TEXAS A&M UNIVERSITY



No. 397 October 1991

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

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FORTHCOMING NMR MEETINGS

13th Eastern Analytical Symposium, Somerset, NJ, November 11-15, 1991; NMR symposia November 12-14; See Newsletter 395, 43.

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

33rd ENC (Experimental NMR Conference), Asilomar Conference Center, Pacific Grove, California, March 29 - April 2, 1992; Contact: ENC, 750 Audubon, East Lansing, MI 48823; (517) 332-3667

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

XV International Conference on Magnetic Resonance in Biological Systems, Jerusalem, Israel, August 16 - 21, 1992; Contact: Prof. Gil Navon, XV ICMRBS, P. O. Box 3190, Tel Aviv 61031, Israel.; Tel. (972-3) 5271111, Fax: (972-3) 5239099.

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

Additional listings of meetings, etc., are invited.

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Last Notice re 1991-92 Invoices

By the time this issue of the Newsletter reaches you, your 1991-92 subscription fee should have been received by me. If my records indicate that your payment has not been received, a notice to that effect is included in your October Newsletter copy. Newsletter finances are such that we cannot afford to service subscriptions which are unpaid by the time the November issue is to be mailed. Please help by making it unnecessary to remove and then re-add your name. Thanks!

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All Newsletter Correspondence

Should Be Addressed To:

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

(415) 493-5971

DEADLINE DATES *

No. 399 (December)----15 November 1991

No. 400 (January 1992) - 13 December 1991

No. 401 (February) ----- 24 January 1992

No. 402 (March)-----21 February 1992

igspace* Please note that some of these dates are somewhat earlier in the month than has usually been the case !!



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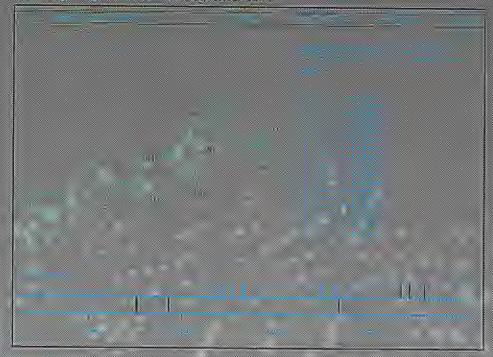
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Sept. 4, 1991 (received 9/7/91)

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

Homoallylic Couplings: Larger Than You Might Imagine

Dear Barry,

We were somewhat puzzled recently while investigating the structure of the following secondary mold metabolite

MeO
$$H_{eq}$$
 H_{ax} Me H_{eq} H_{ax} Me H_{eq} H_{ax} H_{eq} H_{eq

The methylene protons at the 1- and 4-positions each appeared as an 8-line pattern (ddd) and were initally thought to be adjacent to one another. While <u>no</u> large vicinal, diaxial coupling was observed, it was believed that its absence could, perhaps, be due to rapid conformational averaging in the half-chair C-ring. Before a confirming low-temperature study could be conducted, the metabolite was discovered to be the 3-methyl ether derivative of a compound whose structure had been elucidated by Steyn and coworkers. Four of the six observed couplings between the methylene protons are, therefore, homoallylic in nature and not vicinal as first suspected.

The magnitude of the three largest coupling constants were surprising because we have seldom encountered homoallylic couplings in our natural products studies, and then they were in the usual 1-2 Hz range.² However, Sternhell³ and Barfield⁴ stated some time ago that these coupling constants can, in fact, be quite large in rigid molecular systems when both protons are largely perpendicular to the plane of the carbon-carbon double bond:

$$^{5}J_{HH'}=+4.99 \sin^{2}\varphi \sin^{2}\varphi'$$

Note that the protons need <u>not</u> be pointing in the same direction but must only exist in a relatively parallel orientation. With the subject metabolite, as is the case with 6-membered rings, $H-1_{ax}$ and $H-4_{ax}$ point in opposite directions.

Sincerely,

Mike Stack

Gene Mazzola

- 1. P. S. Steyn, P. L. Wessels, and W. F. O. Marasas, Tetrahedron, 36, 1551 (1979).
- 2. E. D. Becker, *High Resolution NMR Theory and Chemical Applications*, 2nd Ed., Academic Press, New York, 1980, p. 96.
- 3. S. Sternhell, Pure Appl. Chem., 14, 15 (1964).
- 4. M. Barfield and B. Chakrabarti, Chem. Revs., 69, 757 (1969).

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ANALYTICAL AND ENVIRONMENTAL RESEARCH
August 27, 1991
(received 8/30/91)

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

ASPECT 3000 - BRUKNET - PATHWORKS® - PC - WIN-NMR

Dear Barry,

In April, 1990, we installed a Bruker ACF-300 with an automatic sample changer and made this spectrometer available for routine use by all the chemists on the site. The chemists need only select the experiments to be run from a menu and enter an identifying title, the computer then carries out the measurements. We are finding that 60 to 90 samples (a mixture of ¹H, ¹³C, ¹⁹F and ³¹P) are being measured each day. When the queue contains only proton experiments, a spectrum is completed every 8 minutes. The quad probe means that we only have to change the probe when we need to measure ³H spectra. Many spectra need additional expansions that are not included in the standard plot. With more than 60 people needing access to the keyboard and display on the ASPECT 3000, there is a problem with doing these additional plots from the keyboard, especially since the automation program usually wants the plotter right in the middle of the individually queued plots. To alleviate this problem and provide for permanent storage of the spectra, we transfer the data via BRUKNET to a MicroVAX II where it can be archived to tape or optical disk and then using Pathworks® to one of several personal computers (mixture of MS DOS and Macintosh). The chemists selected Bruker's WIN-NMR after looking at several PC based NMR programs. In my opinion, Bruker could sell a ton these if the price was more reasonable. Bruker recommends a 386 class machine but we have found that a 286 is sufficient for expansions and plotting of spectra, however you do need a machine capable of adequately supporting MS Windows™. Of course, BRUKNET files and WIN-NMR are incompatible and a conversion routine is necessary. The program I wrote (about 50 lines of FORTRAN code, source and executable available on request) reads the file from the VAX, strips the BRUKNET headers, and writes a DOS format file in the correct directory for DISCONV, the Bruker supplied conversion program for files from Kermit or TCP/IP. A bat file on the PC does the directory switching, establishes the Pathworks® connection to the VAX and runs the FORTRAN program. Had I had the file format for WIN-NMR, I would have included the DISCONV step as well but that and converting the program to be a Windows application will have to wait. Using ethernet (BRUKNET-Pathworks®) for the file transfer is much faster then KERMIT (~30 seconds vs >15 minutes @9600 baud for the usual 32k file) even with the need for some seconds to strip the BRUKNET headers. With this system in place, the chemists can study their data in their own office and if they need additional hard copy, send the plots to one of the network plotters.

