THE
NMR
NEWSLETTER

No. 455
August 1996

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

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

2 For applicable digits, see detailed instrument specifications.

3 Bench cabinets are 17" wide.

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

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FORTHCOMING NMR MEETINGS

XVIIth International Conference on Magnetic Resonance in Biological Systems, Keystone, Colorado, **August 18 - 23, 1996**; Contact: ICMRBS, 1201 Don Diego Avenue, Santa Fe, NM 87501; (505) 989-4735; Fax: (505) 989-1073. See Newsletter 452, 59.

Missouri Magnetic Resonance Symposium (MMRS) and FACSS Meeting, Kansas City, MO, **Sept. 29 - Oct. 4, 1996**; Contact: (MMRS) Frank D. Blum, Dept. of Chemistry, Univ. of Missouri-Rolla, Rolla, MO 65409-0010; 573-341-4451 fblum@umr.edu. (FACSS) 198 Thomas Johnson Dr., S-2, Frederick, MD 21702-4317.

50 Years of NMR At Stanford, An International Symposium, Stanford, CA, **October 4, 1996**; Contact: Robin Holbrook: Tel: 415/723-6270; Fax: 415/723-2253; Email: holbrook@camis.stanford.edu; http://cmgm.stanford.edu/SMRL/50.html. See Newsletter 455, 38.

38th ENC (Experimental NMR Conference), Orlando, FL, **March 23 - 27, 1997**; Contact: ENC, 1201 Don Diego Avenue, Santa Fe, NM 87505; (505) 989-4573; Fax: (505) 989-1073.

4th International Conference on Magnetic Resonance Microscopy "Heidelberg Conference in Albuquerque", **Sept. 21-15, 1997**: Contact: E. Fukushima, The Lovelace Institutes, 2425 Ridgecrest Drive SE, Albuquerque, NM 87108-5127; (505) 262-7155; Fax: (505) 262-7043. See Newsletter 449, 37.

Additional listings of meetings, etc., are invited.

Molecular Sciences Department
Central Research Division
Pfizer Inc
Eastern Point Road
Groton, CT 06340

Central Research
NMR Spectroscopy

July 23, 1996
(received 7/24/96)

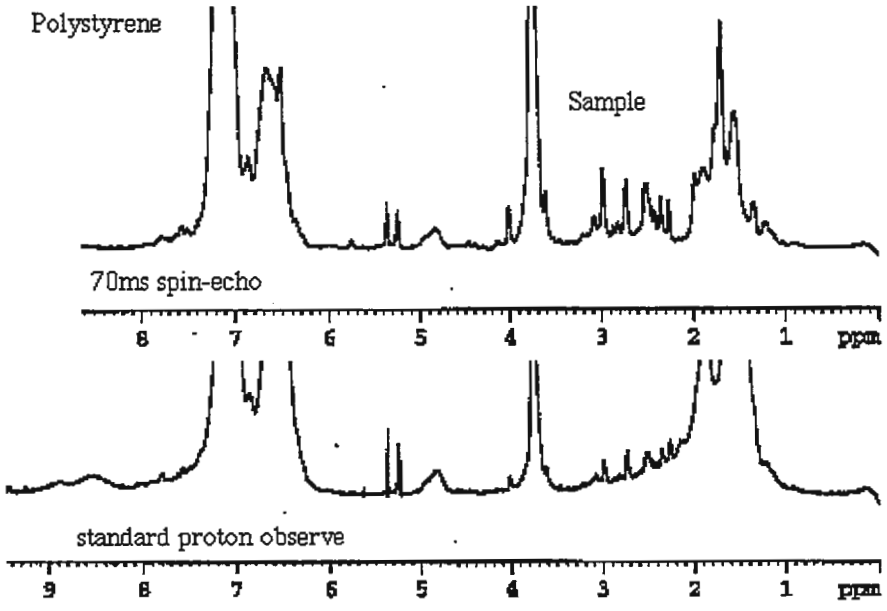
Dr. Barry Shapiro
The NMR Newsletter
966 Elsinore Court
Palo Alto, California 94303

Solid-phase spin echos

Dear Dr. Shapiro,

We have begun to ask whether solid-phase synthesis samples give proton data which is actually useful to a synthetic chemist who must interpret his or her own NMR spectra. Currently, solid-phase samples acquired under weak MAS conditions (liquids instrument, MAS probe, spinning rates of 1-3 kHz) of typical resins give less than ideal results: polystyrene resins obliterate the aromatic part of the spectrum, lines run together (of course), and then there is the problem of spinning sidebands. While it is trivial to distinguish sidebands from peaks when sitting in front of a spectrometer, it's not quite so easy when sitting in front of a single piece of paper which contains your data. Since real chemistry is very rarely clean, it is important to know which peaks the spectrometer put in the spectrum for you.

As part of a demo of MAS-capable instruments for solid-phase synthesis, we asked Dr. Sue Pochapsky of Bruker Instruments to acquire proton data on a sample with and without a spin-echo observe. We figured we could learn whether the broadness of the resin signals was due to chemical shift inhomogeneity or T_2, and possibly get some spectral editing out in the process. Much to our delight (see below) the spectral editing was pretty good:

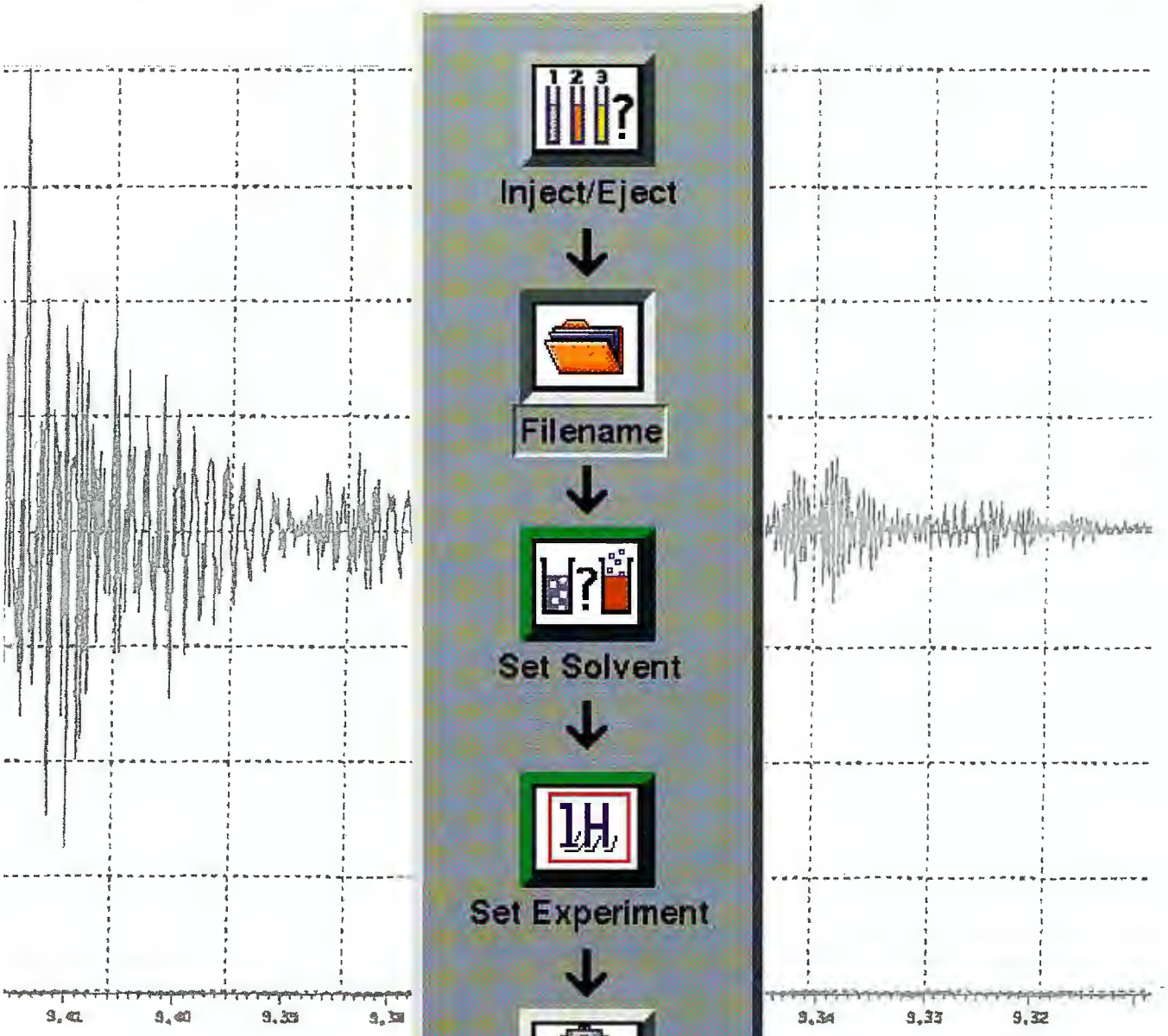


Sincerely,

Walt

Walter Massefski, Jr.
203-441-5962
203-441-4734 (FAX)
email: wwm@pfizer.com

It doesn't get any easier than this:



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UMEÅ UNIVERSITY
Department of Organic Chemistry
Ulf Edlund

1996-06-20
(received 7/1/96)

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

^{1}H-^{13}C Constant Time HSQC using sensitivity-enhanced gradient detection

Dear Barry,

In a collaboration with people from the biochemistry department, who are interested in the characterisation of the folding pathway of Human Carbonic Anhydrase I (HCA I), we have attempted to assign this protein. By employing ^{15}N-labeling for selective amino acids in combination with a number of triple-resonance experiments (cbca(co)nh, hncacb, cbcaco(ca)ha and hnco) on a ^{15}N/^{13}C uniformly labelled sample, it has been possible to obtain the sequential backbone assignment[1].

During the assignment procedure we were interested in getting a ^{1}H-^{13}C-CT-HSQC-spectrum of HCA1 without having to dissolve the ^{13}C/^{15}N-labeled protein in D_2O. To obtain an efficient water suppression and get the maximum sensitivity, we've chosen to use a sensitivity-enhanced gradient version of the originally suggested experiment[2]. The recorded spectrum shows that it was possible to suppress the water resonance down to the noise level (Fig. 1). Moreover, despite the long CT-period (26.6 ms) and the size of this protein it was possible to record a spectrum with acceptable S/N. The pulse sequence, which is displayed along with Fig. 1, used for the experiment was written on a Bruker AMX2 system and accordingly buffered acquisition had to be used.

1. Submitted to J. Biom. NMR.

2. G.W. Vuister & A. Bax, J. Magn. Reson. 98, 428-435 (1992)

Best regards

Ulf Edlund

Ingmar Sethson

Umeå universitet 901 87 Umeå. Telefon: 090-16 69 33. Telefax: 090-16 78 44. E-post: ulf.edlund@chem.umu.se

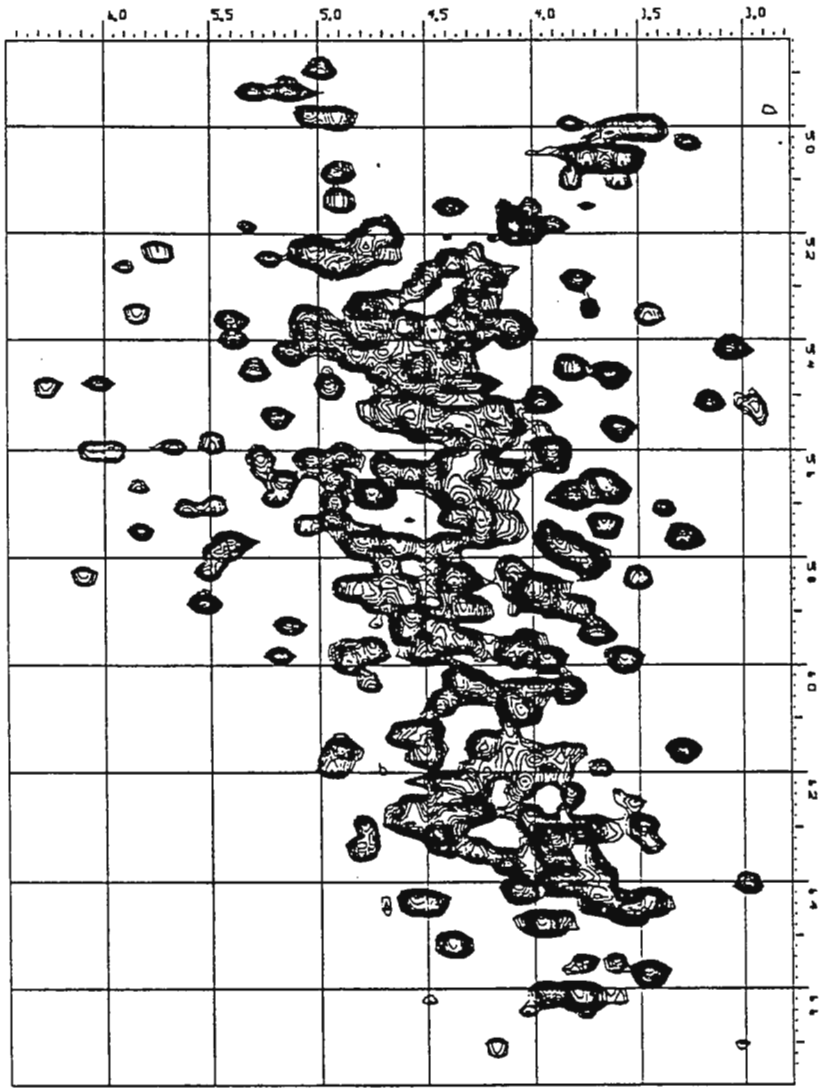


Fig. 1. Gradient-enhanced (^{13}C-^{1}H)-CT-HSQC-exp. on ^{13}C/^{15}N-lab. HCA I displaying the Hα-Cα-region.

```
;2D H-1/X correlation via double inept transfer
;phase sensitive using Echo/Antiecho gradient selection
;constant time version
;NB! NBL=2, NS=1

1 ze
2 d11
 d5*2
3 d11
 d5*4
4 d5*6
5 d5
6 d11*2
7 d1 hl1 dl0 dbl1 dbo do
 (p1 ph1)
 d4
 (d30 p2 ph1) (p4 ph9):d
 d4 unblank
; (d29 p1 ph2) (p3 ph3):d
 p1 ph2
 d16
8 2u:ngrad                ;homospoil gradient
 d27
 lo to 8 times l21
 2u:ngrad
 d16
 (p3 ph3):d
 d13 cpdb                 ;switch 15N-dec. on
 d0
 (d31 p2 ph1) (p13 ph1):dp3
 d13
 d20
 (p6 ph1):dp2
 d6
9 2u:ngrad                ;1st gradient for grad. selection
 d27
 lo to 9 times l21
 2u:ngrad
 d16
 (p13 ph1):dp3
 d13 dbo                  ;switch 15N-dec. off
 (d29 p1 ph1) (p3 ph7):d
 d8
 (d30 p2 ph1) (p4 ph9):d
 d8
 (d29 p1 ph2) (p3 ph8):d
 d4
 (d30 p2 ph1) (p4 ph9):d
 d4
 (p1 ph1)
 d21
 (p2 ph1)
 d13
10 2u:ngrad               ;2nd gradient for grad. selection
 d27
 lo to 10 times l21
 2u:ngrad
 d16 dl1 ;blank                ;delay for gradient recovary
 4u blank
 go=2 ph31 cpd
 d11 do st zd
 d5 ip8
 d5 ip8
 lo to 3 times nbl
 d11 wr #0 st0 zd
 d5 ip3
 d5 ip3
 d5 ip31
 d5 ip31
 lo to 4 times 2
 d5 ip7
 d5 ip7
 d5 ip8
 d5 ip8
 d5 ip31
 d5 ip31
 lo to 5 times 2
 d5
 lo to 6 times l6
 d11 if #0 id0
 d11 dd6
 lo to 7 times l3
exit
```

