

Four-Bond ^{13}C - ^1H Correlations in Aliphatic Molecules	Wray, V.	2
Nuclear Spin Cross-Relaxation in Metal Hydrides	Baker, D. B., Conradi, M. S., and Norberg, R. E.	7
Spectroscopy of Tree Seed Embryos	Amos, L. W.	9
^{13}C NMR Characterization of Cellulose Acetates	Buchanan, C. M.	15
Beware Those Intensity Differences	Goux, W. J.	17
Copper(II) Coordination to Aminosugar Derivatives	van Haveren, J., and Peters, J. A.	23
A Five-Legged Mouse for a Bruker A3000	Briguet, A., Duplan, J. C., Lapray, C., and Pimmel, P.	25
Natural Abundance ^{15}N Studies	Consonni, R., Molinari, H., and Zetta, L.	31
^{31}P NMR Studies of the Action of Dantrolene on Halothane-Membrane Interactions	Gaillard, S., DuFourc, E. J., and Renou, J. P.	33
Peptide Spectrum Simulation Program	Bortmann, P., and Saudek, V.	39
Organoderivatives of the Main Group Elements and Their Precursors	Watkins, C. L., and Krannich, L. K.	41
Enol-Keto Forms in Certain 3-Oxotetrahydrothiapyrans	Berlin, K. D.	42
Obituary - Professor Jean Delmau	Briguet, A.	47
Position Available	Swayne, S.	48
Position Available	Wright, P. E.	48
Jim Shoolery the Mentor	Tschudin, R.	49
Neighbouring Nuclei	Granger, P.	50
TAMU NMR Newsletter: Sundry Notices	Shapiro, B. L.	54
TAMU NMR Newsletter: <u>Policies and Practical Considerations</u> (slightly revised)	Shapiro, B. L.	55

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D-130	Chloroform-d	CDCl ₃	99.8%	1.50	-64	62	0.611
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NO. 386, NOVEMBER 1990

AUTHOR INDEX

Amos, L. W. 9	DuFourc, E. J. 33	Lapray, C. 25	Shapiro, B. L. 54, 55
Baker, D. B. 7	Duplan, J. C. 25	Molinari, H. 31	Swayne, S. 48
Berlin, K. D. 42	Gaillard, S. 33	Norberg, R. E. 7	Tschudin, R. 49
Bortmann, P. 39	Goux, W. J. 17	Peters, J. A. 23	Watkins, C. L. 41
Briguet, A. 25, 47	Granger, P. 50	Pimmel, P. 25	Wray, V. 2
Buchanan, C. M. 15	van Haveren, J. 23	Renou, J. P. 33	Wright, P. E. 48
Conradi, M. S. 7	Krannich, L. K. 41	Saudek, V. 39	Zetta, L. 31
Consonni, R. 31			

TEXAS A&M NMR NEWSLETTER

NO. 386, NOVEMBER 1990

ADVERTISER INDEX

American Microwave Technology, Inc. 5	International Equipment Trading, Ltd. 51
Bio-Rad, Sadtler Division 21	JEOL outside back cover
Bruker Instruments, Inc. 3	MR Resources, Inc. 29
Cambridge Isotope Laboratories 45	Norell, Inc. 37
Chemagnetics 43	Polyflon Company 35
Doty Scientific, Inc. 27	Varian 11
Fremont Magnetic Resonance 13	Volttronics Corporation 53
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FORTHCOMING NMR MEETINGS

Advanced Tomographic Imaging Methods for the Analysis of Materials, Symposium at the Fall Meeting of the Materials Research Society, Boston, Mass., Nov. 26 - Dec. 1, 1990; See Newsletter 378, 57.

In Vivo Magnetic Resonance Spectroscopy Tutorial and Participatory Workshop, St. Louis, Missouri, April 4 - 7, 1991; See Newsletter 385, 58.

32nd ENC (Experimental NMR Spectroscopy Conference), St. Louis, Missouri, April 7 - 11, 1991; Contact: ENC, 750 Audubon, East Lansing, MI 48823; (517) 332-3667.

1991 Keystone Symposia on Molecular & Cellular Biology, Keystone, Colorado: April 8-14, 1991, *Frontiers of NMR in Molecular Biology; Proteolysis in Regulation and Disease; Protein Folding, Structure and Function*; See Newsletter 384, 46.

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

Additional listings of meetings, etc., are invited.

GBFGesellschaft für
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Strukturforschung

Date: 25.9.90

(received 10/2/90)

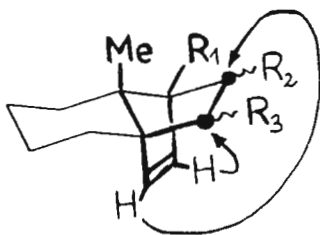
Four-bond ^{13}C - ^1H correlations in aliphatic molecules

Dear Dr. Shapiro,

Although most of the time on our Bruker AM 600 instrument is devoted to the measurement of peptide/protein spectra we have been able to take advantage of the considerable number of new 2D techniques, not available on our other older instruments, for the structure elucidation of small molecules (MW <2000). In particular the use of 2D ^1H -detected direct and long-range ^{13}C - ^1H correlations have proved extremely useful as nearly all spectra are first order and the sensitivity is such that only small amounts of material (few mgs) are required. Data manipulation is particularly easy on our data station where the use of the "skyline" correction routine of the "Aurelia" program (written by Dr. P. Neidig, Bruker, Karlsruhe) has proved invaluable for removing ridges associated with strong signals in such spectra.

In our experience the ^1H -detected long-range correlations are far more helpful than the corresponding ^{13}C -detected COLOC types of correlation in that most if not all, two and three bond correlations can be observed in one experiment. As four-bond heteronuclear couplings are usually zero in aliphatic compounds it is implicitly assumed that such correlations will not be observed. Hence we were initially somewhat perturbed when we had to invoke two four-bond correlations in the structure elucidation of a series of compounds in work undertaken in collaboration with Professor Winterfeld, University of Hannover, FRG. After a careful analysis of the 1D and 2D

^1H and ^{13}C data it was possible to unambiguously assign these and we were left with distinct correlations corresponding to the couplings shown in the figure. With a knowledge of the structure it is perhaps now not so surprising as several mechanisms could contribute to the coupling. Thus there are several bonding pathways between the coupled nuclei and, by analogy with long-range ^1H - ^1H coupling, both are allylic couplings. The angle subtended by the C-C single bond to the plane of the double bond is near 90° , the optimum for such couplings.

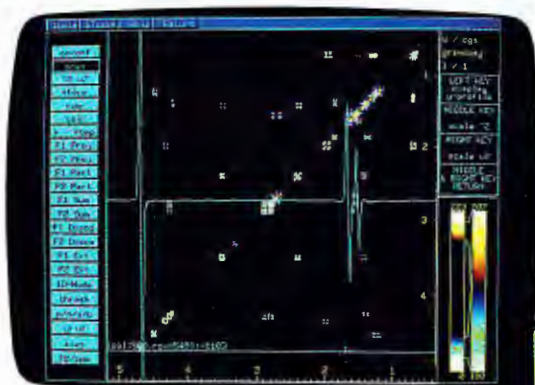


It would be interesting to know what other structural features can lead to detectable four-bond heteronuclear couplings.

Yours sincerely,

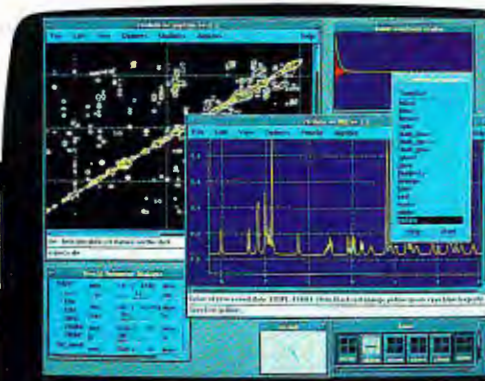
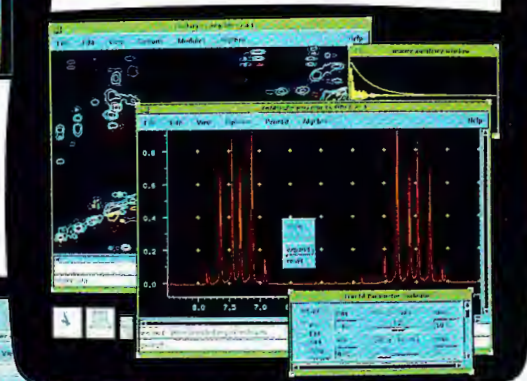
Victor Wray
(Dr.) Victor Wray

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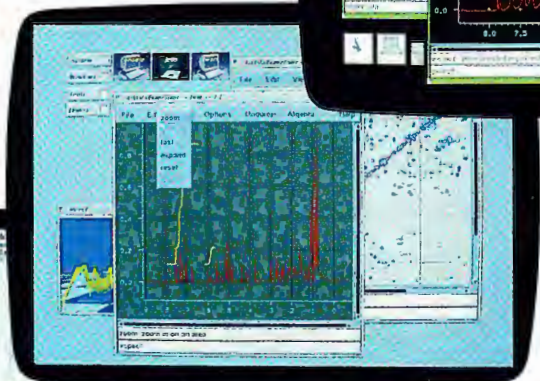


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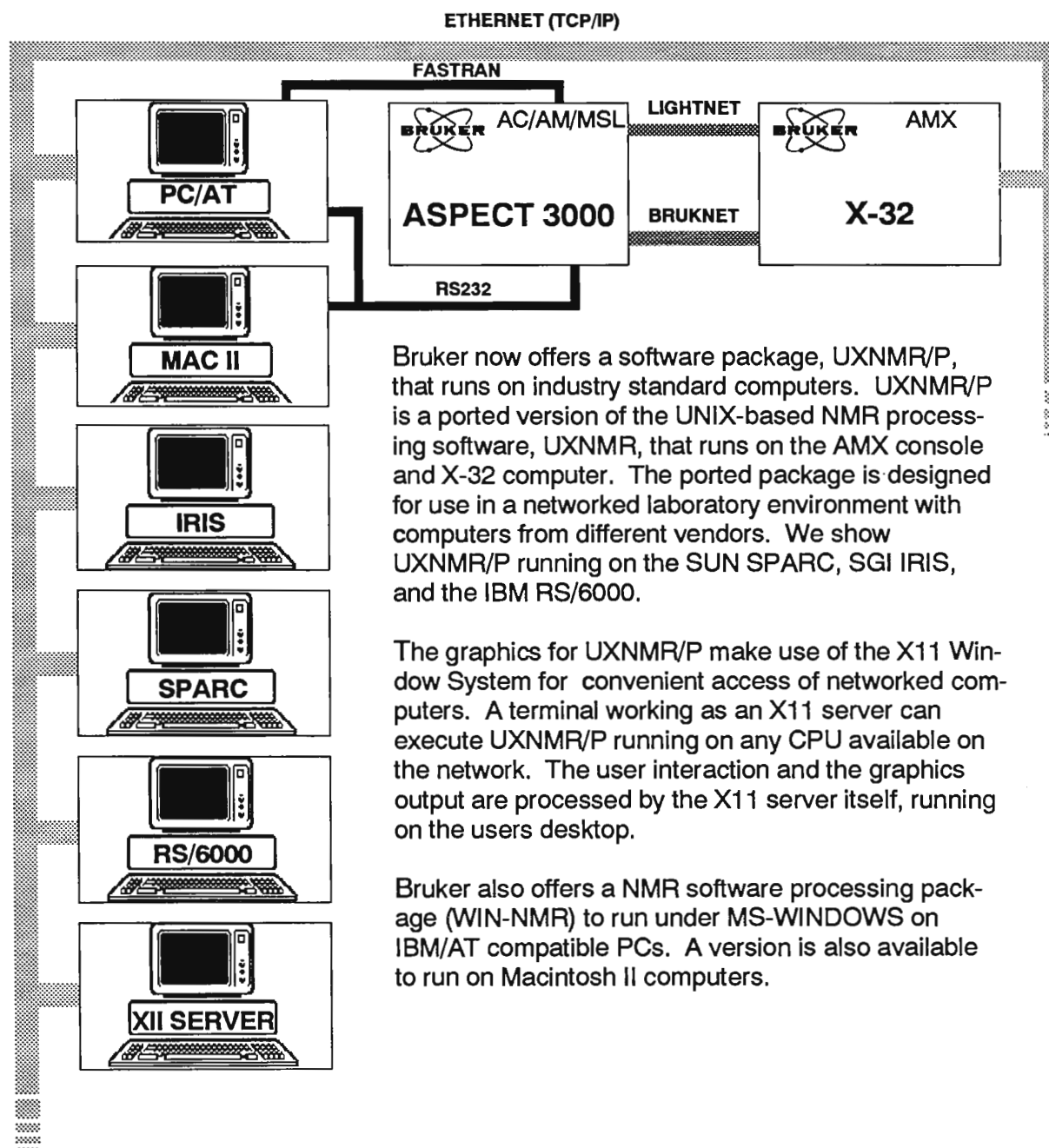


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The graphics for UXNMR/P make use of the X11 Window System for convenient access of networked computers. A terminal working as an X11 server can execute UXNMR/P running on any CPU available on the network. The user interaction and the graphics output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

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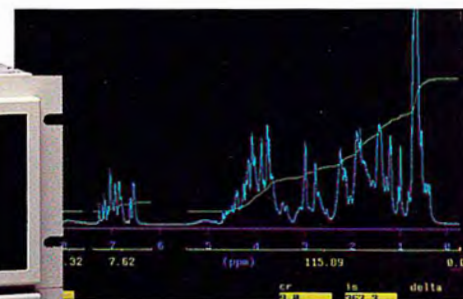
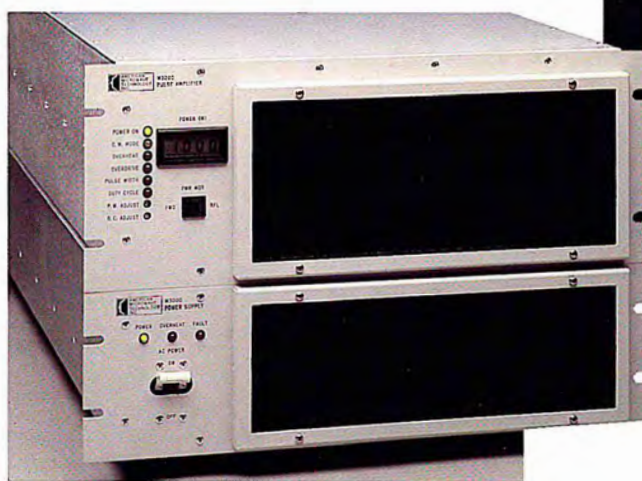
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Department of Physics

September 12, 1990
(received 9/15/90)

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

NUCLEAR SPIN CROSS-RELAXATION IN METAL HYDRIDES

Dear Dr. Shapiro:

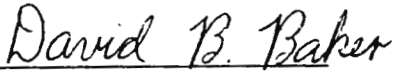
We recently examined a proposed cross-relaxation model involving protons and metal nuclei in metal hydrides.¹ In the proposed model, protons cross-relax to quadrupolar metal nuclei (many metal nuclei have $I > 1/2$) via the proton-metal dipolar interaction. The metal nuclei subsequently relax to the lattice via conduction electrons (Korringa mechanism). *Only* those few metal nuclei ($\sim 10^{-3}$) with resonance frequencies matching the protons (to within the proton dipolar linewidth, ~ 50 kHz) may participate in the cross-relaxation and serve as *relaxation centers* for the proton system. Local disorder of the hydrogen locations near the metal nuclei provides a distribution of electric field gradients (EFG) which inhomogeneously broadens the metal resonance (~ 100 MHz wide in Ta). Without these quadrupole effects, overlap of the proton and metal resonances would not occur ($\gamma_{\text{proton}} \gg \gamma_{\text{metal}}$).