Sincerely yours,

Mike Maddox

PHASE - SENSITIVE GRASP

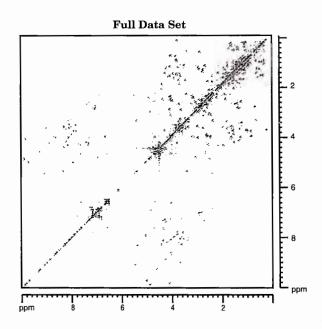
Phase-sensitive gradient DQF COSY of 3mM BPTI in 90% H₂0/10% D₂O on an AMX 600 using a GRASP II accessory including a 5mm triple-resonance probe [¹H {¹³C, ¹⁵N}] with integrated Z-gradient. 60G/cm, no phase-cycling, no baseline correction, symmetrized; (D. Moskau, Spectrospin AG)

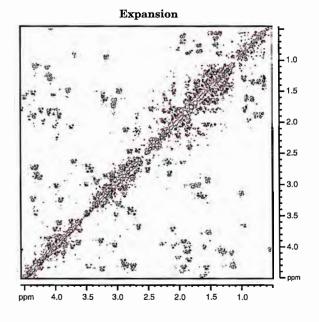
Gradient Spectroscopy (GRASP) has tremendous potential for accelerating the acquisition of long 2D/3D NMR spectroscopy experiments, by eliminating the need for lengthy phase-cycling for quadrature detection in ω_1 . Other GRASP benefits are its superb water-suppression capability, and reduced T_1 -noise.

However, until now GRASP was not practically applicable to NMR spectroscopy of larger molecules, because only magnitude-mode spectra could be obtained (except in artificial low-bandwidth cases of e.g. 1 ppm ranges at 300 MHz). Recently, important advances were reported (1) towards obtaining phase-sensitive GRASP-data at high field over a broad frequency range on an AMX 600 equipped with a GRASP II accessory.

Bruker's GRASP II accessory is available on all Bruker spectrometers with widebore and standardbore magnets at all field strengths. 5mm water suppression probeheads with up to l00G/cm gradients (¹H-only, BB inverse, triple-resonance) are available with excellent RF specifications (S/N, resolution, lineshape, etc.). For more information, call your nearest Bruker representative.

(1) A. L. Davis, E. D. Laue, J. Keeler, D. Moskau, J. Lohmann, Journal of Magnetic Resonance, in press.







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National Institutes of Health National Heart, Lung, and Blood Institute Bethesda, Maryland 20892 September 3,1991 (received 9/7/91)

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

A Question of Shifts

Dear Barry,.

Tappey Jones, in this Laboratory, has prepared a series of imine phosphonates as intermediates in the synthesis of ant venoms. The ¹H spectrum shows an NH, so this familiar sort of tautomerism is going on:

$$(C_2H_5O)_2POCH_2$$
 α
 CH_3
 CH_3
 CH_3
 $CH_5O)_2PO$
 $CH_5O)_2PO$

Imine

Enamine

α-C: δ ¹H 3.51, ¹³C 60.9 ppm ¹J HC 159 CP 198 Hz.1

I was troubled by the fact that neither the ¹H nor the ¹³C spectrum showed chemical shifts characteristic of the =CH group of the eneamine. The ¹H shift was at about 0.7 ppm in higher field than anticipated and the ¹³C a good 40 ppm up. However, the one-bond couplings of the imine form fit the structure perfectly well, both ¹J(HC) and ¹J(CP) being half again as big as that of the imine.

I can find examples of similar ¹H shifts for compounds of this sort in the literature, but no examples of ¹³C shifts, nor any word of explanation. I would welcome one.

Yours very truly,

R. J. Highet



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August 21, 1991 (received 8/26/91)

Dr. B. L. Shapiro Editor/Publisher TAMU NMR Newsletter 966 Elsinore Court Palo Alto, CA U.S.A. 94303

ARE YOUR PEAK RATIOS BETTER THAN YOU THOUGHT?

Dear Barry,

We've been doing some quantitative work, based on collecting spectra in blocks and then doing some statistics on them (see Can. J. Chem., August issue). One of the things that has come up in this work is how to measure peak ratios. If you have blocks of duplicate spectra, you can either average each peak over the blocks and calculate the ratio of the averages, or ratio each block and work out the average of the ratios. We find that in most (but not all) cases, the average of the ratios is better, as shown below. This does not seem to depend much on spectrometer brand, in our experience, but we'd welcome anyone else's experiences.

Yours truly,

Alex D. Bain

Associate Professor of Chemistry

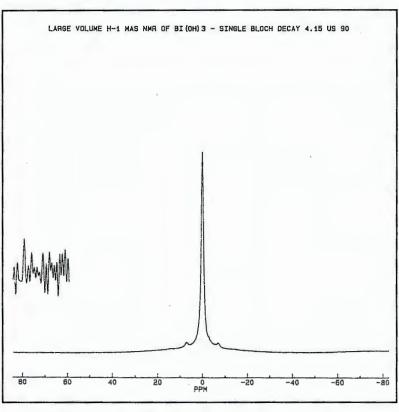
CARBON PEAK HEIGHT RATIOS FOR DUPLICATE SPECTRA

	PEAK 1	PEAK 2	RATIO
	0.5920	0.4133	0.6981
	0.5673	0.3896	0.6868
	0.5737	0.4028	0.7021
	0.5926	0.4201	0.7089
	0.5901	0.4147	0.7028
	0.5958	0.4229	0.7098
	0.5960	0.4170	0.6997
	0.5977	0.4219	0.7059
	0.5952	0.4135	0.6947
	0.5928	0.4216	0.7112
	0.5795	0.4106	0.7085
	0.5636	0.3980	0.7062
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Are Your Enriched Compounds Substituted or Labeled?

August 30, 1991 (received 9/6/91)

Barry Shapiro 966 Elsinore Court Palo Alto, CA 94303

Dear Barry,

In response to the dreaded ultimatum, I'd like to submit the following bit of enlightenment.

