THE NMR NEWSLETTER

No. 471

December 1997

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	Frequency Range	Resolution	Switching Time[1]	Phase-Continuous Switching[2]	Rack-Mount Cabinet Dim.[3]	Remote-Control Interface	Price Example[4]
PTS 040	.1-40 MHz	optional .1 Hz to 100 KHz	1-20μs	optional	5¼"H×19"W	BCD (std) or GPIB (opt)	$5,330.00 (1 Hz resol., OCXO freq. std.)
PTS 120	90-120 MHz	optional .1 Hz to 100 KHz	1-20μs	optional	5¼"H×19"W	BCD (std) or GPIB (opt)	$5,330.00 (1 Hz resol., OCXO freq. std.)
PTS 160	.1-160 MHz	optional .1 Hz to 100 KHz	1-20μs	optional	5¼"H×19"W	BCD (std) or GPIB (opt)	$6,495.00 (1 Hz resol., OCXO freq. std.)
PTS 250	1-250 MHz	optional .1 Hz to 100 KHz	1-20μs	optional	5¼"H×19"W	BCD (std) or GPIB (opt)	$7,440.00 (1 Hz resol., OCXO freq. std.)
Type 1 PTS 310 Type 2	.1-310 MHz	1 Hz	1-20μs	standard	3½"H×19"W	BCD (std) or GPIB (opt)	1 Hz resol., OCXO: $6,425.00 1 Hz resol., OCXO: $5,850.00
PTS 500	1-500 MHz	optional .1 Hz to 100 KHz	1-20μs	optional	5¼"H×19"W	BCD (std) or GPIB (opt)	$8,720.00 (1 Hz resol., OCXO freq. std.)
PTS 620	1-620 MHz	optional .1 Hz to 100 KHz	1-20μs	optional	5¼"H×19"W	BCD (std) or GPIB (opt)	$9,625.00 (1 Hz resol., OCXO freq. std.)
PTS 1000	0.1-1000 MHz	optional .1 Hz to 100 KHz	5-10μs	optional	5¼"H×19"W	BCD (std) or GPIB (opt)	$11,830.00 (1 Hz resol., OCXO freq. std.)
PTS 3200	1-3200 MHz	1 Hz	1-20μs	optional	5¼"H×19"W	BCD (std) or GPIB (opt)	$14,850.00 (1 Hz resol., OCXO freq. std.)
PTS x10	user specified 10 MHz decade	1 Hz	1-5μs	standard	3½"H×19"W	BCD (std) or GPIB (opt)	$3,000.00 (1 Hz resol., OCXO freq. std.)
PTS D310	two channels .1-310 MHz	.1 Hz	1-20μs	standard	5¼"H×19"W	BCD (std) or GPIB (opt)	$8,560.00 (.1 Hz resol., OCXO freq. std.)
PTS D620	two channels 1-620 MHz	.1 Hz/.2 Hz	1-20 μs	standard	5¼"H×19"W	BCD (std) or GPIB (opt)	$13,240.00 (.1 Hz/.2 Hz resol., OCXO freq. std.)

1 Switching Time is dependent on digit (decade) switched; see detailed instrument specifications.

2 For applicable digits, see detailed instrument specifications.

3 Bench cabinets are 17" wide.

4 Prices are U.S. only and include Manual and Remote (BCD) Control; PTS 3200 Digital Front Panel.

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FORTHCOMING NMR MEETINGS

39th ENC (Experimental NMR Conference), Asilomar Conference Center, Pacific Grove, CA, **March 22 - 27, 1998**; Contact: ENC, 1201 Don Diego Avenue, Santa Fe, NM 87505; (505) 989-4573; Fax: (505) 989-1073; Email: enc@enc-conference.org. See Newsletter 460, 41.

Sixth Scientific Meeting and Exhibition, International Society for Magnetic Resonance in Medicine, Sydney, Australia, **April 18 - 24, 1998**. Contact: International Society for Magnetic Resonance in Medicine, 2118 Milvia St., Suite 201, Berkeley, CA 94704; 510-841-1899.

NATO ARW "Applications of NMR to the Study of Structure and Dynamics of Supramolecular Complexes", Sitges (Barcelona), Spain, **May 5 - 9, 1998**. Contact: Prof. M. Pons, Dept. Quimica Organica, Univ. de Barcelona, Mart I Franques 1, 08028 Barcelona, Spain; http://www.ub.es/nato/nato.htm; e-mail: miguel@guille.qo.ub.es.

^{13}C in Metabolic Research, Symposium at the University of Texas Southwestern Medical Center, Dallas, Texas, **May 7, 1988**; For more information, contact Jean Cody at 214-648-5886 or www.swmed.edu/home_pages/rogersmr.

Fifth International Conference on Heteroatom Chemistry, London, Ont., Canada, **July 5 - 10, 1998**. For details, see Newsletter 468, 40.

XIVth International Conference on Phosphorus Chemistry, Cincinnati. OH, **July 12 - 17, 1998**. For details, see Newsletter 468, 40.

NMR Symposium at the 40th Rocky Mountain Conference on Analytical Chemistry, Denver, CO, **July 27 - 30, 1998**. Contact: Dr. Robert A. Wind, Battelle/Pacific Northwest National Laboratory, P.O. Box 999, MS K8-98, Richland, WA 99352; (509) 376-1115; Fax: (509) 376-2303; Email: ra_wind@pnl.gov. See Newsletter 470, 8.

Additional listings of meetings, etc., are invited.

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October 24, 1997
(received 10/29/97)

Barry Shapiro
NMR Newsletter

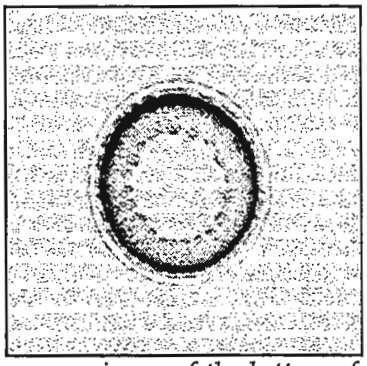
Cut Flowers by NMRI

Dear Barry:

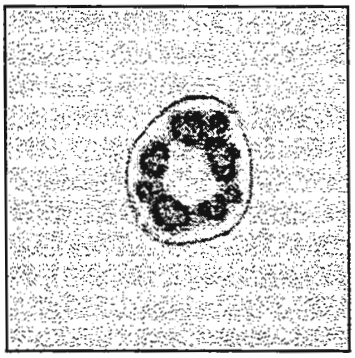
Here at UC Davis the large and diverse agricultural emphasis has intersected again with NMR in the form of a novel (we think) application of MRI to cut flowers, specifically roses.

The failure of roses to rehydrate after storage and transport, and early collapse in the vase, are twin sources of frustration to rose growers, retailers, consumers, and romantics. Despite more than four decades of active research, there is still controversy as to the cause of vascular failure. Apparently, the main reason for the occurrence of a water deficit is a blockage to water flow that develops in the stem.

Only indirect methods have been used to establish the role of the different parameters affecting the water uptake of cut roses. Relatively recent studies have shown the potential of MRI microscopy in plant tissue. NMR is the only noninvasive method available today to investigate water behavior in cut flowers. Preliminary data (see figures) have been obtained on the UCD NMR Facility's GE-NMR Omega horizontal 7 Tesla system show the feasibility of using NMRI to probe the structure of cut rose stems.



Transverse image of the bottom of the stem of a cut rose (Tineke cultivar)



Transverse image of the top -near to the flower- of the stem of a cut rose (Tineke cultivar)

Pixel resolution 40 μm x 40 μm, Slice thickness 2 mm, Acquisition time 3 min

These images were acquired with a homebuilt 15 mm i.d. slotted tube resonator coil and Nalorac 65 mm i.d gradients. Data was acquired with a standard spin-echo sequence, using TE = 18 msec, TR = 0.7 sec, 2 scans/increment, and 128 phase encode steps.

Our principal goals with the NMRI technique will be to obtain water distribution (proton density), water state (relaxation times), and water transport (flow visualization) in the stem. NMRI experiments will help us to determine and understand the role of vase solutions, hydrostatic pressure, xylem structure and other factors involved in rose stems during rehydration, and hopefully eventually lead to a brighter Valentines Day for all recipients of floral bouquets.

Michael S. Reid	Serge Bobroff	Rosa Valle	Jeff de Ropp
Env. Horticulture	Materials Science	Env. Horticulture	NMR Facility

P.S. Please credit this contribution to Gerd La Mar's subscription.

AMT

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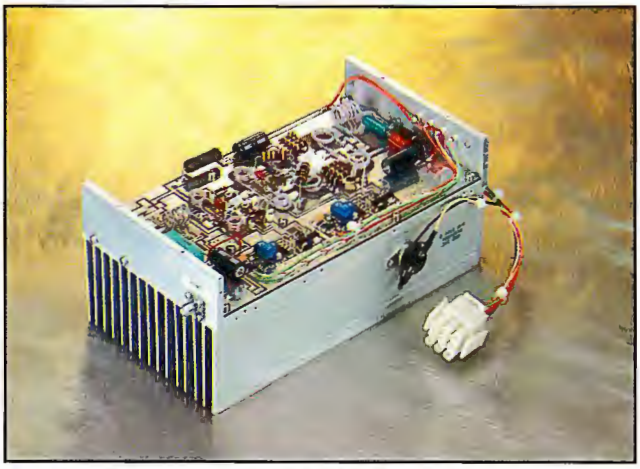
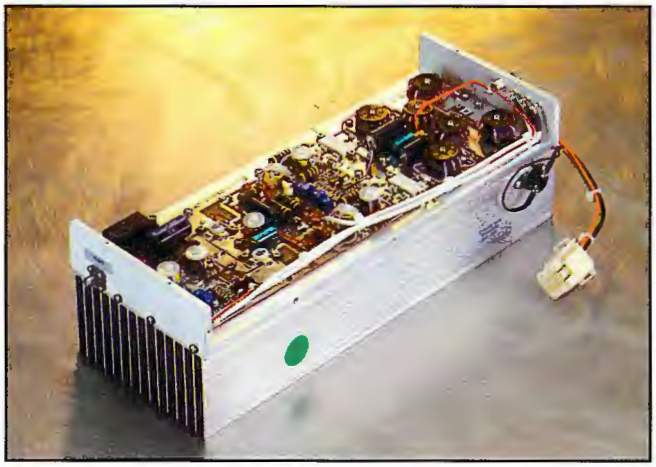
Model 3205 – 6 MHz to 220 MHz, 300 W, NMR Amplifier

Model 3445 – 10 MHz to 130 MHz, 2.0 kW, MRI Amplifier



200 MHz to 500 MHz, 50 W, NMR Module



185 MHz to 500 MHz, 125 W, NMR Module

Model 4T70 – 25 MHz to 175 MHz, 7.0 kW, MRI Amplifier

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SCIENTIFIC & MEDICAL PRODUCTS

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AMT designs, develops and manufactures custom radio frequency (RF) and microwave power amplifiers for the wireless, scientific/medical and application specific industries. The company has been in business since 1984 and currently has over 60 employees, including 20 experienced engineers.

AMT has a worldwide reputation as a leading supplier of high power, solid state power amplifier products that operate at frequencies between 1 MHz and 3 GHz and provide RF power from several watts to several kilowatts. Its products are noted for their exceptional performance, highest quality and superior reliability.

The company's products are sold to numerous major corporations, universities and research centers throughout the world.

FACILITIES

AMT is located in Anaheim, California and occupies a 25,000 square foot facility allocated to engineering, manufacturing, quality assurance, marketing/sales, administration and finance.

