

Model for Non-Exponential Chemical Exchange	Kaplan, J. I.	2
High Field NMR of Solids	Baltusis, L.	5
NMR at the <i>Eastern Analytical Symposium</i> , November 13-14, 1990	Dalgarno, D. C., and Evans, C. A.	8
Sideband ELimination by Temporary Interruption of the Chemical Shift (SELTICS).	Harbison, G. S.	11
^1H Decoupler Offset Cycling Technique to Determine Trace Quantities of Hydride in Trialkylaluminum	Su, B.-M., and Marr, D. H.	15
Rhodium the Easy Way: Inverse ^{31}P - ^{103}Rh Correlation	Bigam, G.	19
TAMU NMR Newsletter: <u>Third Notice re 1990-91 Invoices and Change in Subscription Rates</u>	Shapiro, B. L.	20
Rapid Calculation of Powder NMR Line Shapes: Quadrupolar Nuclei	Power, W. P., and Wasylishen, R. E.	23
$^6\text{Li}/^{15}\text{N}$ Heteronuclear Correlation Spectroscopy	Harrison, A., Fuller, D., Gilchrist, J., and Collum, D.	27
Conformational Analysis of NADP ⁺ and NADPH Coenzymes	Ragg, E., Scaglioni, L., and Mondelli, R.	31
MORASS: A Computer Program for Multispin Overhauser Relaxation Analysis and Simulation	Meadows, R. P., Post, C. B., and Gorenstein, D. G.	35
2D Exchange Powder NMR on an MSL-300; Position Available	Inglefield, P. T., and Zhang, C.	39
Position Available	Van Duser, R.	42
^{31}P and ^{13}C NMR Studies on the Effects of EGF on Breast Cancer Cells	Kaplan, O., and Cohen, J. S.	43
Position Available	Evans, C. A.	44
Reconstructing Special Planes from 3D NMR Data by SDFT	Szalma, S., and Pelczer, I.	47
Isomers of 5-Hydroxy-3-cyclohexene- <i>cis</i> -1,2-dicarboximide	Bowler, D. J., Snyder, J. R., and Tseng, C. K.	51
Bernoulli Cartridge Drives for Aspect Computers	Wessel, T., Saunders, J. K., and Smith, I. C. P.	57
Savings and Short-Cuts for Setting Up a 600	Bubb, W. A.	58
A Geometrical View of Phase Cycling and Coherence Selection	Cottrell, C. E.	61
Neighborliness and Coolth in a Moved Imaging System	Szeverenyi, N. M.	65
Study of Exchange Mechanisms with Selective Pulses	Benesi, A. J., Rakiewicz, E. F., and Jackman, L. M.	66
Positions Available	Bolton, P. H.	68

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NO. 382, JULY 1990

AUTHOR INDEX

Baltusis, L. 5	Fuller, D. 27	Meadows, R. P. 35	Smith, I. C. P. 57
Benesi, A. J. 66	Gilchrist, J. 27	Mondelli, R. 31	Snyder, J. R. 51
Bigam, G. 19	Gorenstein, D. G. 35	Pelczar, I. 47	Su, B.-M. 15
Bolton, P. H. 68	Harbison, G. S. 11	Post, C. B. 35	Szalma, S. 47
Bowler, D. J. 51	Harrison, A. 27	Power, W. P. 23	Szeverenyi, N. M. 65
Bubb, W. A. 58	Inglefield, P. T. 39	Ragg, E. 31	Tseng, C. K. 51
Cohen, J. S. 43	Jackman, L. M. 66	Rakiewicz, E. F. 66	Van Duser, R. 42
Collum, D. 27	Kaplan, J. I. 2	Saunders, J. K. 57	Wasylishen, R. E. 23
Cottrell, C. E. 61	Kaplan, O. 43	Scaglioni, L. 31	Wessel, T. 57
Dalgarno, D. C. 8	Marr, D. H. 15	Shapiro, B. L. 20	Zhang, C. 39
Evans, C. A. 8, 44			

TEXAS A&M NMR NEWSLETTER

NO. 382, JULY 1990

ADVERTISER INDEX

American Microwave Technology, Inc. 33	Magnex Scientific 59
Bio-Rad, Sadtler Division 7	MR Resources, Inc. 21
Bruker Instruments, Inc. 29	Nalorac Cryogenics Corporation 17
Cambridge Isotope Laboratories 53	New Methods Research, Inc. 41
Doty Scientific, Inc. 25	Norell, Inc. 49
Fremont Magnetic Resonance 37	Polyflon Company 45
GE NMR Instruments 13, inside back cover	Otsuka Electronics (U.S.A.) Inc. 9
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FORTHCOMING NMR MEETINGS

Bat-Sheva Workshop on New Developments and Applications in NMR and ESR Spectroscopy, October 14-24, 1990, Israel; Contact: Dr. D. Goldfarb, The Weizmann Institute of Science, Rehovot, Israel. See Newsletter 377, 10.

Eastern Analytical Symposium, Garden State Convention Center, Somerset, NJ; NMR Symposia and Poster Sessions on Nov. 13 and 14, 1990; Contact D. C. Dalgarno or C. A. Evans, Schering-Plough Research, 60 Orange St., Bloomfield, NJ 07003; (201) 429-3957; FAX: (201) 429-3916.

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

Additional listings of meetings, etc., are invited.

All Newsletter Correspondence

Should Be Addressed To:

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

(415) 493-5971

DEADLINE DATES*

No. 384 (September)----- 17 August 1990

No. 385 (October)-----14 September 1990

No. 386 (November)-----12 October 1990

No. 387 (December)-----9 November 1990

*Please note that these deadline dates have been moved a bit forward from those previously in effect.



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May 17, 1990
(received 5/21/90)Dr. B. L. Shapiro
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto, CA 94303*Model for Non-Exponential Chemical Exchange*

Dear Dr. Shapiro:

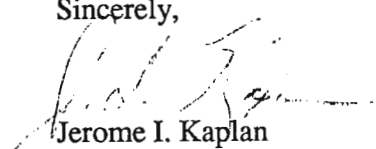
The standard chemical exchange model assumes exponential relaxation. When experimental data does not fit the standard model, one can try to fit to the experimental data by either postulating a distribution of sites with different relaxation times or a single site with non-exponential relaxation. A suggested form for the non-exponential relaxation is the so-called stretched exponential, $\exp[-t/\tau_0]^\alpha$, $\alpha \leq 1$, which can be well approximated by two exponentials with short and long relaxation times.

Al Garroway and I have made a comparison of theoretical lineshapes arising from these two models [J. Mag. Res. 49, 464, (82)]. The resulting lineshapes for these models look significantly different.

In this letter, I report on a model starting from a vibrational normal mode description which leads to non-exponential exchange. The critical concept is that molecules undergo a transformation from one structural form to another structural form via a subset of vibrational normal modes — called hopping modes. The other vibrational modes which do not lead to a change in structural form are called non-hopping modes (they are in the majority). After a molecule has made a transition to one of the forms via a hopping-hopping coupling, it can immediately make a transition back to its previous form as it is already in a hopping mode (short relaxation time) or it can make a transition to a non-hopping mode where it wanders about until it makes a transition back to a hopping mode (long relaxation time).

These ideas have been cast in a density matrix formulation and compound relaxation times and line shapes have been obtained. The manuscript has been submitted to J. Mag. Res. Please credit this letter to B. D. Nageswara Rao's account.

Sincerely,


Jerome I. Kaplan
Professor of Physics

JIK:mrp

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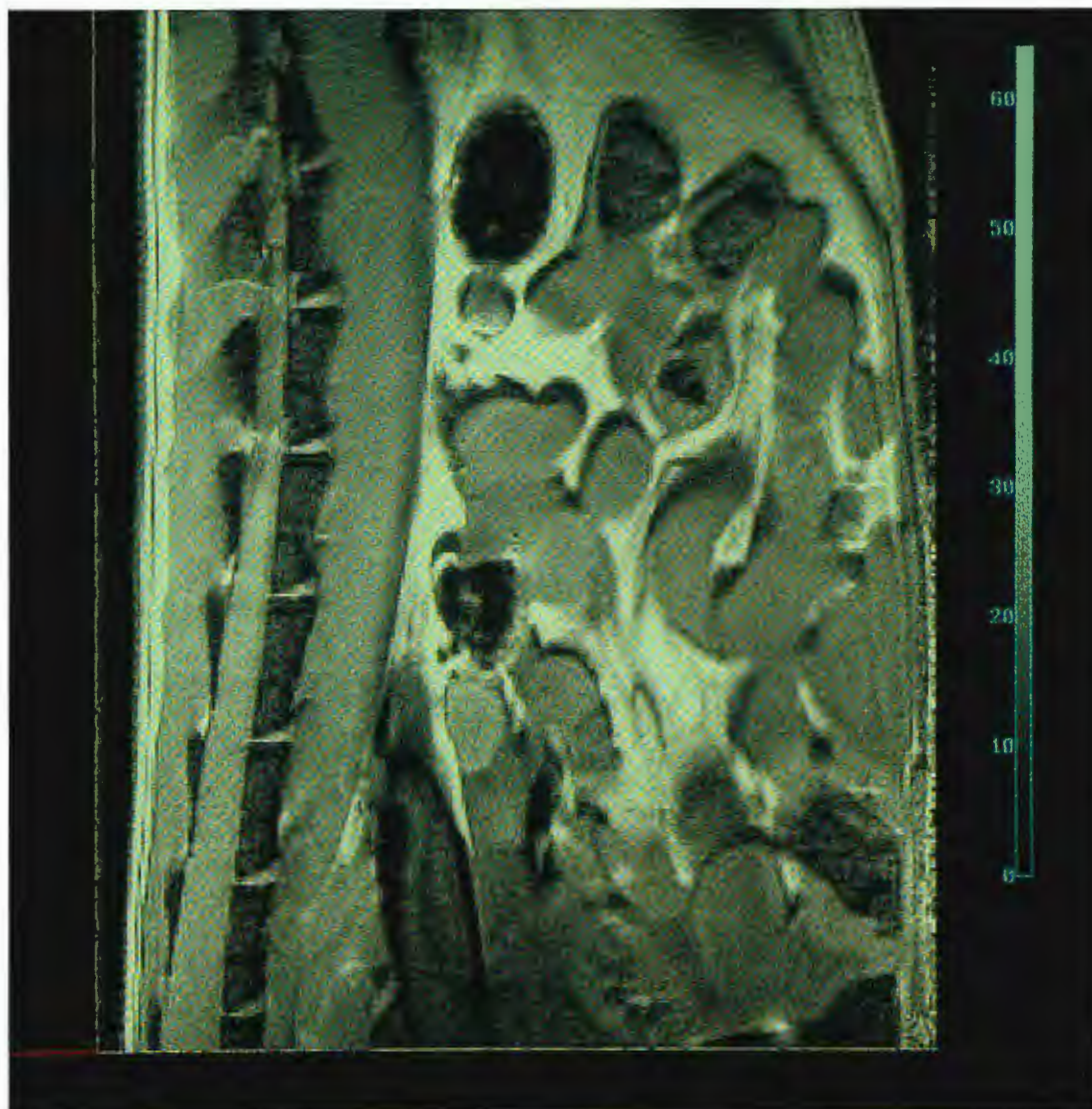
SAGITTAL VIEW OF AN ADULT MOUSE

Obtained on a Varian UNITY-300 NMR Spectrometer, this sagittal view of an adult mouse allows the spectroscopist to investigate the spine as well as other details of the mouse's anatomy using non-invasive microimaging techniques. In addition, the mouse can be studied over time to learn the physiological response to a stimulus.

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June 12, 1990
(received 6/13/90)Dr. Barry Shapiro, Editor
TAMU Newsletter
966 Elsinore Court
Palo Alto, CA 94303

Dear Barry,

The application of high field MAS has been explored by those interested in the study of quadrupolar nuclei with odd spin quantum numbers such as ^{27}Al (5/2), ^{23}Na (3/2), ^{17}O (5/2), ^{11}B (3/2), ^{51}V (7/2), etc. Spectra of these nuclei at lower magnetic fields can be rather complicated due to second order quadrupolar effects for the central $-1/2 \leftrightarrow 1/2$ transition. Since the second order quadrupolar effect is inversely proportional to the square of the magnetic field, it is often desirable to study chemical problems involving quadrupolar nuclei at the highest field strength possible (i.e. 11.7 T (500 MHz ^1H) and 14T (600 MHz ^1H). Other approaches such as double rotating probes and angle flipping probes have also been used to simplify the spectra of quadrupolar nuclei.

We have been investigating the benefits of high field nmr of solids. Attached are a few Unity MAS spectra using a Doty probe at 14 T for a variety of interesting sodium samples. Due to the requirement of high sample spinning speeds at high fields, both the 500 and 600 MAS probes use 5 mm rotors as sample containers. Samples were typically spun at between 8 and 13.5 KHz.

Figure 1A shows a 14 T ^{23}Na MAS spectrum of Na_2SO_4 . Na_2SO_4 is a classic example of a single site sodium used in many articles to illustrate the effect of line shape as a function of magnetic field (qcc (quadrupolar coupling constant) = 2.4 MHz, eta (asymmetry parameter) = 0.65).

Figure 1B shows a 14 T ^{23}Na MAS spectrum of NaMO_4 . This material contains at least two sodium sites with quite different qcc's and eta's. Figure 1C shows a spectrum of the same compound taken on 7 T (300 MHz ^1H) spectrometer. A comparison between Figure 1B and 1C demonstrates how second order effects alter the shape of the powder pattern in a field dependent manner.

Figure 1D shows a 14 T ^{23}Na MAS spectrum of NaVO_3 . As in the case of NaMO_4 , NaVO_3 is a material where there are multiple sodium sites. Due to the spread in chemical shift of the three kinds of species with different qcc's and eta's, it is easier to resolve by eye each of the components.

These spectra demonstrate some benefits of high field nmr of solids. Current and future users of Varian equipment may now have these benefits by adding the appropriate probe.

Sincerely,

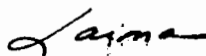
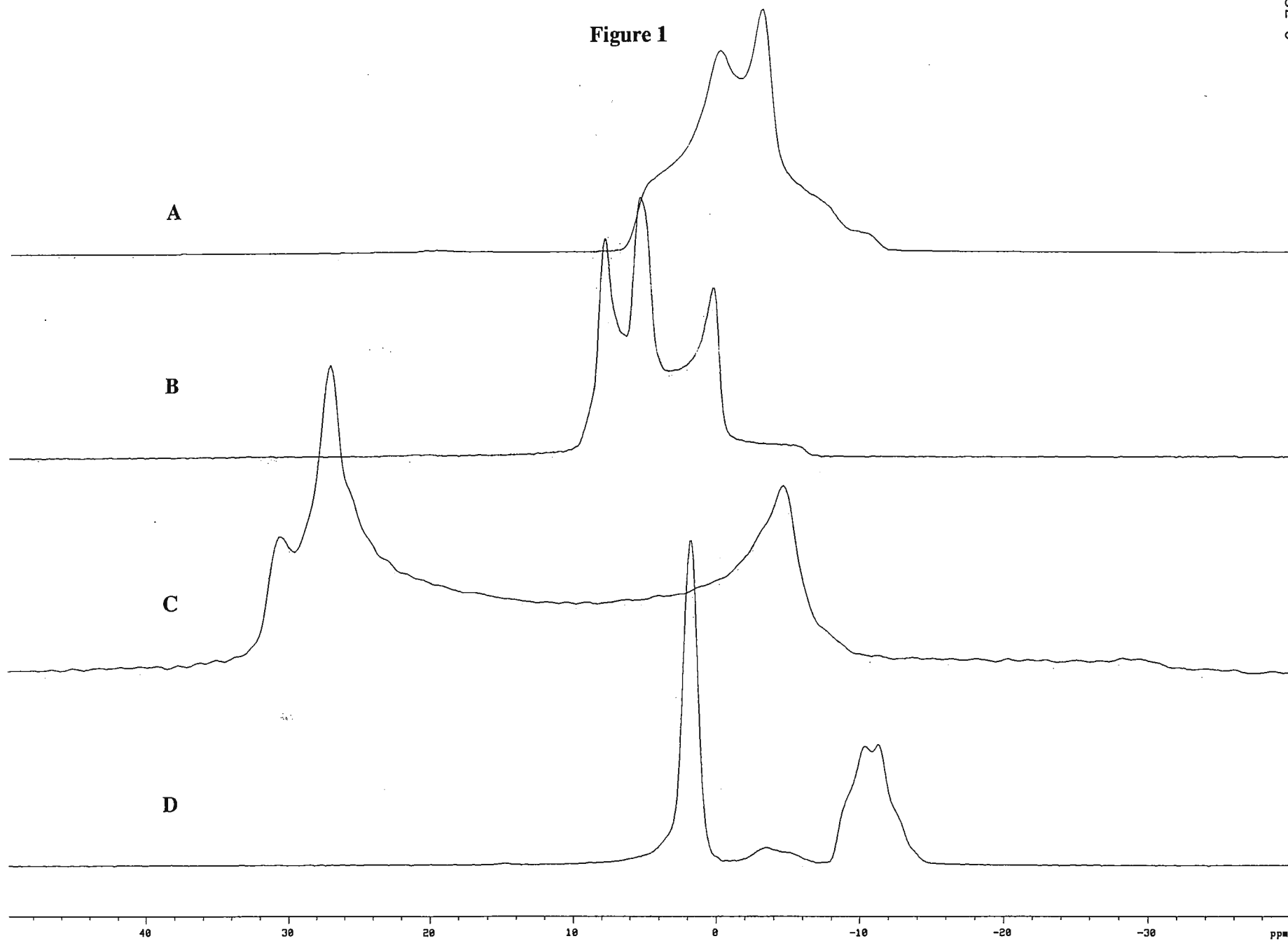
Laima Baltusis
NMR Applications Chemist

Figure 1

When faced with a tough analytical problem . . .

QUESTION

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

ANSWER

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CAS Registry Search
Exact Match Search
Substructure Search
SHUTDOWN.SHI
Source Structure
Number of Hits: 128

Index	NOI	Lib	Entry	Name
1	1	13C1	71	SULFIDOL, 10-ETHYL-PHENYL
2	1	13C1	73	PHENYL DISULFIDE
3	1	13C1	98	BENZENESULFONYL CHLORIDE
4	1	13C1	97	BENZENESULFONYL FLUORIDE
5	1	13C1	206	BENZENESULFONYL FLUORIDE, -
-6	1	13C1	227	P-TOLUENESULFONIC ACID, METHYL ESTER
7	1	13C1	353	P-TOLYL DISULFIDE
8	1	13C1	732	METHANOL, 2-TOLYLSULFONYL-,
9	1	13C1	982	BENZENESULFONIC ACID, HYDRATE
10	1	13C1	983	BENZENESULFONIC ACID, SODIUM SALT
11	1	13C1	987	P-TOLUENESULFONIC ACID, BUTYL ESTER
12	1	13C1	986	BENZENESULFONAMIDE
13	1	13C1	918	P-TOLUENESULFONAMIDE, N-BUTYL-,
14	1	13C1	974	ACETIC ACID, 2-PHENYLTHIO-,
15	1	13C1	997	P-TOLUENESULFONYL CHLORIDE
16	1	13C1	1637	P-TOLUENESULFONAMIDE, N-METHYL-,
17	1	13C1	1359	ANILINE, O-TETRAHYDRO-,
18	1	13C1	1179	SULFIDE, PHENYL 3-PHENYLPROPYL,
19	1	13C1	1296	SULFIDE, P-BROMOPHENYL METHYL,
20	1	13C1	1393	SULFIDE, METHYL N-NITROPHENYL,

STEP 2:

Match Those Derivatives
With Your Spectrum.

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(Total of peaks: 5)
No. Location Intensity
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3 126.70 9
4 128.70 9
5 138.60 1

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

June 6, 1990
(received 6/13/90)

Dear Barry:

The Eastern Analytical Symposium, newly relocated to the Garden State Convention Center in Somerset, NJ, has planned an exciting program in NMR this Fall. We would like to invite TAMU NMR readers to contribute to this program by participating in one or two NMR poster sessions associated with the NMR Symposium. The poster presentations will take place on the mornings of November 13 or 14, 1990. Research and analytical papers from all areas and applications of NMR are welcome.

Please note that each of these poster sessions precede powerful afternoon symposia: Tuesday (Nov. 13) afternoon is the "Symposium on Medical Magnetic Resonance" chaired by Ted Becker and Wednesday is the session entitled "3D and Other Novel Approaches in Protein NMR Spectroscopy" chaired by Angela Gronenborn.

