT) equal 16 or a multiple thereof so that the full phase cycle is realized.

The example below was taken from a molecule we've recently synthesized. The proton assignments were based on a wide variety of information not detailed here. A COSY spectrum, not shown, was run under conditions showing only three bond correlations (NI=64). These correlations are indicated by dotted Xs. The NOESY spectrum was acquired in deuteriochloroform solution with 64 increments in each phase with a 3s preacquisition delay and mix time respectively. The spectrum was processed as usual with a very light exponential window in each dimension. As can be seen, the ortho proton correlations are evident as are the peri interactions between 1-8 and 4-5. Two other molecules of similar size but entirely different structure have also been done with equally satisfying results.

Sincerely,

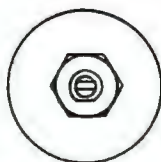
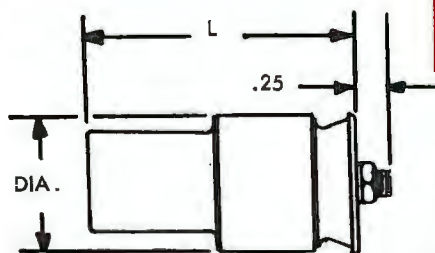
A handwritten signature in cursive script that reads 'Bill'.

William B. Smith
Professor

Polyflon **DESIGN DATA**

RF Variable Capacitors (0.3-25pF)

Non-Magnetic - High Voltage - High Power - High Q
Screwdriver Adjust Rotating Piston Series



Specifications

Capacitance Range	0.3-25pF
Working Voltage	To 10 Kv Peak
Dielectric Strength	200% of working voltage
Quality Factor (Q)	5000 or greater
Insulation Resistance	10 ¹⁶ Megohms
Operating Temperature	-55°C to 150°C
Temperature Coefficient	50 ppm/°C
Tuning Torque	4 inch ounces (max)

Ordering Instructions

RP-VC **10** - **12** - **25**
 Style Kv (Peak) Test Lead Option
 Max. Capacitance rating expressed in picofarads (pF)

Design

PTFE anode and mount surfaces are metalized with electro-deposited copper. Polyflon's proprietary plating process eliminates the possibility of air being trapped between the PTFE and copper interface which could break down under voltage stress.

Part Number	Capacitance Range (pF)	Peak (Kv) Working Volts	Peak (Kv) Test Volts	Max. (RMS) Current (amps)	Dimensions/inch (mm)		Wt./Ounces (Approx)
					L(±.020")	Dia.(±.020")	
RP-VC3-6-*	0.3-3	3	6	0.70	0.800 (20.3)	0.375 (9.5)	0.10
RP-VC5-10-*	0.8-5	5	10	2.40	1.250 (31.8)	0.500 (12.7)	0.25
RP-VC8-6-*	3-8	3	6	1.90	0.870 (22.1)	0.560 (14.2)	0.25
RP-VC10-6-*	0.8-10	3	6	1.70	1.150 (29.2)	0.375 (9.5)	0.20
RP-VC10-12-*	0.8-10	6	12	3.40	1.530 (38.9)	0.625 (15.9)	0.75
RP-VC18-2-*	0.8-18	1	2	1.70	1.150 (29.2)	0.420 (10.7)	0.20
RP-VC25-6-*	5-25	3	6	5.50	1.430 (36.3)	0.875 (22.2)	1.00
RP-VC25-6B-*	5-25	3	6	3.90	1.540 (39.1)	0.625 (15.9)	1.00
RP-VC25-15-*	5-25	10	15	7.20	1.770 (45.0)	1.125 (28.6)	1.75

*Add code number for leads. See lead code number table below.

Notes

- Working volts and maximum RMS current must not occur simultaneously.
- Peak test voltage rating may be used for pulsed voltage applications. Peak pulsed current will be limited by the duty cycle that will produce an average current that does not exceed the maximum RMS current.
- Mounting Dimension: 1/4" diameter "D" hole. Hardware: 1/4-32 hex nut and internal tooth lockwasher. Mounting flange is at the bottom of the capacitor.

Lead Option Table

05	without lead wires
15	3" wire, top only - in line with flat
25	3" wire, top and bottom - in line with flat
35	1-1/2" strap, top only - in line with flat
65	3" wire, top only, opposite flat
75	3" wire, top and bottom - opposite flat
85	1-1/2" strap, top only - opposite flat

CRANE

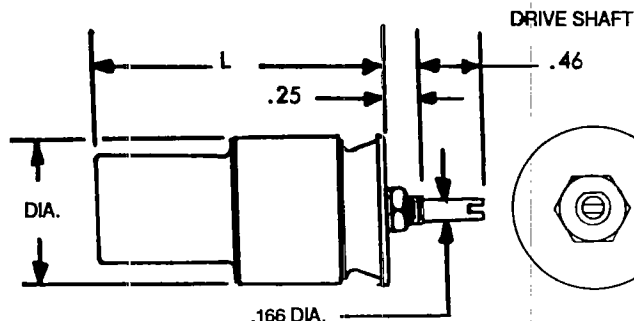
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Polyflon Company, 35 River Street, New Rochelle, NY 10801 • Tel:(914) 636-7222 • FAX: 914-633-3664

RF Variable Capacitors (0.8-85pF)

Non-Magnetic- High Voltage - High Power - High Q

Screwdriver Adjust Non-Rotating Piston Series



Specifications

Capacitance Range	0.8-85pF
Working Voltage	To 10 Kv
Dielectric Strength	200% of working voltage
Quality Factor (Q)	5000 or greater
Insulation Resistance	10 ¹⁶ Megohms
Operating Temperature	- 55°C to 150°C
Temperature Coefficient	50 ppm/°C
Tuning Torque	4 inch ounces (max)

Design

PTFE anode and mount surfaces are metalized with electro-deposited copper. Polyflon's proprietary plating process eliminates the possibility of air being trapped between the PTFE and copper interface which could break down under voltage stress.

Ordering Instructions

NRP-VC	10-	12-	26	A
Style	Kv(peak) test			Lead Option
	Max. capacitance rating expressed in picofarads (pF).			Drive shaft option (see table).

Notes

- Working volts and maximum RMS current must not occur simultaneously.
- Peak test voltage rating may be used for pulsed voltage applications. Peak pulsed current will be limited by the duty cycle that will provide an average current that does not exceed the maximum RMS current.
- Mounting dimension: 1/4" diameter "D" hole. Hardware: 1/4-32 hex nut and internal tooth lockwasher. Mounting flange is at the bottom of the capacitor.