Engineering areas include an R & D laboratory, a tool and die shop, mechanical design and drafting areas, an environmental testing laboratory and document control. The R & D laboratory is equipped with all of the latest design and testing equipment including intermodulation distortion simulators, network analyzers, spectrum analyzers, signal generators, noise figure meters and infrared (IR) scanners. The environmental testing laboratory includes equipment to simulate shock, vibration and thermal environments.

Manufacturing areas include a controlled access stock room, a 10,000 square foot assembly area and a production test area employing automatic testing. Also included is an environmental laboratory used for environmental stress screening of production products.

PRODUCTS

AMT's products vary in complexity from single modules, to rack-mounted amplifiers, to complete transmitter systems. The rack-mounted amplifiers and complete transmitter systems typically include detection/protection circuitry, built-in power supplies, front panel metering and digital and/or analog interface controls. Both forced air and/or water cooling are used, depending on the customer's requirements.

AMT's products feature highly reliable technical solutions designed for producibility and reliability. Producibility is enhanced through the use of surface mount components and circuit designs that eliminate the need for excessive alignment during the production cycle. High reliability is accomplished through the implementation of conservative thermal and RF circuit design and sophisticated self-protection schemes. Reliability is further enhanced during the design phase by employing detailed environmental testing.

These factors, along with computer driven automatic testing and environmental stress screening of the final product, ensure that the performance, quality and reliability meet AMT's exacting standards.

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(received 11/10/97)

Protein NMR, Peer Review and *Esprit de Corps*

Dear Barry,

Your call for contributions that deal not with technical detail, but with the philosophy of the field is more than timely and I am happy to respond.

There exists a long standing legend in Structural Biology that protein crystallographers are a model community built on mutual respect and recognition and active mutual support. The counter-legend is that the biological NMR community is the opposite, torn apart by excessive criticism, put-downs and undercutting. It may be difficult to assess how much truth there is to these legends, but the verifiable fact remains that the average support of a protein NMR laboratory in the US is less than a third of the average support of an established protein crystallography program - even though the costs of modern NMR are higher than those of crystallography. An unmistakable sign that there *is* a problem, and *some* truth to the legends, appears when one is confronted by a distinguished University president - and several eminent journal editors - with the blunt question: "What is wrong with you people in biological NMR? We can't appoint anyone in your field - no one has a good word to say about anyone else."

There is every reason to be surprised that a question of this kind would ever be raised. Protein NMR has by now found a unique place in the armamentarium of Structural Biology - it has allowed us to look at solution structures and recognize that solution structures are not always the same as those seen in the crystals. It has made it possible to study the structure of proteins that cannot be crystallized and has taught us much more about protein dynamics than other experimental methods ever could. Its potential in the study of molecular interactions and information transfer has barely been tapped. Many more than a single individual are responsible for its success and the development of an exceptionally wide range of techniques and ingenious applications. The field has every reason for collective pride. Yet the perception is that there is more criticism than pride. To be sure, criticism is necessary to set the standards in a field and being critical is a laudable trait of a good scientist - though only as long as the scientist remembers to be critical of his own criticisms as well.

If one reads carefully the critiques on the basis of which funding in the field is denied, cut or at the very least delayed, papers are rejected or sent back for revision, and appointment and tenure decisions are adversely affected, one can indeed readily find statements that should be questioned.

A very favorite cause for hasty rejection in grant reviews is "insufficient preliminary data" - as some have put it: for crystallographers - no crystal, no grant, for NMR - no 2D spectrum, no

Express/Courier Address: Edwards Bldg., Room R320, Stanford University Medical Center, 300 Pasteur Drive
Email: jardetzky@camis.stanford.edu

grant. At first glance, it sounds eminently reasonable, but it quickly becomes unreasonable, especially when qualifiers such as "an interpretable" spectrum are added or a demand made that "a 3D spectrum would be more convincing." For most interesting larger proteins the basic NMR spectra are not interpretable and it takes extensive isotopic spectral editing to make them so. This requires funds and time that exceed the legitimate scope of preliminary results which have to be obtained with little or no funding. Even working out the conditions for successful NMR spectroscopy on interesting proteins can take more work than is possible without funding. A similar blanket rule - "insufficient number of NOEs per residue" - has often been invoked to reject protein structure papers, even when a more careful look would show that part of the molecule was well defined by more than enough NOEs per residue, and part was ill-defined for physical reasons, not through the sloppiness of the authors. Simple minded application of such simple criteria is destructive.

Even more dangerous are misstatements of fact ("the applicant does not..." when in fact the applicant does) or negative comments that have no bearing on the quality of the work. A comment encountered more than once is "the problem is not in the mainstream of current research" (or "too difficult"). The danger of creating and enforcing fashions by such glib value judgments should be self-evident. Significant innovation often does not fit preconceived notions and accustomed values. The most dangerous are those who have learned to use computer programs for a particular model and argue as referees that phenomena not predicted by the model cannot exist and those who investigate them don't know what they are doing. In the study of NMR relaxation such criticisms have become commonplace.

Many other reviewer comments which have had adverse effects without just cause can be cited. A protein chemist famous for having focused his entire career on the solution of one major problem was refused a grant on the grounds that his research was unfocussed. An applicant identified as a "fine investigator" and judged to have an "excellent hypothesis" had an application declined on the grounds that it was "overly ambitious" and another because it "contained too much methodological detail." Another established group had a paper turned down because "it failed to solve the precipitation problem" even though it clearly stated that all results were obtained at 25°C where there was no precipitation. Among the most charming was the denial of an application for a modern instrument to one of the best known groups in the field on the grounds that "this group cannot justify a 600 MHz spectrometer, because it is doing such elegant work with the 500."

Criticism unrestrained by self-criticism is not uncommon in the early phases of a new development, when the standards are still emerging, the rules of the game themselves are still in the making and everyone still believes in the initial burst of enthusiasm to hold a monopoly on truth. Biological NMR has undergone an explosive growth in the past decade, the number of papers per year rising by a factor of 10, and the number of meetings by almost as much. Not surprisingly, the climate bears many of the earmarks of a gold rush era - aggressive staking of claims, rushing to publication, paying little attention to rigor and to the work of others. Yet the time has come to sort out the reasonable from the unreasonable and to consider the consequences of not doing so.

Spurious criticism can harm not only the person against whom it is directed, but the critic and his entire field. The critic who habitually uses sweeping negative judgments eventually discredits himself, and the prevalence of such criticisms in a research area discredits the field. Even one responsible university officer who finds the field has this problem is one too many. Vague criticism can also easily be wrong. Richard Ernst in his historical article in the NMR Encyclopedia

points out that his 1966 paper which now forms the basis of all modern NMR was rejected by the Physical Review as "trivial" and "unoriginal."

Responsible criticism has clear standards. It focuses on issues that are important in making good science - importance of the problem, clarity and originality of the conception, adequacy of the methods and the track record of the investigator. It is specific and well-reasoned. It avoids sweeping generalities and issues of secondary importance - such as the amount of detail in an application. It acknowledges the creativity of others and does not judge by rules that may apply to the familiar special case, but may not apply in general. It recognizes that the new way one favors may not be the only good way to make progress. And it stays clear of cynicism that uses doing well as a reason to prevent others from doing better. Far too many NMR reviewers' comments that I have seen in recent years do not meet these standards.

The vicious cycle set off by spurious criticisms may be difficult to break. Reviewers are human. If treated harshly by their colleagues, they are likely to become harsh themselves. As a result, less work will be supported, opportunities will be lost and less will be accomplished. The beneficiaries will be fields of research in which the experts are more supportive of each other - even though they may be less productive. The outside onlookers are easily led to the natural conclusion - if each of the experts thinks no one except himself is any good in this field, they must collectively be right - no one is much good and the field should not be supported. To have lasting success, to be maximally productive, to be respected and maximize its opportunities, a field must have self-respect, an *esprit de corps*. An *esprit de corps* is not created by the well-known battle cry of a decade ago "we will put the crystallographers out of business." It is created by treating every contribution to the field - proposed or accomplished - with care and respect and basing recommendations for support or publication on major strengths, not on minor weaknesses.

Now that the dust is settling and protein NMR has come of age we would do well to scrutinize the cogency of our arguments and search our souls for the purity of our motives when judging our colleagues. If we remember that "do unto others as you would have them do unto you" was one of the best pieces of advice ever given, it may not be too late to develop an *esprit de corps* - or rather - restore it: An *esprit de corps* did exist in the first twenty years when the groundwork was laid but the field was still small. It was not until some members of the next generation claimed monopoly for all significant achievements and began aspiring to a personality cult, quoting only themselves and ignoring or belittling the contributions of others, that rancor spread. It is now up to the youngest generation to recapture good will, balanced judgment and good humor - and earn the field as a whole the appreciation and respect it deserves.

Oleg Jardetzky

NORTHWESTERN UNIVERSITY

Joseph B. Lambert
Clare Hamilton Hall Professor of Chemistry

November 13, 1997
(received 11/17/97)

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

Dear Barry:

Last year we reported (JACS, **118**, 7867-7868 (1996)) the first example of a stable β-silyl carbocation, $Et_3SiCH_2CPh_2^+$ $(C_6F_5)_4B^-$. The noninteracting structure of the anion and the low nucleophilicity of the arene solvent led to ions that are stable for weeks at room temperature, in contrast to magic acid conditions, under which ions normally do not survive above -100°C. Because we were unable to obtain crystals for X-ray analysis, we relied on NMR for the structure proof.

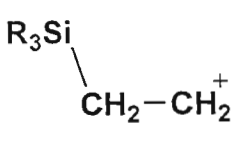
We now have prepared the germanium version of this cation. The tin version apparently does not form. In addition, we have obtained the first γ-silyl carbocation. All these materials were characterized primarily by various NMR probes, which are listed in the table below.

cation	$\delta(C^+)$	$\delta(CH_2)$	$\delta(C_p)$	$^1J(C_\beta\text{-H})$, Hz	δ(Si)
$H\text{-}CH_2\text{-}CPh_2^{+a}$	228.1	30.7	146.8	141.4	
$Et_3Si\text{-}CH_2\text{-}CPh_2^+$	225.4	56.2	141.1	138.5	46.2
$Bu_3Ge\text{-}CH_2\text{-}CPh_2^+$	213.4	66.4	138.6	146.5	
$CH_2{=}CPh_2$	150.1	114.1	127.6	158.1	
$Et_3Si\text{-}CH_2CH_2\text{-}CPh_2^+$	210.2	42.0, 45.1			59.6

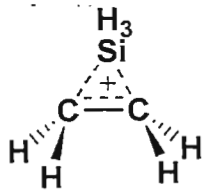
[a]Measured as the fluorosulfonate in CD_2Cl_2 at -40°C.

The first entry is for the unstabilized carbocation, and the next to the last entry is for the starting material alkene. These two species represent the extremes of no hyperconjugation and complete hyperconjugation (hyperconjugativity of 0 and 1.0). For these four entries, the carbocation carbon, the methylene carbon adjacent to the carbocation carbon, the para carbon, and the one bond coupling constant between hydrogen and the methylene carbon next to the carbocation carbon all provide measures of the hyperconjugation continuum. Moreover, plots of any one of these factors versus any other are linear with correlation coefficients between 0.95 and 1.00, indicating that they all are responding to the same structural factors. Simple calculation then led to hyperconjugativities of 0.31 for the β-silyl carbocation and 0.43 for the β-germyl carbocation. The values for the γ-silyl carbocation comprise the last entry, and these indicate a carbocationic structure as well.

These data indicate that the β-stabilized ions exist in the open, vertically (hyperconjugatively) stabilized form (1) rather than the bridged, nonvertically stabilized form (2). The γ-stabilized ion is percaudally stabilized by homohyperconjugation.