I recently submitted a manuscript originally titled "A Convenient Method for the Preparation of ¹³C-enriched D-Fructose Phosphates...". The journal editor in agreement with one of the referees suggested that ¹³C-substituted be used in place of ¹³C-enriched. Furthermore it was suggested that in agreement with present rules on nomenclature ¹³C-substituted be used in place of ¹³C-labeled. Substituted positions should be designated with round and not square parenthesis, i. e., D-(1-¹³C)glucose.

I quickly surveyed the literature and found what I more or less expected; different author and journals used different nomenclature. Furthermore isotopically substituted compounds are almost universally termed labeled, even by the companies who sell them. A call to the editor landed me a copy of the 1979 edition of IUPAC's Nomenclature of Organic Chemistry. Here's a summary:

An **isotopically modified** compound has a macroscopic composition such that the isotopic ratio of nuclides for at least one element deviates measurably from that occurring in nature. These can be either isotopically **labeled** or **substituted**.

An **isotopically substituted** compound has a composition such that essentially all the molecules of the compound have *only* the indicated nuclide(s) at each designated position.

An **isotopically labeled** compound is a mixture of an isotopically unmodified compound with one or more analogous isotopically substituted compound(s).

The formula of an isotopically substituted compound is written as usual with the label notation "hard-up" against the compound name. The notation is contain in *parenthesis*, *not square brackets*.

What isn't clear from all of this is what "essentially all the molecules with the indicated nuclide" means. Does this mean one, ten or a hundred deviations away from natural abundance? Who writes these rules anyway?

Sincerely Yours,

Warren J. Goux

Associate Professor of Chemistry

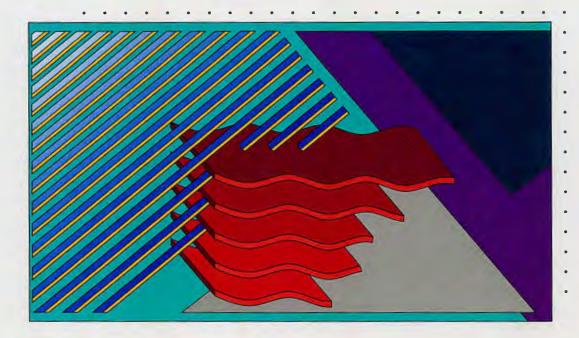


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Dr. B. L. Shapiro TAMU NMR Newsletter 966 Elsinore Court Palo Alto, CA 94303

(received 8/19/91) August 13, 1991

One-dimensional NMR on the Structure and Stability of Serum Retinol Binding Protein: Investigations of a 'Lazarus' Protein

Dear Dr. Shapiro,

During our investigations of the thermodynamic stability of serum retinol binding protein (RBP), a 21 kD protein, we have begun NMR studies to complement the work we have done by other methods (UV-VIS, CD, fluorescence, and DSC). One of the great limitations of studying proteins greater than 15 kD by NMR is that excessive line widths, increased overlap of resonances, and problems with spin diffusion phenomena in such large systems makes complete assignments of such proteins by conventional methods difficult. Indeed, these problems account for only modest increase in resolution at higher fields as is illustrated in Figure 1 of RBP run on our NT-300 and on a Bruker 500. Further, rapid T2-relaxation processes cause not only linewidth problems but limit the use of many 2D methods².

One should not lose hope, there is still a wealth of information to be gleaned even at low field strengths. 1D NMR can yield important information concerning many aspects of protein structure, stability and folding characteristics. For example, prior to thermodynamic analysis, it is necessary to establish the reversibility of the protein unfolding process. Figure 2 shows an overlay of the upfield region of native RBP at 30 C and RBP that has been heated to 85 C for 20 minutes then cooled back to 30 C. The NMR spectrum of RBP at 85 C (not shown) is as would be expected for a random coil structure, corroborating our CD studies that the protein is completely unfolded at this temperature. This result provides evidence that RBP, like Lazarus, can rise from the dead.

Other interesting information is found in the upfield (< 0.8 ppm) region of the spectrum. This region of protein spectra contains mostly methyl resonances that are shifted due to ring current effects from aromatic amino acids. RBP shows several resonances in this region (Figures 1 & 2). The use of ring current shifts in analysis of protein structure is well documented³. Simple calculations of expected ring current shifts using X-ray crystal structure coordinates can lead to initial guesses for determination of resonance assignments. Our initial calculations for shifted methyl resonances coupled with modeling of the subsequent spectrum has yielded some encouraging results. We are now applying a method described by Reid and Saunders⁴ using ring current and NOE calculations with experimental 1D NOE to make assignments of the resonances in this region of the spectrum of RBP.

Please credit this submission to the account of Charles Watkins in our department.

Sincerely,

D. Vincent Waterhous

Don Muccio

- 1. A. Kalk and H.J.C. Berendsen, <u>J.Mag.Reson.</u>, 24, 343 (1976).
- 2. R.Ernst, G. Bodenhausen, and A. Wokaun, Int.Ser.Monogr.Chem., 14,283 (1987).
- 3. S. Perkins, Biol. Magn. Reson., 4, 193 (1982).
- 4. D. Reid and M. Saunders, J. Biol. Chem., 264 (1989).

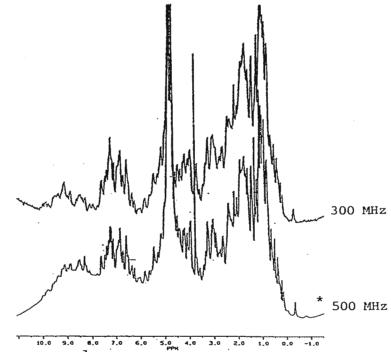


Figure 1. H NMR spectra of RBP

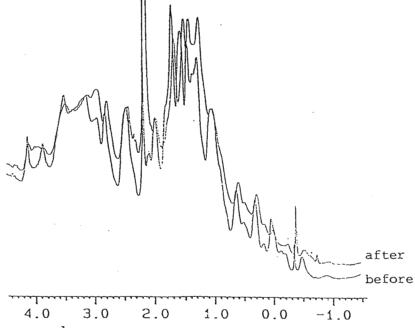


Figure 2. H NMR spectra of RBP before and after heating to 85 C.

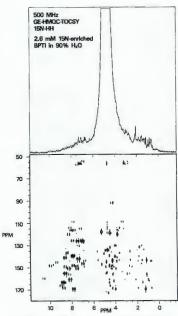
^{*} Grateful acknowledgement to Dr. Russ Timkovitch at University of Alabama in Tuscaloosa for spectra run at 500 MHz.

