THE
NMR
NEWSLETTER

No. 476

May 1998

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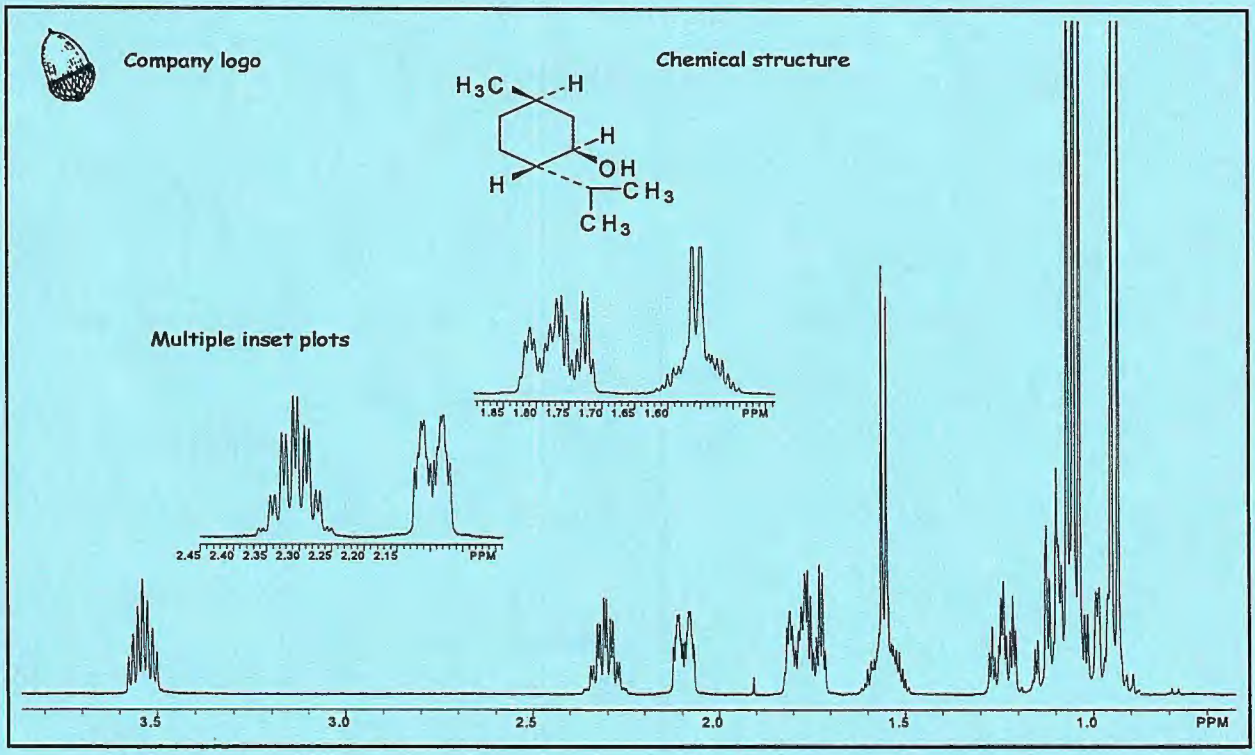

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FORTHCOMING NMR MEETINGS

^{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.

New Mexico Regional NMR Meeting, Las Cruces, NM, **May 9, 1998**; Contact: Y. Shachar-Hill, Dept. of Chem. and Biochem., New Mexico State Univ., Las Cruces, NM 88003; Tel. 505-646-3218; Fax. 505-646-2649; Email: yairhill@nmsu.edu. For details, see Newsletter 475, 54.

14th European Experimental NMR Conference, Bled, Slovenia, **May 10-15, 1998**. Contact: The Secretariat of 14th EENC, Dept. of Physics, University of Ljubljana, Jadranska 19, 1000 Ljubljana, Slovenia; Phone: +386-61-1766500; Fax: +386-61-217-281; E-mail: eenc98@fiz.uni-lj.si; http://www.fiz.uni-lj.si/~stipe/eenc98/eenc98.html.

Workshop on Magnetic Resonance of Connective Tissues and Biomaterials, Philadelphia, PA, **June 18-20, 1998**; For more information. Contact International Society for Magnetic Resonance in Medicine, 2118 Milvia Street, Suite 201, Berkeley, CA 94704; (510) 841-1899; fax (510) 841-2340; info@ismrm.org; http://www.ismrm.org.

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.

XVIIIth International Conference on Magnetic Resonance in Biological Systems, Tokyo Metropolitan University, **August 23 - 28, 1998**. Contact: Professor Masatsune Kainosho, Department of Chemistry, Tokyo Metropolitan University; +81-426-77-2544; Fax: +81-426-77-2525; e-mail: kainosho@raphael.chem.metro-u.ac.jp; http://icmrbs98.chem.metro-u.ac.jp

Continued on p. 14

ROSKILDE UNIVERSITY

Professor Poul Erik Hansen, Department of Life Sciences and Chemistry

e-mail poulerik@virgil.ruc.dk; FAX +4546743011 P.O.Box 260, DK-4000 Roskilde, Denmark

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

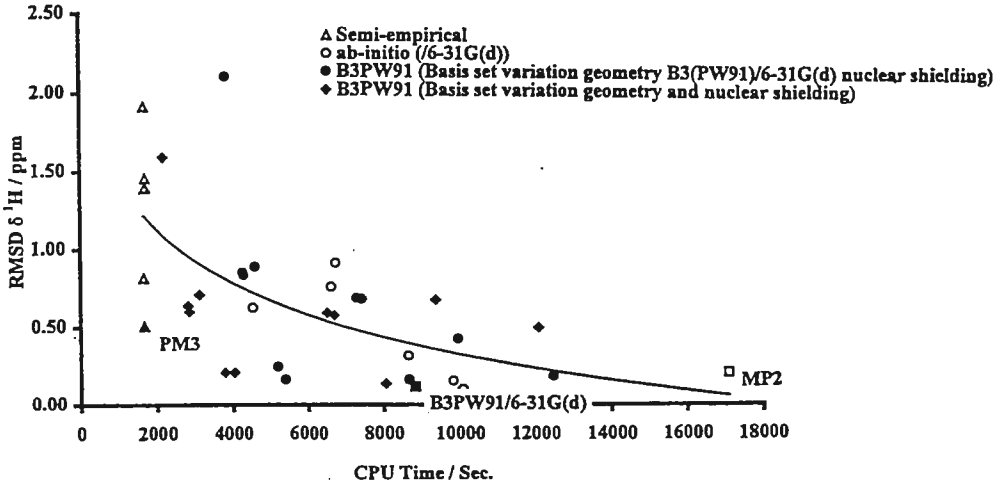
DATE OUR REFERENCE YOUR REFERENCE

March 11 1998 (received 3/31/98)

"Ab Initio calculations of nuclear shieldings and isotope effects"

Dear Professor Shapiro

Ab initio calculations of chemical shifts (nuclear shildings) have become very reliable using e.g. the Gaussian94 suite of programs. Followingly, calculations of isotope effects can also be done.[1,2] Jens Abildgaard has investigated the accuracy and time spent using a number of semiempirical and ab initio methods using salicylaldehyde as an example. It is revealed (see also figure) that the density functional method (DFT) B3PW91/6-31(d) yields very good geometries, vibrational frequencies and ^{1}H (figure) and ^{13}C nuclear shieldings.



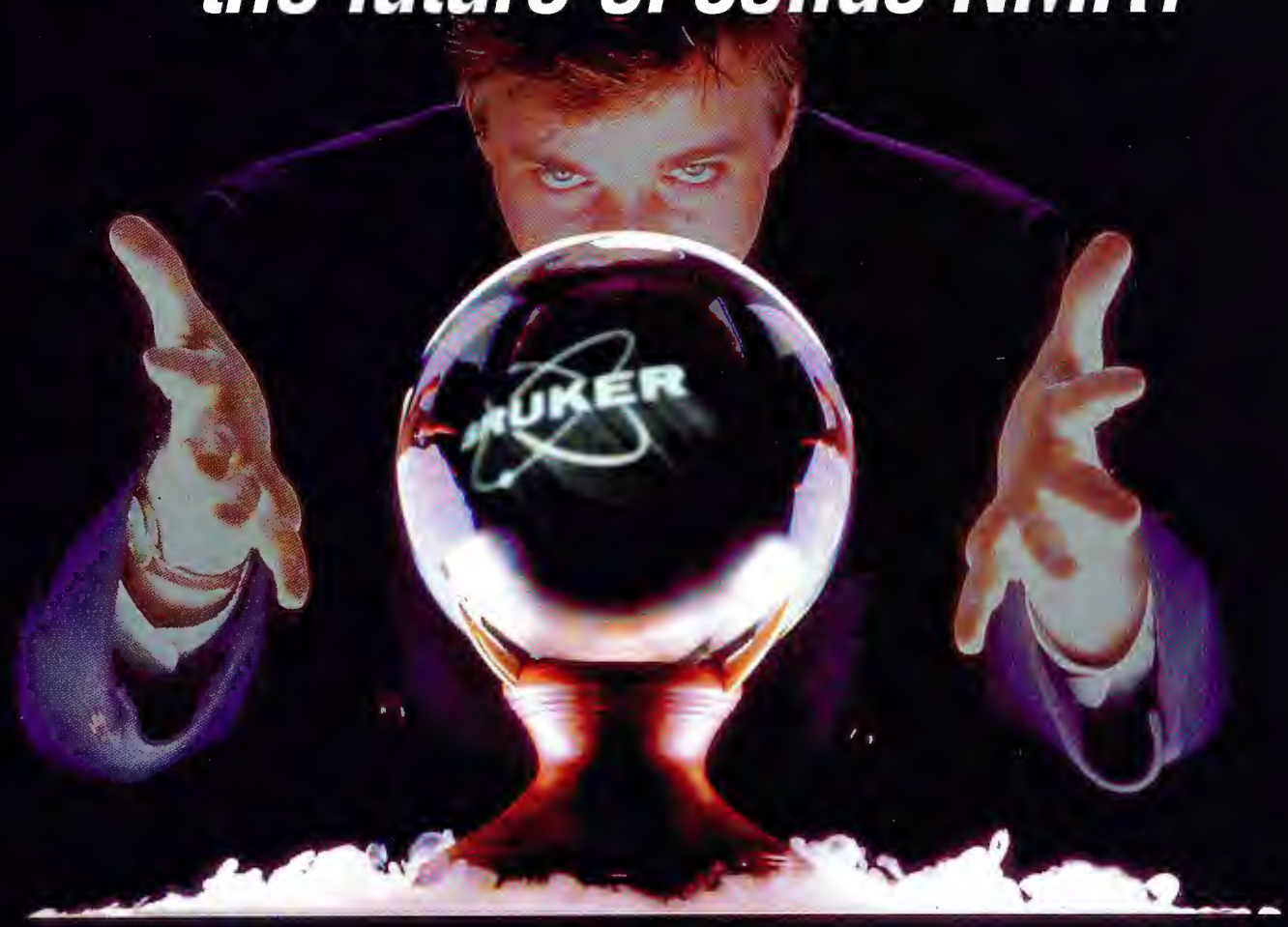
Even fast PC´s may be used for this type of molecules. The full story is available in english from the authors.[3]

Yours sincerely

Poul Erik Hansen

[1] M.Munch, Aa.E.Hansen, P.E.Hansen, and T.D.Bouman, Acta Chem.Scand. 46 (1992) 1065.
[2] P.E.Hansen, Isotope Effects on Chemical Shifts as Tools in Structural Studies. Roskilde University Press (1997).
[3] J. Abildgaard and P.E.Hansen, Quantum Mechanical Model Calculations in Chemistry: Structure, Vibrations, Chemical Shifts and Isotope Effects on Chemical Shifts. Papers from Institute of Life Sciences and Chemistry, RUC, 22, 1998.

Now what's happening to the future of solids NMR?