Phase Cycling Notation and Implementation

(received 7/25/96)

Alfred Redfield, Biochemistry Department, Mail Stop 009, Brandeis University, Waltham, MA 02254

For about a decade I've been using an algebraic notation for phase cycling in NMR. It has the advantage over the currently used strings of numbers, or ±y, ±y, of brevity, clarity, check-ability, and physical basis. I have no illusion that this notation (which is obvious) will be adapted by anyone else, but I'll get it off my chest anyway.

The physical basis for the notation is that first, almost all phase shifts now used are in steps of 90°, and can be represented by two bit binary numbers of which bit 0 represents a relative 90° shift, and bit 1 is a relative 180° shift; and that the different steps in a phase cycle are kept track of by a cycle counter that can usually be represented by a binary number whose bits are b0, b1,..bn for a total of 2^{n+1} steps in a cycle. Examples of the notation follow, where the left column is in conventional notation (with 0, 1, 2, 3 ≡ x, y, -x, -y ≡ 0°, 90°, 180°, 270° shift):

0123 Notation	Algebraic notation
0	(0, 0)
1	(0, 1)
2	(1, 0)
3	(1, 1)
0,1,2,3	(b1, b0)
0,3,2,1	-(b1, b0)
1,2,3,0	(b1, b0)+(0, 1)
0,1,2,3,2,3,1,0	(b2, 0)+(b1, b0)=(b2∧b1, b0)
0,2,2,0,0,2,2,0,2,0,0,2,2,0,0,2	(b3∧b1∧b0, 0)
0,2,2,0,2,0,0,2,1,3,3,1,3,1,1,3	(b2∧b1∧b0, b3)

The algebraic notation is to be interpreted as a two bit number (j, k) and gives bits j and k as a function of the bits b0... The + and - signs have their usual modulo 4 meaning, and ∧ is Boolean exclusive-or (xor).

That's all there is to it. The bits to the left of the comma obviously specify 180° shifts (all other bits being constant), and those to the right are 90° shifts. Clarity comes because any bit occurring in the data-taking phase has to be matched with a bit in a coherence-transferring pulse cycle (or a 180 shift matched by a 90 shift in an active 180° pulse), and the minus signs must each be used for cycling upstream of a non-shifted 180° focus. We also make States-TPPI phase-cycling explicit, with bits b5, b4 being 180°, 90° shifts in D2, b7, b6 for D3 and b9, b8 for D4. This notation is used in our program listings for compact in-line comments on any line that encodes a pulse or composite pulse.

Our pulse program assembler could most likely be programmed to read this notation directly, but instead, we maintain a library of 64 phase cycles in a small memory, each called by a number from 0 to 3f. These are programmed by writing an array in c-language that is "included" (in the technical sense of c-language) in the c-program that programs this memory. A typical line is: {0×2e,11,10,12,0,13,1}. This says that the 2e'th sequence is (b1, b0) + (b2, 0) + (b3, 1) = (b1∧b2∧b3, b0) + (0, 1) which is 1,2,3,0,3,0,1,2,3,0,1,2,1,2,3,0. Pairs of digits (only up to three pairs) after the enumerator (2e in this example) code simple pairs of bits, which are added modulo 4. The notation is 1x ≡ bx, x = 0-9; y ≡ y, y = 0-3. There are similar separate arrays for proton and heteronuclei pulses, and for 45° proton shifts.

A fairly simple assembler converts these (c-program) arrays into a file that is read into a memory so that the six hardware lines levels which encode one of the 64 phase sequences, and the ten hardware levels b0-b9, are converted into five output lines (90°, 180° shifts for protons, heteronuclei, and 45° shift). Hardware simplifies this since only two of the six bits b4-b9 occur in a given sequence (except for data recording where these six bits are handled in the usual way by the computer that generates them, to route data to the appropriate hypercomplex buffer). Also, bits b5, b7, b9 are restricted to 180° shifts, which can be xor'd together.

The operator can instruct the computer to omit any of the bits b0-b3, and also the 180° shift bits b5, b7, or b9, shortening the cycle without rewriting code. The ignored bits are then zero. The usual binary order of the toggling of the remaining bits does not need to be followed; I've considered randomizing the order but haven't done it.

The phase shifts encoded as described above are added by hardware to other independent shifts encoded elsewhere, for composite pulses. The six hardware lines that specify the phase cycle are also used for other purposes for non-rf-pulses, such as specifying gradients.

Although pulse sequences are, of course, constantly being changed, the library of phase cycles is seldom changed, because such a change requires revising the codes of many sequences. This works well, but a more powerful assembler that understands the algebraic notation directly and eliminates the 64 phase codes would be feasible if I had the time and inclination to write it.

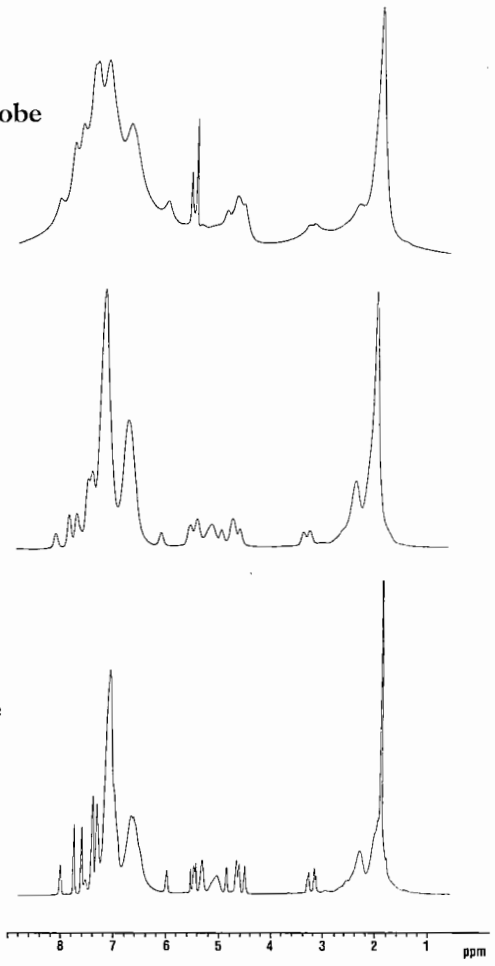
In conclusion, an unrelated complaint: When will you-all start writing decent comments, in-line, in your pulse programs (yes, you too, Varian-C users) so that outsiders can follow them, and so that you can, too, in 20 years? I mean really long complete ones, like "this is the first carbonyl 90" or "useless but required delay". The same goes for data-reduction macros.