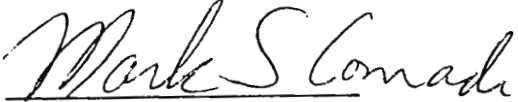
The experiments which test the cross-relaxation model involve ac field modulation and sample rotation. In the field modulation experiments the proton resonance is periodically varied by applying an ac magnetic field (~ 250 G p-p, at 10 kHz). The modulation of the proton frequency brings an increased number of metal nuclei into contact with the protons. In the sample rotation experiments (with axis perpendicular to field) the quadrupolar metal nuclear frequencies are varied periodically by changing the orientation of the EFG (which are fixed within the sample) with respect to the external magnetic field. Again increased spectral (frequency) overlap occurs which increases the proton relaxation rate T_1^{-1} .

The results are presented in Figs. 1 and 2. The striking effects are unambiguous evidence of the cross-relaxation. Note the factor ~ 50 increase in Fig. 2 with 5 Hz spinning.

¹L. R. Lichty, J.-W. Han, D. R. Torgeson, R. G. Barnes, and E. F. W. Seymour,
Phys. Rev. B (accepted for publication).

Sincerely yours,


David B. Baker


Mark S. Conradi


R. E. Norberg

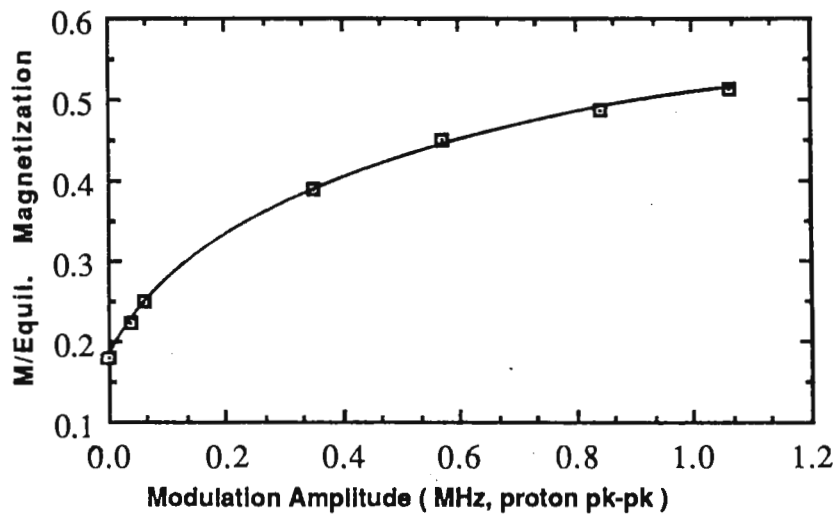


Fig.1: Fraction of recovered magnetization M/M_0 as a function of field modulation amplitude at a fixed waiting time.

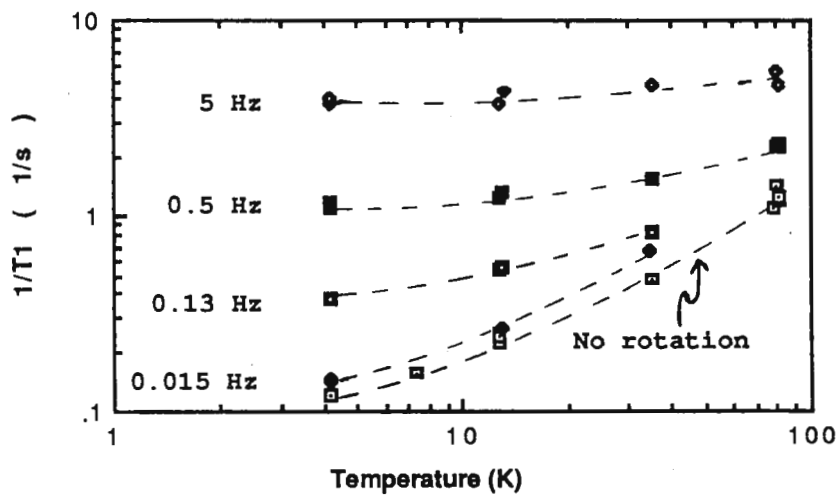


Fig.2: Proton relaxation rate T_1^{-1} as a function of temperature at several sample rotation frequencies. Note the factor of ~ 50 increase upon 5 Hz rotation at 4.2 K.



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September 12, 1990
(received 9/15/90)

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

SPECTROSCOPY OF TREE SEED EMBRYOS

Dear Dr. Shapiro:

Thanks for the reminder. This and the Ultimatum will cross in the mail; also, you can expect the subscription renewal shortly.

Among my recent labors have been NMR examinations of tree embryos, that portion of seeds which sprout into plants when everything works right. These explorations are expected to yield some indications of the types and relative amounts of components, those of interest to our biologists being lipid, protein and carbohydrate.

Two carbon experiments have proven useful so far (attached). The top spectrum is a direct-polarization 25-MHz ^{13}C NMR spectrum with MAS at 2200 rps. This experiment discriminates in favor of liquid-like components, and the sharp signals in this spectrum, readily assigned to fatty acid triglycerides, represent lipid. A very broad component centered at 70-80 ppm also appears, in amounts varying with embryo source; it probably arises from carbohydrates.

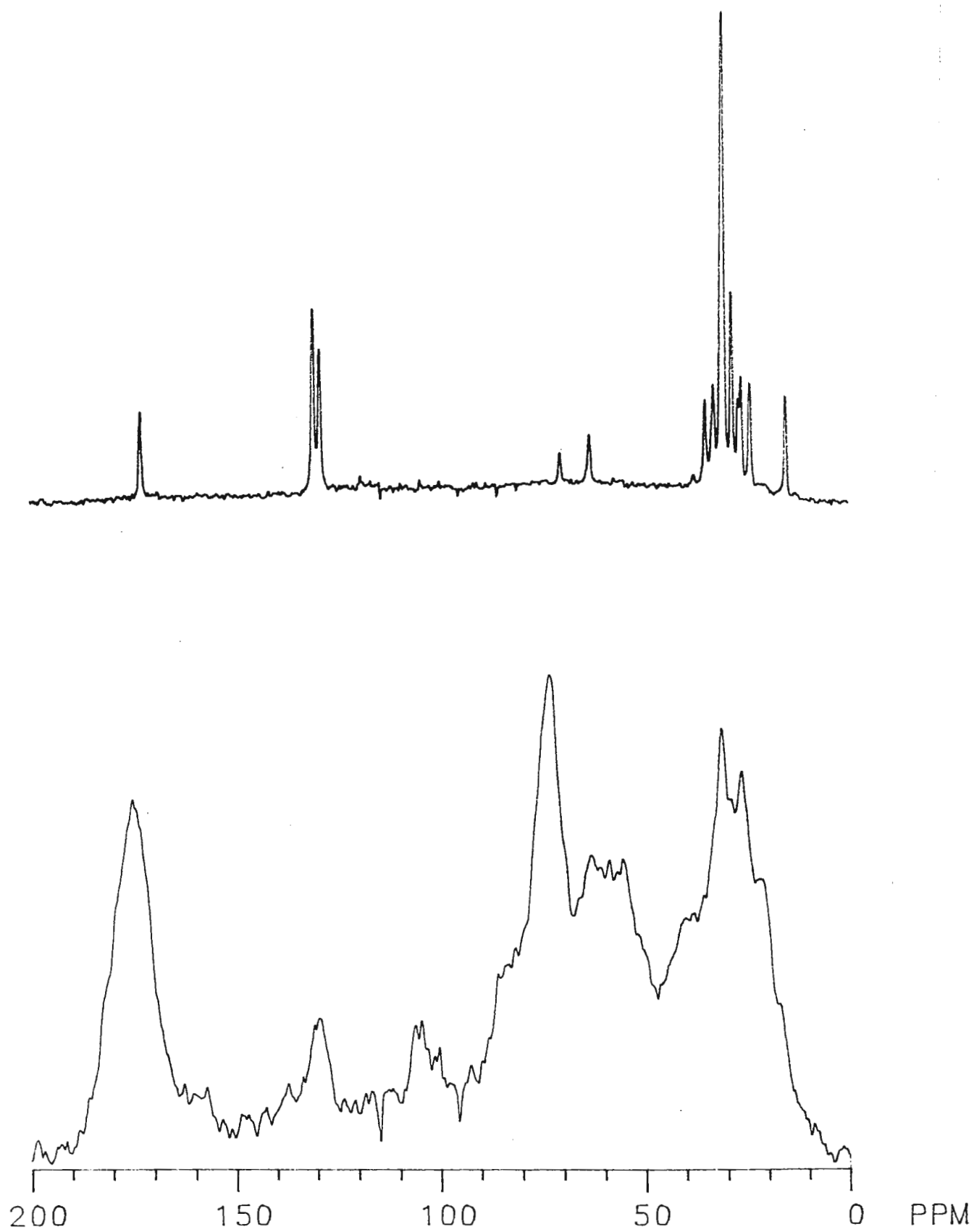
The bottom spectrum is the 25-MHz CP/MAS spectrum of the same sample, also at 2200 rps, and reveals the more rigid, solid-like protein and polysaccharide materials. Note the apparent absence of lipid signals in this spectrum which vividly demonstrates the differences in the DP and CP experiments. Results to date indicate that relative amounts of lipid, protein and carbohydrate vary widely in embryos of different source. We are considering trying microimaging to determine where these components are located in the embryo.

Our forestry biologists prepare these samples by excising the embryos from a few hundred seeds yielding 50-300 mg for an NMR sample. The DP and CP experiments are run back-to-back at night. An occasional nuisance is the expulsion of lipid from high-lipid samples, which finds its way out of the rotor, greases the inside of the probe, and requires cleaning.

Hoping this will sustain my standing with the Newsletter, I am

Sincerely yours,

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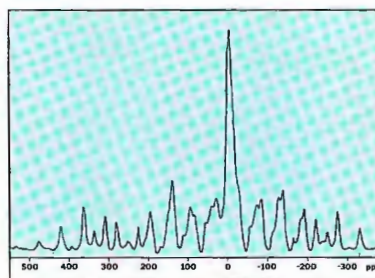
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NMR WITH A FUTURE

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SPIN 1 STATIC POWDER PATTERN SIMULATION

Varian's new Solids Analysis Software for UNITY spectrometers allows both the simulation and fitting of solid-state NMR spectra. The FID for a spin 1 quadrupolar nucleus with a non-zero electric field gradient is calculated by summing FID's from each orientation in a powder. The Solids Analysis Software requires as input the quadrupolar coupling constant, asymmetry parameter, and the 90° pulse width. One of the attributes of this simulation program is the ability to allow for the effects of finite

pulse width, as seen in the spectra below (pulse widths from left to right are 1 μsec , 2 μsec , 3 μsec , 4 μsec , and 5 μsec). If the 90° pulse length is short (ie, 1 to 2 μsecs) there is little, if any, noticeable distortion. When pulse lengths are longer, however, there are noticeable distortions that this program takes into account when calculating the FID. For these simulations, the value of the quadrupolar coupling constant was 180 KHz; the value of the asymmetry parameter was 0.





Fremont Magnetic Resonance

3315 Seldon Court
Fremont CA 94539
(415)623-0722
(415)623-0851 FAX

Felix / PC

NMR Data Processing Software

FMR cooperates with Hare Research in providing the NMR community with Felix/PC (tm) NMR data processing software and software utilities for IBM compatible PCs. The software is available in either a 1D or Multi-D package. Felix/PC is a "toolbox" of NMR data processing routines which allow the operator to perform all common and many unusual processing functions. It is an extremely powerful processing package rivaling many packages on "more powerful" computers.

1D Package:

- Full range of apodization routines.
- Forward and reverse transforms.
- On screen "real time" phasing, expansion and difference routines.
- Several types of baseline correction routines.
- Automatic and manual peak picking and labeling.
- Total spectrum and "broken" integration routines.
- 1D data table sizes up to 64K words.
- Complete macro functions.
- "Locate" menuing system.
- Graphics support for HPGL and Postscript.

1D / 2D Package:

- Process up to 4 dimensional without transposition.
- 1D data table sizes up to 32K words.
- 2D data table sizes up to 2K x 2K.
- Color coded contour displays and plots.

Felix/PC requires a 100% compatible IBM PC computer (8088, 8086, 80286 or 80386) with an with 640 K of memory, a 80x87 coprocessor, a hard disk and an IBM compatible CGA, EGA or VGA graphics adapter.

If Felix/PC is to be used with data from NMR spectrometers, data format translation is required. Data format translation is the responsibility of the buyer. Data format translation software is available as a separate purchase.

Felix software is also available for other computers such as SUN and IRIS systems. Felix/PC and Felix is available from Hare Research for only a small handling charge (\$150.00) to all academic and government institutions. Demo software packages are available.

Data Translation Software.

Both Nicolet/GE and Bruker provide Kermit and X-Modem data transfer software for their spectrometers. This software can easily communicate with a PC running any one of the many software communication packages using Kermit or X-Modem transfer protocol at transfer speeds up to 38K Baud.

Once the data is transferred using X-Modem or Kermit protocols, with FMR's GOSSIP or by any other means, data translation software packages are available to convert the Nicolet/GE 1280 20 bit word or the Bruker Aspect 24 bit word into floating point Felix/PC words. Key parameters are also converted from the Nicolet/GE and Bruker integer and floating point header parameters into the respective file headers for Felix/PC:

- Spectrometer Frequency.
- Sweep Width.
- Data Table Size.
- Non-Quadrature / Quadrature Data.

Some Varian conversions are available from other vendors. The capability to process several manufacturers' data with a single software package can make life in a mixed instrument laboratory easier for many users.

Parallel port 1280 to PC transfers are in development.

BXR Data Transfer & Translation Software

BXR is a set of programs that transfers data files from the Bruker Aspect computers to PC computers. BXR stores the data in translated files that Felix/PC can read. Parameters related to data processing are transferred for use by Felix. Transfer rates of up to 19200 baud are usually routine (> 100 KBytes per minute). In normal operation the PC and the Aspect are connected with a communication cable and the BXR transfer program started on the PC. The unattended PC then waits for files to be transferred by the Aspect. There is no need to halt the PC program. You can start and stop the transfer program on the Aspect without stopping and restarting BXR on the PC. Under this condition, the PC waits for additional files from the Aspect until you halt it. This is for convenient data transfers to an unattended PC.



Fremont Magnetic Resonance

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GOSSIP

GOSSIP is a new two-way data transfer program written by FMR for data transfer between IBM compatible PC computers and the Nicolet/GE 1280. Data transfers can be done at rates up to 38.4K baud (about 700 Nicolet Words per second with overhead). This function gives the 1280 user a new spectrum of capabilities:

Inexpensive mass data storage. Once the data is on the PC, large capacity, inexpensive and reliable magnetic and optical disks and tape backups are abundant. 330 MByte disks sell for as little as \$2000. This makes long term data backups and personal spectral archives practical.

Many alternate NMR data processing packages are available for the PC. When GOSSIP is combined with data translation software and one of these data processing packages, convenient and inexpensive desktop NMR processing becomes possible.

The processed data is immediately available for direct incorporation into many popular **word processing and desktop publishing** packages.

TMON

With the TMON operating system for the 1280, transfers to the PC can be done in two ways.

FILTRN - RS-232 transfers can be done using the Nicolet/GE FILTRN program from the TMON operating system to GOSSIP on the PC. This can be done at rates up to 38.4K baud (about 700 Nicolet Words per second with overhead).

NMR Programs (QE, GN and NT) - Transfers can be done from inside the NMR programs. These programs support foreground and background RS-232 transfers at rates up to 38.4K baud (about 700 Nicolet Words per second with overhead). These transfers can be automated under MACRO control of the 1280 so that when the experiment is finished the data is automatically transferred. Overnight and/or sample changer operations can automatically store copies of the data on a waiting PC.