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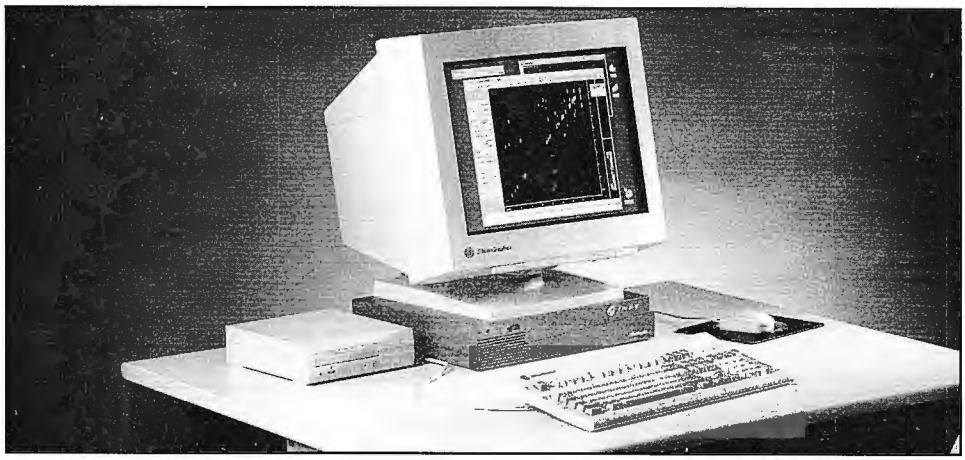

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Aksel A Bothner-By
6317 Darlington Rd.
Pittsburgh, PA 15217

CAN A STRONG MAGNETIC
FIELD DISTORT THE MOLECULE?

11 April 1998 (received 4/14/98)

Dear Barry,

When we first got into this game of orienting molecules in the magnetic field, I would be asked fairly often whether the forces orienting the molecule might not be strong enough to actually alter the structure. I didn't think very much about it, and answered that I thought the forces to be so small that they couldn't produce any significant change. Of course if the field were gigantic enough, they would eventually produce some effect, and if the molecule were flexible enough (I've always thought that proteins for example were pretty squishy), something observable might happen.

Since fields are in fact getting stronger all the time, I thought it would be worthwhile to try to predict more quantitatively what to expect. Herewith my first attempts in this direction, carried out on the molecule diphenyl, in which the two benzene rings can rotate with respect to each other about the single bond.

I assume a rotational barrier, V_O, the coplanar state being the most stable. The equilibrium between the coplanar and perpendicular forms, $p_{\parallel}/p_{\perp}=f_r$, which is equal to $e^{-V_o/kT}$. The effect of the magnetic field will be to try and orient each of the rings with its plane aligned with B_O. The energy difference, $V_m = H^2\Delta\chi/2$, and $f_m = e^{-V_m/kT}$. Then one can calculate the relative populations of a set of 12 extreme states, as follows:

config.	B_O	B_O	B_O
	$f_r f_m^2$	$f_r f_m^2$	f_r
	f_m^2	f_m	f_m
	f_m^2	f_m	f_m
	$f_r f_m^2$	f_r	$f_r f_m^2$

Then one can do a statistical-mechanical-like thing and get the partition function, calculate $p_{\parallel}/p_{\perp}$, etc. etc. I get

$$p_{\parallel}/p_{\perp} = f_r(2f_m^2+1)/(f_m^2+2f_m)$$

For a benzene ring in a 600 MHz spectrometer, f_m =0.999976 -- and putting that into this formula, gives

$$p_{\parallel}/p_{\perp} = f_r \times 1.000000001$$

hardly a noticeable effect. f_m goes up nearly as the square of the field, and it looks like one would have to work at about ten million megahertz, to be able to see an effect.

Finally, it's amusing to note that if the field gets large enough so that only the terms containing f_m^2 in the table are significant,

$$p_{\parallel}/p_{\perp} = 2f_r$$

Looks to me like we really don't have to worry about this.

Sincerely,

The NMR Newsletter - Book Reviews

Book Review Editor: István Pelczer, Dept. of Chemistry, Princeton University, Princeton, NJ 08544

"A Complete Introduction to Modern NMR Spectroscopy"

by

Roger S. Macomber

John Wiley & Sons, Inc., 605 Third Ave., New York, NY 10158-0012; 1988; 212-850-6011; 212-850-6008 fax; ISBN 0-471-15736-8, 382 pages, $54.95 (softback).

In recent years there have been a number of smallish, soft cover, Introductory texts for teachers of NMR spectroscopy. Each of these have strong and weak points determined, in part, by the constraints of length and the author's concepts of what such texts require. My personal experience has been that many of these are usable though none has completely covered everything my own prejudices wanted in subject matter. This text by Macomber may well eliminate that point of criticism.

The scope of the text may be suggested by the list of chapter titles: 1. Spectroscopy: Some Preliminary Considerations; 2. Magnetic Properties of Nuclei; 3. Obtaining an NMR Spectrum; 4. A Little Bit of Symmetry; 5. The ^{1}H and ^{13}C NMR Spectra of Toluene; 6. Correlating Proton Chemical Shifts with Molecular Structure; 7. Chemical Shift Correlations for ^{13}C and Other Elements; 8. First-Order (Weak) Spin-Spin Coupling; 9. Factors That Influence the Sign and Magnitude of J: Second-Order (Strong) Coupling Effects; 10. The Study of Dynamic Processes by NMR; 11. Electron Paramagnetic Resonance Spectroscopy and Chemically Induced Dynamic Nuclear Polarization; 12. Double-Resonance Techniques and Complex Pulse Sequences; 13. Two-Dimensional Nuclear Magnetic Resonance; 14. NMR Studies of Biologically Important Molecules; 15. Solid-State NMR Spectroscopy; 16. NMR in Medicine and Biology: NMR Imaging.

Each chapter has worked examples, a chapter summary, and references. Some chapters finish with a set of problems, with answers given in an Appendix at the end of the text. There are indices for both subjects and chemical compounds, a useful addition.

I found the portions I read in detail to be well written. Given the price and number of pages, I warrant this text to be the nearest I am aware of to fulfilling the title promise of *complete*.

William B. Smith
Department of Chemistry
Texas Christian University

Family Matters

From left to right:

Don McReynolds
Earthy Lee Young
Remonda Lavinghouse
James Brewer
Gary Skidmore

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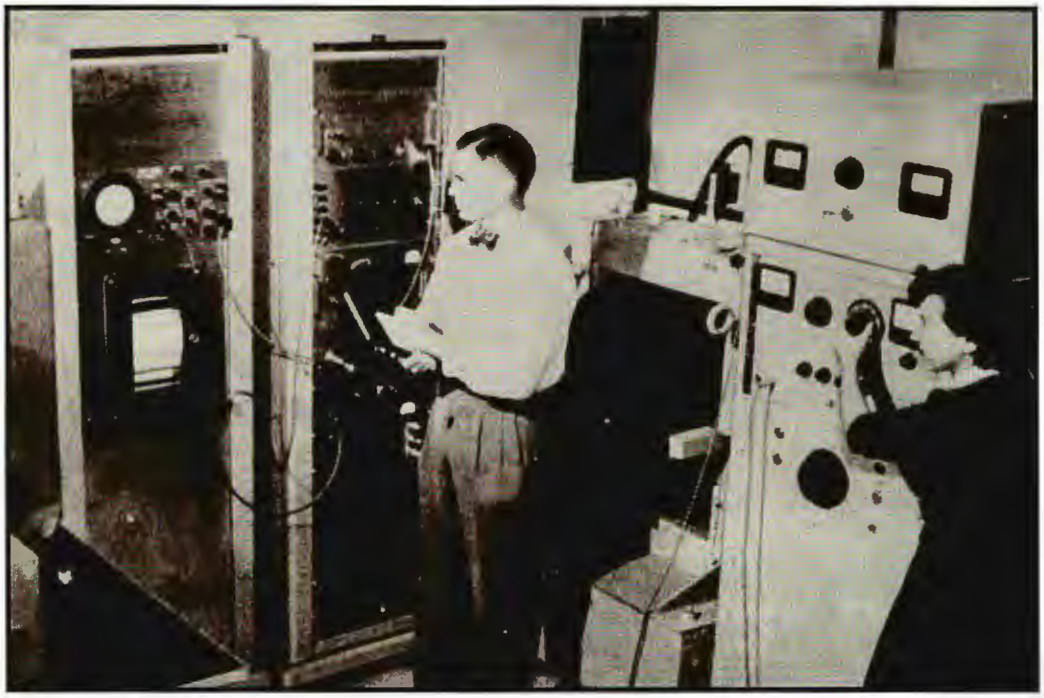

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Dr. B.L. Shapiro
The NMR Newsletter
966 Elsinore Court
Palo Alto, CA 94303

April 13, 1998
(received 4/20/98)

Molecular Diffusion Measured Using ^{31}P NMR

Dear Barry:

It was delightful to hear about the history of the NMR Newsletter at the Varian User's Conference prior to the ENC. Lately, we have been utilizing NMR diffusion measurements to characterize molecular association. The important role of phosphorus in biology makes it desirable to utilize ^{31}P NMR in conjunction with, or as an alternative to, ^{1}H NMR to determine the diffusion

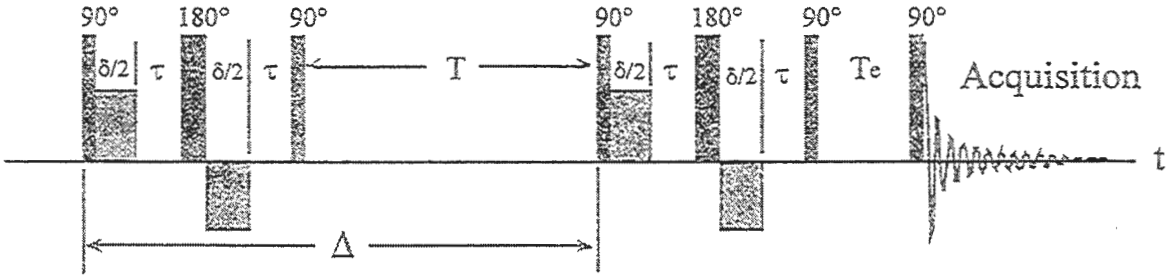

Figure 1 - Pulse sequence for the bipolar pulse pair (BPP) LED.

characteristics of samples containing phosphorus. The experimental setup for ^{31}P NMR diffusion measurements is similar to that of ^{1}H although (as noted below) the smaller γ for ^{31}P requires altering gradient strengths and magnetization evolution times to acquire data with similar signal attenuation due to diffusion.

To demonstrate the utility of ^{31}P NMR to measure diffusion coefficients, we measured the translational diffusion coefficient for the SV40 Okazaki fragment using a Varian Unity500 spectrometer. The bipolar pulse pair (BPP) LED experiment[1] (Figure 1) was used for both ^{31}P and ^{1}H (202.29 MHz and 499.73 MHz respectively) experiments. The amplitude of the observed PFG NMR signal can be expressed as:

$$A=A_o \exp[-\gamma^2 g^2 \delta^2 D(\Delta - \delta/3 - \tau/2)]. \quad [1]$$

The gradient strength, g, was calibrated by fitting the residual ^{1}H water signal at 25 °C to Equation 1, and back-calculating g, assuming the diffusion rate for HDO is 1.9 x 10^{-5} cm^2/sec.[2] In the phosphorus experiments, due to the smaller gyromagnetic ratio of ^{31}P, it was necessary to use

stronger gradient fields, g, (3-13 Gauss/cm for ^{1}H and 3-20 Gauss/cm for ^{31}P) as well as longer evolution times, T. For the ^{1}H experiment an evolution time of 0.1 seconds was used, while for ^{31}P, 0.5 seconds was used.