1



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Sincerely,

Joseph B. Lambert

Yan Zhao

UltraShield™ Magnets Win the Space War

Introducing the BRUKER SPECTROSPIN 600 MHz/52 mm UltraShield™ High Resolution NMR Magnet

Siting is now much easier than ever before because the space required for NMR systems has just become considerably smaller. The magnetic stray field has been significantly reduced by redesigning the coil of a standard magnet and adding a superconducting active shield. The volume enclosed by the 5 Gauss surface for an UltraShield™ magnet is ten times smaller than for a comparable standard magnet, without sacrificing any specifications.

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SPECTROSPIN is a member of the BRUKER family of companies and is located near Zurich, Switzerland. SPECTROSPIN is the world's largest manufacturer of superconducting NMR magnets. Our more than 30 years of experience in development and production of superconducting magnets enables us to deliver NMR magnets with exceptional performance and reliability. Many SPECTROSPIN superconducting magnets built in the late 70's and early 80's are still on field, providing quality data and dependable service.

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SPECIFICATIONS

MAGNET

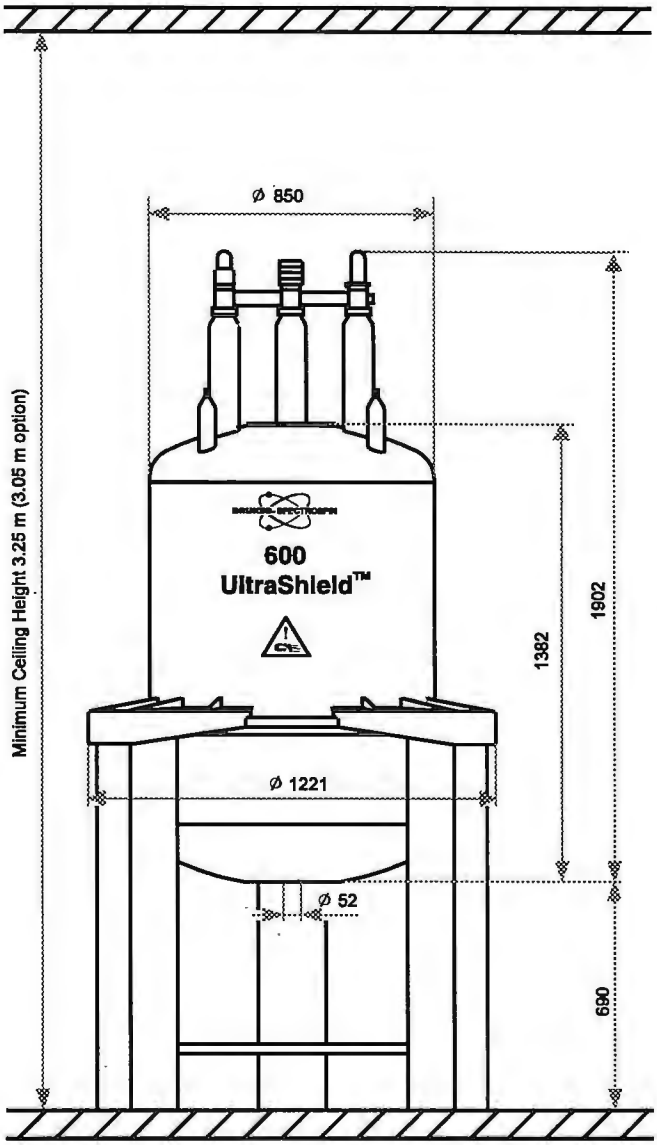
Central Field	14.1 Tesla
NMR Frequency	600 MHz
Field Drift	< 9 Hz/hr
Superconducting Shims	z, z^2, z^3, x, y, xz, yz, xy, x^2-y^2
Axial Range with Field Homogeneity better than 10 ppm (w/o RT Shimming)	~ 60 mm
5 G Line from the Magnetic Center	
-radially	< 1.8 m
-axially	< 2.5 m
Resolution at 50% 1% $CHCl_3$ 5 mm spinning	< 0.45 Hz
Lineshape 1% $CHCl_3$ 5 mm non-spinning	
at 0.55%	< 6 Hz *
at 0.11%	< 12 Hz *
Spinning Sidebands	< 1%

* Typical values obtained with the BOSS II™ shim system.

CRYOSTAT

Helium Evaporation Rate	~ 40 ml/hr
Helium Refill Volume	~ 125 liters
Helium Hold Time	> 130 days
Nitrogen Evaporation Rate	~ 400 ml/hr
Nitrogen Refill Volume	~ 163 l
Nitrogen Hold Time	> 17 days
Magnet Stand	included
Anti-Vibration Columns	included
Weight Without Cryogens	1150 kg
Weight Including Cryogens	1300 kg
Minimum Ceiling Height	3.25 m
Reduced Minimum Ceiling Height	3.05 m

600 MHz / 52 mm UltraShield™ Magnet



Dimensions in millimeters unless stated otherwise

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November 14, 1997
(received 11/17/97)

Dr. B.L. Shapiro
The NMR Newsletter
966 Elsinore Court
Palo Alto, California 94303

How Far Does the Phosphoryl Group Move in a Kinase Reaction?

Dear Dr. Shapiro,

Kinase reactions play a critical role in a variety of cellular processes. In these reactions, a phosphoryl group is reversibly transferred between ATP and a phosphorylated second substrate (X):

$$ATP + X \overset{M(II)}{\rightleftharpoons} ADP + XP$$

M(II) is an obligatory cation, Mg(II), which may be substituted by paramagnetic cations such as Mn(II) and Co(II). Relaxation effects due to the paramagnetic ions have been used to determine the distances between the cation and selected nuclei in the reaction complex. For example, measurement of ^{31}P relaxation rates in E•CoATP (or E•CoADP) complexes (E=enzyme) allows the determination of Co(II)-^{31}P distances in these complexes (G.K. Jarori *et al..*, *Biochem. 24*, 3487-3494 (1985)). Note that these experiments are performed on fully enzyme-bound complexes, but with only a fraction (<10%) of the complexes with Co(II). Exchange between diamagnetic and paramagnetic species E•ATP and E•CoATP is an unavoidable part of the measurement.

We have recently extended these measurements to enzyme bound equilibrium mixtures. These experiments are harder to perform. Besides, there is a theoretical complication. In addition to the exchange between the paramagnetic and diamagnetic species, noted above, operating on both sides of the reaction, there is an additional exchange process due to the interconversion of the substrates and products on the surface of the enzyme (E•CoATP•creatine↔E•CoADP•P-creatine). Specifically, for the ^{31}P nuclei, these exchanges are: α-P(ATP)↔α-P(ADP), β-P(ATP)↔β-P(ADP), and γ-P(ATP)↔P(P-creatine). The theoretical problem of the multiple exchange process was recently analyzed by us (B.D. Nageswara Rao,*J. Magn. Reson. B108,* 289-293 (1995)). It was shown that under the conditions prevalent in these measurements, the experiment could be analyzed in terms of two relaxation-rate constants in order to determine the endpoint distances associated with itinerant moieties.

The figure below shows a ^{31}P T_1-stack-plot (at 202 Mhz) for an enzyme-bound equilibrium mixture of creatine kinase. Experimental conditions are in the caption and the peaks are labeled. It is qualitatively clear that the P-creatine signal relaxes significantly slower than the signals from the nucleotides. The relaxation decay curves have been analyzed using the theory mentioned above. This analysis yields

$$(r(\text{P-creatine})/(r(\gamma\text{-P(ATP)})=1.46\pm0.05$$

Co(II)- γ-P(ATP) was determined previously to be about 3.0±0.2 Å. Since the cation and the two positions of the itinerant ^{31}P nucleus in γ-P(ATP) and P-creatine form a trinagle

with Co(II) at the apex, it leads to the conclusion that the phosphoryl group moves at least 1.4 Å as the reaction proceeds on the surface of the enzyme.

We are also examining equilibrium mixtures of other enzymes with this methodology. Information of this kind is of potential value in visualizing catalytic events on the enzyme surface. We do not know of any other technique that can yield information on such coherent structural movements occuring in the bound reaction complex during catalysis.

Sincerely,

B.D. Nageswara Rao Bruce D. Ray

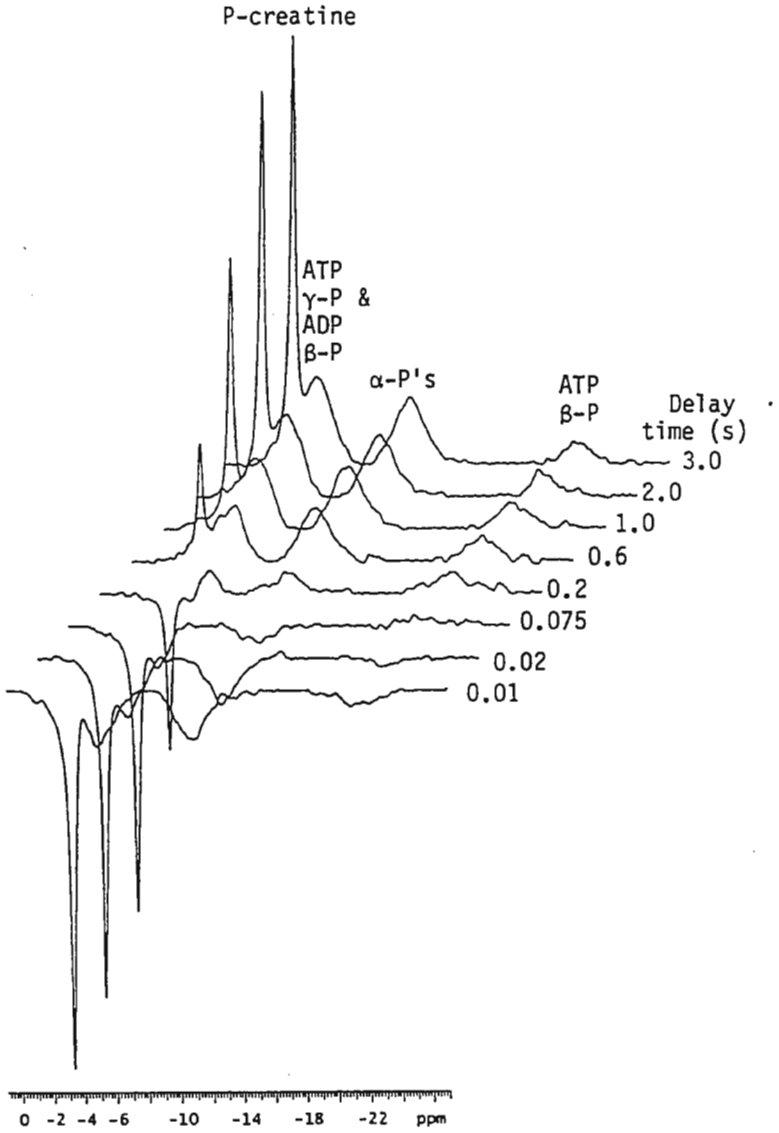


Figure: Selected traces from a typical T_1 measurement at 202 Mhz and 5° C for ^{31}P nuclei in the creatine kinase equilibrium mixture. Measurements were made for a range of Co(II) concentrations with sample conditions: 7.5 mM enzyme sites, 4.36 mM ATP, 6.45 mM creatine and 130-680 μM $CoCl_2$. NMR parameters: transients, 256; π/2 pulse width, 26 μs; sweep width, 8000 Hz; data size, 8K; line broadening, 40 Hz, and recycle delay 3 s.