Gradient Enhanced Spectroscopy: a new, practical answer

By Frank Huang, PhD, Paul Calderon, MS, and Boban John, PhD

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Research Implications

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

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- Provides lineshape independent water suppression via diffusion differences for large molecular weight samples.
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Gradient-Enhanced 15N HMQC

The pulse sequence and the coherence pathway diagram for a ¹⁵N GE-HMQC are shown in Fig. 1. The pulse sequence was a standard ¹³C GE-HMQC experiment (I) with different gradient amplitudes to account for the difference between the gyromagnetic ratios of ¹³C and ¹⁵N. The 90° proton pulse creates transverse magnetization which evolves into an anti-phase state with respect to J(NH) coupling at the end of the period \triangle (where \triangle = ½ J (NH)). The antiphase components are converted into heteronuclear zero- and double-quantum coherence by the ¹⁵N 90° pulse and the multiple quantum coherences are allowed to evolve during t₁. The 180° ¹H pulse in the center of the evolution period serves to eliminate the 1H chemical shift evolution, yielding pure 15N chemical shifts along that axis. The zero- and double-quantum signals are then coherence-order labeled by the gradient pulses G1 and G2. After conversion into antiphase proton magnetization by the last ¹⁵N 90° pulse, the desired components are refocused by the gradient G3 and detected. The application of a gradient pulse results in a phase factor being applied to the magnetization which is dependent upon gradient strength, duration, the distance from the gradient isocenter, the gyromagnetic ratios of the coupled nuclei, and the desired coherence order. The relative amplitudes of the labeling and refocusing gradient pulses will determine the selection of a specific coherence pathway and are calculated to suppress magnetization components arising from the solvent and other protons not coupled to 15N spins.

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

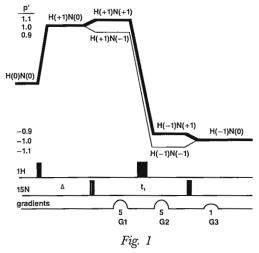
$$G_1 p' 1 + G_2 p'_2 + G_3 p'_3 = 0.$$
 [1]

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

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

and ^{p1}H and ^{p15}N are the coherence orders for the ¹H and ¹⁵N spins respectively.

In the coherence pathway diagram, the relevant values of p' are given to the left and the relative gradient areas



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

(gradient strength x duration) are given next to each gradient pulse. The following pathway (shown in Fig. 1):

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

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

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

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

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

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

which results in a net phase factor:

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

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

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

Gradient-enhanced experiments provide a viable alternative to traditional phase-cycling methods for the selection of coherence pathways. In cases where the sensitivity is adequate, gradient selection can substantially reduce the collection time in multi-dimensional experiments. The ¹⁵N GE-HMQC data presented here has none of the t₁-noise from cancellation artifacts usually present in phase-cycled versions of the HMQC experiment. In addition, since the suppression of the single-quantum signals is done prior to acquisition, the receiver gain may be increased, which results in a substantial increase in signal-to-noise. For these reasons, gradient pulses should be the method of choice for coherence selection in HMQC experiments.

Reference

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

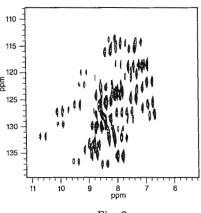


Fig. 2

A 2P Ge-HMQC spectrum of 15N enriched BPTI. The sample was 2.6mM in 90% H2). The data collection time was 2.6 hours.



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22 August 1991 (received 9/6/91)

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

A User-Friendly Selective Scheme Applied to COSY

Dear Professor Shapiro,

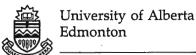
Selective excitation COSY experiment is useful when high resolution is demanded in a particular region of a spectrum while experimental time is limited. Problems associated in this method includes an uncorrectable phase distortion for a simple shaped pulse such as a Gaussian or half Gaussian. The problem can be solved by more complicated shaping of pulses. However, this would require higher precision of the instrument, since more complicated shapes are more sensitive to a small change in the shape. In addition the selectivity is usually low. We employ a simple selective scheme [X. -L. Wu, P. Xu, and R. Freeman, J. Magn. Reson. 83, 404 (1989)] which does not require any shaping in the time domain and is insensitive to the fidelity of the pulse shaping, the total flip angle, and inhomogeneity in the excitation field. As an example, this scheme was applied in a DQ-COSY experiment using a sample of protein-G in 80% H2O. A high-resolution spectrum of the fingerprint region is required in order to obtain J-couplings for structural refinement. The signals are close to the water frequency and are relatively weak compared with other signals. To obtain selective excitation of this fingerprint region, a complete supression of all other signals is needed to avoid aliasing effect in the ω_1 dimension. The resultant spectrum shown below gives a clean spectrum of the region of interest; no phase correction was applied in the ω_1 dimension. The fine structures in the ω_1 are clearly shown, thus permitting the spin systems to be defined and more accurate J coupling constants to be measured. The scheme can be applied to 3D or higher dimensional experiments, where saying of the data storage and experimental time is necessary.

Please credit this contribution to Gordon Roberts' subscription.

Yours sincerely,

Xi-Li Wu

Lu-yun Lian



Department of Biochemistry

Canada T6G 2H7

474 Medical Sciences Building, Telephone (403) 492-5460 Fax (403) 492-0886

August 19, 1991 (received 8/31/91)

TAMU NMR Newsletter c/o Bernard L. Shapiro 966 Elsinore Court Palo Alto, CA 94303

Dear Barry,

Natural Abundance ¹³C hmqc's of Proteins

We have had quite a few changes here (NMR wise) since our last contribution. This includes the installation of a Varian 600 MHz NMR, upgrade of our VXR-500 to a Unity-500, and upgrade of our 300 to a Unity-300 equipped for solids. All three instruments are operating extremely well, especially the 600 which has superb sensitivity and lineshape.

As an example, Figure 1 shows the *natural* abundance ¹³C-¹H hmqc spectrum of a 4mM thioredoxin sample. Shown is the αCH region, the spectrum is ¹³C decoupled during acquisition. Acquisition time for this spectrum was ~24 hrs, although the S/N is such that it could have been less.

This certainly opens many new applications.