Highest Resolution for Solid-Phase Synthesis

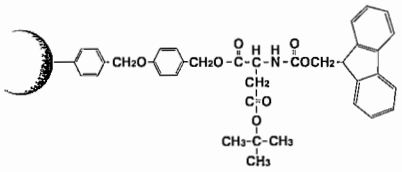
A. Vertical spinning probe

B. CP/MAS probe

C. ^{1}H Nano•nmr probe



^{1}H spectra of Fmoc-L-aspartic-ß(t-butyl) ester Wang resin in CD_2Cl_2 obtained utilizing a vertical spinning probe (A), a CP/MAS probe (B), and a ^{1}H Nano•nmr probe (C). J. Magn. Reson. (1996), in press.



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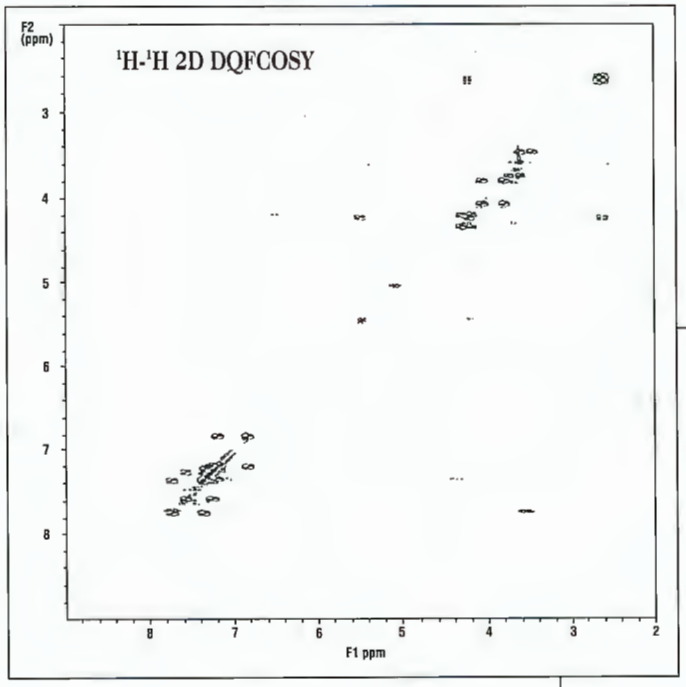
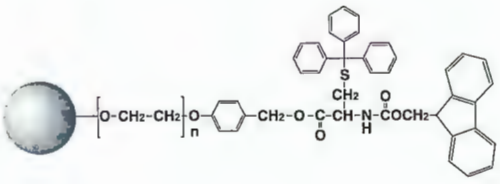
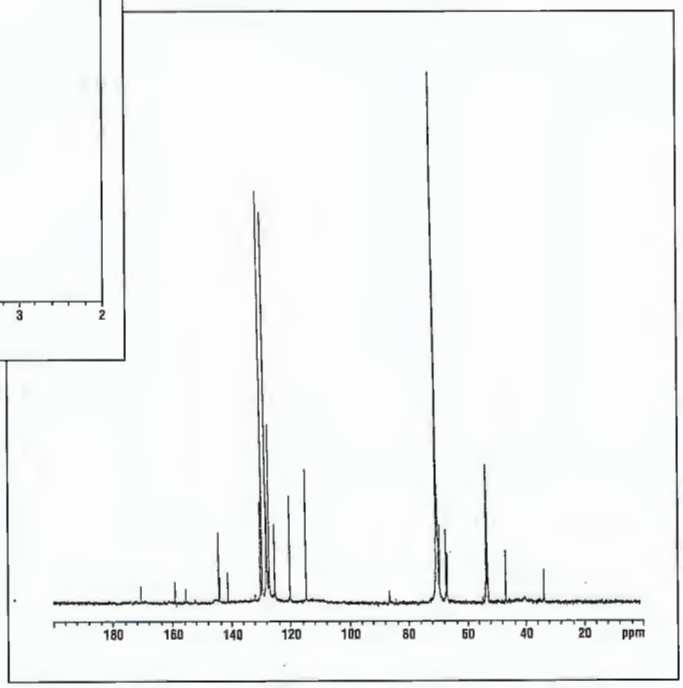
High Resolution ^{1}H 2D Spectra from Solid-Phase Synthesis Gels







High Resolution ^{13}C Spectra from Solid-Phase Synthesis Gels

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July 16, 1996 (received 7/24/96)

DECEPTIVELY SIMPLE SPECTRA (AGAIN)