NOE-Difference Experiments on Rotationally Biased Ferrier-Dimers

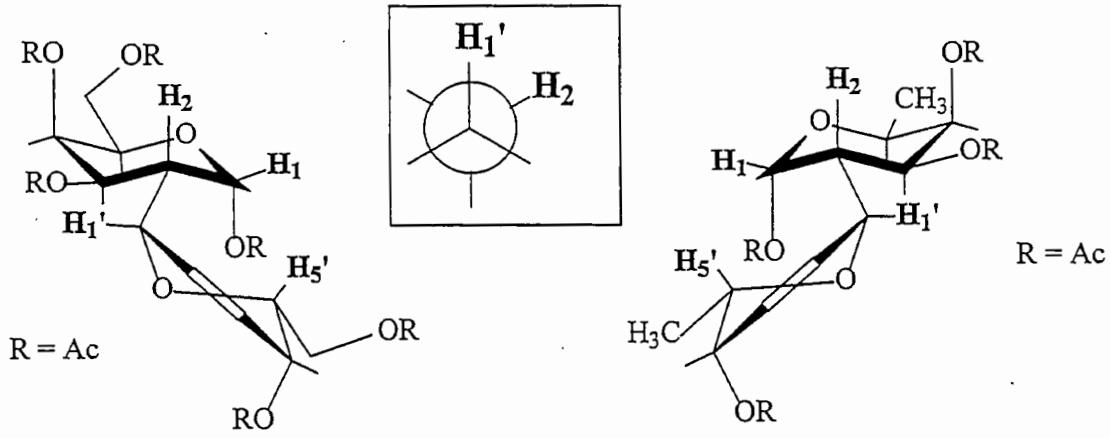
Andreas H. Franz, Paul H. Gross, Michael Minch
Chemistry Department, University of the Pacific, 3601 Pacific Avenue, Stockton, CA, 95211, USA

(received 11/4/97)

Dear Dr. Shapiro:

Rotationally biased molecules such as C-glycosidic Ferrier-Dimers are interesting systems for $^1H/^1H$-coupling experiments and NOE investigations. Rotationally biased systems show one dominant rotamer about a specific bond. In the case of the Ferrier-Dimers, the C2-C1' bond cannot rotate freely due to steric reasons. Compounds **1** and **2** were synthesized according to the literature[1] and analyzed for conformational behavior in NOE-difference experiments.

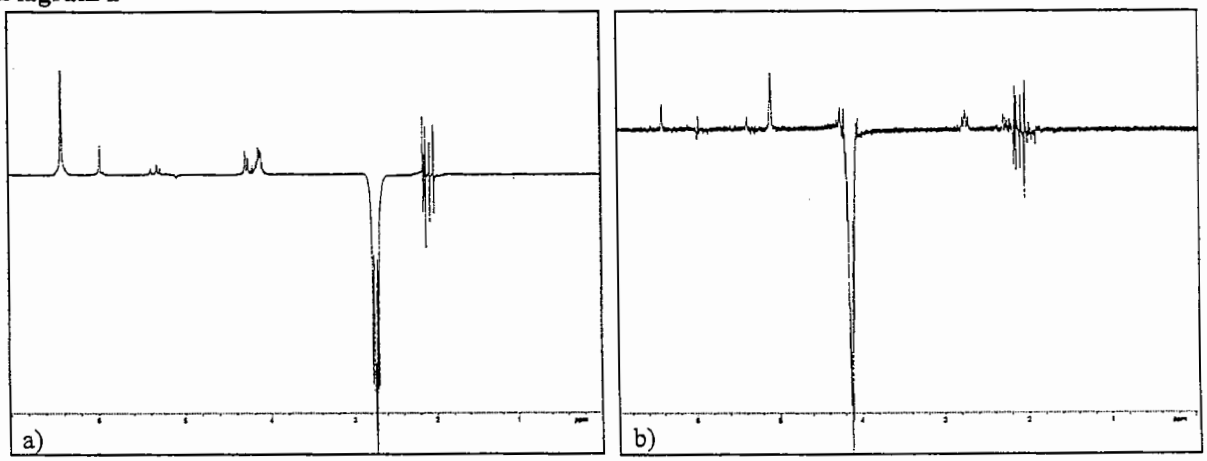
Diagram 1



1 **2**

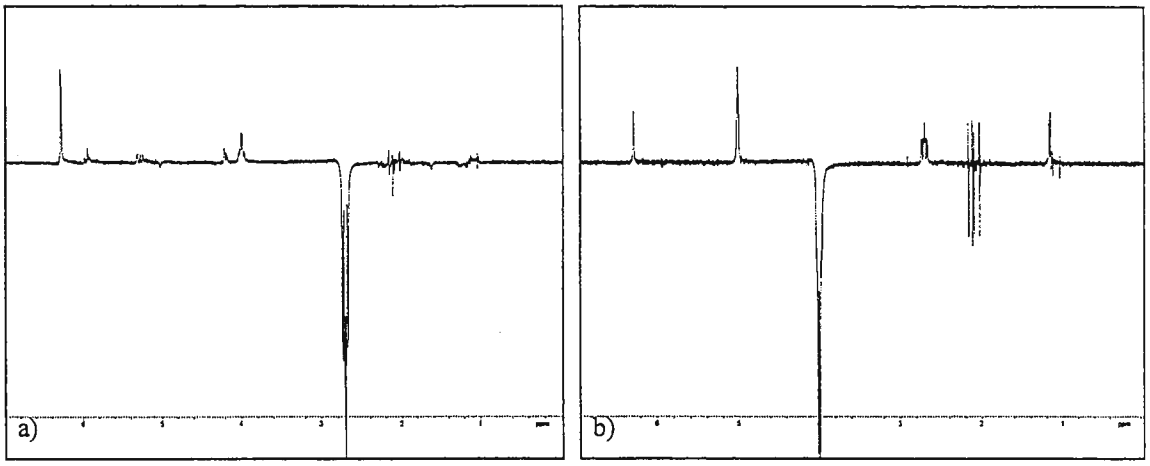
In the following discussion carbohydrate nomenclature applies and primed labels refer to atoms in the unsaturated ring. Conformational symbols are used as "D/L-enantiomers" (D-$_5H^O \rightarrow$ L-$^OH_5$) Compound **1** was irradiated at H2 (Diagram 2a).

Diagram 2



A strong response at H1 (cis, 6.39 ppm), a moderate response at H1' (4.25 ppm), at H5' (4.11 ppm) and at H2'/H3' (5.95 ppm), and a weak response at H3 (trans-diaxial, 5.27 ppm) were observed. The moderate NOE at H5' can only be explained by a $_5H^O$-conformation of the unsaturated ring (Diagram 1). H5' is in spatial vicinity of H2. As a control experiment, H5' was irradiated (Diagram 2b). NOEs were found at H1 (6.39 ppm), H4' (cis, 5.06 ppm), H2 (2.71 ppm) and $CH_2$6' (4.13 ppm). The observed NOE between H2 and H5' can be explained with a $_5H^O$-conformation of the unsaturated ring. The sterically demanding saturated sugar ring is in pseudo-axial position.
Compound **2** was irradiated at H2 (Diagram 3a). The strong response at H1 (cis, 6.26 ppm) and the weak response at H3 (trans-diaxial, 5.27 ppm) correspond to the configuration of the saturated α-*fuco*-ring. NOEs at H1' (4.17 ppm) and H5' (3.98 ppm) were observed also. In the former case a small effect was observed. In the latter a moderate effect was found.

Diagram 3



In the model (Diagram 1), the strong response at H5' can only be explained by a $^OH_5$-configuration of the unsaturated ring placing H5' into close spatial vicinity of H2. This finding calls for a pseudo-axial position of the saturated ring at C1'. The angle between H2 and H1' is greater than 90° which explains the small NOE response at H1' (4.17 ppm) when irradiating H2, and is in agreement with $J^{2,1'} = 9.0$ Hz.
H5' was irradiated subsequently (Diagram 3b). A strong NOE was found at H1 (6.26 ppm), H4' (cis, 5.00 ppm), H2 (2.69 ppm) and $CH_3$6' (1.17 ppm) as a confirmation of the above findings.

References

1. Andreas H. Franz, Paul H. Gross*, Carbohydr Lett. 1997 (in press)

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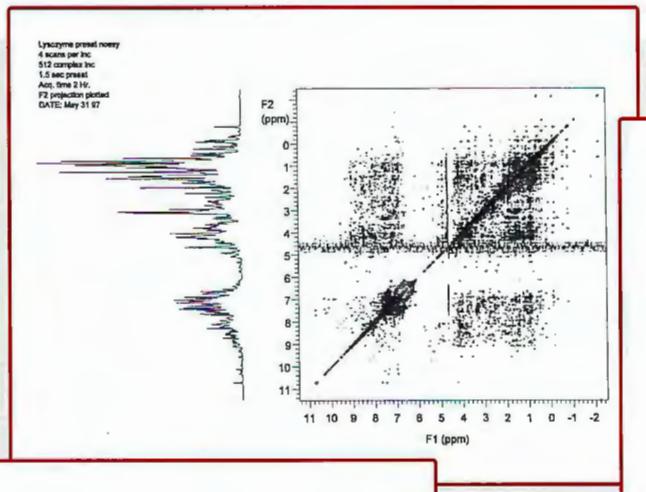
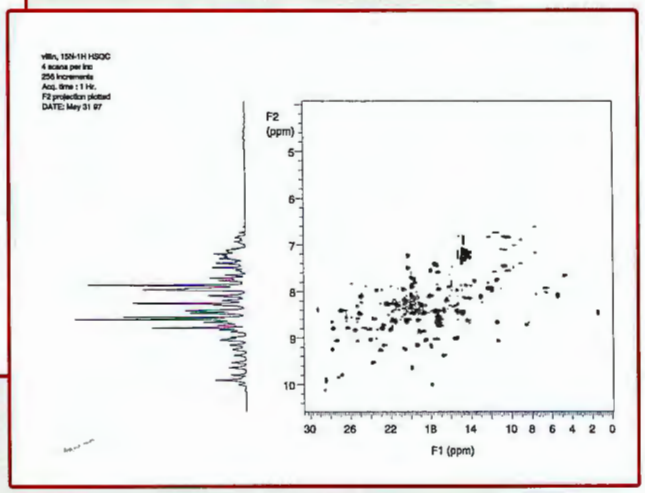
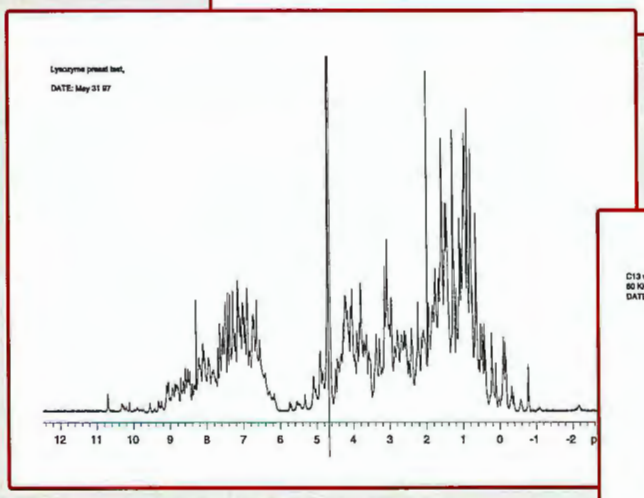
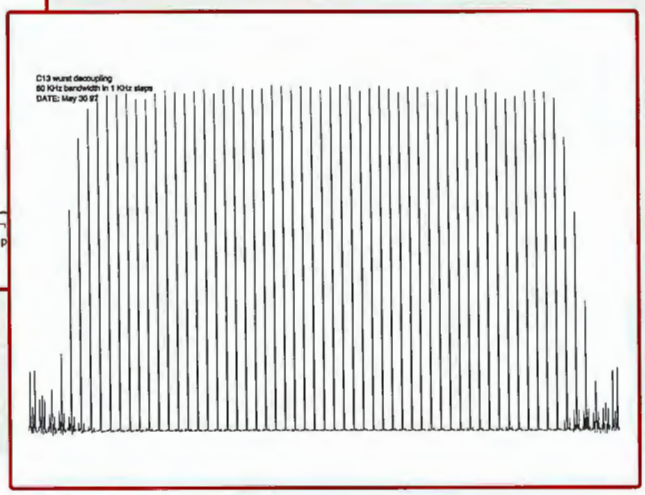


Multidimensional Heteronuclear NMR





Water Suppression Using Presaturation



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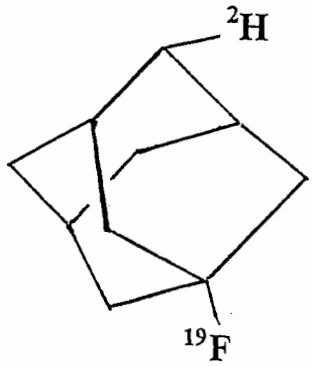
Dr. B. L. Shapiro
The NMR Newsletter
966 Elsinore Court
Palo Alto, CA 94303

Berne, november 14th 1997
(received 11/22/97)

HETERONUCLEAR ^{5}J-COUPLING

Dear Dr. Shapiro

very recently we got a mixture containing two isomeric compounds for a heteronuclear NMR study, with the aim to unequivocally identify the two isomeric forms on the basis of their spectral properties.