Using these parameters, the experimental data (shown in Figure 2) were fit to equation 1 resulting in diffusion coefficients, D, of 1.77×10^{-5} cm^2/sec for the ^{1}H experiment and 1.79×10^{-5} cm^2/sec for the ^{31}P experiment. The difference between the experimental results for the two nuclei are well within experimental error. While measurement of the diffusion coefficient using ^{31}P is more time consuming, due to its longer T_1, it may be advantageous, when available, to use ^{31}P to confirm that diffusion rates measured using ^{1}H are not affected by hydrogen exchange processes.

1. D. Wu, A. Chen, and C. Johnson, Jr., *J. Magn. Reson. Series A*, **115**, 260 (1995).
2. L. G. Longsworth, *J. Phys. Chem.*, **64**, 1914 (1960).

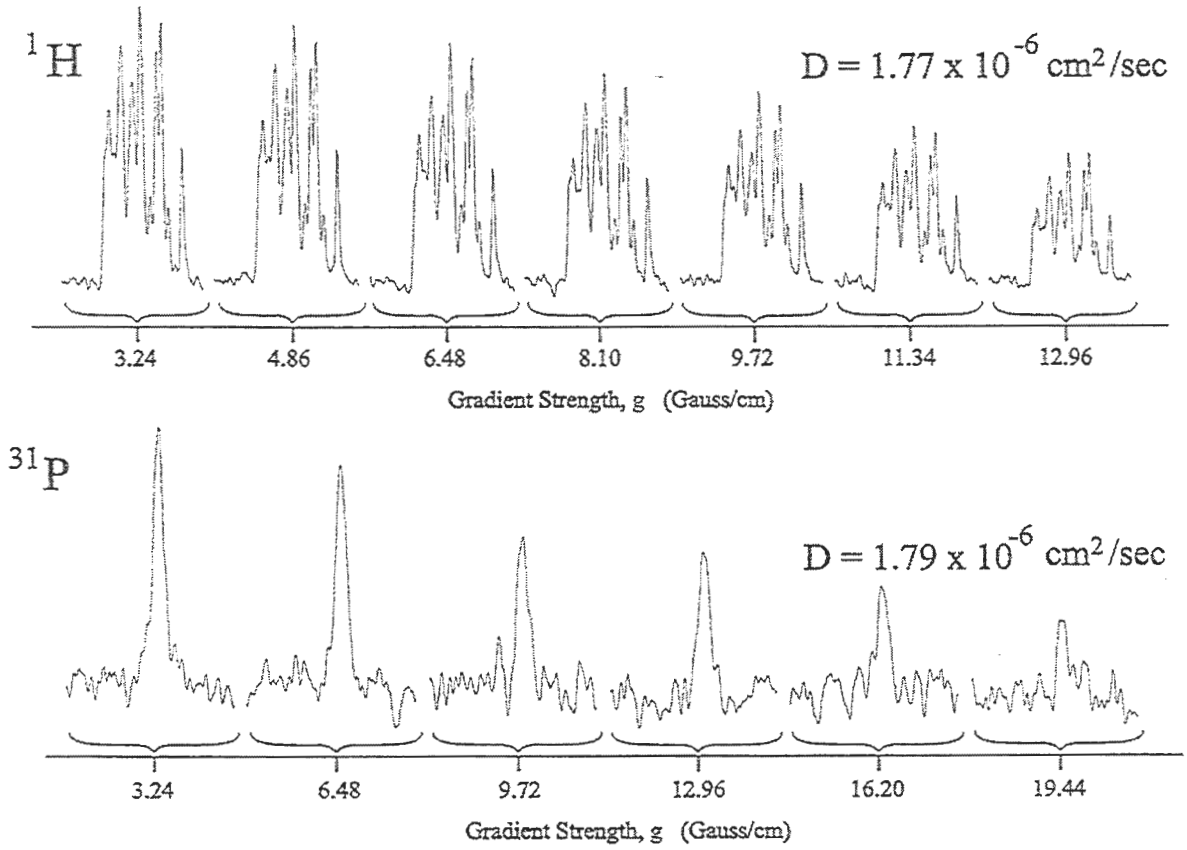

Figure 2 - Diffusion of the SV40 Okazaki Fragment measured using ^{1}H (top) and ^{31}P (bottom).

Sincerely yours,

Chris Hudalla

Bill Gmeiner

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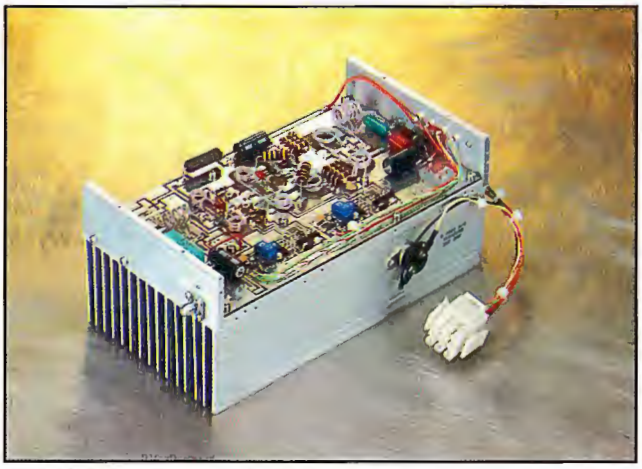

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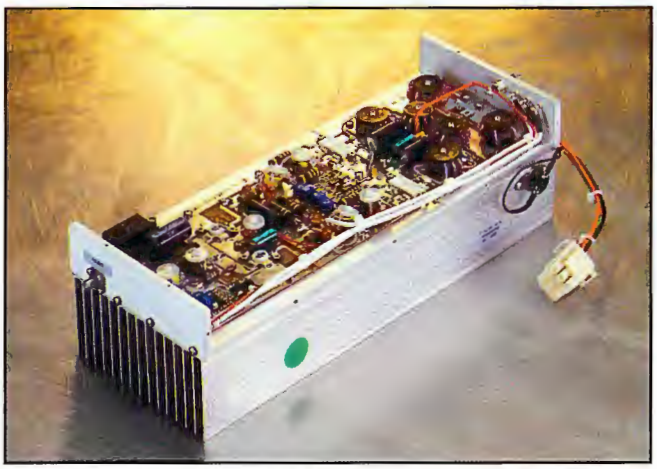

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SCIENTIFIC & MEDICAL PRODUCTS

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FACILITIES

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PRODUCTS

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April 2, 1998
(received 4/6/98)

Dr. B. L. Shapiro
The NMR Newsletter
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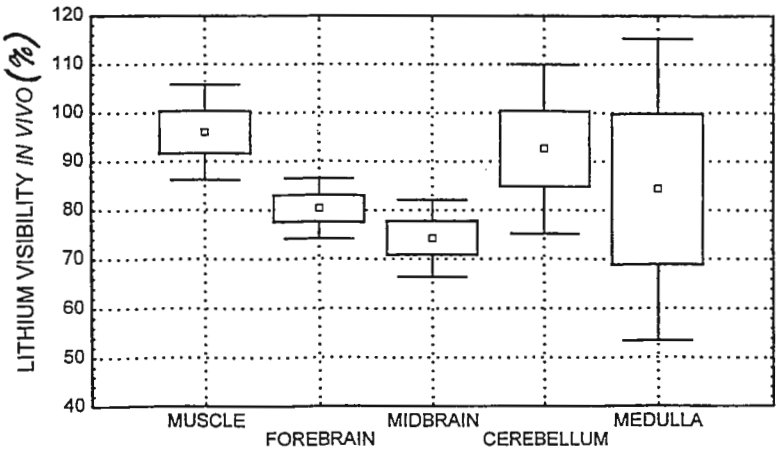
Title: ^{7}Li VISIBILITY IN RAT BRAIN AND MUSCLE *IN VIVO* BY ^{7}Li NMR IMAGING
Also: **New Address**

Dear Barry:

Lithium (Li) is the treatment of choice for manic-depressive illness. The magnitude of the pharmacologic effect of Li depends on the concentration at the receptor sites in the brain, which may not be reflected in the serum concentration. ^{7}Li NMR is potentially a noninvasive, *in vivo* measure of Li concentration, particularly one that can be applied to humans. Alkali-metal NMR studies on a wide variety of tissues have shown that the ion concentration determined by NMR is often substantially less than that determined by other analytical methods. The extent to which the ^{7}Li NMR signals from biological tissues, such as brain and muscle, exhibit such reduced visibility *in vivo* has not been determined previously. My coworkers (John Pearce and Dr. J.E.O.Newton) and I have determined the apparent concentration of Li *in vivo* for several regions in the brain and muscle of rats by ^{7}Li NMR imaging at 4.7 T with inclusion of an external standard of known concentration and visibility. The average apparent concentrations were 10.1 mM for muscle, and 4.2-5.3 mM for various brain regions under the dosing conditions used. The results were compared to concentrations determined *in vitro* by high resolution ^{7}Li NMR spectroscopy of extracts of brain and muscle tissue from the same rats. The comparison provided estimates of the ^{7}Li NMR visibility of the Li cation in each tissue region. These results are shown in the figure. Although there was considerable scatter of the calculated visibilities among the five rats studied, the results suggested essentially full visibility (96%) for Li in muscle, and somewhat reduced visibility (74-93%) in the forebrain and midbrain regions. A full description of this work has been accepted for publication in *J. Magn. Reson.*



On another matter, we have just moved our laboratory from our current site to the VA Hospital in North Little Rock, AR, although the institutional affiliation will remain the same. Please address future correspondence to:

NMR Lab (151K/NLR)
VA Medical Center
2200 Fort Roots Drive
North Little Rock, AR 72114
phone: (501)378-5025
FAX: (501)378-5026
e-mail: rkomoros@radlan.uams.edu
Lab phone: (501)661-1202, ext.7666

Richard A. Komoroski
Professor

Forthcoming NMR Meetings, continued from page 1:

NMR Spectroscopy of Polymers, Breckenridge, Colorado, **January 24-27, 1999**; an International Symposium Sponsored by the Division of Polymer Chemistry, American Chemical Society; Organizers: P. T. Inglefield and A. D. English: Registration contact: Neta L. Byerly, Division of Polymer Chemistry, Inc., Virginia Tech, 201 Hancock Hall, M.C. 0257, Blacksburg, VA 24061; 540-231-3029; Fax: 540-231-9452; email: nbyerly@vt.edu.

40th ENC (Experimental NMR Conference), Clarion Plaza Hotel, Orlando, Florida, **February 28 - March 5, 1999**;immediately preceding Pittcon in Orlando; Contact: ENC, 1201 Don Diego Avenue, Santa Fe, NM 87505; (505) 989-4573; Fax: (505) 989-1073; Email: enc@enc-conference.org.

Spin Choreography - a symposium in appreciation of Ray Freeman, Cambridge, England, **April 8-11, 1999**; web site: http://mchsg4.ch.man.ac.uk/mcmr/RF.html; fax: c/o M.H.Levitt +46-8-15 2187; email: mhl@physc.su.se.

41st ENC (Experimental NMR Conference), Asilomar Conference Center, Pacific Grove, CA, **April 9-14, 2000**; Contact: ENC, 1201 Don Diego Avenue, Santa Fe, NM 87505; (505) 989-4573; Fax: (505) 989-1073; Email: enc@enc-conference.org.