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

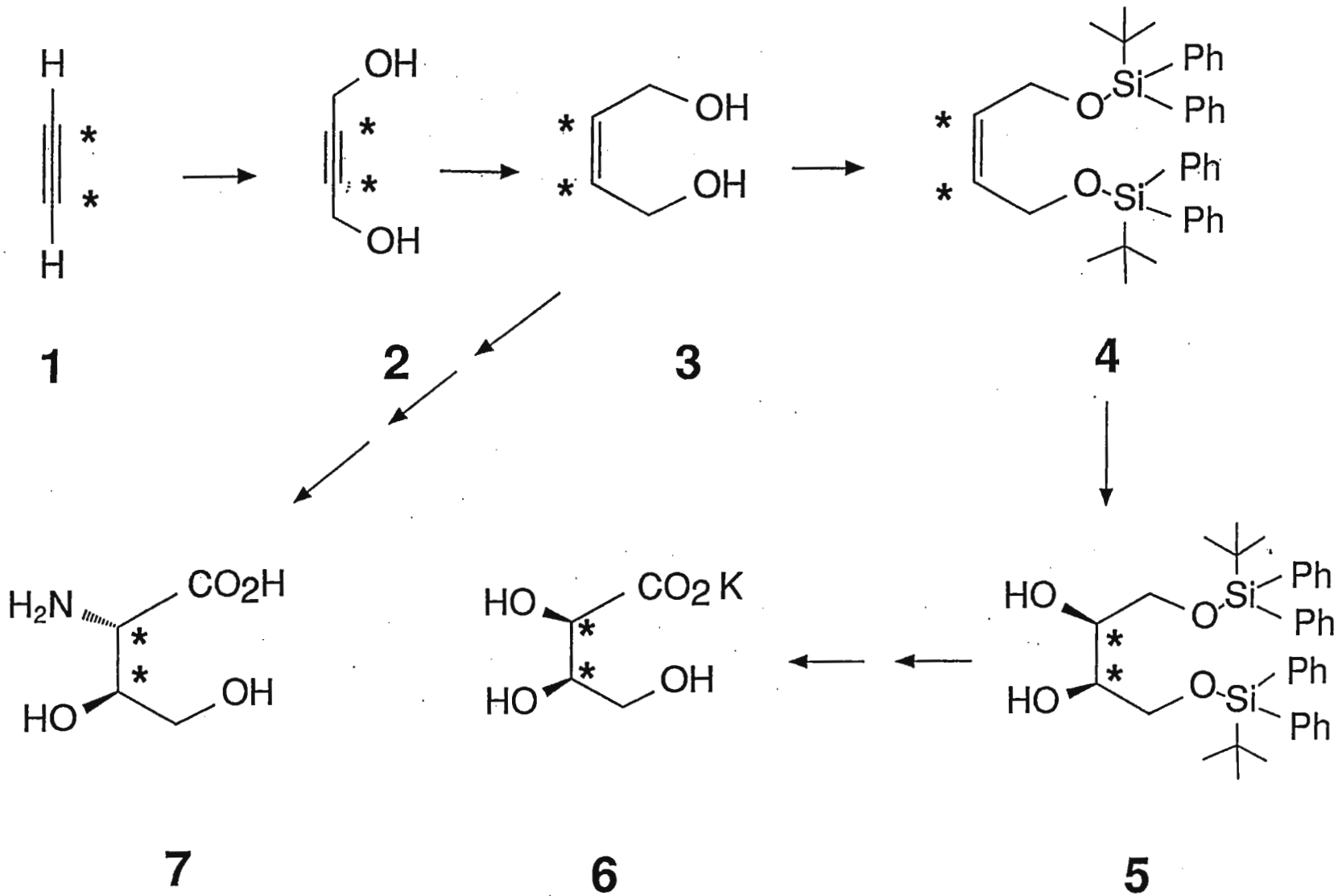
Dear Barry,

Every five years or so, it seems that people re-discover strong coupling effects and remember that they are important. In this case it was deceptively simple spectra (Abraham and Bernstein, Can. J. Chem. **39**, 216-230 (1961)). Ian Spenser in our department was looking at contiguously ^{13}C labelled acetylene derivatives (scheme 1). As long as the substitution was asymmetric, the spectra looked normal, but the symmetrical molecules became AA'X type systems. For instance, the CH_2 protons in [2,3-$^{13}C_2$]but-2-yne-1,4-diol (compound 2) are apparently a triplet due to ^{13}C coupling with a splitting of 1.3 Hz. In the related molecule propyne, the two-bond J_{CH} is -10.6 Hz and the three-bond coupling is +4.8 Hz. The deceptively simple spectra show a pseudo-triplet, with the average of these two couplings, hence the small observed splitting.

The question was then whether the spins themselves are deceived. We used SIMPLTN to simulate a series of DEPT-135 spectra of this AA'X_2 system as a function of the delay. The two carbons had the same chemical shift, the propyne values were used for the proton-carbon couplings, and the carbon-carbon coupling was 170 Hz. The results are shown in the figure. The modulation is complex, as one might expect. However, the first peak in the graph is at 50 ms, just where you would expect for a 10 Hz coupling. It seems that the spins are smarter than we are.

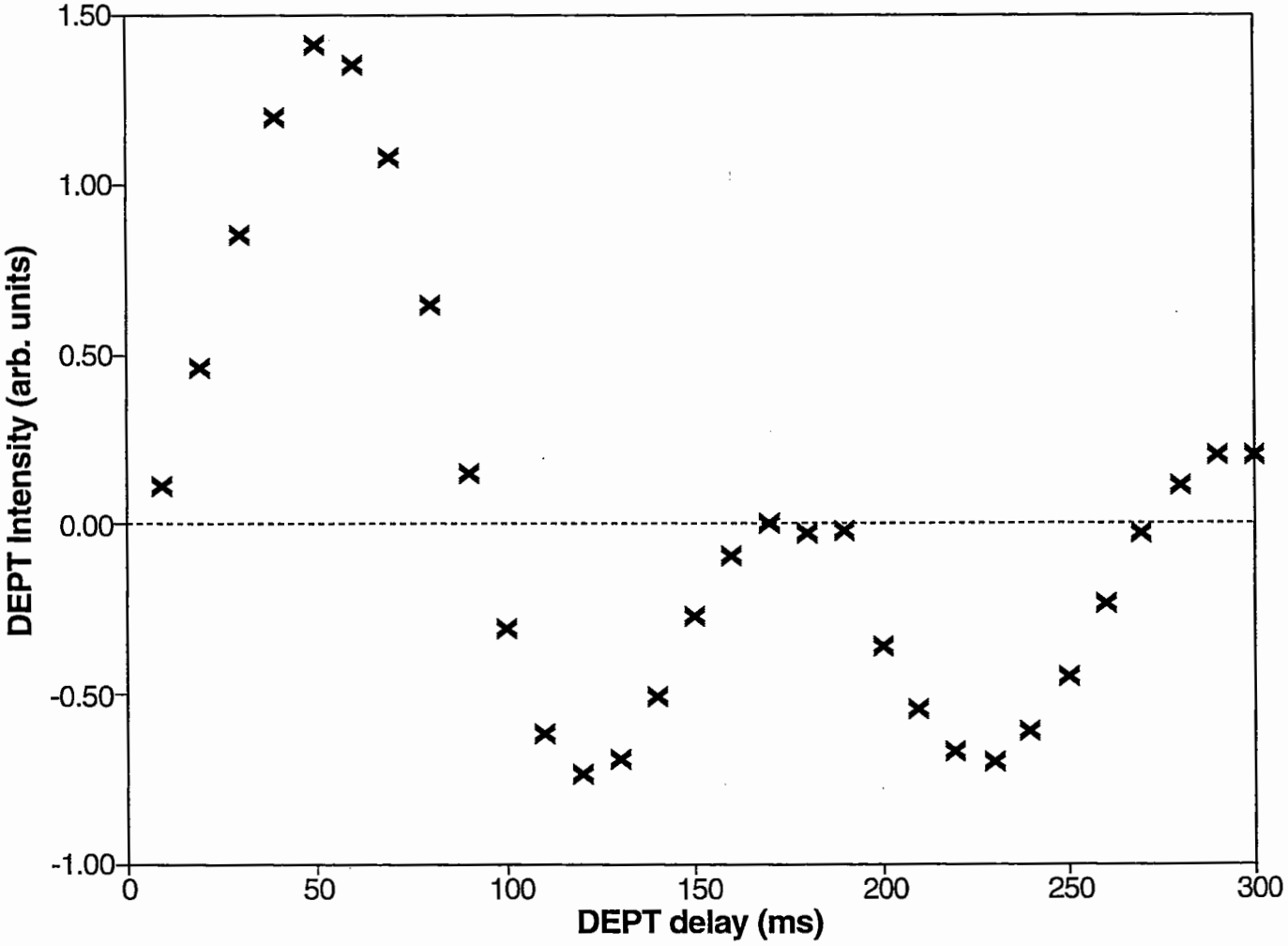
Yours truly,

Alex D. Bain
Professor of Chemistry
bain@mcmaster.ca



* = 99 % ^{13}C

Scheme 1


1.50
1.00
0.50
0.00
-0.50
-1.00
DEPT Intensity (arb. units)
0
50
100
150
200
250
300
DEPT delay (ms)

CARLSBERG LABORATORY **DEPARTMENT OF CHEMISTRY**

Flemming M. Poulsen

July 9, 1996

(received 7/9/96)

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

Dear Dr. Shapiro,

COMPUTER-ASSISTED NMR ASSIGNMENT TOOL PRONTO/3D^2 IS NOW FREELY AVAILABLE ON THE WWW:

The development of software for the assignment of NMR spectra of large molecules is a never-ending story. The latest version of Pronto/3D, (protein NMR tool), called Pronto/3D^2, contains the following new facilities:

- Display of molecular structures.

  Reading of PDB structure files. Display of structures as sticks, spheres, ball-and-stick and as cartoons. Display of NOE's between atoms. Export of display in a format ready for popular raytracing software. The display tools work most powerful with GL graphics on a Silicon Graphics workstation, but primitive displays are supported on any X11 display.

- Powerful interactive language, PILS (Pronto Interactive Language System).

  This language has a BASIC-like syntax. The language has a set of statements that makes it easy to write readable, structured programs. The data types include structures that resemble the records in the Pronto data base. It is possible to write programs that reads, writes, or updates the information in the data base. The programs can read in information into the data base, extract information and present them in an ASCII file or as graphics, and it can make sophisticated searches in the database.

To download a copy of the Pronto/3D program, please visit our home page:

`http://unidhp.uni-c.dk/~carlmk`

The software is available for all institutions.