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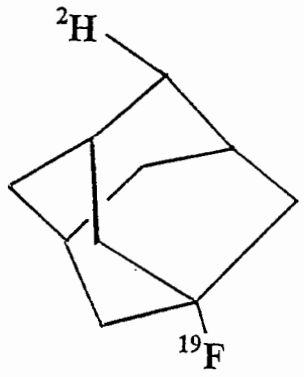
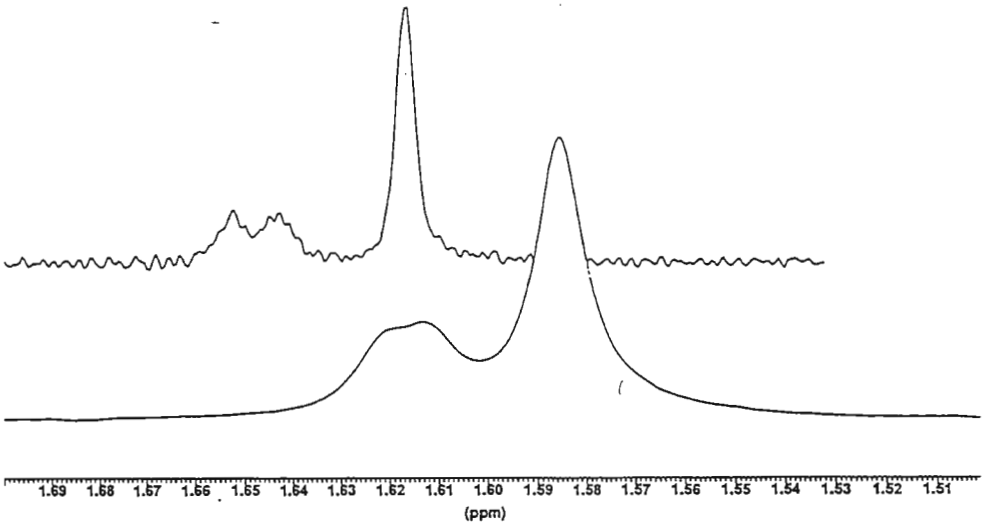


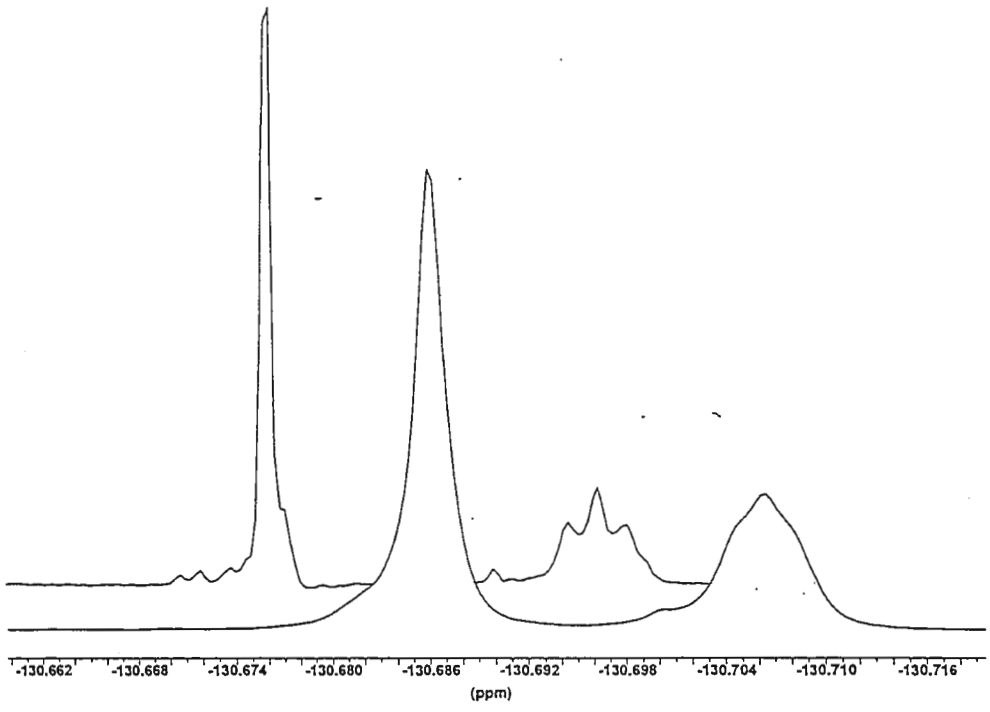
B

When analyzing the corresponding ^{2}H- and ^{19}F-spectra, both measured with ^{1}H-broadband decoupling, we not only found line broadenings originating from a heteronuclear ^{5}J coupling for one of the isomers (**B**), but found the corresponding coupling - after adequate data processing - resolved in both spectra, a doublet in the ^{2}H- and a 1:1:1 triplet in the ^{19}F-spectrum. The size (≈0.7Hz) of the corresponding coupling constant is astonishingly large in view of the five-bond coupling pathway and the gyromagnetic ratios of the coupled nuclei

^{2}H spectrum (upper trace with resolution enhancement)



^{19}F spectrum (upper trace with resolution enhancement)



Yours sincerely

Peter Bigler

The NMR Newsletter - **Book Reviews**

Book Review Editor: **Istvan Pelczer**, Chemistry Department, Princeton University, Princeton, NJ 08544

"Biological NMR Spectroscopy"

Edited by

J.L. Markley and S.J. Opella

Oxford University Press, Inc., 198 Madison Avenue, New York, New York 10016. Tel.: 212/726-6000 or 800/451-7556. 1997. ISBN 0-19-509468-9, x+360 pages. $65.00.

In March of 1994, a Symposium on Biological NMR was conducted at Stanford University to honor the 65th birthday of Professor Oleg Jardetzky, one of the great pioneers in the application of NMR to the investigation of biological problems. The Symposium was opened by Nobel Laureate William Lipscomb, Dr. Jardetzky's Ph.D. advisor. Nobel Laureate Linus Pauling, also a former mentor of Dr. Jardetzky, made his last public address before his death at this symposium. Owing to the long and outstanding contributions of Stanford University to the advancement of the science and art of NMR, the list of speakers was weighted with scientists who are or have been associated with the University. This excellent text is the result of this Symposium. The text begins with a Foreword addressed by Nobel Laureate Richard Ernst to Professor Jardetzky. The book should be required reading for all scientists and students interested in, or involved with, the application of NMR to biological problems. In addition to an excellent overview of the state of the art in the field, it contains an equally important overview, in my opinion, of the history of the field - a history which many younger scientists need to learn in order to appreciate how far we have come.

The highlights of the history of the application of NMR techniques to biological problems presented in the Epilogue of Professor Jardetzky's Chapter are in themselves noteworthy. Jardetzky reminds us of the importance to daydream and wonder about science. He challenges us to be

continued

explorers of science, not to be just exploiters of existing knowledge. Professor Jardetzky leaves us with these words of wisdom: "A society that increasingly thinks only in terms of directing and channeling craftsmanship in the pursuit of clearly visible goals is cutting itself off from the source of all innovation." His thought-provoking epilogue should be required reading for all scientists, specifically those who sit on grant review panels.

The text contains twenty three chapters divided into four sub-sections dealing with all areas of the application of NMR to the investigation of biological problems. The history of biological NMR spectroscopy is discussed in the first section. These four chapters (Jardetzky, Cohn, Shulman and Ackerman) provide an insight into the major advances and great challenges over the past 30 to 40 years in Biological NMR. For the younger scientists who are accustomed to and familiar with the application of modern NMR techniques for the determination of the structure of biomolecules, these chapters provide a much needed insight into the development of these techniques. This historical perspective provides a valuable insight into the creativity, as well as the hard work, which went into the development of these powerful techniques. The application of NMR methods for the determination of protein structure is presented in the second section of the text. The thirteen Chapters in this section discuss the application of solution and solid state NMR methods to determine protein structure, protein folding, protein specificity, ligand receptor binding and enzyme action in a clear and concise manner. The three Chapters in the third section are devoted to the study of nucleic acids. Topics discussed include determination of the structure of ribosomal RNA, characterization of DNA and determination of conformational transitions. The final section consisting of three Chapters presents a discussion of the application of Magnetic Resonance Imaging methods to the study of the brain and cancer cell metabolism.

This text is an excellent reference source for the beginning graduate student and the experienced researcher as well. The text provides an excellent mix of current research and the history of our science.

Rickey P. Hicks
Department of Chemistry
Mail Stop 9573
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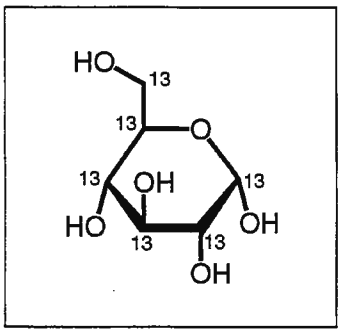

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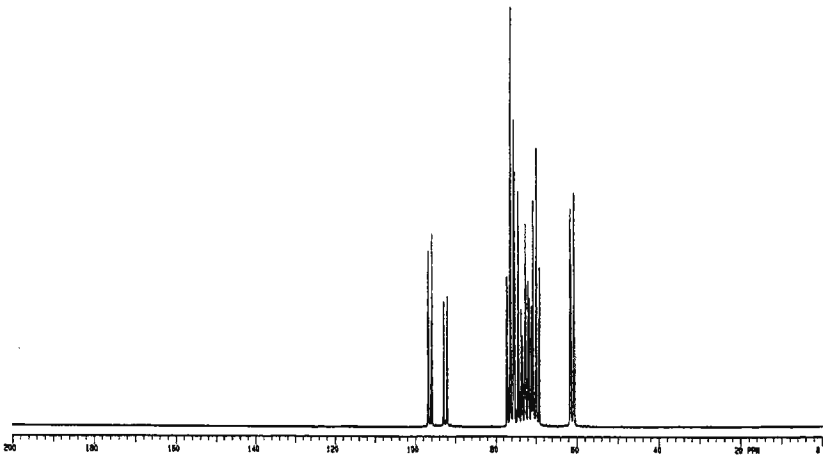

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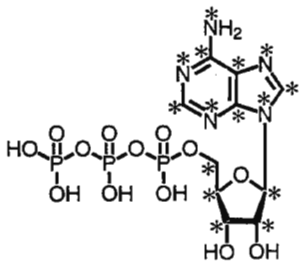