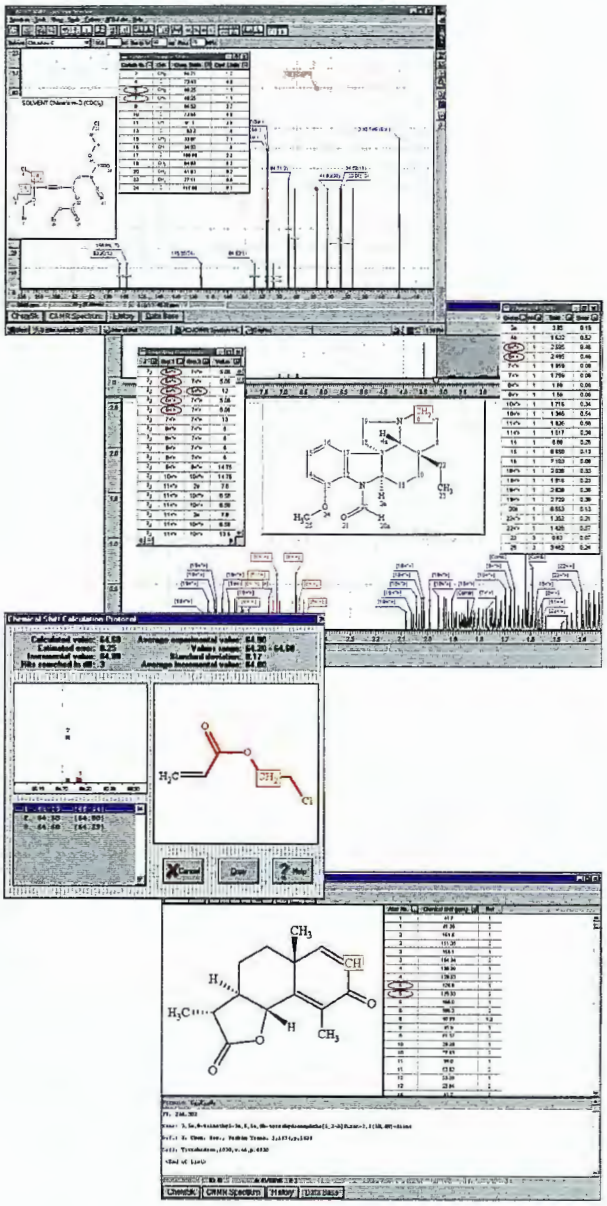
Additional listings of meetings, etc., are invited.

E-mail Addresses Wanted

Please include your e-mail address on all correspondence, including technical contributions, or send me an e-mail message. This will make it more convenient - and economical - to contact you. Thanks.

BLS
shapiro@nmrnewsletter.com

ACD/HNMR,CNMR Predictors



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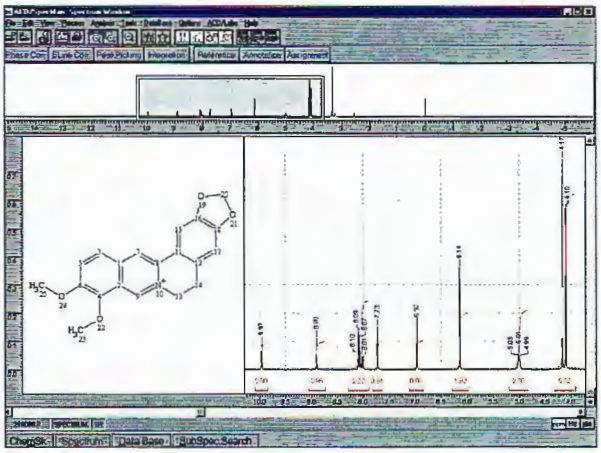

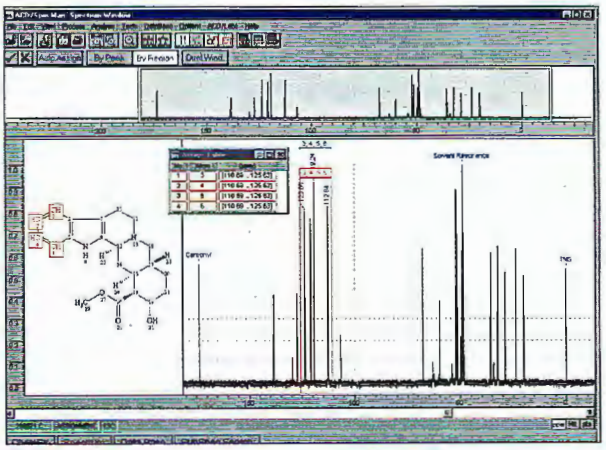

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April 6, 1998
(received 4/20/98)

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

IMPROVED HYDROGEN DETERMINATION IN PETROLEUM STREAMS USING A BENCH TOP PULSED NMR ANALYZER

Accurate feed and product hydrogen measurements are key to determining hydrogen consumption required in today's refining environment. Total hydrogen has been measured in aviation fuels (ASTM D3701[1]) and in other petroleum streams (ASTM D4808[2]) using low resolution continuous wave (CW) spectrometers and low resolution pulsed NMR spectrometers.[3,5] Hydrogen content can also be measured by combustion (ASTM D5291).[4] Hydrogen balances for process units are accomplished by determining the hydrogen content of feeds and products by one or more of these methods. Unfortunately none of the methods can measure total hydrogen with precision better than 0.1 - 0.2 % over the entire range of streams or content. Significant effort has been directed at improving the precision of routine hydrogen analyses using inexpensive low resolution bench top NMR analyzers.[5] However, since these methods are not directly applicable to high boiling, waxy samples and total liquid product samples containing light ends, there continues to be a need to improve hydrogen analyses for refinery and pilot units.

In this contribution, we report method development at sample temperatures up to 60°C using a density normalized input that has resulted in significant improvements in the precision (repeatability ≤ 0.05 wt %) and accuracy of hydrogen determinations on a broad range of process streams. This revised and simplified low resolution pulsed NMR approach is very efficient and expands the range of samples which can be routinely analyzed for hydrogen to include all samples that are liquids at ≤ 60°C. Samples are quickly and non-destructively measured without dilution. Volatile samples containing light ends can also be handled using sealed tubes with the density normalized approach described below. A Bruker Minispec 120 pulsed NMR Analyzer equipped with a 10 mm zero background variable temperature probe and an external heating and cooling device capable of temperature control of ±0.05°C is used for this measurement. A block diagram of the instrument configuration is given in Figure 1. Just as with previous weight normalized methods, temperature control is the most critical parameter for accurate measurements. The sample preparation, calibration, and instrument parameters are based on this careful control of temperature. The instrument is set up using the preprogrammed software application named fid_n_v supplied by Bruker which allows a calibration curve to be generated using calibrants with known hydrogen contents and densities. A calibration curve is generated using dodecane, butylbenzene, phenylheptane, and methylnaphthalene as calibrants. Choice of calibrants are dependent on two criteria: i) calibrants should cover the range of hydrogen contents for the experimental samples, and ii) calibrants should be homogeneous liquids at the measurement temperature. Every calibration should be performed using fresh calibrants. A calibration curve with a correlation coefficient of $\geq .9995$ should be obtained prior to any measurements on experimental samples. Samples, having been tempered in the heating block for 10 minutes at the temperature to be measured are inserted into the magnet of the Minispec. The sample is allowed to equilibrate to the probe temperature (the temperature of the probe is controlled by the same circulating bath as the densitometer). Absolute H measurements on unknowns are done by comparing the normalized NMR response to this calibration curve. Sample preparation using the density normalised approach is greatly simplified. There is no need for accurate sample weights and sample heights. All that is necessary is that the coil volume be filled (which is easily accomplished by overfilling) and the sample density be known. Samples are heated in an oven at 60°C to ensure homogeneity and prepared neat in a 10 mm flat bottom tube to a minimum height of ~40mm. Samples are placed in a heating block for at least 10 minutes to equilibrate samples at the specified temperature. Density measurements are made on an ANTON PARR mPDS 2000 densitometer at 60°C. Prior to measurements the densitometer is calibrated following the manufacturer's instructions and the procedures in ASTM method D4052.[6]

This approach has greatly expanded the range of materials for which wt.% hydrogen can be routinely measured. Our experience with the application of the density normalized approach at elevated temperature has shown that the short term precision is also <0.05 wt. %.The density normalized method with a variable temperature probe offers the advantages of simpler sample preparation and applicability to a wider range of materials than the weight normalized approach with an absolute probehead. These new and simplified methods offer potential for the more accurate determination of hydrogen balances of refinery processes.

Gordon J. Kennedy

Gordon J Kennedy

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1. ASTM D3701 (1993) "Hydrogen content of aviation turbine fuels by low resolution nuclear magnetic resonance spectroscopy". American Society for Testing and Materials, Philadelphia.
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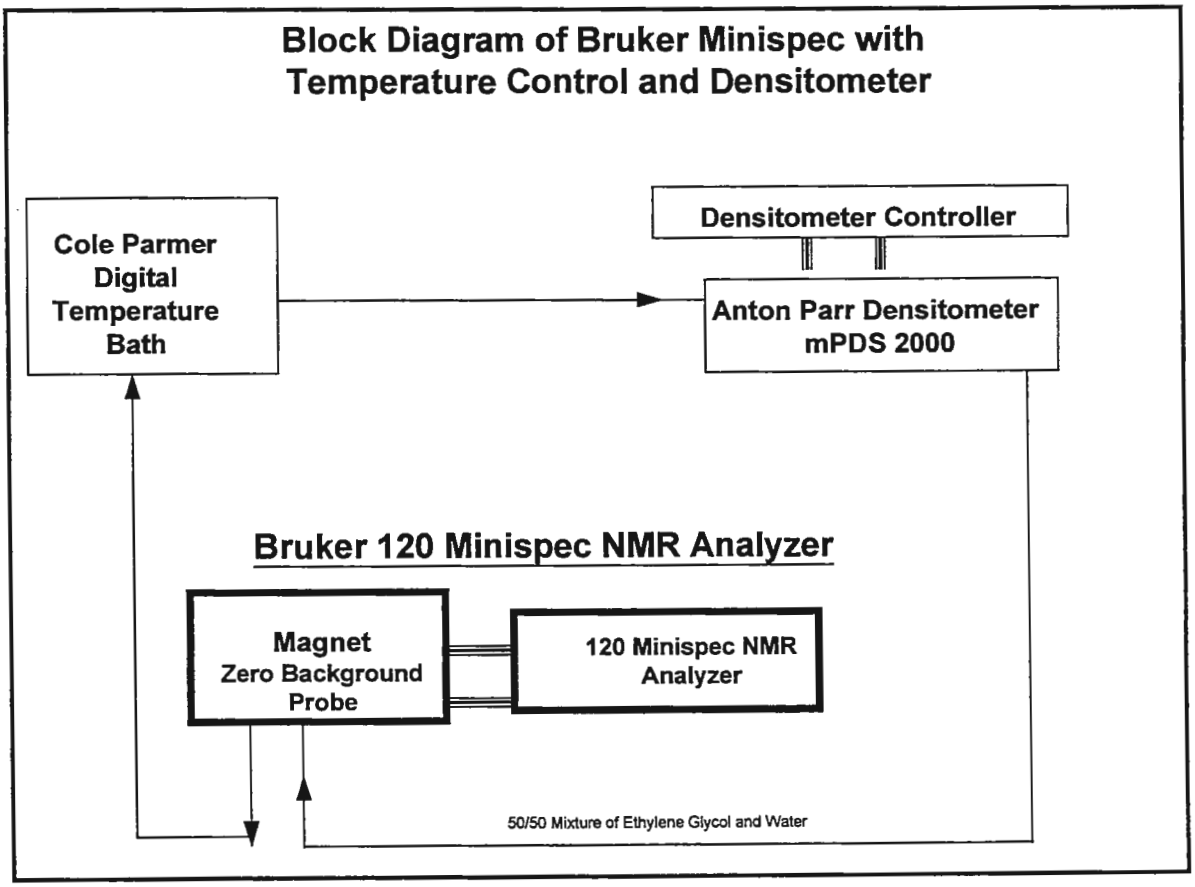