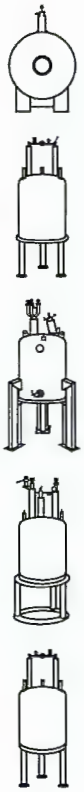
For further information, send an email to: carlmk@unidhp.uni-c.dk

Yours sincerely,

Flemming M. Poulsen and Mogens Kjær*

*Carlsberg Laboratory, Dept. of Chemistry, Gamle Carlsberg Vej 10, DK-2500 Valby, Denmark.
Phone: +45 33 27 53 25, Fax: +45 33 27 47 08, E-mail: carlmk@unidhp.uni-c.dk

Beware of imitations



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Magnetic field Strength (^{1}H-MHz)	Room Temperature Bore Diameter (mm)	Field Stability (^{1}H-Hz/Hour)	Maximum Helium Refill Interval (Days)	Minimum Operational Ceiling Height (m)
750	51	15	60	3.8
600	51	10	120	3.4
500	51	10	150	3.2
400	54	8	365	2.8
360	54	8	365	2.8
300	54	3	365	2.8
270	54	2.7	365	2.8
200	54	2	365	2.8
100	54	1	365	2.8
500	89	15	120	3.4
400	89	10	180	2.8
360	89	10	365	2.8
300	89	3	365	2.8
270	89	2.7	365	2.8
200	89	2	365	2.8
100	110	1	119	2.8

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The University of Virginia

ROBERT G. BRYANT
Commonwealth Professor

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28 June 1996
(received 7/5/96)

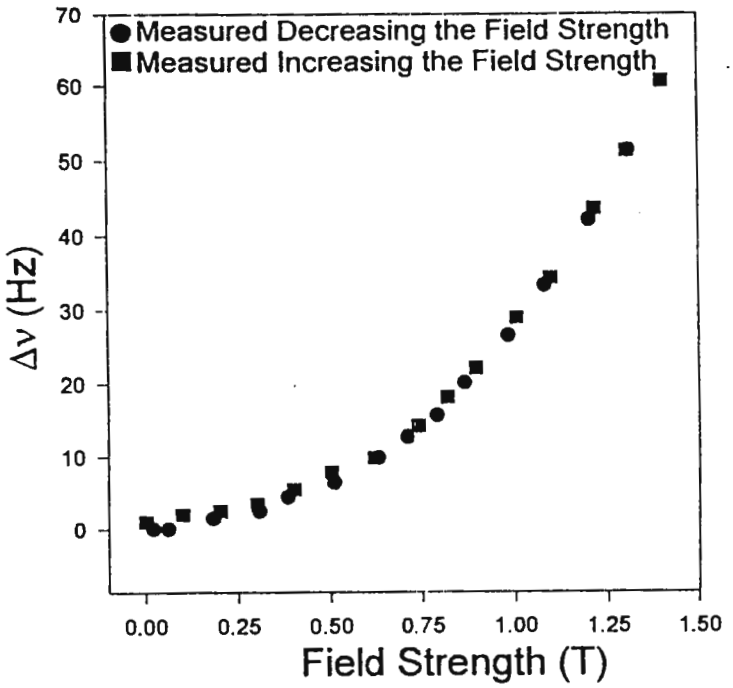
B.L. Shapiro
The NMR Newsletter
966 Elsinore Court
Palo Alto, CA 94393

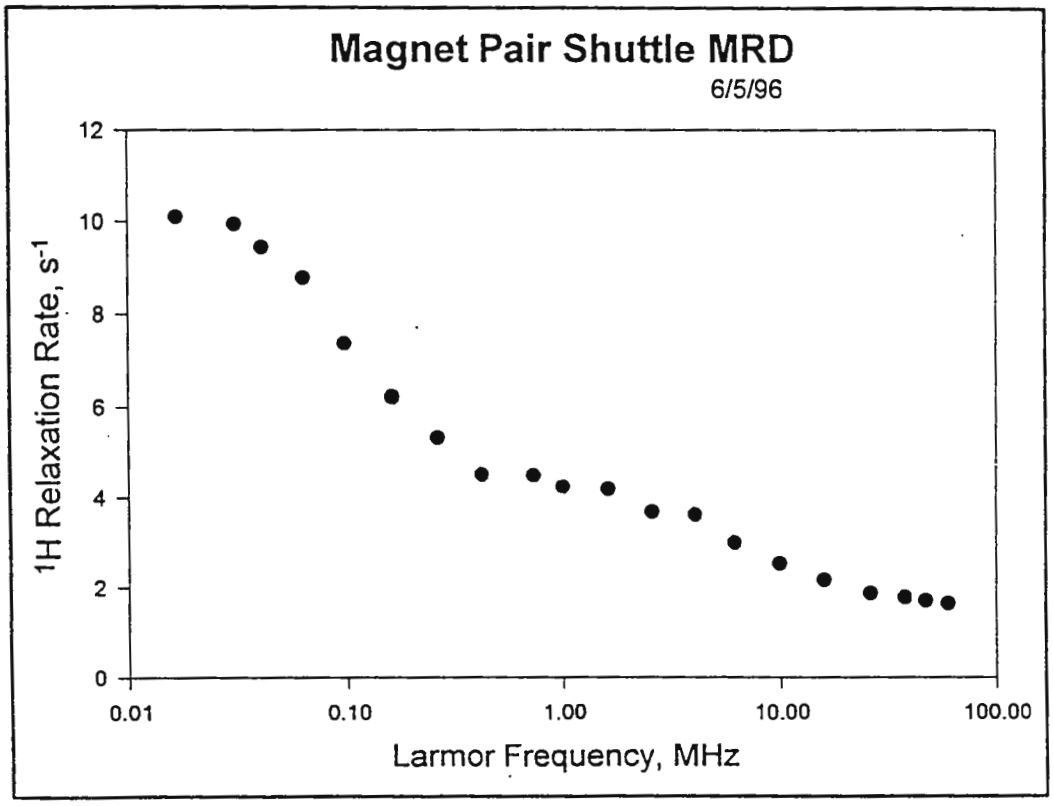
Dear Barry:

We have nearly completed construction of a new instrument that is unusual in that it uses two independent magnets which are physically close together but magnetically isolated. The high field magnet is a 7 T Magnex super conducting solenoid that has been reinforced to withstand the forces associated with an iron shield placed 212 mm from the Dewar bottom flange. Beneath the shield we have a GMW 4-inch electromagnet which we may ramp from 0 to 1.6 T. The idea of this bizarre configuration is to measure the magnetic field dependence of spin-lattice relaxation rates by polarizing the spins in the high field, shuttling the sample to the low field where it will relax for a variable time, then returning it to the high field where the magnetization may be detected with high resolution and sensitivity.

The resolution of the high field magnet is really quite good. We have no trouble achieving 10 Hz linewidths on nonspinning samples and the resolution is not degraded on successive returns of the smple to the high field region. This resolution, which is poor by some standards, is still better than 0.04 ppm and represents pure joy after working with our field switched relaxation dispersion spectrometer since 1982 where the linewidth exceeds the whole proton shift range by a factor of nearly a factor of ten. More important, the sensitivity of the new instrument is that appropriate to a 300 MHz spectrometer. Our field switched instrument by comparison uses a 30 MHz soak field and a 7 or 21 MHz detection field and the resultant sensitivity is similar to what Hahn may have been excited about in 1950. In any case, the idea works well.