Because of EAS publicity requirements we are asking you please to respond to us as soon as possible. We are facing a deadline of late August for getting abstracts into the final program. If your readers would like to make a presentation but will not be able to meet this deadline, we are still very interested in having their presentation at the symposium. Missing this deadline simply means that your abstract will not be included in the final program. We plan to hand out a complete set of abstracts to attendees of the poster sessions.

Whether or not your readers contribute a paper to the poster sessions, we hope that they will be able to come to the EAS NMR symposium. We look forward to seeing you there, also.

Sincerely,

David C. Dalgarno

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Preamplifier	Broadband
Noise Figure	< 2dB

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Output Power	Computer Controlled: 12 Bit DAC
Power Control	0 - 100 %
Linearity of Power Control	±1%
RF Amplifier Power Output	100W CW

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Field (T)/Bore (cm)	2/31	2/30.5	2.4/40	4.7/20	4.7/31	4.7/40	7/20
Bore With RT Shims And Gradients (cm)	26.5	26	33	15	26.5	31	15
Helium Evaporation (ml/hr)	50	50	50	50	55	60	50
Nitrogen Evaporation (ml/hr)	400	400	400	400	450	500	400
Half Length (mm)	350	275	570	396	460	735	575
5 Gauss Line — Radial From Center (m)	3.4	3.2	4.7	3.7	5.3	6.4	5.1
— Axial From Center (m)	4.4	4.0	5.9	4.7	6.7	8.1	6.4

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17 May 1990 (received 5/21/90)

Dr. Barry L. Shapiro
Editor, TAMU Newsletter
California

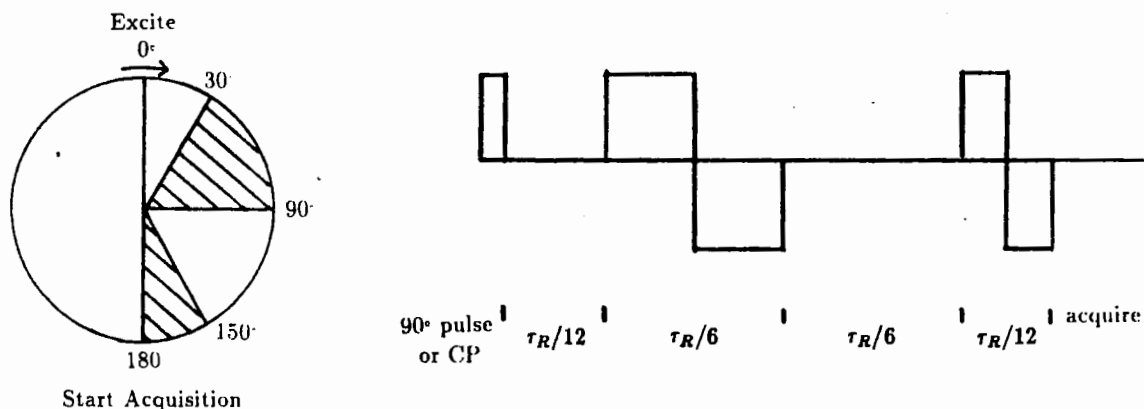
Sideband ELimination by Temporary Interruption of the Chemical Shift (SELTICS)

Dear Barry:

Those of us who do magic-angle spinning NMR usually adhere to one of three philosophical schools when it comes to rotational sidebands. These are the Stoics, who regard sidebands as a necessary evil about which comparatively little can be done; the Utilitarians, who believe sidebands are a useful source of information about shielding tensor magnitudes and orientations; and the Reductionists or Nihilists (depending on how well the sequence works), who aim at their total suppression. I have until recently been a devout Utilitarian; I'd like to use the newsletter as a public forum to confess to a brief lapse into Nihilism. This heresy, like most such, had its positive side; it generated a nice acronym, and incidentally a pulse sequence which seems to remove sidebands more efficiently than any previously proposed.

The SELTICS experiment, like all good ones, is in fact an infinite set of pulse sequences, although in our experience the simplest one works the best. It aims to accomplish what Dixon's TOSS previously set out to do — prepare a state of the magnetization where the signals from individual crystallites are aligned in such a way that the part of the magnetization that oscillates at $N\omega_R$ cancels out over the powder average, leaving only the centerband. The way this aligned state is prepared is however quite different. To reach nihilist nirvana (or reductionist rapture?), TOSS uses four π pulses arranged with fairly esoteric timings over somewhat more than 2 rotor cycles. Since π pulses are poorly behaved entities, concatenating four of them, along with a fairly lengthy time during which normal relaxation operates, results (in our undoubtedly incompetent hands) in poor suppression of sidebands and significant loss of centerband intensity.

Rather than attempting to align the magnetization vectors actively with π pulses, one can produce the same state by simply turning off evolution of the anisotropic part of the chemical shift Hamiltonian for a couple of precisely timed intervals in the rotor cycle. Without belaboring the theory here, the effect one aims at is to turn the powder integral over the Euler angle γ , which ordinarily looks like a modified Bessel function with frequency components at $N\omega_R$, into a delta function at zero frequency. The simplest sequence which accomplishes this is shown in Fig. 1 below, where the rotor cycle is represented as a circle, and the periods during which chemical shift evolution is suspended are hatched areas. The sequence, as can be seen, is $\tau_R/2$ long, less than $1/4$ of the shortest possible TOSS sequence.



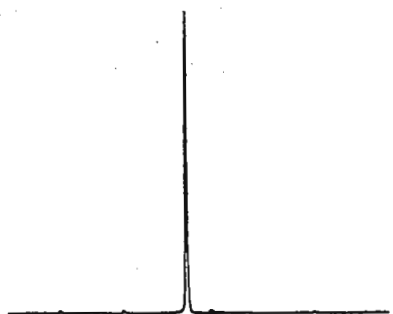
How does one turn off the chemical shift? The simplest method is to spin-lock the magnetization with a strong r.f. field; the spin locking intervals should be divided into two periods during which r.f. pulses of equal amplitude and opposite phase are applied; this ensures that the magnetization really is in an identical position in the complex plane after the spin-locking as it is before. This scheme is illustrated in Fig. 2. The power levels of either the spin-lock or the decouple channel should be Hartmann-Hahn mismatched to avoid losing signal due to back-cross-polarization. If it is possible to do so, it also helps to have the length of the spin-locking intervals an integer multiple of a 4π pulse, since a $2\pi - 2\pi$ sequence compensates for r.f. offset effects to second order as well as first order. This is merely a refinement; the sequence works quite adequately without it. Finally, the phase of the spin-locking pulses is completely arbitrary, and so they can be cycled relative to the CP with complete freedom.

The problem of finding pulse combinations which do nothing has of course confronted NMR spectroscopists before, most recently in implementing TOCSY-type sequences for observing rotating frame NOEs. It is interesting to note that in our hands none of the obvious short sequences used for such purposes (e.g. MLEV-4) performs as well as combinations of 2π pulses. We speculate that such sequences compensate more poorly than conventional spin-locking for *time dependent* r.f. offset, such as one has in MAS.

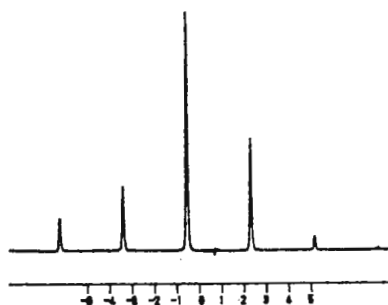
Finally, just to demonstrate that the elimination thing actually works, below we show, plotted to scale, a couple of examples of SELTICS spectra compared with conventional CP-MAS. These show the excellent sideband suppression and significant centerband enhancements obtained with the new sequence. A more detailed treatment, including sequences which refocus the isotropic shift, is available from the author.

Please credit this to Charlie Springer's account, and send the backnumbers that through my negligence we've been missing.

Diammonium Hydrogen Phosphate, sideband suppressed. 2.64 KHz

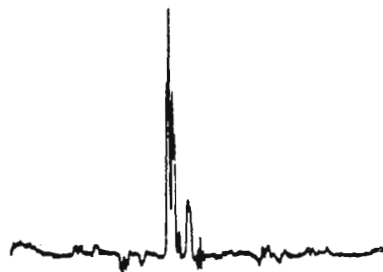


Diammonium Hydrogen Phosphate, CP-MAS. 2.64 KHz

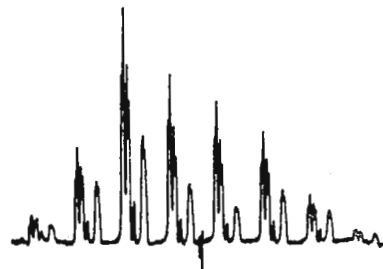


FREQUENCY (kHz)

ATP, sideband suppressed. 3.69 KHz



ATP, CP-MAS 3.69 KHz



FREQUENCY (kHz)

Sincerely,

Gerard S. Harbison, Part-time Nihilist.

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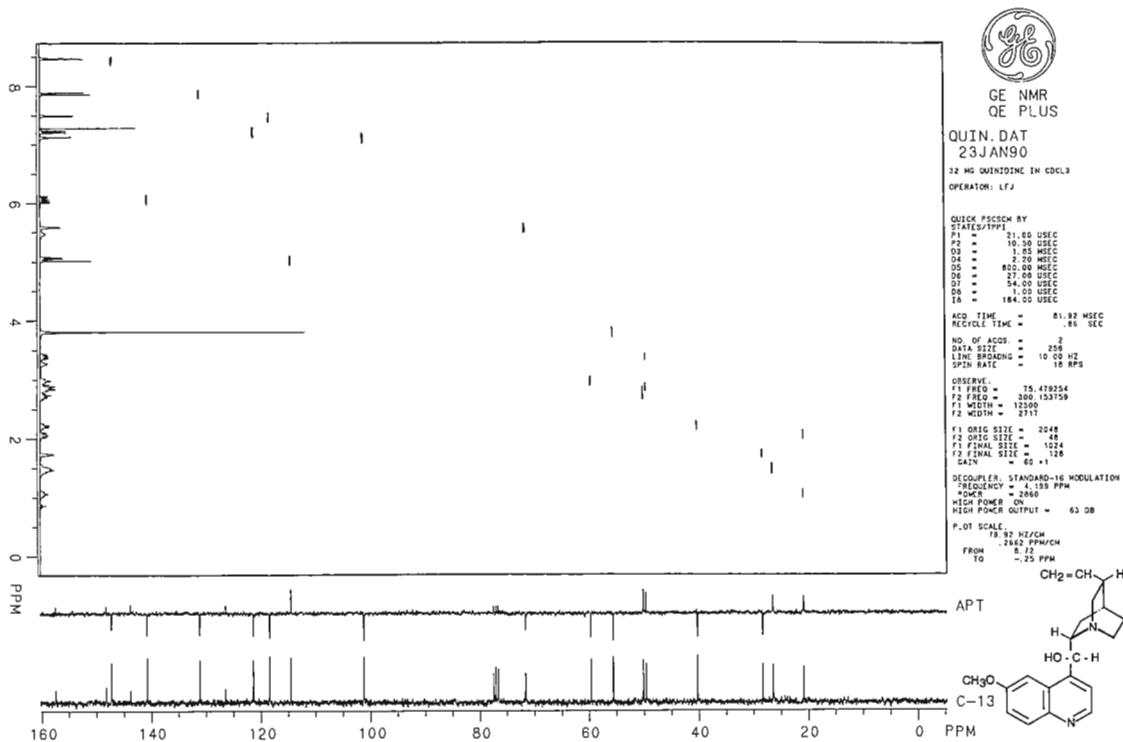
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Chemical Division

May 18, 1990
(received 6/16/90)

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

Title: *¹H NMR Decoupler Offset Cycling Technique to Determine
Trace Quantities of Hydride in Trialkylaluminum*

Dear Barry:

Thanks for your reminder and herewith is our attempt to stave off a more vigorous one.

A quantitative ¹H NMR decoupler offset cycling (DOCYCL)⁽¹⁾ technique at elevated sample temperature, 55 ° or 60 ° C, has been developed to determine trace levels (5 to 30 ppm) of hydride in trialkylaluminum samples. To avoid dynamic range problems, the DOCYCL technique was used to suppress the strong peaks from alkyl groups and solvent (i.e., hexane). At elevated temperature, hydride peaks from monomer and dimer will coalesce to form a single peak thereby improving the detection limit at low hydride levels. To avoid further dilution of the sample with deuterated solvent, only the reference compound (toluene) was added to the hexane solutions of trialkyl aluminum. Therefore all the experiments were run without field/frequency lock. The NMR samples were prepared in a nitrogen box and then sealed.

Figure 1 shows a normal ¹H NMR spectrum of a concentrated diethylaluminum hydride in hexane. The hydride region shows two signals. Figure 2 shows a general DOCYCL ¹H NMR spectrum of a triethylaluminum/hexane sample doped with a known amount of toluene. To avoid the NOE effect, methylene protons of the ethyl group were not irradiated. Figure 3 is the expanded spectrum of the hydride and toluene regions. With a known amount of toluene added to the sample and the ¹H NMR area, one can determine the % hydride present. The unsaturation present arises from decomposed alkyl group.

Sincerely,

Bing-Ming Su, David H. Marr

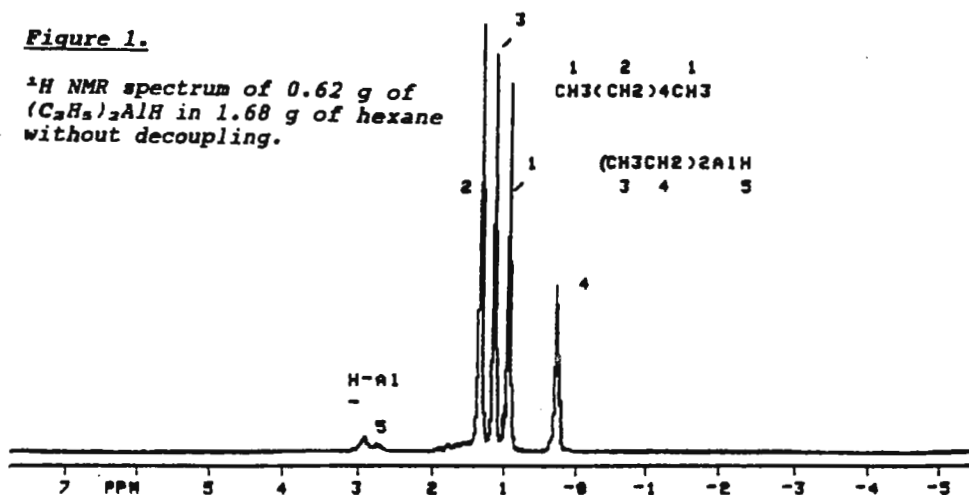
(1) M. Kinns, J.K.M. Sanders, J. Magn. Reson. 56, 518-520 (1984)



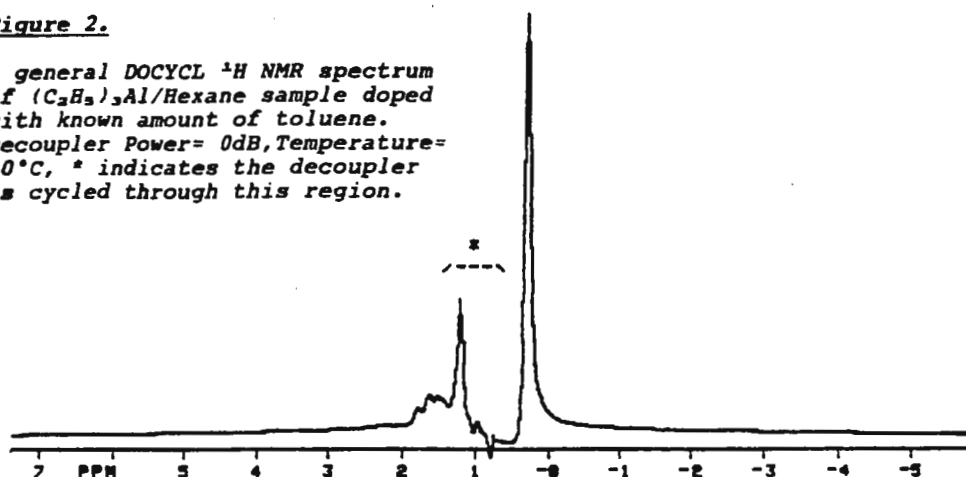
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Figure 1.

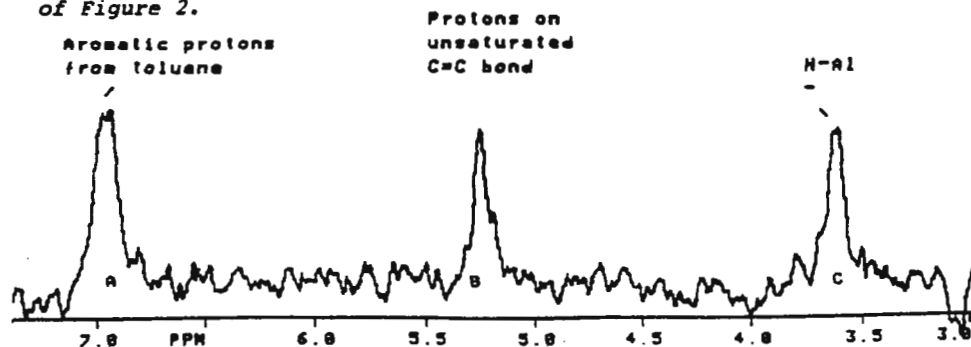
^1H NMR spectrum of 0.62 g of $(\text{C}_2\text{H}_5)_2\text{AlH}$ in 1.68 g of hexane without decoupling.

**Figure 2.**

A general DOCYCL ^1H NMR spectrum of $(\text{C}_2\text{H}_5)_2\text{Al}$ /Hexane sample doped with known amount of toluene. Decoupler Power= 0dB, Temperature= 60°C , * indicates the decoupler is cycled through this region.

**Figure 3.**

Expanded ^1H NMR down-field region of Figure 2.



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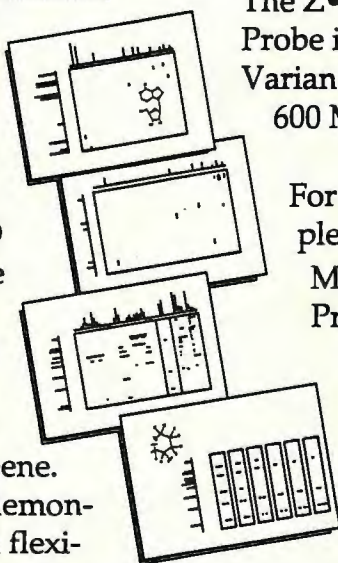
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For more information, please contact Toby Zens, Manager of the Z•Spec Products Group.



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Z·SPEC Indirect Detection Spectra

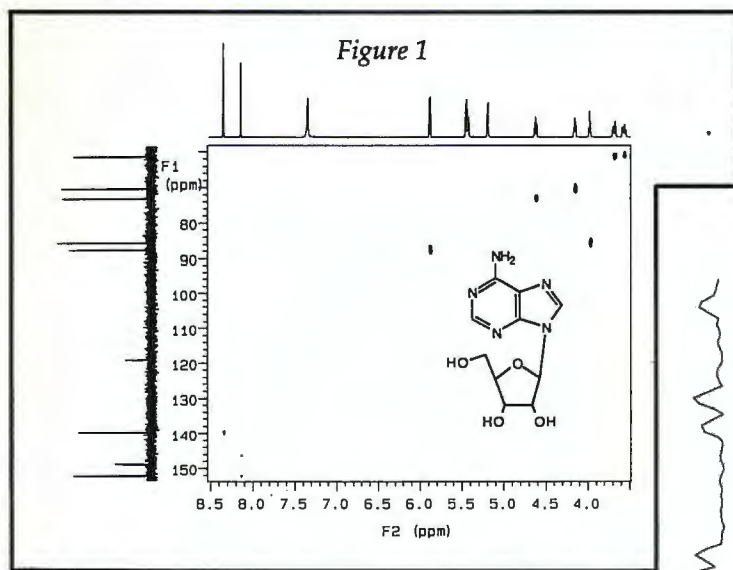


Figure 1: Z·Spec ID probe, HMQC experiment run on a 1 mg/ml DMSO_{d6} solution of Adenosine. Full chemical shift region, PW90=10 μ sec ^{13}C , 8 transients/FID, total time 19 minutes.

Figure 2: HMQC Ribose region Adenosine, 125 $\mu\text{g}/\text{ml}$. Total acquisition time 2.2 hours, nt = 224.