DEXTER

With the DEXTER operating system for the 1280, transfers to the PC can be done in two ways.

FILTRN - RS-232 transfers can be done using the Nicolet/GE FILTRN program from the DEXTER operating system to GOSSIP on the PC. This can be done at rates up to 38.4K baud (about 700 Nicolet Words per second with overhead).

NMR Programs (NT) - FMR provides a package of software which includes an *overlay for the 1280 NMR program* and the GOSSIP PC program. With this package transfers can be done from inside the NMR program IN BACKGROUND and at rates up to 38.4K baud (about 700 Nicolet Words per second with overhead).



Professor Barry Shapiro
966 Elsinore Ct.
Palo Alto, CA 94303

October 1, 1990
(received 10/5/90)

Dear Prof. Shapiro:

Water soluble and water absorbant polymers are extremely important commercial products. Despite being known since 1929, cellulose esters with an affinity for water, as typified by water soluble cellulose monoacetate (CMA), are conspicuously absent from the list of commercially available water soluble and water absorbant polymers. There are many reasons for the lack of commercial exploitation of this biodegradable and renewable resource as a source of water soluble polymer. One reason is that the analytical methods suitable for detailed characterization of CMA have not been available. Recognizing this problem, Kamide and others have devoted significant research effort to this rather formidable problem. Based on their work, it is proposed that the two basic requirements for water soluble CMA are (i) that the degree of substitution (DS, the number of substituents per anhydroglucose ring where the maximum DS is 3) be in the range of 0.5-1.1 and (ii) that the relative degree of substitution (RDS) among the three possible substitution sites be roughly equal.

To address the analytical problems, we have prepared and characterization by NMR spectroscopy (COSY, INAPT) a series of cellulose acetates labeled at the carbonyl carbons with carbon 13. We identified a total of 16 carbonyl carbon resonances which were assigned to carbonyls attached to either a C2, C3, or a C6 hydroxyl (Table 1). In the case of the C2 and C3 carbonyls, the carbonyl resonances were assigned to specific monomers in the polymer backbone. Although we are limited by resolution and by our inability to assign all of the C6 carbonyl resonances, we have found the assignments to be invaluable in addressing structure-property relationships of CMA.

When we applied these assignments to CMA prepared by metal catalyzed, carboxylic acid promoted, and aqueous hydrolysis (Table 2), we found that the principle differences in monomer composition between water absorbant CMA and water soluble CMA are the relative ratios of the 3-monoacetyl monomer to the 2,3-di, the 3-tri, and/or total 3-substituted monomer. The ratio of 3-monoacetyl monomer DS to total DS also provides a clear distinction between water soluble and water absorbant CMA. In general, we find that for water absorbant CMA: $(3-m/2,3-di) < 0.5$, $(3-m/3-tri) < 1.1$, $(3-m/total\ 3\ acetyl) < 0.25$. For water soluble CMA, the inequalities are reversed. For water absorbant CMA, the ratio of DS due to the 3-mono monomer to the total DS is generally 0.11 or less whereas for water soluble CMA this ratio is greater than 0.11. These data show that the structural requirement, as expressed in terms of monomer composition, for water solubility is that the number of 3-monoacetyl monomer repeat units be greater than 10-11 per 100 monomers. It is very easy to focus only on the acetyl substituents and neglect the unsubstituted hydroxyls. However, the ratio of 3-monoacetyl monomer to the remaining 3-acetyl monomers clearly illustrates the importance of the C2 hydroxyl to water solubility. In conjunction with current knowledge of cellulose crystal structure, we feel that the data suggest a minimum level of 3-acetyl substituents must be present for disruption of intra- and/or intermolecular hydrogen bonding while still maintaining a delicate balance of C2 hydroxyl to insure the hydrophilicity of the polymer

backbone. Apparently the C6 hydroxyl is too remote from the central axis of the polymer backbone to have a significant hydrophilic influence and, as such, can only exert a negative influence on water solubility of CMA. (Please credit this contribution to the account of Eastman Chemical Company.)

With Best Regards



Charles M. Buchanan
Senior Research Chemist
ECC Research Laboratories
P. O. Box 1972

Table 1. Chemical shifts for the carbonyl resonances of CA.

Carbonyl Chemical Shift (ppm) ^a	Monomer
170.01 (0.01, 0.02)	6
169.92 (0.01, 0.02, 0.03)	6-triacetyl
169.83 (0.01, 0.02, 0.03)	6
169.78 (0.01)	6
169.56	3-monoacetyl*
169.48 (0.01, 0.02, 0.04)	3-monoacetyl
169.44	2,3-diacetyl*
169.36 (0.01, 0.02, 0.03)	2,3-diacetyl
169.25	3,6-diacetyl*
169.14	3-triacetyl*
169.13 (0.06)	3,6-diacetyl
169.05 (0.02, 0.03, 0.05)	3-triacetyl
168.83 (0.01, 0.02)	2,3-diacetyl
168.72 (0.01)	2-triacetyl
168.63 (0.01)	2-monoacetyl 2,6-diacetyl

(a) The chemical shifts that are given are for the first observed resonance(s) for that monomer. The numbers given in the brackets gives the downfield shift for that resonance with increasing hydroxyl level.

* This resonance has been shifted by hydrogen bonding

Table 2. Ratio of 3 acetyls for CMA.

	3-m/2,3-di	3-m/3-tri	3-m/total 3 acetyl	3-m DS/DS
Absorbs Water				
1	0.28	1.58	0.19	0.11
2	0.22	0.47	0.13	0.06
3	0.00	0.00	0.00	0.00
4	0.30	1.00	0.18	0.18
5	0.46	1.19	0.24	0.11
Water Soluble				
6	0.86	2.37	0.36	0.20
7	0.54	∞	0.35	0.19
8	0.61	1.10	0.26	0.10
9	1.00	1.45	0.33	0.15
10	1.20	1.30	0.32	0.13



THE UNIVERSITY OF TEXAS AT DALLAS

BOX 830688 RICHARDSON, TEXAS 75083-0688

October 2, 1990 (received 10/5/90)

Dr. Barry Shapiro
Texas A&M Newsletter
966 Elsinore Court
Palo Alto, CA 94303

Beware Those Intensity Differences

Dear Barry,

I am writing you near the eve of the due date of the dreaded ultimatum. Let me first open with a statement "life never ceases to amaze me" and then go on to present some "simple" 1D ^{13}C NMR spectra for which I invite explanations.

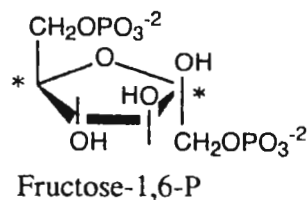
One of our goals is to scale up an enzymatic synthesis of ^{13}C labeled sugars from simpler three atom precursors. A first step is to prepare doubly labeled ^{13}C fructose-1,6-diphosphate from ^{13}C pyruvate using a reverse glycolysis pathway (I promise no more biochemistry). A convenient way of checking our yield is by ^{13}C NMR. We desire not to attenuate any of the labeled carbon resonances in the spectra (so as to get a good estimate of yield) and at the same time acquire the spectra in a reasonable short period of time (so as to set aside more time to play with 2D pulse sequences for *other more challenging problems*). Recently we have been preparing [2,5- ^{13}C]fructose-1,6-diphosphate from 2- ^{13}C pyruvate. Because this sugar has a phosphate at C6 it can only assume a furanose (5 membered ring) configuration in both the anomeric α and β forms. One of the isotopically labeled carbons, C2 (the anomeric carbon), is nonprotonated while C5 has a single proton attached.

Before running my spectra, I knew the T1 of C2 would be much longer than C5 (although at the time I knew neither) and figured I could compensate for the effects of a short delay by using a short pulse width. I thought I might also run the experiment with the proton decoupler gated on only during acquisition in order to compensate for any NOE differences. Fig. A shows the spectrum of a reaction mixture run on a GN-500 using a 20° pulse width and a 5 sec pulse delay. Each of the furanose anomer contributes a resonance for labeled carbons C2 (at about 102 and 105 ppm) and C5 (at about 80.5 and 81.5 ppm). What is striking is that the carbon with the longer relaxation time (C2) is much more intense than C5. Integration shows that C5 is only 0.6 the intensity of C2. Fig. B shows the same sample (with the same spectral parameters) acquired in the presence of full decoupling. Although C5 still appears less intense, integration shows that within experimental error, both signals have the same intensity. C5 is just broader, probably as a result of coupling to the phosphorous two bonds away.

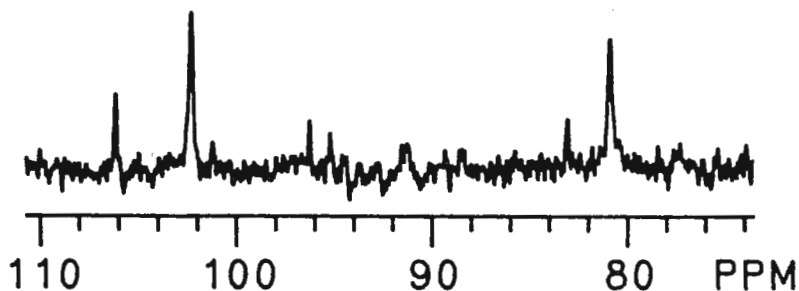
In order to make some sense of all this I measured the T1's of both resonances. C2 had a T1 of about 7 sec and C5 of 0.7 sec. If I carefully waited 5 T1's between acquisitions (35 sec), then both signals had the same intensity either in the presence of full decoupling or gated on during acquisition.

My best guess at what's happening from a "nuts and bolts" approach is that even though in the case of the gated experiment the decoupler is on only a short time, it is enough for C2 to acquire some equilibrium NOE. While C5 also picks up some NOE during this time it quickly decays because of the more efficient relaxation pathways. Does someone have a better explanation ?

A. 5 sec delay. Decoupler gated on during 0.5 sec acquisition time. 20 deg pulse.



B. Same spectral parameters as in A. continuous decoupling.



Sincerely Yours,

Warren J. Goux
Associate Professor of Chemistry

Not even a coffee break.



Seven days a week, twenty-four hours a day. For over three years, a QE automated NMR analytical spectroscopy system ran experiments at Searle Labs in Chicago with only a few hours of downtime.

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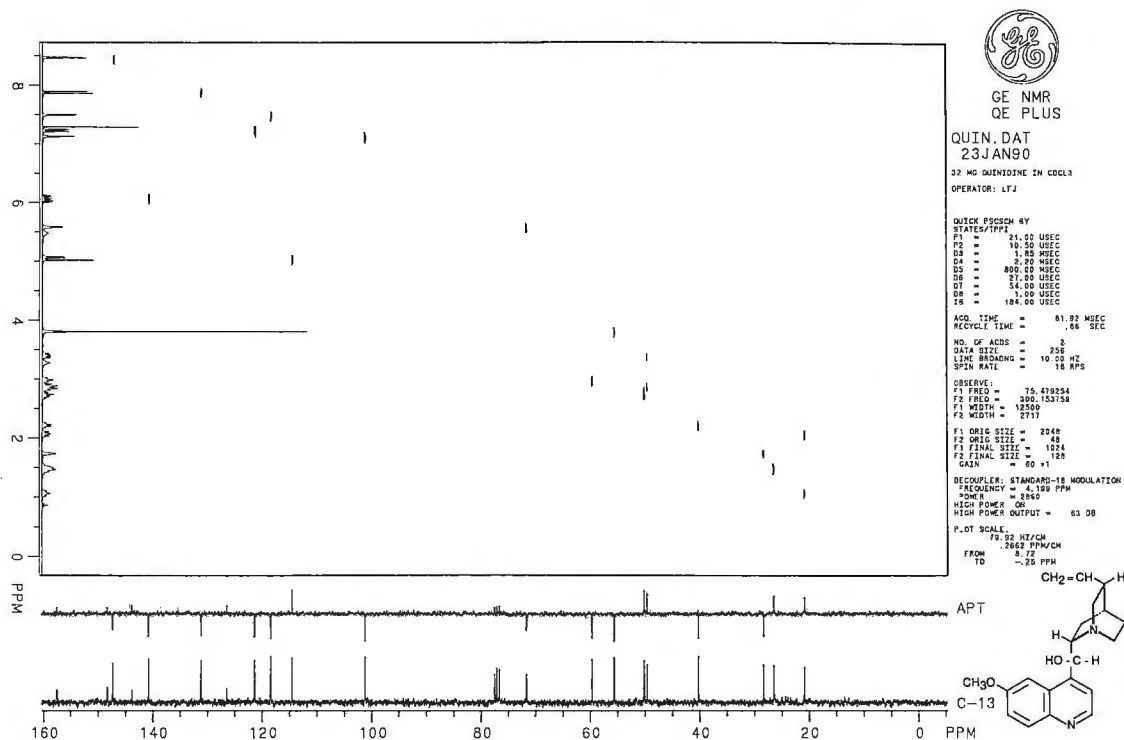
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An automated run using MACRO mode operation on a sample of 32 mg of quinidine in 0.5 ml chloroform-d (0.20M). Data were obtained using the 5 mm broadband probe. ¹H, ¹³C, APT and phase sensitive 2D data were collected, processed and plotted—including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

When faced with a tough analytical problem . . .

QUESTION

How do you find all the thiophenol derivatives matching YOUR CARBON SPECTRUM?

ANSWER

Sadtler Structure Assignment Library

STEP:1

Search For
Thiophenol Derivatives.

Structure Search Version 1.0

CAS Registry Search

Exact Match Search

Substructure Search

Number of Hits: 128

Index	HQ1	Lib	Entry	Name
1	1	13C1	70	PHENYL DISULFIDE
2	1	13C1	79	BENZENESULFONYL CHLORIDE
3	1	13C1	90	BENZENESULFONYL CHLORIDE
4	1	13C1	207	BENZENESULFONYL FLUORIDE
5	1	13C1	208	BENZENESULFONYL FLUORIDE
6	1	13C1	227	P-TOLUENESULFONIC ACID, METHYL ESTER
7	1	13C1	355	P-TOLYL DISULFIDE
8	1	13C1	732	METHANOL, P-TOLYLSULFONYL-
9	1	13C1	982	BENZENESULFONIC ACID, HYDRATE
10	1	13C1	993	BENZENESULFONIC ACID, SODIUM SALT
11	1	13C1	997	P-TOLUENESULFONIC ACID, BUTYL ESTER
12	1	13C1	998	BENZENESULFONAMIDE
13	1	13C1	910	P-TOLUENESULFONAMIDE, N-BUTYL-
14	1	13C1	974	ACETIC ACID, PHENYLTHIO-
15	1	13C1	997	P-TOLUENESULFONYL CHLORIDE
16	1	13C1	1037	P-TOLUENESULFONAMIDE, N-METHYL-
17	1	13C1	1129	ANILINE, O-METHYLTHIO-
18	1	13C1	1178	SULFIDE, PHENYL 3-PHENYLPROPYL
19	1	13C1	1296	SULFIDE, P-BROMOPHENYL METHYL
20	1	13C1	1303	SULFIDE, METHYL N-NITROPHENYL

STEP 2:

Match Those Derivatives
With Your Spectrum.

NMR Search 2.12

File Search View Library Print Utility Import Page

Unknown Entry

PEAK SEARCH PARAMETERS

Peak Tolerance: 0.3 ppm [value between 0.1 and 2.0]

Minimum No. of Peak Match: 4

Algorithm

☐ Forward Search

☒ Reverse Search

Cancel

OK

YOUR SEARCH IS OVER!