There is almost complete isolation between the two magnets. Ramping the low-field magnet from 0 to maximum field results in a chemical shift measured in the 7 T magnet of less than 100 Hz for protons and there is very little hysteresis as shown in the top graph. We are still testing the reliability of the relaxation rates using well characterized systems, but will shortly be looking at interesting samples. The standard test is the water proton relaxation in an aqueous manganese(II) sulfate solution. The data for a 0.2 mM solution are shown in the bottom graph. The low frequency inflection is from the electron-nuclear hyperfine coupling and the inflection frequency reports the electron T_1. The high field inflection is from the ω_s term of the electron-nuclear dipole-dipole term for which the correlation time is the rotational correlation time of the metal complex. These data are actually distorted a little bit at higher field strengths because the moving sample does not follow the effective field adiabatically when it drops through the shield. This has been fixed by adding a solenoid winding on the shuttle tube which maintains the field in an axial orientation when the sample moves through the shield region.





Shawn Wagner

Robert G. Bryant

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Pulse width	20 ms	20 ms
Duty cycle	Up to 10%	Up to 10%
Amplitude droop	5% to 20 ms typ.	5% to 20 ms typ.
Harmonics	Second: -25 dBc max. Third: -24 dBc max.	
Phase change/output power	10° to rated power, typ.	
Phase error overpulse	4° to 20 ms duration, typ.	
Output noise (blanked)	< 10 dB over thermal	
Blanking delay	< 1 µ s on/off, TTL signal	
Blanking duty cycle	Up to 100%	
Protection	1. Infinite VSWR at rated power 2. Input overdrive 3. Over duty cycle/pulse width 4. Over temperature	

Supplemental Characteristics:

Indicators, front panel	1. AC power on 2. CW mode	4. Overdrive 5. Over pulse width	6. Over duty cycle 7. LCD peak power meter
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AC line voltage	208/230 VAC, 10%, 1Ø, 47-63 Hz		

	3445	3446
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SmithKline Beecham
Pharmaceuticals

July 23, 1996
(received 7/26/96)

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

MR Microscopy of Guinea Pig Stifle Joints

Dear Barry,

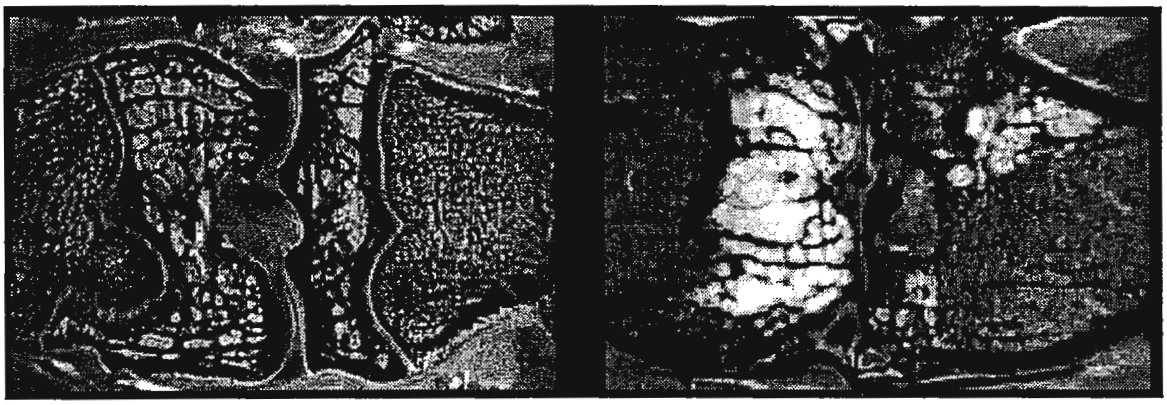
Osteoarthritis (OA) is a major joint disease, common among aged population, in which articular cartilage degenerates over a period of time, eventually leading to erosion of the joint surface. There are several animal models, reported in the literature, in which experimental degenerative joint disease has been induced in a variety of ways including metabolic, chemical, biomechanical and immunologic methods. In addition there are also reports of spontaneous cartilage degeneration in mature guinea pigs.

Since magnetic resonance imaging allows serial measurement of animals for a particular disease process, we have chosen to study the onset and the progression of cartilage degeneration in guinea pigs as a function of age using MRI. In order to better characterize the MRI markers for cartilage degeneration, we have initially begun this study using MR microscopy on ex vivo samples of stifle joints extracted from guinea pigs of different age groups.

MR microscopy images were obtained at 9.4 T with a resolution of 54 x 54 x 250 μm. A representative set of images of stifle joints from a 4 month old and a 22 months old guinea pigs are shown in Figure 1. Significant age-related and possibly disease-related differences are apparent in the images. They include a marked attenuation of the physis (growth plate), osteopenia (trabecular bone thinning/loss) and remodeling of the joint surfaces. Other differences between the images are being characterized and confirmed using histologic measurements.

709 Swedeland Road, PO Box 1539, King of Prussia, PA 19406-0939. Telephone (215) 270 4800. Fax (215) 270 7777.

Figure 1



4 months old Guinea pig **22 months old guinea pig**

We hope to report the characterestic changes before another 'colorful ultimatum' appears from you.