Part Number	Capacitance Range (pF)	Peak (Kv) Working Volts	Peak (Kv) Test Volts	Max. (RMS) Current (amps)	Dimensions/Inch (mm)		Wt./Ounces (Approx)
					L(±.020")	Dia.(±.020")	
NRP-VC5-10*	0.8-5	5	10	2.30	1.560 (39.6)	0.500 (12.7)	0.40
NRP-VC10-12*	0.8-10	6	12	3.60	1.830 (46.5)	0.625 (15.9)	0.80
NRP-VC25-6*	5-25	3	6	5.70	1.620 (41.1)	0.910 (23.1)	1.25
NRP-VC25-6B*	5-25	3	6	4.20	1.620 (41.1)	0.625 (15.9)	1.25
NRP-VC25-15*	5-25	10	15	7.20	1.770 (45.0)	1.125 (28.6)	2.00
NRP-VC30-6*	3-30	3	6	10.00	2.250 (57.2)	1.500 (38.1)	4.00
NRP-VC50-6*	5-50	3	6	12.50	2.250 (57.2)	1.500 (38.1)	3.50
NRP-VC70-15*	3-70	10	15	20.00	3.250 (82.6)	1.625 (41.3)	4.50
NRP-VC85-6*	5-85	3	6	20.00	3.250 (82.6)	1.500 (38.1)	4.10

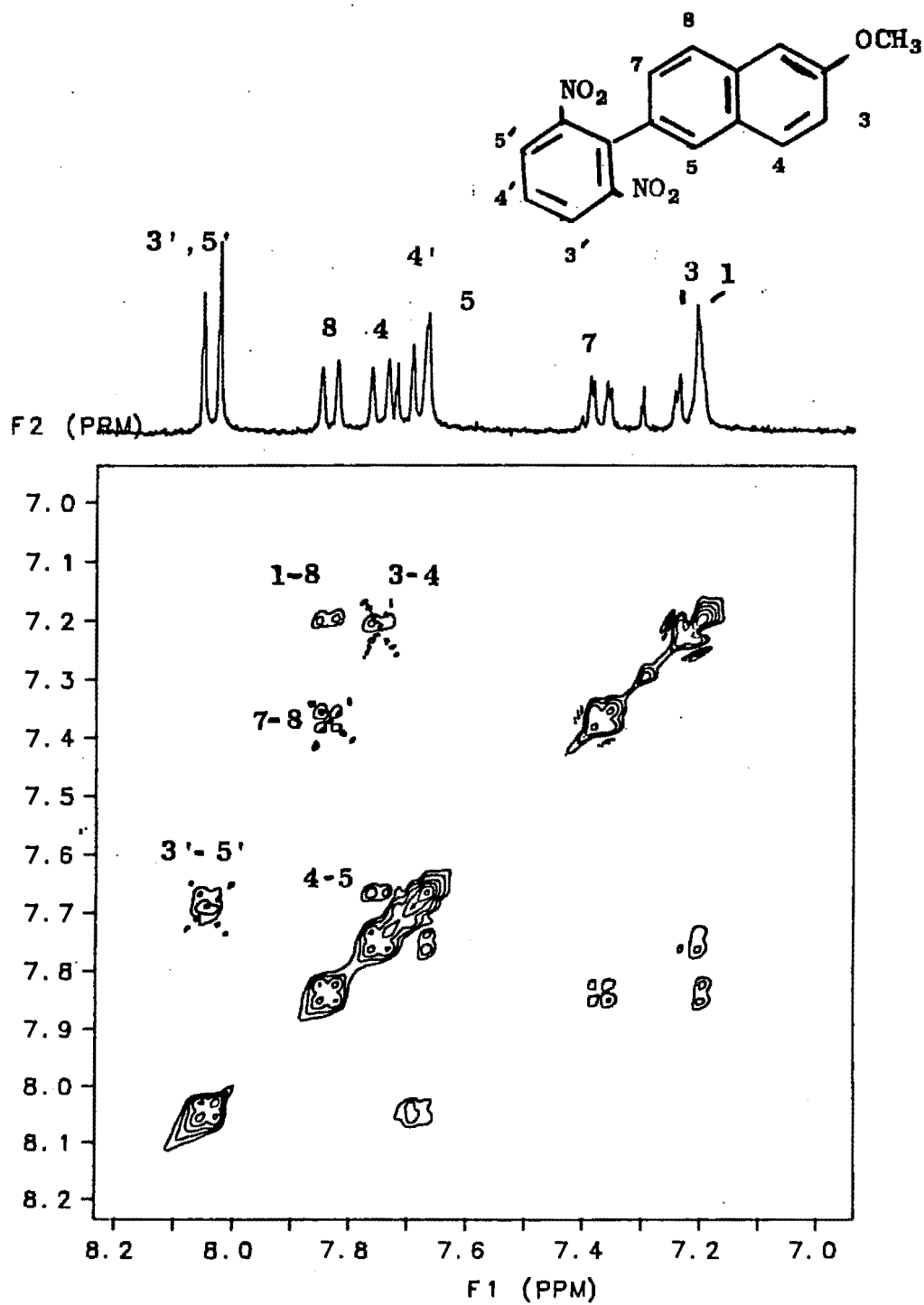
*Add code number for leads. See lead code number table below.

Drive Shaft Option Table

Option	Description
A	Extension Shaft: .125" dia. x 3" long delrin rod with .500" dia. x .250" thick knurled delrin knob.
B	Coupling Adapter: .375 O.D. x .170 I.D. (female) x .500" long to .250" dia. (male). Overall length is 1". (Has set screws)
C	Coupling Adapter: .437" O.D. x .170" I.D. (female) to .250" dia. (female) overall length is 1". (Has set screws)

Lead Option Table

06	without lead wires
16	3" wire, top only - in line with flat
26	3" wire, top and bottom - in line with flat
36	1-1/2" strap, top only - in line with flat
66	3" wire, top only, opposite flat
76	3" wire, top and bottom - opposite flat
86	1-1/2" strap, top only - opposite flat





INSTITUTE FOR CANCER RESEARCH ■ 7701 BURHOLME AVENUE ■ PHILADELPHIA, PENNSYLVANIA 19111

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

November 11, 1987
(received 11/17/87)

215/728-6900

RE: RF Spinning Sidebands

Dear Professor Shapiro:

A recent letter (TAMU 346-36) raised the question of how to reliably distinguish RF spinning sideband signals from spinning sidebands due to an inhomogeneous B field. In an excellent discussion of the NMR receiver D.I. Hoult (1) considers several aspects of the NMR probe including RF spinning sidebands. Hoult states that for a saddle-shaped coil the absence of complete cylindrical symmetry of the coil results, when the sample is spun, in sidebands which can not be removed by "shimming" or greater spinning speeds.

