TEXAS A&M UNIVERSITY

NMR NEWSLETTER

No. 411

December 1992

Continued on Inside Back Cover

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TABLE 1 DEUTERATED SOL

Cat. No.	Description	Formula	Min. Isotopic Purity (%D)	Density (g/cc)	MP (°C)	BP (°C)	$-\chi_v \times 10^6$ @ (°C)
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D-13	Acetone-d_6	CD_3COCD_3	99.8%	0.87	-94	57	0.460 (20)
D-121	Aceonte-d_6 + 1% TMS		99.8%				
D-129							
D-14							543 (20)
D-21							0.611
D-122							
D-130							
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FORTHCOMING NMR MEETINGS

International Symposium on Solid-State NMR Spectroscopy of Polymers, Keystone, Colorado, **February 28 - March 3, 1993**; Contact: Barbara D. Hogan, Du Pont, CR&D, P. O. Box 80356, Wilmington, Del. 19880-0356; Phone: (302) 695-4394; FAX: (302) 695-8207.

PITTCON'93, Atlanta, Georgia, **March 8-12, 1993**; Contact: Dept. 60, Suite 332, Penn Center Blvd., Pittsburgh, PA 15235-5503; Phone: (412) 825-3220, Toll-free: 1-800-825-3221; Fax: (412) 825-3224.

1993 Keystone Symposia on Molecular & Cellular Biology, Taos, New Mexico: **March 8-14, 1993**, *Frontiers of NMR in Molecular Biology - III*; Organizers: T. L. James, S. W. Fesik, and P. E. Wright; Information: Keystone Symposia, Drawer 1630, Silverthorne, CO 80498; Telephone: (303) 262-1230.

34th ENC (Experimental NMR Conference), St. Louis, Missouri, **March 14-18, 1993**; Contact: ENC, 815 Don Gaspar, Santa Fe, New Mexico 87501; Phone: (505) 989-4573; Fax: (505) 989-5073. See TAMU NMR Newsletter 408, 45.

Advanced School on Magnetic Resonance and Protein Dynamics, Erice, Sicily, Italy, **March 15-21, 1993**; Contact: Prof. Oleg Jardetzky or Stanford Magnetic Resonance Lab., Stanford Univ., Stanford, CA 94305-5055; Fax: (415) 723-2253; See TAMU NMR Newsletter 411, 50.

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

35th Rocky Mountain Conference on Analytical Chemistry, Denver, Colorado: **July 25-29, 1993**; Contact: Patricia L. Sulik, RML, Inc., 456 S. Link Ln., Ft. Collins, CO 80524; Phone: (303) 530-1169.

1993 FACSS Meeting, Detroit, Michigan, **October 17-22, 1993**; Contact: H. N. Cheng, Hercules, Inc., Research Center, 500 Hercules Road, Wilmington, DE 19808; Phone: (302) 995-3505; Fax:. (302) 995-4117. See TAMU NMR Newsletter 411, 10.

Additional listings of meetings, etc., are invited.

411-2

Lilly

Lilly Research Laboratories
A Division of Eli Lilly and Company

Lilly Corporate Center
Indianapolis, Indiana 46285
(317) 276-2000

October 27, 1992
(received 10/30/92)

Prof. B. L. Shapiro
968 Elsinore Court
Palo Alto, CA 94303

Tipping Over a Can

Here at Lilly, as with many companies these days, we are in a constant search for more laboratory space. For example, when our Analytical Development group recently fell heir to a badly needed spectrometer, they had to scramble to find space for it. They finally settled on crowding it between two existing spectrometers in a lab that was already pretty busy. Unfortunately, this laboratory was also one that people were accustomed to walking through, and experience had shown it necessary to put up those temporary, half-height walls around spectrometers. You know the type of walls I mean: the type that you push a couple of inches toward your neighbor's office when he is away, and which he pushes right back when he returns. In any event, it was decided to put up some of these walls around the new spectrometer to keep out the kibitzers that look over the operator's shoulder and ask if there are any interesting games on the computer.

And so eventually the workmen showed up with a stack of half-height walls. As it turned out, they showed up during a week that the senior spectroscopist of the lab, Steve Maple, was on vacation. In addition, they ignored the instructions that Steve had left for them to assemble the walls elsewhere, hauled the stack of panels into the lab, and leaned them against a hood. (You can see what is coming, can't you?) While they were deciding just how to get started, someone bumped against one of the panels leaning against the hood, which tipped over against its neighbor, which in turn.... But then we have all of us played with dominoes this way, haven't we?

Anyhow, readers, please take my word for the fact that there is no sadder sight than to see than a nice, shiny superconducting cryostat lying on its side with a pile of half-height walls stacked on it. I arrived some 15 minutes after the event, having been called in with the hope that I would have some clever idea what to do. The best I could come up with was to turn on the write-protect of the disk, turn off the computer, and then discourage a couple of cruel mass spectrometrists who were trying to ruin Steve's vacation by finding a way to apprise him of the event. After examination of the innards of the cryostat a week later, Steve and his coworkers wisely decided to have a new one built, and they are now waiting for delivery. While insurance is paying for the replacement of the hardware, there is no way to recoup the lost time and efficiency.

There is a lesson here; come to think of it, there are probably a couple of lessons. But the one that I am trying to make is that spectrometers are very special beings that need their own space, and that putting them into high traffic areas is something to be avoided at all costs.

Best Regards,

Doug

Doug Dorman

Hoffmann-La Roche

Hoffmann-La Roche Inc.
340 Kingsland Street
Nutley, New Jersey 07110-1199

Direct Dial

(received 11/3/92)

October 26, 1992

SAME STRUCTURE + DIFFERENT SEQUENCE = SIMILAR CHEMICAL SHIFTS?

Dear Barry:

We are studying the solution structure of the Interleukin-1 Receptor Antagonist (IL-1ra) and its interaction with the IL-1 receptor. We have made nearly complete assignments of the backbone ^{1}H, ^{13}C and ^{15}N resonances of IL-1ra$_{(des1-6)}$ in 25 mM sodium acetate pH 6.3 at 35°C. At this preliminary stage, we have about 700 NOE-derived distance constraints, which were applied in CHARMM to evaluate the backbone conformation. It is very similar to that of IL-1β, although the two proteins share only 26% sequence identity. The chemical shifts of IL-1β under somewhat comparable conditions (100 mM sodium acetate, pH 5.4, 36°C) have been reported in the pioneering studies of Clore and co-workers (1,2). This seemed like a unique opportunity to assess the contribution of backbone structure to chemical shifts in the presence of substantially different primary sequences. In particular, we wondered whether it would be possible to predict the chemical shifts of proteins with similar structures which are not yet assigned (IL-1α and FGF, for example).

Chemical shifts were corrected for residue type by subtracting the conformationally-averaged "standard" values given by Wishart, Sykes, and Richards (3,4). Comparison plots are shown in Figure 1. The structure-dependent deviations from standard values were surprisingly similar for the Cα protons. The comparison was not as encouraging for the NH, $^{13}C\alpha$, and $^{15}N\alpha$ shifts.

Nevertheless, we pulled out some old (^{1}H, 400 MHz) data on IL-1α. We took the average deviations for IL-1ra and β and, using sequence homolgy (again, about 25%), back-calculated the expected CαH chemical shifts of IL-1α. Of the 20 residues with predicted CαH values greater than 5.2 ppm, 3 were alanines: A 38, 6.11; A 129, 5.83; & A 62, 5.46. In the COSY spectrum we observed only 3 alanine-like connectivities with CαH protons downfield of 5.2 ppm: 5.77; 5.57; & 5.53. Our initial feeling is that the homology method cannot yield precise predictions, but may be useful for suggesting assignment candidates.

Sincerely yours,

David Fry, David Greeley, Kirsten Berghmans, Sudha Narasimhan, and Ross Pitcher

1. Driscoll, P.C., Clore, G.M., Marion, D., Wingfield, P.T., & Gronenborn, A.M. (1990) *Biochemistry* **29**, 3542.
2. Clore, G.M., Bax, A., Driscoll, P.C., Wingfield, P.T., & Gronenborn, A.M. (1990) *Biochemistry* **29**, 8172.
3. Wishart, D.S., Sykes, B.D., & Richards, F.M. (1991) *J. Mol. Biol.* **222**, 311.
4. Wishart, D.S., Sykes, B.D., & Richards, F.M. (1992) *Biochemistry* **31**, 1647.

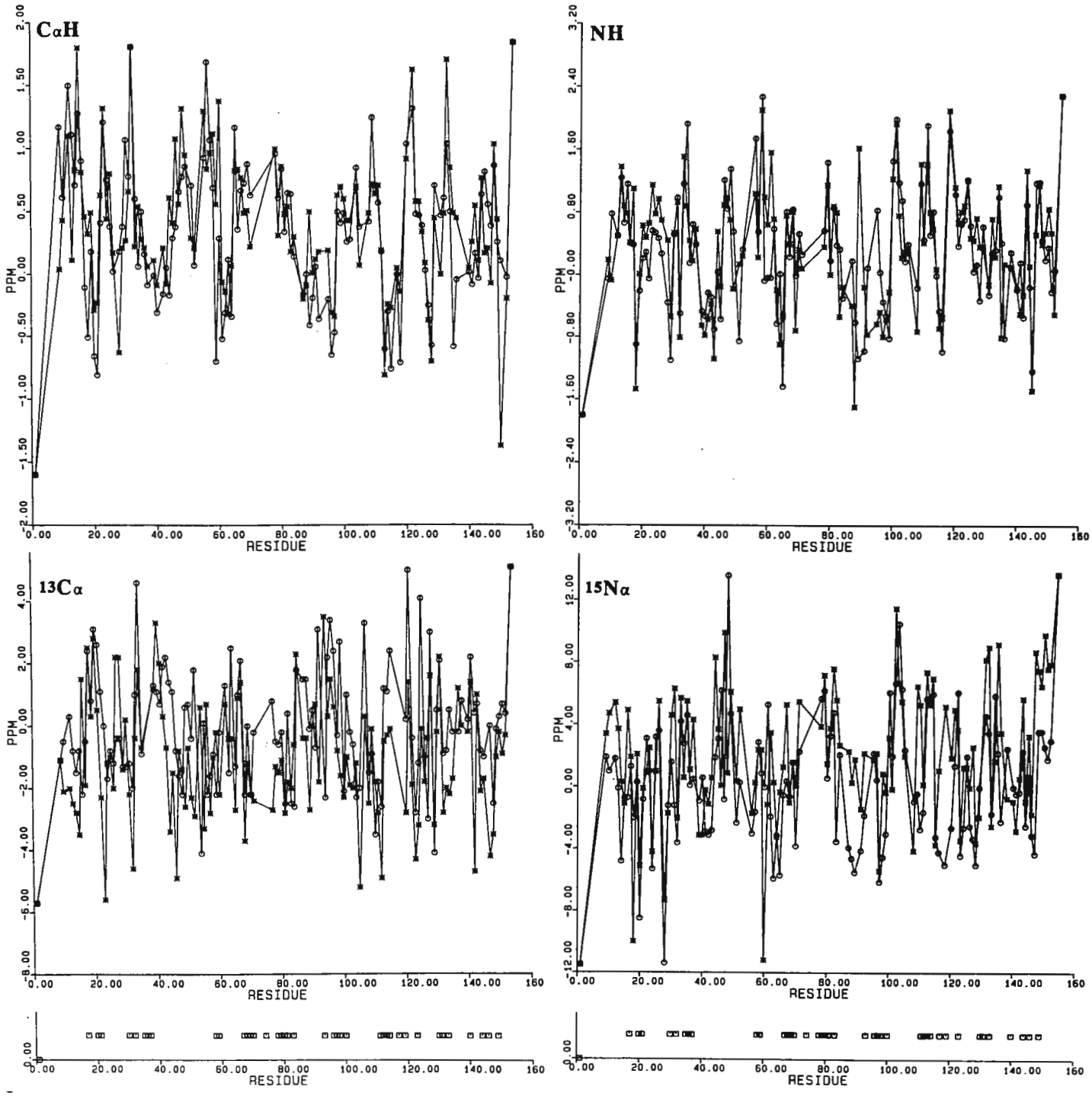

FIGURE 1: Differences in ppm between observed chemical shifts and standard values for IL-1ra$_{(des1-6)}$ (*) and IL-1β (O). Standard values were taken from the following sources: CαH - Table II of ref. 4; NH - "All" column of Table 7, ref. 3; ^{15}Nα - "All" column of Table 10, ref. 3; ^{13}Cα - "All" column of Table 8, ref.3. The residue numbers refer to IL-1ra; IL-1β was aligned by sequence homology. Squares indicate identical residues. Dummy points at residues #1 and #154 are for scaling purposes only. Missing data points represent residues for which assignments were not available for one or both of the proteins. Chemical shifts for IL-1β were taken from refs. 1 & 2.

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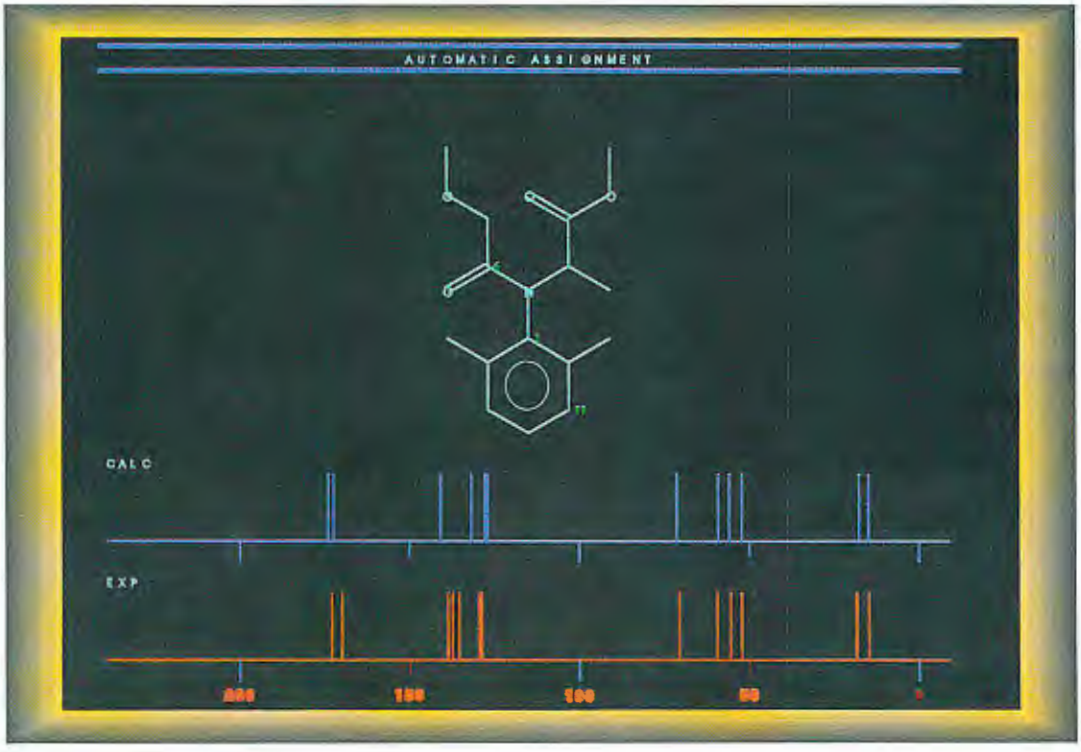

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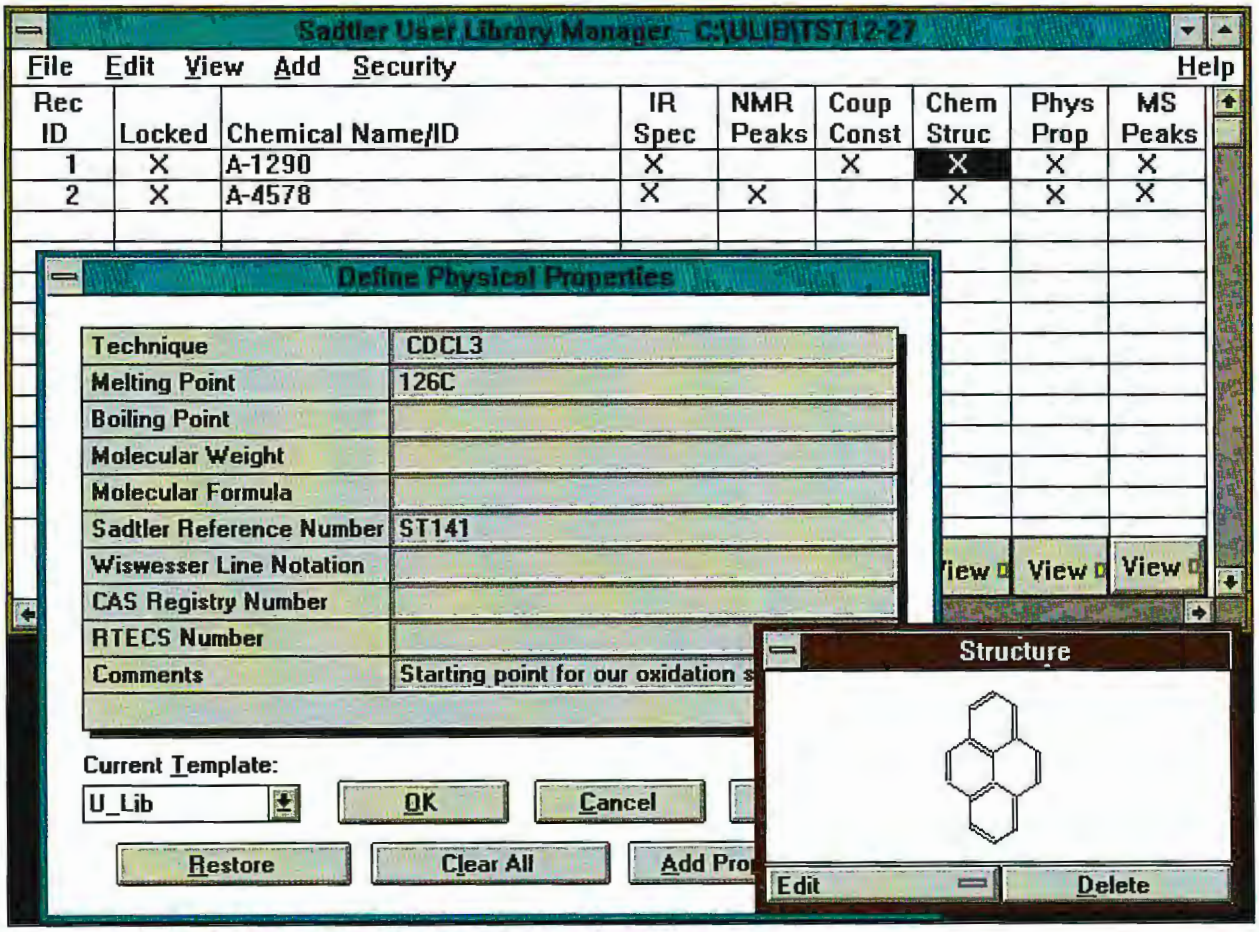

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2200 BONISTEEL BOULEVARD
ANN ARBOR, MICHIGAN 48109-2099

October 29, 1992
(received 11/2/92)

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

Dear Dr. Shapiro:

In working with some relatively small DNA fragments we have run into problems with zero-quantum transfers in our short-τ_m NOESY datasets. The classical method to suppress these transfers is to randomly vary the τ_m mixing time or the position of a 180 pulse in the τ_m mixing time from increment to increment. For this particular sample with narrow lines this approach gave rise to excessive noise in the 2D data set. We have also used the alternative approach by shifting a 180 pulse in τ_m systematically with the t_1 increment. This gives rise to ZQ peaks symmetrically displaced around the cross peaks and the diagonal. These shifted peaks interfere with other NOE cross peaks, especially since they have an antiphase-dispersion lineshape.