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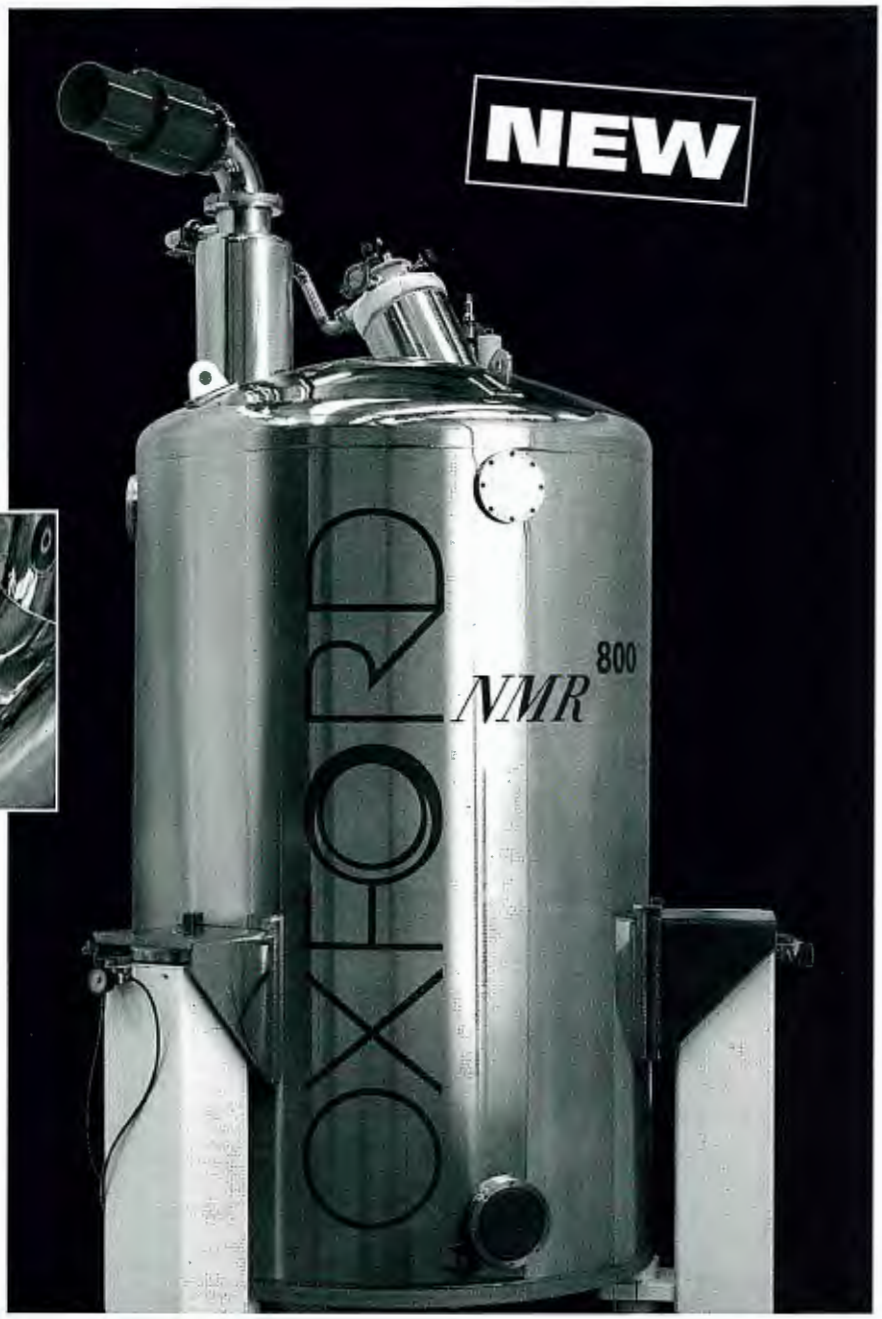

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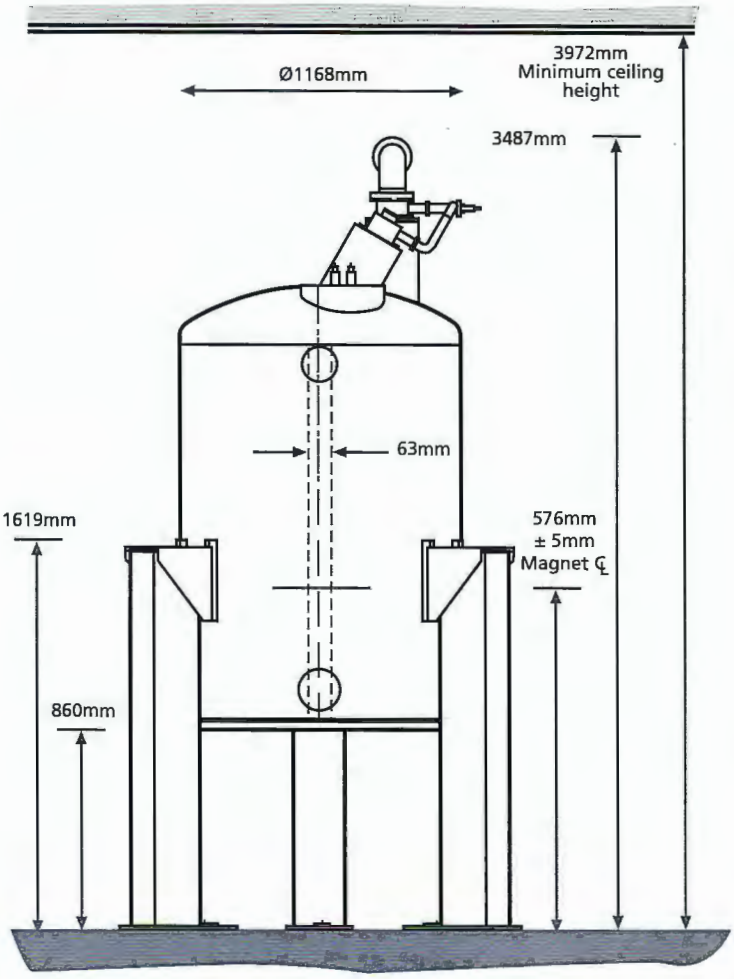

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April 15, 1998
(received 4/20/98)

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

Crystalline Wax Content in Asphalts

Dear Barry:

Recent studies have shown that the wax content is an important parameter affecting the rheological properties and road performance of an asphalt.[1-3] The amorphous and crystalline wax phases in asphalt have been identified using ^{13}C CP/MAS and dipolar dephasing techniques.[4] We report here the quantitation of the crystalline wax content and its correlation with DSC measurements. The CP/MAS NMR spectra of five asphalts (Figure 1) of varying wax content were obtained at -45°C. The spectra were obtained at -45°C to increase the C-H cross-polarization efficiency resulting from the reduction in the molecular segmental and rotational motion of the aliphatic carbons at the low temperature. In addition, the increase in cross-polarization efficiency resulted in an enhanced signal-to-noise ratio.

The spectra were deconvoluted and the mass percent of crystalline methylene carbon at 32 ppm was calculated for each asphalt from the percent carbon, fraction of aliphatic carbons and the fraction of crystalline methylene carbon. The mass percent calculated from NMR data and the mass percent of crystalline wax from DSC measurements are plotted as shown in Figure 2. A 1:1 correlation is obtained but we have found that the correlation depends upon the DSC time-temperature profile, method of computing the crystalline fraction, and the enthalpy value used for n-alkanes. A value of 180 J/g was used which is an average value most often reported in the literature for paraffinic material in asphalts.

Obviously, it is more costly to measure the crystalline wax content by NMR, but it may provide a better means of studying physical aging at low temperature and steric hardening of asphalts at temperatures above T_g both of which depend on the formation of crystallites. Details of the NMR and DSC studies will appear in a journal later this year.

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Ph 307-721-2370
Fax 307-721-2345
e-mail: dnetzel@uwyo.edu

Francis P. Miknis

Laurent C. Michon

Thomas F. Turner

References

1. P. Claudy, J. M. Letoffe, F. Rondelez, L. Germanaud, G. King, and J. P. Planche, A New Interpretation of Time-Dependent Physical Hardening in Asphalt Based on DSC and Optical Thermoanalysis, Preprints, Am. Chem. Soc. Div. Fuel Chem., 37(3): 1408 (1992).

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4. Daniel A. Netzel, Low Temperature Studies of the Amorphous, Interfacial, and Crystalline Phases in Asphalts Using Solid-State ^{13}C NMR, Preprint No. 980829. 77th Annual Transportation Research Board Meeting, Washington, D. C., January 11-15, 1998.

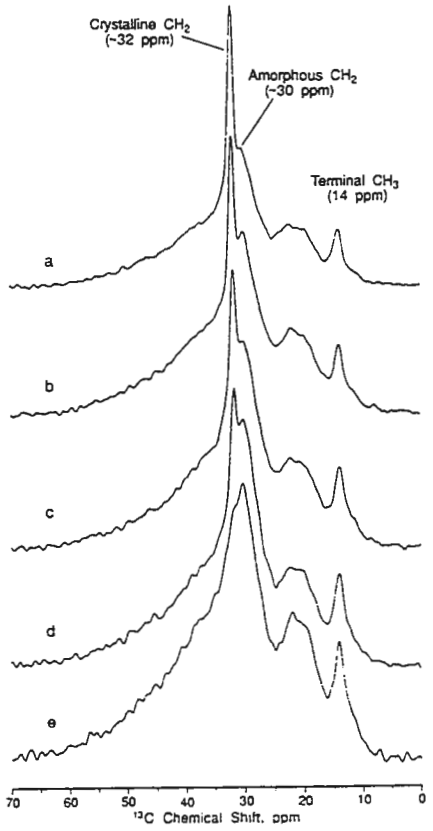

Figure 1. ^{13}C CP/MAS NMR Spectra at -45 °C for Five Asphalts

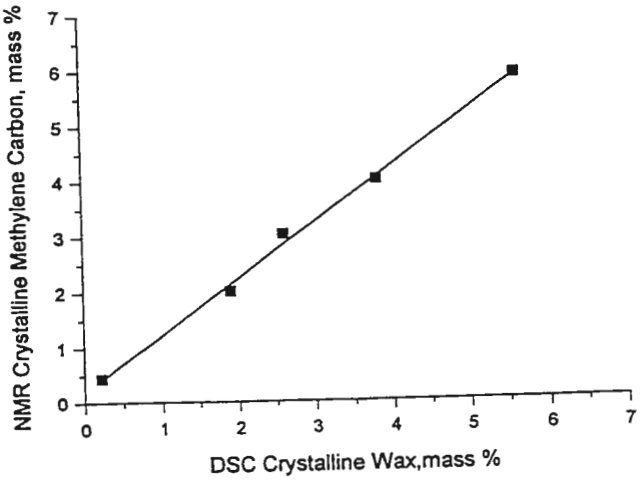

Figure 2. A Correlation Plot of NMR Crystalline Methylene Carbon Content versus the Crystalline Wax Content from DSC Measurements for Five Asphalts

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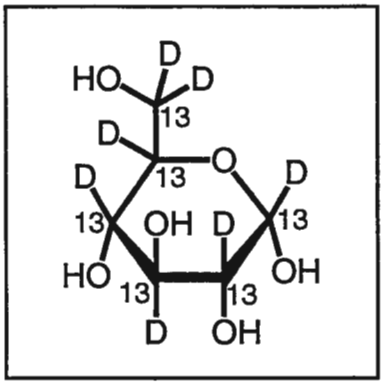

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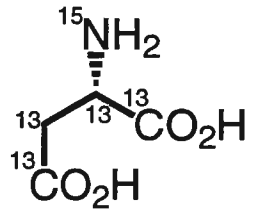
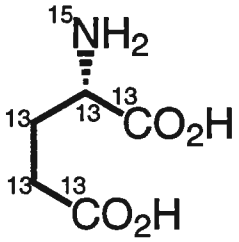
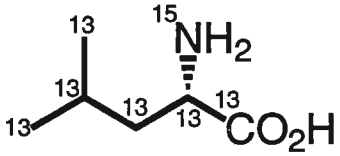


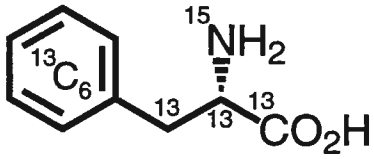
L-Aspartic Acid-$^{13}C_4$,^{15}N



L-Glutamic Acid-$^{13}C_5$,^{15}N



L-Leucine-$^{13}C_6$,^{15}N



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Glycine-$^{13}C_2$,^{15}N
L-Isoleucine-$^{13}C_6$,^{15}N
L-Leucine-$^{13}C_6$,^{15}N
L-Lysine-$^{13}C_6$,$^{15}N_2$
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March 28, 1974 [sic]

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

Shtuyot Bemitz Agvaniyot

Dear Barry:

We have constructed a device which is quite useful for nmr in all types of systems. This has several novel features, e.g.:

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5. The machine smiles during a spin tickling experiment.
6. Equipped with a 45 KW power transmitter. This is supplied with an answering service to reply to complaints from radio amateurs in Siberia.
7. Optical detection is optional. Sensitivity is so enormous that half a photon has been detected. The other half is being kept spin-locked under close guard of a hyperfine component.
8. The device operates in any frame including the rotating, pulsating, and randomly fluctuating frames.
9. Can do DEFT, TDFT, PRFT, WEFT, LEFT, GLBM, etc., by selecting a combination of four letters. If no experiment exist for a selected combination, the device will invent one and publish a preliminary communication.