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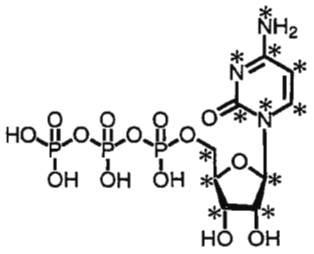

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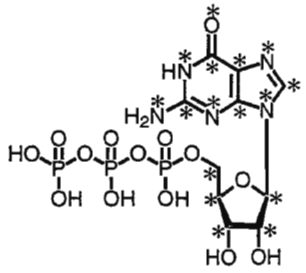

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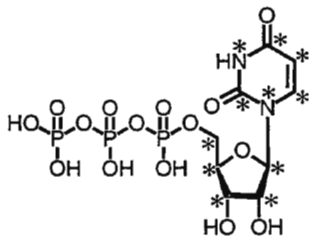

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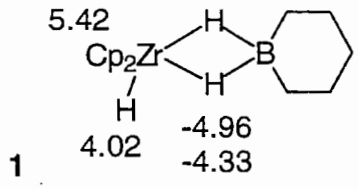
October 22, 1997 (received 10/27/97)

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

Hydride exchange
within Zirconacene
boracyloalkanehydrides

Dear Barry:

Sheldon Shore's group has made two interesting zirconacene boracycloakanehydride derivatives, **1** and **2** (toluene-d_8 solution), shown, with selected proton shifts,



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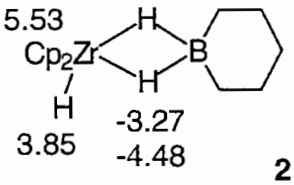

2

which exhibit in their ^{11}B decoupled proton NMR spectra lineshape changes indicative of intramolecular mutual exchange among the bridge and ZrH hydrogens. For example with increasing temperature above 0 °C the bridge and ZrH hydrogens resonances begin to broaden and by 40 °C Zr**H** appears as a broad line as does the bridge proton resonance. With increasing temperature above 40 °C the resonances for all three hydrogens broaden further and almost disappear into the baseline. Proton lineshape analysis fits best if we assign two different hydrogen exchange processes - either of the bridge hydrogens changing sites with Zr**H** and the faster exchange between the bridge hydrogens alone. For intra bridge exchange activation enthalpies (kcal/mole) and entropies (eu) are respectively for **1** 1.4 and -0.9. While for **2** the values are 8.1 and -22. In the case of bridge ZrH exchange the parameters are for **1** 20 kcal/mole and +18 eu while for **2**, 12.6 kcal/mole and -8 eu.

The ^{11}B coupled (normal) proton NMR of **1** and **2** show evidence of ^{11}B bridge proton coupling as well as ^{11}B quadrupole induced relaxation. Thus in the case of **1** between -60 °C and -40 °C the two bridge and ZrH proton resonances are essentially single lines. By 20 °C the bridge resonances have broadened to an almost trapezoidal shape indicative of ^{11}B, ^{1}H coupling (but not ZrH). Further warming results in the line averaging described above for the ^{11}B decoupled spectra. Similar effects are seen among proton NMR data for **2** except at +20 °C the bridge hydrogen resonance shows poorly

resolved splitting again indicative of ^{11}B bridge-H coupling. These results also reveal the operation of ^{11}B quadrupole relaxation, whose rate is slow enough at 20 °C to indicate ^{11}B, bridge proton coupling and which increases on cooling the sample, effectively decoupling ^{11}B from bridge protons. The rates of ^{11}B quadrupole induced relaxation and hydrogen exchange change in opposite directions with increasing temperature, thus ^{11}B bridge H coupling is only revealed within a fairly narrow temperature window.

The boron coupled proton NMR lineshapes have also been analyzed. Quadrupole relaxation is accounted for by use of the Block operator $\sum_{\alpha} \vartheta_2[\vartheta, [\vartheta^*, \vartheta]]$ assuming extreme narrowing. An approximation of the spectral densities is easily obtained from the proton decoupled ^{11}B spectra for **1** and **2**. That provides a composite relaxation parameter, r, which we apply to proton data.

$$r = \left(\frac{e^2qQ}{4I(2I - I)h}\right)^2 \tau$$

Finally one needs the ^{11}B, bridge H coupling constant, barely detectable among the NMR data for **1**. However ^{11}B NMR of **2** in diethylether-d_{10} clearly reveals this coupling to be 55 Hz. Combining the dynamic results obtained with ^{11}B decoupled proton NMR samples with the ^{11}B, H coupling constant (it works best for 60 Hz) and estimates of the ^{11}B quadrupole relaxation parameters allowed full analysis of the ^{11}B coupled NMR data. Fortunately full analysis of both "normal" and ^{11}B decoupled proton NMR data provided closely similar dynamic data for the proton exchange.

Effects due to the ^{10}B isotopomers on the ^{11}B decoupled spectra could be neglected since ^{10}B relaxes fast enough within the temperature range used to decouple ^{10}B from the bridge hydrogens.

I hope this keeps us off your pink sheet list for the time being.

With best regards,

Gideon

Gideon Fraenkel
Professor of Chemistry

Albert Chow
Research Associate

[1]The symbols have their usual meanings.

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Dr Bernard L. Shapiro
The NMR News Letter
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(received 11/3/97)

Flow-diffraction and pumped bulk flow

Dear Barry,

'Flow-diffraction' is an effect that can be manifest in plots of the ^{1}H pulsed field-gradient spin-echo (PFGSE) signal intensity, of flowing samples such as water, versus the magnitude of the field-gradient pulses employed in the experiment; the latter magnitude is proportional to that of the spatial wave number vector $\mathbf{q} = (2\pi)^{-1}\ \gamma\ \mathbf{g}\ \delta$, where γ is the nuclear magnetogyric ratio of the protons, g is the magnitude of the field gradient pulses, and δ is the pulse duration (*1, 2*). Recently we demonstrated the phenomenon of flow diffraction using a very simple experimental setup. We used a conventional peristaltic pump (Gilson) with a bubble trap and long Tygon tubing circuit of 5.5 m that dampened pulsatile flow. The Tygon tube was passed down the bore of the magnet and simply threaded through the NMR probe, down the conduit that normally houses the heater coil. Deionised water of total volume ~60 mL was circulated by the pump. The volume-flow rate of the water was measured using a preweighed flask and recording the volume delivered in a 10, 20 or 33 min period; thus, from a measurement of the Tygon tube diameter, the linear flow velocity through the NMR probe was calculated.

Figure 1 shows q-space plots for three different flow rates. The interference-type features of the plots are clearly evident. The linear flow velocity was calculated on the assumption that the signal attenuation due to the convection (coherent motion) is greatly in excess of that due to self-diffusion (incoherent motion); this is a realistic assumption given that the time over which the motion was measured was $\Delta = 40$ ms; and the signal loss due to flow out of the detection volume of the probe was negligible. From a simple theoretical analysis it can be shown that the minima occur when q has the value:

$$q_{\mathrm{min,n}} = n/(2\ a\ \Delta) \qquad (1)$$

where n is a positive integer that corresponds to the 'diffraction order'. Thus, experimentally, the linear- flow velocity, a, is measured using;

$$a = n/(2\ q_{\mathrm{min,n}}\ \Delta) \qquad (2)$$

In Fig. 1A the minimum in the curve is at $q_{\mathrm{min,1}} = 1.54 \times 10^4$ m^{-1}, and given that $\Delta = 0.04$ s this yielded an estimate of the velocity of 8.12×10^{-4} m s^{-1}; this value compared favourably with the gravimetrically-determined value of 8.34×10^{-4} m s^{-1}. In Fig. 1B the minima are at $q_{\mathrm{min,1}} = 9.36 \times 10^3$ m^{-1} and $q_{\mathrm{min,2}} = 1.96 \times 10^4$ m^{-1} which corresponded to estimates of a of, 1.34×10^{-3} m s^{-1} and 1.28×10^{-3} m s^{-1}, respectively; and these values compared favourably

with the gravimetric estimate of 1.32 x 10^{-3} m s^{-1}. Finally, the first three flow-diffraction minima in Fig. 1C (5.96 x 10^{3}, 1.19 x 10^{4} and 1.79 x 10^{4} m^{-1}) yielded estimates of a of 2.10 x 10^{-3} m s^{-1}, 2.10 x 10^{-3} m s^{-1}, and 2.09 x 10^{-3} m s^{-1}, respectively; again, these compared favourably with the gravimetric estimate of 2.15 x 10^{-3} m s^{-1}.

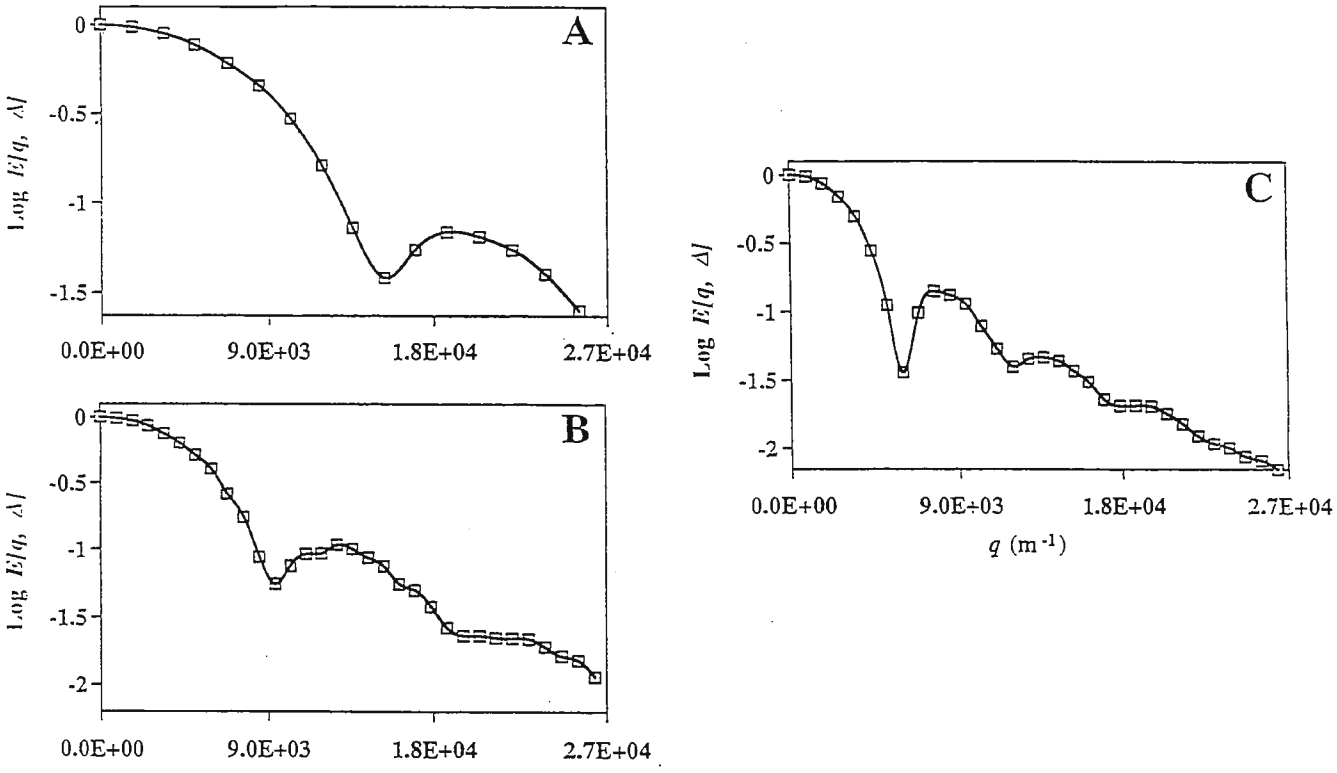

FIG. 1. 'Diffraction' evident in the q-space plots (logarithm of the signal intensity, $E[q, \Delta]$ versus q) of ^{1}H PFGSE NMR data from flowing water. The NMR spectrometer was a Bruker AMX 400 with an Oxford Instruments 9.4 T wide-bore magnet. A Bruker high-gradient diffusion probe (maximum ~ 10 T m^{-1}; but in these experiments the maximum used was *only* 0.03 T m^{-1}) with the gradient along the z-axis, and carrying a ^{1}H exchangeable r.f.-coil insert, was used. The power-supply for the gradient coils was made by Bruker, and the gradient intensity was under software control (*uxnmr*). The pulse sequence was a conventional Hahn spin-echo with 8-step phase cycle (*3*); the field gradient pulses were 2 ms in duration (d) and separated by 40 ms (D); and 16 or 32 spectra were acquired for each q-space plot, each derived from summing 16-64 transients, with an inter-transient delay of 4.0 s. The fids were transferred to a program written in MatLab, which performed the phasing of each spectrum in the series and generated the q-space plot (*3*).