Here, we report on our efforts to use a three-dimensional approach for separating NOE and zero-quantum transfer cross peaks. By actually recording the 2D NOESY experiment as a 3D data file

$$RD - 90 - t_1 - 90 - (\tau_m - t_2)/2 - 180 - (\tau_m + t_2)/2 - 90 - t_3$$

we obtain for the NOE cross peak and the zero-quantum transfer cross peak between between the scalar coupled A and B resonances the modulations

$$\cos(\Omega_A t_1)\cos(\pi J_{AB} t_1)\cos(\Omega_B t_3)\cos(\pi J_{AB} t_3)$$

and

$$\cos(\Omega_A t_1)\sin(\pi J_{AB} t_1)\cos((\Omega_A - \Omega_B) t_2)\cos(\Omega_B t_3)\sin(\pi J_{AB} t_3)$$

respectively.

Upon 3D transformation, the NOE cross peaks stay in the first (axial) ω_2 plane while the zero-quantum transfer cross peak are shifted out in three-dimensional space along ω_2. As a result, the NOE plane is free of the zero-quantum interferences and also remarkedly devoid of noise.

This is shown in the figure where a comparison of the C5-C6 cross peak areas of a "jittered" 2D and the first ω_2 plane of the 3D spectrum is shown. The 2D and 3D data were acquired in the same time with the same total number of scans: 2D, $512(t_1)*2048(t_2)$ and 48 scans per increment ; 3D, $512(t_1)*12(t_2)*2048(t_3)$ and 4 scans per increment. In the 3D data, t_2 was extended to 32 points by linear prediction prior to Fourier transformation (Felix 2.05; Hare Research Inc.).

The experiment is also a demonstration of the fact that increase in dimensionality is not accompanied by loss in sensitivity provided that no additional (incrementable) delays are used - as is the case here.

Sincerely,

Hong Wang Gary Glick Erik R.P. Zuiderweg

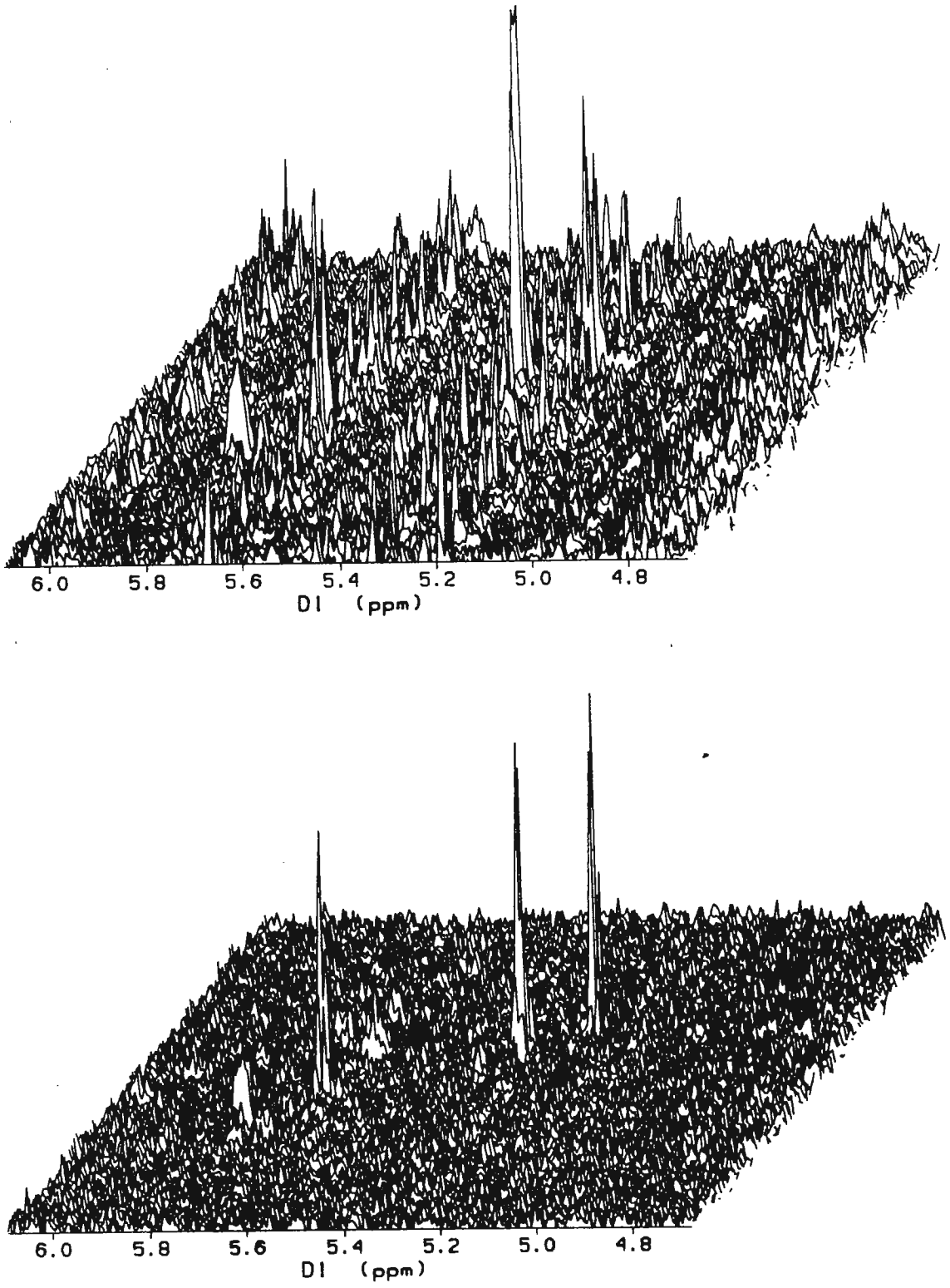

Figure (Wang et al.). Comparison of the C5-C6 region of hairpin dodecamer DNA NOESY spectra. Top, the "jittered" spectrum; τ_m = 25 ms. Bottom, the first plane of the 3D spectrum, τ_m = 25 ms. Processing and vertical scale is identical for the two data sets. D1 is the direct dimension.

ISIS
PHARMACEUTICALS

September 18, 1992
(received 10/23/92)

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

1H-1H AND 1H-13C OLIGONUCLEOTIDE COUPLING CONSTANTS

Dear Barry:

We have been interested in estimating the conformational properties of single nucleotides in a DNA duplex as a function of temperature. This is important for the developing antisense field, where modifications at one site can produce dramatic (or no) changes in local helical structure. These changes are manifested as increases or decreases in duplex stability which can alter biological properties. The "standard" for determining nucleic acid duplex stability is measurement of thermodynamic properties such as Tm and ΔG measured from UV spectroscopy. It has been known for many years that NMR "Tm" values estimated from imino proton exchange rates or changes in chemical shift related to changes in base stacking generally are lower than the corresponding optical Tm values. We wished to explore the sensitivity of the sugar coupling constants for detecting small changes in local conformation. Site-specific 13C enrichment offers a useful handle for measuring proton-proton and proton-carbon coupling constants from a single nucleotide via conventional 1D 1H-13C editing methods. The coupling constants can then be monitored as a function of temperature and converted into crude structural constraints.

A sample of dGCG(2',5'-13C2)UGCG and its compliment was prepared and kindly provided by Dr. M. Manoharan (Isis) and Prof. J. Gerlt (U. of Maryland). The desired sugar proton signals were edited out using a conventional high-resolution (0.1 Hz) 1D HMQC-TOCSY sequence. A "clean"-TOCSY (MLEV-17) was employed during the 100 msec locking time. Carbon decoupling was applied during the lock but not during acquisition in order to measure the long-range couplings. The temperature was varied over a range of 19-46°C, all below the optical "Tm" of the sample. The proton-proton, proton-phosphorous, and proton-carbon coupling constants were measured directly from the spectra, or estimated from lineshapes using the Varian-supplied simulation routine. The values are listed in the table below. The largest changes are observed in the 3'H-O-P, 3'-4', and 1'-2' coupling constants. The percentage of the S conformation has been calculated using standard models from the 1'-2',2" and the 3'-4' proton couplings, and decreases from 86% (19°C) to 72% at 46°C. The 2'-13C2 coupling decreases in magnitude as the 1'-2' and 2'-3' proton couplings increase. The results suggest the long and short-range H-C couplings can be used for the estimation of sugar conformation as an aid in determination of "premelt" structure in oligonucleotides.

J	Temp(°C) 19	28	37	46
1'-C5	1.2	1.2	1.2	1.2
1'-2'	7.6	8.0	8.5	9.0
1'-2"	6.8	6.8	7.0	7.0
2'-C2	144.5	143.2	141.7	140.5
2'-2"	13.3	13.7	14.3	14.5
2'-3'	3.7	4.0	4.3	4.3
2"-3'	2.0	2.0	2.3	2.3
3'-P	6.6	7.2	8.0	8.7
3'-4'	1.2	1.7	2.2	2.7
4'-5"	3.0	3.0	3.3	3.5
4'-C5	1.0	1.0	1.0	1.0

Sincerely,

Richard H. Griffey Patrick Wheeler

HERCULES

Hercules Incorporated
Research Center
500 Hercules Road
Wilmington, DE 19808, U.S.A.

November 16, 1992
(received 11/20/92)

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

Dear Dr. Shapiro:

Call for Papers - 1993 FACSS Meeting

The 20th annual meeting of the Federation of Analytical Chemistry and Spectroscopy Societies (FACSS) will be held in Detroit on October 17-22, 1993. As usual, the meeting will include symposia, instrument exhibits, workshops, and short courses. A major thrust of this meeting is the characterization of materials. For this purpose, NMR is one of the premiere techniques. In view of this emphasis on materials, I am organizing a symposium on the use of NMR spectroscopy in materials characterization.

At present, there are three or four sessions that are reserved for NMR. Original research papers are solicited in all areas of materials (organic and inorganic materials, polymers, blends, composites, metals and ceramics). The technique used may be solid state NMR or solution NMR. Papers involving theory, methods development, spectra/properties correlation, simulation, and analysis are all welcome. Hopefully a suitable balance of basic and applied research can be represented.

Anyone interested may write or call me before January 31, 1993; telephone: (302) 995-3505; fax: (302) 995-4117.

Yours very truly,

H. N. Cheng
Research Associate

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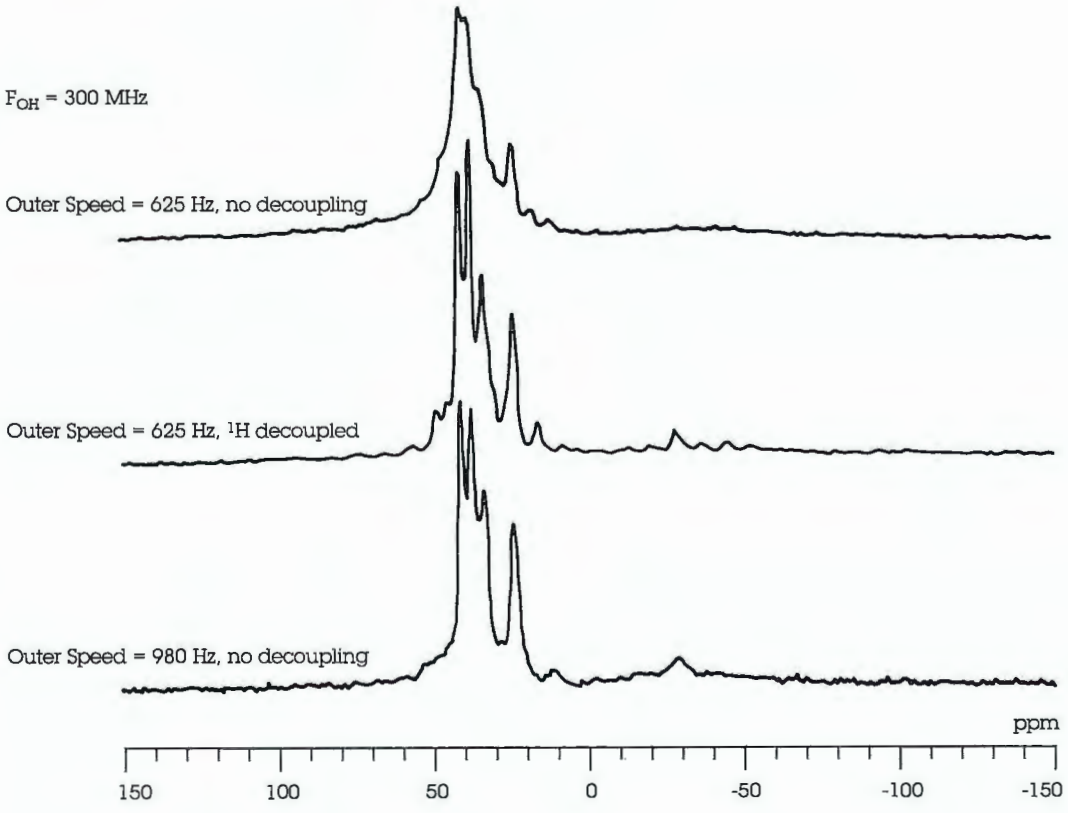

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Date 22nd October 1992 (received 10/31/92)

Dr B L Shapiro
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Detection of Hydration-Induced Mobility of Protein Amino Acids by Solid State ^1H NMR

Dear Dr Shapiro

Hydration of a dry protein initially results in increased mobility of parts of the molecule. This is of interest for a number of reasons, e.g. it relates to the moisture content above which enzyme activity is observed [1]. It may be possible to rationalize the effects of moisture content and temperature on molecular mobility in terms of the concepts of plasticization and the glass transition temperature, well-known for synthetic polymers [2]. We are interested in the potential of ^1H nmr for detecting the onset of mobilisation.

Figure 1 shows two ^1H spectra from a sample of ovalbumen (Sigma, Grade V) which was hydrated to 0.2 g H_2O/g dry solid over saturated salt solutions. The spectra were obtained using a Bruker 300 MHz CXP spectrometer incorporating a MAS double-bearing probe and with the sodium salt of 3-(trimethylsilyl)-1-propanesulphonic acid as a reference. A broad water peak centred at 4.8 ppm was suppressed by using T_2 weighting [3]. The remaining resonances from the protein protons exhibit additional peaks at 2.0 and 8.6 ppm when the temperature is raised from 30 to 50°C. We interpret this as mobilisation of CH_2 and NH_2 protons, possibly associated with lysine and arginine sidechains. These two resonances are also observed at 30°C when the moisture content is increased from 0.2 to 0.25 g H_2O/g dry protein.

Please credit this contribution to the account of Dr Mike Hey.

Sincerely

L P Hartley, S E Hill, J R Mitchell

1. Careri, G. & Rupley, J.A. 1991 Adv.Protein Chem. **41**, 37.
2. Slade, L. & Levine, H. 1991, Crit. Rev. Food Sci. Nutrition, **30**, 115.
3. Rabenstein, D.L. & Isab, A.A. 1979 J.Magn.Reson. **36**, 281.