NMR Search 2.12

File Search View Library Print Utility Import Page

LIB PEAK LOCATION

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Sadtler ID: ST71

==== First Peak Table ===

(Total of peaks: 5)

No.	Location	Intensity
1	15.60	4
2	124.90	5
3	126.70	5
4	128.70	9
5	138.60	1

Source Library: 13C1 - C-13 NMR STANDARDS

<<Unknown Buffer>>

File Name: THIDEMO.NPF

Chem. Name: ODIFEROUS SPECIES

CAS Registry Number: unknown

Comment: process shutdown \$2/9/89, green

READY

STRUCTURE

Print View

Chemical structure of 3-methylthiophenol (SMILES: CS1=CC=CC=C1)

**... look to SADTLER
for the knowledge to end your search.**

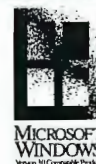
BIO-RAD

**Sadtler
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3316 Spring Garden Street,
Philadelphia, Pennsylvania 19104.

Telephone: (215) 382-7800.

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Sadtler ^{13}C NMR Structure Assignment Library

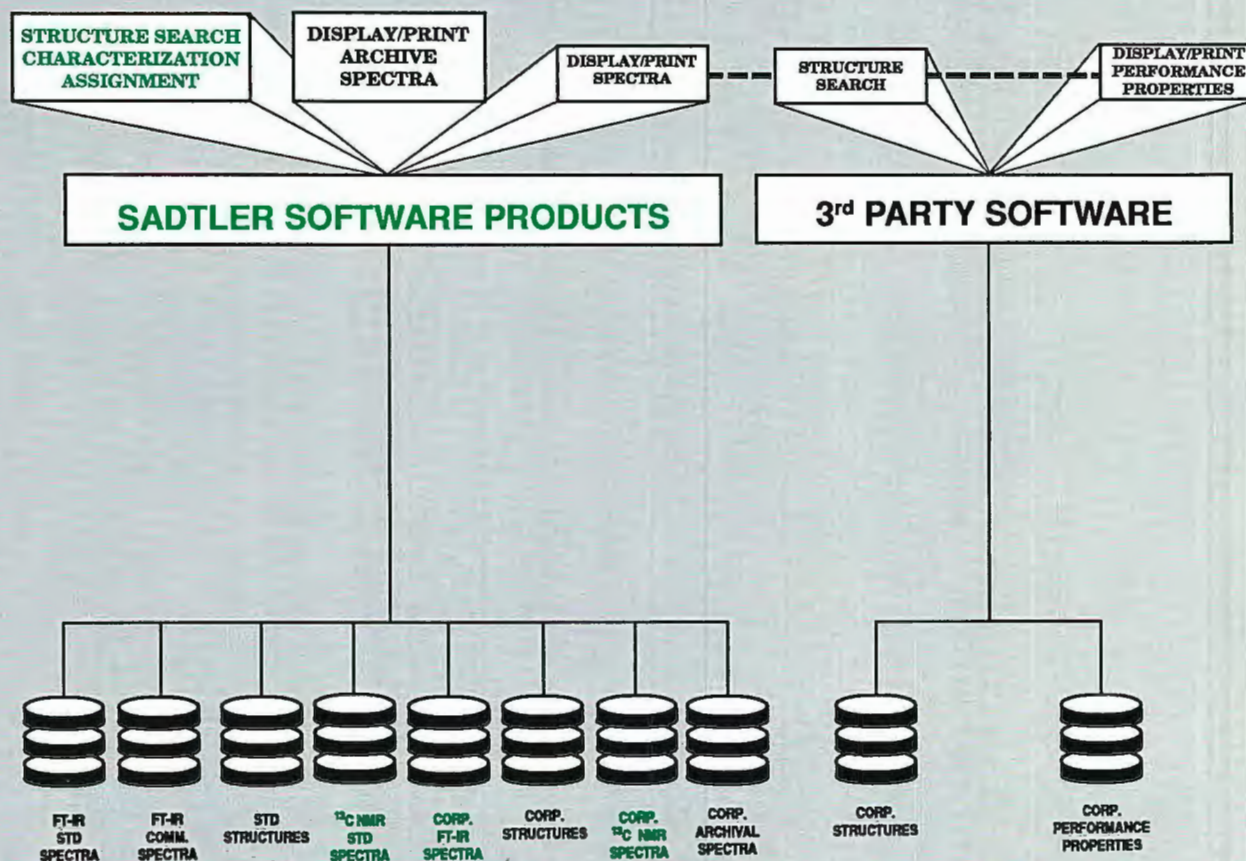
The Sadtler ^{13}C NMR Structure Assignment Library consists of:

The Sadtler Standard ^{13}C NMR Spectra Library
containing entries for 30,000 pure compounds

Sadtler ^{13}C NMR Search Software

Sadtler ^{13}C NMR Molecular Structure Library
(the corresponding 30,000 chemical structures)

Sadtler Molecular Substructure Search Software



The assignment library provides a unique access to Sadtler's ^{13}C NMR spectra that correlates molecular properties with chemical shifts. Structure elucidation is facilitated by searching anticipated structural features and measured chemical shifts.

Dr. B.L. Shapiro
TAMU NMR Newsletter
966 Elsinore Court
PALO ALTO, California 94303
U.S.A.

Your reference and date

Our reference

Office telephone

Date

JAP/mk/90464

(015) 78 5892

September 19, 1990
(received 9/27/90)

Subject

Sub-division

"Copper(II) coordination to aminosugar derivatives"

Dear Dr. Shapiro,

The aminosugar derivatives 2-amino-2-deoxy-D-glucose oxime (**1**) and 2-amino-2-deoxy-D-gluconate (**2**) (Figure 1) are good sequestering agents for transition metal ions. The Cu(II) coordinating abilities are comparable to that of the well-known metal ion sequestering agent D-gluconate.

We have investigated the Cu(II) coordination of **1** and **2** by ^{13}C longitudinal and tranverse relaxation rate measurements. At pH 6.9 and pH 10.7 varying amounts of Cu(II) were added to solutions of **1** or **2**. From the induced longitudinal relaxation rate enhancements (T_{1p}^{-1}) the average Cu(II) distances to the carbon nuclei of **1** and **2** were calculated, using the relations $T_{1p}^{-1} = \rho q T_{1, \text{compl.}}^{-1}$ and $T_{1, \text{compl.}}^{-1} = C \tau_c R^{-6}$. In these formulae ρ represents the ratio of the paramagnetic ion to the ligand, q is the number of the ligands in the coordination sphere, $T_{1, \text{compl.}}^{-1}$ is the relaxation rate of the complex, τ_c is the correlation time of the complex, C is a constant and R is the distance between the Cu(II) and the various carbon nuclei.

The results show that the coordination of the ligands is dependent on the pH. Of **1** only the E-isomer coordinates to Cu(II). At pH 6.9 it binds Cu(II) in a bidentate fashion via the amino and oxime nitrogen. The distances of Cu(II) to the donor sites of the ligand are about equal. At pH 10.7 the oxime oxygen is also bound to Cu(II).

The results for **2** at pH 6.9 (see Figure 2), indicate that in a 1:2 complex the Cu(II) is coordinated via two carboxylate and two amino groups in approximately a square planar geometry with the C3 hydroxyl groups hydrogen bonded to axially coordinated water molecules. At pH 10.7 the ligand is coordinating with the amino and C3 hydroxyl group in a square plane and the carboxylate group in axial position.

These results demonstrate the usefulness of paramagnetic relaxation rates in structural studies of metal ion complexes.

Yours sincerely,

J. van Haveren




J.A. Peters

Reference:

1. A.S. Mildvan and M. Cohn, Adv. Enzymol. 1970, 33, 1.

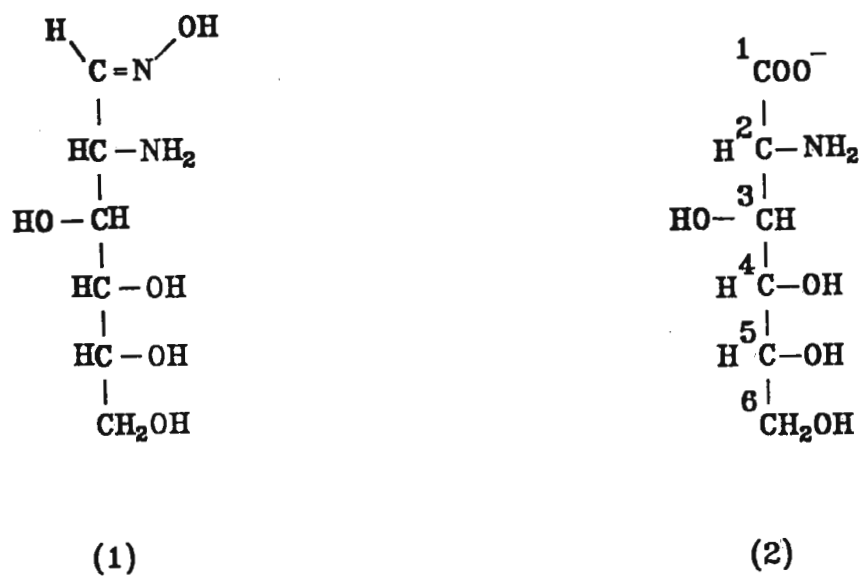


Figure 1: Structures of 2-amino-2-deoxy-D-glucose oxime (1) and 2-amino-2-deoxy-D-gluconate (2)

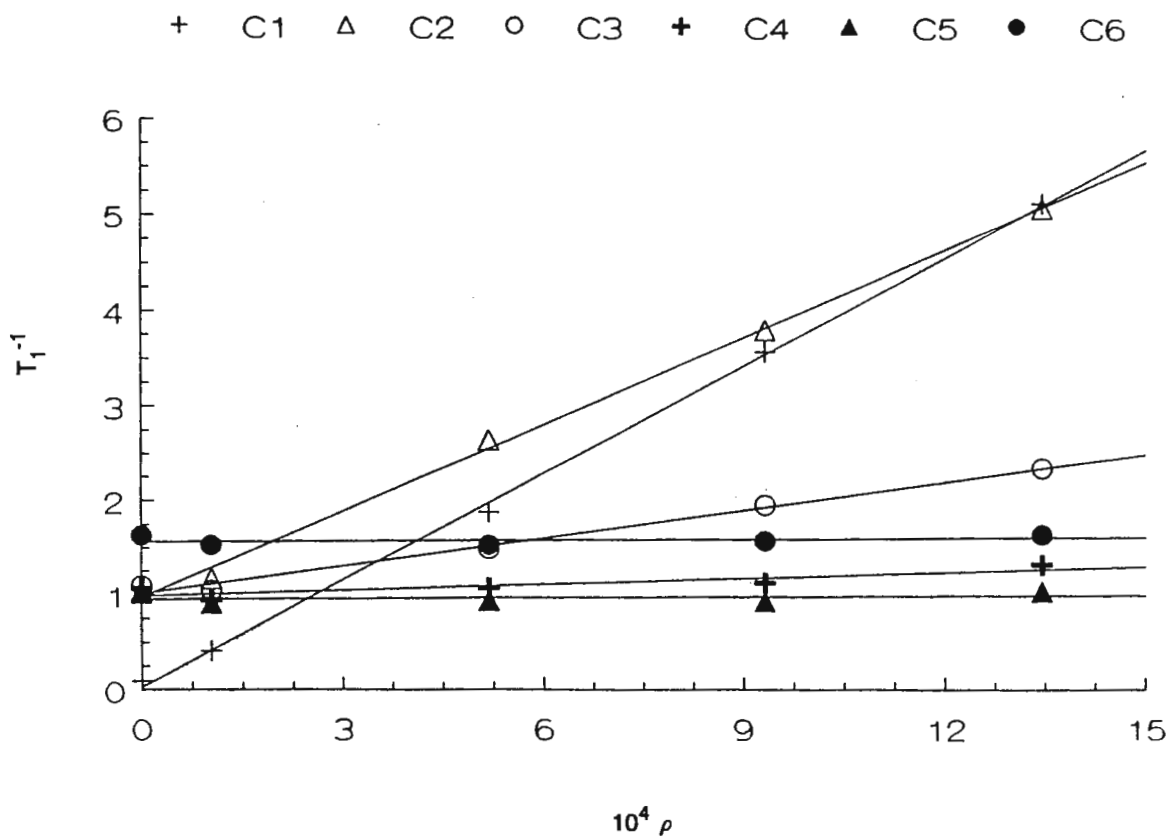


Figure 2: Relaxation rates (sec^{-1}) of 2-amino-2-deoxy-D-gluconate in the presence of varying amounts of Cu(II) at pH 6.9.

UNIVERSITÉ CLAUDE BERNARD LYON I

LABORATOIRE DE RÉSONANCE MAGNÉTIQUE NUCLÉAIRE

Bâtiment 721

43, boulevard du 11 Novembre 1918
69622 VILLEURBANNE Cedex

Tél. 72 44 80 00 (secrétariat poste 33.70)

Villeurbanne le 20 Septembre 1990

(received 9/24/90)

A five leg mouse for A3000

Dear Professor Shapiro,

We apologize for the delay.

We describe here a circuit which replaces a mouse or a track ball for image display with the Bruker A3000 computer and IGDP.

The commands which are necessary to move the cursor are applied through five push buttons: Two are for the X axis, forward and backward, two are for the Y axis, up and down, and the last one is used to modulate the speed of the displacement of the cursor.

The required signals are sent to the computer (port n°5 of the I circuit) through a print I/O port (DB25 type connector). Pinouts are indicated in circled numbers in Figure1. The system works in the following fashion:

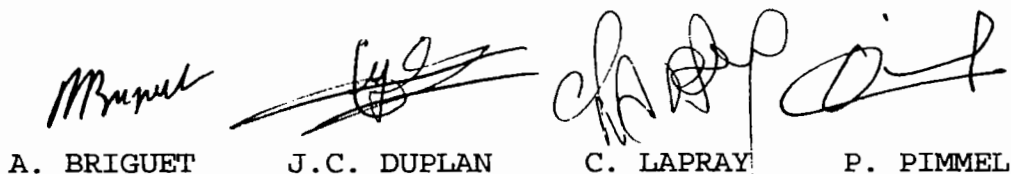
- In the iddle state, **Xaxis**, **Yaxis** and **SRQ** are at low logic level.
- When a push button **X_{AV}** or **X_{AR}** is pressed, the clock signal (from a 555 timer) is enabled to trigger the type D flip-flop. So we get **Xaxis** =1 and **SRQ**=1.

Once the **SRQ** is ON, the computer resets the circuit to the iddle state by sending an acknowledge pulse **ACK** (low level active) , see pin 17. A simple **RS** flip-flop, set by **X_{AV}** or reset by **X_{AR}**, gives the direction of the displacement along the X axis. The Y channel uses the same procedure.

The clock rates have been fixed at 15 Hz for low speed or 130 Hz for rapid motion. The +5V voltage is from the A3000 and it is applied to the pin 24 of the port n°5 of the I circuit. Be careful, if some doubt consult Bruker Services.

This low cost device replaces advantageously the track ball necessary for image processing. Further informations are available on request.