Best regards,

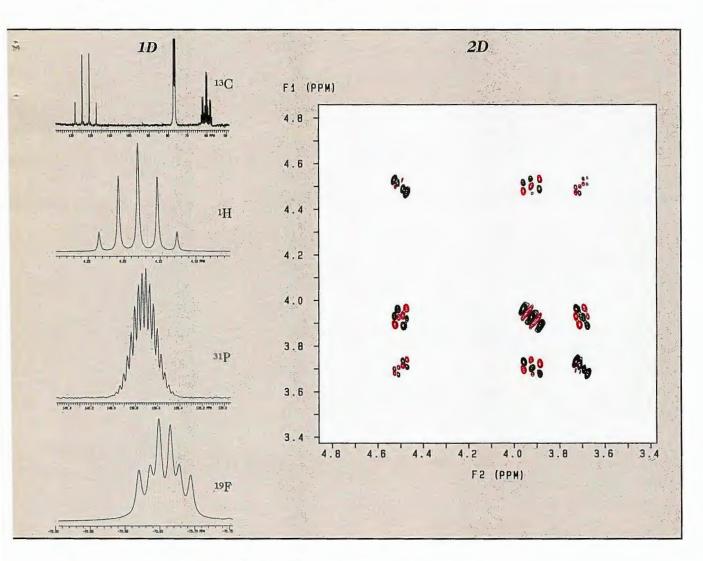
Brian D. Sykes, PhD Professor of Biochemistry

David Wishart, PhD

P.S. Believe it or not, we're getting a lot of golf in here this summer.

approved -BLS.

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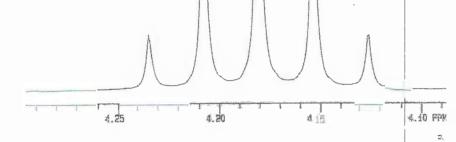
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August 21, 1991 (received 8/24/91)

Professor B. L. Shapiro

2515

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Postadresse: ETH-Zentrum

TAMU NMR Newsletter 966, Elsinore Court CH-8092 Zürich Palo Alto, CA 94303 E-mail: jabr@nmr.lpc.ethz.ch

U.S.A.

Dear Barry,

A COMPUTER-OPTIMIZED CLEAN HOMONUCLEAR TOCSY PULSE SEQUENCE

We would like to report on a computer-optimized pulse sequence for homonuclear coherence transfer in the rotating frame. Our goal has been to maximize TOCSY transfer over an extended resonance offset range and, simultaneously, to suppress contributions from dipolar cross relaxation (ROESY effects), i.e. leading to a "clean" spectrum. It is well-known that during the mixing period, undesired magnetization transfer through cross relaxation can occur simultaneously to coherent magnetization transfer. The resulting cross peaks have oppposite intensity such that overlap with TOCSY cross peaks may lead to cancellation. It may also give rise to additional cross peaks in the final spectrum rendering its interpretation even more difficult. Fortunately, cross relaxation can be inhibited by so-called "clean" pulse sequences which are designed in such a way that the spin trajectory created by the mixing pulse sequence forces the transverse and the longitudinal crossrelaxation effects to compensate each other. This compensation works only for large molecules where the transverse and the longitudinal cross-relaxation rates are of opposite sign.

We have found a new mixing pulse sequence which results from an extensive multiparameter computer optimization of differently designed pulse sequences. The cross-relaxationcompensated pulse sequence consists of a basic unit S which is a composite π pulse:

$$d - \pi_y - 2d - \pi_y - d - R - d - \pi_y - 2d - \pi_y - d$$
.

The time delays d are used to allow for enough influence of the longitudinal cross-relaxation rate (NOESY effects) and are adjusted to suppress cross-relaxation effects. The pulse sequence unit R is also a composite π pulse and its optimized form turns out to be a three-pulse sequence: (-48°_{x}) 276°_{x} , -48°_{x}). The basic unit S is subsequently expanded into a supercycle SSSS. By due respect to the environment, this sequence is henceforth referred to as the clean CITY (clean Computer-Improved TocsY) sequence. However, we are not quite yet into the "recycling" business where we can make good use of the suppressed "wastes" originating from dipolar cross relaxation.

The performance of clean CITY was experimentally evaluated on BPTI, a small globular protein with 58 residues. A "dirty" 300 MHz TOCSY spectrum obtained with the MLEV-17 pulse sequence is shown in Figure 1A. The cross peaks painted in black are due to cross relaxation. A "clean" TOCSY spectrum obtained with clean CITY is illustrated in Figure 1B. For a more quantitative comparison of clean CITY and the previously known clean MLEV-17 sequence, a series of TOCSY spectra with different mixing times were recorded with both pulse sequences. The integrated cross peak intensities show a 40-50 % improvement in the one-step transfer efficiency Tyr^{23} NH- α H (Fig. 2A). The one-step transfer Tyr^{23} ϵ H- δ H (Fig. 2B), on the other hand, is the same for both pulse sequences, as expected, since the offset difference is very small. This work is reported in more detail elsewhere (1).

Sincerely yours,

Jacques Briand

Richard R. Ernst

Millan!

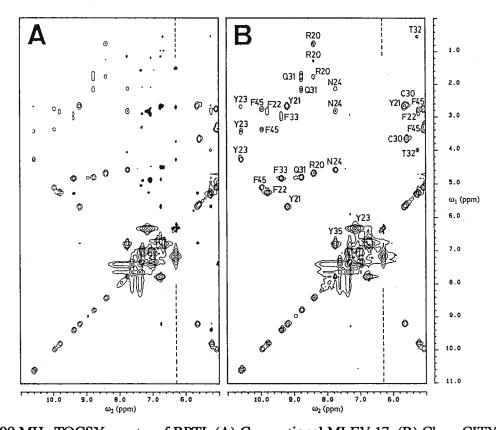


Fig. 1. 300 MHz TOCSY spectra of BPTI. (A) Conventional MLEV-17. (B) Clean CITY.

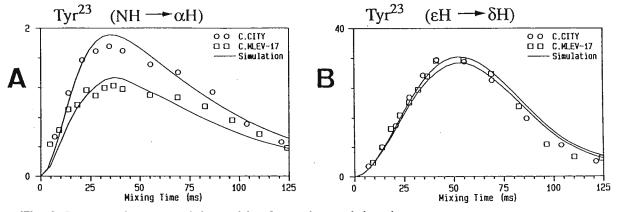
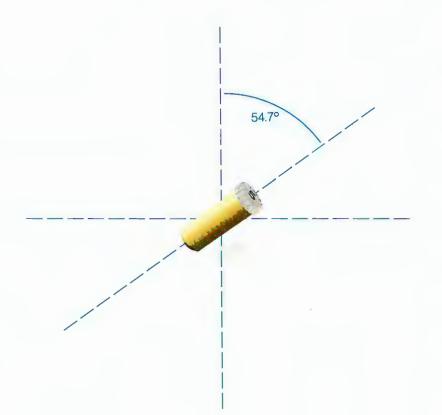


Fig. 2. Integrated cross peak intensities for various mixing times.