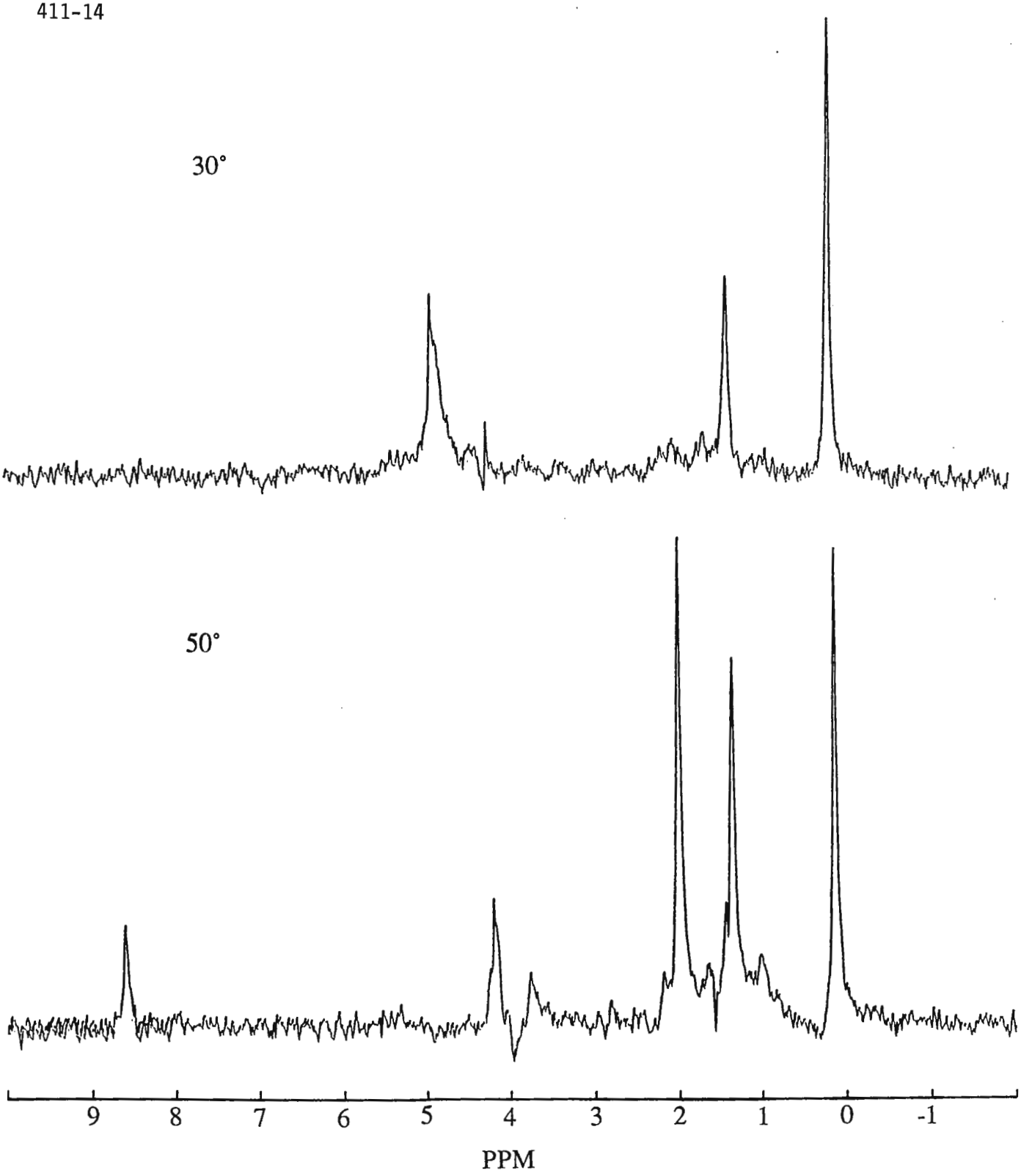

Fig. 1 T_2 Weighted Spectra for Ovalbumen Hydrated to 0.2 g H_2O/g dry solid at 30°C and 50°C.

CENTRE FOR MAGNETIC RESONANCE

PROFESSOR DAVID M. DODDRELL
DIRECTOR

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

(received 10/28/92)

Dear Professor Shapiro,

Shielded Gradient Designs for *In Vivo* IDS in Vertical Magnets

We have spent much number crunching time over the last year optimizing the design of shielded gradients for Image Directed Spectroscopic (IDS) applications in vertical bore magnet systems. The design process has developed from the animal outwards and resulted in Birdcage resonator/Surface Coil receiver RF probes with a controllable patient positioning device setting the gradient coil inner dimensions. Gradient sets and RF probes have been developed for wide bore (WB) and super-wide bore (SWB) magnet systems with the gradients exhibiting excellent linearity and shielding characteristics. Figure 1 is a 2% contour plot of the departure from a perfectly linear gradient of a SWB transverse gradient. A typical WB gradient produces 0.5 G/cm/A and the SWB 0.35 G/cm/A. Figure 2 shows a radial field plot of the same gradient and indicates the shielded of the set (< 1.0% rms shielded/unshielded field at 5 mm radially from the outer diameter of the set). Figure 3 is a landscape view of the field from a longitudinal WB coil.

Please credit this contribution to Prof. D.M. Doddrell and thanks for the dreaded ultimatum.

Stuart Crozier Craig D. Eccles Wolfgang Roffmann

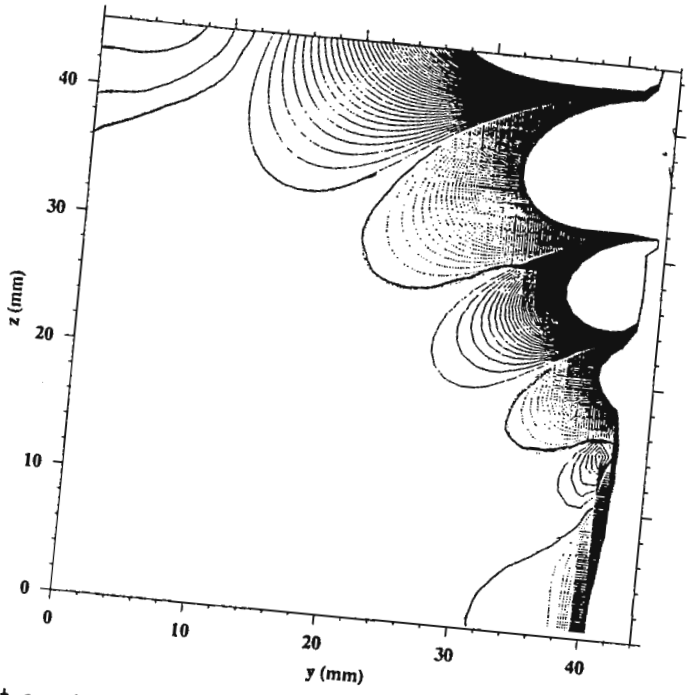


Figure 1 2% gradient contours from a SWB (85/115 mm) transverse gradient

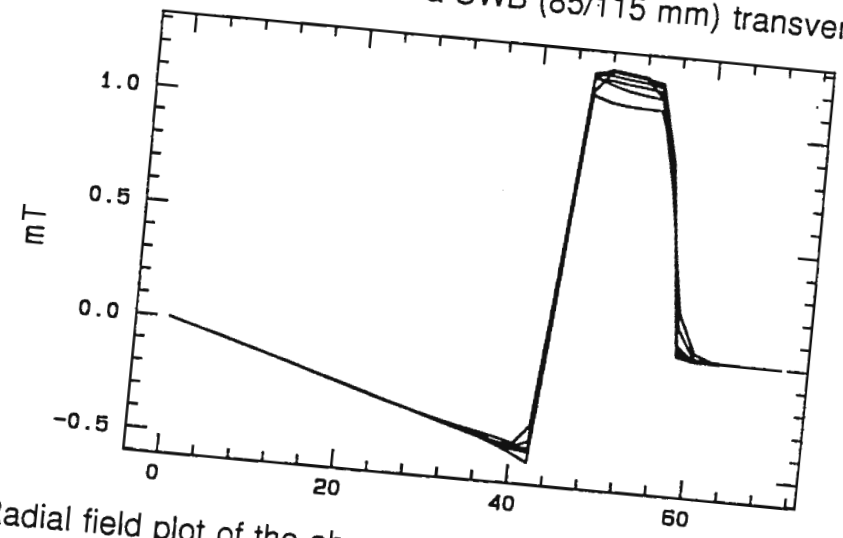


Figure 2 Radial field plot of the above gradient coil

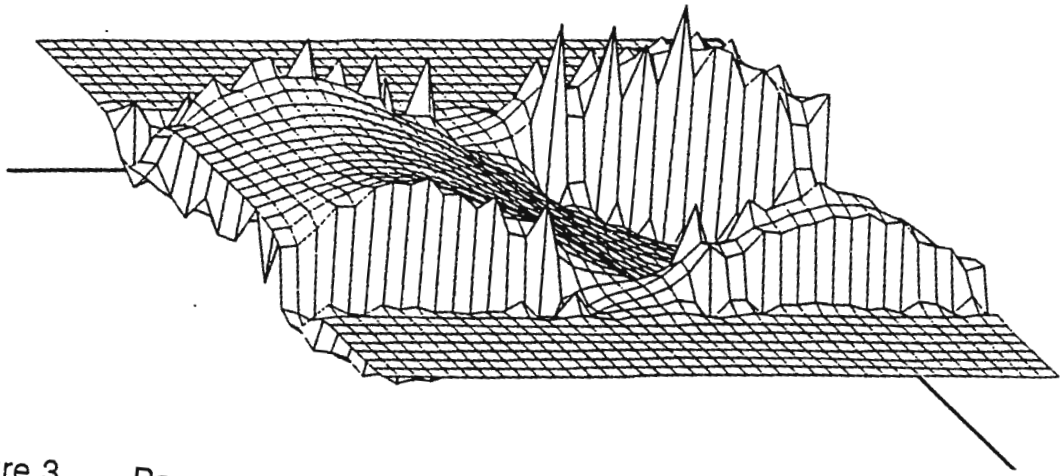


Figure 3 Panoramic Z gradient field.

Peace on Earth

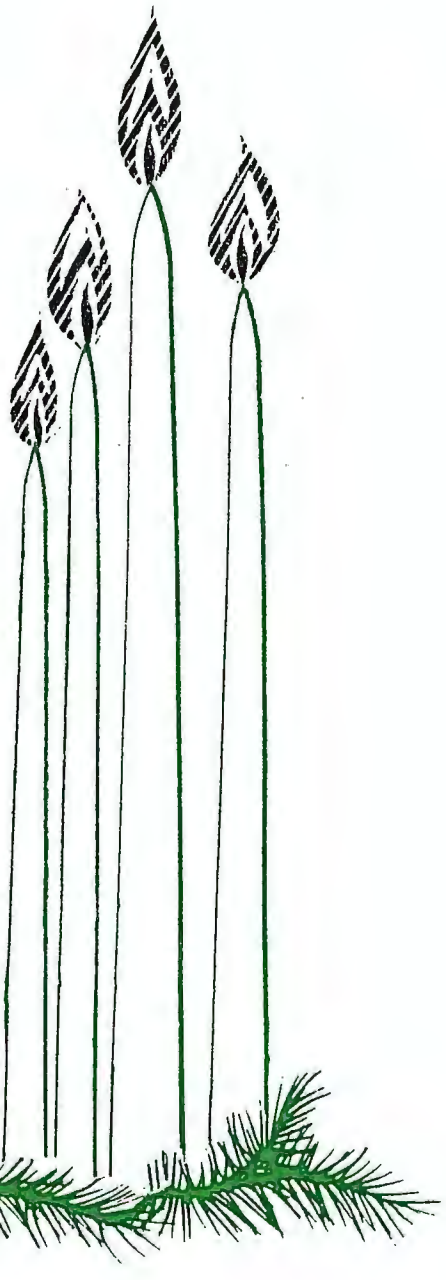
I heard the bells on Christmas Day
Their old familiar carols play,
And wild and sweet the words repeat
Of peace on earth, good-will to men.

I thought how, as the day had come,
The belfries of all Christendom
Had rolled along the unbroken song
Of peace on earth good will to men.

And in despair I bowed my head:
"There is no peace on earth," I said.
"For hate is strong, and mocks the song
Of peace on earth, goodwill to men."

Then pealed the bells more loud and deep:
"God is not dead, nor doth He sleep;
The wrong shall fail, the right prevail,
With peace on earth, goodwill to men."

Henry W. Longfellow, 1807 - 1882



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October 20, 1992
(received 10/29/92)

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

Noise Bridge for Impedance Matching

Dear Barry,

We would like to start a subscription in Dr. Heinrich Roder's name. What does it cost?

This circuit serves to tune and match an NMR probe quickly at any of its design frequencies. The idea has been around for a while and is described for example in the ARRL Handbook for matching antennas (1). The present circuit is applied to the tuning/matching of NMR probes and has some modifications which make it more suitable for that purpose. The use of an audio detector and meter for example was found superior to listening to a speaker because the null we achieve is not as deep as with an antenna match, even though the resulting match is a good 50 ohms.

A non-mathematical explanation of the relative shallowness of the null: the RF noise excites ringing in the high Q tuned circuit of the probe in the same way that an RF pulse would do. The ringing appears at the noise bridge as though it were part of the reflection due to mismatch, even though it is not. Fortunately, minimizing the "NULL" signal still results in a good 50 ohm tune/match.

In a more conventional tune/match scheme either cw or swept cw would be fed into an RF bridge. The probe port's impedance would be compared to 50 ohms; the bridge output would be detected and a null reached when the bridge was correctly balanced. In our system (Fig. 1) an uncalibrated noise source (IN751 Zener diode) provides very broadband RF noise (more than 300 MHz) which is amplified and passed through a Mini Circuits directional coupler PDC-10-21 which serves nicely as an RF bridge from 1 to 1000 MHz. The spectrometer is tuned to a narrow band in the normal way and produces audio noise, which is minimized by tuning and matching the probe. It is as though we were using a noisy signal generator instead of a broadband noise source.

The procedure is simple enough to be done quickly at the start of an NMR experiment:

1. Disconnect the probe cable from the T/R and preamp. Do not yet connect it to the noise bridge's "PROBE" port.
2. Connect the T/R and preamp to the noise bridge's null output.
3. Connect the audio output of the spectrometer to the noise bridge's audio input (it may be left there permanently).
4. The bridge is now badly mismatched since the probe is not connected. Find the receiver gain setting of the spectrometer to give somewhat less than one volt on the Digital Voltmeter (20V full scale, 3-1/2 digits).
5. Connect the probe cable to the "PROBE" port of the bridge and minimize the DVM reading, ultimately concentrating on the final digit only. Experience may show you that only the TUNE control of the probe needs to be adjusted, although you can check by iterating the MATCH control and retuning. The nulling does not have as nice a feel as when an RF sweeper is used, partly because the null is not as deep, but there is excellent agreement between the results of the two methods.

ACKNOWLEDGMENTS

I wish to thank Dr. Britton Chance for putting me onto the idea of using a noise bridge. This work was supported by National Institutes of Health Grants CA-06927 and RR-05539, and also by an appropriation from the Commonwealth of Pennsylvania.

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1. The Radio Amateur's Handbook, 1989, Chapter 25, pp. 32-34.

Sincerely,

James L. Engle

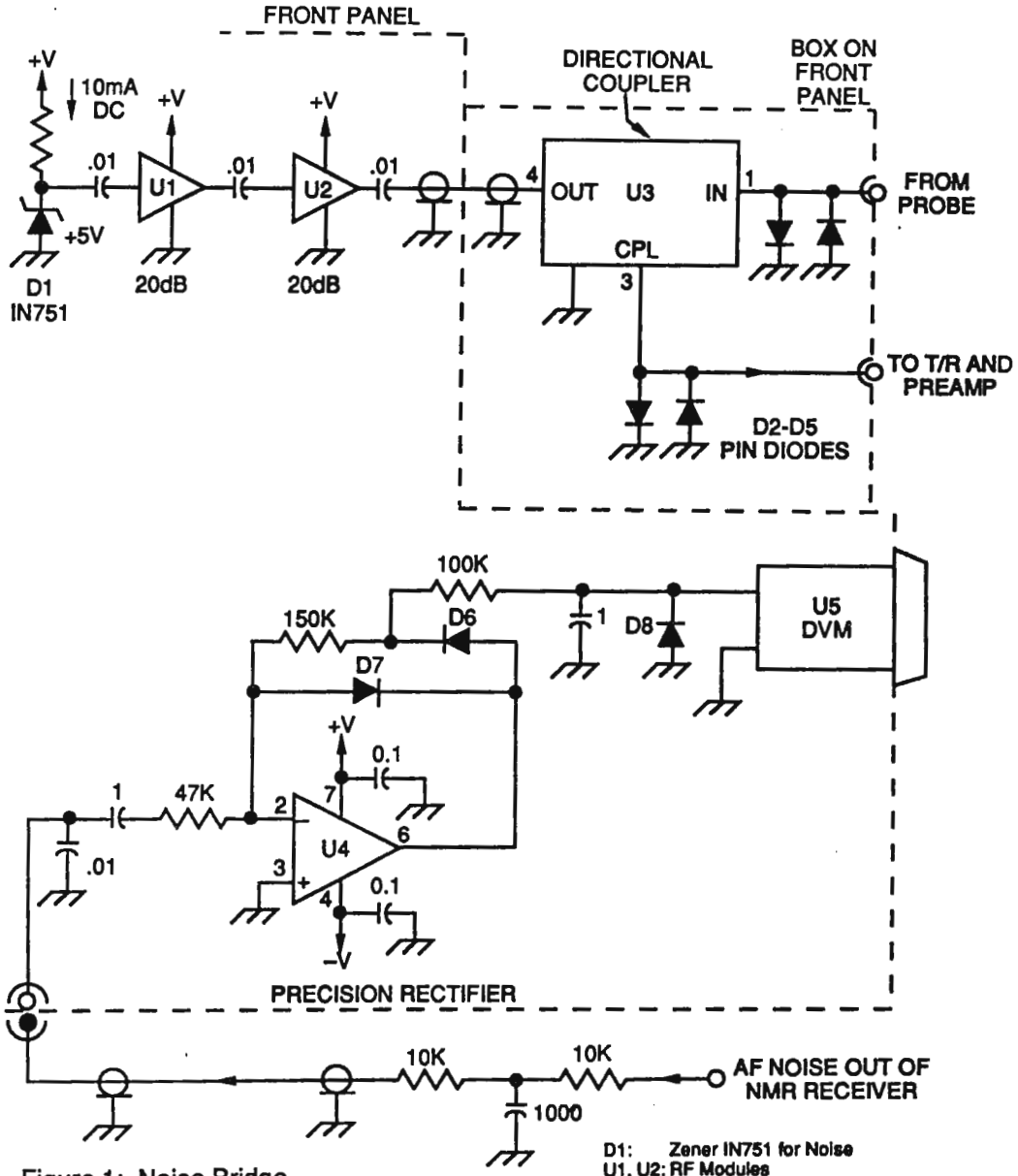

Figure 1: Noise Bridge

D1: Zener IN751 for Noise
U1, U2: RF Modules
U3: PDC-10-21 1 to 1000 MHz Mini Circuits
U4: LF351 Operational Amplifier
U5: DVM 20v Full Scale
D2-D5: GC43077-15
Frequency Sources, Inc.
D6-D8: IN914

The University of Akron

Department of Chemistry (216) 972-7372
Akron, Ohio 44325

October 29, 1992 (received 11/7/92)

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

Title: $^{1}H/^{19}F/^{13}C$ Triple Resonance NMR Spectra of Polymers

Dear Dr. Shapiro:

For sometime we have been using triple resonance techniques in conjunction with isotopic labeling to study problems in polymer chemistry.[1] Attached are the first spectra generated via the use of $^{13}C\{^{19}F\}$ INEPT experiment. Utilization of this experiment enables one to obtain a clear view of the carbons which are fluorinated without the interference of the non-fluorinated carbon resonances.