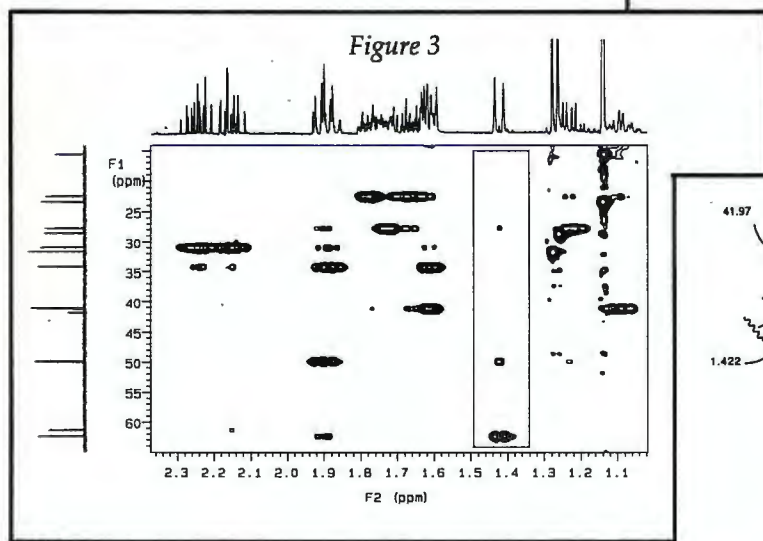
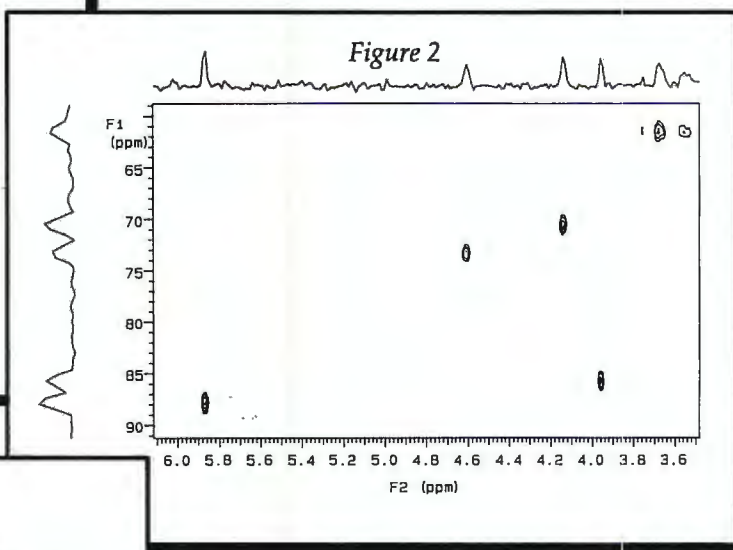
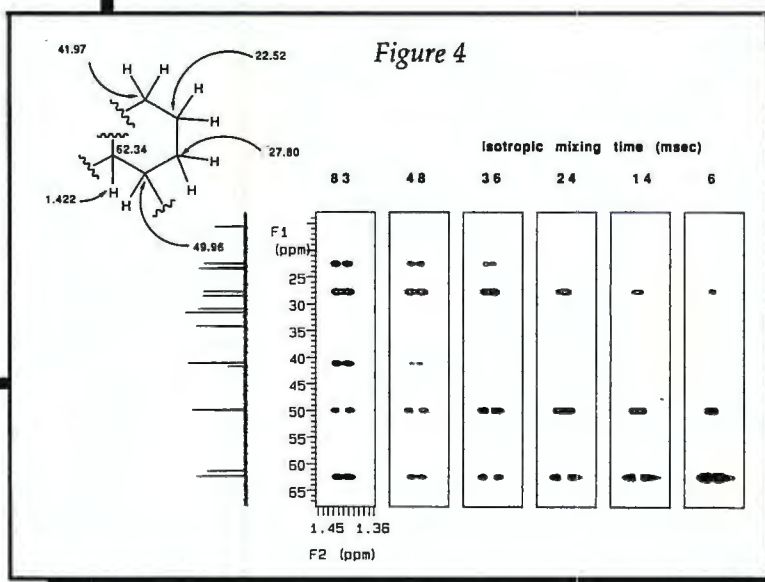


Figure 3: HMQC/TOCSY experiment run on a 8 mg/ml solution of a Marine Sesquiterpene decomposition product. Total acquisition time 2 hours, 10 minutes.

Figure 4: Expansions at the 1.4 ppm proton resonance showing the propagation of magnetization to the carbons with increasing mixing time. Note the excellent F1 phase characteristics throughout.



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June 6, 1990
(received 6/14/90)

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

Rhodium the easy way: Inverse ^{31}P - ^{103}Rh Correlation

Dear Barry:

Several groups in our Department are engaged in research on transition metal complexes which frequently contain ^{103}Rh and ligands containing ^{31}P . Since the oxidation state of ^{103}Rh is a sometimes controversial subject, it has long been a desire to measure ^{103}Rh chemical shifts, which have characteristic ranges for different states.

Because the receptivity of ^{103}Rh is so low, we elected to observe Rhodium with the 4-pulse inverse ^{31}P - ^{103}Rh correlation experiment which is at least 1300 times more sensitive than direct detection and is governed by shorter ^{31}P T_1 's.

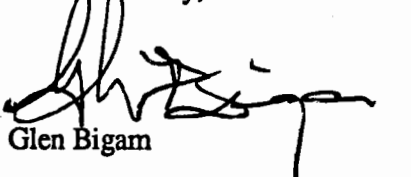
With the experiment defined, all we had to do was to build our own probe (^{31}P , ^{103}Rh , ^1H , ^2H) for the WH-200 and scavenge various bits and pieces from a retired WH-400 console. After about six months of "This is never going to work!", we now have an experiment which is ridiculously easy to perform. A 0.01 M solution in a 5 mm NMR tube yields a rhodium projection with a 20/1 S/N ratio in 55 minutes. Indeed, for most samples to date, the acquisition time is dictated by the length of the phase cycle rather than by S/N.

Even though $(\gamma B_1)_{\text{Rh}}$ is rather small (2800 Hz, $\tau_{90} = 90 \mu\text{s}$), the experiment is very tolerant. Setting ω_{Rh} 15,000 Hz off-resonance produced useable signals (although a null was observed at $\Delta\omega \approx 10,000$ Hz). Similarly, mis-setting $\pi/2_{\text{Rh}}$ to $\pi/4$ reduced the S/N ratio, but only by about 50%.

Our probe accommodates 10 mm NMR tubes, but we use 5 mm tubes exclusively, so far. A 5 mm probe should have even better S/N and a much shorter $\pi/2_{\text{Rh}}$ pulse. We strongly recommend the smaller insert to anyone contemplating this most useful technique.

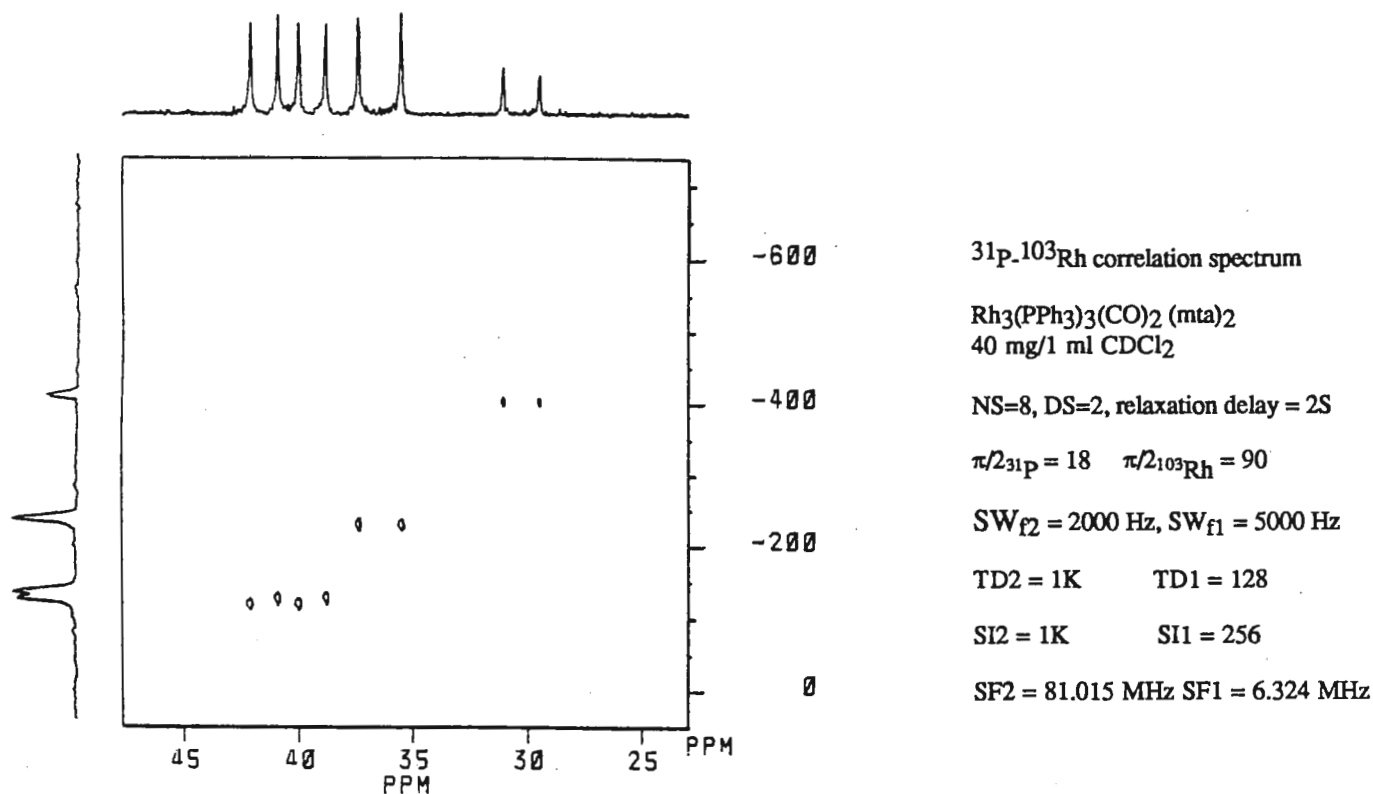
Please credit this contribution to Tom Nakashima's account.

Yours sincerely,



Glen Bigam

GB:lf



Third (and Final Version) Notice re 1990-91 Invoices and Change in Subscription Rates

Subscription renewal invoices for the October 1990 - September 1991 year were mailed out on 22 June. If you ought to receive such an invoice, and do not have it in your hands by the time you read this, please call or write me promptly. Payment of these invoices must be received by me no later than September 5, 1990 to ensure uninterrupted mailing of the Newsletter issues. Please do not delay execution of any necessary paperwork! Also, please be sure that the instructions on the invoice are followed precisely.

You will have noted that it has become necessary to make a modest increase in the subscription rate. This is the first such increase in three years. I regret that inflationary pressures in general, and a substantial incipient increase in the postage rates in particular, make the subscription rate increase necessary. Advertising rates will also increase modestly, and our loyal Sponsors will be asked to increase their contributions. The surcharge for Air Mail Printed Matter mailing will also need to be incremented.

Thank you for your understanding and cooperation.

B. L. Shapiro
1 July 1990



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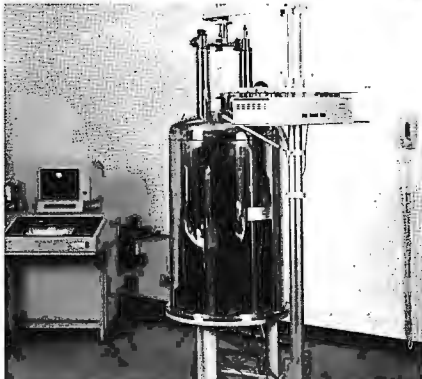
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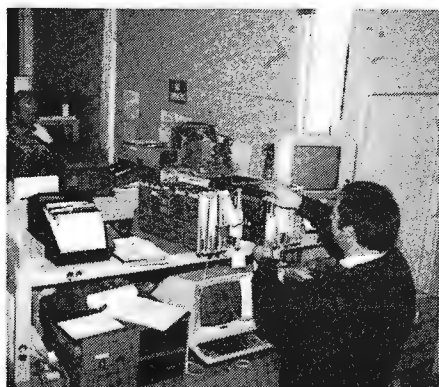
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June 6, 1990
(received 6/14/90)

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

Rapid Calculation of Powder NMR Line Shapes:
Quadrupolar Nuclei

Dear Barry,

Recently, there has been considerable interest and reinvestigation of nmr powder line shapes resulting from quadrupolar nuclei, of which the most significant result has been the study of nuclei whose efg tensors and chemical shielding tensors are not coincident.¹ One of the prime reasons why such considerations have not been attempted up to now is the incredible computational demands required for simulation of such spectra. There are 8 parameters that must be described, namely the magnitude and asymmetry of the efg tensor, the three principal components of the chemical shielding tensor, and the three Euler angles denoting the relative orientations of these two interactions.

Our research in this area has led us to develop two FORTRAN-77 programs incorporating the POWDER interpolation routine of Alderman, Solum and Grant,² which can be performed quickly on desktop microcomputers. One program simulates 1st order quadrupolar - chemical shielding line shapes, such as that shown in fig. 1. This simulation of the 7 transitions of a ¹³³Cs nmr powder spectrum required 3 min. 15 s. on a 286 (PC-AT) system, and only 52 s. on a 386 system, both of which contained math coprocessor chips. The simulation of the central transition of a 2nd order quadrupolar - chemical shielding line shape, shown in fig. 2, required only 60 s. on the 286 and 16 s. on the 386 computers. This second program can also calculate full powder patterns (i.e., all transitions) of a second-order quadrupolar - chemical shielding line shape. Both of these programs are also capable of calculating multi-site spectra. Developments such as these can be expected to stimulate increased activity in this area of research.

Bill

Bill Power

Rod

Rod Wasylishen

-
- ¹ (a) W.P. Power, R.E. Wasylishen, S. Mooibroek, B.A. Pettitt and W. Danchura, J. Phys. Chem., 1990, 94, 591.
(b) J.T. Cheng, J.C. Edwards and P.D. Ellis, J. Phys. Chem., 1990, 94, 553.
(c) P.J. Chu and B.C. Gerstein, J. Chem. Phys., 1989, 91, 2081.

² D.W. Alderman, M.S. Solum and D.M. Grant, J. Chem. Phys., 1986, 84, 3717.

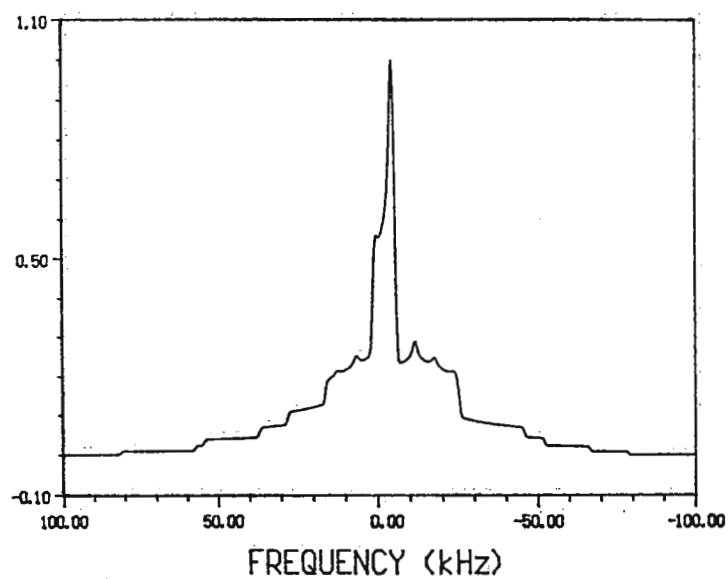


Figure 1.

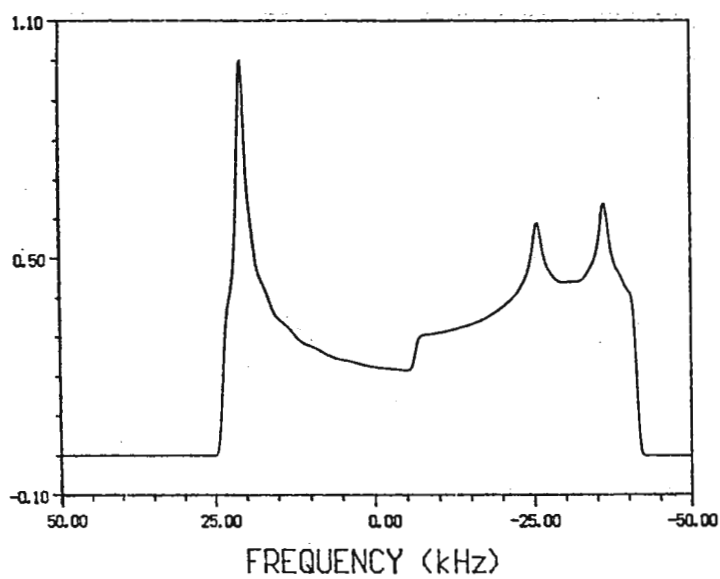


Figure 2.

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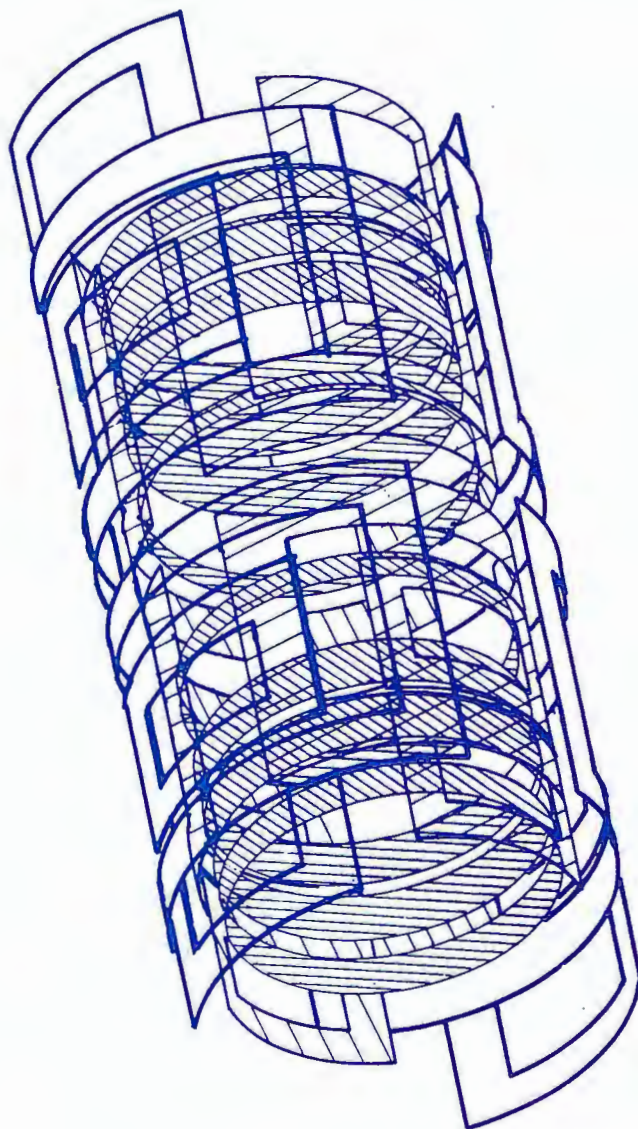
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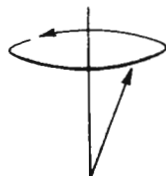
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Cornell University

Department of Chemistry
Baker Laboratory
Ithaca, New York 14853-1301 USA

May 26, 1990
(received 5/29/90)

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

Dear Barry,

Before describing some of our recent work on the NMR spectroscopy of organolithium compounds, we are pleased to announce that Dr. David Zax will be joining us at Cornell as an Assistant Professor of Chemistry. Dr. Zax is a specialist in solid state NMR spectroscopy and is likely to be a major contributor to the news letter in the future.

$^6\text{Li}/^{15}\text{N}$ Heteronuclear Correlation Spectroscopy

In our last Letter (TAMU 373/41) we described the application of single frequency decoupling of ^{15}N from ^6Li for the determination of structures of N-lithiated species. We now wish to report that the corresponding $^6\text{Li}/^{15}\text{N}$ inverse heteronuclear correlation spectra (HETCOR) can be acquired, allowing us to observe resonance correlations and deconvolute overlapping multiplets.