Sincerely yours,

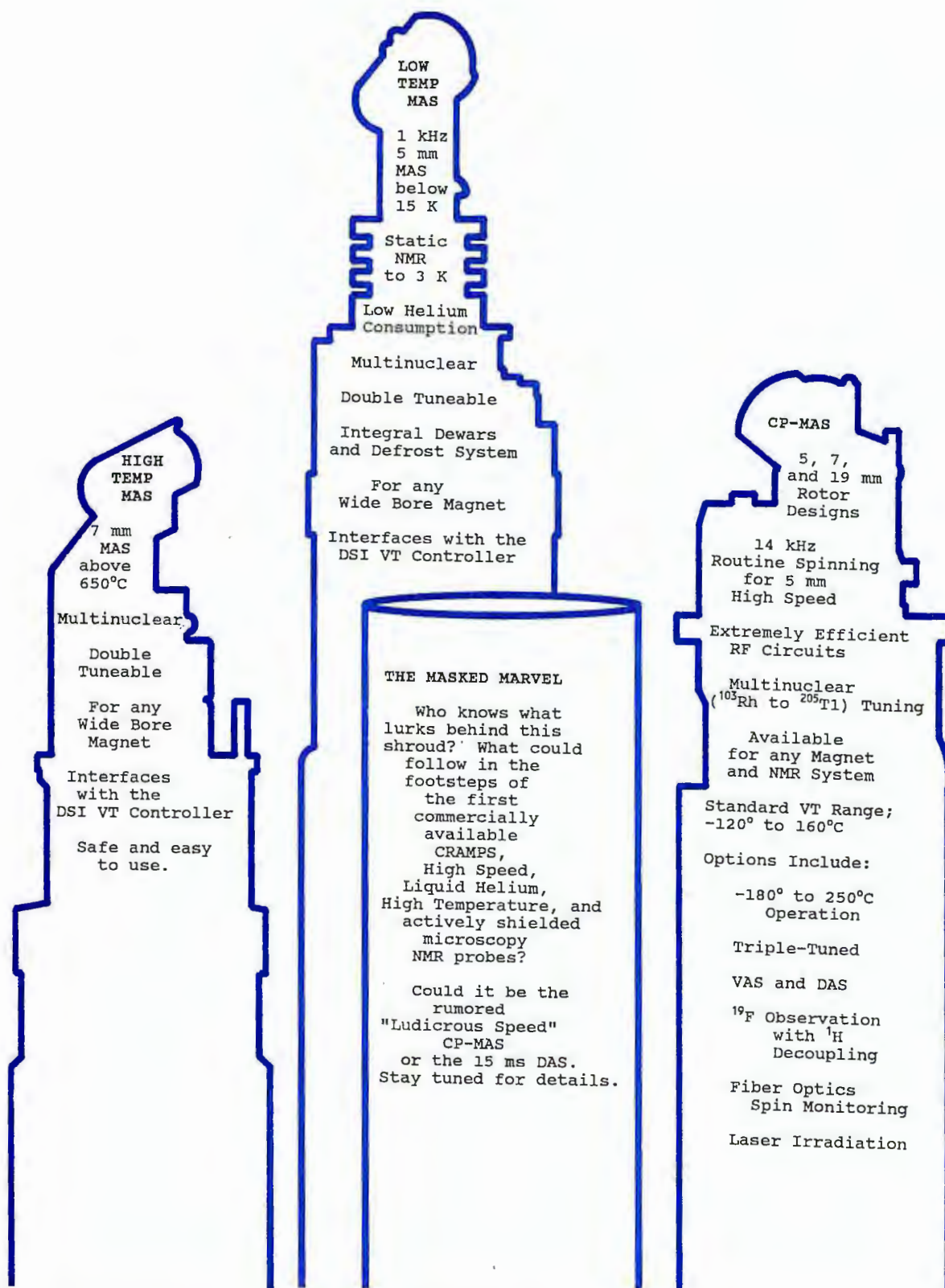


A. BRIGUET J.C. DUPLAN C. LAPRAY P. PIMMEL



Doty Scientific



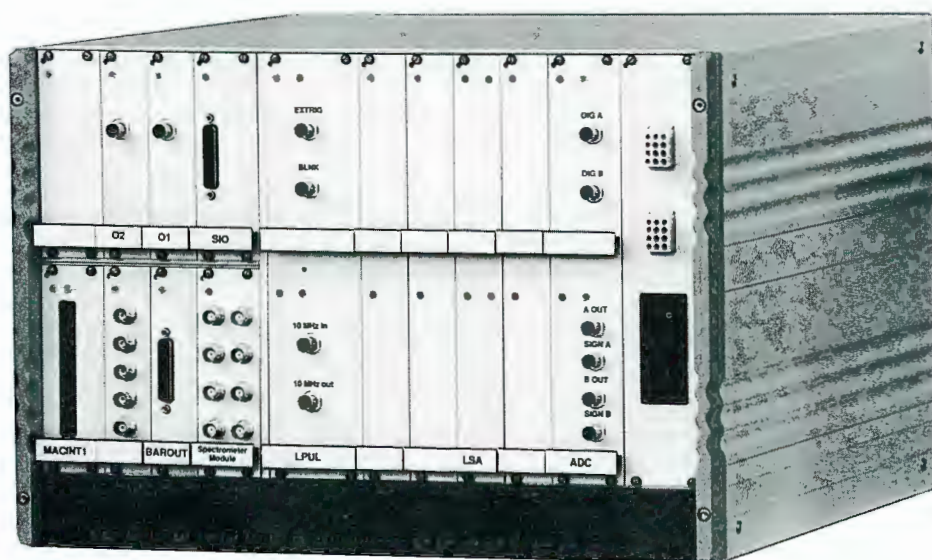


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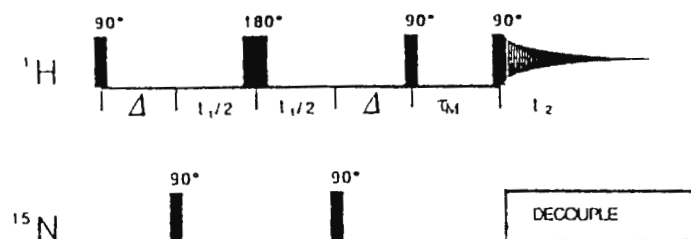
August, 3 1990
(received 10/6/90)

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

NATURAL ABUNDANCE ^{15}N STUDIES

Dear professor Shapiro:

we present here the first example of a natural abundance ^{15}N HMQC-NOE experiment, using reverse detection. This experiment was performed on a linear peptide CTTTNSRGTTT, which has been synthesized as an example of rational drug design. The structural element -NSRG- has been suggested to be recognised by antirabic antibodies, while the six threonines have been introduced in order to stabilize the structure. At the very beginning we carried out ^1H homonuclear NOE and HOHAHA experiments, both at 500 MHz, but the severe overlapping of the NH signals of the six threonines did not let us to assign the individual resonances. Then we performed the reported ^{15}N experiment, first proposed by Opella et al. (1), which combines ^{15}N - ^1H correlation spectroscopy and ^1H homonuclear NOE to give 2D spectra with cross peaks from backbone amide protons to other protons in close spatial proximity. The combined data from ^{15}N spectra and ^1H NOE led us the complete assignment of ^{15}N resonances (fig. 1). The HMQC-NOE experiment (see scheme 1) was performed with 40 mM sample in DMSO- d_6 using the following parameters: mixing time 180 msec, spectral width 5000 Hz in F_2 and 2000 in F_1 , 94 t_1 increments with 2560 transients each. The delay Δ was 5.25 msec ($1/2J$) and the heteronuclear decoupling was achieved employing the GARP-1 scheme, using a low power ^{15}N pulse of 94 μsec .



Scheme 1



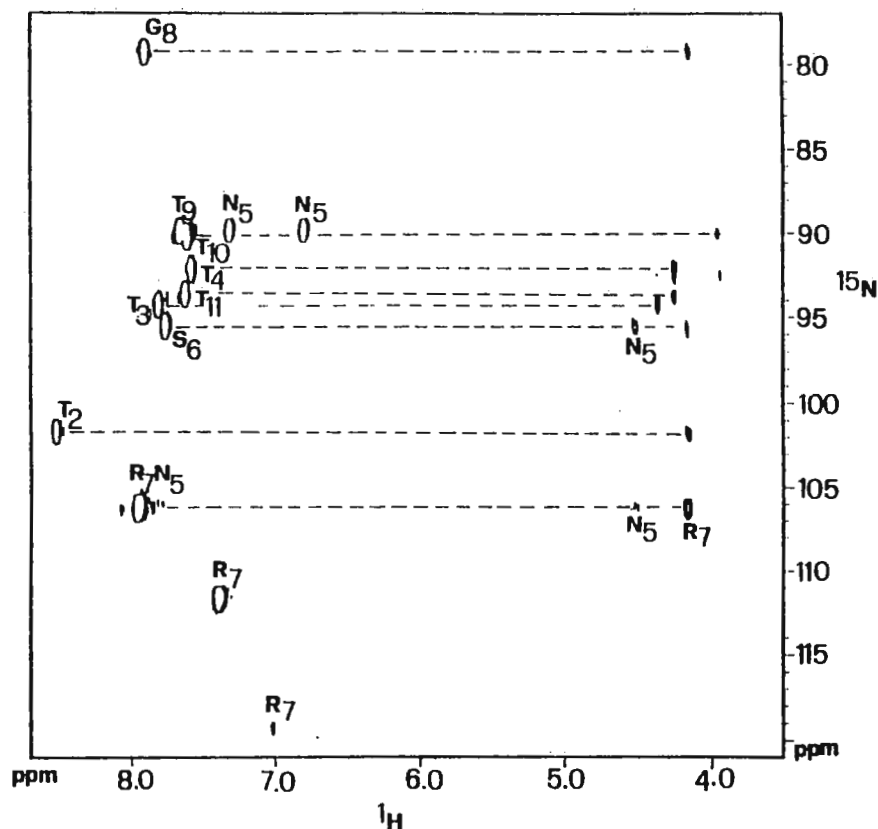
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Fig.1 Phase-sensitive heteronuclear ^{15}N - ^1H spectrum of CTTTNSRGTTT 40 mM in DMSO obtained by employing the HMQC-NOE sequence. Mixing time was 180 ms.



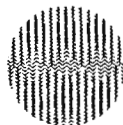
1) Shon, K. & Opella, S. (1989) J. Magn. Reson. 82, 193-195

Sincerely,

Consonni Roberto

Molinari Henriette

Zetta Lucia



INRA

INSTITUT NATIONAL DE LA RECHERCHE AGRONOMIQUE

 RÉPUBLIQUE FRANÇAISE
 MINISTÈRE DE LA RECHERCHE ET
 DE LA TECHNOLOGIE
 MINISTÈRE DE L'AGRICULTURE

STATION DE RECHERCHES SUR LA VIANDE

Prof. B. L. Shapiro
 966 Elsinore court, Palo Alto
 CA 94303

11 september 1990
 (received 9/17/90)

^{31}P NMR studies of the action of dantrolene on halothane-membrane interactions.

Dear Professor Shapiro

Malignant Hyperthermia is a pharmacogenetic disorder of skeletal muscle in susceptible humans and animals. It is triggered by stress or pharmacological agents like the volatile anaesthetic halothane. The syndrome characterized by skeletal muscle rigidity can be reversed by dantrolene, a relaxing agent.

We used solid state ^{31}P -NMR to study the effect of dantrolene on interactions of halothane-lipidic membrane. The effect was studied for different halothane-to-lipid molar ratio R_i , and for different dantrolene-to-lipid ratio R_d .

The second moments of spectra were always greater than those determined for halothane-dipalmitoylphosphatidylcholine system. In the fluid phase, the percentage of isotropic line which were dominant on spectra from halothane-lipidic membrane system decreased as well for any R_i (figure 1).

It is pointed out that dantrolene has an inhibition effect on the halothane-lipidic membrane system. Therefore these observations can reflect a stabilisation of the structure and / or dynamics of the phospholipid headgroups by addition of dantrolene.

Yours sincerely

S. GAILLARD
 SRV INRA Theix
 F 63122 Ceyrat

E. J. DUFOURC
 CNRS P. Pascal
 F 33600 Pessac

J.P. RENOU
 SRV INRA Theix
 F 63122 Ceyrat

Rd**0.13**

4.4

2

Ri

0.8

0

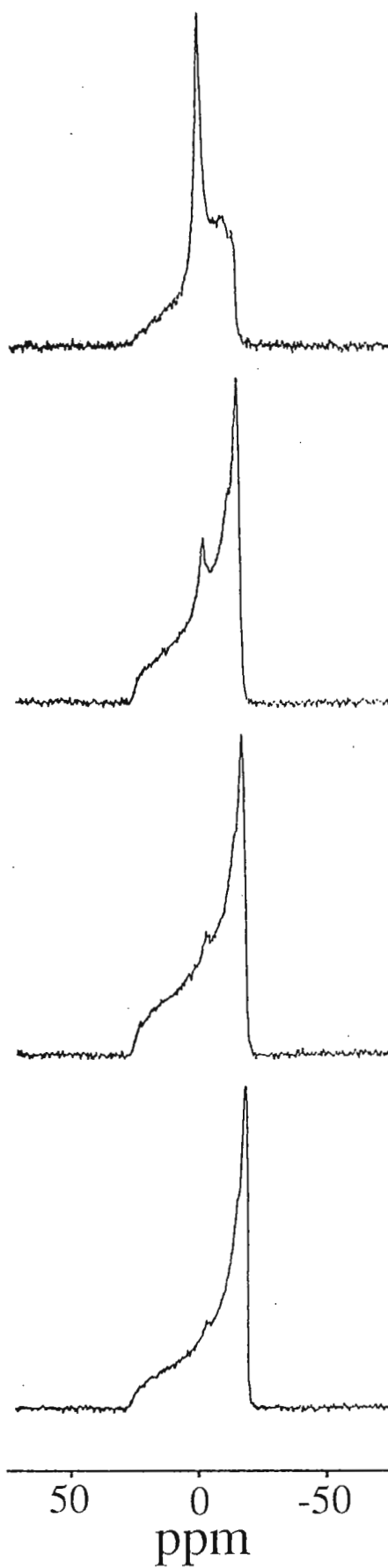
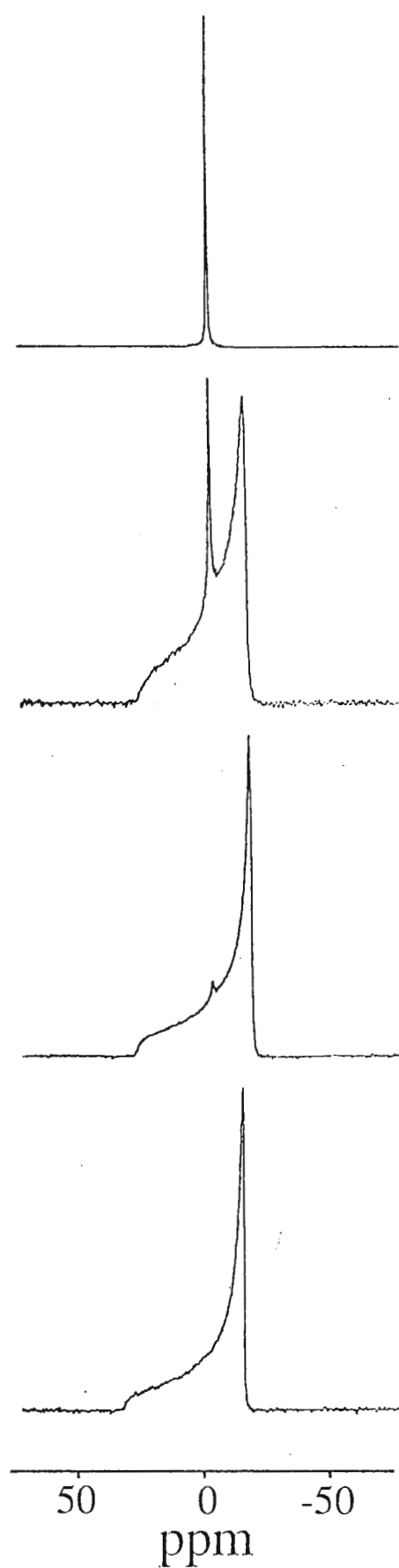


Figure 1 : Solid state ^{31}P -NMR spectra of DPPC dispersions at 41°C for different halothane / lipid ratios (Ri) without dantrolene $R_d = 0$ (left) and with dantrolene