When RF spinning sidebands and sidebands due to inhomogeneities in B are present simultaneously the results may be confusing due to the changing phase relationship between these signals. The following procedure is offered as an exercise to detect RF spinning sidebands in a proton probe without interference from sidebands due to the B field.

1. Connect the ^1H probe and ^1H decoupler for homonuclear decoupling or a presaturation experiment.
2. Set the observe pulse width to zero (no RF pulse).
3. Insert a D_2O sample, and tune and impedance match the probe.
4. Set the observe carrier frequency 1 kHz away from the D_2O resonance, and set the decoupler frequency within a few hertz of the carrier frequency.
5. Set the spectral width to 1 kHz, data block size to 4 k, and receiver gain to a low value.
6. Set the decoupler power to the lowest power level and CW (no modulation).
7. Use the spectrometer's set-up mode (GS for Bruker, TU for Nicolet etc.) for repetitive one-scan experiments which are shown on the spectrometer display.

A low frequency sine-wave signal should appear on the display, adjust receiver gain to provide a suitable amplitude signal. When the sample is spun a modulation of the sine-wave signal may be seen. A clearer picture is available if the signal is transformed to produce the frequency domain spectrum. A 1 Hz line broadening, FT, and phase correction produced the spectra shown in Figure 1 (1A spinning at 20 Hz, 1B non-spinning).

The spectra in Figure 2 were obtained using a D_2O sample in a loaner probe (we hope for an improvement in the final product) and the typical one-pulse NMR experiment. Figure 2A results from one scan, and Figure 2B is from 8 scans. The "virtue" of RF spinning sideband signals is their amplitude, with respect to the main line, decreases with increasing scans since they are not coherent with the NMR experiment.

Sincerely,


Robert W. Dykstra

(1) D.I. Hoult, in "Progress in NMR Spectroscopy" (J.W. Emsley and L.H. Sutcliffe, Eds.) Vol. 12, Pergamon, Great Britain, 1977.

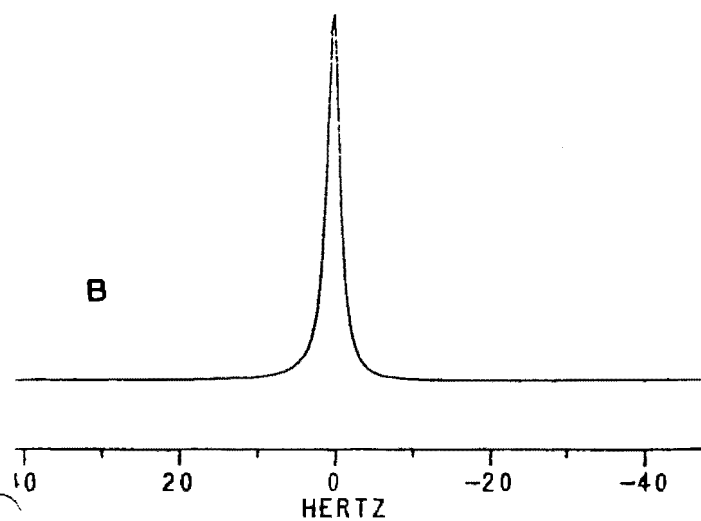
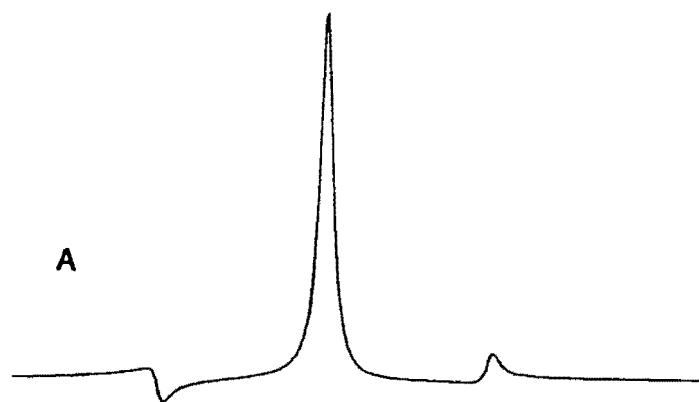


FIGURE 1

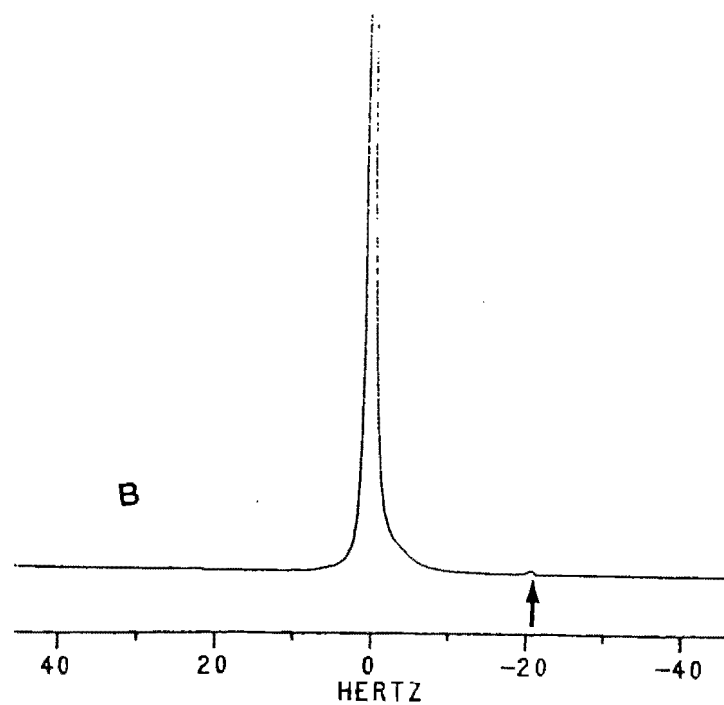
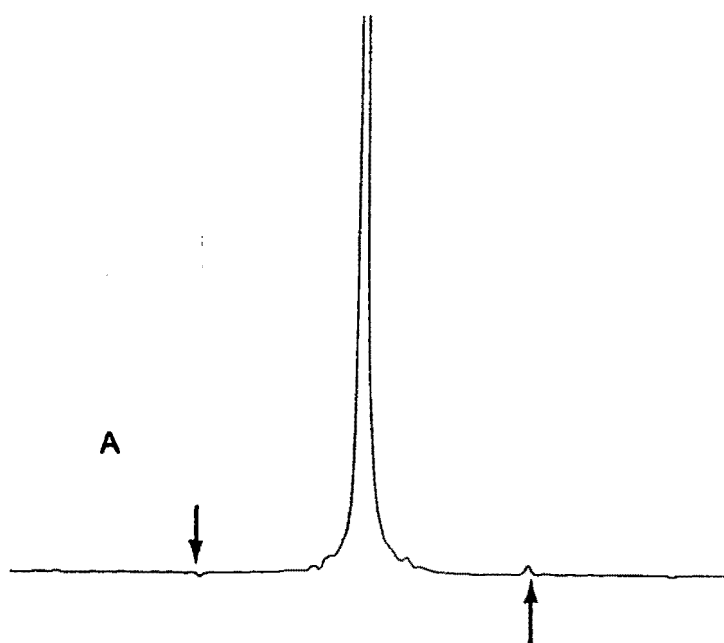