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Philip Kuchel

Bob Chapman

New Products for

NMR Spectroscopy

At Aldrich, we are continually adding new NMR spectroscopy products to our inventory, as well as new unit sizes to improve the handling and storage of existing products. Some of our most recent additions are presented here.

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Acetone-d_6, 99.5 atom % D
(contains 0.03% v/v TMS, 1pkg = 10 x 1.0mL)
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Chloroform-*d*, 99.8 atom % D
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Deuterium oxide, 99.9 atom % D
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Chloroform-*d*, 99.8 atom % D (contains 0.1% v/v TMS)
43,487-6 **100g $23.30; 125g $32.50;500g $96.30; 10 x 100g $181.00; 50 x 100g $710.00**

Methyl-d_3 alcohol-*d*, 99.8 atom % D (contains 0.1% v/v TMS)
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(Methyl sulfoxide)-d_6, 99.9 atom % D (contains 0.1% v/v TMS)
43,903-7 **10g $17.35; 25g $40.70; 50g $78.15; 10 x 10g $144.50**

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Benzene-d_6, 99.95 atom % D
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Chloroform-*d*, 100.0 atom % D
45,328-5 **0.5pkg $11.70; 1pkg $19.10; 5 x 1pkg $85.00**

Deuterium oxide, 100.00 atom % D
45,335-8 **0.5pkg $26.00; 1pkg $39.75; 5 x 1pkg $159.00**

Deuterium oxide, 100.0 atom % D
45,333-1 **0.5pkg $10.10; 1pkg $15.90; 5 x 1pkg $74.25**

Dichloromethane-d_2, 99.95 atom % D
45,329-3 **0.5pkg $68.90; 1pkg $116.60**

Methyl-d_3 alcohol-*d*, 99.95 atom % D
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44,471-5 **0.5pkg $55.00; 1pkg $91.00; 5 x 1pkg $400.00**

Acetone-d_6, 99.9 atom % D
44,485-5 **0.5pkg $10.75; 1pkg $18.50; 5 x 1pkg $86.25**

Acetone-d_6, 99.8 atom % D
44,129-5 **0.5pkg $10.50; 1pkg $18.00; 5 x 1pkg $83.10**

Acetone-d_6, 99.5 atom % D
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Acetonitrile-d_3, 99.95 atom % D
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Acetonitrile-d_3, 99.6 atom % D
44,131-7 **0.5pkg $15.25; 1pkg $25.35; 5 x 1pkg $119.65**

Benzene-d_6, 99.6 atom % D
44,132-5 **0.5pkg $11.25; 1pkg $18.60; 5 x 1pkg $83.25**

Chloroform-*d*, 100.0 atom % D
44,473-1 **0.5pkg $16.00; 1pkg $28.00; 5 x 1pkg $120.00**

Chloroform-*d*, 99.8 atom % D
44,133-3 **0.5pkg $6.20; 1pkg $7.70; 5 x 1pkg $36.40**

Deuterium oxide, 100.0 atom % D
44,136-8 **0.5pkg $15.50; 1pkg $26.90; 5 x 1pkg $86.65**

Deuterium oxide, 99.9 atom % D
44,137-6 **0.5pkg $8.50; 1pkg $12.50; 5 x 1pkg $48.00**

Dichloromethane-d_2, 99.8 atom % D
44,610-6 **0.5pkg $57.70; 1pkg $96.10; 5 x 1pkg $386.25**

***N,N*-Dimethylformamide-d_7**, 99.5 atom % D
44,134-1 **0.5pkg $101.65; 1pkg $169.50**

Methyl-d_3 alcohol-*d*, 99.95 atom % D
44,475-8 **0.5pkg $66.00; 1pkg $118.50; 5 x 1pkg $534.75**

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Products for

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Techniques employed for the detection of compounds separated by HPLC have traditionally included refractive index, UV, fluorescence, IR, and mass spectrometric detection. Despite the noninvasive nature and wealth of information obtainable from NMR spectroscopy, this technique was not utilized as a detection tool until relatively recently.[1] Advances in NMR instrumentation (i.e., improved sensitivity, increased solvent peak suppression, and increased lock stability)[2] have resulted in its use in the detection of drug metabolites[3] and impurities in drug substances,[4] in studies of reaction pathways,[5] and in other applications.[6]

The usefulness of this technique would make it applicable to the detection of minute quantities of components contained in natural or synthetic mixtures such as body fluids and plant extracts, or to the determination of molecular weight distributions in polymers.

Aldrich offers deuterated and protonated HPLC grade solvents, buffers, and related products for research in this area. Additionally, our use of modern manufacturing methods allows us to manufacture large quantities of deuterated solvents economically. Talk to us about your product needs. You'll be glad you did!

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Deuterated NMR Solvents

Acetonitrile-d_3, 99.6 atom % D
15,180-7 — **5g $16.15; 10g $30.70; 25g $72.80; 50g $126.10; 10 x 10g $273.00**

Deuterium oxide, 99.9 atom % D
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Note: *The chemical purity of these products is equal to or better than that of the HPLC grade, protonated solvents. The percent purity assigned represents the deuterium content.*

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Deuterium oxide, 10 atom % D
43,578-3 — **100g $7.50**

Methyl-d_3 alcohol-*d*, 95+ atom % D
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Methyl alcohol, 99.9+%, ACS HPLC grade
27,047-4 — **100mL $14.20; 1L $18.70; 2L $28.05; 6 x 1L $92.40; 4 x 2L $84.00; 4 x 4L $128.20; 18L $110.25**

Toluene, 99.8%, HPLC grade
27,037-7 — **100mL $14.10; 1L $18.40; 2L $27.65; 6 x 1L $93.00; 4 x 2L $82.80; 4 x 4L $147.80**

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October 20, 1997
(received 10/27/97)

NMR Micrsocopy of Freezing Injuries in Plants

Dear Barry,

NMR Imaging has long been used in medical studies but has only recently begun to be seriously used in plant studies. Recently we have, with considerable success, been taking advantage of the ability of high-resolution NMR microscopy[1] to provide detailed information on the distribution of unfrozen water in plant tissues. This provides a powerful technique for non-invasively visualizing freezing behaviors (extracellular freezing, extra-organ freezing, deep supercooling) in various tissues in cold hardy woody plants. There are a number of factors that affect image intensity including temperature, however, the water to ice transition with its concomitant catastrophic decrease in water T_2 is by far the most dominant.[2] NMR microscopy non-invasively provides visual information on tissue specific and tissue-tissue interactions. So far we have studied the freezing behaviour in a number of plant species including azalea, maple and conifers.[3-6] We hope that it will help to understand the complex mechanisms and diversity involved in freezing behaviors such as: 1) identifying the barrier against ice propagation from already frozen tissues into the supercooled tissues in extra-organ freezing;[7] 2) elucidating the relationship of freezing behaviors to bud morphology and/or phylogeny;[8] 3) determining freezing behaviours in complex or interconnecting tissues which are not otherwise identifiable.[4,5] However, it is necessary to combine the results on wintering buds from other methods of analyses, both analytical and phenomenological, with the NMR microscopic images to obtain functional information.

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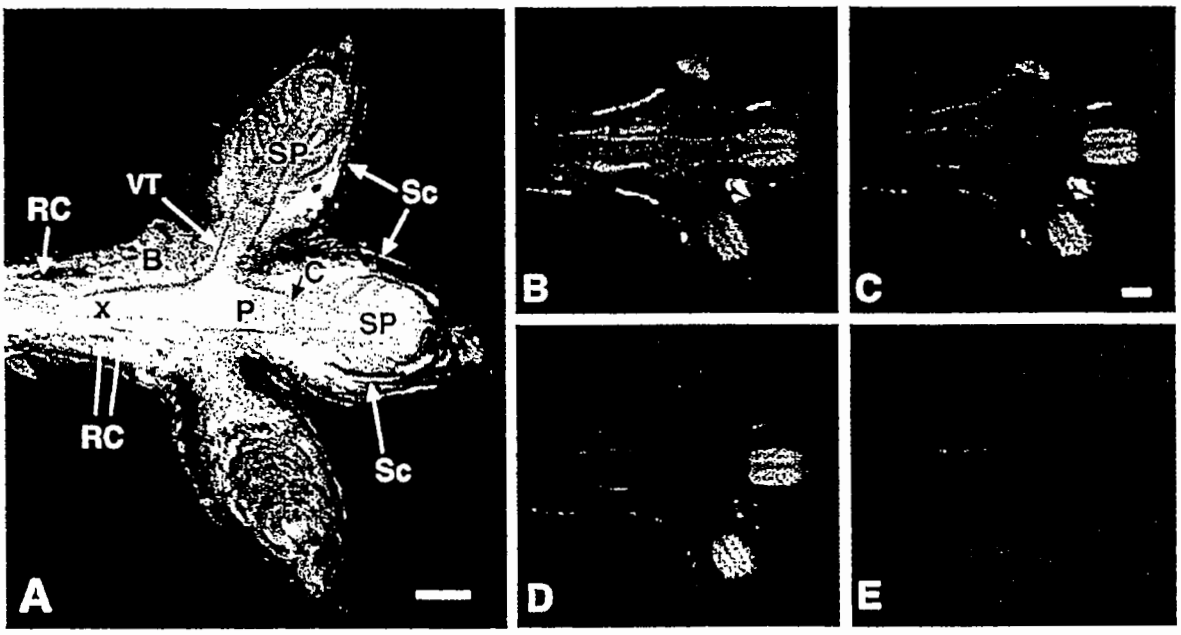

An optical image of a longitudinal section of *Abies firma* leaf. In the images the various bud tissues are indicated as follows: B, bark (periderm, cortex, phloem); C, crown tissue; P, pith; RC, resin canal (duct); Sc, leaf bud scales; SP, shoot primordium; VT, vascular tissues; X, xylem. The leaves had been removed to allow cleaner sectioning. Longitudinal NMR images taken at (A) 1 °C, (B) -7 °C, (C) -14 °C, (D) -21 °C of the same *A. firma* leaf buds collected in mid December. Imaging parameters were the same as in Fig. 3. The bars represent 1 mm. The corresponding DTA profile is displayed in Fig. 7D.

Yours sincerely

W.S. Price 井出博之 石川雅也 荒田洋治

William S. Price Hiroyuki Ide Masaya Ishikawa Yoji Arata

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November 3, 1997 (received 11/7/97)

A New Affiliation, a nontechnical contribution.