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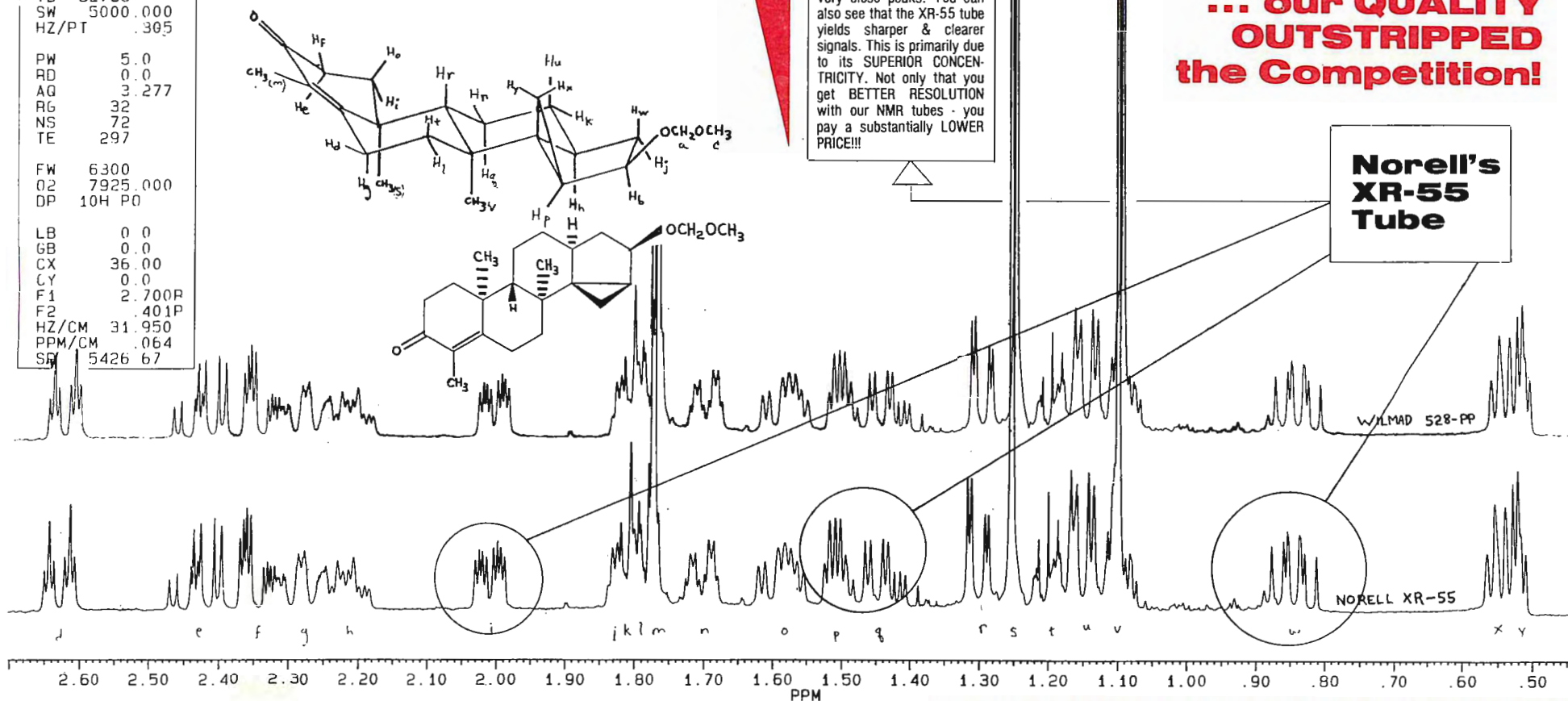
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October 4, 1990

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

Strasbourg Research Center

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(received 10/10/90)

PEPTIDE SPECTRUM SIMULATION PROGRAM

Dear Dr Shapiro,

We would like to report on a tool we are using here in our NMR laboratory at the Strasbourg Research Center. We are interested in peptide structure determination, and we currently use a standard set of 2D NMR experiments. As they are quite time-consuming, we thought of a simple preliminary test to evaluate the opportunity of applying them to peptides of unknown structure.

It is well established that the chemical shifts of the protons in a well structured peptide are significantly different from those in the "random coil" conformation. On the other hand, a denatured or unstructured mobile molecule displays chemical shifts which are nearly "random coil". We therefore wrote a program that can simulate the 1D proton spectrum of any given peptide (provided its sequence is known) in an extended conformation.

The program RCS (Random Coil Simulations) has been written using Microsoft FORTRAN version 5.0 and runs on IBM PCs and clones. It consists of three separate modules. First, a user-friendly sequence editor allows the input, visualisation, editing and storage of peptide sequences. The default database contains the spectral characteristics of the 20 common aminoacids. The second module is an editor that makes it possible to create new databases (e.g. for unusual or synthetic aminoacids or for different experimental conditions such as temperature, pH and solvent). Finally, the calculation itself, using a combination of one database and one sequence plus spectral parameters (i.e. linewidth, resolution and spectral window) produces, in a few seconds, a 16 Kbytes simulated spectrum in the AVIV format. We chose this format because it is particularly easy to emulate and because resulting files can be imported into a very versatile software package, Spectra-Calc (available from Galactic Industries Corp.). We use Spectra-Calc to process the simulated spectrum and to plot it. Of the many output devices supported by this program we have tried two (HP Laserjet+ and Zeta-8) with satisfying results. The experimental spectrum can either be superimposed directly within Spectra-Calc (Bruker files can be imported after transfer via a RS-232 line) or plotted separately and examined qualitatively using a light-box.

The two figures on the next page are examples of the experimental and calculated spectra for two peptides with very different structural features. The comparison suggests that one peptide is highly structured, whereas the second is in a conformation which is nearly "random coil". This conclusion is in very good agreement with our detailed studies based on 2D experiments. This assay, used *a priori*, would have helped us to save much spectrometer time !

Of course, both source and executable files together with directions for use are available for free, if anyone is interested.

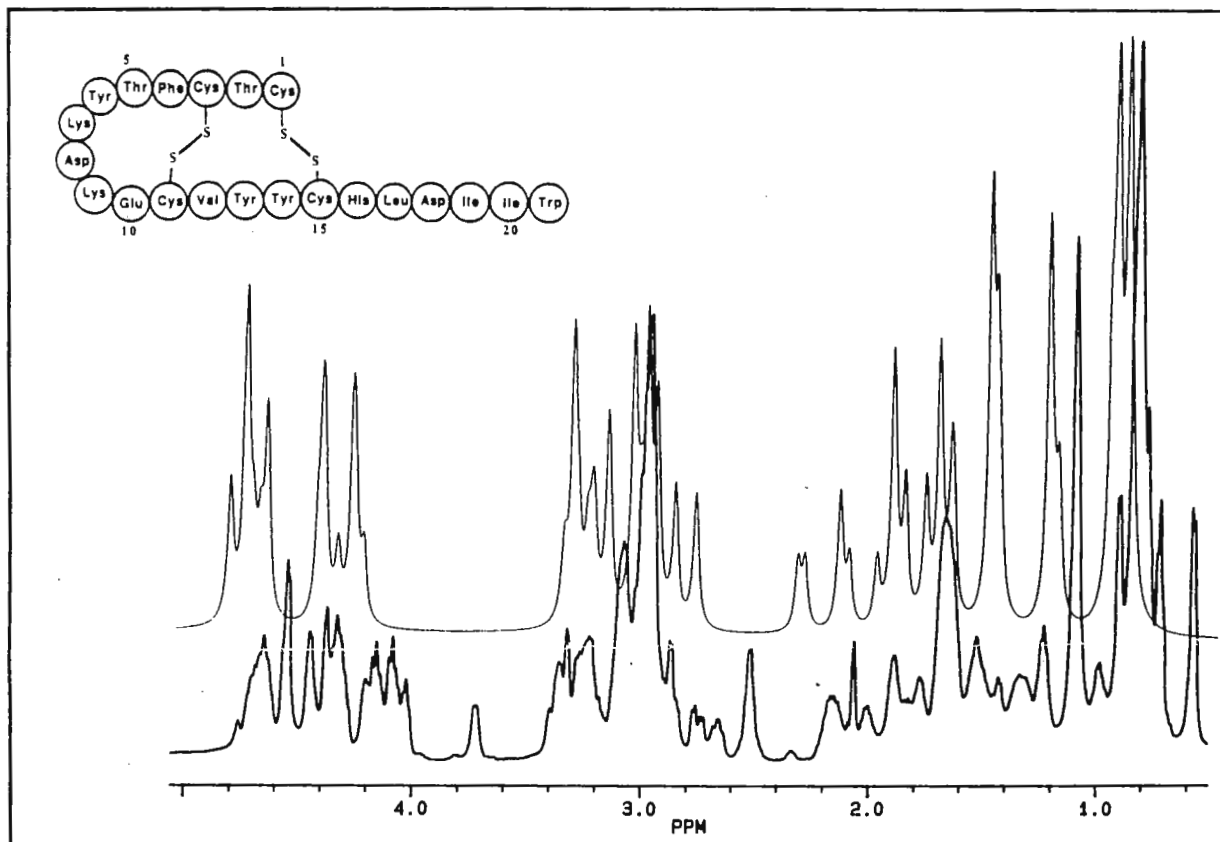
Sincerely,

P. Bortmann

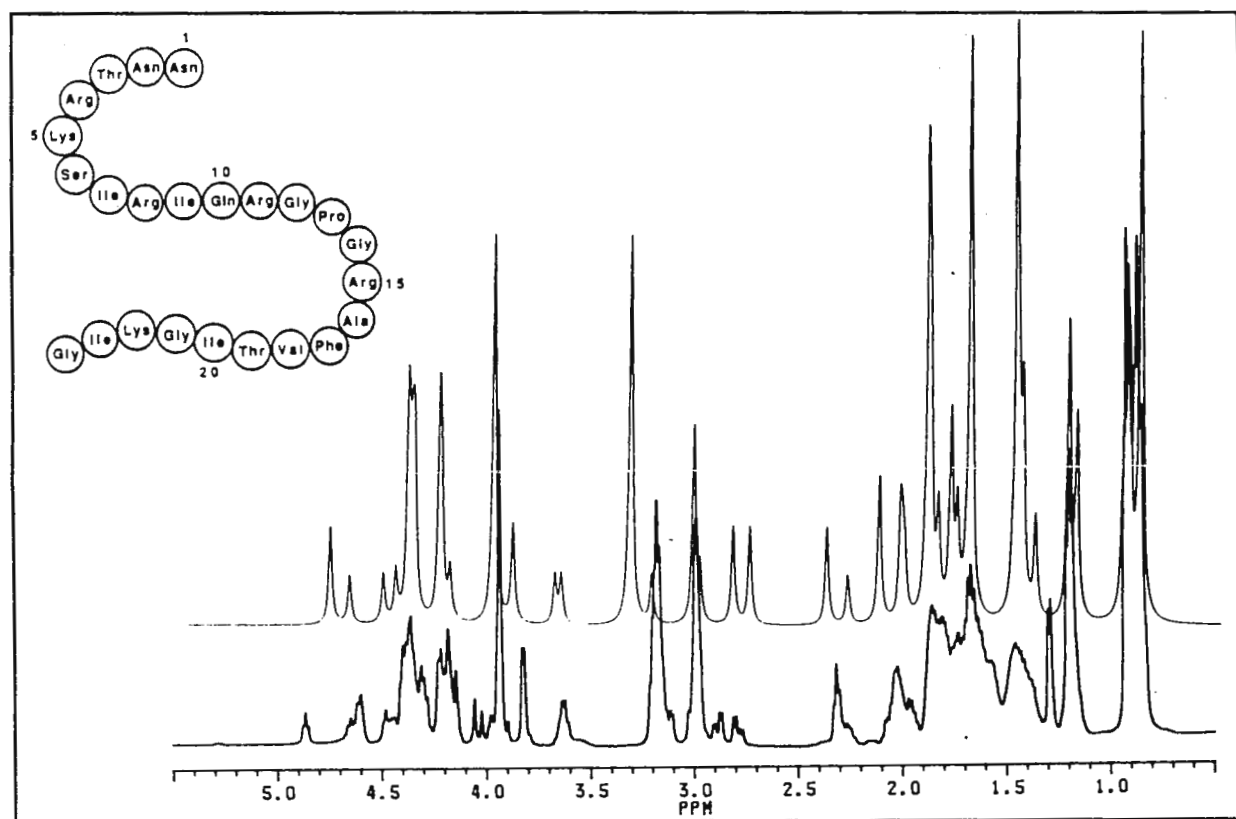


V. Saudek





Example 1 : Experimental (bottom) and simulated (top) ^1H NMR spectra of Endothelin-3, a bicyclic 21 residue peptide which is known to be folded in a compact globular structure.



Example 2 : Experimental (bottom) and simulated (top) ^1H NMR spectra of RP-135, a 24 residue fragment of the HIV envelope protein GP120, which exhibits almost no secondary structure.



The University of Alabama at Birmingham

Department of Chemistry

205/934-4747

FAX 205/934-2543

Telex 888826 UAB BHM

September 12, 1990

(received 9/17/90)

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

Dear Barry:

Recently, there has been considerable interest in the chemistry of materials (compound semiconductors; ceramics). This has led to a renewed research effort in main group chemistry. Some interesting comparisons between the NMR parameters of organoderivatives of the main group elements and their organic precursors can be made.

(1) δ_c data for analogous species in a group cannot necessarily be analyzed in terms of a simple parameter for the group members. Other contributions to δ_c may become important, especially for latter members of the group. For example, for the series R_3N , R_3P , and R_3As , the δ_c values for R_3As are anomalous with respect to R_3N and R_3P when only the electronegativity of the central element is considered.

(2) The δ_c and δ_H of the alpha carbon in an alkyl group may be very unusual for certain species. For example, in Et_2Zn , δ_c for CH_2 = 6.50 ($CDCl_3$, TMS) while δ_c for CH_3 = 10.11 ppm. Yet, for $n-Pr_2Zn$, $[Zn-C_1-C_2-C_3]$, δ_c = 19.27, 19.78, and 21.12 ppm for C_1 , C_2 , and C_3 , respectively. These values cannot be simply explained by substituent effect arguments.

(3) Interligand substituent effects may be noted. For example, δ_c for Me_3As , Me_2AsH , and $MeAsH_2$ are 11.17, 1.43, and -8.73 ppm, respectively ($CDCl_3$, TMS). Also, substitution of H for Me alters significantly the ^{13}C T_1 of the remaining methyl groups.

(4) Finally, 1H NMR spectral patterns may change dramatically with simple substitution. Et_3As exhibits an A_2B_3 (almost A_2X_3) pattern, $EtMe_2As$ a more pronounced A_2B_3 pattern, and Et_2MeAs a very complicated ABC_3 pattern for the ethyl group.

Other interesting effects may occur as well (such as spin-spin coupling to heavy elements) which may provide an interesting intellectual exercise.