FIGURE 2

Los Alamos

Los Alamos National Laboratory
Los Alamos, New Mexico 87545

DATE: 13-November-1987
MAIL STOP: MS G740

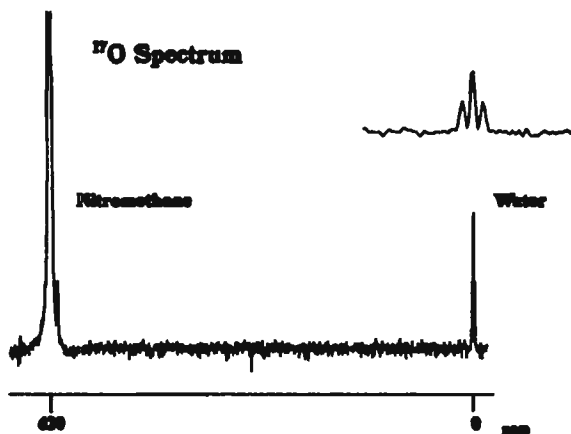
(received 11/19/87)

Prof. Barry Shapiro, Editor
Texas A&M University NMR Newsletter
966 Elsinore Court
Palo Alto, Calif. 94303

Dear Barry,

TITLE: Simple Problems Need Simple Solutions

We have been doing a lot of work on nitromethane, attempting to understand the chemistry involved in its detonation. (Contrary to the belief of many organic chemists and dragster drivers, nitromethane is neither a solvent nor a fuel but rather a relatively inefficient, insensitive high explosive.) We found reference in the Russian literature to 8 wt. % solutions of water in nitromethane and this was contrary to our subjective experience, i.e., we didn't think that you could get that much in solution. How to determine the approximate solubility? Proton NMR, of course. I ran a proton NMR spectrum of nitromethane that had been saturated with water at room temperature. Sure enough, the proton resonances completely overlap. The proton resonance for nitromethane is rather broad, presumably due to some kind of couplings to the nitrogen. The chemical shift is 4.4 ppm from TMS, and consequently the tiny water resonance cannot be seen under the nitromethane peak. I thought that I was going to have to resort to some kind of multidimensional/multiquantum experiment or (God forbid) Karl Fischer titrations. The solution is quite simple ^{17}O NMR. In such small molecules, with modern multinuclear NMR spectrometers, ^{17}O NMR is rather simple and the spectrum below took only a couple of hours.



Prof. Barry Shapiro, Editor

- 2 -

13-November-1987

The spectrum shows the ^{17}O resonances of water and nitromethane in a rather poorly shimmed magnetic field, without sample rotation. The chemical shift of nitromethane is 620 ppm from water, the linewidth is about 97 Hz, and the ^{17}O - ^1H coupling of 81.5 Hz can be seen in the water spectrum.

The concentration of water in this saturated solution was found to be about 8 mole % which is right about at the concentration which the Russian workers claim to see detonation effects.

PROBES TO GIVE AWAY

I have moved to a different group here at Los Alamos and am in the process of eliminating a lot of old hardware. To that end, we have a bunch of old Varian probes and associated hardware (including dewars) which we would be willing to give to someone who could use them. They include: A total of 9 V4230B probes from a "DP-60" (2-4, 4-8, and 8-16 Mcs). A model V4331A probe tuned for 60 MHz, a 4331B probe tuned for 56.4 MHz, and several inserts and assorted hardware for them. A total of 4 probes from an XL-100 (for the wide gap magnet) including: a V4415 probe for 2-12 MHz, a 4418 probe, and a 12 mm probe for ^{13}C with deuterium lock and proton decoupling. Anyone interested in these or anyone knowing of possibly interested parties can contact me at the address above or by telephone at (505) 667-0773. I will keep these for several months in hopes of finding a home for them.

EQUIPMENT NEEDED

We would like to acquire a field frequency lock for a Bruker CXP-200 and I wonder if anyone has one that they are interested in getting rid of. If anyone has any suggestions or improved circuitry for locking a CXP, I would be most interested in hearing from them.

Sincerely,

Bill

William L. Earl.

CALIFORNIA INSTITUTE OF TECHNOLOGY

Pasadena, California 91125

Division of Chemistry and Chemical Engineering
Gates and Crellin Laboratories of Chemistry

John D. Roberts
Institute Professor of Chemistry

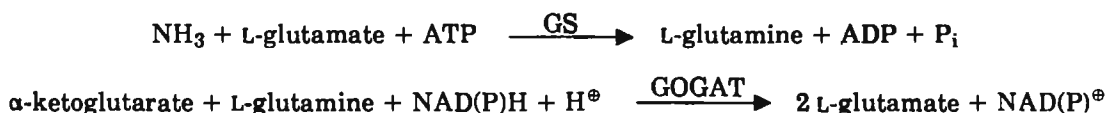
Professor Bernard L. Shapiro
Department of Chemistry
Texas A&M University
College Station, Texas 77843

November 18, 1987
(received 11/23/87)

Enzyme-Activity Measurement by ^{15}N NMR

Dear Barry,

The glutamine synthetase (GS)/glutamate synthase (GOGAT) pathway



constitutes one of the important pathways of ammonia assimilation into glutamic acid in microorganisms and plants. The question of how the synthesis of GOGAT is regulated in response to nitrogen availability is not as well understood as that of GS and is under active investigation through measurements of the specific activities of GOGAT in microorganisms growing with different nitrogen sources.