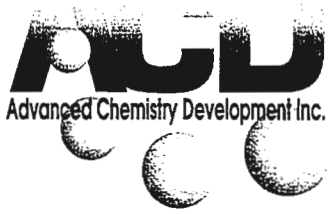
This work was hardly supported.

Best regards,

Alex

Alex Pines

AP:crh



JCAMP - The continuing saga of a "Standard" File Format for NMR

April 9th, 1998 (received 4/16/98)

Dear Barry,

In the continuing exchange of commentary and opinion within the NMR newsletter regarding the JCAMP *standard* file format for NMR, let me now add my statements on behalf of Advanced Chemistry Development Inc. During recent exchanges (NMR Newsletter and the JCAMP meeting at ENC '98) Virginia and Woody expressed concern regarding the need for the NMR industry to finally decide what the acceptable JCAMP format should be. In response, Mick Grzonka was kind enough to give us a historical overview regarding attempts to declare and invoke an NMR standard which, according to Mick's note, was actually declared to be the JCAMP format at a meeting in 1990..

Recently at ENC '98 yet another attempt was made to define and defend both the need and the details of a universally acceptable NMR data format appropriate to both 1D and multi-D spectroscopy. During this conversation Tony Davies of the IUPAC JCAMP sub-committee gave a seminar detailing development of the JCAMP standard which can actually take five separate forms: Fixed Length, DIF, PAC, SQZ and DIFDUP. The conclusion of the meeting *appeared* to lead to the decision that there was a need to develop a form of JCAMP which would deal with the complexities of multi-dimensional NMR data and, assuming funding, we can only wish Tony and the committee the best of luck in developing an appropriate *standard.*

During the same meeting concern was expressed regarding the need for the vendors to decide on which of the five JCAMP formats would become the standard export format. The driving force behind this discussion appeared to be the need to limit the number of JCAMP import filters that need to be coded into home-grown and third party vendor spectroscopy software. As was so elegantly summarized by Tony Davies, the idea behind the JCAMP format is to allow access to stored data years from now when vendor file formats will have shifted dramatically. Since the JCAMP format has already been around for a number of years and has delivered the five iterations named above, it is therefore necessary for third party software vendors to support all present JCAMP versions as imports as well as any new issues which may occur. Since the number of desktop packages for both 1D and multi-dimensional processing has increased it would be expected that these packages would also support the export of the JCAMP formats. In the initial communication in this series the availability of time domain data in JCAMP format was questioned. Since there may be a need to reprocess data using modified processing parameters it would certainly be appropriate for software to support the export of time domain data.

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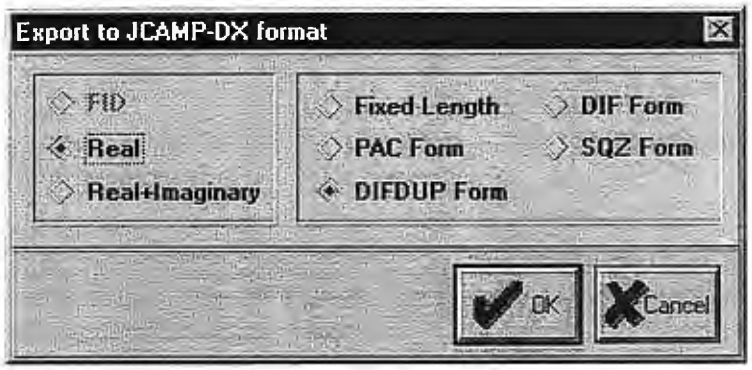
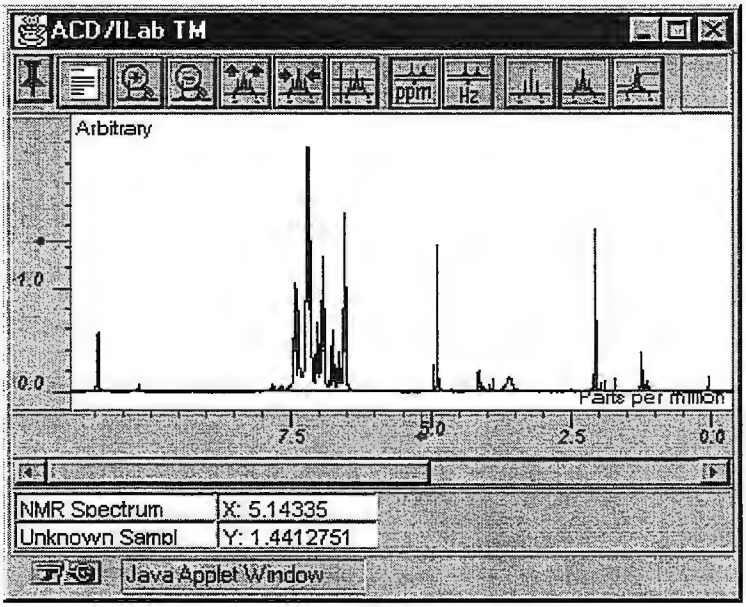
With all of these issues in mind, and with our present development of SLIMS, a fully web-based LIMS environment for Samples, Structure and Spectra, we have ensured that our desktop processing and spectral management package ACD/NMR Manager has both import and export capability for ALL of the JCAMP formats defined to date for NMR. As the dialog box below indicates, we have the capability to export both time domain data, and frequency domain data as both REAL and REAL/IMAGINARY data.

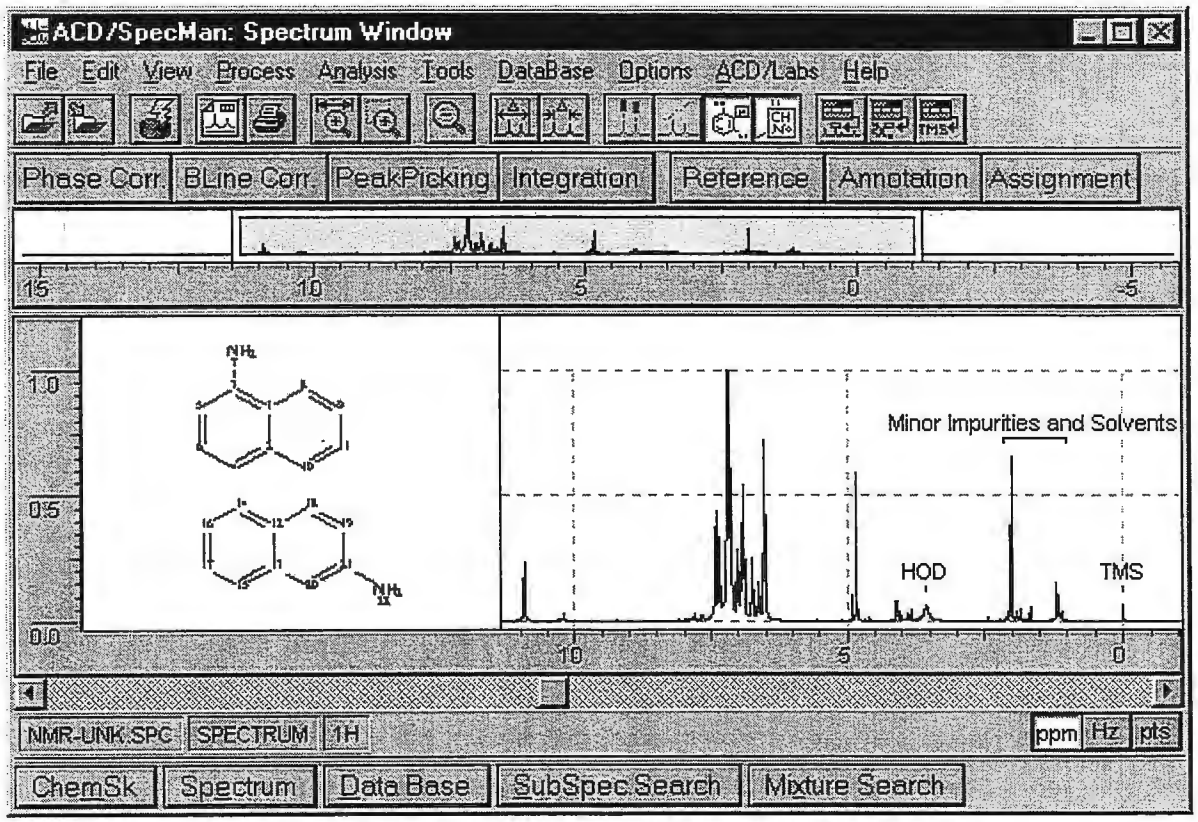


Using the capability for the spectral export of NMR spectra from ACD/NMR Manager, spectra can be exported to the spectral database within the SLIMS package (www.acdlabs.com/slims) for direct display on the web in our Java-based spectral display applet as shown below.



This applet is a platform independent Java application that runs within a web browser and therefore offers platform independent viewing of any spectra populated into the database. The standard applet toolset includes zooming, changing axis units, display of discrete lines and integration capability. The applet has the capability to display NMR, MS, IR, UV-Vis, Raman, X-ray spectra and LC curves using the data formats of SPC, JCAMP and NetCDF. Use hyperlink download capabilities platform *dependent* processing

packages can be triggered as helper applications for remanipulation of the data. The window below displays the NMR spectrum shown in the Java applet above, when the NMR manager is loaded as the helper application by the browser. *Note that the spectrum is indeed the one shown in the spectrum applet.* In this case additional manipulation included baseline correction, adding a structure through the integrated Chemsketch interface and performing on-screen annotations.



With the continuing development of the Java Development Kit and the increasing installation of intranet enterprise capabilities, we can only assume that third party processing and spectral display software vendors will be developing plug-in or Java applet capabilities for browsers in the near future. In this venture ACD Inc. are finding that established data standards such as JCAMP are extremely useful in enabling this technology. To support this enabling technology, all spectrometer vendors are invited to make JCAMP export a simple option in their standard software.