(1) J. Briand and R. R. Ernst, Chem. Phys. Letters, in press.



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DEPARTMENT OF CHEMICAL ENGINEERING BERKELEY, CALIFORNIA 94720 Sept. 12, 1991 (received 9/14/91)

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

Re: ¹³C Chemical Shift Assignment for Vectra

Dear Barry,

After having survived my Ph.D defense, I joined Jeff's group at U. C. Berkeley and tried to learn how to relax by examining the relaxation times of protons in thermotropic liquid crystal polymer (TLCP) samples. As the resolution of proton solid state NMR spectra of TLCP is too low to observe the proton relaxation time of each component in a polymer blend, we have utilized the technique of ¹³C observation of ¹H inversion recovery through cross polarization. One problem we faced was the overlap of the spinning sidebands of some carbons with the isotropic resonances of other carbons, making chemical shift assignment extremely difficult. This problem is now solved. Thanks to a clever sideband elimination pulse sequence (SELTILS) designed by Gerry of SUNY at Stony Brook (see TAMU P.11, July 1990).

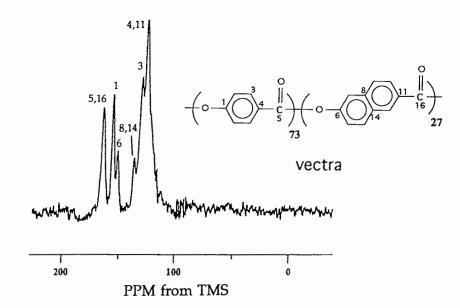
Let me use Vectra as an example to show the success of this sideband elimination method. The figure below shows the first high-resolution solid state ¹³C NMR spectrum of Vectra. The chemical shift values for the assigned carbons may be useful for my colleagues in liquid crystal polymer NMR research.

Sincerely Yours,

Pei Tang

Postdoctoral Researcher

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Gain (typ.) Gain flatness		65 dB 60 dB ±4 dB ±3 dB
Input/Output impedance Input VSWR		0 ohms 2:1
Pulse width		0 ms
Duty cycle Amplitude rise/fall time Amplitude droop	2	p to 10% 00 ns typ. 150 ns typ. % to 10 ms typ; 7% max
Phase change/power output	1	0° to rated power, typ.
Phase error overpulse Noise figure		<pre>to 10 ms duration, typ. 1 dB typ. 8 dB typ.</pre>
Output noise (blanked)		20 dB over thermal
Blanking delay	<	2 μ s on/off, TTL signal
Protection	2	. VSWR: infinite VSWR at rated power . Input overdrive: up to +10 dBm . Over duty cycle/pulse width . Over temperature
Supplemental characteristic		. Over comperature
Connectors, rear panel	2	 RF input: BNC (F) RF output: Type N (F) Noise blanking: BNC (F) Interface: 25 pin D(F), EMI filtered
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TAMU NMR Newsletter

Policies and Practical Considerations

(Revised September 1991)

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 beginning its thirty-fourth 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. <u>Participation is the prime requisite for receiving the TAMU NMR Newsletter:</u> 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 <u>Subscription</u> fee for the October 1991 - September 1992 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 <u>Sponsors</u> of the Newsletter. Sponsors' names appear in each month's Newsletter, and copies of the Newsletter are provided to all Sponsors. The continuation of

this non-commerical 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 <u>Advertising</u>. 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 8.5×11^n (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 $0.5 0.75^n$ (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 8.5×11^n pages. We are not equipped to handle pieces of paper larger than 8.5×11^n (21 x 27.5 cm).

Please do not fold, clip, or staple your pages. Protect the condition of your letters from the ravages of the mails by enclosing what you send in a cardboard or plastic folder, etc.

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, 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. <u>Suggestions</u>: They are always welcome.

3. L. Shapiro

Address for all correspondence:

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

Telephone: (415) 493-5971. Please confine telephone calls to the hours from 8:00AM - 10:00PM, Pacific Coast Time.



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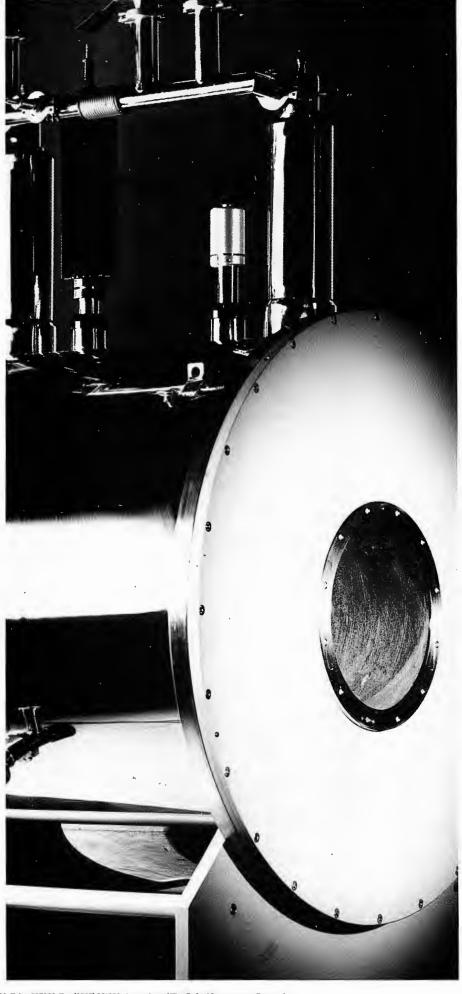
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POSITIONS AVAILABLE

BIOMOLECULAR RESEARCH INSTITUTE NMR LABORATORY

Applications are invited for two Research Scientist positions in the NMR Laboratory of the Biomolecular Research Institute. The primary focus of the BRI will be determination of the structure and structure-function relationships of biological macromolecules, with particular emphasis on molecules having therapeutic potential. Extensive collaboration will be undertaken with tertiary and medical research institutions throughout Australia.