With Best Regards,

Charlie

Charles L. Watkins
Professor

Lam

Larry K. Krannich
Professor and Chairman



Oklahoma State University

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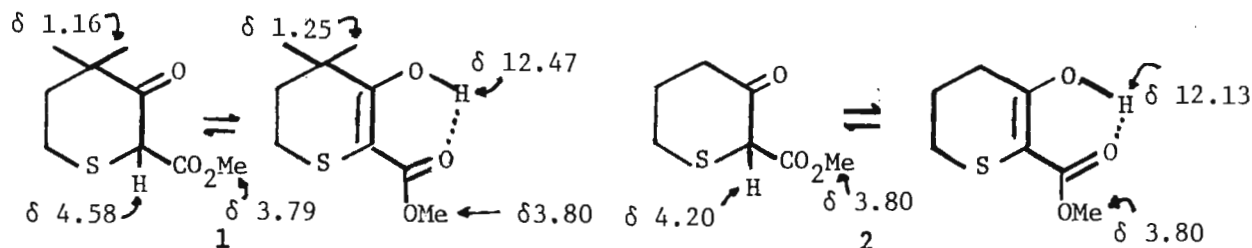
September 18, 1990
(received 9/24/90)

Dr. N. L. Shapiro
966 Elsinore Court
Palo Alto, CALIFORNIA 94303

SHORT TITLE: Enol-Keto Forms in Certain 3-oxotetrahydrothiapyrans

Dear Dr. Shapiro:

Recently we have been examining the chemistry of certain tetrahydrothiapyrans and the title systems have exhibited some interesting enol-keto tautomerism. Although this system has been reported once,¹ no indication of the ratio of the two forms was given. Our proton measurements indicate a ratio of 4:1 enol:keto system. Interestingly, comparing the ratio in **1** with that in **2**² disclosed that the latter was 3:2 enol:keto forms. Consequently, the gem dimethyl group in **1** may influence this ratio, an observation we are investigating further. Some characteristic shifts are shown on the structures. The inability of **1** to undergo



C-alkylation may be related to the geometry of the molecule **1**. The extreme complexity of the C-13 spectrum of **1** hints at perhaps more than one tautomeric form. This area of tetrahydrothiapyranones is not well studied but the compounds could be important synthons to natural products in which sulfur replaces a CH₂. Thus we plan to continue our examination of these novel heterocyclics.

We trust this will serve as our contribution for this time. Best regards.

Sincerely yours,

K. Darrell Berlin
Regents Professor

¹Baas, J. L.; Davies-Fidder, A.; Husiman, H. O. *Tetrahedron*, **1966**, 22, 285-291

²Young, T. E.; Heitz, L. J. *J. Org. Chem.* **1973**, 38, 1562-1566.



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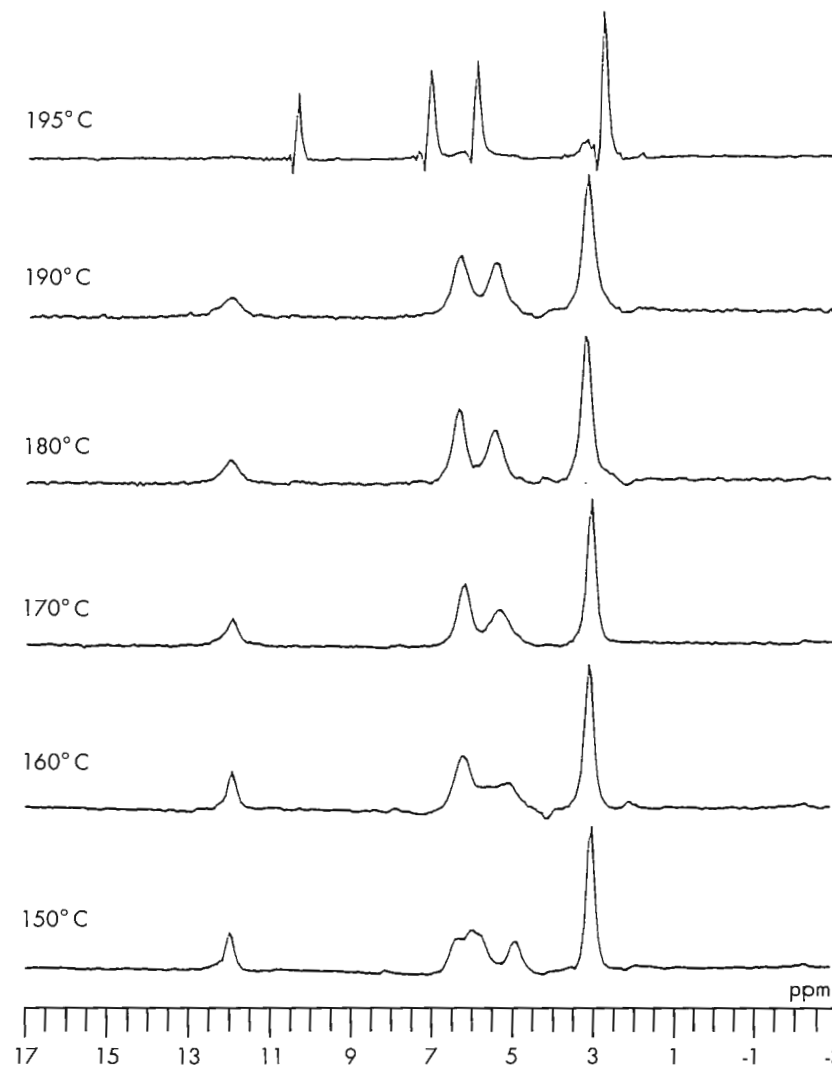
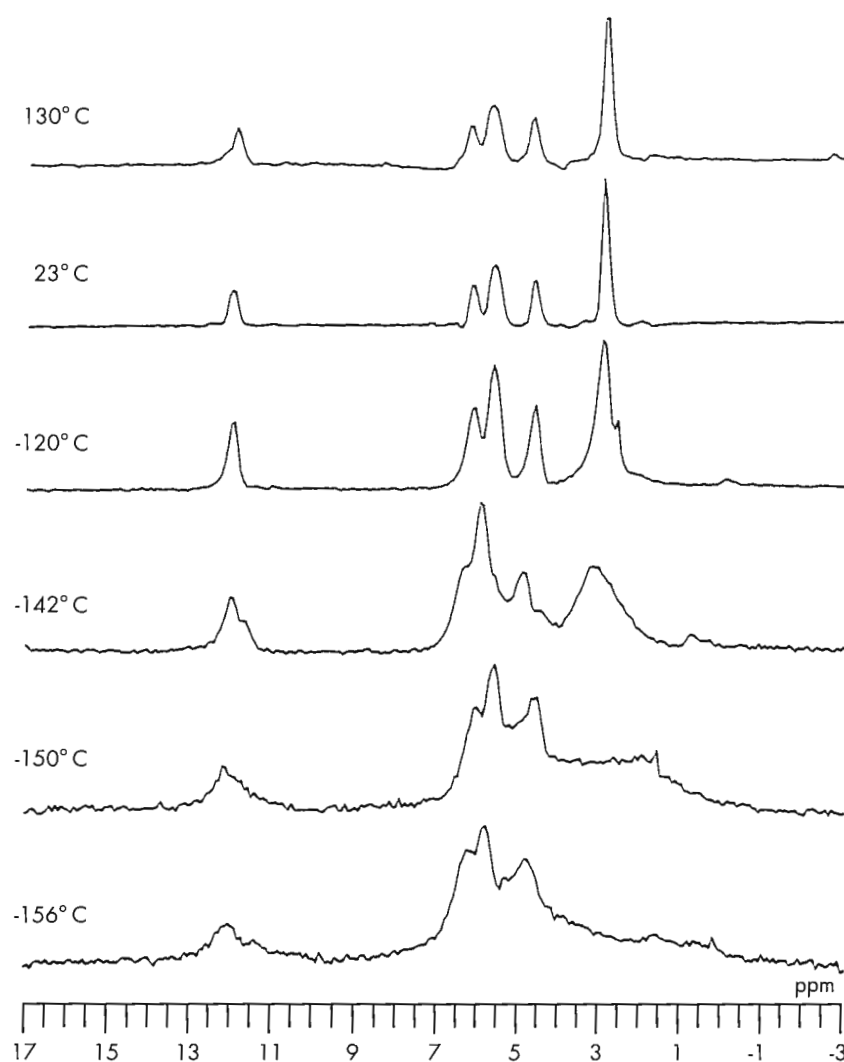
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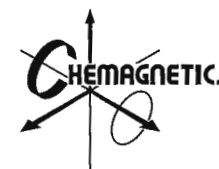
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Villeurbanne le 20 Septembre 1990

(received 9/24/90)

Dear Professor Shapiro,

I have the sad duty to inform you that Professor Jean DELMAU died at 60, in Perpignan on the end of June. He is now buried in Bron (suburb of Lyon) with his wife.

At the beginning of the sixties, Professor Jean DELMAU pioneerized conformational studies on dioxanes. His research activities were developed in Geneva and then in Lyon. Since 1966 he was Professor of Physics at the University of Lyon. In 1976 he was elected as the Dean of the Faculty of Sciences for five years. During the last six years he had to suffer a very strong Parkinson attack. Nevertheless he was continuously present in the laboratory, until September 1989.

However the activities of our group are increasing with high resolution nmr in the "Centre Commun d'Analyse par RMN" which was created by Professor Jean DELMAU in 1975 and is now managed by Dr. J.C. DUPLAN. Due to the large part of Medicine in our University we have also to deal with Imaging and more recently with "in vivo" spectroscopy.

I would ask you to continue to receive the NMR News Letters, and on behalf of my colleagues, I apologize for the present delay.

Yours truly,



Professor André BRIGUET

Abbott Laboratories' Pharmaceutical Products Division currently has an immediate opening for a COMPUTER PROGRAMMER/NMR SPECTROSCOPIST. As part of a team whose goal is to assist in designing new pharmaceutical agents by determining the three-dimensional structures of proteins and enzyme/inhibitor complexes by NMR, this position is responsible for aiding the development of computer programs for the automated analysis of multi-dimensional NMR data. A Ph.D. degree or equivalent experience along with knowledge of the requisite NMR methodology and/or experience in protein molecular modeling is required. Demonstrated skill in producing well-engineered software is a must.

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NMR Manager

We are looking for an individual with experience in NMR spectroscopy to manage the NMR facilities in our laboratory. Specific duties will include the care and maintenance of Bruker AM600, AM500, AMX500 and MSL300 spectrometers, instruction of students and postdoctoral fellows in the use of the instruments, and systems management responsibilities for related computer equipment. Involvement in the research activities of the laboratory is strongly encouraged. Experience in the operation and maintenance of modern NMR spectrometers is mandatory, and familiarity with computer programming and basic electronics is highly preferred. The focus of the research activities in the laboratory is the use of multi-pulse, multi-dimensional solution-state NMR spectroscopy to study the structure and function of proteins and nucleic acids. Applications, including salary history and the names of three individuals who can be contacted for references, should be sent to:

Dr. Peter E. Wright
Dept. of Molecular Biology, MB2
Research Institute of Scripps Clinic
10666 N. Torrey Pines Rd.
La Jolla, CA 92037

National Institutes of Health, Bethesda, MD.

Dear Barry:

10. October, 90

(received 10/12/90)

Jim Shoolery the Mentor.

I have just read the special section on Jim Shoolery in the September issue of the Newsletter. Many eloquent words by NMR's most eminent practitioners honoring the man and his contributions. When I finished, I felt that something was missing. It is a 'thank you' from the little guy.

In the heydays of NMR, the late fifties and early sixties, people were needed to build those cumbersome and awkward machines. To test them, to sell them, to install them in the customers' dungeons and to demonstrate the simple results.

We came from all walks of life. We learned to spell NMR the first day on the job with Varian, and soon our heads were filled with questions. Lucky were some of us who met Jim. No matter how busy his schedule, he would find the time to sit down and answer those questions. Not on his terms. He would find out how little we understood, and explain on our terms, in small steps, to give us some answers. He was a firm believer in the famous quote by one of the Varian brothers that a man has the right to know the significance of what he is doing, and he practiced it. And for this, we were all able to do better jobs.

I believe I can speak for many of us when I say: Thank you, Jim. You made our job a lot easier, you gave us purpose in what we were doing and a sense of belonging.

Sincerely
Rolf Tschudin





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France **Pierre GRANGER**

Strasbourg, le 10 October 1990
(received 10/16/90)

Neighbour Nuclei

Dear Bary

You perhaps ask you whether one day, I will enter the TAMU family. I often promise you that I will write some letter but time is passing. I take now my pen to write this contribution.

As you know, we are mainly interested by the NMR of "exotic" nuclei (if they are always exotic!) and we were looking for the ^{63}Cu NMR in a metallic cluster. Only three resonances in clusters are reported, to our knowledge, in the literature (Organometallic 4, 1224 (1985)) and they are highly shifted toward the low frequencies as it may be expected in such a case. We have observed on our compound a broad resonance exactly at the same chemical shift as the published ones, but our cluster was of a quite different nature. This seemed strange and we have performed a new spectrum in the same conditions without complex. The line was again present.

After some moments we have understood that we were observing the resonance of ^{23}Na contained in the different pieces of glass of the probe and of the tube. Returning to the publication, we have seen that it was a ionic complex having a sodium atom as a counterion and consequently the published results only concern sodium.

This situation may sometime arise and we have met for instance this problem when we have observed with Dr. Brevard ruthenium complexes containing potassium ions. As, to my knowledge, no list of possible coincidences exists, I have prepare such a list joint to this letter.

I hope you will enjoy my arrival to TAMU.

Best regards

P. GRANGER

TABLE of NEIGHBOUR NUCLEI

On the left is the nucleus at the higher frequency.

Underlined characters indicate practical cases.

Italic characters indicate cases where at least one nucleus has never been observed.

$^{49}\text{Ti} - ^{47}\text{Ti}$	<u>267 ppm</u>	$^{51}\text{V} - ^{123}\text{Te}$	5080 ppm
$^{87}\text{Rb} - ^{115}\text{Sn}$	673 ppm	$^{139}\text{La} - ^9\text{Be}$	5250 ppm
$^{185}\text{Re} - ^{99}\text{Tc}$	724 ppm	$^{131}\text{Xe} - ^{37}\text{Cl}$	5360 ppm
$^{193}\text{Ir} - ^{179}\text{Hf}$	1070 ppm	$^{23}\text{Na} - ^{51}\text{V}$	<u>5665 ppm</u>
$^{115}\text{In} - ^{113}\text{In}$	<u>2130 ppm</u>	$^{138}\text{La} - ^{133}\text{Cs}$	5950 ppm
$^{53}\text{Cr} - ^{49}\text{Ti}$	2660 ppm	$^{201}\text{Hg} - ^{97}\text{Mo}$	6370 ppm
$^{39}\text{K} - ^{109}\text{Ag}$	<u>2770 ppm</u>	$^{93}\text{Nb} - ^{45}\text{Sc}$	7530 ppm
$^{63}\text{Cu} - ^{23}\text{Na}$	<u>2870 ppm</u>	$^{99}\text{Ru} - ^{105}\text{Pd}$	8280 ppm
$^{69}\text{Ga} - ^{121}\text{Sb}$	2950 ppm	$^{109}\text{Ag} - ^{99}\text{Ru}$	<u>8530 ppm</u>
$^{191}\text{Ir} - ^{197}\text{Au}$	3500 ppm	$^{103}\text{Rh} - ^{177}\text{Hf}$	8650 ppm
$^{13}\text{C} - ^{79}\text{Br}$	<u>3580 ppm</u>	$^{51}\text{V} - ^{27}\text{Al}$	9370 ppm
$^{50}\text{V} - ^{135}\text{Ba}$	3600 ppm	$^{205}\text{Tl} - ^{203}\text{Tl}$	9720 ppm
$^{123}\text{Te} - ^{27}\text{Al}$	4333 ppm	$^{39}\text{K} - ^{99}\text{Ru}$	<u>11300 ppm</u>

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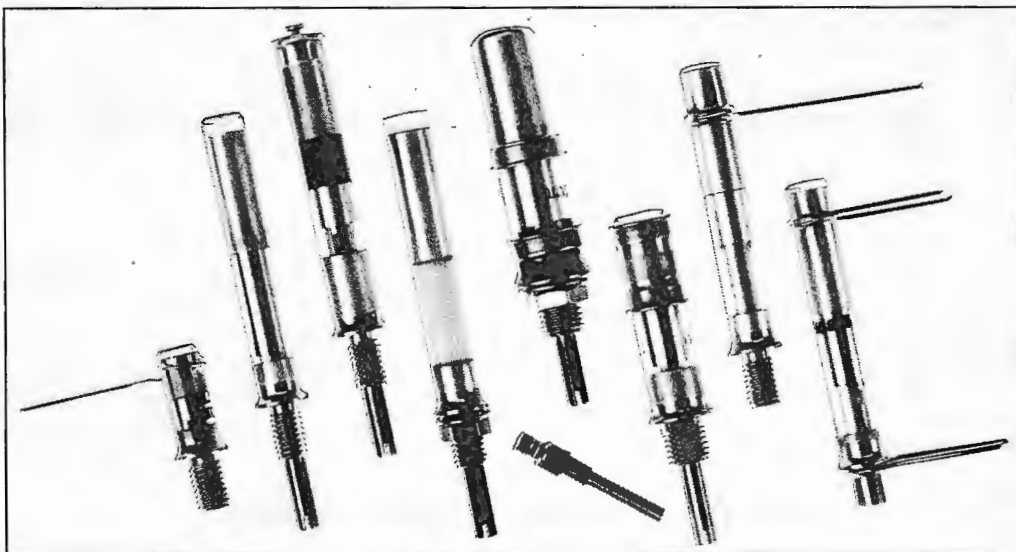
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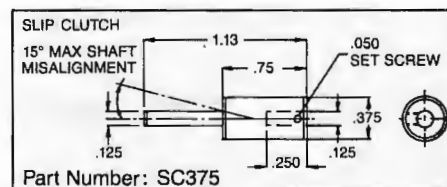
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If the mailing label on your envelope of this issue is adorned with a large red dot or circle: this decoration means that you will not be mailed any more issues until a technical contribution has been received by me.