The activity of GOGAT in cell extracts is usually measured by addition of cell extracts to an assay solution containing 5 mM L-glutamine, 5 mM α -ketoglutarate and 0.25-0.3 mM NAD(P)H in a buffer at pH 7.3, and observation of the time-dependent decrease in UV absorbance at 340 nm arising from oxidation of NAD(P)H. The initial concentration of NAD(P)H in the assay solution is low, $\sim 1/20$ th of that of the other substrate, in order to maintain the UV absorbance in the range of 1.0 to 1.5. One problem with this method is that, when the cell extract contains NAD(P)H oxidase activity at a level much higher than that of GOGAT, detection of the GOGAT activity is difficult because of the rapid oxidation of NAD(P)H by the NAD(P)H oxidase. Under such circumstance, we have found that ^{15}N NMR provides a convenient method for assaying the GOGAT activity because it allows the addition of equimolar, or excess, NAD(P)H and detection of the product without separation from the substrates.

Fig. 1 (A-C) shows some typical ^{15}N NMR spectra of GOGAT assay solutions containing 5 mM [$\gamma\text{-}^{15}\text{N}$]-glutamine, 5 mM α -ketoglutarate and 15 mM NADPH at three time intervals after the addition of the cell-free extracts of N_2 -fixing *Bacillus macerans*. After the reaction was terminated by acidification to pH 2.0 to denature and precipitate the enzymes, the ^{15}N NMR spectra were taken at 50 MHz on a Bruker AM-500 NMR spectrometer. The formation of [^{15}N]glutamine acid, as observed from the increase in the intensity of its peak at 335.1 ppm, was linear with time for 30 min. The nmoles of [^{15}N]glutamine acid formed were determined from the peak intensity by calibration with a standard. No [^{15}N]glutamic acid was formed when either NADPH or α -ketoglutarate was omitted from the assay solution (Fig. 1D and 1E). A GOGAT activity of 4.5 nmoles \cdot min $^{-1}\cdot$ mg protein $^{-1}$ was obtained from these data. The method which, to the best of our knowledge, has not been used previously, provides a useful, rapid way to measure GOGAT activity in the presence of NAD(P)H oxidase activity.

Sincerely yours,

Keiko Kanamori *Jack*

Keiko Kanamori

John D. Roberts

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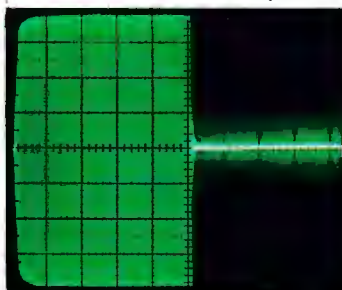
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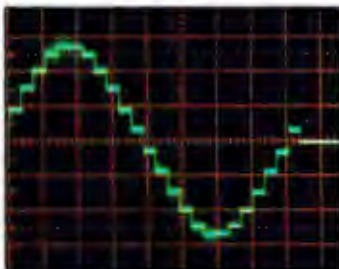
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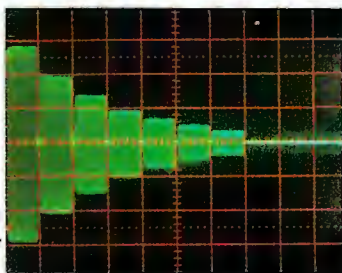
A 5 μ sec ^1H observe pulse. ▼



15° phase shift through 360°. ►



Shaped decoupler pulse. ►



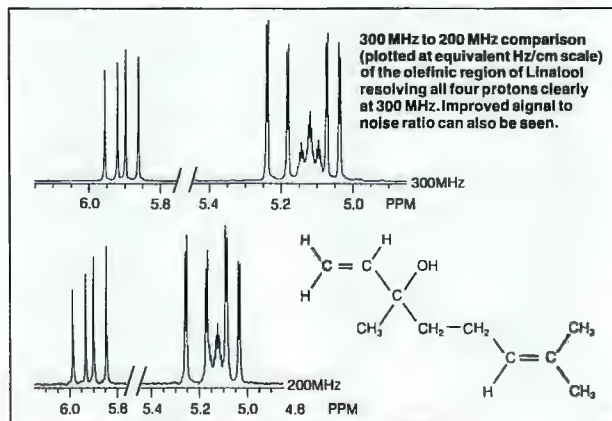
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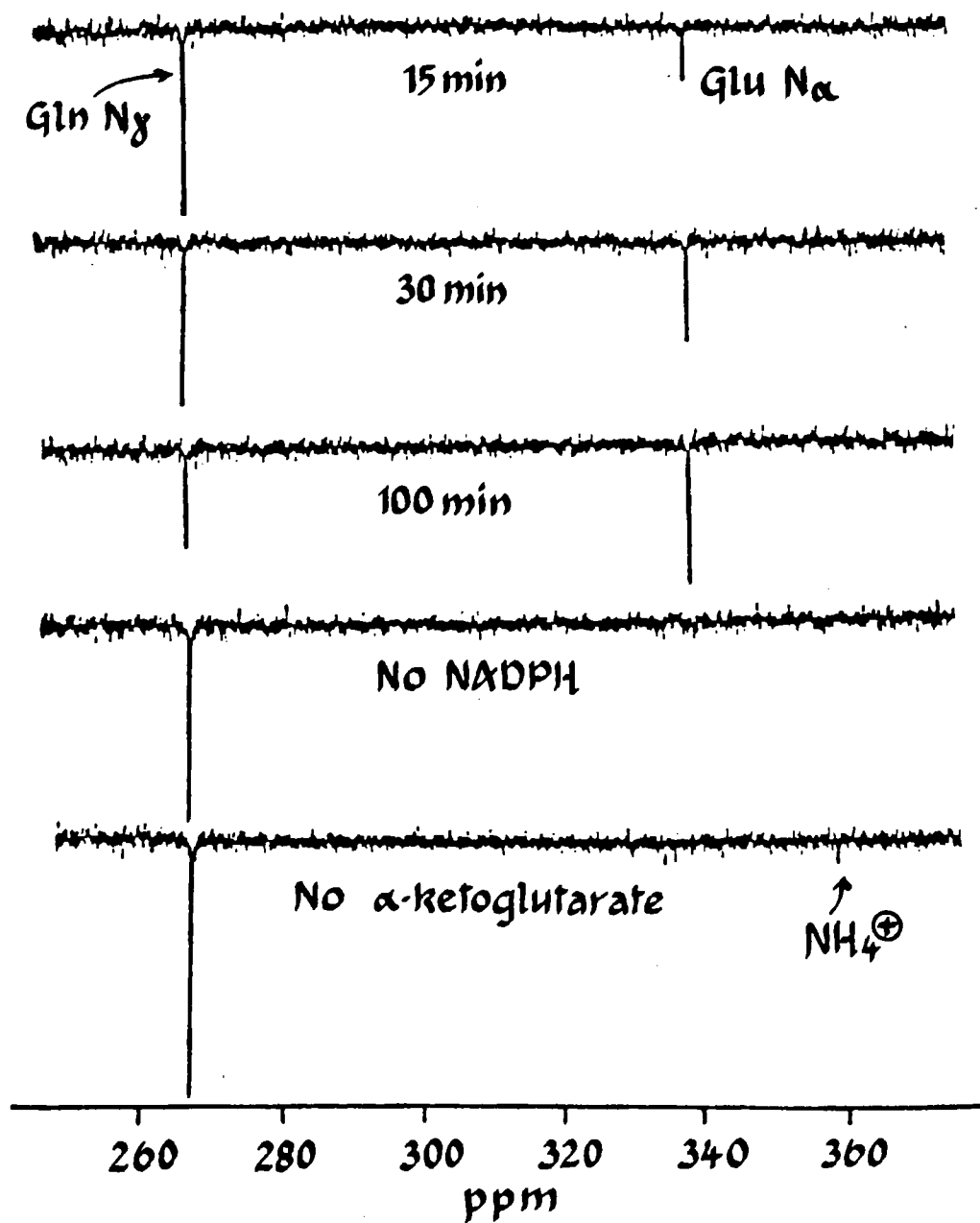
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Figure 1