In our initial experiments, we have used a 4 pulse inverse detection sequence on a Bruker AC300 without decoupling during acquisition. We observe ^6Li (spectral width approximately 300 Hz) with 64 increments at an acquisition time of approximately 0.5 sec and a relaxation delay of 10 s. The overall acquisition time is approximately 1.5 hr. Figure 1 shows the HETCOR spectrum of $[\text{}^6\text{Li}, ^{15}\text{N}]$ -lithium 2,2,6,6-tetramethylpiperidide/ $[\text{}^6\text{Li}]$ -LiCl mixed aggregates. One of two resonances of a minor mixed aggregate that is just barely resolved in a high resolution one dimensional spectrum is readily observed in the HETCOR spectrum. (The second resonance fails to resolve from the baseline in both one and two dimensional spectra at these concentrations.)

We would like to thank Jim Simms of MIT and Brian Andrew of Brüker for several very helpful discussions.

Sincerely, -

Aidan Harrison

David Fuller

James Gilchrist

David Collum

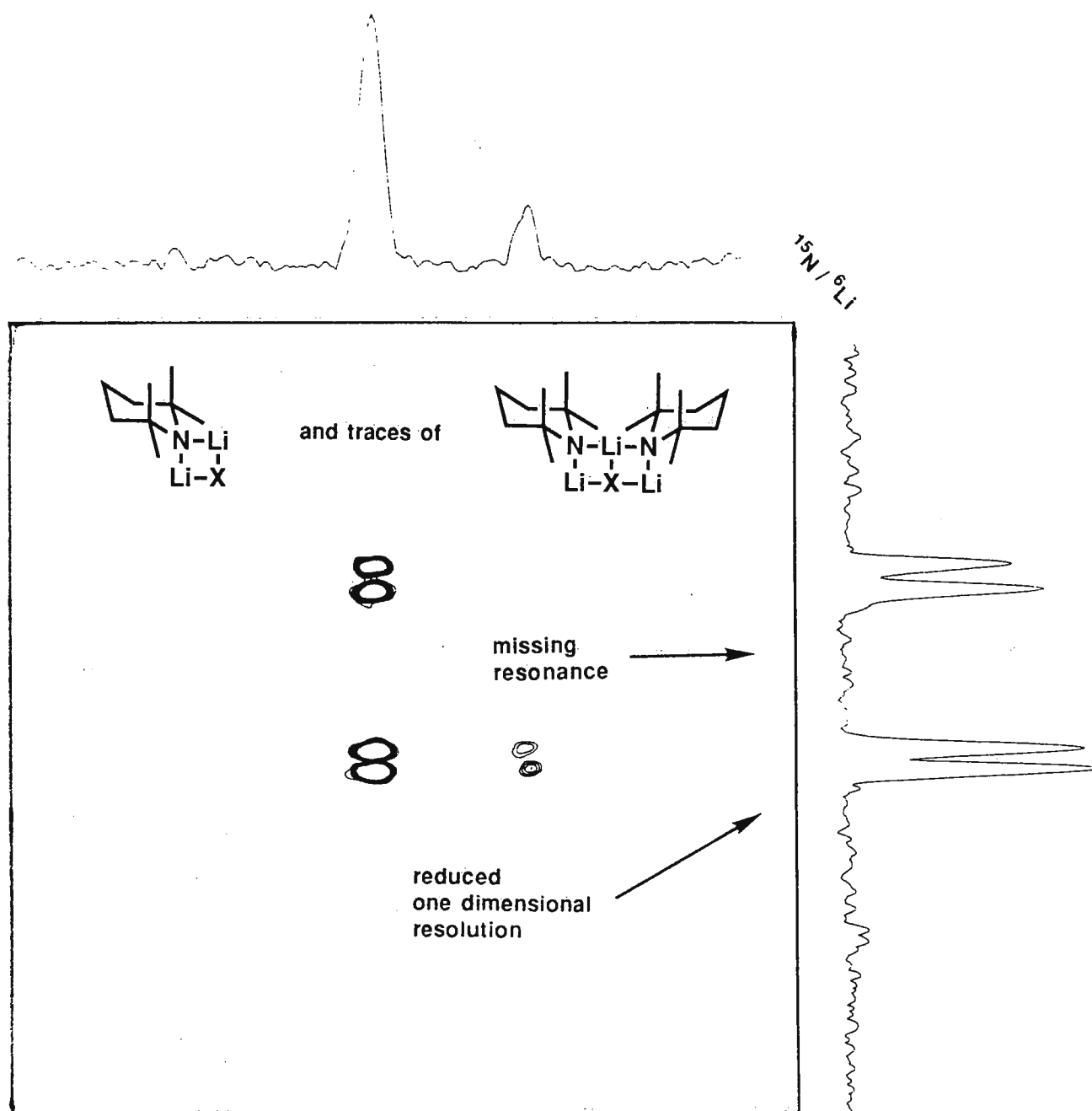
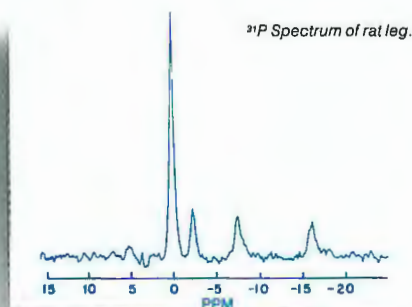


Figure 1. HETCOR spectrum of a solution of [^6Li , ^{15}N]-lithium 2,2,6,6-tetramethylpiperidide/ ^6Li -LiCl (0.10 M, 1.1:1.0 ratio) recorded at -94°C on a Bruker AC300 spectrometer operating at ^6Li and ^{15}N frequencies of 44.19 and 30.42 MHz, respectively.

Real Answers.

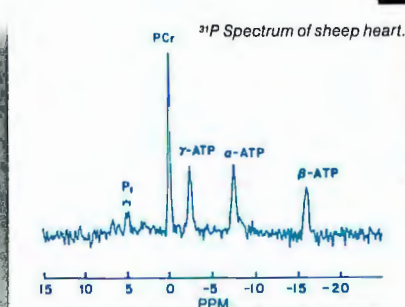
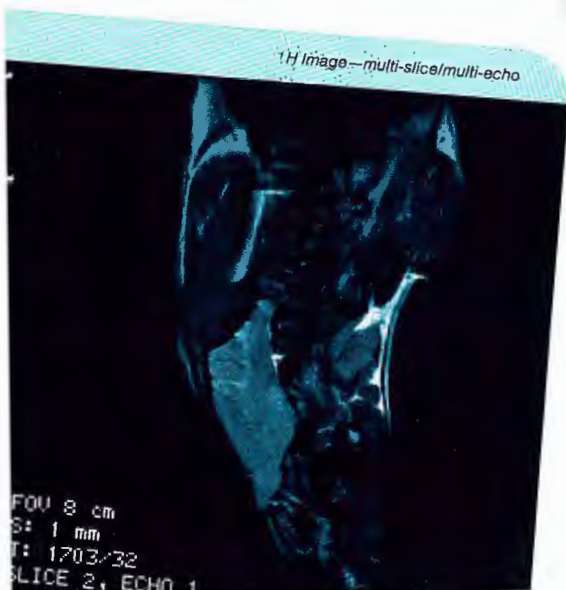


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Multi-slice/Multi-echo.

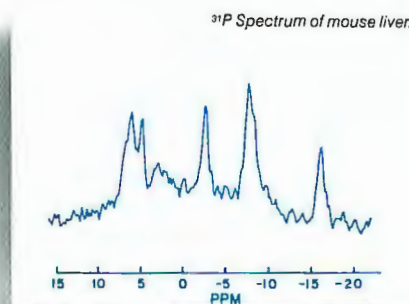
High quality image detail. An MSME image (256 x 256) taken of a rat head (TE = 33 msec, TR = 1700 msec). The slice thickness is 1mm and FOV is 8 cm. Details of the cerebellum, pharynx and the mystacial pads can be clearly discerned.



(Courtesy of Pittsburgh NMR Center)

Large bore.

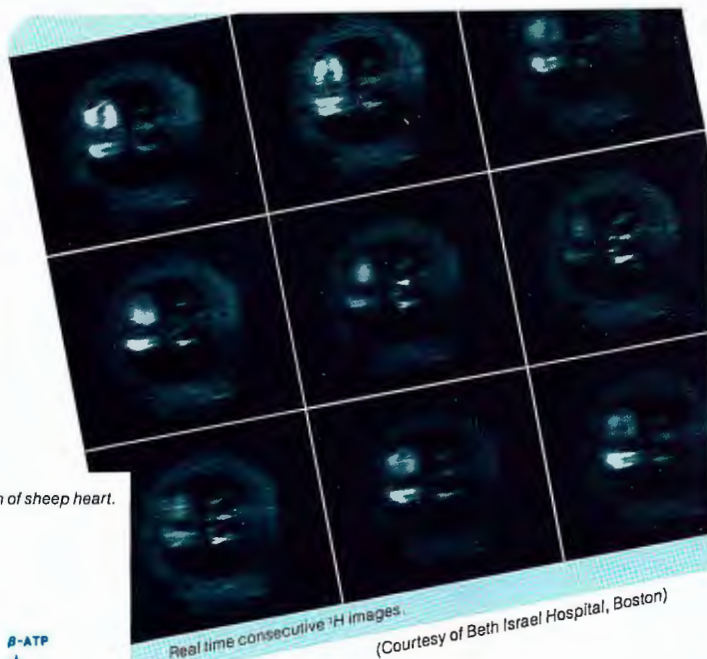
Wide access for large animal studies. ³¹P NMR spectrum obtained at 81 MHz from heart muscle in a live sheep (64 x 90° pulses at 2 sec. intervals) using a circular surface coil of diameter 35mm.



(Courtesy of Pittsburgh NMR Center)

In-vivo 7.0 tesla.

Horizontal-axis magnet systems at 7.0T. ³¹P NMR spectrum obtained at 121 MHz from mouse liver (128 x 90° pulses at 5 sec. intervals) using a circular surface coil of diameter 10mm.



Real time consecutive ¹H images.
(Courtesy of Beth Israel Hospital, Boston)

Real time.

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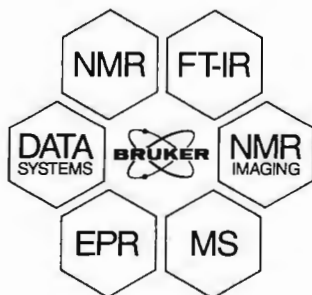
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Prof. B.L. SHAPIRO
Editor TAMU NMR Newsletter
966 Elsinore Court, Palo Alto
CA 94303 U.S.A.

May 25, 1990
(received 5/31/90)

**NMR STUDIES AND CONFORMATIONAL ANALYSIS
OF NADP⁺ AND NADPH COENZYMES**

Dear Barry,

I hoped to arrive before receiving your first pink letter, sorry I did not!

During an investigation on the products obtained from a one-electron reduction of the coenzyme nicotinamide adenine dinucleotide phosphate (NADP⁺), we needed to analyse the ³¹P NMR spectra of this coenzyme in the oxidized (NADP⁺) and reduced (NADPH) forms. We found a great confusion in the literature about the NMR data, especially for the H,H and P,H coupling constants; in particular the interactions of the ribose protons H-4', H-5' and H-5'' with the phosphorus atoms of the pyrophosphate chain have never been measured. We obtained all the H,H, P,H and the most important of the P,C coupling constants for both NADP⁺ and NADPH, by using 1D spectra (2D spectroscopy was used for the assignments).

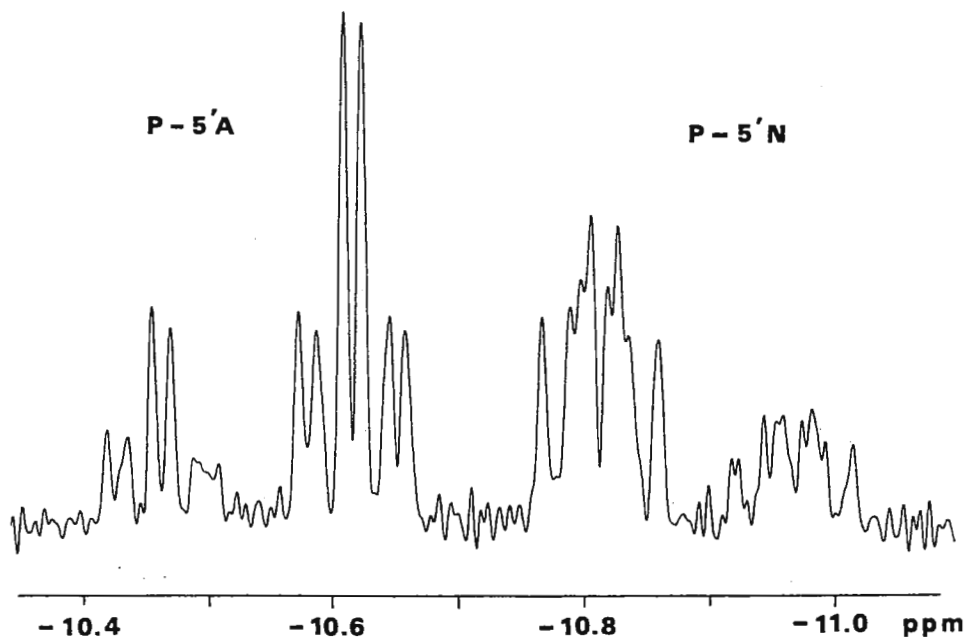
The conformation of the nicotinamide coenzymes has been the subject of many papers, but the preferred conformations in solution have not yet been unambiguously determined. We analyzed the conformational equilibrium of the ribose rings, following the Altona procedure, and we obtained the relative population of the N-type and S-type puckering modes. The S and N families of conformers have almost equal population for the adenine-ribose, whereas for the nicotinamide-ribose rings the S-type reaches the 90% in both forms of the coenzyme.

The conformational preference of the pyrophosphate chain was deduced relating to the rotation about the C(5')-O(5') and C(4')-C(5') bonds, which is defined by the torsion angles β and γ respectively. The relative populations of the conformers calculated from the values of $J(P,H5')$, $J(P,H5'')$ and $J(P,C4')$ for β and from $J(H4',H5')$, $J(H4',H5'')$ and $J(P,H4')$ for γ respectively. We found a high preference (75-80%) for the trans conformer β^t in all ribose rings, whereas the γ rotamers are distributed between the gauche γ^+ and the trans γ^t forms. The adenine-ribose displays 63% of γ^+ and 34% of γ^t ; the nicotinamide ribose reaches the 90% of γ^+ in the oxidized form, whereas NADPH shows only the 50%.

The dihydropyridine ring protons of NADPH were also examined and the results will appear in a near publication.

We have very recently installed our new AMX-600 Bruker spectrometer, which works very well. We did not find problems, but we spent a lot of time for the preparation of the room. We are

particularly satisfied with the easy shimming and with the water peak-suppression experiments in 90% H₂O samples.



³¹P NMR spectrum (121.4 MHz) of the pyrophosphate fragment of NADP⁺ in D₂O/NH₃ solution, pH /7.2 external reference 85% H₃PO₄.

With best greetings, sincerely yours

E. Ragg

L. Scaglioni

R. Mondelli

Ezio Ragg

Leonardo Scaglioni

Rosanna

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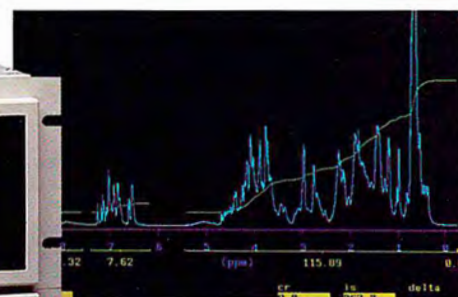
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Amplitude Droop	5% to 10mS typ; 7% max		
Phase Change/Power Output	10 $^{\circ}$ to rated power typ.		
Phase Error Overpulse	4 $^{\circ}$ to 10mS duration typ.		
Noise Figure	11dB typ.	8dB typ.	
Output Noise (blanked)	< 20dB over thermal		
Blanking Delay	< 5uS on, 2uS off, TTL signal		
Protection	1. VSWR- will withstand infinite VSWR at rated power		
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	2. Output- Type N (F)		
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Indicators, Front Panel	1. Peak power meter	5. CW Mode	
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	3. Over duty cycle		
	4. Over pulse width		
System Monitors	1. Thermal		
	2. DC power supply fault		
	3. Over duty cycle		
	4. Over pulse width		
Front Panel Controls	1. A.C. power	3. Duty cycle	
	2. Pulse width		
Cooling	Internal forced air		
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DAVID G. GORENSTEIN, DIRECTOR

June 12, 1990
(received 6/15/90)

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

MORASS: A Computer Program for **M**ultispin **O**verhauser **R**elaxation **A**nalysi**S** and
Simulation

Dear Dr. Shipiro,

Interproton distances from nuclear Overhauser effect spectroscopy (NOESY) have historically been derived from the two-spin approximation. Recently however, analysis of NOESY data using a complete relaxation rate matrix approach has been shown to be an effective method for obtaining a large number of accurately determined interproton distances. In many cases, a matrix approach allows one to accurately quantify distances from nOe 's under conditions where the distances derived from a two-spin analysis would be unsatisfying (1-3).

We have developed a computer program MORASS (**M**ultispin **O**verhauser **R**elaxation **A**nalysi**S** and **S**imulation) to aid in our studies of bio-macromolecular structure. The program uses a relaxation rate matrix solution of the simultaneous Bloch equations and may be used to evaluate the relaxation rate matrix given a set of experimental volumes, or simulate the Noe (either time dependence or full NOESY spectra) from model structures. We have also incorporated a facility to produce a hybrid volume matrix in which elements calculated from a structural model are merged with experimental NOESY volumes. The hybrid matrix approach has been helpful in those cases where a complete experimental volume matrix cannot be measured due to experimental limitations (4,5).

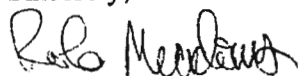
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An experimental NOESY plot (figure A) is compared with a MORASS calculated NOESY plot (Figure B) of the base to H2'/H2'' special region for a duplex DNA fragment resulting from a combined MORASS hybrid matrix and constrained molecular dynamics refinement (6). The NOESY spectrum was plotted using in house software.

MORASS is written in FORTRAN and is available upon request.

Please credit this contribution to Carol Post's account.

Sincerely,



Robert P. Meadows
Box 614
Dept. of Chemistry
Purdue University
West Lafayette, IN 47907

Sincerely,



Carol B. Post
Dept. of Medicinal Chemistry
Purdue University
West Lafayette, IN 47907

Sincerely,



David G. Gorenstein
PUBMRL Director
Dept. of Chemistry
Purdue University
West Lafayette, IN 47907

- (1) Dobson, C. M., Olejniczale, E. T., Poulsen, F. M., Ratcliffe, R. G. (1982), J. Magn. Res. 48, 97-110.
- (2) Keepers, J. W., James, T. L. (1984), J. Magn. Res. 57, 404-426.
- (3) Post, C. B., Meadows, R. P., Gorenstein, D. G., J. Am. Chem. Soc., in press.
- (4) Boelens, R., Koning, T. M. G., Kaptein, R. (1988), J. Mol. Struct. 173, 299-311.
- (5) Nikonowicz, E. P., Meadows, R. P., Gorenstein, D. G. (1990), Biochemistry 29, 4193-4204.
- (6) Nikonowicz, E. P., Meadows, R. P., Gorenstein, D. G., submitted.