Page Length Request Instruction

Attention overseas subscribers: If you must use paper which is longer than 11", *please* take care that all material (including signatures, addresses - everything!) ends no more than 10" from the top of each of your pages. It is costly to make reductions, and henceforth I reserve the right to chop the excess length off any page, no matter what the result.

Beware of the dreaded guillotine! Your cooperation in this matter will be greatly appreciated. Thank you.

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. 388 (January)-----* 7 December 1990
No. 389 (February) ----- 18 January 1991
No. 390 (March)-----15 February 1991
No. 391 (April) ----- 15 March 1991

**Please note that this deadline date is early in the month!!.*

* * * * *

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TAMU NMR Newsletter

Policies and Practical Considerations

(Revised October 1990)

The TAMU NMR Newsletter (formerly the IIT NMR Newsletter, and originally the Mellon Institute NMR Newsletter) continues with the same name, under the aegis of Texas A&M University, although the undersigned Editor/Publisher now resides in California. The Newsletter, now in its thirty-third year of consecutive monthly publication, continues under the same general policies as in the past. All communication with the Newsletter must be directed to the address overleaf.

1. Policy:

The TAMU NMR Newsletter is a means for the rapid exchange of information among active workers in the field of NMR spectroscopy, as defined broadly, including imaging. As such, the Newsletter will serve its purpose best if the participants impart whatever they feel will be of interest to their colleagues, and inquire about whatever matters concern them.

Since the subscriber/participant clearly is the best judge of what he or she considers interesting, our first statement of policy is "We print anything." (This usually is followed by the mental reservation, "that won't land us in jail.") Virtually no editorial functions are performed, although on rare occasions there is the need to classify a contribution as 'not for credit'. I trust that the reasons for this policy are obvious.

The TAMU NMR Newsletter is not, and will not become, a journal. We merely reproduce and disseminate exactly what is sent in. Foreign participants should not feel obliged to render their contributions in English.

2. Public Quotation and Referencing:

Public quotation of Newsletter contents in print or in a formal talk at a meeting, etc., is expressly forbidden (except as follows), and reference to the TAMU NMR Newsletter by name in the scientific literature is never permissible. In order to quote results or use material from the Newsletter, it is necessary, in each individual case, to obtain the prior permission of the author in question and then to refer to the material quoted as a "Private Communication". If your copy of the Newsletter is shared with other readers, it is your obligation as the actual recipient of the Newsletter to see that these other readers of your copy are acquainted with, and abide by, these statements of policy.

3. Participation is the prime requisite for receiving the TAMU NMR Newsletter:

In order to receive the Newsletter, you must make at least occasional technical contributions to its contents.

We feel that we have to be quite rigorous in this regard, and the following schedule is in effect: Eight months after your last technical contribution you will receive a "Reminder" notice. If no technical contribution is then forthcoming, ten months after your previous contribution you will receive an "Ultimatum" notice, and then the next issue will be your last, absent a technical contribution. Subscription fees are not refunded in such cases. If you are dropped from the mailing list, you can be reinstated by submitting a contribution, and you will receive back issues (as available) and forthcoming issues at the rate of nine per contribution.

Frequent contributions are encouraged, but no "advance credit" can be obtained for them. In cases of joint authorship, either contributor, but not both, may be credited. Please indicate to whose account credit should be given. Please note that meeting announcements, as well as "Position Available," "Equipment Wanted" (or "For Sale"), etc., notices are very welcome, but only on a not-for-credit basis, i.e., such items do not substitute for a *bona fide* technical contribution. Similar considerations must occasionally be applied to a few (quasi-)technical items.

4. Finances:

The Newsletter is wholly self-supporting, and depends for its funds on advertising, donations, and individual subscriptions.

The Subscription fee for the October 1990 - September 1991 year is US\$150.00, with a 50% academic or personal subscription discount. Subscriptions are available only for the twelve monthly issues which begin with the October issue and run through that of the following September. However, a subscription can be initiated at any time, and the issues back to the previous October will be provided as long as copies remain available.

Companies and other organizations are also invited to consider joining the list of Sponsors of the Newsletter. Sponsors' names appear in each month's Newsletter, and copies of the Newsletter are provided to all Sponsors. The continuation of

Continued

this non-commercial Newsletter depends significantly on the interest and generosity of our Sponsors, most of whom have been loyal supporters of this publication for many years. We will be happy to provide further details to anyone interested.

Another major, indeed most essential, source of funds for the Newsletter is Advertising. We earnestly encourage present and potential participants of the Newsletter to seek advertising from their companies. Our rates are very modest - please inquire for details.

5. Practical Considerations:

- a) All technical contributions to the TAMU NMR Newsletter will always be included in the next issue if received before the published deadline dates.
- b) Please provide short titles of all topics of your contributions, so as to ensure accuracy in the Table of Contents.
- c) Contributions should be on the *minimum* (NOTE!!) number of 8.5 x 11" (21 x 27.5 cm) pages, printed on one side only. Contributions may not exceed three pages without prior approval. Each page must have margins of at least 0.5 - 0.75" (1.3 - 2.0 cm) on all sides. Please observe these limits. Black ink for typing, drawings, etc., is essential. All drawings, figures, etc., should be mounted in place on the 8.5 x 11" pages. We are not equipped to handle pieces of paper larger than 8.5 x 11" (21 x 27.5 cm).

Foreign subscribers are reminded that regardless of the standard paper length you use, all material - letterhead, text, figures, addresses printed at the page bottom, everything - must not exceed 10" (ca. 25.3 cm) from top to bottom.

Significant savings of Newsletter pages and total space can be made by exercising close control over the formatting and type sizes of the contributions. Please consider the following:

i) For those with computers, try using a smaller type font. The body of this page is printed in 10 point type, which I believe is adequate for most purposes. Even 12 point is acceptable, I suppose. Those who are computerized can also employ non-integral spacing of lines so that sub- and superscripts don't collide with lines below and above.

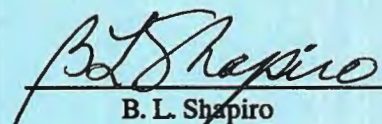
ii) PLEASE avoid excessive margins. *Instruct your secretaries to avoid normal correspondence esthetics or practices, however time-honored or 'standard'!* This page has margins on both sides of 0.6" (ca. 1.55 cm), which is very adequate. Margins of the same size at the top and bottom are sufficient also, but don't worry if there is more space at the end of your document, for I can often use such spaces for notices, etc.

Also, please avoid large amounts of unused space at the top of letters. Give thought to the sizes of figures, drawings, etc., and please mount these so as to use the minimum space on the page.

iii) 'Position Available', 'Equipment Wanted', and Similar Notices. These are always welcome, without charge, but not for subscription credit, of course. Such notices will appear, however, *only* if received with these necessarily rigid constraints: a) Single spaced; b) both side margins 0.6 - 0.7" (1.5 - 1.7 cm.) - NOT WIDER; c) the minimum total height, please, but definitely no more than 4.5" (11.5 cm.) This will let me place such notices wherever a bit of space occurs.

iv) AVOID DOUBLE SPACING LIKE THE BLACK PLAGUE ! ! ! This is extremely wasteful of space. Even sans computer, small type and 1.5-line (if needed) spacing can be had with a little effort.

6. Suggestions: They are always welcome.


B. L. Shapiro

Address for all correspondence:

Dr. Bernard L. Shapiro
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto, California 94303
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Telephone: (415) 493-5971. Please confine telephone calls to the hours from 8:00AM - 10:00PM, *Pacific Coast Time*.

30 October 1990.

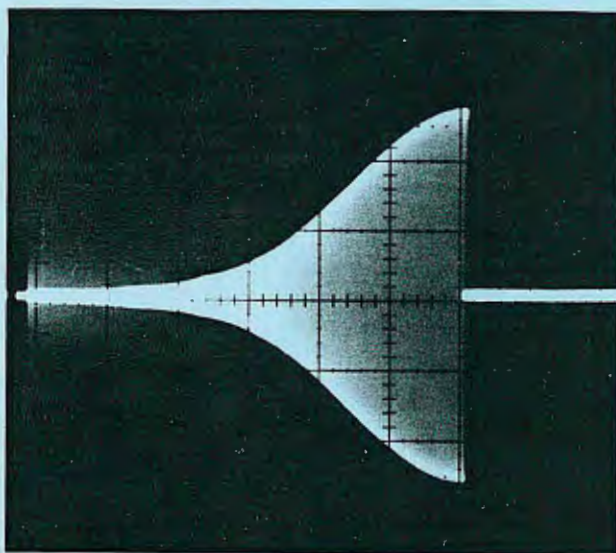
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Pulse Shaping Made Simple

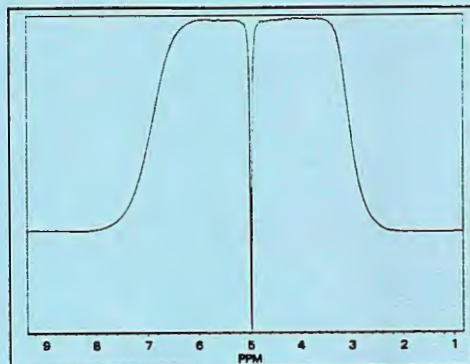
Omega PSG (Pulse Sequence Generator) boards provide very flexible control of both amplitude and phase on each transmitter channel. Through a unique combination of instruction and waveform memory, waveform libraries can be

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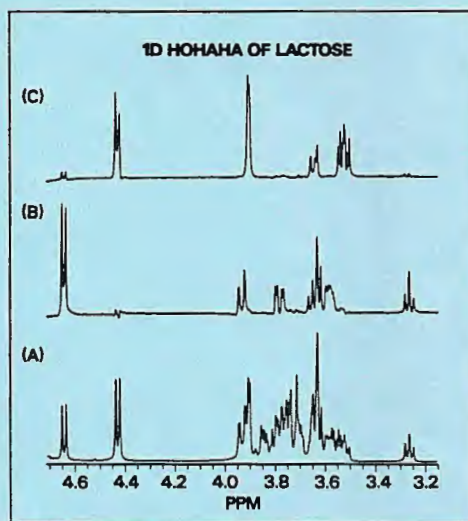
Wave Shaping on the Omega 500 PSG



An oscilloscope trace of a half-Gaussian pulse. The pulse is defined by 250 points and the duration is 10 ms.



The result of applying a 180° half-Gaussian pulse to a sample of doped water. The water resonance has been broadened by introducing a large Z1 current in the room temperature shims. The half-Gaussian pulse width is 200 ms and the width of the "burned hole" is 12 Hz.



TOCSY of Lactose (10mM in D_2O). Bottom spectrum (A) is a simple one pulse spectrum. Middle spectrum (B) is a 1D-TOCSY spectrum, where anomeric proton at 4.43 ppm has been selectively irradiated with a half-Gaussian pulse. Top spectrum (C) is a 1D-TOCSY spectrum when the anomeric proton at 4.65 ppm has been selectively irradiated with a half-Gaussian pulse.

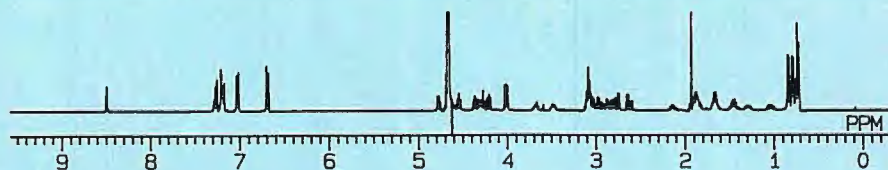
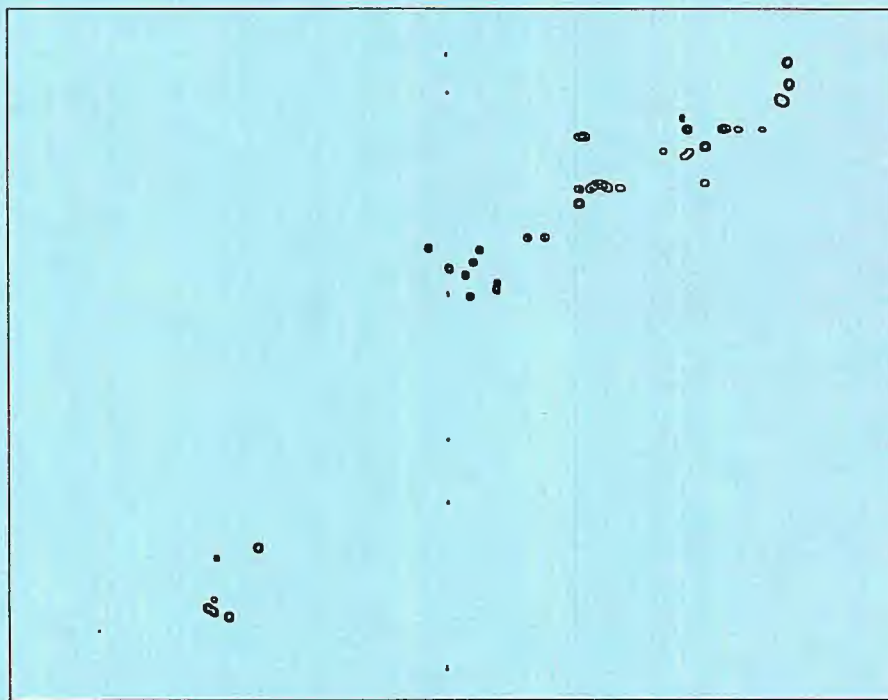


GE NMR Instruments

AFTER THE WORK IS FINISHED...

THE OTHER CPF COMES OUT TO PLAY

When all of the day's production samples have been run and the piles of spectra plowed through, JEOL's CPF can quickly switch operation modes. This same highly automated machine which is used for all of the production work can produce very high quality research data for those non-routine problems that always seem to appear. In many cases all that is necessary to make the change is to log into the research account. This changes the CPF from an automated, limited instrument into a wide-open research-grade spectrometer. With the addition of the appropriate accessories the low cost CPF is capable of running the most sophisticated research type experiments including CP MAS solids. Now that the CPF is available at a field strength of 400 MHz in addition to the very popular 270 MHz, the high field research spectrometer has become very affordable. The above 400-CPF data is a reverse detection ^{13}C experiment run on Angiotensin-II. The instrument used for this experiment is a standard CPF with the addition of the optionally available reverse detection Broad Band probe. This data clearly shows that the routine need never be the only thing you can do.



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