^{13}C $\{^{19}F\}$-INEPT spectra were obtained on a Varian VXR 300–MHz spectrometer modified with a home-built broad-banded fourth rf channel to permit simultaneous decoupling and pulsing at the ^{1}H, ^{19}F and ^{13}C resonance frequencies while maintaining a ^{2}H lock. This channel was connected to four software-controlled auxiliary lines on the spectrometer where one line controls pulse-gating of the decoupler, two lines are used for generation of phase shifting and one line simultaneously controls a hardwired WALTZ modulator and an attenuator. The $^{13}C\{^{19}F\}$-INEPT spectra were obtained with a 75 watt ^{13}C 90° pulse of 8.0 μs, a 0.2 s acquisition time, and a 1.0 or 2.0 s relaxation delay was used for polymers and small molecules, respectively. Combining the outputs from the instrument's standard ^{1}H and ^{19}F decoupler rf channel and that of the broadband channel constructed at The University of Akron was accomplished via the use of an auxiliary power combiner. The output from the power combiner was fed through the ^{1}H / ^{19}F decoupler coil of a Varian 4 nuclei probe to obtain continuous ^{1}H WALTZ-16 modulated decoupling and ^{19}F irradiation .

Sincerely yours:

D. R. Hensley, M. Tokles, G. S. Hatvany, H. J. Harwood, P. L. Rinaldi

[1] Rinaldi, P.L., N.J. Baldwin, *J. Am. Chem. Soc.*, 105, 7523, **1983**.

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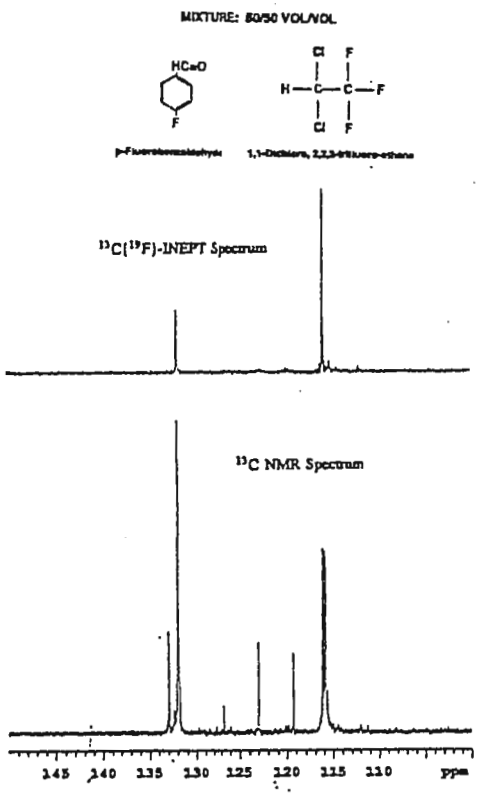

Figure 1. ^{13}C NMR spectrum and $^{13}C\{^{19}F\}$- INEPT spectrum of a 50/50 by volume mixture of p-Fluorobenzaldehyde and 1,1-Dichloro, 2,2,2-trifluoro-ethane.

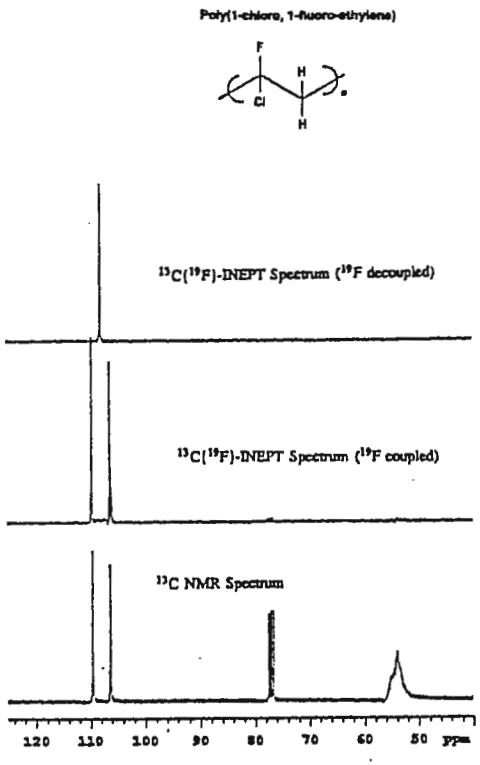

Figure 2. ^{13}C NMR spectrum and $^{13}C\{^{19}F\}$- INEPT spectra of Poly(1-chloro, 1-fluoro-ethylene).

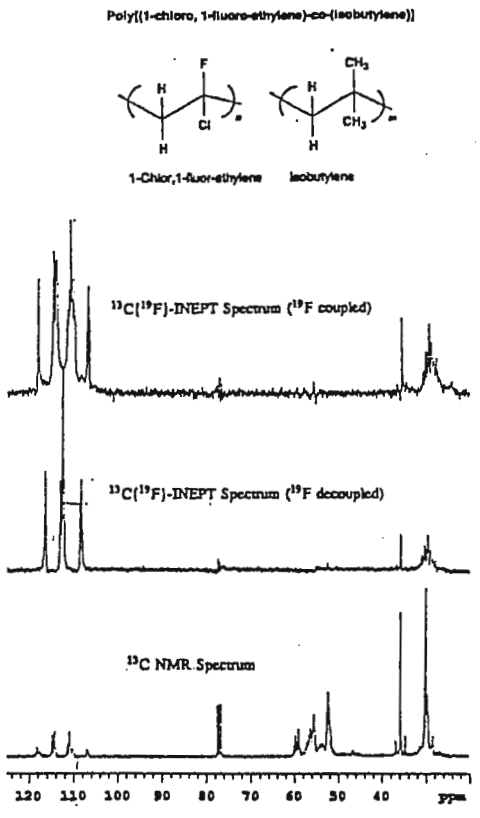

Figure 3. ^{13}C NMR spectrum and $^{13}C\{^{19}F\}$- INEPT spectra of Poly[(1-chloro, 1-fluoro-ethylene)-co-(isobutylene)].

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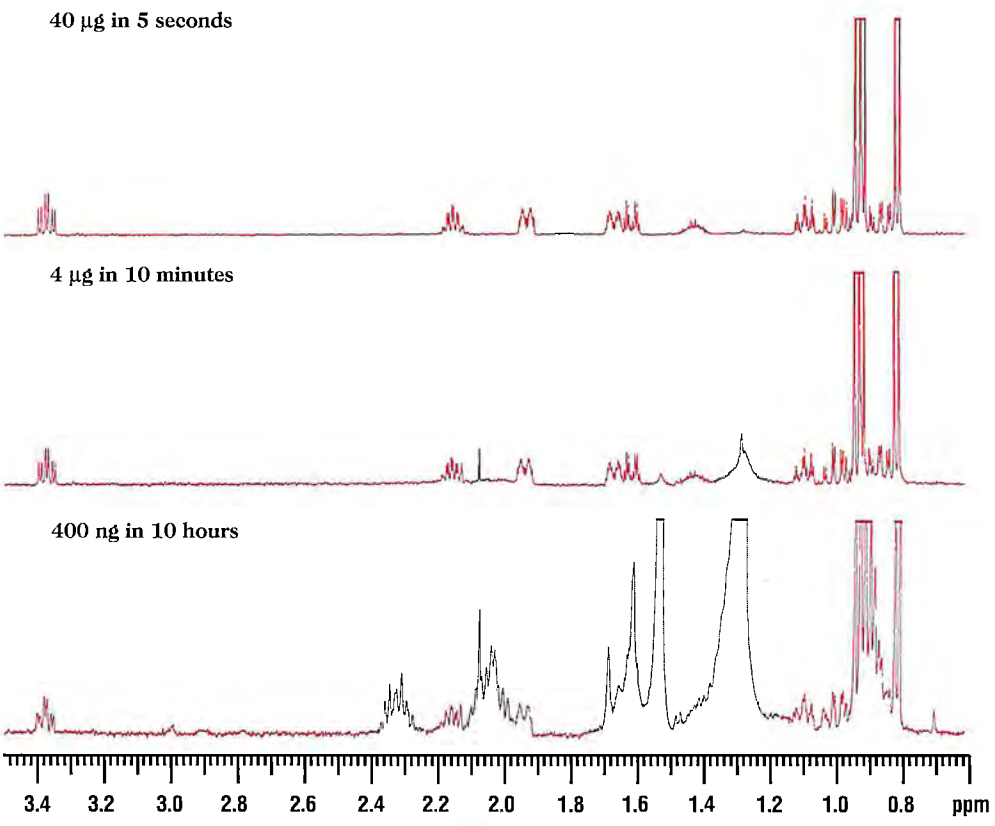

These spectra of menthol in CD_2Cl_2 were obtained on a UNITY 500 MHz spectrometer with the Nano•nmr probe.™ The new peaks in the lower spectra (shown in black) are from solvent impurities.

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

(received 11/2/92)

Dear Dr. Shapiro,

SEARLE

Title: Spurious Affliction Spurned Ethically

Proton spectra from our XL-200 NMR spectrometer have been afflicted by the presence of spurious signals, for some time. Fortunately, most of the spurious signals fall outside of the chemical shift range of 10 ppm and are usually out of phase with the real NMR signals. However, the magnitude of these artifacts vary unpredictably from sample to sample. Increased signal averaging reduces the magnitude of the spurious signals relative to the real NMR signals, at a significant cost of experimental time.

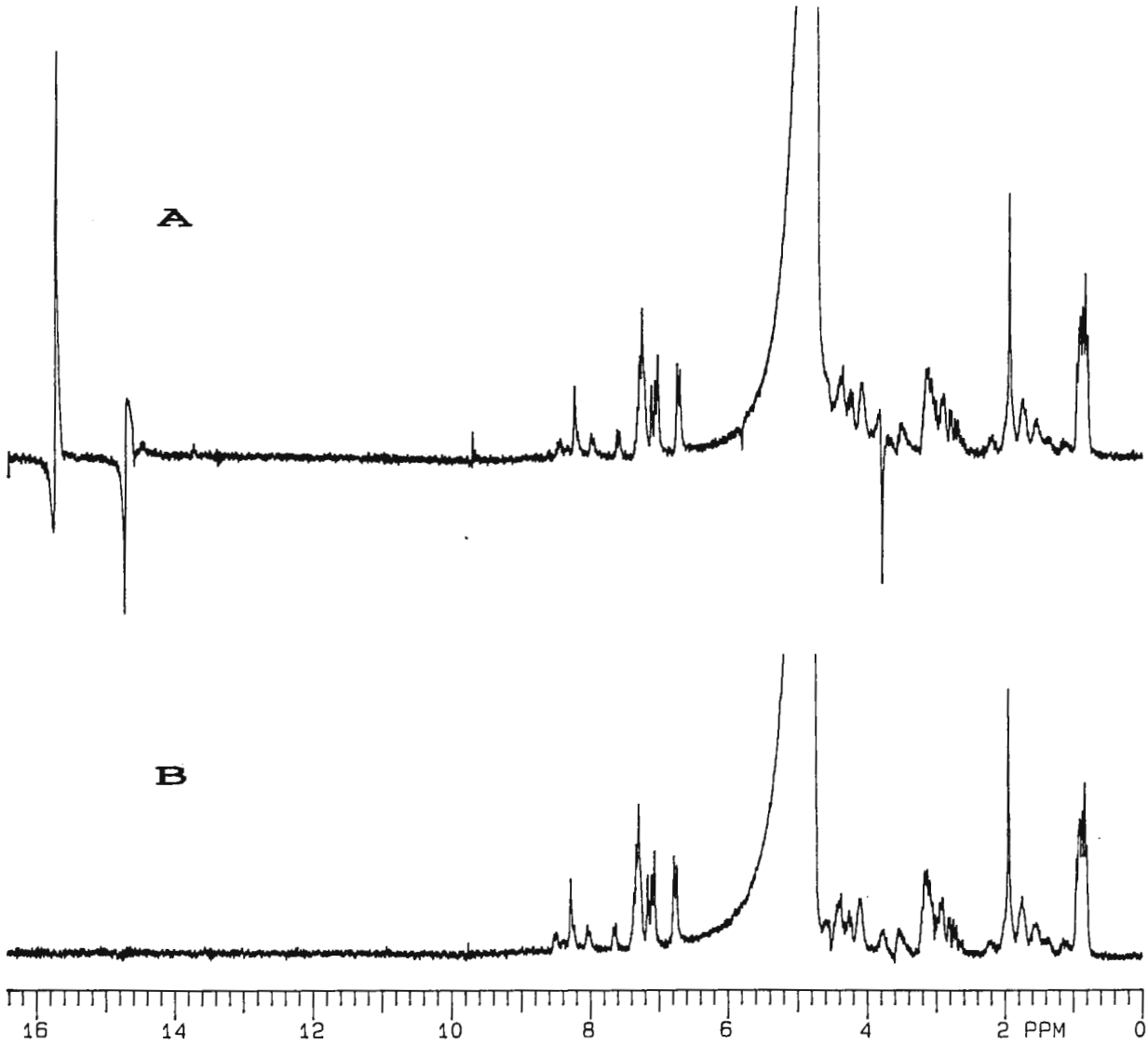
We have determined that the interference is due to a portion of the pulsed lock transmitter coupling into the observe preamplifier through the probe. Hence, removing the lock cable from the probe eliminated the interference. Operating without the deuterium lock is, of course, not an appropriate remedy.

This troublesome interference has been eliminated by installing a deuterium trap filter in the proton transmission line at the probe. In this filter, frequencies in the vicinity of 30.7 MHz are attenuated by 30 dB, which is sufficient to avoid cross-modulation interference in the preamplifier. The filter was constructed in a shielded enclosure with BNC connectors to facilitate connection to the probe.

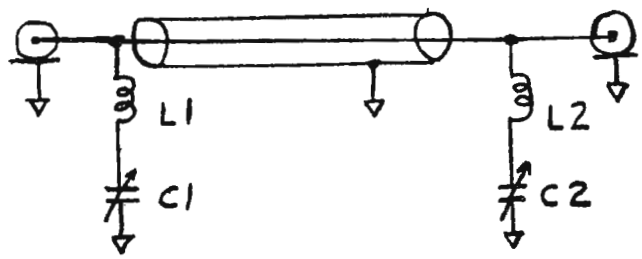
The improvement in the quality of proton NMR spectra due to this inexpensive filter is illustrated by the spectra shown in Figure 1. The filter is shown schematically in Figure 1, also.

Best wishes,

Robert W. Dykstra



C



C1, C2 = 5-25 pF
L1, L2 = 20 turns 0.012" o.d. enameled copper wire, tightly wound on a 0.225" o.d. former. (a 1 meg ohm, 1 watt resistor provides a suitable former)

Figure 1. A and B are proton NMR spectra (200 MHz) of 5mM angiotensin II in D2O from 128 transients using a presaturation experiment. Spectrum A is from the XL-200 before the modification, and B was taken after the deuterium trap filter (shown in C) was installed at the probe.

WESTERN RESEARCH INSTITUTE
The University of Wyoming Research Corporation

November 4, 1992 (received 11/9/92)

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

RE: Application of Solid State NMR to Environmental Science

Dear Barry,

We are presently investigating the use of solid state NMR in solving problems of an environmental nature. We have conducted some very preliminary experiments to see if we could observe pentachlorophenol (PCP) on coal fly ash which was washed with water. PCP is a wood preservative for railroad ties and has become an environmental concern at some EPA Superfund Sites. However, PCP may be stabilized/immobilized by fly ash. Such stabilization phenomena are useful in waste minimization, i.e., finding a use for the waste fly ash through its use as an absorbent of the hazardous waste, PCP. Figures 1a and 1b show the ^{13}C single pulse excitation (SPE) spectrum of solid PCP and the ^{13}C SPE spectrum of 1 g of coal fly ash on which 0.02 g PCP was deposited. Resonances below 90 ppm are due to probe background. The C-1 carbon of PCP in Figure 1a had shifted in the spectrum of coal fly ash with sorbed PCP. This shift suggests that the phenolic carbon is aligned near the surface of the fly ash. The ^{13}C SPE spectrum of the ash leached with water shown in Figure 1c appears to be similar to spectrum 1b. Thus, it can be assumed, without additional data to the contrary, that the PCP is strongly adsorbed on the surface of the coal fly ash. The enhanced intensity of the C-1 carbon shown in Figure 1c remains an unexplainable observation at this time. The CP/MAS spectrum of the water leached fly ash (Figure 1d) shows little or no C-1 carbon signal even under different contact times. It is assumed that the paramagnetic impurities in coal fly ash may have shortened the ^{1}H relaxation time such that the cross polarization for this H-O-C bond becomes quite inefficient. If other readers have another explanation we would like to hear from them.