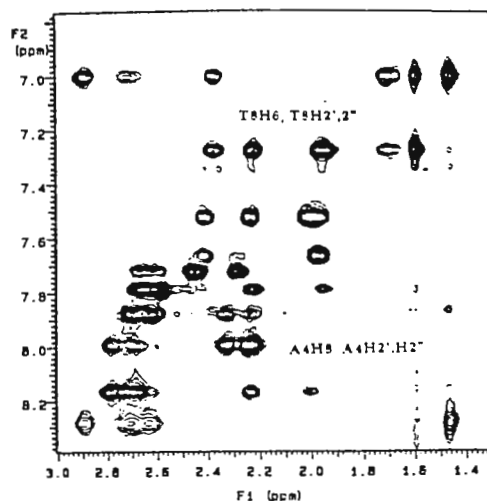


Figure A: Observed

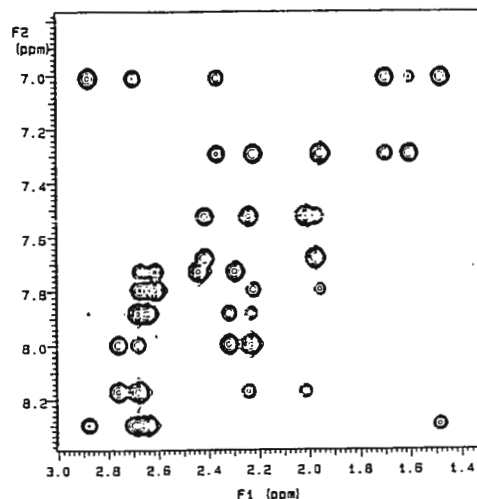


Figure B: Calculated



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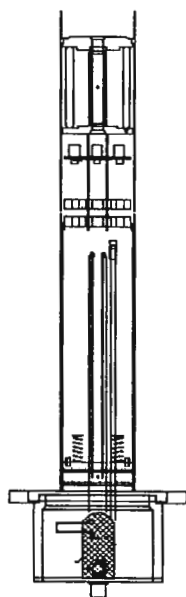
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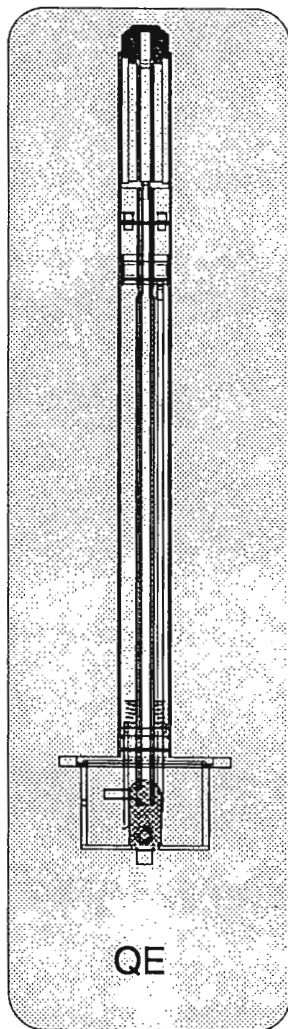
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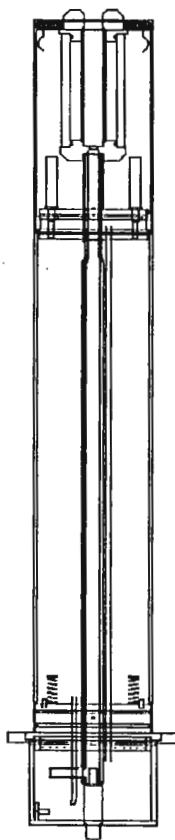
Technology available to do many probe modifications to correct probe deficiencies such as poor water suppression due to lead pickup.



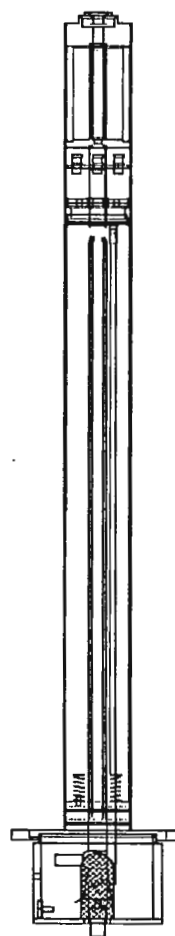
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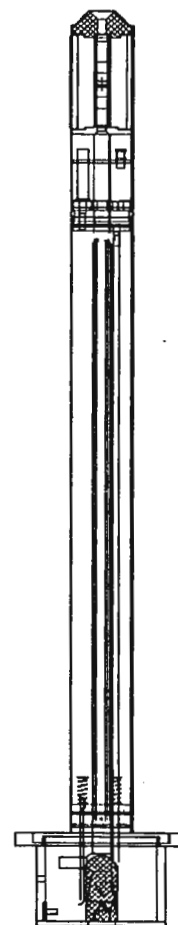
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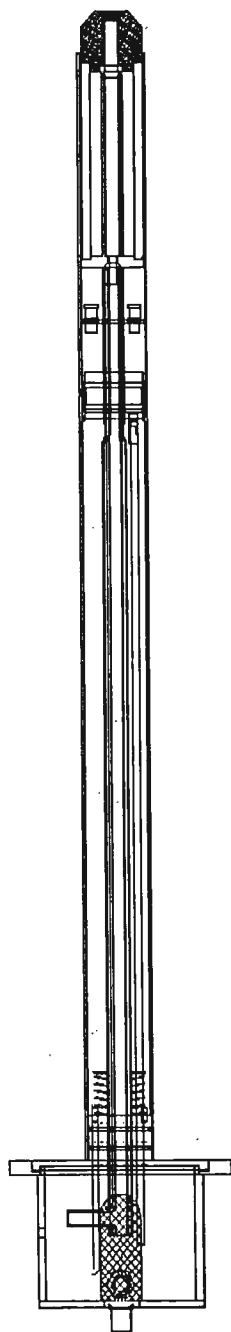
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Department of Chemistry

Telephone (508) 793-7116

June 12, 1990
(received 6/18/90)

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

- (1). 2D exchange powder NMR on MSL 300
- (2). Post-doctoral position available

Dear Barry:

We have been doing solid state ^{31}P 2D exchange experiments on our recently installed MSL 300. These experiments are done in the mode of the ^{13}C and ^2H 2D experiments of Spiess et al. It appears that for spin 1/2 decoupled CSA patterns the experiments are able to be carried out and processed via the Aspect 3000. Simulation of the resulting patterns is done off-line and probably processing of ^2H quadrupole experiments is better accomplished off-line. The ^{31}P 2D powder spectra shown were obtained at 121 MHz using magnetization generated by a conventional CP sequence as shown. The ^{31}P $\pi/2$ pulse was 3-8 μs and dead time estimated at 5 μs . A sweep width of 250KHz with 256 data points in τ_1 (evolution) and 512 in τ_2 (detection) was employed. In order to avoid the phase twist problem and obtain a pure absorption mode display the method of RuSH was used and the real (cosine) and imaginary (sine) parts of the tranverse magnetization were collected in two separate experiments, then combined.

The patterns shown are done with a mix time (τ_m) of 1s and show the signal in the presence and absence of exchange. The sample here is a phosphate plasticizer in a polymer matrix and shows clearly the onset of plasticizer reorientation at the higher temperature, in fact, at the same temperature at which this material exhibits mechanical loss at this same timescale.

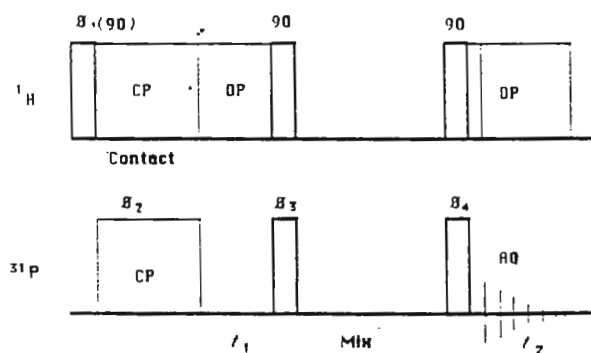
Post-doctoral position: We have a post-doc position available starting in the fall to do solid state NMR experiments in multi-component polymer systems to probe structure and dynamics. Anyone interested can contact me (P.T.I.) at (508) 793-7753 or send a C.V. to the above address.

Regards,

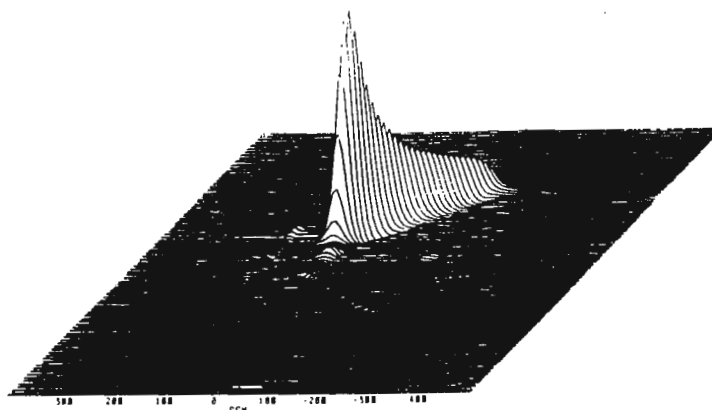
Paul

Changan Zhang

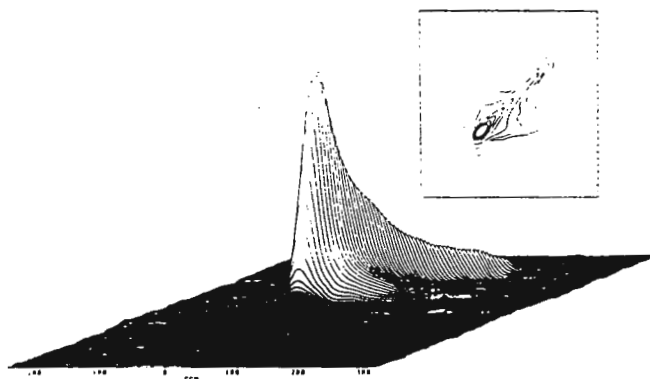
Paul T. Inglefield & Changan Zhang



2D CSA Pattern at 344 K,
No Exchange.



2D CSA Pattern at 356 K,
Exchange Present.



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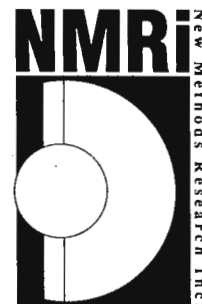
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GEORGETOWN UNIVERSITY MEDICAL CENTER

Department of Pharmacology

June 10, 1990
(received 6/15/90)
Dr. Bernard L. Shapiro
NMR Newsletter
966 Elsinore Court
Palo Alto CA 94303

^{31}P and ^{13}C NMR studies on the effects of EGF on breast cancer cells.

Dear Barry:

I have recently moved to the Pharmacology Department of Georgetown University Medical School, and Peter van Zijl and Ofer Kaplan have joined me here as Research Assistant Professors. Please consider this contribution as the first from my new laboratory.

We have studied the metabolic effects of epidermal growth factor (EGF) on MDA-468 human breast cancer cells by ^{31}P and ^{13}C NMR, and by glucose utilization measurements. These cells exhibit a large number of EGF receptors (EGFR), and have been reported to be inhibited *in vitro* by EGF (1). This is surprising since EGF is a growth factor that usually stimulates cell division. We found that this inhibition can be minimized by elevating the glucose concentrations in the growth medium (2).

Cells were cast in agarose threads, perfused with the growth medium containing very high concentrations of EGF (up to 200 ng/ml), and simultaneous ^{31}P NMR and glucose consumption and lactate production measurements were performed. EGF induced a striking increase in glucose utilization in MDA-468 cells, while no changes were noted in the ^{31}P NMR spectra for up to 36 hs of EGF perfusion, and 95% of the cells remained viable. No changes in glucose metabolism were observed when MCF-7 human breast cancer cells, which have very low numbers of EGFR (3), were perfused with EGF as controls.

The rates of glucose transport and phosphorylation were quantitated by ^{13}C NMR, utilizing [6- ^{13}C]-2-DG (4), and a 97% increase was found in MDA-468 cells following EGF administration. The enhancement of glucose utilization by EGF, together with the lack of toxicity in the perfused system, indicate that the inhibition observed *in vitro* is mediated through the effects on glucose metabolism, and results from glucose depletion and the accumulation of waste materials in cells grown in culture flasks. Clinical observations that

high EGFR tumors are correlated with poor prognosis (3,5) support this hypothesis. In these *in vivo* conditions (similar to our perfusion experiments), endogenous EGF apparently stimulates the cancer cells, in contrast to its inhibitory effect *in vitro*.

Sincerely,

OKaplan

Ofer Kaplan

Jack S. Cohen

1. J. Filmus, et al. BBRC 128: 898-905, 1985.
2. O. Kaplan, et al., J. Biol. Chem., in press.
3. N.E. Davidson, et al. Mol. Endocrinol. 1: 216-223, 1987.
4. G. Navon, et al. FEBS Lett. 247: 86-90, 1989.
5. J. R. C. Sainsbury, et al. Lancet I: 364-366, 1985.



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Research

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Bloomfield, New Jersey 07003
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Biophysical NMR Spectroscopist Positions

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Polyflon's CuFlon® Pure PTFE microwave substrates electroplated with copper, matches the peel strength of copper clad laminates at 8 lb/sq. in. (min.). Custom applications utilizing this process have typically exceeded peel strengths of 15 lb/sq. in.

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NMR AND DATA PROCESSING LABORATORY

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

.....

June 6, 1990
(received 6/14/90)

Reconstructing Special Planes from 3D NMR Data by SDFT

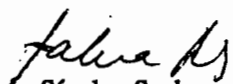
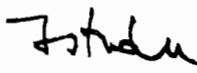
Dear Dr. Shapiro:

We have been using the frequency selective version of DFT (SDFT) [S. Szalma, I. Pelczer, P. N. Borer, G. C. Levy, submitted] instead of widely applied FFT for 2D and 3D NMR data processing. An interesting feature of this approach is that it requires less data traffic and under certain circumstances less computing than conventional processing.

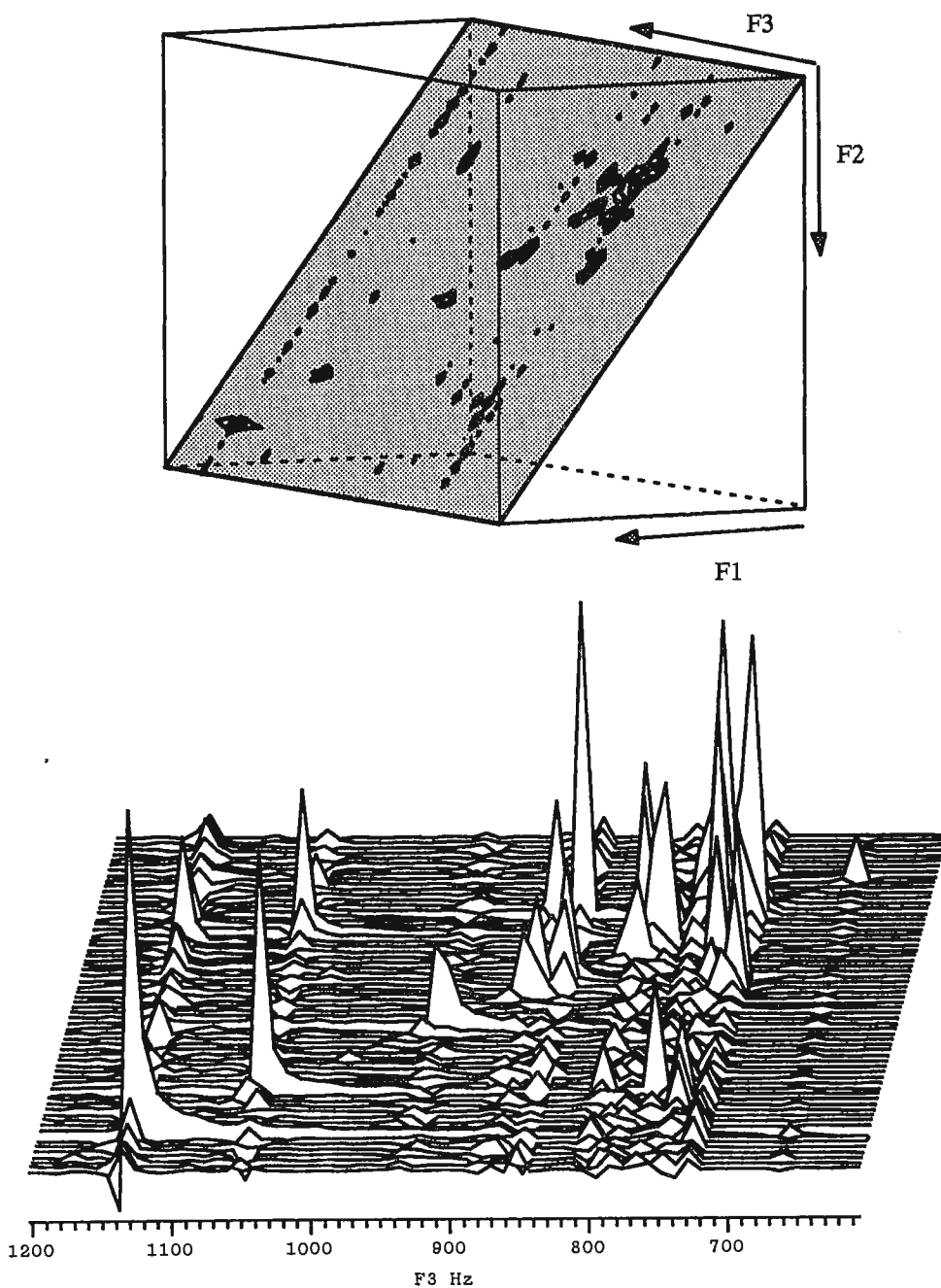
Another unique characteristic of the SDFT, is that any point in the frequency domain can be reconstructed at will. This can be especially useful for producing subvolumes or surfaces of interest from a multidimensional time domain data set, without calculation of the full frequency domain.

A straightforward application of this possibility is easy and fast reconstruction of typical planes of a 3D NMR spectrum. Cross-diagonal and back-transfer planes can be separately produced as shown in the figure for the 3D ROESY-HOHAHA correlation of the model benzantracene. The calculation of the $F1 = F2$ cross-diagonal plane took 77s CPU time on a Stellar GS-1000 graphics supercomputer (the time domain size was $32 \times 64 \times 256$ "hyper-hypercomplex" points. The resulting spectrum contains 64×128 real points using "negative zero-filling", another feature unique to SDFT). The same application can run on a Sun Sparcstation 1 also, where the reconstruction time is roughly 3-20 min, depending on final size. Further interesting applications are under investigation in our lab.

Yours sincerely,

 
Sándor Szalma and István Pelczer

P.S. Please credit this contribution to Prof. G. C. Levy's account.



Mailing Label Adornment: Is Your Dot Red ?

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carried out at

HARVARD UNIVERSITY

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Standard tube length: 178mm (7 inches);
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i.d. 4.20 ± 0.025 mm (0.001 in.);
camber ± 0.013 mm (0.0005 in.).
For additional length,
add \$0.24 per cm (\$0.60/in.).

1-99 tubes...\$4.50 ea.
100-299.....\$4.25 ea.
300 +\$4.00 ea.

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top
quality
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Standard tube length: 178mm (7 inches);
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camber ± 0.025 mm (0.001 in.).
For additional length,
add \$0.21 per cm (\$0.50/in.).

1-99 tubes...\$3.00 ea.
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i.d. 4.20 ± 0.025 mm (0.001 in.);
camber ± 0.025 mm (0.001 in.).
For additional length,
add \$0.16 per cm (\$0.40/in.).

1-99 tubes...\$2.50 ea.
100-299.....\$2.45 ea.
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i.d. 4.20 ± 0.025 mm (0.001 in.);
camber ± 0.03 mm (0.0015 in.);
For additional length,
add \$0.08 per cm (\$0.20/in.).

1-99 tubes...\$2.20 ea.
100-299.....\$2.00 ea.
300 +\$1.90 ea.

No. 505-P PRECISION for medium and high resin. NMR

Standard tube length: 178mm (7 inches);
o.d. 4.97 ± 0.03 mm (0.001 in.);
i.d. 4.20 ± 0.03 mm (0.001 in.);
camber ± 0.04 mm (0.002 in.);
For additional length,
add \$0.08 per cm (\$0.20/in.).

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100-299.....\$1.75 ea.
300 +\$1.70 ea.

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Standard tube length: 178mm (7 inches)
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i.d. 4.20 ± 0.05 mm (0.002 in.);
camber ± 0.07 mm (0.003 in.);
For additional length,
add \$0.08 per cm (\$0.20/in.).

1-99 tubes...\$0.80 ea.
100-299.....\$0.75 ea.
300 +\$0.70 ea.

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No. 1008-UP ULTRA PRECISION for ultra high resolution NMR

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i.d. 8.76 ± 0.025 mm (0.001 in.);
camber ± 0.007 mm (0.0003 in.).
For additional length,
add \$0.20 per cm (\$0.50/in.).

1-5 tubes...\$14.50 ea.
6-25.....\$13.75 ea.
26 +\$12.95 ea.

No. 1005-P PRECISION for medium and high resin. NMR

Standard length: 178mm (7 inches);
o.d. 10.00 ± 0.013 mm (0.0005 in.);
i.d. 8.76 ± 0.025 mm (0.001 in.);
camber ± 0.013 mm (0.0005 in.).
For additional length,
add \$0.20 per cm (\$0.50/in.).

1-5 tubes...\$9.50 ea.
6-25.....\$9.25 ea.
26 +\$9.00 ea.