Best wishes,

Tony Williams

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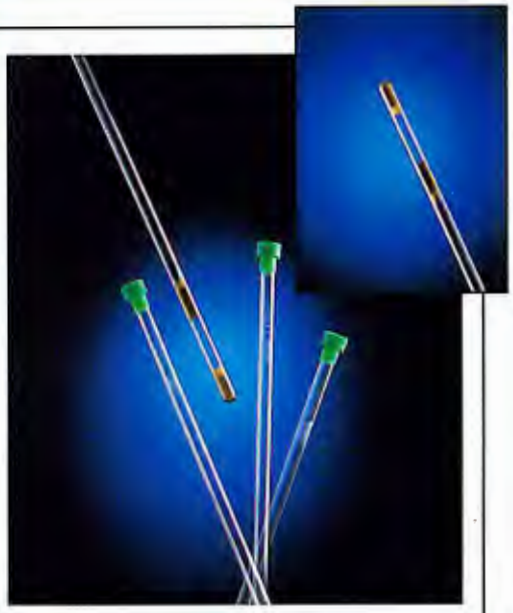

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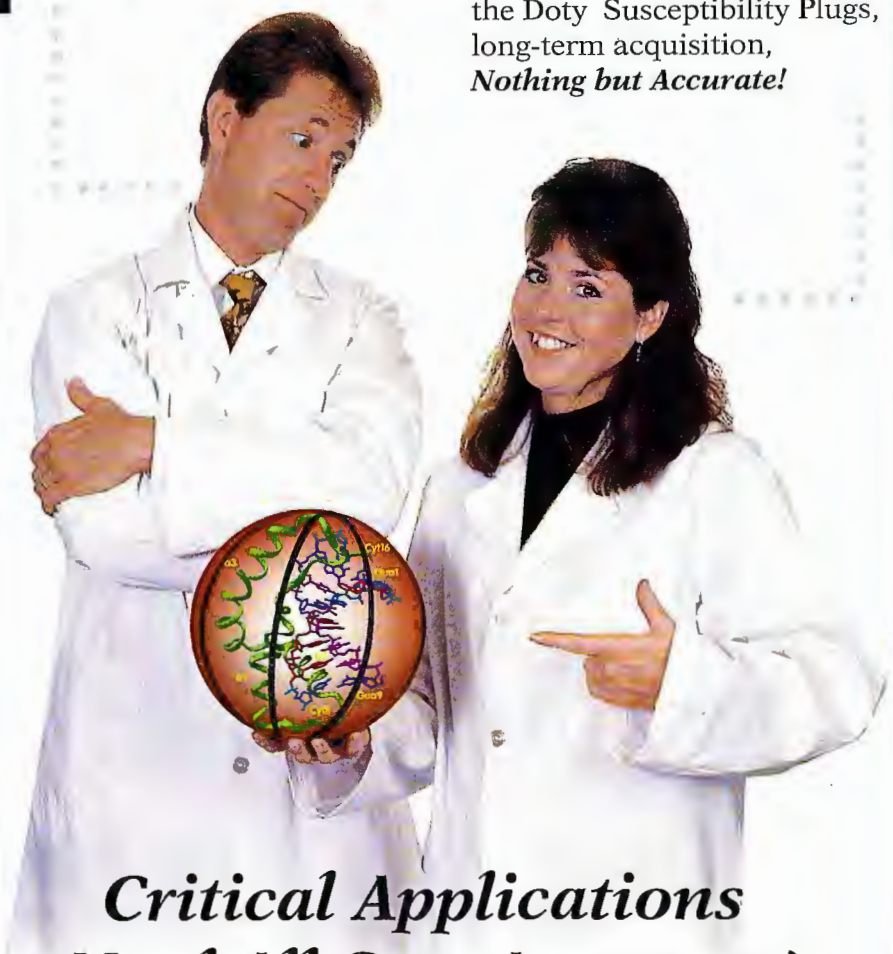

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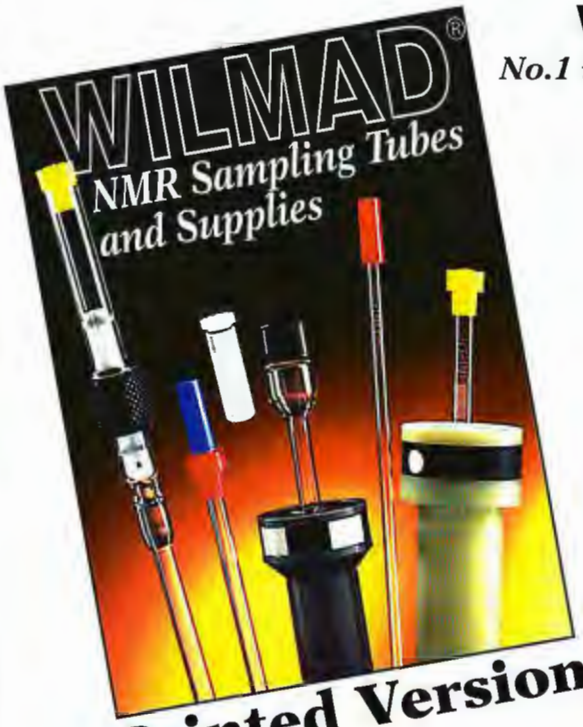

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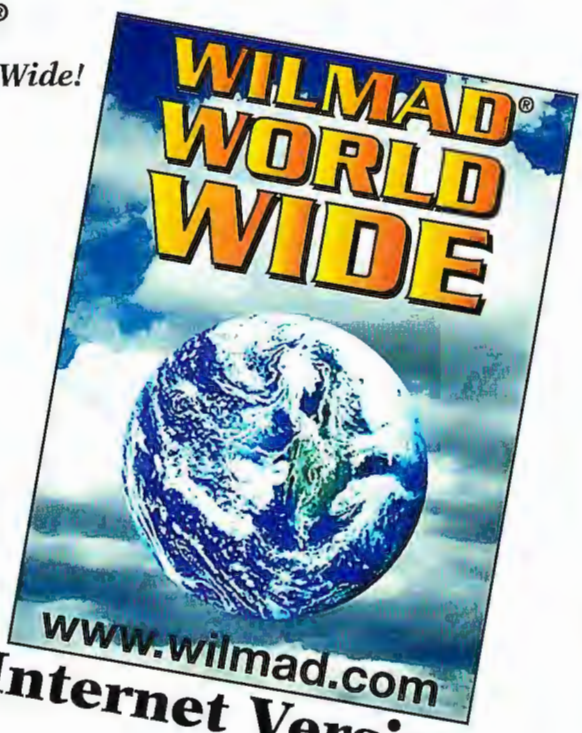

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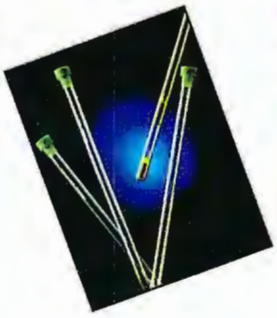

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PO Box 117200
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April 14, 1998
(received 4/20/98)

Dear Barry:

Since I have retired as editor of JMR, I have had opportunity to review some of the interesting work which we have been carrying out in the U.F. laboratories on the fluorine-19 NMR of highly fluorinated molecules. Your readers may be interested in the results which we have obtained and the correlations we have been able to make.

One category of materials from which we have examined a variety of samples includes compounds which are mostly perfluorinated but which have one or more chlorine atoms also present. When the fluorine atoms observed are on the same carbon as are the chlorines, the direction of the shift depends upon whether the group in question is a methylene or a methyl group. In a methylene group, the fluorine which is left behind on substitution always appears at a lower frequency (higher field) than would one in a CF_2 group in the same environment. In contrast, substitution of one chlorine in a CF_3 group causes a large shift to higher frequency. Surprisingly, substitution of a second chlorine has the opposite effect, so that the order of resonance frequencies from low to high is: CF_3– $CFCl_2$– CF_2Cl–

If one examines the effects of chlorines on shifts of fluorines along the chain away from the position of substitution, they are all in the unshielding direction (to higher frequency), show fairly good additivity, and decrease in magnitude with number of bonds. They are fairly well represented by an additivity scale as follows:

Each Cl on an alpha carbon	+6 ppm
Each Cl on a beta carbon	+3 ppm
Each Cl on a gamma carbon	+0.5-1.0 ppm

As an example, the shifts of a fluorine in a –CFCl– group are shown in the following table of representative data:

STRUCTURE	EXPERIMENTAL SHIFT	PREDICTED SHIFT
$-CF_2CF_2CFClCF_2$	–136.0	(Reference)
$-CF_2CF_2CFClCF_2CFCl$	–134.0	–133
$-CF_2CF_2CFClCF_2Cl$	–131.7	–130
$-CF_2CF_2CFClCF_2CFCl_2$	–130.7	–130
$-CF_2CF_2CFClCF_2CCl_3$	–128.8	–127
$CF_2ClCFClCFClCF_2Cl$	–120.0	–121

Introduction of a –CFCl– group in a fluorocarbon chain corresponds to creation of an asymmetric center, which leads to nonequivalence of the fluorine pairs in nearby $-CF_2$ groups. One might expect this effect to be the largest in the methylene groups adjacent to the asymmetric center, but this is not observed. Rather the effect is greatest for the fluorine pair on the second carbon from the center. We interpret this as a consequence of steric interactions (somewhat like the "repulsive unshielding" noticed many years ago in substituted ethanes) along with a preference of the chlorofluorcarbon chain for an extended conformation.

Yours,
Wallace
Wallace S. Brey

Frank D. Blum

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fblum@umr.edu

UNIVERSITY OF MISSOURI-ROLLA
Missouri's Technological University

Professor B. L. Shapiro
TAMU NMR Newsletter
966 Elsimore Court
Palo Alto, CA 9430

(received 4/22/98)
Apr. 14, 1998

^{29}Si MAS NMR and Gravimetric Analysis of an Acrylic Silane Coupling Agent

Dear Barry,

The silane structures of an acrylic silane, APMS (CH_2=CH-C(O)-O-CH_2-CH_2-CH_2-Si-(OCH_3)$_3$)were studied using NMR and gravimetric analysis APMS after heat treatments at 110 and 600 °C. APMS was hydrolyzed in acidic aqueous solution and condensed at 110 °C. The condensed APMS was then pyrolized at 600 °C.

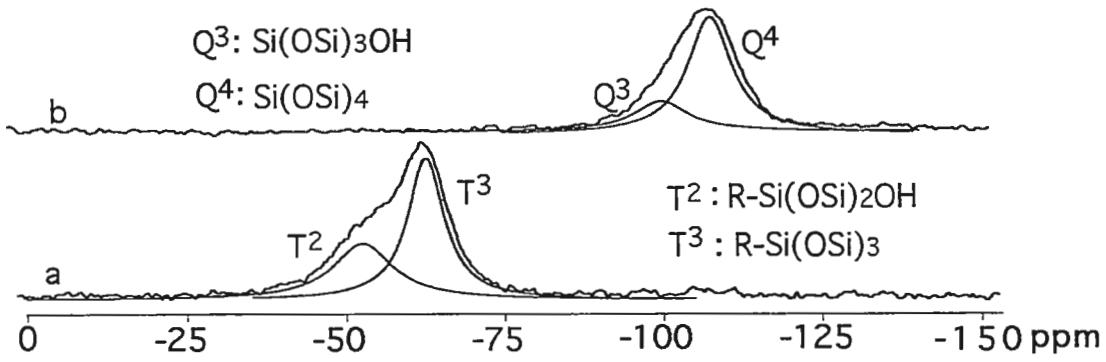
^{29}Si MAS spectra (below) showed that most of the APMS silanol groups were highly condensed by the heat treatment at 110 °C, and that most of the APMS alkyl groups had been replaced by oxygen during pyrolysis at 600 °C. The fully relaxed ^{29}Si MAS NMR spectra of APMS show the amounts of T and Q species of APMS. The spectrum (a) of APMS that was treated at 110 °C had only T species and was fit quite well with two components: 25% T^2 and 75% T^3 species. The spectrum (b) of APMS that was treated at 600 °C showed only Q species and was modelled with two components: 20% Q^3 and 80% Q^4 silane species.

From gravimetric analysis, the differences in the weights of APMS following various heat treatment steps made it possible to confirm the nature of the chemical species present. After the heat treatment at 110 °C, 26% T^2 species and 74% T^3 species were found. This suggested that a more highly condensed silane resulted from the heat treatment. For the 600 °C heat-treated APMS, the weight of APMS was reduced to 26.3 % of their original weights and there were 18% Q^3 species and 82% Q^4 species. These gravimetric analysis results agreed nicely with results of ^{29}Si MAS NMR.