Sincerely,

D.A. Netzel F.M. Miknis D. Lane

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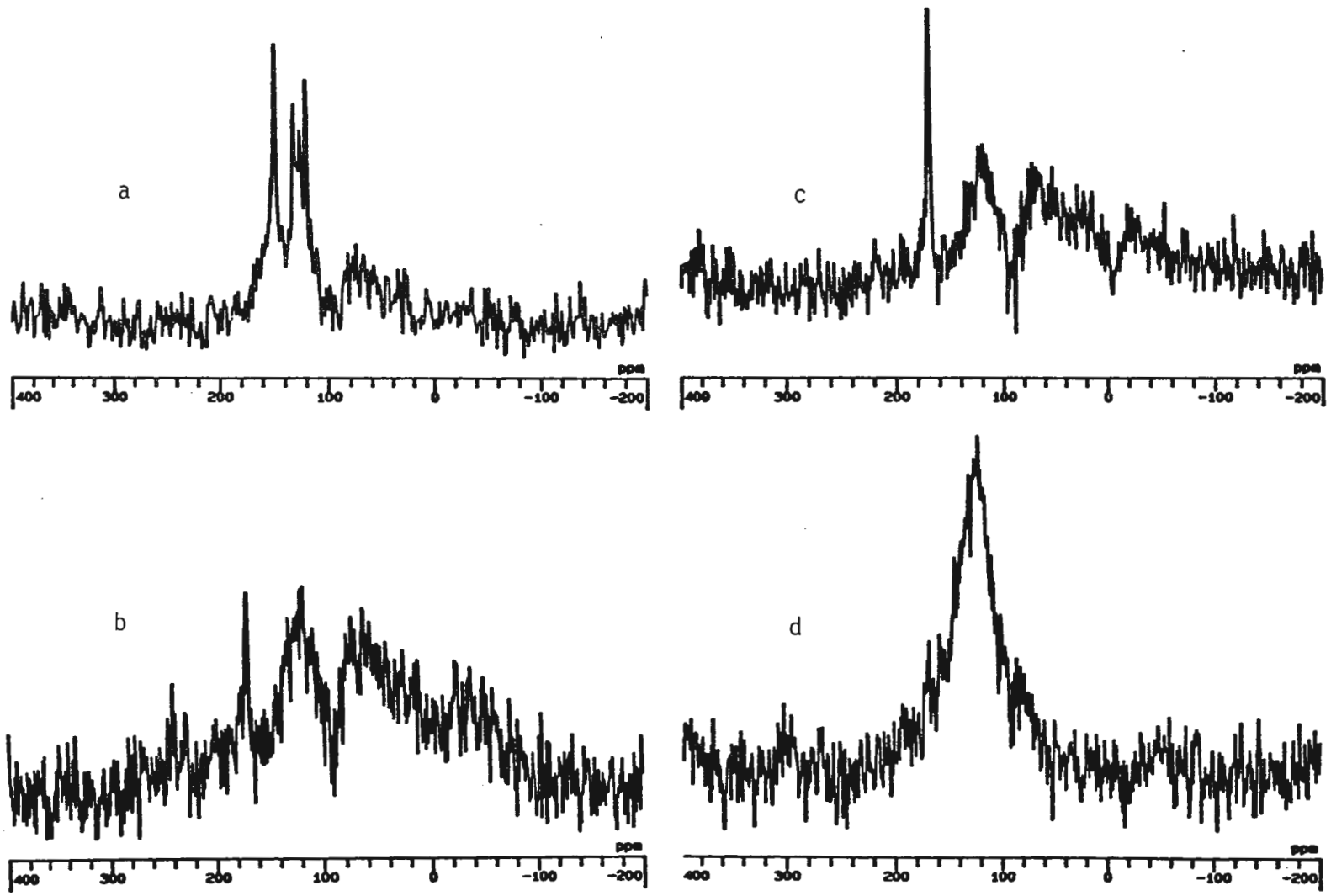

Figure 1. a) ^{13}C SPE Spectrum of Pentachlorophenol (PCP), b) ^{13}C SPE Spectrum of PCP on Coal Fly Ash, c) ^{13}C SPE Spectrum of PCP on Coal Fly Ash after Leaching with H_2O, d) ^{13}C CP/MAS Spectrum of PCP on Coal Fly Ash after Leaching with H_2O.

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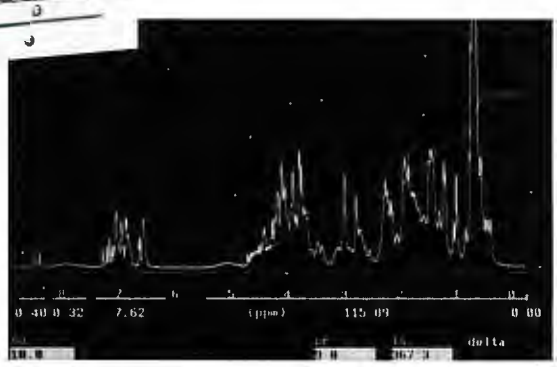

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Amplitude droop	5% to 20 ms typ.
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Front panel controls	1. AC power 2. Pulse width 3. Duty cycle
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October 12, 1992
(received 10/30/92)

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

Processing and Archiving Aspect 3000 Files using UXNMR/P and MacX

Dear Dr. Shapiro:

Approximately a year ago we decided to use a Sun SPARCstation 2 as both a workstation and an archiving center for our Bruker AM 300 NMR spectrometer. The ultimate goal was to create a NMR database and NMR processing system that would be accessible to Macintosh users. We concentrated our efforts on Macintosh computers since our principal customers who are synthetic chemists primarily use Macintosh computers.

The first step that we used in setting-up a useful NMR processing network between our Aspect-derived data and Macintosh computers involved the placement of a UNIX workstation as a node between the Macintosh and Aspect computers. This was accomplished using a network configured with a SPARCstation 2, Aspect 3000, and various Macintosh computers. Each computer was connected to an Ethernet network that supported either Brulinet (Bruker's Ethernet protocol) or TCP/IP communications protocols. This type of configuration permits data files that are acquired on the Aspect 3000 computer to be transferred to the SPARCstation using Bruker's Brulinet communications protocol. The Macintosh computers can then access the data on the SPARCstation via our facility network that supports the TCP/IP communications protocol. To process the data, the Macintosh computers on the facility network would run X applications remotely from the SPARCstation using Apple's MacX. The NMR data is both processed and stored on the SPARCstation, while the Macintosh computer is simply used as an X-terminal for the SPARCstation.

The processing software used on our SPARCstation is Bruker's UXNMR/P version 1.1.1. To open an X window session on the Macintosh computer, we used the following remote command: usr/tape/binSun4/xterm -display "display address":0.2. The "display address" in the remote command directs the output of "xterm" to the appropriate Macintosh terminal. The .2 at the end of the remote command is necessary to define a color rootless window on the Macintosh terminal. The only other parameters which must be defined were the connection setting method (MacTCP tool) and the proper host name. The host name is the IP address for the

SPARCstation in the /etc/hosts file. After opening an X window session, either UXNMR/P can be executed for NMR data processing or an additional X window can be opened for another application, e.g. search routines.

Currently, spectra processed on the SPARCstation are plotted on a SPARCprinter which requires an SBus printer card and NEWSprint software as a postscript filter. Spectra that are processed remotely on the Macintosh are plotted either on the SPARCprinter or the Apple laser printer. Spectra that are plotted on the Apple laser printer require that a plotfile be directed into FrameMaker®. Once the plotfile is in FrameMaker®, the spectrum can be further modified before plotting on the Apple laser printer.

After we provided processing capabilities to our synthetic chemists, the next step was to provide a NMR database that would permit keyword search capabilities of archived data. We envisioned a search routine using the DISNMR TI (title) information already present with each Aspect-derived FID. We could then correlate each archived file name to a keyword description of that file, i.e. title information. This was an important aspect since we had approximately 12,000 files archived on 2400 ft streaming magnetic tapes and did not want to go back and enter any additional information for each file.

The database was assembled by first converting all the 24 bit ASPECT-derived data to a 32 bit format compatible with UXNMR/P. This was accomplished by doing a global conversion ,supplied by Dr. Beat U. Meier of Spectrospin AG, for each month of the Aspect 3000 archived data. As part of the conversion routine a title file is created for each FID (..../#/pdata/#/title) containing the title information that was entered with the DISNMR TI command. Following the global conversion, a find command is executed for that month's data (find . -name title -exec cat {} \; -exec echo "" \; > title.month). The find command locates the title file of each converted file, closes the contents in the title file, and appends the title to a file named title.month. The title.month file is ,therefore, the summation of all the title files that are created by the global conversion of a particular month. Each title.month file is then appended to a single master file (title.all). Simple shell script routines then search the title.all file using some variation of the grep command.

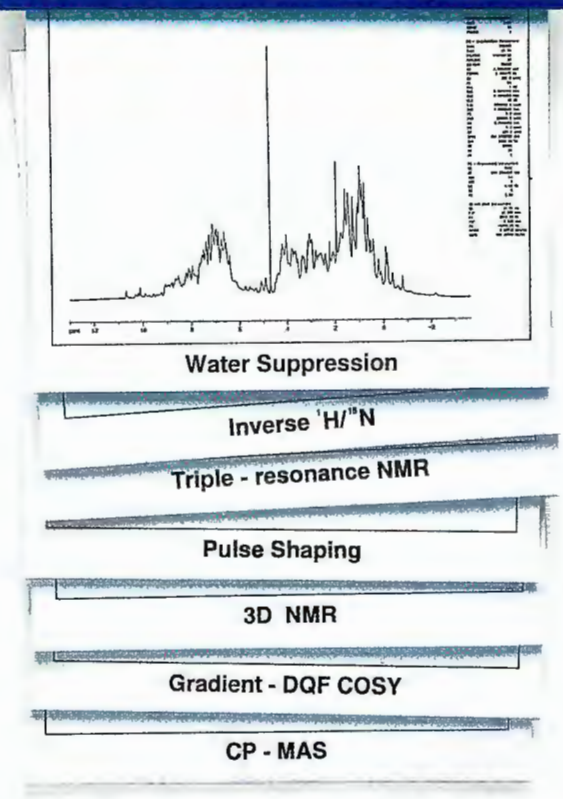
At the present time we archive all the 1D FIDs on 1.3 Gbyte 4mm DAT tapes. The 1D files are archived in 50 Mbyte tar files which also can be searched. However, as you can imagine, this is not a very satisfactory means of recalling data. For the immediate future we plan to install a 2.5 Gbyte magnetic disk that will contain only 1D files. Eventually, we plan to install a mass storage optical disk jukebox on the network that will hopefully accommodate all our archiving needs.

Best Regards,

Kurt

Kurt Wollenberg

ARX



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

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

Console	Dimensions and weights are approximate; voltage +10/ – 5% max. variation; other line freq. and voltage upon request
dimensions electronics cabinet	w 130 x d 0.75 x h 1.20 m
weight	450 kg
power (dissipation)	220 V/50 Hz/16 A (ca. 4 kW)

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HEALTH SCIENCES CENTER
DEPARTMENT OF BIOCHEMISTRY

November 12, 1992
(received 11/16/92)

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

High Resolution NMR Studies of Human Glutathione Transferase Mu

Dear Dr. Shapiro:

On this side of the Atlantic (see TAMU 409-13) we have been using NMR to study a class mu glutathione transferase from Human Muscle. This class of glutathione transferases appears to play a role in the protection of an individual from carcinogenic aromatic hydrocarbons.

One of our interests is to obtain information on the effects of ligand binding on the dynamics of the enzyme by measuring relaxation rates and exchange kinetics of amide groups in ^{15}N labeled proteins. An example of ligand induced changes is shown in Figures A and B. Figure A shows the 2D HMQC spectra of ^{15}N Tyr labeled protein in the presence of product (glutathione-dinitrobenzene). There are 12 Tyr residues in the monomer and it is clear that all 12 Tyr signals are present in this spectrum. However, if the product is absent (Figure B) several changes occur in the spectrum. First, the resonance from Y116 completely disappears, presumably due to exchange broadening. A number of additional resonances are either broadened or split (Y33, Y23, Y62) when the ligand is removed. All of this indicates that there is considerable flexibility in the protein in the absence of ligand and addition of ligand appears to tighten up the protein. We are in the process of measuring amide exchange rates and relaxation parameters of a number of isotopically labeled sites to more completely map out changes in protein dynamics due to ligand binding.

What has tacitly been assumed in the above discussion is the assignments of these 12 resonances lines from the Tyr amide group. The big problem with working with proteins as large as GST (52-54KDa) is trying to get resonance assignments. It is pretty clear that because of the fast proton spin-spin relaxation rate that sensitivity will be a problem in coherent transfer of proton magnetization. Although in the long run ^{15}N edited NOESY and 3D Heteronuclear NOESY spectra will give some sequential assignments (problems of spin diffusion not withstanding), we have initially elected to take a simpler route to these assignments.

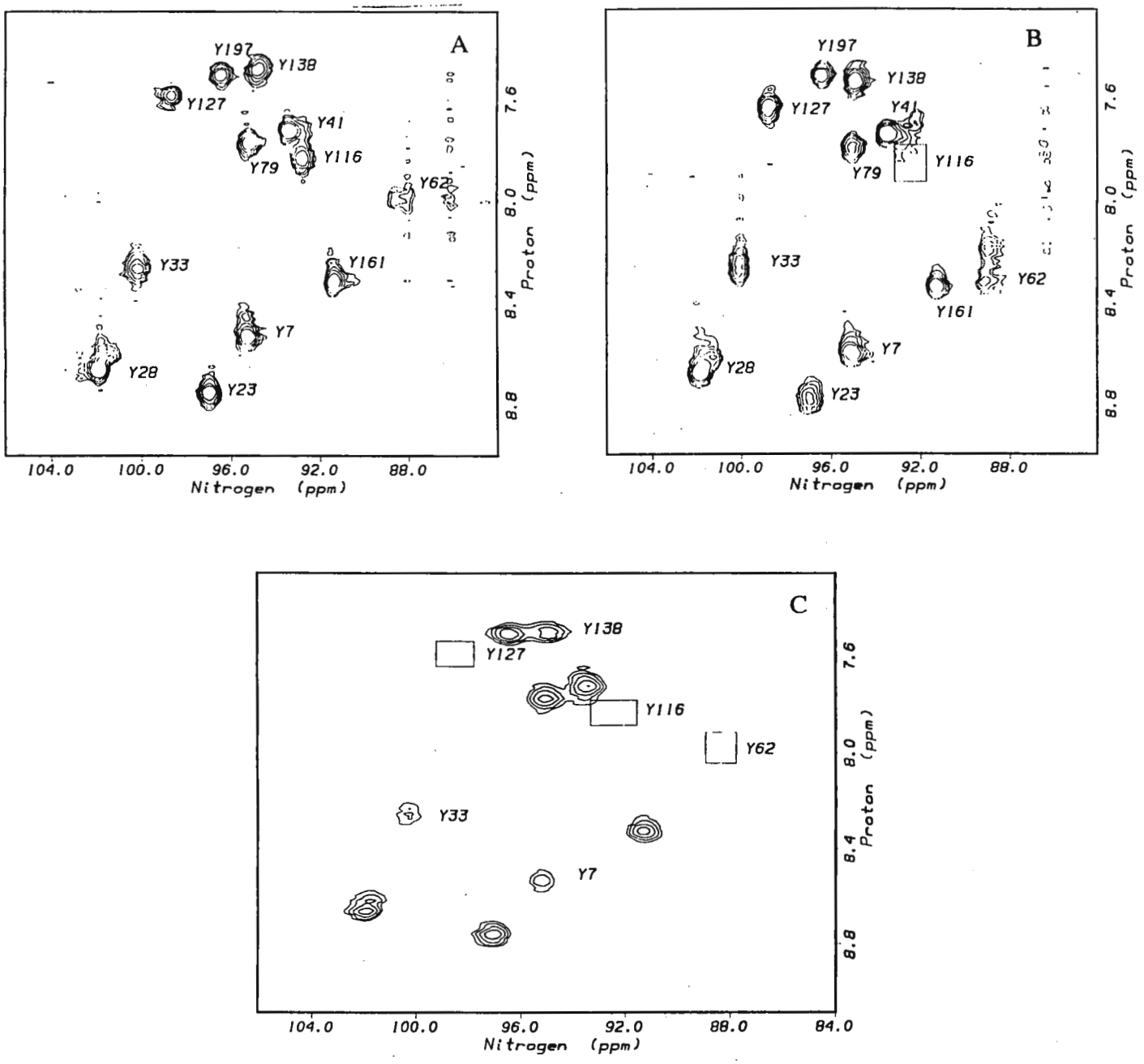
Paramagnetic broadening has been extensively used to obtain distances between nuclear and electron spins. In the case of glutathione transferases, the unpaired electron is introduced into the system by coupling a nitroxide group to the SH group of glutathione. Nitroxide labeled glutathione is an ideal relaxation probe for these enzymes because it targets the nitroxide to the active site, and when bound the nitroxide group becomes immobilized. If the crystal structure of the protein is known (as is the case for a number of transferases), then the paramagnetic broadening can be used to obtain assignments. A HMQC spectrum of ^{15}N-Tyr labeled enzyme, obtained in the presence of spin-labeled glutathione, is shown in Figure C. A number of resonances are broadened and thus disappear from the contour plot. The assignments of those which do broaden are indicated in Figure C. The remaining 6 Tyr residues are outside the range of detectable broadening (about 17Å) and these cannot be assigned using distance measurements. To assign the remainder of these Tyr resonances, and to check on the validity of the assignments based on

distance measurements, a panel of 12 Tyr → Phe mutants was constructed. HMQC spectra of each of these mutants showed the absence of a single peak, without large chemical shift changes in the remaining 11 peaks.