20mm o.d. THIN WALL NMR SAMPLE TUBES

No. 2005-P PRECISION for medium and high resin. NMR

Standard length: 178mm (7 inches);
o.d. 20.00 ± 0.01 mm;
i.d. 17.70 ± 0.02 mm;
camber ± 0.03 mm.
For additional length,
add \$1.00 per cm (\$2.50/in.).

1-5 tubes...\$38.00 ea.
6 +\$37.25 ea.

NOTE: Other tube sizes
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Rods for all Vortex Plugs.....\$10 each.

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SF 500.135
 SY 83.0
 O1 7900.000
 SI 32768
 TD 32768
 SW 5000.000
 HZ/PT 305

PW 5.0
 RD 0.0
 AQ 3.277
 RG 32
 NS 72
 TE 297

FW 6300
 O2 7925.000
 DP 10H P0

LB 0.0
 GB 0.0
 CX 36.00
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 F1 2.700P
 F2 .401P
 HZ/CM 31.950
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 SF 5426.67

I have evaluated Norell's XR-55 Precision NMR Sample Tubes and found them to be:

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Type of NMR equipment used in the above evaluation: BRUKER AM-500

Date Nov 30, 1988 Name SCOTT VIRGIL Title GRADUATE STUDENT

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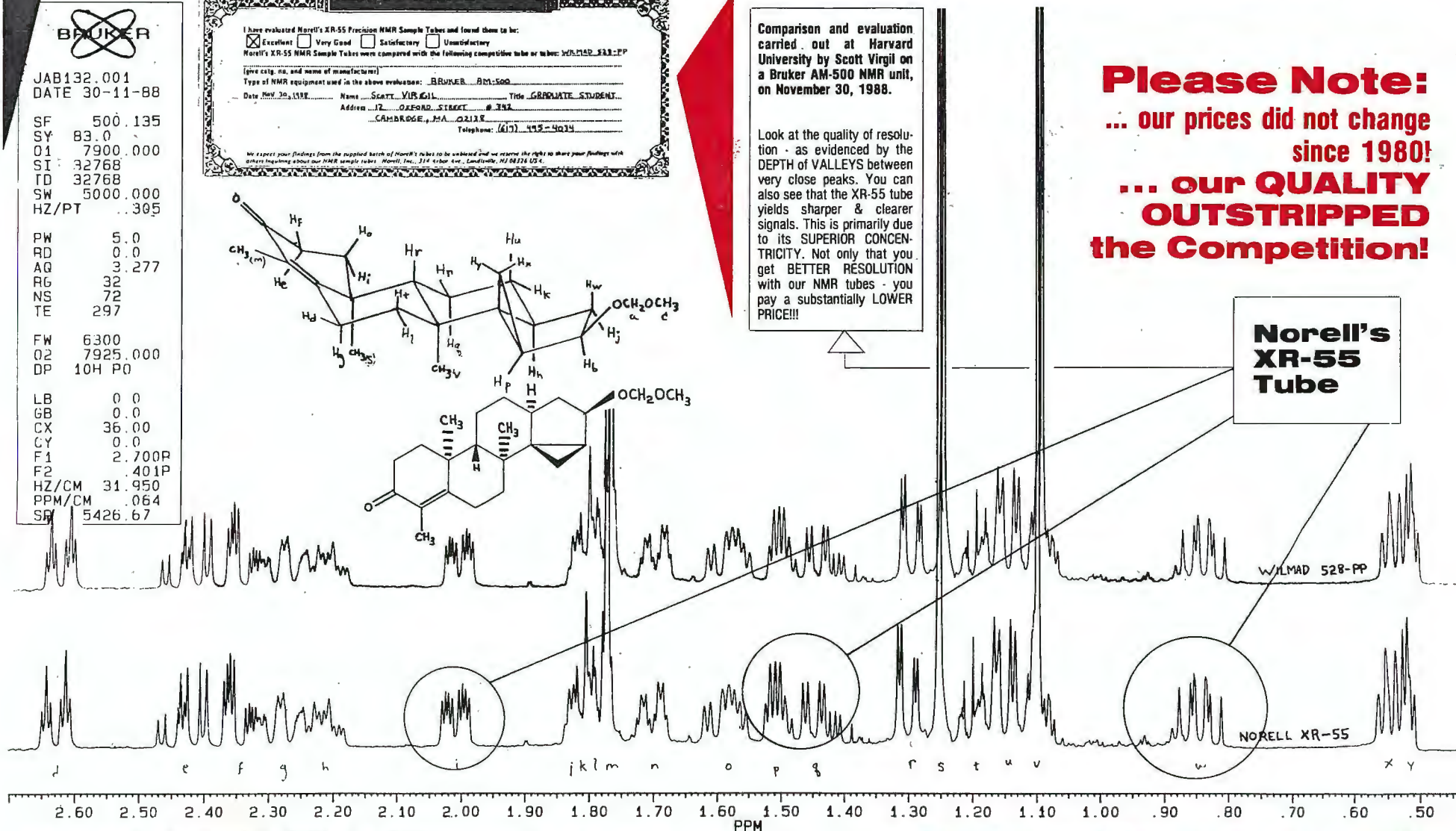
We expect your findings from the supplied batch of Norell's tubes to be unbiased and we reserve the right to share your findings with others inquiring about our NMR sample tubes. Norell, Inc., 22 Marlin Lane, Mays Landing, NJ 08330 USA.

Comparison and evaluation
 carried out at Harvard
 University by Scott Virgil on
 a Bruker AM-500 NMR unit,
 on November 30, 1988.

Look at the quality of resolution - as evidenced by the DEPTH of VALLEYS between very close peaks. You can also see that the XR-55 tube yields sharper & clearer signals. This is primarily due to its SUPERIOR CONCENTRICITY. Not only that you get BETTER RESOLUTION with our NMR tubes - you pay a substantially LOWER PRICE!!!

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May 29, 1990
(received 5/30/90)

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Fax (415) 231-1368
Telex 172197

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

Subject: Isomers of 5-Hydroxy-3-cyclohexene-cis-1,2-dicarboximide

Dear Barry;

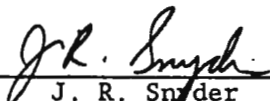
Depending on whether the hydroxy group is cis or trans to the five member ring, 5-Hydroxy-3-cyclohexene-cis-1,2-dicarboximide can exist in I (cis) and II (trans) isomers.

The proton coupling constants were measured and are listed in Table 1. The two sets of coupling constants are quite similar in magnitude except for $J_{1,6e}$ and $J_{1,6a}$, the values of which suggested that H_1 is gauche to both 6 position protons in II but anti to H_{6a} and gauche to H_{6e} in I. Inspection of a Dreiding model showed that H_1 should be anti to H_{6a} and gauche to H_{6e} in the cis isomer but would be gauche to both H_{6e} and H_{6a} in the trans isomer.

NOE difference measurements provide additional support for the isomer assignments. When H_5 of isomer I was irradiated, the multiplet corresponding to H_1 was enhanced showing that these two protons lie on the same side of the cyclohexene ring. H_{6e} , and H_4 also showed enhancement. When H_5 of isomer II was similarly irradiated, only H_{6e} and H_4 showed enhancement. Interestingly, H_2 of the cis isomer showed no enhancement upon irradiation of H_5 . This result suggests that H_5 and H_2 are not close together. Indeed, the Dreiding model shows that when the cyclohexene ring is in the lower energy half-chair conformation, H_5 is closer to H_1 than H_2 . Only when the cyclohexene ring is twisted into the higher energy boat conformation, does H_5 become close to H_2 . An alternative explanation to the lack of enhancement at H_2 is that it is an example of the "three spin effect"². Further NOE work is in progress.

Sincerely,


D. J. Bowler


J. R. Snyder


C. K. Tseng

¹P. W. Rabideau (Ed.), Conformational Analysis of Cyclohexenes, Cyclohexadienes, and Related Hydroaromatic Compounds, VCH Publishers, N.Y., 1989.

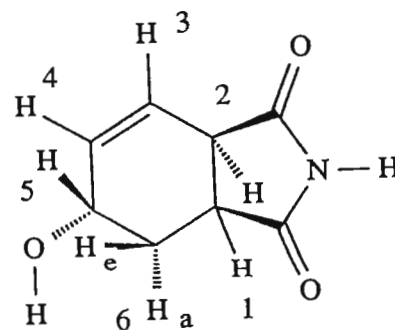
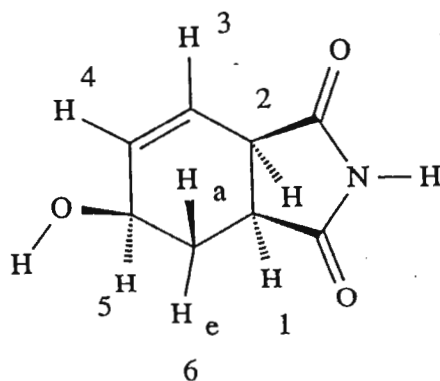
²J. H. Noggle and R. E. Schirmer, The Nuclear Overhauser Effect, Academic Press, N.Y., 1971.

TABLE 1
COUPLING CONSTANTS (in Hz) FOR ISOMERS I AND II¹

	H ₁	H ₂	H ₃	H ₄	H ₅	H _{6e}	H _{6a}	δ ²
H ₁	-	8.7	-	-	-	6.5	10.1	3.12
	-	8.1	-	-	-	4.4	6.1	3.34
H ₂	8.7	-	3.8	2.5	2.5	-	-	3.47
	8.1	-	4.4	2.2	-	-	-	3.50
H ₃	-	3.8	-	10.1	1.7	-	-	5.83
	-	4.4	-	10.1	2.0	-	-	5.76
H ₄	-	2.5	10.1	-	2.7	0.8	-	6.01
	-	2.2	10.1	-	2.2	0.9	-	6.01
H ₅	-	2.5	1.7	2.7	-	4.2	8.0	4.24
	-	-	2.0	2.2	-	5.1	9.3	4.08
H _{6e}	6.5	-	-	0.8	4.2	-	12.7	2.16
	4.4	-	-	0.9	5.1	-	13.1	2.35
H _{6a}	10.1	-	-	-	8.0	12.7	-	1.58
	6.1	-	-	-	9.3	13.1	-	1.62

¹Upper row for each proton refers to isomer I, lower row to isomer II.

²Chemical shift from internal TMS. Solvent was Acetone-d₆.



The Synthesis

NEW 300 MHZ FT-NMR



New 300 MHz FT-NMR

In keeping with its policy of offering "the highest quality products available anywhere," CIL has expanded its analytical facilities with the purchase of a new General Electric QE-300 MHz Supercon FT-NMR. The addition of this state of the art instrument allows us to expand our QA/QC program for all of our high purity NMR solvents and carbon-13-labeled compounds.

Prior to this purchase, samples had to be sent out of house for FT-NMR analysis, occasionally causing production delays. Now we can obtain instant results for our NMR solvent, custom synthesis and research product groups.

CIL has also boosted its QC department with the addition of analytical and preparative HPLC systems and a Chirasil-Val capillary column GC for the measurement of optical purity of protein amino acids. This new equipment supplements CIL's other state of the art analytical instrumentation and enables CIL to provide its customers better quality products and services than ever before.

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In order to meet the increasing demand for our products, we recently acquired over 7000 square feet of additional space. This new space has allowed us to relocate our administrative offices to an adjacent building and to double our laboratory capacity in the original facility. Our new, custom designed laboratories will accommodate the continued expansion of our contract research and catalog product areas.

Increased space has allowed us to restructure our production department into eight groups, as well as adding five new laboratories, three new senior chemists and three new associate scientists. In combination with its isotope separation engineering group and its expanded laboratory facilities and technical staff, CIL has emerged as one of the world's leading manufacturers of stable isotope labeled compounds. There is no company more qualified to fill your stable isotope requirements.

CARBON-13 PLANT UPDATE

On-line production at CIL's carbon-13 separation facility in Xenia, Ohio will begin this spring, making CIL the world's largest manufacturer of carbon-13. The plant will produce 25 kilograms of carbon-13 per year.

Installation of the isotope separation columns and support equipment has been completed. Extensive testing has been performed to ensure the integrity of the hundreds of welded/soldered joints in the three miles of stainless steel tubing contained in the separation columns. In addition, more than five miles of wire and thermocouples have been installed and tested.

CIL's separation column design is more advanced than typical installations of the past. This new complexity added significant challenges in the construction phase of the project, but promises several major advantages during operation. For example, the modular design of the CIL plant will provide flexibility in operating efficiency over a range of production rates. More importantly, the manner in which the columns are grouped and the specific measures taken to provide redundancy in critical areas assure that the plant can continue production if malfunction of individual columns should occur. In many typical designs of the past, a single leak or failure could result in a complete shutdown of production.

CIL is committed to being the world's primary source of high quality carbon-13. We welcome inquiries regarding the progress of the plant and the availability of carbon-13.



Carbon-13 Separation Plant

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Third Row: Heather Cavalier, Marketing; Eric Duffy, Ph.D., Technical Service; Tasha Blandini, Customer Service.

NEW NMR PRODUCTS

CDLM-844	Chloroform (^{12}C , 99.96%; D, 99%D) (very low ^{13}C content)	1 g	\$55
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		5 g	\$90
DLM-1558	Dichlorofluoromethane (Freon-21) (D, 99%D) (additional charge for lecture bottle) (for use in low temperature NMR studies)	10 g	\$175
DLM-1802	Ethylene Glycol (D_6 , 99.7%D) (high enrichment)	1 g	\$140
		5 g	\$550
DLM-132	Ethylene Glycol (D_6 , 99%D)	5 g	\$190
DLM-3006	Hexamethylphosphoric Triamide (HMPT, HMPA) (D_{18} , 98%D)	5 g	\$330
DLM-2173	Lithium Deuteroxide (D, 99.5%D) (3N in D_2O)	25 g	\$36
		100 g	\$120
DLM-1988	N-Methyl-2-pyrrolidone (NMP) (D_9 , 97%D)	1 g	\$125
		5 g	\$450
DLM-3037	Nitric Acid (D, 99%D) (70% solution in D_2O)	5 g†	\$36
		25 g†	\$146
DLM-56	Tetrahydrofuran "100%" (D_8 , 99.95%D)	10x0.5 ml	\$148
		10x0.75 ml	\$210
DLM-36	Tetrahydrofuran (D_8 , 99.5%D)	5x1 g	\$125
		3x(10x1) g	\$450
DLM-1803	Tetramethylene Sulfone (D_8 , 98%D)	1 g	\$64
		5 g	\$254
DLM-58	Trifluoroethanol "100%" (D_3 , 99.94%D)	1 g	\$105
		5 g	\$390
DLM-27	Trifluoroethanol (D_3 , 99%D)	1 g	\$36.50
		5x1 g	\$165

† grams of solution contained

NEW PRODUCTS AND PRODUCT UPDATES

DLM-2201	Acetanilide (acetyl-D ₃ ; 98% D)	1 g	\$350
CNLM-121	Adenine (¹³ C ₅ , 99%; ¹⁵ N ₅ , 99%)	0.01 g	\$650
NLM-2168	D-Alanine, N-t-BOC/protected (¹⁵ N, 99%)	0.25 g	\$350
OLM-2159	Aluminum Hydroxide (¹⁸ O ₃ ; 97% + ¹⁸ O)	1 g	\$190
CLM-865	DL-Aspartic Acid (3- ¹³ C, 99%)	0.25 g	\$360
DLM-2218	Cortisol (9,10,11,11-D ₄ ; 95% D)	0.01 g	\$950
DLM-1819	Cotinine (methyl-D ₃ ; 98% D)	0.01 g	\$390
DLM-2006	Decanoic Acid (methyl-D ₃ ; 98% D)	0.5 g	\$250
NLM-2304	Dimethylamine (¹⁵ N, 99%) (gas) (additional charge for lecture bottle)	1 g	\$440
DLM-2181	Dopamine (ring-D ₃ ; 98% D)	0.1 g	\$260
CLM-2187	Ethane (1- ¹³ C, 99%) (gas) (breakseal flask at no additional charge)	0.25 L	\$650
CLM-1553-90	D-Fructose (¹³ C ₆ ; 90% ¹³ C)	0.25 g	\$425
CLM-1509-90	D-Glucose (¹³ C ₆ ; 90% ¹³ C)	0.5 g	\$240
CLM-2024	L-Glutamic Acid (1,2- ¹³ C ₂ ; 99% ¹³ C)	0.25 g	\$450
CLM-2008	L-Glutamic Acid, gamma-Benzyl Ester, N-t-BOC/protected (1,2- ¹³ C ₂ ; 99% ¹³ C)	0.1 g	\$350
CLM-1902	L-Glutamine, N-t-BOC/protected (1,2- ¹³ C ₂ ; 99% ¹³ C)	0.1 g	\$450
CLM-2001	L-Glutamine (1,2- ¹³ C ₂ ; 99% ¹³ C)	0.1 g	\$440
CLM-1397	Glycerol (2- ¹³ C, 99%)	0.25 g	\$240
CLM-1510	Glycerol (1,2,3- ¹³ C ₃ ; 99% ¹³ C)	0.25 g	\$550
DLM-2306	Hydroquinone (D ₆ ; 98% D)	1 g	\$275
NLM-2167	L-Isoleucine, N-t-BOC/protected (¹⁵ N, 99%)	0.25 g	\$480
CLM-2119	Linoleic Acid, Sodium Salt (1- ¹³ C, 99%) (chemical purity 95%)	0.25 g	\$600
DLM-2108	Morpholine (D ₉ ; 95% D)	1 g	\$350
DLM-1818	Nicotine (methyl-D ₃ ; 98% D)	0.01 g	\$390
NLM-814	Nitroglycerine (¹⁵ N ₃ ; 99.6% ¹⁵ N) (1000 ± 100 µg/ml in ethanol solution)	1.2 ml	\$290
NLM-2174	L-Ornithine · HCl (5- ¹⁵ N, 99%)	0.1 g	\$250
CLM-2120	Palmitic Acid (2- ¹³ C, 99%)	0.5 g	\$750
CLM-2253	Palmitic Acid (methyl- ¹³ C, 99%)	0.1 g	\$390
NLM-835	L-Proline (¹⁵ N, 99%)	0.25 g	\$550
CLM-1579-90	Sodium Lactate (1,2,3- ¹³ C ₃ ; 90% ¹³ C) (30% solution in H ₂ O)	0.25 g†	\$490
DLM-2097	Tetramethyl Bisphenol A (isopropyl-D ₆ ; 98% D)	1 g	\$250

† grams of product contained

¹³CO₂ BREATH TESTS

During the past year, CIL has expanded its analytical services to include breath test analyses. CIL now supplies carbon-13-labeled substrates, breath collection kits and stable isotope ratio analysis of breath samples.

Breath gas analysis is an important tool for diagnostic and medical research. Diagnostic uses currently under review include tests for *Campylobacter pylori* (a bacteria suspected of causing peptic ulcers), chronic hepatitis and cirrhosis. Research applications include studies of fat malabsorption, bile acid metabolism, hepatic function, glucose metabolism, nutrition balance and energy expenditure, bacterial overgrowth syndromes, and gastric emptying disorders.

A number of important metabolic pathways may be monitored by measuring the ¹³C/¹²C ratio in expired CO₂. This method relies on the fact that a subject with a metabolic disorder may absorb and oxidize a labeled substrate at a different rate than a subject with normal metabolism. Studies begin with the oral administration of a ¹³C-labeled substrate to a subject. The specific substrate is designed to contain a particular target functional group, the breakdown of which will produce ¹³CO₂. The ¹³CO₂ is measured in the subject's exhaled breath. Tracking the changes in the carbon-13 enrichment over time indicates the rate at which the substrate was metabolized.

Breath gas analysis will offer several advantages over other available diagnostic methods. It is non-invasive, requiring only a small oral dose of a labeled substrate. It is safe, using the stable isotope ¹³C rather than the radioactive isotope ¹⁴C. Also, it is a simple, painless, quick procedure which can be administered in an outpatient clinic or private office. Sample collection takes less than two hours. These advantages make the procedure comparatively inexpensive.

¹³C-labeled substrates such as urea-¹³C, triolein-¹³C, acetate-¹³C and methacetin-¹³C and breath collection kits are available from CIL. CIL will also analyze individual breath samples by ¹³C stable isotope ratio analysis (SIRA). For further details, please contact our Technical Services Department.

CUSTOMER SERVICE

CIL now has four customer service representatives who are pleased to receive your orders, answer your questions and solve your problems.

ANDRIA AFFANNATO has been with CIL for a year and a half. Andria graduated from the University of Massachusetts at Amherst with a B.S. in microbiology. Outside of work she is busy planning her June wedding. Andria enjoys sailing in the summer and downhill skiing in the winter.