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MARBURG, DEN 19.11.87 (received 11/23/87)

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

Fossel Test with Automatic Sample Changers

Dear Professor Shapiro,

it is not nice to send an "Ultimatum" before having send a "Reminder". As many others we were intrigued by the report of Fossel et al [1] on Tumor detection by simple NMR. In collaboration with Dr. T. Kämpchen from Marburg Pharmacy, Dr. K.-H. Pflüger and J. Fischer from Haematology we started a project of plasma measurements.

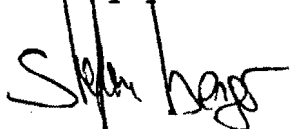
Both Chemistry and Pharmacy in Marburg have NMR instruments equipped with automatic sample changers, one a Bruker AC-300 and the other a JEOL GX-400. We found, that the process is easily adopted to full automation. As already reported by others [2] we use a combination of presaturation and inversion recovery sequence to eliminate both the water and the lactate signal. Due to our instrumental setup we were able to check on a large scale (by now about 160 plasma samples) the reproducibility of the measurements for the same plasma sample with two different nmr instruments of different manufacturers at two different field strength, the spectra evaluated by many different persons.

Bruker 300 MHz results correlate well enough with Jeol 400 MHz measurements if the lactate problem is eliminated. The average of the 300 MHz values is by 3.6 Hz lower than the 400 MHz average, and for "normal" controls our 400 MHz results are 5 Hz lower than the average reported by Fossel. We find, that the methyl resonance is not reliable and will in future focus on the methylene group, where we have a reproducibility better than 1 Hz, regardless of handling techniques, or operator influence. Only storing at room temperature increases the linewidth of the methylene peak by 3 Hz after 24h.

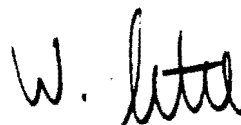
Recently we had six apparently healthy persons undergo a day profile, taking blood early in the morning before breakfast, shortly before lunch and one hour after lunch. We did not find a larger variation than 1.5 Hz, thus it seems not essential, to define a certain time or condition, when the blood of a patient is being taken.

Despite all these experimental efforts we are not yet ready to affirmate the findings by Fossel, nor as several others [3] to question their results. The patient samples so far measured in our hands were all more or less treated cancers and thus the variety of linewidth is too large to have clear predictive value. We will continue our efforts in this project until we can conclude on a firm basis about the usefulness of such a cancer probe.

Sincerely yours

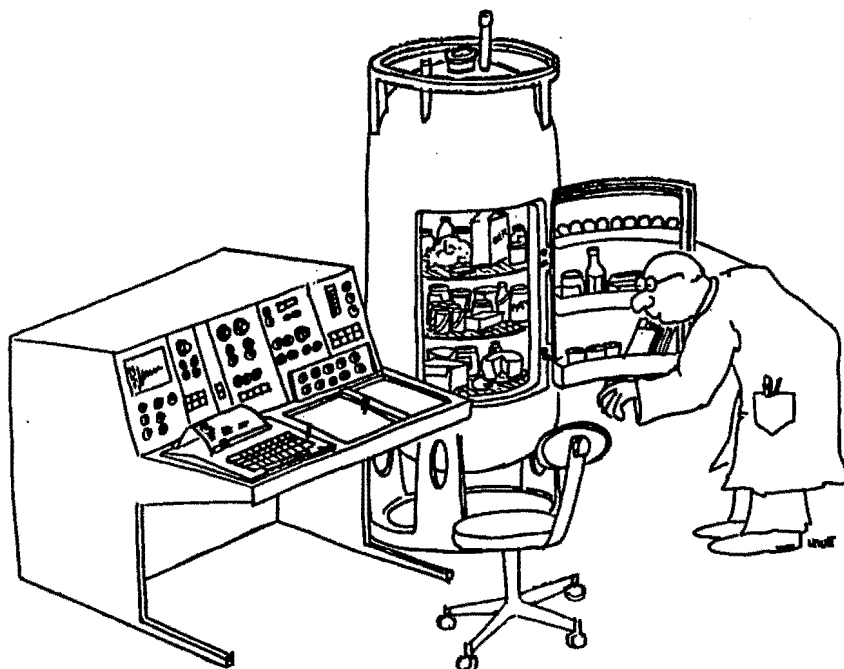


(S. Berger)



(W. A. Etzel)

- [1] E. T. Fossel, J. M. Carr and J. McDonagh, New Engl. J. Med. **315**, 1369 (1986)
- [2] G. D. Williams and K. R. Metz, TAMU **346**, 4 (1987)
- [3] See Correspondence in New Engl. J. Med. **316**, 1411 (1987)



(reproduced through the courtesy of Dr. Uwe Oehler,
Department of Chemistry, University of Guelph, Canada.)

DEPARTMENT OF MEDICINE

630 West 168th Street

November 4, 1987

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

Dear Dr. Shapiro:

I would appreciate your posting the following advertisement for a POST-DOCTORAL position in the NMR research center at The College of Physicians and Surgeons of Columbia University.

A Postdoctoral training position for Ph.D.'s or M.D.'s is available immediately in biologic cardiovascular MRS (*in vivo* and *in vitro*). The position requires a one or preferably two year commitment and is funded through an NIH Cardiovascular Training grant. Interested applicants should send a C.V. to Dr. Andrew M. Keller, Department of Medicine, Cardiology Division, 8-Stem, Columbia University, 630 W. 168th Street, New York, NY, 10032. A brief description of our facilities follows.