In summary, a large number of assignments of resonances that arise from residues within 17Å of the active site can be obtained by the combined use of selective labeling, paramagnetic broadening agents, and the judicious use of site-directed mutagenesis.

Please credit this contribution to Jeff Ellena (U.Va. Department of Chemistry).

Gordon S. Rule



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PS/BMNC/92/37

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

12 November, 1992
(received 11/16/92)

Dear Barry,

DEUTERIUM NMR IN DRUG METABOLISM

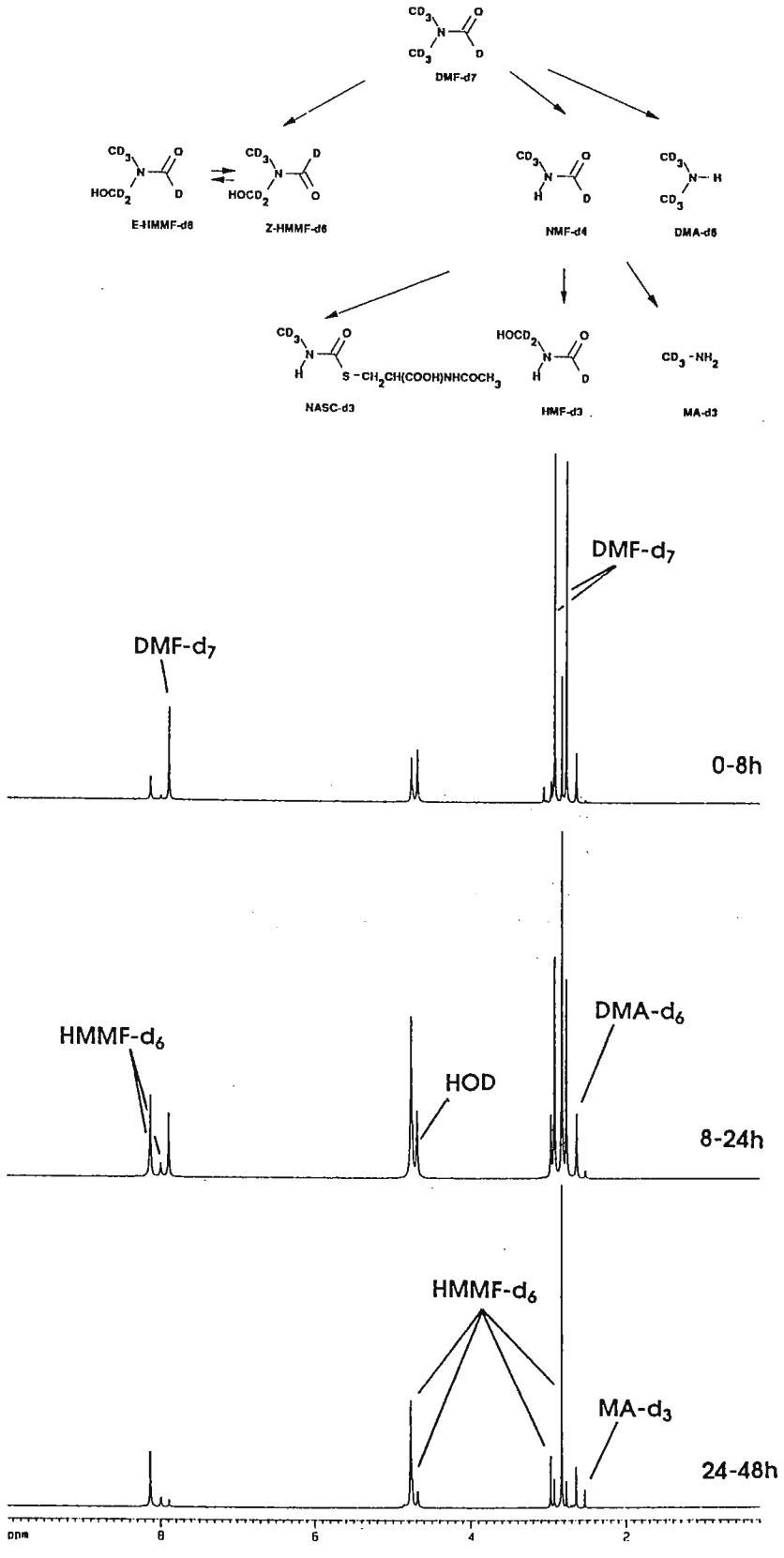
We have been investigating the usefulness of deuterium NMR of small molecules as a way of elucidating drug metabolic routes. Deuterium NMR has some advantages and some disadvantages over other isotopic probes for metabolism. and has been used in a number of mass spectrometry approaches. It is not radioactive like ^{3}H or ^{14}C and so the contamination possibilities are minimised, there is little background signal if significant enrichment is used, lines are relatively sharp for small molecules, T1s are short, sensitivity is nearly as good as ^{13}C for equal numbers of nuclei but on the other hand not as good as ^{1}H or ^{3}H and the chemical shift range is rather small in frequency terms.

Anyway, to test out the approach, we dosed a rat with 1g/kg of DMF-d_7, the well known NMR solvent and anticancer agent, collected the urine and measured its ^{2}H NMR spctrum at 92.1 MHz (corresponding to 600 MHz for ^{1}H). The figure shows the result of 128 scans for urines collected from 0-8 hr, 8-24 hr and 24-48 hours after dosing. Clearly the DMF-d_7 signals can be seen to decrease over this period, with both rotameric isomers of the major metabolite, HMMF-d_6 (abbreviations given in the scheme), increasing. Closer examination of the spectra, using known ^{1}H chemical shifts, also enabled us to pick out several minor metabolites, namely the N-acetylcysteine adduct of NMF-d_4, HMF-d_3, methylamine-d_3 and dimethylamine-d_6, and, as they are deuterated, the last two cannot be the endogenous forms of these molecules.

It seems that deuterium NMR at high field of intact biofluids may have some potential in drug metabolism and we are exploring this further.

Yours sincerely,

John C. Lindon Jeremy K. Nicholson R. Duncan Farrant Belinda C. Cupid


DMF-d7
E-HMMF-d6
Z-HMMF-d6
NMF-d4
DMA-d6
NASC-d3
HMF-d3
MA-d3
DMF-d7
DMF-d7
0-8h
HMMF-d6
HOD
DMA-d6
8-24h
HMMF-d6
MA-d3
24-48h
ppm
8
6
4
2

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Output	Level:	+3 to +13 dBm (1V into 50 Ω), metered in dBm and volts (rms)	
	Flatness:	±0.5 dB	
	Impedance:	50 Ω	
	Control:	Manual by front panel control; remote by analog voltage	
Spurious Outputs	Discrete:	-70 dBc (-55 dBc, 1/2 & 3/2 f_{out} above 310 MHz)	
	Harmonics:	-30 dBc at full output (-40 dBc at lower level)	
	Phase Noise:	-63 dBc (0.5 Hz to 15 KHz) including effects of internal standard	
	£(1 Hz):	100 Hz/100 dBc, 1 KHz/110 dBc, 10 KHz/120 dBc, 100 KHz/125 dBc	
	Noise Floor:	-135 dBc/Hz	
Frequency Standard	Internal:	OCXO 3×10^{-9}/day $\pm 1 \times 10^{-8}$/0–50°C 1×10^{-6}/year	or TCXO 1×10^{-8}/day $\pm 1 \times 10^{-6}$/0–50°C 2×10^{-6}/year
	External Drive:	10 MHz, 0.4 Vrms into 300 Ω; 5 MHz, 0.5 Vrms into 300 Ω	
	Aux. Output:	10.000 MHz, 0.4 Vrms into 50 Ω (Note: internal or external standard required for operation)	
General	Oper. Ambient:	0–55°C, 95% R.H.	
	Power:	105–125V, 50–400 Hz, 50W (100, 220, 240V optional)	
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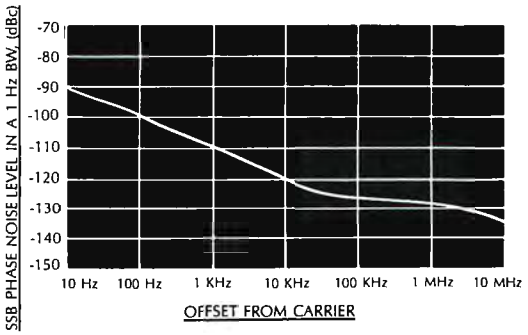
Frequency	Resolution:	0.1 Hz (0.2 Hz, 310–620 MHz)	0.1 Hz (0.2 Hz, 310–620 MHz)
	Optional Phase Rotation:	0–360° in .36° steps (in .72° steps, 310–620 MHz)	N/A
Switching Time		(within phase continuous range) <1 µs transient, 2 µs delay	
Spurious Outputs	Discrete:	-60 dBc	-70 dBc
Prices (domestic)		$10,025.00 including 0.1 Hz resolution, manual and remote controls, OCXO frequency standard. Other configurations available; prices are a function of resolution.	



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

October 23, 1992
(received 10/29/92)

Chaotropic Anion - Membrane Interactions

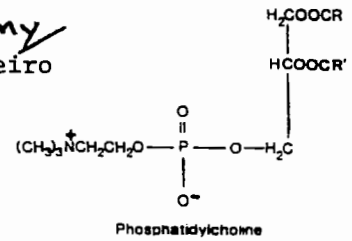
Dear Barry,

The figures below are expansions of 600 MHz (Varian - Unity) 1H NMR spectra showing the effects of the chaotropic anions, SCN^- and $SeCN^-$, on small (~200 A^o) bilayer vesicles of egg phosphatidylcholine in D20. The choline $N(CH_3)_3$+ signal of these vesicles has previously been observed to be split into 2 components (1-4). Indeed, in the absence of anion, the $N(CH_3)_3$+ signal is split (Fig. 1). At the same time, the CH_2N+ signal is a single resonance (Fig. 2) and the O_3POCH_2 signal appears as a tall, lowfield resonance with a shoulder resonance 15 Hz upfield (Fig. 3). Addition of 0.2M anion increases the 11 Hz $N(CH_3)_3$+ splitting to ~22 Hz (SCN^-) or to ~24 Hz ($SeCN^-$). The CH_2N+ resonance, initially a single peak, is split by either anion (~20 Hz for SCN^- and ~23 Hz for $SeCN^-$). The O_3POCH_2 splitting increases from ~15 Hz to ~23 Hz for SCN^- and ~25 Hz for $SeCN^-$. Note that the anion effects appear about equal for all three phospholipid choline groups, while the backbone glycerol sn1 and sn3 CH_2 resonances are not detectably affected by either anion. Note also that the lowfield components move downfield in the presence of anion, while the upfield signals remain at their original positions. Seelig and coworkers have recently suggested a tilt of the phospholipid head group in the presence of charged molecules (5), and our NMR observations may have some relevance here.

Sincerely, Tony

Anthony A. Ribeiro

1. G.Jendrasiak Chem. Phys. Lipids 9 133 (1972).
2. R.Kostelnik and S. Castellano J. Magn. Reson. 9 291 (1973).
3. M.Sheetz and S.I. Chan Biochemistry 11 4573 (1972).
4. C.Brouillete et al. Biochemistry 21 4569 (1982).
5. J.Seelig et al Biochemistry 28 7720 (1989).



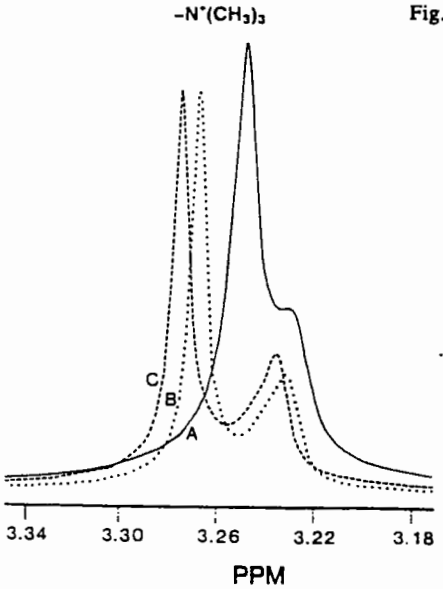


Fig. 1 Choline N(CH3)3+ resonances of EPC alone (A), with 0.2M KSCN (B), and with 0.2M KSeCN (C).

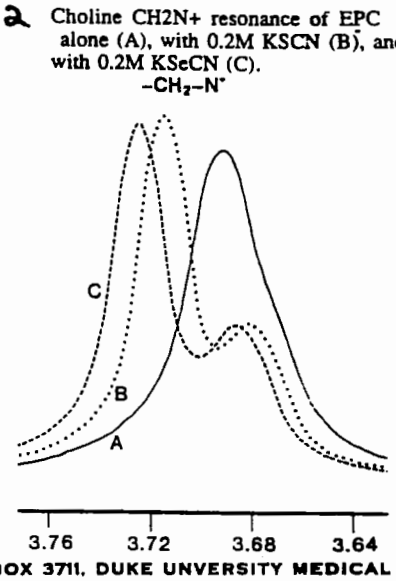


Fig. 2 Choline CH2N+ resonance of EPC alone (A), with 0.2M KSCN (B), and with 0.2M KSeCN (C).

BOX 3711, DUKE UNVERSITY MEDICAL CENTER
DURHAM, NC 27710

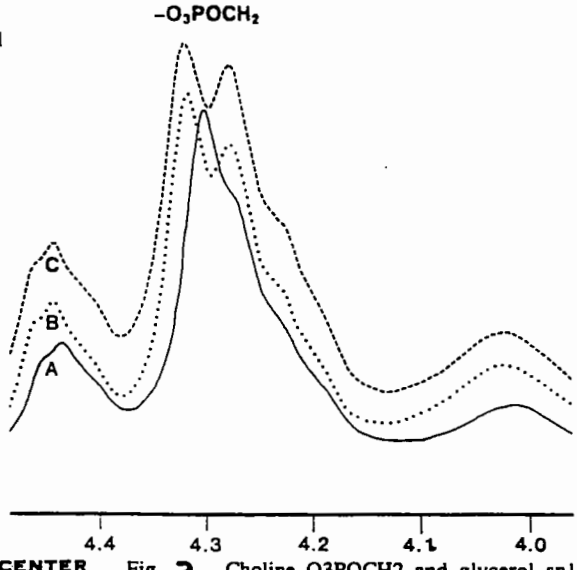


Fig. 3 Choline O3POCH2 and glycerol sn1 and sn3 CH2 resonances of EPC alone (A), with 0.2M KSCN (B), and with 0.2M KSeCN (C).

411-42 ICI **Pharmaceuticals Group**

Stuart Pharmaceuticals/ICI Pharma

October 26, 1992 (received 10/31/92)

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

Dear Dr. Shapiro,
I am writing this letter for that specific group of Bruker AMX users who are interested in using the ethernet file transfer protocol (FTP) to bring their NMR data to a Macintosh or other PC for off-line processing. *Please credit this to J.R. Snyder's account: ICI Western Research Center,Richmond, CA.*

FTP'ing Bruker AMX data to a Macintosh or PC with a minimal infliction of pain

We have off-line processing programs for the Macintosh, VersaTerm-PRO to "FTP" the data over our ethernet to an AMX-X32 UNIX-like computer. Since "RCP" transfers are not supported by VersaTerm-PRO, Telnet and most generic transfer protocols one is subject to a major limitation of FTP namely that FTP transfers files easily but not folders containing files. Since the Bruker AMX data format consists of a data folder containing an experiment folder containing files and a processing folder, etc..., to use FTP one would literally have to re-create this multi-folder structure on the Mac and move between the appropriate folders to put the correct files in. Looking at my half million dollar NMR instrument I quickly rejected this tedious scenario. For a while Kermit (a very very slow but time old favorite) started looking rather good. Fortunately, my Mac/Unix guru suggested some alternatives.

The first alternative was to write a shell script on the AMX that literally reads the files to be transferred and re-creates the folder structure and transfers the files appropriately. After writing an awkward two page script to do this my Mac/Unix guru felt sorry for me and condensed it down to a couple of scripts each about 1/3 of a page. The data folders are put into a specific location transferred to the Mac via FTP and just in case there is a problem (i.e. the Macintosh is off or the network is down) the files are not deleted from the AMX but automatically moved to a temporary location. To make this even more mindless a process I started a cron on the AMX that looks for files in a given location and transfers them automatically to the Mac at specified times of the day. This alternative is excellent for our needs because most of the data "FTP'ed" originated from our AM systems (via lightnet to the AMX) whose operators do not have access to the AMX computer at all. The current limitation is that these scripts assume you are transferring exp folder "1" and proc folder "1" but these scripts can be easily modified to not assume this.

The second alternative was to find "freeware" or "shareware " that could address this problem. A friend from U of Penn. told me about a "freeware" program called "suntar". Instead of "taring" onto tape you tar the data folder into a "bundled" form (using "tar cvf -" command) that can be easily FTP'ed to the Mac as a file and "unbundled" by suntar. This alternative is best for a multi-user environment. The actual transfer process via FTP is faster while the bundling and unbundling of data takes more user time but less networking time.

If you are interested in the first alternative, the FTP script, send me a blank formated Mac disk and I will gladly send you a copy with instructions on setting it up or contact BTR Software Tools at P.O Box 9152 Wilmington, DE 19809. If you are interested in the freeware package I suggest you write to the "suntar" people (vai Email: speranza@cirfid.unibo.it or via SnailMail: Suro Speranza via Cappuccini 18, 40026 Imola, Italy) or download it from an internet bulletin board such as "sumex".

Sincerely,

Debra Banville

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APPLICATION

The Z•SPEC family of microsample probes allow you to complete the analysis of your quantity limited samples (140µl) in one-half the normal time compared to your standard 5mm probe. Shown on the next page are HMQC data from 12µg of cryptolepine (Figure 1), HMBC data from 35µg of cryptolepine (Figure 2), HMQC data from 140µg Digoxin (Figure 3), and TOCSY data from 140µg Digoxin (Figure 4).