TASHA BLANDINI joined the CIL staff last November. Tasha is a recent graduate of the University of Lowell with a B.S. in Marketing. Tasha is also planning a 1990 wedding; hers will be in October. Tasha enjoys swimming and aerobic dancing. Welcome aboard, Tasha! We're glad to have you on the CIL team.

DEBBIE GAGNON has been with CIL for over two years. Debbie is a graduate of Keene State College with a B.S. in Education. She enjoys downhill skiing and aerobic dancing.

PAMELA DEGOU, a five year veteran with CIL, was recently given a new position as our full-time International Sales Representative. She holds an Associates Degree from Northern Essex Community College as an Executive Secretary in Accounting. Pam now coordinates requests from our team of international representatives. Pam is a new mother who enjoys spending time with her husband and their 22 month old son.

NEW U-¹³C AMINO ACIDS

CLM-2184	L-Alanine (¹³ C ₃ , 98%+)	0.1 g	\$330
CLM-2265	L-Arginine · HCl (¹³ C ₆ , 98%+)	0.1 g	\$1050
CLM-1801	L-Aspartic Acid (¹³ C ₄ , 98%+)	0.1 g	\$650
CLM-1800	L-Glutamic Acid (¹³ C ₅ , 98%+)	0.1 g	\$690
CLM-2264	L-Histidine · HCl (¹³ C ₆ , 98%+)	0.05 g	\$750
CLM-2248	L-Isoleucine (¹³ C ₆ , 98%+)	0.05 g	\$540
CLM-2262	L-Leucine (¹³ C ₆ , 98%+)	0.1 g	\$790
CLM-2247	L-Lysine · HCl (¹³ C ₆ , 98%+)	0.1 g	\$840
CLM-2250	L-Phenylalanine (¹³ C ₉ , 98%+)	0.1 g	\$790
CLM-2260	L-Proline (¹³ C ₅ , 98%+)	0.05 g	\$540
CLM-1574	L-Serine (¹³ C ₃ , 98%+)	0.1 g	\$750
CLM-2261	L-Threonine (¹³ C ₄ , 98%+)	0.1 g	\$840
CLM-2263	L-Tyrosine (¹³ C ₉ , 98%+)	0.1 g	\$840
CLM-2249	L-Valine (¹³ C ₅ , 98%+)	0.1 g	\$750

CIL ALGAL PRODUCTS

CIL now offers the following products derived from algae. Uniform incorporation of carbon-13 and deuterium into algal biomass provides a resource from which fully labeled products are isolated.

CLM-1548	Algal Amino Acid Mixture (U- ¹³ C, 98%+)	0.5 g	\$515
DLM-2044	Algal Amino Acid Mixture (U-D, 98%)	1 g	\$300
NLM-2161	Algal Amino Acid Mixture (U- ¹⁵ N, 98%)	0.5 g	\$450
ULM-2314	Algal Amino Acid Mixture (Unlabeled)	1 g	\$80
CLM-2063	Algal Lipid Mixture (U- ¹³ C, 98%+)	0.5 g	\$900
DLM-2064	Algal Lipid Mixture (U-D, 98%)	1 g	\$200
CLM-2065	Algal Lyophilized Cells (U- ¹³ C, 98%+)	0.5 g	\$180
DLM-2066	Algal Lyophilized Cells (U-D, 98%)	1 g	\$95
NLM-2162	Algal Lyophilized Cells (U- ¹⁵ N, 98%)	1 g	\$150
ULM-2177	Algal Lyophilized Cells (Unlabeled)	10 g	\$100
CLM-2046	Algal Whole Hydrolysate (U- ¹³ C, 98%+)	0.5 g	\$400
DLM-2045	Algal Whole Hydrolysate (U-D, 98%)	1 g	\$275
NLM-2163	Algal Whole Hydrolysate (U- ¹⁵ N, 98%)	0.5 g	\$450
ULM-999	Algal Whole Hydrolysate (Unlabeled)	5 g	\$175

NEW ENVIRONMENTAL PRODUCTS

EM-2286	bis-Acetyl 4-Chlorocatechol (Unlabeled)	0.1 g	\$250
CLM-3235	Biphenyl (¹³ C ₁₂ , 99%) (100 µg/ml in nonane)	1.2 ml	\$580
DLM-1148	Diazinon (diethyl-D ₁₀ , 98%)	5 mg	\$550
CLM-1544	Dibenzo-p-dioxin (¹³ C ₁₂ , 99%) (50 µg/ml in nonane)	1.2 ml	\$700
CLM-1561	Dibenzofuran (¹³ C ₁₂ , 99%) (50 µg/ml in nonane)	1.2 ml	\$800
DLM-2132	Di-n-butylamine (D ₁₈ , 98%)	0.1 g	\$250
DLM-1934	1,1-Dichloroethane (2,2,2-D ₃ , 98%)	0.1 g	\$195
DLM-3073	2,4-Dimethylphenol (ring-D ₃ , 98%)	0.1 g	\$130
DLM-3173	4,6-Dinitro-2-methylphenol (3,4-D ₂ , 98%)	0.1 g	\$205
DLM-2133	Diphenylamine (D ₁₀ , 98%)	0.1 g	\$200
DLM-2140	Fluoranthene (D ₁₀ , 98%)	0.1 g	\$90
CLM-2110	Hexachlorocyclopentadiene (¹³ C ₄ , 99%)	5 mg	\$325
CLM-2003	Hexachloroethane (1- ¹³ C, 99%)	0.1 g	\$225
CLM-2287	Kepone (¹³ C ₈ , 99%)	1 mg	\$750
DLM-2277	2-(4-Methylphenyl) propane (D ₁₄ , 98%)	1 g	\$250
CLM-2078	Mirex (¹³ C ₈ , 99%)	1 mg	\$590
DLM-2130	N-Nitrosodimethylamine (D ₆ , 98%)	0.1 g	\$300
DLM-2131	N-Nitroso-di-N-propylamine (D ₁₄ , 98%)	0.1 g	\$550
DLM-2053	Tetrahydrophthalic Anhydride (1,1,2,3,4,4-D ₆ , 98%)	0.01 g	\$480
DLM-2054	Tetrahydrophthalimide (1,1,2,3,4,4-D ₆ , 98%)	0.01 g	\$480
DLM-2143	2,4,5-Trichlorophenol (3,6-D ₂ , 98%)	0.1 g	\$250

CAREER OPPORTUNITIES AT CIL

CIL, a world leader in the manufacture of stable isotope labeled compounds, has several excellent career opportunities for the right individuals.

The Manufacturing Department is always interested in hearing from Ph.D., M.S., and B.S. organic chemists and biochemists who are skilled in the art of synthesis.

Our sales group offers a unique challenge to people who have strong communication skills and a technical background in organic or analytical chemistry or biochemistry.

Current openings exist for the following positions: Regional Sales Representative and Environmental Products Manager. CIL offers an excellent salary, benefits package, and stock option plan. Send resume and salary requirements to Diane.

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Ottawa, Canada
K1A 0R6

May 31, 1990
(received 6/10/90)

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

Re: Bernoulli Cartridge Drives for Aspect 1000 and 3000 Computers


Dear Barry,


Since April of 1988 we have been using a Bernoulli cartridge disk drive on a Bruker Aspect 1000 computer. The Bernoulli drive is produced by IOMEGA corporation and retails for less than \$2000 (Cdn) as an IBM pc add-on. We designed and built the necessary SCSI host adapter which plugs into the Aspect's I/O bus. The driving software was written in Aspect assembly language and occupies only 6K words of memory. Each Bernoulli cartridge provides up to 20 Mbytes of data storage in a removable 5.25 inch package, which roughly translates into three thousand 2K NMR data files.

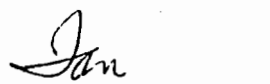
Prior to April of '88 we were restricted to using a 9-track tape drive for backup and retrieval of data files. Since large numbers of data files are generated on a daily basis, individual users were required to back up their data files to tape regularly for later retrieval and processing. This resulted in user frustration since the average access time for tape files is 15 minutes.

Although our tape drives are still in service, our users have a strong preference for Bernoulli cartridges, because they are fast, reliable and compact. For the last two years the Bernoulli system has run without a hitch and we can recommend such a data storage system to anyone with an Aspect 1000 or 3000 who requires off-line high density storage at moderate cost.

Yours sincerely,


T. Wessel


J.K. Saunders


I.C.P. Smith

Canada



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Dr. B.L. Shapiro
TAMU NMR Newsletter

May 16, 1990
(received 5/25/90)

Savings and Short cuts for Setting up a 600

Australia's first 600 MHz spectrometer has recently been installed in our Department as part of a joint NMR facility operated with the Department of Organic Chemistry and the nation's largest government research organisation, CSIRO. As site planning guides tend to be written in very general terms, we hope that some of the details of our installation will prove useful to other laboratories.

With no specific funds provided for the installation, a domestic liquid helium price of about \$US25 per litre and estimates of helium requirements for commissioning of between 400 and 1000 l, we were anxious to recover as much helium gas as possible from the initial cool down. A recovery system, comprising a 300 cu ft gas bag and compressor rated at 10 cu ft per minute linked to 2 banks of 5 G size cylinders, proved inadequate for handling the cooling of a 400 wide bore some 5 years earlier. However, the helium liquefaction plant in the University's School of Chemistry has recently set up a similar compressor, housed in a box trailer with a bank of 6 cylinders, to recover helium from magnets which are linked only to gas bags. The combination of the 2 compressors and 3 cylinder banks proved just adequate for complete recovery of helium gas during cool down of the magnet (capacity 135 l) which required a total of 330 l of liquid helium. The arrangement of a mobile compressor and cylinder banks, which can be shared amongst various users and linked together when required, is worth serious consideration by all who are interested in helium recovery.

Our initial feelings of satisfaction were rapidly dissipated when the entire contents of the magnet boiled off (into the recovery system) within 36 hours, presumably due to misalignment of the helium and nitrogen vessels although this was not unequivocally established. After reassembling and refilling with helium, the boil off was entirely normal and now averages a respectable 28 ml/h. Our magnet needed to be brought to field while pumping on the helium vessel and we were unable to put the installation engineer's mind at ease with any of our schemes for gas recovery during this operation, so that approximately 60 l of liquid was lost to atmosphere. Nevertheless, recovery of most of the helium boil off during commissioning of the magnet resulted in a saving of over \$US13000.

Initially the chloroform test sample had very poor lineshape and sidebands could not be reduced below 5%. After exhaustively excluding all of the easy fixes, the Bruker engineers responsible for the installation suggested that the field distortions may have been due to iron in a concrete pillar which intersected the 50 gauss line. After our earlier problems, we were not enthusiastic about the prospect of running the magnet down to test a theory. However, this was not necessary and a plan was devised to move the magnet, at field, 0.9 m so that it was then symmetrically located with respect to a similar pillar on the opposite side of the room and each pillar just clipped the 20 gauss line.

There being limited scope for manoeuvring above the magnet, with a ceiling height of 3.7 m, the original assembly had been achieved with the aid of a cable winch mounted in the room above, with the cable fed through a small hole immediately above the magnet. A similar hole was drilled in the desired new location, debris being collected in a plastic bin held flush against the ceiling. The magnet was secured to its base, with clamps made from aluminium plates, and lifted on to hardwood tracks. The cable winch was then mounted on a steel rail and the rail, in turn, positioned on the floor against a doorway with the winch cable connected to the magnet base. The wooden tracks and winch were located so that the whole magnet assembly could be eased in very small increments towards the desired location where it was subsequently lifted from the tracks and levelled in the usual way. The whole operation proceeded very smoothly with the helium level falling a mere 5%. In the new position the specifications for lineshape and sidebands were easily achieved.

Please credit this contribution to the subscription of Professor Philip Kuchel, the ideas of various Bruker engineers for sparing us the pain of cooling the magnet a third time, and the untiring efforts of Ross Taylor and Andrew Townsend who built the recovery system and Peter Watson who produced the liquid helium.

W.A. Bubb
William A. Bubb.

MAGNEX HIGH RESOLUTION NMR MAGNET SYSTEMS

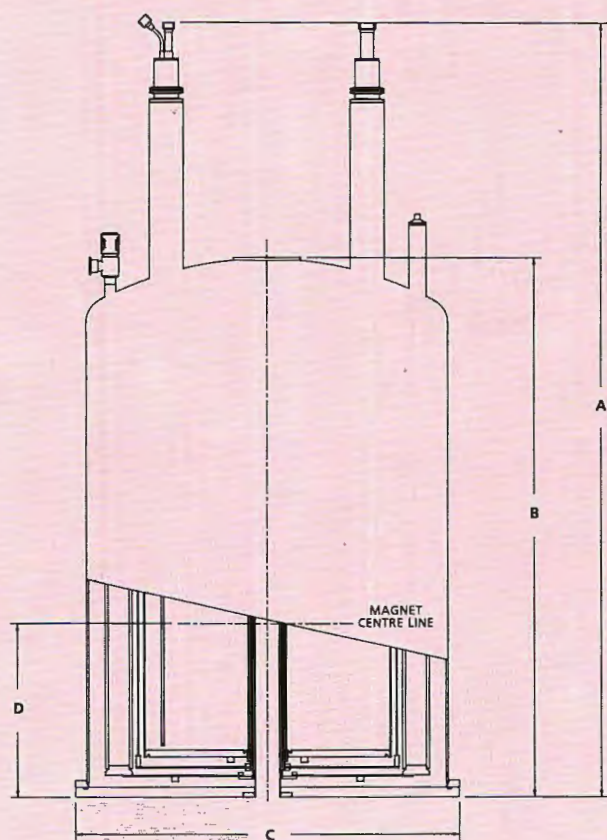
SHORT-FORM SPECIFICATION TABLE FOR MRCA MAGNET TYPES

MRCA magnet type	200/54	360/54	400/54	500/62	360/89	200/89	400/89	360/104
NMR frequency (¹ H MHz)	300/54				300/89		500/89	
Nominal bore mm								
Room temperature bore in mm – minimum access	53.8	53.8	53.8	62.0	88.1	88.1	88.1	104.4
Minimum helium hold-time between refills (days)	140	140	140	100	140	140	60	60
Minimum nitrogen hold-time between refills (days)	14	14	14	14	12	12	12	12
Minimum homogeneity volume diameter (mm) × length (mm)	10×10	10×10	10×10	10×20	20×20	20×20	20×20	25×25
Homogeneity half height line width ¹								
With supercon shims	0.1	0.1	0.1	0.1	0.2	0.2	0.2	0.2
With supercon and RT shims	0.01	0.01	0.01	0.01	0.02	0.02	0.02	0.02
With supercon, RT shims & spinning	0.001	0.001	0.001	0.001	0.002	0.002	0.002	0.002
Spinning side-bands – less than (%)	1	1	1	5	1	1	1	1
Field stability – better than (ppm/hr)	0.01	0.03	0.03	0.03	0.03	0.01	0.04	0.03

Note 1: All specifications measured using MAGNEX probes

MRCA SERIES

NMR MAGNET SYSTEMS



System Type (MRCA)	Dimensions (mm)			
	A	B	C	D
200/54	1516	975	775	312
300/54	1516	975	775	312
360/54	1516	975	775	332
400/54	1516	975	775	332
500/62	1738	1210	860	390
200/89	1530	1000	860	334
300/89	1530	1000	860	334
360/89	1530	1000	860	334
400/89	1550	1010	860	377
500/89	1900	1370	860	435
360/104	1390	955	816	350

**Magnex Scientific Limited**

13, 15 & 19 Blacklands Way, Abingdon Business Park
Abingdon, Oxfordshire, OX14 1DY, England
Telephone: 0235 534488 Fax: 0235 554917

NMR Magnex Scientific Inc.

2550 Appian Way, Suite 209, Pinole, CA 94564, U.S.A.
Telephone: 415 758 7406 Fax: 415 758 0405



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 Alan G. Marshall
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 FAX: 614/292-1685
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120 West 18th Avenue
 Columbus, OH 43210-1173
 Phone 614-292-2251
 FAX 614-292-1685
 TELEX 332911
 Answer Back Code: OSU CHEM UD

15 June, 1990
 (received 6/18/90)

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

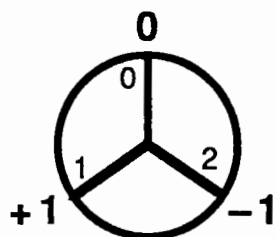
A GEOMETRICAL VIEW OF PHASE- CYCLING AND COHERENCE SELECTION

Dear Barry,

Some time ago it was necessary to write pulse sequences for our Bruker AM-500 which were used to select triple-quantum coherence in both one- and two-dimensional NMR. Papers by Bain; Bodenhausen, Kogler, and Ernst; and Piveteau *et al.* were important in designing phase cycles which select the desired coherence and reject unwanted signal pathways. However, it is not intuitive how phase cycles and phase shifting can be used to select coherence pathways, and an explanation of such processes to non-experts is difficult.

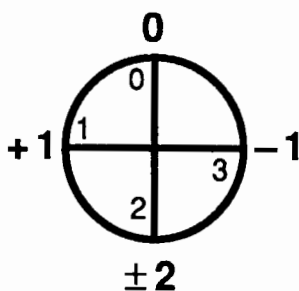
In this note, we present a pictorial representation of the relationship between phase shifts and coherence levels. Several examples are given below. In these diagrams, the large numbers correspond to coherence levels and the small numbers, n correspond to the phase shifts in degrees, given by $360 \cdot n/N$, $0 \leq n \leq N-1$. Some "predictions" can readily be made even if after the fact from these diagrams.

1. With 120° phase shifts, ± 1 coherences can be separated.
2. With 90° phase shifts, ± 2 coherences cannot be separated, and pulse width effects must be relied upon to select coherences.
3. With 60° phase shifts, ± 2 coherences can be separated whereas ± 3 are not separable.



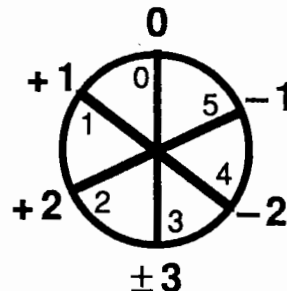
$N = 3$

Phase Shift: 120°



$N = 4$

90°



$N = 6$

60°

```

;INAD60.AU
;by CEC    OSU/CCIC
;PARTIALLY BASED ON "SCREW-PULSE" PHASE CYCLING
;D. PIVETEAU, ET. AL.  J. MAG. RES., 63 255 (1985)
;BODENHAUSEN, KOGLER, ERNST, J. MAG. RES., 58,370 (1984)

1 ZE
2 D1 S1 CPD          ;S1 FOR NOE  D1=RELAXATION DELAY
   D3 S2             ;S2 FOR GOOD DECOUPLING
3 P1 PH1             ;90 DEG PULSE
4 D2                 ;
5 P2 PH2             ;1/4J DELAY  180 DEG PULSE
6 D2                 ;
7 P1 PH3
8 D0                 ;MQ EVOLUTION
9 P3 PH4             ; P3=120 DEG   PH4 HAS 60 DEG PHASE SHIFTS
10 GO=2 PHO          ; FOR SEPARATION OF +2 FROM -2 COHERENCES
11 WR #1
12 IF #1
13 IN=1
14 D3 S1
15 D0
EXIT

PH1=  0 0 0 0 0 0 1 1 1 1 1 1
      2 2 2 2 2 2 3 3 3 3 3 3
PH2=  0 0 0 0 0 0 1 1 1 1 1 1
      2 2 2 2 2 2 3 3 3 3 3 3
PH3=  0 0 0 0 0 0 1 1 1 1 1 1
      2 2 2 2 2 2 3 3 3 3 3 3

;SHIFTS ARE 60 DEGREES BUT SET IN 30 DEGREE STEPS TO
;ALLOW EASY CHANGES OF 90 DEGREES FOR QUAD CYCLING

PH4=(12)  0 2 4 6 8 10 0 2 4 6 8 10
           0 2 4 6 8 10 0 2 4 6 8 10
           3 5 7 9 11 1 3 5 7 9 11 1
           3 5 7 9 11 1 3 5 7 9 11 1
           6 8 10 0 2 4 6 8 10 0 2 4
           6 8 10 0 2 4 6 8 10 0 2 4
           9 11 1 3 5 7 9 11 1 3 5 7
           9 11 1 3 5 7 9 11 1 3 5 7

PHO=  R0 R2 R0 R2 R0 R2 R2 R0 R2 R0 R2 R0
      R0 R2 R0 R2 R0 R2 R2 R0 R2 R0 R2 R0
      R3 R1 R3 R1 R3 R1 R1 R3 R1 R3 R1 R3
      R3 R1 R3 R1 R3 R1 R1 R3 R1 R3 R1 R3
      R2 R0 R2 R0 R2 R0 R0 R2 R0 R2 R0 R2
      R2 R0 R2 R0 R2 R0 R0 R2 R0 R2 R0 R2
      R1 R3 R1 R3 R1 R3 R3 R1 R3 R1 R3 R1
      R1 R3 R1 R3 R1 R3 R3 R1 R3 R1 R3 R1

;D1 RELAXATION DELAY
;P1 P2 ARE 90 AND 180
;D2 IS 1/4J
;D3 SWITCHING TIME FOR POWER LEVEL CHANGE  CA 5 MSEC

```

In the case of one-dimensional multiquantum experiments, it is usually not necessary to separate the \pm -MQ coherence. However, in two-dimensional experiments, only one of the MQ coherences is usually desired, and it is necessary to suppress the other either by pulse width effects or by phase cycling. A phase cycle which completely suppresses the -2 coherence peaks for an INADEQUATE experiment is listed above. In this case, the read pulse is a 120° pulse, because that value transfers the maximum signal intensity. Please credit this contribution to the account of Alan G. Marshall. Thank you.