All biologic work is performed on one of two wide bore Bruker AM 3000 spectrometers at either 300 MHz or 400 MHz. All equipment is located at the NMR center of Columbia University College of Physicians and Surgeons in Manhattan which is directed by Dr. Dinshaw Patel. The 300 MHz magnet is equipped with 20 mm ^{13}C , ^1H , ^{23}Na , and ^{31}P singly tuned probes, and the 400 MHz is equipped with a 20 mm broad band probe. In addition a ^1H surface coil for rats (300 MHz) has recently been constructed, and a 500 MHz narrow bore magnet is available for high resolution experiments. Finally complete facilities are present for sample desiccation and biochemical analysis.

Sincerely,



Andrew M. Keller, M.D.
Assistant Professor of Medicine and Radiology



STANFORD MAGNETIC RESONANCE LABORATORY
STANFORD UNIVERSITY
STANFORD, CALIFORNIA 94305-5055

Director: Oleg Jardetzky, M.D., Ph.D.
Professor of Pharmacology

415/723-6153

November 17, 1987

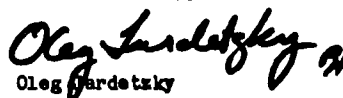
SMRL OFFICE:
415/723-6270

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

Dear Barry,

We currently have an NMR Spectroscopist/Research Engineer position open at Stanford Magnetic Resonance Laboratory. Responsibilities include maintenance and upgrading of SMRL spectrometer systems (Bruker AM-500, HX-360, MSL-100 [Biospec-II] and modified Varian XL-100), implementation and development of instrumentation and NMR techniques including probe design and construction and experimental design, supervision of instrument use and collaboration on research projects. Proficiency in NMR electronics, hardware and software essential. Candidates should have an advanced degree in engineering, physics or physical chemistry. Rank and salary depend on specific qualifications and experience. Interested applicants should contact me at the above address. Stanford is an EO/AA employer.

Yours sincerely,



Oleg Jardetzky

WARNER-LAMBERT/PARKE-DAVIS PHARMACEUTICAL RESEARCH COMPANY**POSITION AVAILABLE**

The Parke-Davis Pharmaceutical Research Division has an excellent employment opportunity in its Ann Arbor, MI facility for an NMR spectroscopist. Presently, the NMR laboratory is equipped with 100, 200 and 300 MHz NMR systems and three laboratory computers. A 250 MHz spectrometer and NMR modeling computer have been ordered and will be added to the laboratory before the end of 1987. The purchase of a new 500 MHz NMR spectrometer is planned for 1988.

A Ph.D. in chemistry with 1-5 years of experience in organic or biochemical NMR applications with a sound knowledge of computers is required. The successful candidate will be expected to administer the development of a 250 MHz spectrometer and modeling computer as well as assist in the purchase of the proposed 500 MHz NMR spectrometer. A demonstrated ability to perform advanced NMR experiments, maintain NMR instrumentation, supervise laboratory staff, and collaborate with a variety of investigators is necessary to be effective in our research program.

For confidential consideration please send your resume directly to:
Dr. Gary McClusky, Spectroscopy Group Leader, 2800 Plymouth Road,
Ann Arbor, MI 48105

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INDUSTRIAL MAGNETIC RESONANCE DIVISION

20 November 1987

Dr. Barry Shapiro
TAMU Newsletter
966 Elsinore Court
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Contact Ellory Schempp, Auburn International, Inc., P.O. Box

TEXAS A&M UNIVERSITY



No. 351
December 1987

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* * * * *

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* * * * *

Concerning the Physical Format and Nature of Contributions to the Newsletter Contents

The Newsletter subscription list continues to grow, but this does put increasing pressure on both the economics and the logistics involved. I am most reluctant to entertain any idea of a backlog or an inventory of letters or other contributions. I'm sure everyone would much rather continue to have each Newsletter contain *all* material received by the deadline date for that issue. Accordingly, permit me to urge each Newsletter contributor/participant to make a suitable effort toward saving space - in these days of the ubiquitous computer, small type (but not less than 10 point, which is the size used for this page, s.v.p.) helps markedly. Several contributors have already made this important discovery

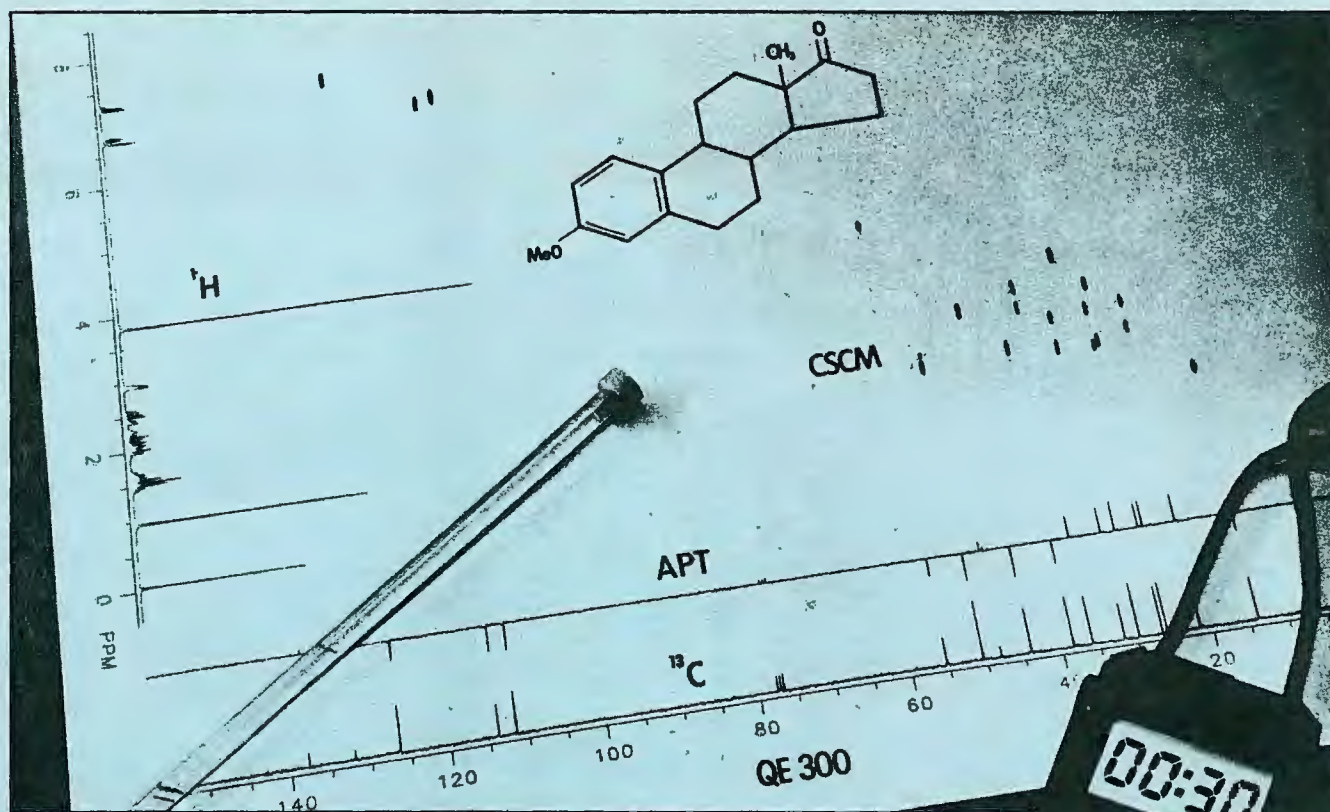
Please give the following matters (some of them new) your best attention:

1. Please provide titles for all topics of your contributions. Failure to provide titles - suitably succinct ones! - may result in my doing so, with possible attendant loss of signal. Ask Alan Marchand
2. "Positions Available", "Equipment Available", and similar notices should be formatted so as to use the *minimum vertical space*. Please do not double space such notices, or leave wide margins at the sides. I believe that such notices should rarely if ever need to exceed one-half of a page, and henceforth, such notices which exceed 4.5" (11.4 cm) in the vertical dimension, *including letterhead, address, etc.*, will be respectfully declined, and returned for condensing (This will usually result in the notice being delayed until the next Newsletter issue.). A notice which really needs to be more than 4.5" tall must be agreed to in advance - give me a phone call - am I not the most reasonable of men?

These space restrictions do not apply to meeting notices, etc., but your cooperation here will also be appreciated.

3. Contributions may not exceed three pages without prior approval.
4. All contributions must be formatted so as to leave a 1" (25.4 mm) margins on all four sides. This is especially important for foreign subscribers, whose paper is usually larger than that used in the U.S.A. and Canada.

B.L.S.



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^1H , ^{13}C , APT, and 2D NMR in $\frac{1}{2}$ hour - automatically!

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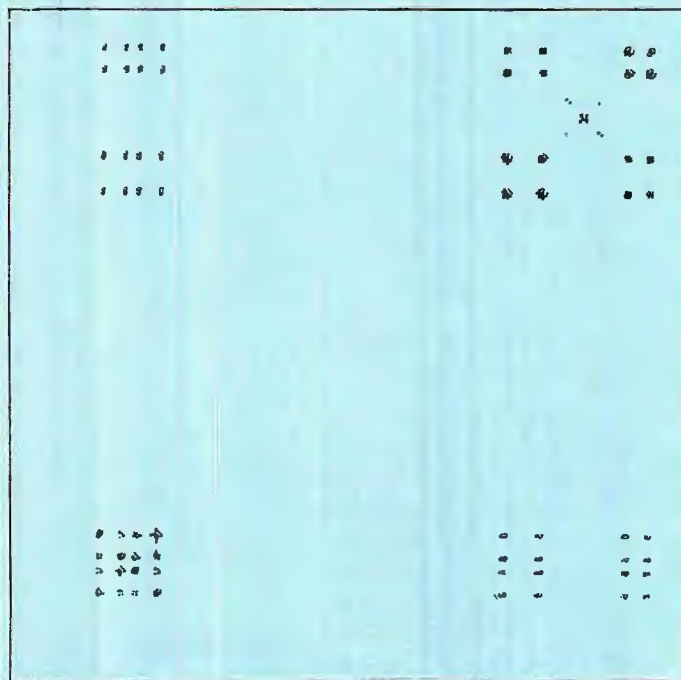
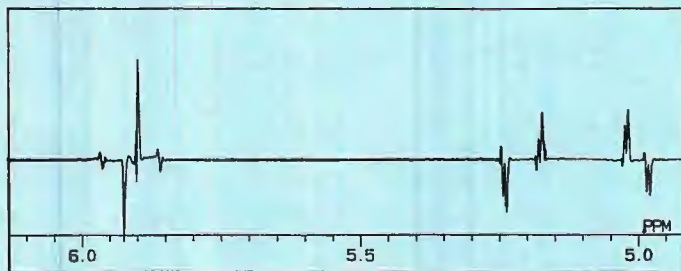
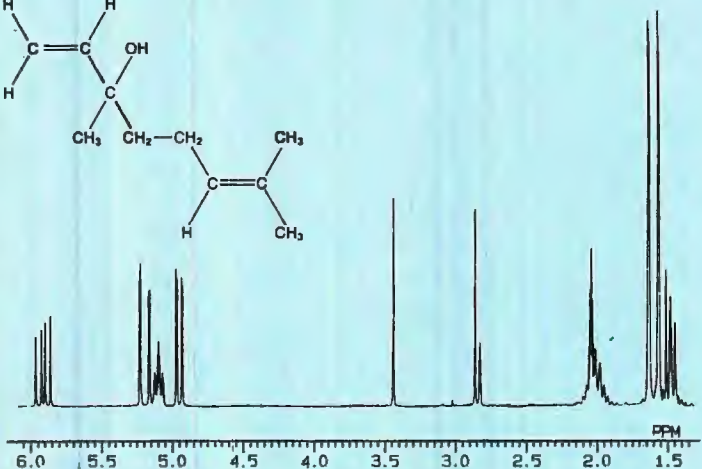
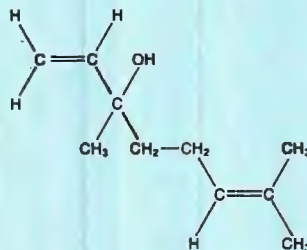
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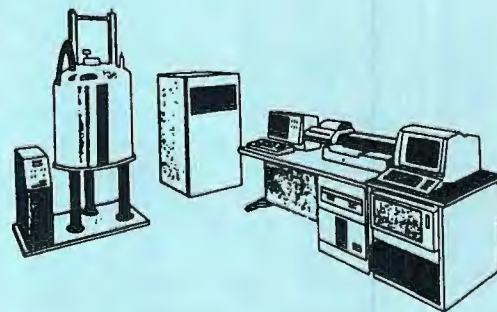
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