These 500MHz spectra demonstrate the significant gain in absolute signal to noise performance and speed of analysis that can be achieved when using the Z•SPEC Microsample Inverse Detection Probe.

COMPATIBILITY

The top loading design of the Z•SPEC microsample probe assures convenient sample changing and compatibility with automatic sample changers. Z•SPEC probes interface directly to Bruker, General Electric, or Varian spectrometers operating at a ^{1}H frequency of 200, 250, 270, 300, 360, 400, 500, or 600 MHz.

CUSTOMER SUPPORT

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NALORAC

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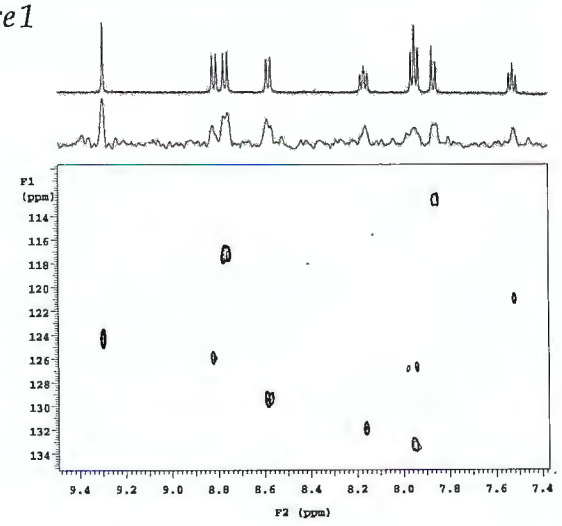
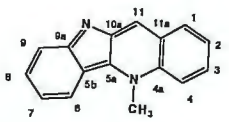
Z·SPEC® Microsample Spectra

Figure 1: HMQC Spectra of 12µg of cryptolepine obtained in 16 hours with Z•SPEC MID500 probe. Normal ^1H spectrum (256 transients) for the 12µg sample plotted on top.

Figure 2: HMBC Spectra of 35µg cryptolepine obtained in 22 hours with Z•SPEC MID500 probe. Aromatic F_2 window. Normal ^1H spectrum (64 transients) for 35µg sample plotted on top.

Figure 1





·HCl

Figure 2

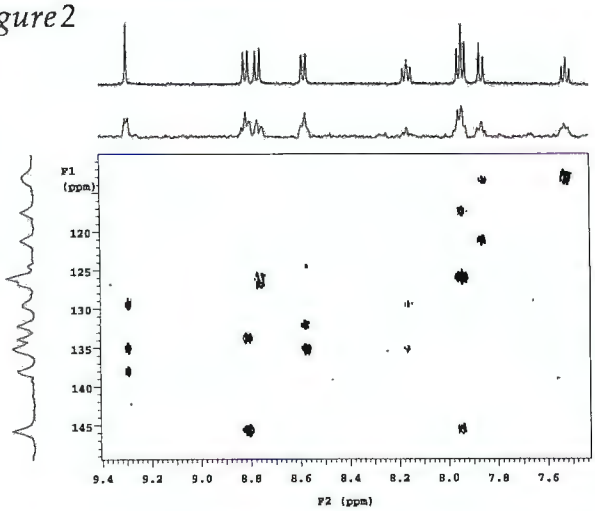
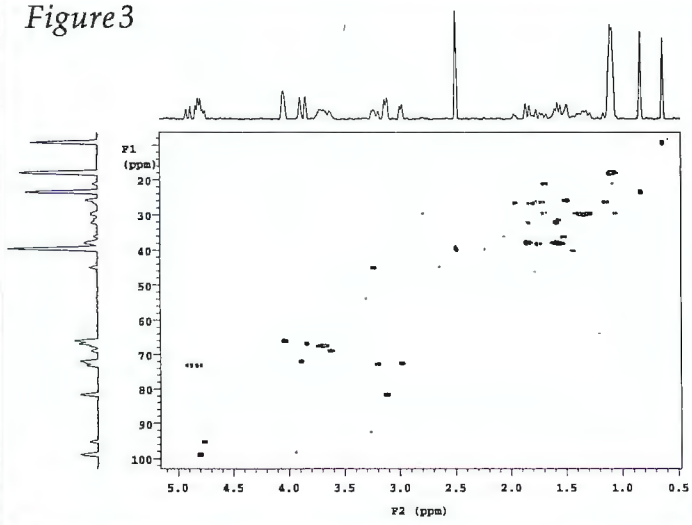


Figure 3



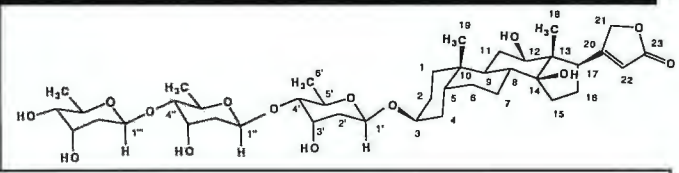


Figure 4

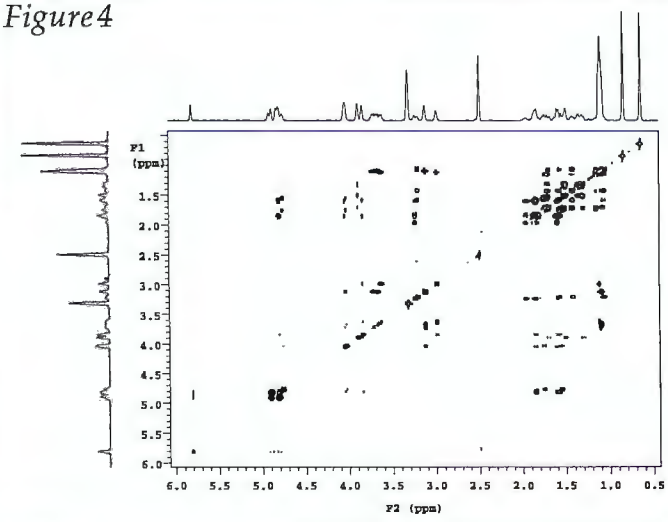


Figure 3: HMQC Spectra of 140µg Digoxin obtained in 14.5 hours with Z•SPEC MID500 probe.

Figure 4: TOCSY Spectra of 140 µg Digoxin obtained in 58 minutes with Z•SPEC MID500 probe.

Please review the following publications for additional examples of results obtained with Z•SPEC Microsample Probes: Micro Inverse-Detection: A Powerful Technique for Natural Product Structure Elucidation, Ronald C. Crouch and Gary E. Martin, *Journal of Natural Products*, **55 (9)**, pg 1343-1347 (1992); Comparative Evaluation of Conventional 5mm Inverse and Micro-Inverse Detection Probes at 500MHz, Ronald C. Crouch and Gary E. Martin. *Magnetic Resonance in Chemistry*. Inpress.

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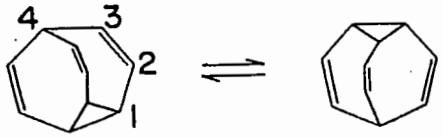
October 22 1992
(received 11/2/92)

Professor B. Shapiro
TAMU NMR Newsletter
966 Elsinor Court
Palo Alto, CA 94303
U.S.A.

Carbon-13 Dynamic MAS Spectra of Solid Bullvalene

Dear Barry,

The question of whether bullvalene undergoes the Cope rearrangement in the solid state remained open for many years until in 1985 Meier and Earl[1] showed unequivocally that the reaction does occur. Their proof is based on the carbon-13 MAS spectra of bullvalene which as function of temperature exhibit characteristic coallescence effects similar to those observed in liquid solutions. A detailed quantitative analysis of these MAS shows however that this reaction is not the only one that takes place in solid bullvalene: in addition the molecules also undergo independent three-fold jumps about their C_3 axes. This is consistent with other recent studies of solid bullvalene.[2,3]



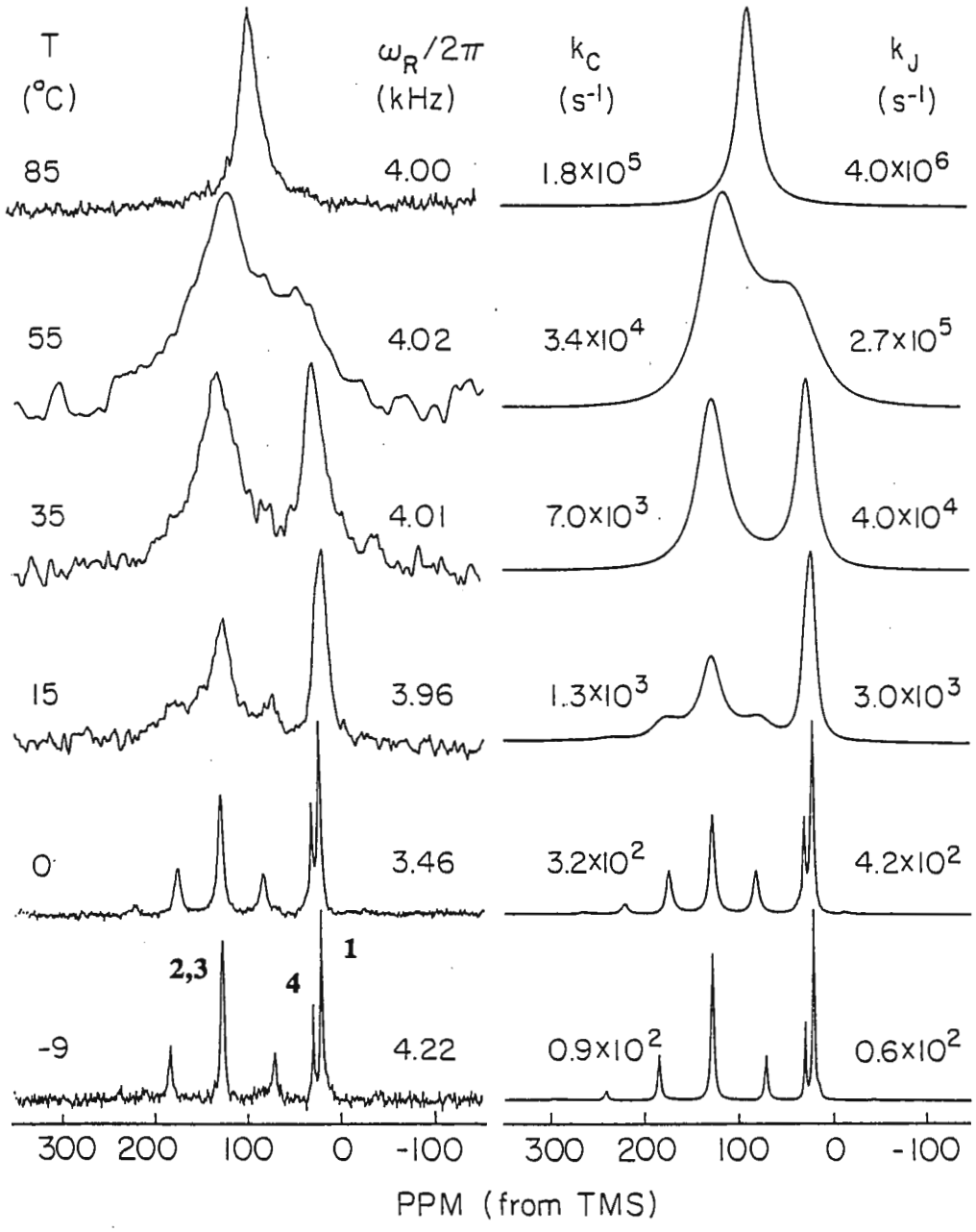
Experimental and calculated spectra are shown in the Figure. For the calculation the theory of Schmidt and Vega[4] was used. Two dynamic processes were assumed, (i) Cope rearrangement coupled with a molecular reorientation that restores the original molecular orientation in the crystal and (ii) three-fold jumps. As may be seen, inclusion of both mechanisms yields dynamic lineshapes that faithfully reproduced the experimental spectra. This example demonstrates the power of the lineshape theory of Schmidt and Vega.

Sincerely,

R. Poupko. Z. Luz

R. Poupko Z. Luz

1. B.H. Meier and W.L. Earl, J.Am.Chem.Soc. 107, 553 (1985).
2. J.J. Titman, Z. Luz and H.W. Spiess, J.Am.Chem.Soc. 114, 3765 (1992).
3. S. Schlick, Z. Luz, R. Poupko and H. Zimmermann, J.Am.Chem.Soc. 114, 4315 (1992).
4. A. Schmidt and S. Vega, J.Chem.Phys. 87, 6895 (1987).



Left: Experimental dynamic MAS carbon-13 spectra of bullvalene as function of temperature. Right: Simulated spectra using the indicated rate constants for the Cope rearrangement, k_C, and the three-fold jumps, k_J.

Postdoctoral Position

We expect to have an opening for a Postdoctoral Fellow beginning September 1993 in the Laboratory of Analytical Chemistry, National Institute of Diabetes, Digestive and Kidney Diseases. The successful candidate will be involved in synthesis, structural and enzymatic studies of isomeric nucleosides using NMR spectroscopy. He or she is also expected to provide support and establish collaboration with researchers using the NMR facilities in this Laboratory which is currently equipped with two Varian 300 MHz and a Varian 500 MHz NMR spectrometers. The Fellow must have less than three years postdoctoral experience at the time of appointment.. Applicants should supply a resume and names of three references to Dr. Herman Yeh, Building 8, Rm B2A-22, Laboratory of Analytical Chemistry, NIDDK, Bethesda, MD 20892.

RECRUITMENT

The Frederik Philips Magnetic Resonance Research Center of the Department of Radiology, Emory University School of Medicine seeks a Ph.D. with NMR spectroscopy or imaging experience for a one to two year postdoctoral fellowship. Knowledge of spin behavior is essential, and skill in medicine, biology or biochemistry, or analog electronics or computer science would be useful.

Two 1.0 m clinical imagers operating at 1.5T and a 30cm 200MHz instrument, all fully programmable, are available. The department is part of an 800 bed tertiary care complex providing ample opportunity for clinical collaboration. Five Ph.D. scientists and one M.D. in the Department of Radiology are currently involved in full time NMR research.

Current research at the center includes measurement of diffusion and perfusion *in vivo* and monitoring rates of intensity changes following administration of contrast agents, organ stimulation, and drug therapy. New pulse sequence development is emphasized and many applications are directed at cancer.

Qualified candidates please contact:

W. Thomas Dixon, Ph.D.
Frederik Philips Magnetic Resonance Research Center
Department of Radiology
Emory University School of Medicine
1364 Clifton Road, room AG15
Atlanta, GA 30322

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ENC 34th Experimental Nuclear Magnetic Resonance Conference
March 14–18, 1993, Adam's Mark Hotel St. Louis, Missouri

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

Dear Barry:

The 34th Experimental NMR Conference will be held at the Adam's Mark Hotel in St. Louis, Missouri from March 14-18, 1992. There will be sessions covering high resolution and multi–dimensional NMR in liquids, materials imaging, biological imaging, hardware, NMR in solids, calculations and data processing, and that old standby, miscellaneous. Two poster sessions will be held, and we anticipate having adequate space for the posters at the Adam's Mark. **The deadline for submitting poster abstracts will be December 30, 1992** (ENC is early this year!), so start planning now.

We have set dates and sites for future ENC's as well, and you should mark the following dates on your busy calendar:

March 14-18, 1993	Adam's Mark Hotel, St. Louis, MO
April 10-14, 1994	Asilomar Conference Center, Pacific Grove, CA
March 26-30, 1995	Boston Marriott Copley Place, Boston, MA
1996	to be determined
March 23-27, 1997	Clarion Plaza Hotel, Orlando, FL

A conference announcement will be mailed to those on our mailing list (*ie*, registered conferees from the past three ENC's) in October. Anyone wishing to be added to the mailing list should contact the Conference Office, address and phone at the bottom of this letterhead. **Please note that the office address and phone have changed from last year.**

I look forward to an exciting conference in St. Louis, and hope that you and your readers will be able to participate.

Sincerely,

Mary

Dr. Mary W. Baum, Chair, 34th ENC
fax: 908-594-1530
email: baum@msdrl.com

Executive Committee:

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Merck Research Laboratories
PO Box 2000, RY50SW
Rahway, NJ 07065-0900
908-594-6301

Bernhard Blümich, Chair Elect
Max Planck Institut für
Polymerforschung
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D-6500 Mainz,Germany

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Erice, Trapani, Sicily, Italy
15-21 March 1993

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Program and Lecturers

Basic Methods for the Study of Macromolecular Dynamics

Modern NMR Spectroscopy	Richard R. Ernst, ETH Zentrum, Zurich
3D and Multidimensional NMR	Hartmut Oschkinat, EMBL Heidelberg
Molecular Dynamics Studies of Protein-Nucleic Acid Complexes	Michael Levitt, Cell Biology, Stanford
NMR and Optical Fluorescence in the Study of Macromolecular Dynamics	Rudolf Rigler, Karolinska Inst., Stockholm
The Definition of Motional Processes in Proteins	Gerhard Wagner, Harvard Medical School

Combinations of Biological and Physical Methods and the Implications of Dynamics

Protein Dynamics and their Implications for Protein Structure Determination by NMR	Oleg Jardetzky, SMRL, Stanford
NMR Studies of Protein Folding	Christopher M. Dobson, Oxford
Studies of Flexible Proteins: NMR and other Physical Methods	Michael Weiss, Harvard Medical School
Isotopic Spectral Editing in NMR Studies of Protein Structure and Dynamics	John L. Markley, University of Wisconsin
The Use of Site-Directed Mutagenesis in NMR Structural Studies	Sture Forsén, University of Lund, Sweden
Structure and Flexibility of Biological Active Peptides Determined by NMR	Jean-François Lefèvre, IBMC, Strasbourg

Studies of Specific Systems

NMR Studies of the Structure and Dynamics of Large Proteins: the Antibody System	Yoji Arata, University of Tokyo
Structure and Dynamics of Metalloproteins	Ivano Bertini, University of Florence
Muscle Proteins	Brian D. Sykes, University of Alberta
Structure and Dynamics of Nucleic Acids	Cornelius W. Hilbers, NSR Centre, Nijmegen
Mechanisms of Intermembrane Lipid Transport	Fritz Paltauf, Technische Universität Graz
NMR Studies of Macromolecular Interactions	Gordon C. K. Roberts, NMR Centre, Leicester
Dynamics of Polysaccharides	Robert G. Shulman, Yale Medical School

PURPOSE OF THE SCHOOL

The purpose of the School is to present the different methods for analyzing molecular dynamics, illustrated by the most advanced studies ranging from the determination of very fast internal motion in macromolecules to the observation of the slow process of protein folding. These analyses also consider the biological activity of the protein. The implication of the dynamics for the determination of three-dimensional structure will also be developed. The subjects which will be covered during the school fall into three broad categories:

1. Basic Methods for the Study of Macromolecular Dynamics
2. Combinations of Biological and Physical Methods and the Implications of Dynamics
3. Studies of Specific Systems

NMR has reached the stage where both three-dimensional structure and dynamics can be studied quite accurately. An integrated approach between these two aspects should be utilized more and more in future research. The proposed course should bring together the interesting features of such an integrated approach.