The successful applicants will participate in a range of projects on the structure and dynamics of biopolymers and their interaction with ligands. They will also have the opportunity to contribute to the development and implementation of new methods for acquiring and processing multi-dimensional NMR data and for generating molecular structures. The NMR Laboratory is equipped with Bruker AMX-600 and -500 spectrometers, supported by a network of UNIX workstations. A Convex C-210 computer is also available.

A PhD in the chemical or biological sciences and experience with NMR spectroscopy are essential. A strong background in computing will be an advantage. The positions are available immediately, with appointment for three years in the first instance. Salary will be A\$35-45,000 depending on experience.

Inquiries may be directed to the Head of the NMR Laboratory, Dr. Ray Norton. Phone: 61-2-697 2037, FAX: 61-2-313 6271. email: norton@csdvax.csd.unsw.oz.au.

Applications (including the names and addresses of at least two referees) to the Director, BRI, 343 Royal Parade, Parkville 3052, AUSTRALIA.

FOR SALE, VARIAN XL200. Beckman Instruments, Inc., Research and Applications Department, Palo Alto, offers for sale a VARIAN XL200. Price--best offer. Purchased new in 1985, currently in use, the instrument is in good working order. The probe is broadband/switchable (20-80/200 MHz), the magnet has rubber (adjustable) seals, and the host and acquisition computer systems are stock issue. The instrument includes a Topaz Power Conditioner and HP7475A Plotter (6-pen). Please contact Mr. Jeffrey Smith, Beckman Instruments, Inc., 1050 Page Mill Road, Palo Alto, California, 94304; (415)859-1565.

POST-DOCTORAL POSITIONS

As part of a new initiative in structural biology several new NMR laboratories with interests in protein structure and function are being established in Toronto, Ontario, Canada. Research interests of the new labs include protein structure, folding and dynamics (J.F.-K. & L.E.K), design and optimization of multidimensional NMR experiments (L.E.K), protein-DNA and protein-protein interactions (C.H.A. & M.I.), in addition to the established program of J.P.C. (polysaccharide and glycoprotein structure and dynamics). There will be the opportunity for collaboration among these laboratories as well as with other molecular modelling and crystallographic groups in the city. Currently available instrumentation includes 500 MHz (4) and 600 MHz (1) spectrometers (4 of these are brand-new with multichannel, multinuclear capabilities). An additional 500 and 600 will be acquired within the next two years. Candidates should have experience in one or more of the following areas: multidimensional NMR, structure determination of macromolecules, isolation and purification of proteins, molecular biology, and/or scientific computing. To apply for positions, interested candidates should send their CV to the appropriate investigators listed below. Openings are available starting from Dec. 1991 onward.

Dr. Cheryl H. Arrowsmith
Ontario Cancer Institute and Dept.
of Medical Biophysics, U. of Toronto
500 Sherbourne St.
Toronto, Ont. M4X 1K9
FAX (416) 926-6529

Address until Nov. 1991
Stanford Magnetic Resonance Lab.
Stanford University
Stanford, CA 94305-5055
FAX (415) 723-0010

Dr. Mitsuhiko Ikura
Ontario Cancer Institute and Dept.
of Medical Biophysics, U. of Toronto
500 Sherbourne St.
Toronto, Ont. M4X 1K9
FAX (416) 926-6529

Address until Dec. 1991
National Institutes of Health
NIDDK/Lab of Chemical Physics
Building 2, Room B2-08
Bethesda, MD 20892
FAX (301) 496-0825

Dr. Julie Forman-Kay
The Hospital for Sick Children
Biochemistry Research Division
555 University Avenue
Toronto, Ont. M5G 1X8

Address until Jan. 1992
National Institutes of Health
NIDDK/Lab of Chemical Physics
Building 2, Room B2-11
Bethesda, MD 20892
FAX (301) 496-0825

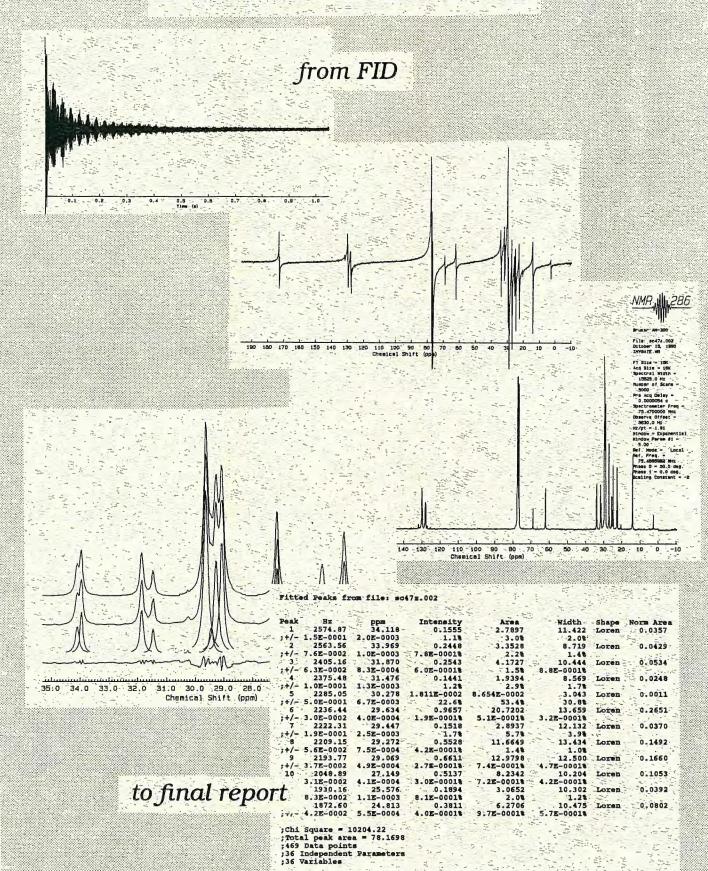
Dr. Lewis E. Kay
Depts. of Medical Genetics and Chemistry
Medical Sciences Bldg.
University of Toronto
Toronto, Ont. M5S 1A8
FAX (416) 978-4194

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Building 2, Room B2-08
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- Manual. A comprehensive reference manual comes with NMR-286. It explains not only

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THE CHEMICAL LABORATORIES

September 6, 1991 (received 9/12/91)

Dr. Barry L. Shapiro 968 Elsinore Ct. Palo ALto, CA 94303

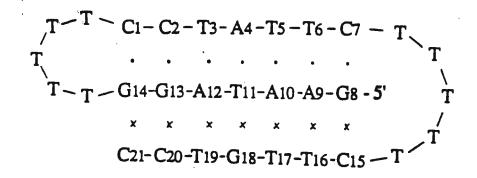
Dear Barry:

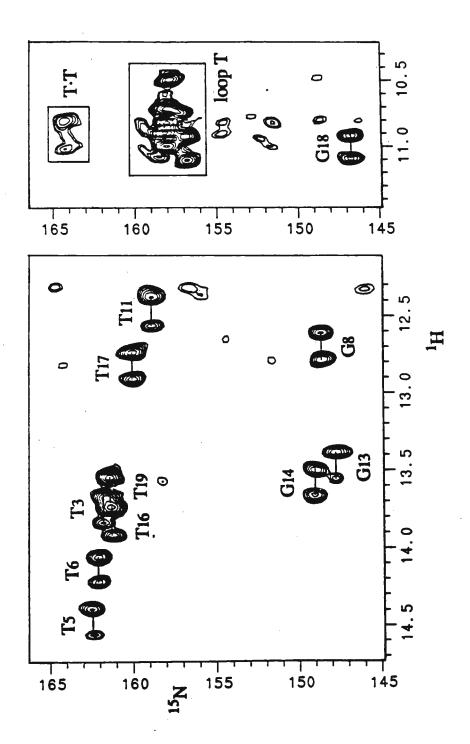
¹⁵N NMR of Triple Helix Oligonucleotide

In our continuing exploration of heteronuclear NMR of oligonucleotides in collaboration with Dinshaw Patel's lab, we have examined the ¹⁵N NMR at natural abundance of a 31 residue oligo that organizes itself into a triple helix with the three strands connected by two loops of T's. This molecule further incorporates an unusual G'TA triple as proposed by Griffin and Dervan (Science, 245, 967-971 (1989)). A H - 15N spectrum is shown in the accompanying figure. As a consequence of the length of the oligonucleotide and its unusual conformation, the imino la spectrum is complex as is the 15N spectrum, with T and G imino proton resonanaces not clearly distinguishable on the basis of their $^{1}\mathrm{H}$ chemical shifts. In the $^{15}\mathrm{N}$ dimension there is a clear dinstinctio between the shifts of the more downfield T and more upfield G resonances, even though the $^{15}{\rm N}$ shifts of each kind of base is spread over a considerable range. Of interest is the large cluster of ¹H signals between about 10.5 and 11.0 ppm. The bulk of these are from T's in the loop segments, however ¹H studies had already indicated that signal from the G in the G·TA triple occurred in this region. The ¹⁵N shift clearly separates this from the others. There are additionally cross peaks shifted downfield in the ¹⁵N dimension from the main group of T resonances. These may arise from wobble base pairs in the loops that were suggested from $^1\mathrm{H}$ data. Assignments are based on analysis of the ¹H spectrum. The spectra were taken on a 0.4 ml sample in 95%H₂O containing about 1000 OD units of oligonucleotide using the coupled HMQC experiment. 128 blocks of data were collected over a 60 hr period. The spectra were run on a GN-500 spectrometer at Emory University, a major part of whose purchase cost was underwritten by my NSF grant DMB 8604304.

Sincerely yours,

David Live





.

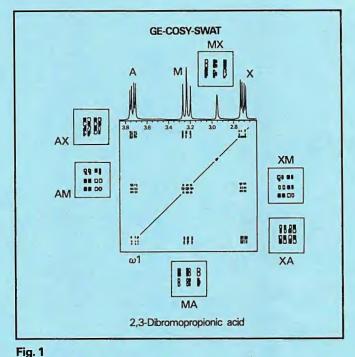
Gradient Enhanced Spectroscopy SWAT

GE introduces the use of Switched Acquisition Time (SWAT) gradients to achieve pure phase 2D spectra with quadrature detection in both the acquisition (ω_2) and evolution (ω_1) dimensions without any phase cycling and without an additional set of t_1 data.

One example of a pure phase gradient enhanced COSY spectrum of a solution of 2,3-dibromopropionic acid in benzene-d6 is shown in Fig. 1. SWAT gradients and a single acquisition per block were used. Data was collected on an Omega 300WB with Microstar actively-shielded gradients. A 5mm inverse probe was built for use within the gradient coils.

Digital resolution of 1.2 Hz in ω_1 and 2.4 Hz in ω_2 was achieved by collection of a 512 x 512 matrix with t_1 evolution time of 840ms and a t_2 acquisition time of 420ms. A single acquisition per t_1 evolution data block and an average recycle time of 1.84s resulted in a 15 minute total collection period.

Since the SWAT gradient method encodes the necessary information in a single t₂ acquisition time, it avoids the collection of additional data blocks required by traditional pure phase methods. This time efficiency is especially important for collection of large multidimensional data sets.

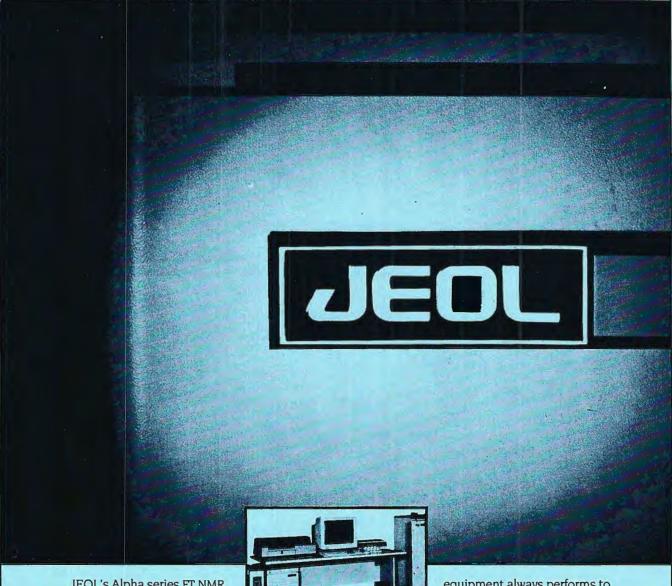


Contour plot of a 300 MHz pure-phase COSY spectrum of a solution of 2,3-dibromopropionic acid in benzene-d6 acquired with only a single acquisition per t₁ evolution time increment using the GE-COSY-SWAT method. Cross peaks are shown in expanded insets with positive peaks as darkened contours and negative peaks as open contours. A one dimensional spectrum is plotted across the top of the 2D spectrum.



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