Sincerely Yours,

Hyoryoon Jo

Frank D. Blum



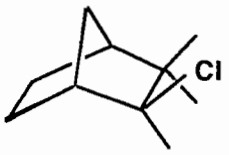
TCU

DEPARTMENT OF CHEMISTRY

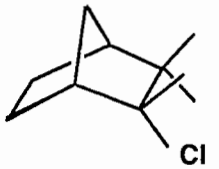
(received 3/30/98)
March 24, 1998

Dear Barry:

One can only wonder how long empirical shift prediction programs will survive given the rapidly advancing methods of calculating such by accurate ab initio methods. A classical problem (meaning buried in obscurity) is the configuration of camphene hydrochloride. Recently in answer to a question regarding this matter a world famous player in the field of cation mechanisms replied: "Everybody knows the chlorine is exo." I don't think the statement is a proof of structure. A thorough search of the literature fails to show any real proof. The best evidence seems to be that the rearrangement to isobornyl chloride follows the expected path for Wagner-Meerwein rearrangements. However, even the proof for the latter structure is somewhat dubious.



Camphene.HCl



Isocamphene.HCl

Recently, Forsyth and Sebag (JACS **1997,** *119* , 9483) have produced a neat way to get around the problem of having to compare calculated magnetic susceptibilities with those of TMS to get chemical shifts. Their method is applied in the accompanying figure which has three kinds of data points shown. The tiny dots, used to determine the line, are forty chemical shifts (calculated vs experimental) for a series of substituted norbornanes taken from the text by Levy and Nelson ("Carbon-13 Nuclear Magnetic Resonance for Organic Chemists", John Wiley & Sons, New York, 1972). The line shown has an R^2 of 0.9799. The dashed lines are 95% confidence levels. Geometries were calculated at the BPW91/6-311G* level and GIAO susceptibilities were at the BPW91/6-311G** level. The idea was to see if the calculated shifts for the two structures allowed a clear cut choice as to the correct configuration. The calculated points for camphene hydrochloride are given as squares and for isocamphene hydrochloride by Xs. Clearly, this plot fails to allow a decision..

However, the situation changes if one looks at the the proton chemical shifts for the methyl groups. The proton chemical shifts determined by two groups in the literature for the C-2 endo- methyl, C-3 exo-methyl and C-3 endo- methyl as follows: 1.56(1.59), 1.17(1.20) and 0.97 (0.98) ppm respectively. The computed values for these methyls for camphene hydrochloride (isocamphene hydrochloride) are 1.57(1.58), 1.22(1.00) and 0.96 (1.45). These results are clearly in line with the conventionally accepted structure for camphene hydrochloride. I might add: "Thank heavens."

Bill

(William B. Smith)

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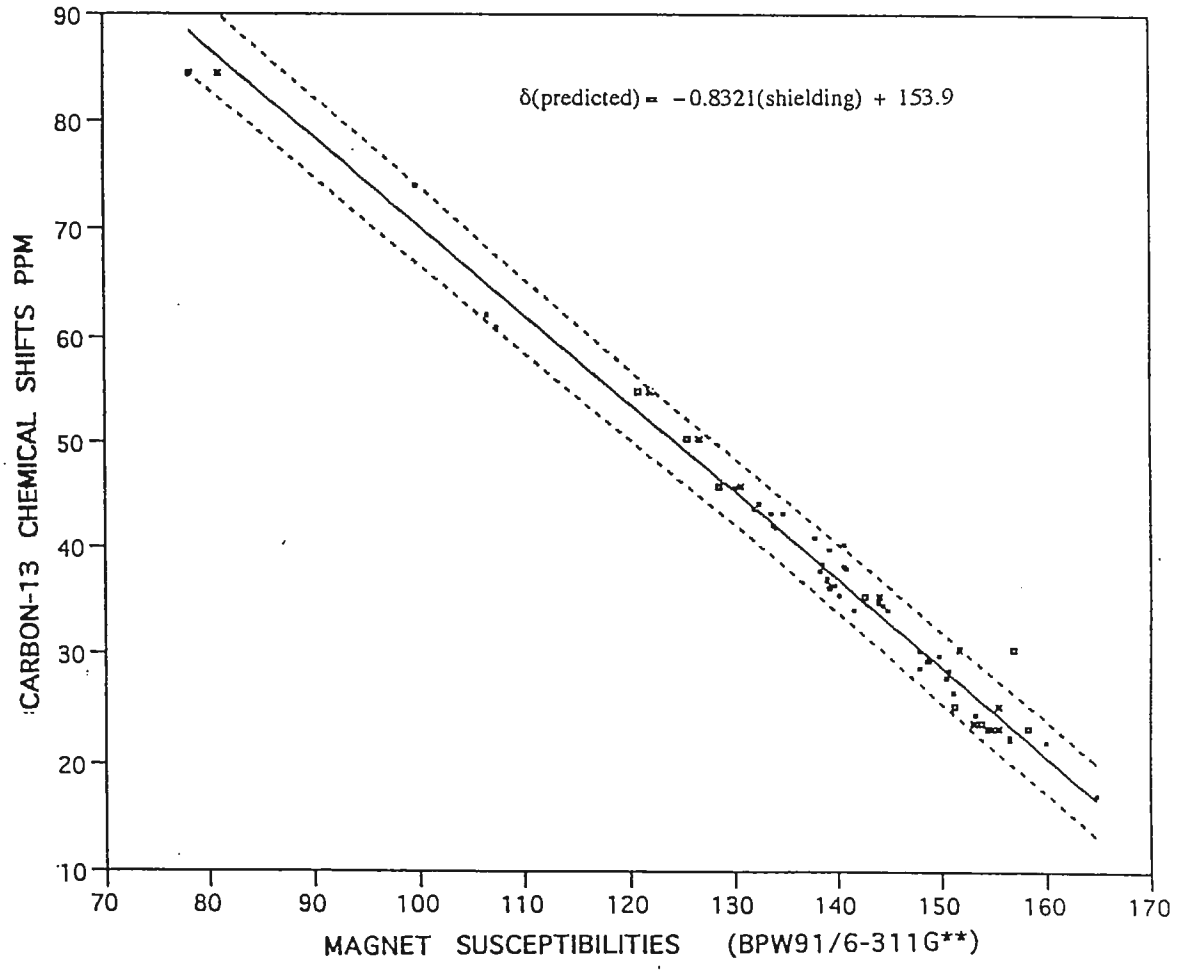

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April 3, 1998 (received 4/4/98)

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Knowledge required:

Fundamental knowledge of image processing and analysis, including necessary programming skills, at the B.S. or M.S. level, with proficiency in the Unix operating system, MATLAB software, and an understanding of image properties and manipulation generally. Programming knowledge of Tcl/Tk is desirable for MedX scripting. Ability to communicate this information to laboratory investigators is required. Knowledge of techniques to write effective documentation and on-line help for customized software is also required. Basic knowledge of neuroanatomy and MRI and PET techniques is desirable.

Duties:

The MRI/PET Computer Specialist supports our research program on structural and functional brain changes as predictors of cognitive and memory impairment and Alzheimer's Disease; develops methods for processing and statistical analysis of longitudinal neuroimaging data, including magnetic resonance images (MRI) of brain structure and positron emission tomography (PET) scans of brain activity and blood flow; performs image processing and analysis under the supervision of the principal investigator and trains other laboratory personnel in these methods.

The incumbent organizes and maintains image files, including file transfers, compression, and backups. The incumbent is responsible for tracking image processing work flow and performing components of image processing, e.g. stripping of extracranial tissues from images, registration of PET-to-MRI and PET-to-PET images, image segmentation, and semi-automated regional analysis. The incumbent works closely with neuroimaging project scientists to preprocess images in preparation for statistical analysis. In addition, the incumbent is responsible for customizing existing image processing software, such as MedX or Statistical Parametric Mapping, to fit the needs of our longitudinal research questions. The incumbent must be able to debug software modifications and participates in development of paradigms for validation and testing of new software.

Please respond to:

Dr. Susan Resnick
Laboratory of Personality and Cognition
NIH/NIA/GRC
5600 Nathan Shock Drive
Baltimore, MD 21224

E-mail: resnick@lpc.grc.nia.nih.gov

Address all Newsletter correspondence to:

Dr. B. L. Shapiro
The NMR Newsletter
966 Elsinore Court
Palo Alto, CA 94303.
650-493-5971* - *Please call only between 8:00 am and 10:00 pm, Pacific Coast time.*

Deadline Dates

No. 477 (Jun.)	22 May 1998
No. 478 (July)	26 June 1998
No. 479 (Aug.)	24 July 1998
No. 480 (Sept.)	21 Aug. 1998
No. 481 (Oct.)	25 Sept. 1998

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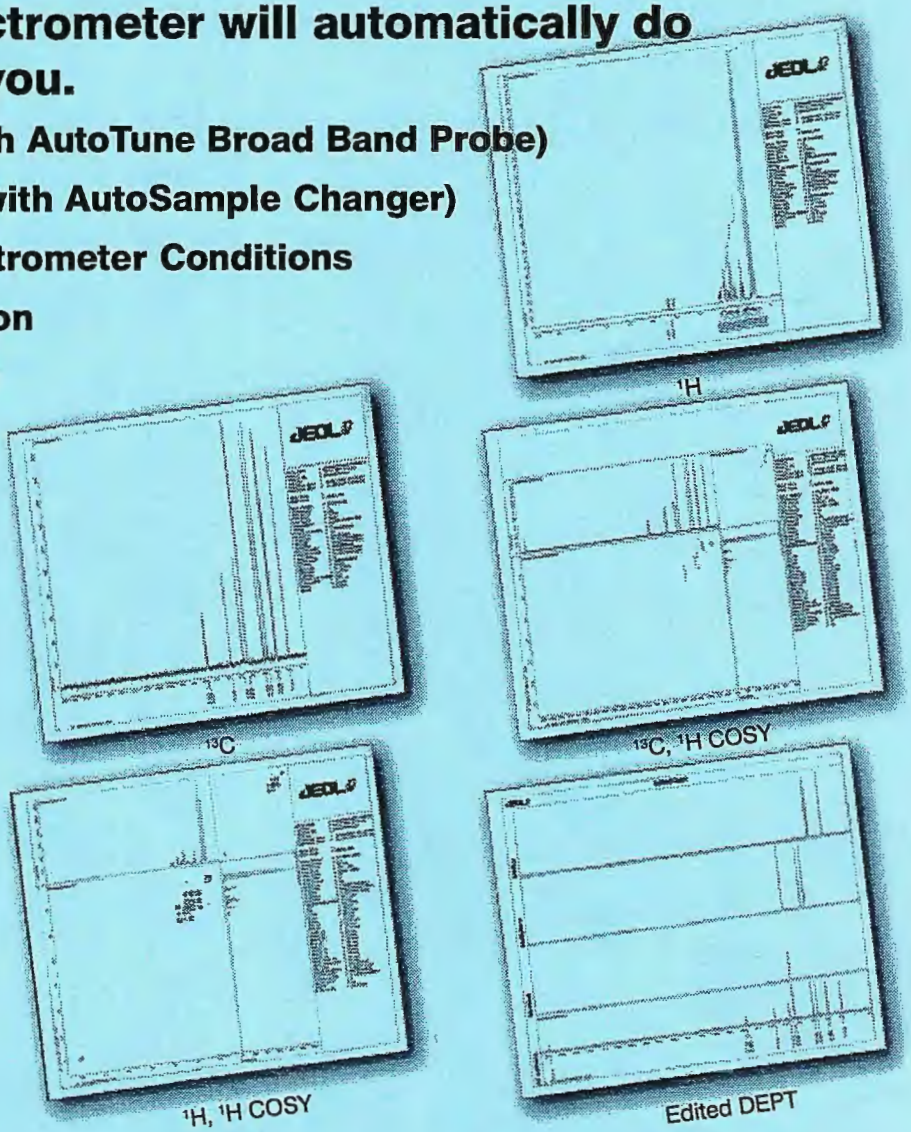

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