Dear Dr. Shapiro,

We introduce New Mexico Resonance, a nonprofit research company specializing in magnetic resonance. It was incorporated in June and became an operational entity on October 1 with Steve Altobelli, Arvind Caprihan, Joe Seymour, and Eiichi Fukushima leaving The Lovelace Institutes to join Kendra Vitek, our Administrative Officer, who had set up the company's infrastructure. We are at the same address as before, renting offices from Lovelace and sharing spaces and equipment with Dean Kuethe who remains with Lovelace.

Our focus remains on NMR studies of flowing systems although thoughts are being given to less orthodox NMR such as STRAFI and earth's field NMR. In fact, Joe has gone south for the second phase of Massey University's earth's field NMR project to study saline pore structures in Antarctic sea ice.

We wish to maintain contact with our colleagues and friends as well as make new acquaintances. You are welcome to visit us when you are in or near Albuquerque. Our email addresses are X@nmr.org where X is salto, arvind, jseymour, kendra, and eiichi for Steve, Arvind, Joe, Kendra, and Eiichi, respectively. Our URL is http://www.nmr.org.

The 4th Int'l Conference on Magnetic Resonance Microscopy and Macroscopy was our swan song at Lovelace. It was very successful, we think, and have listed its interesting program on our webpage whose address was mentioned above.

Sincerely yours,

Eiichi Fukushima

POSITION AVAILABLE

NMR Operator/Researcher with the NMR Facility at Clark University. Duties involve responsibility for maintaining instrumental operation of the Facility. Modern instrumentation including solid state (Bruker MSL-300 WB) and high field liquid (Varian 3-channel Unity 500) is available. Opportunities for collaborative research in establishing programs in solid state NMR of polymers and/or high resolution NMR of biomolecular structure are available. The position requires a PhD with expertise in NMR operation, maintenance and good computer skills. The instrumentation is computer interfaced with a series of workstations for both host control and off-line processing. The position involves system management for the computers. Clark University is an EEO/AA Employer. Resume to: Paul T. Inglefield, Chemistry Department, Clark University, Worcester, MA 01610-1477

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Professor of Molecular Pharmacology

Tel: 415/723-6153
Fax: 415/723-2253
New Area Code is 650
New Zip Code is 94305-5337

POSTDOCTORAL POSITION

Postdoctoral Fellowship available immediately to work on the physical mechanisms of allosteric control in proteins. Good programming skills and familiarity with Molecular Dynamics, Monte Carlo methods and NMR relaxation theory are essential. To apply send c.v. to Prof. Oleg Jardetzky by fax at 650/723-2253 or as an email attachment to jardetzky@stanford.edu.

POSTDOCTORAL POSITIONS AVAILABLE

Post-doctoral positions are available for NMR studies of structure and dynamic behavior of main group organometallic compounds and related carbanionic species. Applicants should be skilled at organometallic synthesis and NMR technology. Arrange for three letters of recommendation and send a CV.

Gideon Fraenkel
Chemistry Department
Ohio State University
100 West 18th Avenue
Columbus, OH 43210

THE NMR NEWSLETTER

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Various

1. On page 19, you will notice that the Newsletter has a new Book Review (and Software Review) Editor, **Dr. Istvan Pelczer** of the Princeton University Chemistry Department. Istvan succeeds **Prof. William B. Smith** of Texas Christian University, who has served as Book Review Editor for many years, writing a very substantial number of reviews himself and inducing his local and other colleagues to provide additional reviews. Bill asked to step aside from this time-consuming chore, and I reluctantly agreed. I believe he did a superb job and I thank him for it. Working with him has been (almost always!) a delight, and rewarding even when he saw fit to disagree with me. I trust we will continue to read his words of wisdom from time to time.

I am sure that Istvan will appreciate volunteers to write reviews. Reviewers should contact him directly at the address on page 19 of this issue, or by email to ipelczer@phoenix.princeton.edu. Reviewers will (most often) get a copy of the book or software item, and may also have one quantum of non-financial subscription credit for the Newsletter.

2. Note the number of 'Positions Available' in this, and indeed in most of the recent issues of the Newsletter. Yet another sign of the health and vigor of the field! Readers are invited to make use of this service, as well as analogous 'Position Wanted', 'Equipment Available' (or Wanted) notices in the Newsletter. There is no charge for subscribers, sponsors or advertisers, and only a nominal fee for others.

3. It would be useful to Newsletter readers (and to me) if you would include your e-mail address in any written communications to The NMR Newsletter, or send this address to me separately by e-mail.

BLS

THE NMR NEWSLETTER

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Policies and Practical Considerations

(Slightly revised October 1997)

The NMR Newsletter (formerly the **TAMU NMR Newsletter**, the **IIT NMR Newsletter**, and originally, the **Mellon Institute NMR Newsletter**), now in its fortieth year of consecutive monthly publication, continues under the same general policies as in the past.

1. **Policy**:

The 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 serves its purpose best if the participants impart whatever they feel will interest their colleagues, and inquire about whatever matters concern them. Technical contributions should always contain a significant amount of information that has not already been published or that will appear in the formal literature within a few weeks of the appearance in the Newsletter.

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 is followed by the reservation, "that won't land us in jail or bankruptcy court.") Virtually no editorial functions are performed, although on rare occasions there is the need to classify a contribution as 'not for credit'. The Newsletter is not, and will not become, a journal. We merely reproduce and disseminate exactly what is submitted.

2. **Public Quotation and Referencing:**

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

3. **Participation is the prime requisite for receiving the Newsletter: In order to receive the Newsletter, you must make at least occasional technical contributions to its contents.**

We feel that we have to be quite rigorous in this regard, and the following schedule is in effect: Seven months after your last technical contribution, you will receive a "Reminder" notice. If no technical contribution is then forthcoming, nine 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, only one contributor may be credited. 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.

4. **Finances**: The Newsletter is wholly self-supporting, and its funding depends on Advertising, Sponsorships, and individual Subscriptions. The **Subscription fee** for the October 1997 - September 1998 year is US$190, with a 50% academic or personal subscription discount. Subscriptions are available for a minimum of the twelve monthly issues which end with a September issue. However, a subscription can be initiated at any time, with the price for more than twelve issues being prorated.

continued

Corporations are also invited to join the list of **Sponsors** of the Newsletter. Sponsors' names appear in each month's Newsletter, and copies of the Newsletter are provided to all Sponsors. The continuation of the Newsletter depends significantly on the generosity of our Sponsors, most of whom have been loyal supporters of this publication for many years. We will provide further details to anyone interested.

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

5. **Practical Considerations**:

a) All technical contributions to the Newsletter will be included in the next issue if received on or before the published deadline dates.

b) Please provide short titles of all topics of your contributions, to ensure accuracy in the Table of Contents.

c) Contributions should be on 8.5 x 11" (21 x 27.5 cm) pages, printed on one side only. Contributions may not exceed three pages without prior approval. Each page must have margins of at least 0.5" (1.3cm) on all four edges. Black ink for typing, drawings, etc., is essential. All drawings, figures, etc., should be mounted *in place* on the 8.5 x 11" pages. We are not equipped to handle pieces of paper larger than 8.5 x 11" (21 x 27.5 cm).

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.*

When formatting your contributions, please consider the following:

i) 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 11 or 12 point type is acceptable if the particular font is not too large. 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. Type smaller than 8 point should not be used.

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. 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.).

iv) AVOID DOUBLE SPACING LIKE THE BLACK PLAGUE ! ! ! This is extremely wasteful of space.

6. **Suggestions**: They are always welcome.

B. L. Shapiro
October 1997

*Telephone: 650-493-5971. Please confine telephone calls to 8:00AM-10:00PM, *Pacific Coast Time.*
*Fax: 650-493-1348 (Do not use for technical contributions which are to appear in the Newsletter, for Fax quality is not adequate.)
*E-mail: shapiro@nmrnewsletter.com
*http://www.nmrnewsletter.com

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650-493-5971* - *Please call only between 8:00 am and 10:00 pm, Pacific Coast time.*

Deadline Dates

No. 472 (Jan.)	19 Dec. 1997
No. 473 (Feb.)	23 Jan. 1998
No. 474 (Mar.)	27 Feb. 1998
No. 475 (Apr.)	27 Mar. 1998
No. 476 (May)	24 Apr. 1998

* Fax: 650-493-1348, at any hour. Do not use fax for technical contributions to the Newsletter, for the received fax quality is very inadequate.

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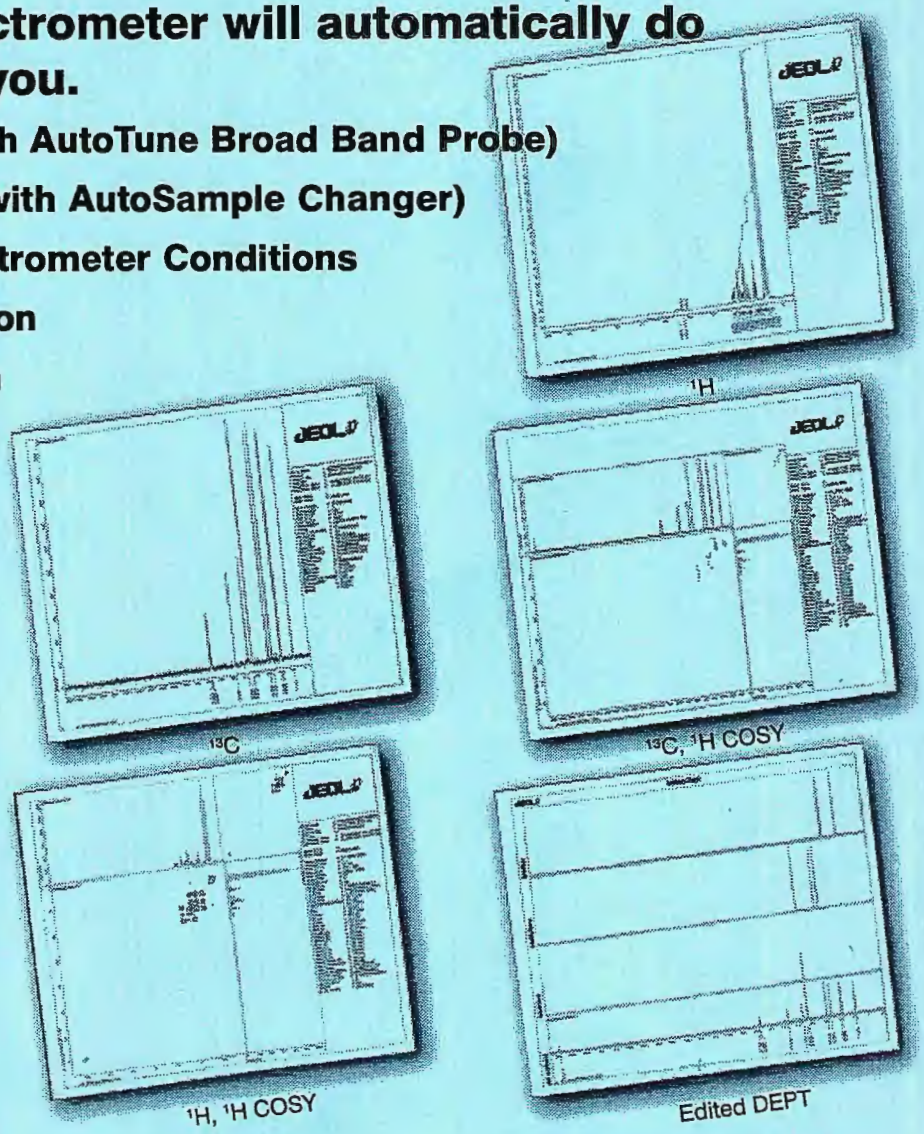

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