VENUE

The Ettore Majorana Centre for Scientific Culture was founded in 1963 in the pre-medieval mountain town of Erice near Palermo as a Conference Centre, taking its inspiration from the Italian Physicist, Ettore Majorana. The Centre's lecture halls are located in two restored monasteries and the ancient Plazzo Ventimiglia. School participants are housed in the Centre Institutes or local hotels.

GENERAL INFORMATION

Prospective participants should apply to either:

Prof. Oleg Jardetzky or
Stanford Magnetic
Resonance Laboratory
Stanford University
Stanford, CA 94305-5055
USA
fax: +415/723-2253
telex: 348402 STANFRD STNU A

Prof. Jean-François Lefèvre
Institut de Biology
Moleculaire et Cellulaire
15 rue Descartes
67084 Strasbourg Cedex
France
fax: +33/88 61 06 80

stating: (1) date and place of birth, nationality, qualifications and present position; (2) address, fax and phone numbers; and (3) list of publications.

Young researchers with little experience should enclose a letter of recommendation from the head of their research group or from a senior scientist active in the field.

Applicants interested in submitting unpublished results should send the title and an outline in about 200 words. Selected papers will be presented and discussed in special sessions.

The total fee, including full board and lodging (arranged by the School) is US $700. Limited financial aid available. Participants should arrive by 5 p.m. on the 15th.

THE CLOSING DATE FOR RECEIPT OF APPLICATIONS IS JANUARY 15, 1993. NO APPLICATION FORM IS REQUIRED.

Attendance will be limited to ~50 students, to be selected by the Co-Directors. Further details will be mailed with the acceptance letter.

Oleg Jardetzky
Co-Director of the School

A. Zichichi
Director of the Centre

Jean-François Lefèvre
Co-Director of the School

The University of Texas Medical Branch at Galveston

School of Medicine
Graduate School of Biomedical Sciences
School of Allied Health Sciences
School of Nursing

Marine Biomedical Institute
Institute for the Medical Humanities
UTMB Hospitals

Department of Human Biological Chemistry & Genetics

October 15, 1992
(received 10/22/92)

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

"UNRECOGNIZED SYNTHESIS PROBLEMS USING ^{17}O-WATER"

Dear Barry:

We have examined ^{17}O NMR spectra of thirty-two C_{19}- to C_{27}-steroids, including alcohol, ketone, ester, and carboxylic acid derivatives. We find that the ^{17}O signals (CD_3CN, 75°, external water reference) from such oxygen functionality correlate with structure in keeping with prior spectra-structure disclosures of others for simpler organic compounds.

The difficulty of such studies is the necessity of isotope enrichment that may require specific synthesis procedures, and we have employed well recognized means for introduction of ^{17}O from ^{17}O-water as isotope source into steroid functional groups. In conducting these relatively direct approaches we encountered a few little problems that impeded progress, but solutions to the problems now accord improved awareness of the use of ^{17}O-water for such work.

Acid-catalyzed (HCl) exchange of ^{17}O from ^{17}O-water into ketone carbonyl by the method of Turro et al.[1] (acetonitrile as solvent) has been advanced as general means of labeling ketones,[2] and we find that there is low level incorporation of isotope, witness ^{17}O-ketone signals in the range δ_o 504-570. However, more prominent, narrower signals at δ_o 332-338, 309-323, and 271-275 (apparently concentration dependent) were also encountered, these obviously not those of ketone carbonyl. Rather, these signals are recognized as those of acetamide, acetamide.HCl salt or complex, and acetic acid, respectively, clearly establishing that solvent acetonitrile is partially hydrolyzed, thus consuming rare and costly ^{17}O-water and limiting the amount of ^{17}O-water available for incorporation by the slower exchange reaction into ketone carbonyl.

Although solvent hydrolysis could be predicted from simple chemistry, report of this difficulty has not appeared to our knowledge. Change to tetrahydrofuran as solvent under conditions of Anderson et al.[3] used for ^{18}O-water exchange into steroid ketones gave the clean reaction needed.

A more helpful problem with either solvent can be turned to advantage. In exchanging the 17-ketone oxygen of 3ß-acetoxyandrost-5-en-17-one a second prominent signal was observed at δ_o 363.7, in the range of fatty acyl carbonyl oxygen, thus evincing isotope incorporation into the 3ß-acetoxyl carbonyl. Also, exchange (in tetrahydrofuran) of the 3-ketone oxygen of methyl 3-oxo-5ß-cholan-24-oate led to ketone (δ_o 561.8) and ester carbonyl (δ_o 360.2) signals, establishing that the steroid ester carbonyl had been labeled (Fig.1). Some ester hydrolysis also occurred, giving rise

to the carboxyl (δ_o 271.4) signal.

It is, of course, well recognized that water oxygen exchange into carboxyl oxygen occurs in ester hydrolysis, but exchange into ester carbonyl without ester hydrolysis has not been heretofore recognized. The discovery may offer advantage to others in devising means of labeling esters with ^{17}O for NMR studies.

Yours truly,

Leland L. Smith Joseph E. Herz Edward L. Ezell

1. N. J. Turro, M. A. Paczkowski, and P. Wan, J. Org. Chem., **50**, 1399-1404 (1985).
2. G. W. Kabalka and N. M. Goudgaon, in D. W. Boykin (editor), ^{17}O NMR Spectroscopy in Organic Chemistry, CRC Press Inc., Boca Raton,FL, 1990, pp.21-37.
3. W. G. Anderson, C. Y. Byon, C. R. Eck, and M. Gut, J. Labelled Compounds Radiopharm., **21**, 59-64 (1984).

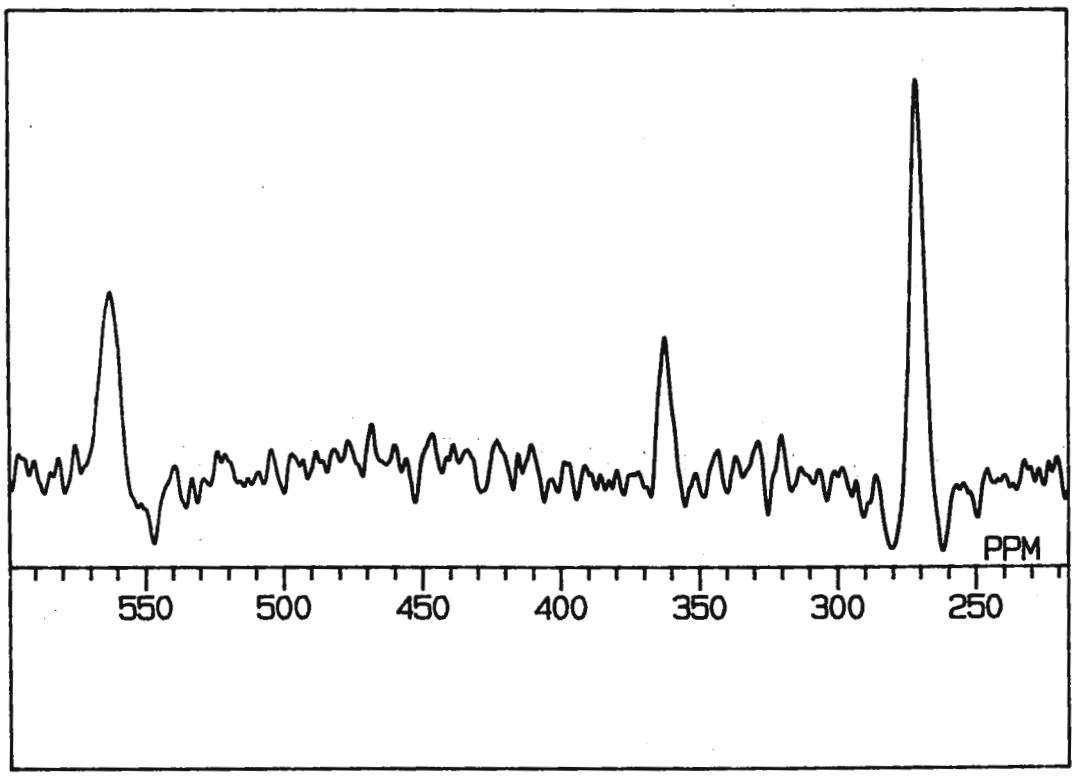

Fig.1. ^{17}O Spectrum of product mixture containing methyl [3,24-$^{17}O_2$]-3-oxo-5ß-cholanoate and [3,24-$^{17}O_2$]-3-oxo-5ß-cholanic acid (CD_3CN, 75°; 100 Hz broadening factor, 2% trapezoidal apodization to remove baseline roll).

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NL-1081 HV Amsterdam
The Netherlands

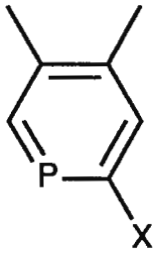
November 6, 1992
(received 11/17/92)

vrije Universiteit *amsterdam*

Title: Long-range ^{1}H-^{31}P couplings

Dear Dr. Shapiro,

H.T. Teunissen, Ph.D. student in the research group of Prof. Bickelhaupt in this Laboratory, has prepared a number of 2-substituted-4,5-dimethylphoshines,



and their tungsten complexes, P-W(CO)$_5$ (1). The ^{1}H NMR spectra show doublets for the aromatic protons H(6), $^{2}J(HP)$ = 25 - 39 Hz, and H(3), $^{3}J(HP)$ = 5 - 24 Hz, and a doublet, J(HP) = 3 - 7 Hz, and a singlet for the methyl protons. Recently, for X=Cl, the doublet has been assigned to the 5-CH_3 protons and the singlet to the 4-CH_3 protons (2), thus $^{4}J(HP) > {}^{5}J(HP)$. However, for the compounds we studied, e.g. X = I, ZnI, $SnPh_3$, we find $^{5}J(HP) > {}^{4}J(HP)$. This is based upon ^{1}H-^{13}C COSY, ^{1}H-NOE and ^{13}C-1D-INADEQUATE spectra which allow unequivocal assignments.
Please credit this contribution to the subscription of Dr. J. Bulthuis. Would you please forward the next issues of the newsletter to me.

Yours sincerely,

F.J.J. de Kanter

(1) H.T. Teunissen and F.Bickelhaupt, Tetrahedron Letters 33, 3537 (1992)
(2) P. Le Flock and F. Mathey, Tetrahedron Letters 30, 817 (1989)

University of Alberta
Edmonton
Canada T6G 2G2

Department of Chemistry
Faculty of Science
E3-44 Chemistry Bldg., Tel. (403) 492-3254 Fax (403) 492-8231

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

October 30, 1992
(received 11/6/92)

Re: Data archiving on streaming magnetic tape

Dear Barry:

Disk space has always been a problem on NMR computer systems and will continue to be especially with the growing popularity of 2D and more recently 3D NMR techniques. A fast and efficient means of data archiving is necessary on any NMR spectrometer. We recently installed a Varian Unity 500 Spectrometer in our laboratory and before the system arrived there were some concerns regarding multiple tape archives on the 150 Mbyte streaming magnetic tape. Some of the readers may be interested in our experiences/observations over the last year.

Some of the UNIX tape commands are as follows (with our corresponding aliases found in the .cshrc file): (the numbers in parentheses after each entry represent approximate speed in Mbytes/minute):

alias rew	'mt -f / dev/nrst0 rew'	(fast)
alias skip	'mt -f /dev/nrst0 fsf'	(6)
alias tapedir	'tar -tvf /dev/nrst0'	(1.5)
alias archive	'tar -cvf /dev/nrst0'	(1.2)
alias extract	'tar -xvf /dev/nrst0'	(6)

For the first tape archive, simply change to the directory to be stored, 'rew' the tape and 'archive' the file. Every archive creates an end-of-file (EOF) marker; the next file must begin after this marker. Fortunately, there is a UNIX tape command that allows 'skip'ping past these markers; thus for the n+1th archive, 'rew' the tape, 'skip' n markers and 'archive'. 'extract'ing (tape to disk) files and getting 'tapedir'ectories are performed similarly. Tape directories and archiving data are about 5 times slower than skipping and extracting; however, to extract a single file from within an archive file takes about 5 times longer than extracting the whole file.

We would recommend the following for using the magnetic tape unit:

1. Since extracting and skipping take about the same time, keep the size of the archive files reasonably small. This is because disk space is always limited and one does not want to extract a single file from within an archive.
2. Keep good notes. One does not want to take tape directories.

The disadvantages of the tape unit is that it is a serial device, the cartridge is erasable near a magnetic field and it can be over written accidentally and the software is silent even when the 150 Mbyte limit is exceeded.

Sincerely,

Tom

TTN:lf

Tom Nakashima

Varian NMR Users

CFT-20

FT-80

FT-80A

XL-100

XL-200

XL-300

PseudoCorp Data Machines Group manufactures 100% software compatible computer hardware to replace the hard to repair computer and associated components of the control console for the above listed Varian NMRs.

- **History**

At the heart of these systems are Varian Data Machines mini-computers. In 1977 Varian sold the Mini-computer Operations to Sperry-Univac. Sperry-Univac discontinued the Varian line in 1985 and Varian Instruments changed to different processors on subsequent models. In July 1992, PseudoCorp delivered it's first 2100x series replacement processor.

(See reverse for details)

Products and Services offered in the upgrade process

- **PDM2100x [tm] Series Processor**
  - Direct single board replacement processor for Varian 620i, l , f, V72, V73, V74, V75, V76, V77-200, 400, and 600 series processors.
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  - 10% Discount on PseudoStore 7000 [tm] (Must take delivery by 3/31/93)

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For More Information, Or To Order Call Jack Primavere at

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DEADLINE DATES

No. 413 (February) --------- 22 January 1993
No. 414 (March)----------19 February 1993
No. 415 (April) ----------19 March 1993
No. 416 (May) ----------23 April 1993

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NMR

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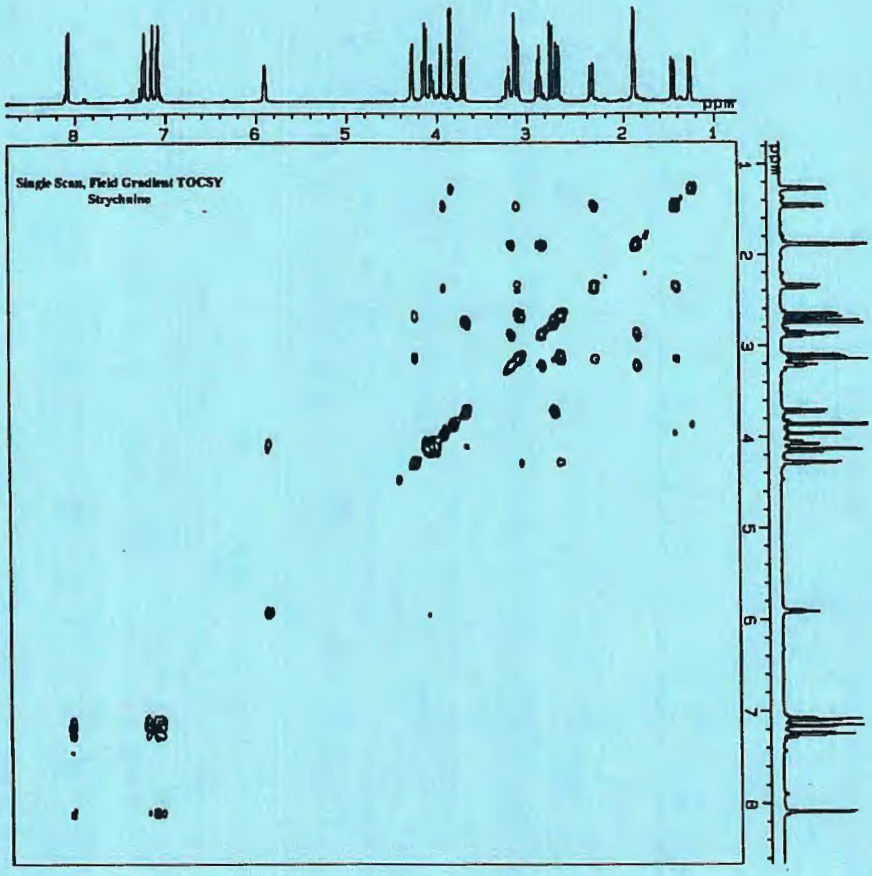

The above data is a 512 by 512 *TOCSY* spectrum and was obtained on an **Alpha**- 500 in less than 5 minutes with **JEOL**'s gradient probe. This same probe is also designed to do reverse and water suppression experiments. Pulse shaping, three channel operation, dynamic Rf control, matrix shims, as well as unsurpassed field reliability are all standard with the **Alpha**.

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