Charles E. Cottrell, NMR Manager
OSU Campus Chemical Instrument Center

C. Ruck



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Hewlett Packard 5840A with 7671A
Hewlett Packard 5880A with FID,
ECD, NPD
Hewlett Packard 3392A integrator
Perkin Elmer Sigma 1B, dual TCD
Perkin Elmer Sigma 2000 with
HS-100 auto-injector
Perkin Elmer 3920B, dual FID
Tekmar ALS II Autosampler
Tekmar LSC II Purge and Trap
Tracor 540 with ECD, FID
Varian 6000 FID, FPD capillary and
packed column with model 8000
autosampler

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Dionex 2000/sp ion chromatograph
Hewlett Packard diode array
detector 1040M + HP 9000
(9843A), 20 Meg drive and printer
Perkin Elmer LC-75 UV detector
Perkin Elmer LC-85 UV detector
Varian Varichrome UV detector
Varian 5060 + UV-100 and Vista 401
Waters 6000A pump
Waters 440 detector
Waters 441 detector
Waters 710B wisp Autosampler

MASS SPECTROMETERS:

Finnigan 4021
Finnigan 1020/1050
Hewlett Packard 5987 with 5890 GC
with RTE-6 operating system

UV-VIS:

Bausch & Lomb Spectronic 2000
Beckman DU-7
Hitachi 100-80
Perkin Elmer Lambda 3B
Varian 2290

ATOMIC ABSORPTION:

Instrumentation Labs 457 with D2
and 440 AVA
Jarrell Ash 951 with D2
Jarrell Ash 655 Atomizer
Jarrell Ash EDL Power Supply
Jarrell Ash Smith-Hieftje Video 12E
with 188 Furnace Atomizer and
440 AVA
Leeman Labs Plasma Spec III
simultaneous and sequential
Perkin Elmer EDL Power Supply
Perkin Elmer 403
Perkin Elmer 373 with D2
Perkin Elmer 560 with D2
Perkin Elmer 703 with D2
Perkin Elmer 2380 with printer
Perkin Elmer 5000 with D2
Perkin Elmer HGA-400
Perkin Elmer HGA-500
Perkin Elmer HGA-2200 furnace
Perkin Elmer AS-40 Autosampler
Perkin Elmer AS-50 Autosampler
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June 6, 1990
(received 6/11/90)

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

Dear Barry:

We have successfully moved our 2 Tesla 31 cm horizontal bore imaging system to a new laboratory in Weiskotten Hall. The location of the facility on the first floor is ideal. (We have windows in nearly every room). Initially there was concern about the fringe field extending into our neighbors' space on the floors above and below the magnet, but floors are separated by 13' and it seemed the 5 Gauss line would be contained in the floor of the neighbor upstairs and above head height for the neighbor downstairs. Due to the influence of steel beams and reinforcements rods, the fringe fields were well inside what was calculated and everyone was happy with the magnet site.

What no one had anticipated was that the noise of the switching gradients from the magnet would be conducted so efficiently to the floor below. Following a week of operation, the downstairs neighbor was in our lab asking whether "your machine always makes that noise". We placed 3/8" thick rubber sheets under 4"x4" (3/8" thick) aluminum pads under each foot of the magnet, but this had negligible effect on the dB's below. An electric motor shop provided the solution by suggesting we try the same material used in mounting noisy motors. The material is supplied as pads of 1/2" cork laminated between two 1/4" sheets of ribbed neoprene rubber. This 1" thick layer was placed under the 4"x4" aluminum foot pads and provided complete isolation from the magnet noise. (The magnet was gently lifted one side at a time with a long 2'x4' and fulcrum while the pads were installed.)

We now also observe 8 week helium fill intervals compared to the 5 week intervals following the initial installation 3 years ago. I am not sure if this reflects the quality of the recent Oxford reassembly job or if isolation and/or damping of vibrations by the pads account for reduced helium consumption.

Sincerely,

Nikolaus M. Szeverenyi

NMS/wdp

PENN STATE


 Department of Chemistry
 College of Science

 152 Davey Laboratory
 The Pennsylvania State University
 University Park, PA 16802

 May 15, 1990
 (received 5/21/90)

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

Elucidation of Exchange Mechanisms with Selective Pulses

Dear Barry,

Our apologies for allowing our technical contributions to become delinquent. We do have vague memories of pink ultimatum sheets, but we didn't really notice anything until we stopped receiving the Newsletter.

We have been investigating the structure and the exchange mechanism of lithium phenolate aggregates with added salts using variable temperature ^{13}C , ^7Li , and ^6Li liquid state NMR. In the case of lithium 3,5-dimethylphenolate with added lithium perchlorate, we were confident of the structure shown in figure 1, and we knew that the three types of lithium in a diethylether solution were in slow exchange with each other at 228 K. However, we did not know anything about the nature of the exchange process.

In order to elucidate the exchange process, we applied the difference selective inversion recovery experiment described by Freeman.¹ In this experiment, the time-dependence of chemical or dipolar exchange is followed by obtaining the difference between the normal $(-\frac{\pi}{2})_{\text{non-selective}}$ FID and the FID obtained after a $\pi_{\text{selective}} - \tau -$

$(-\frac{\pi}{2})_{\text{non-selective}}$ inversion recovery experiment. In the absence of exchange, only the selectively inverted resonance appears in the spectrum, and its τ dependence yields T_1 . In the presence of chemical or dipolar exchange, the difference spectrum reveals not only the decay of the selectively inverted peak with τ , but also the rise and fall of the magnetization for the exchange partner(s) of the selectively inverted peak. Rate constants and T_1 's for the peaks may be obtained from the data.²

In our case, we observed the ^6Li resonances (73.6 MHz) at 228 K. We chose ^6Li rather than ^7Li because the relaxation times of the former are 40 sec. We used a 75 msec Gaussian selective inversion pulse and a 10 mm BB probe. The results are shown in figures 2, 3, and 4. Even without quantitative analysis, the interpretation is clear. The resonance at 0Hz from the three equivalent Li atoms at the corners of the cube exchanges with both the

Fig. 1

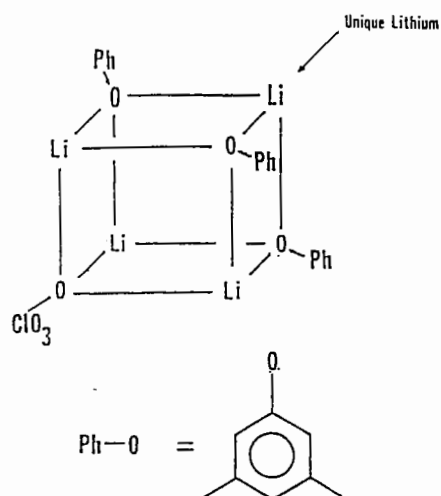


Fig. 2

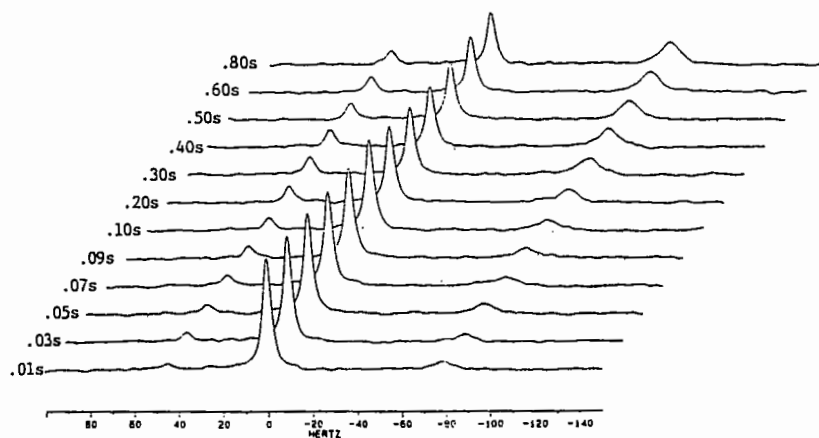


Fig. 3

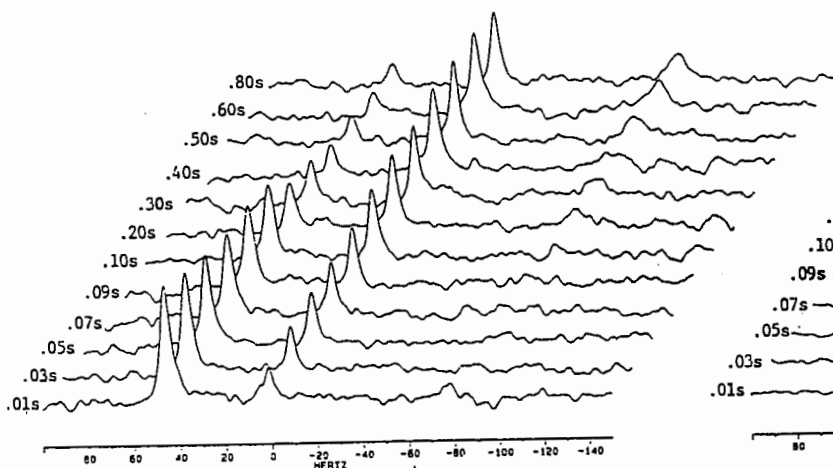


Fig. 4

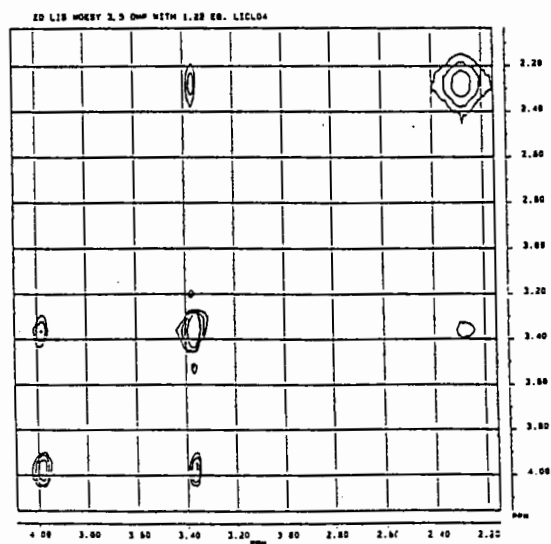
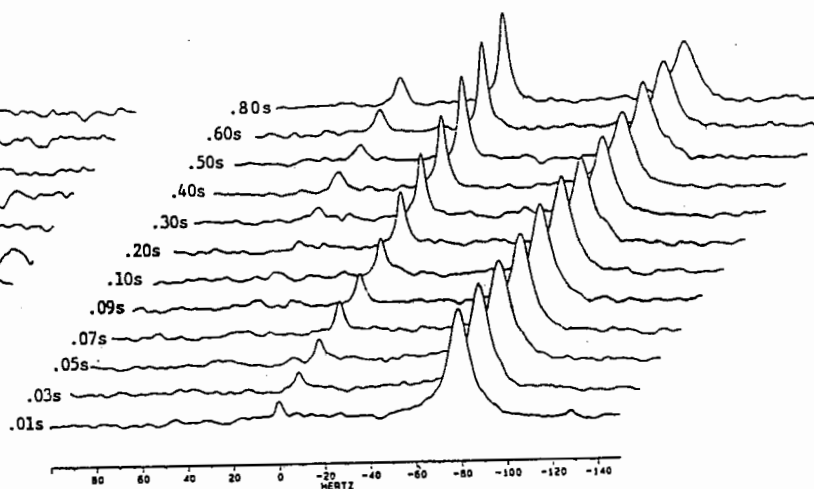
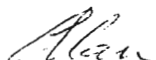


Fig. 5

Dr. B. L. Shapiro
Page 2
May 15, 1990

upfield LiClO_4 resonance and the downfield unique corner Li atom at about the same rate (figure 2). The unique resonance exchanges first with the resonance at 0Hz, and this then exchanges with the LiClO_4 resonance (figure 3). It is apparent from the figures 3 and 4 that no appreciable exchange occurs between the upfield LiClO_4 and unique resonance up to 0.10 sec at this temperature, so we ran a chemical exchange NOESY with a mixing time of 0.10 seconds. As expected, the only cross peaks observed were those between the LiClO_4 resonance and the 0Hz resonance, and between the 0Hz resonance and the unique downfield resonance (figure 5).

Sincerely,



Alan J. Benesi



Edward F. Rakiewicz



Lloyd M. Jackman

1. Ray Freeman, A Handbook of Nuclear Magnetic Resonance, pp. 200-201, Longman, 1988.
2. G. A. Morris and R. Freeman, J. Magn. Reson. **29**, 433 (1987)

Department of Chemistry
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Philip H Bolton
Wesleyan University

Middletown, Connecticut 06457
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6/10/90
(received 6/13/90)

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Please send vita and three references.

CSI 2T Applications

Shielded Gradients: Theory and Design

NMR imaging and localized spectroscopy depend on the use of pulsed magnetic field gradients. As these techniques have grown more complex, it has become apparent that eddy currents created in the magnet cryostat and other structures by pulsed gradients have become the chief limitation to many sophisticated applications.

Figure 1a illustrates the design problem for unshielded gradients. Figure 1b illustrates the shielded gradient arrangement. Figures 2 and 3 show the contours of constant flux for an unshielded and shielded Z gradient coil, respectively. This demonstrates that, for the shielded gradients, most of the flux has been kept away from the magnet bore.

The dramatic reduction of eddy currents which can be made over the conventional, unshielded gradients is shown in Figures 4a, b. These graphs show frequency as a function of time following the application of a long, constant amplitude gradient pulse which is suddenly cut off. Soon after cut off, a 90° pulse is applied and the complex FID recorded. The instantaneous frequency is then obtained from the FID and normalized by dividing by the frequency offset at the sample during the gradient pulse.

Figure 4a shows a typical decay of extra magnetic fields in a CSI 2T instrument caused by eddy currents in the conventional, unshielded gradient set with compression.

Figure 4b shows the decay of the uncompensated shielded Z gradient and Figure 4c shows the Z gradient decay with compensation. Note that the time scale for 4b and 4c is five times shorter than that for the unshielded gradients.

Fig. 1a.

CONVENTIONAL GRADIENT COIL

- Currents are constrained to flow on a single cylinder.
- There is only one degree of freedom.

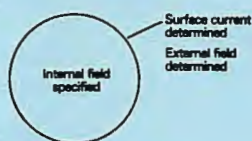


Fig. 1b.

SHIELDED GRADIENT COILS

- Currents on two cylinders.
- Two degrees of freedom.

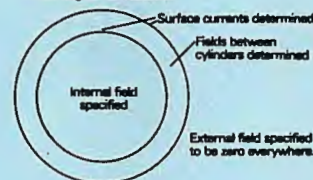


Fig. 1a—Design problem for unshielded gradients. The field inside the winding is specified to be a linear gradient and the current pattern on the cylinder is determined. **Fig. 1b**—Design arrangement for shielded gradients. The field inside the inner cylinder is specified to be a linear gradient and the field beyond the outer cylinder is specified to be close to zero. The current patterns on both inner and outer cylinders are then determined.

Fig. 2.

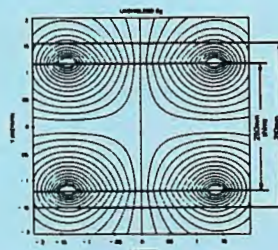
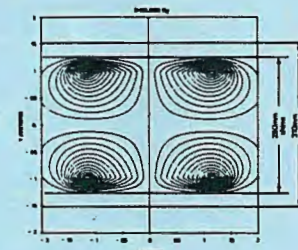


Fig. 3.



Lines of constant flux for Z-gradient. **Fig. 2**—Unshielded gradient. Note that flux lines extend well beyond the cryostat bore. **Fig. 3**—Shielded gradient. Flux lines are kept within the outer gradient cylinder.

Fig. 4a.

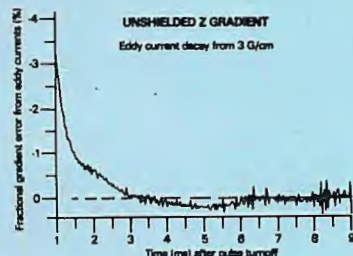


Fig. 4b.

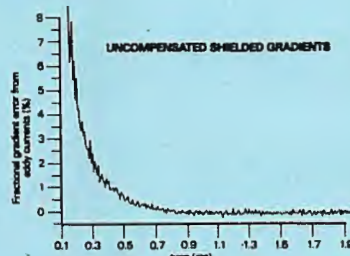
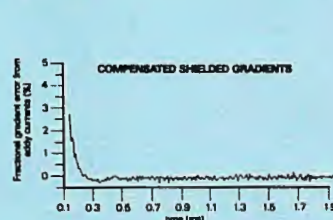


Fig. 4c.



Decay of field following application of square gradient pulse. **Fig. 4a**—Unshielded gradients. **Fig. 4b**—Shielded S150 gradient with no waveform compensation. **Fig. 4c**—Shielded S150 gradient with waveform compensation.

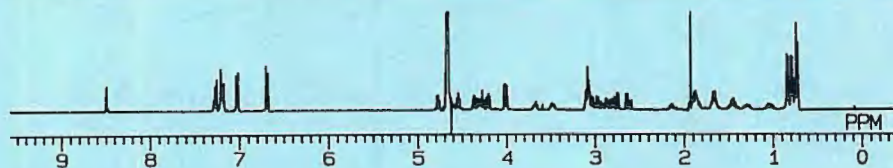
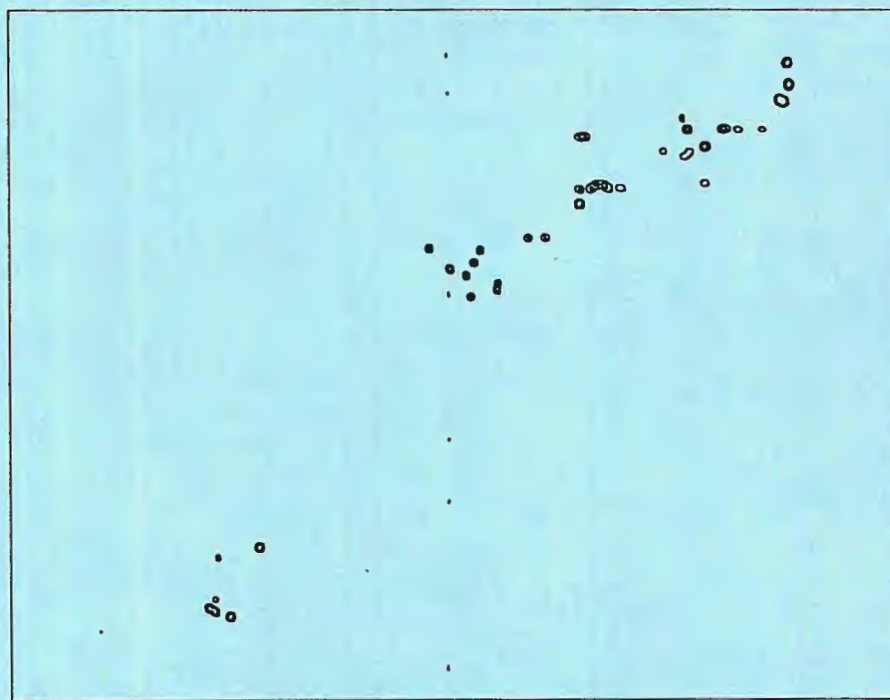


GE NMR Instruments

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For more information please contact:

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