Regards,

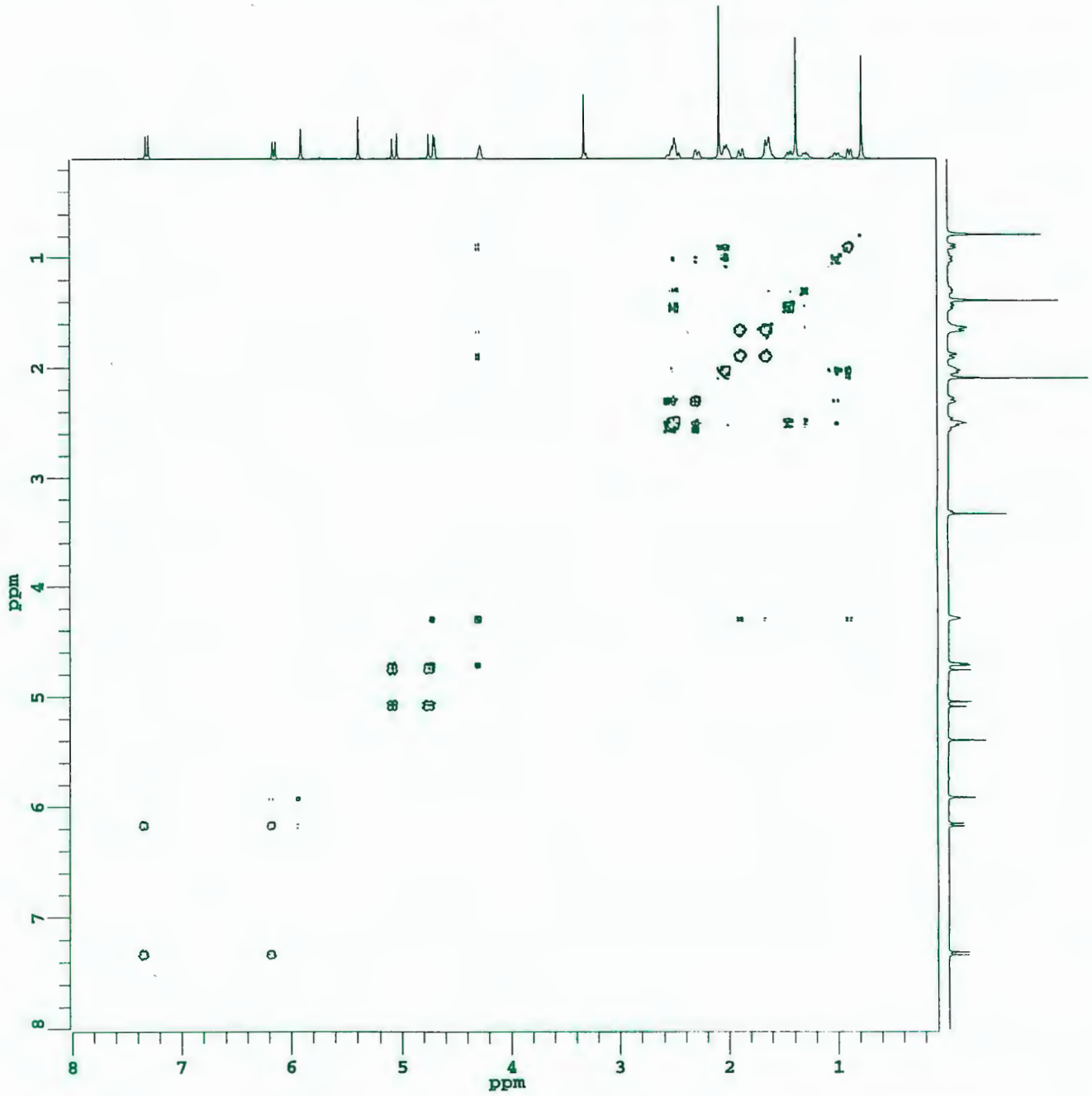
Sincerely yours

Rasesh D. Kapadia Robert Coatney Jeremey Bradbeer Hugh Zhao

Alison Badger Michael Lark Susanta K. Sarkar

Chemagnetics

Otsuka Electronics USA Inc.



This spectrum was acquired as 1024 rows of 1024 complex points each.

^{1}H-^{1}H PHASE-SENSITIVE PFG DOUBLE QUANTUM FILTERED COSY OF PREDNISOLONE 21-ACETATE

2607 MIDPOINT DRIVE • FORT COLLINS, COLORADO 80525 • 800-4-OTSUKA • www.otsuka.com

Shown here is the ^{1}H-^{1}H PFG DQFCOSY spectrum of prednisolone-acetate in DMSO-d_6 collected on the *Chemagnetics*™ 400 MHz CMX Infinity Spectrometer.

- Excellent signal-to-noise and resolution are obtained in this spectrum. Only two scans per row were necessary for the signal-to-noise seen here.
- Spinsight™ software features multiple viewports which can contain acquisition data and processed data of multiple dimensions. Parameters can be exchanged between the viewports for easy experiment setup.
- Using pulsed field gradients dramatically reduces experiment time.
- Compared to the traditional phase-sensitive COSY experiment, the double quantum filtration version suppresses single quantum coherences.
- This filtration simplifies the spectrum along the diagnonal thereby allowing for more straightforward structural analyses. Because we are observing higher order coherences, greater sensitivity is necessary to detect these weak couplings

Chemagnetics
Otsuka Electronics USA Inc.

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The NMR Newsletter - *Software* Reviews

Software Review Editor: **William B. Smith**, Texas Christian Univ., Fort Worth, TX 76129

ACD/CNMR and ACD/HNMR

and support programs.

from

Advanced Chemistry Development, Inc.,
141 Adelaide St. West, Suite 1501, Toronto, Ontario M5H 3L5, Canada
Phone: 800-304-3988. Email: acdlabs@acdlabs.com. Web site http://www.acdlabs.com

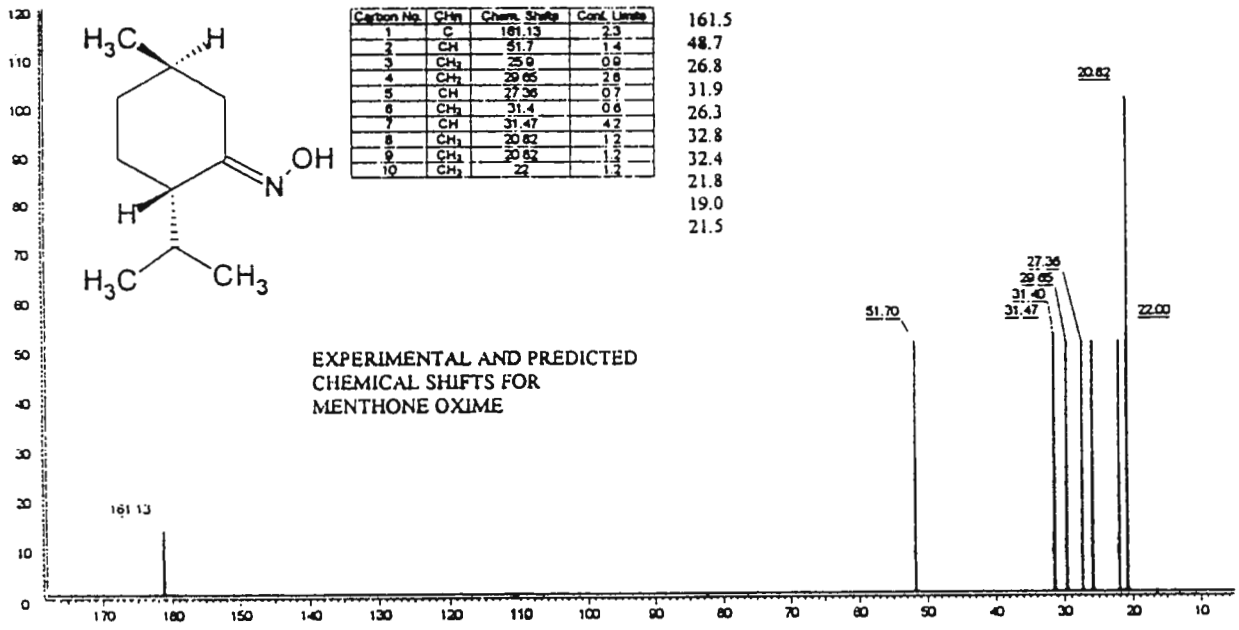
Price (all prices in US$) for total package: Industry $6175, Government $4625, Academic $3125. The package can be broken into smaller modules: for HNMR, Industry $4999, Government $3749, Academic $2499; for CNMR, Industry $1499, Government $1125, Academic $799. Requires a PC with Windows 3.1 or later (486 or above recommended.)

Developed in Russia, this software is a combination of modules designed to provide graphical structure input for the prediction of both carbon-13 and proton NMR spectra, including both chemical shifts and proton coupling constants. In addition to the drawing program, there is a dictionary which provides 48,000 systematic and non-systematic chemical names with their associated structures. Oriented towards organic, medicinal and biochemical interest groups, these names and structures are categorized into over 200 therapeutic areas as well as inhibitors for more than 500 different enzymes. There are ancillary units which also search for tautomers and calculate boiling points and logP (a measure of hydrophobicity); these units will not be reviewed here.

Each section of the total package is accompanied by a detailed users' manual. These are among the best written manuals I have seen and might well serve as models for others who write about complex computer software programs. However, much of the program is sufficiently user friendly that one may intuit the operation with considerable success.

For the C-13 prediction program, having drawn the structure with the ChemSketch program one simply clicks a button to generate the C-13 spectrum. The structure, spectrum and a table of chemical shifts are then transferred to an editing window from which the whole may be printed out. While in the spectrum window, one may do spectral editing for different types of carbons à la DEPT or may check assignments by clicking on various peaks whose assignments are then highlighted in the table. The algorithm used to predict the chemical shifts calls on a data base of 170,000 experimental values derived from 18,000 experimental spectra along with data from 3,000 structural fragments. The worse case scenario is ±5ppm. For the numerous cases I've examined, values were very much better than this. One may also add data of one's own to augment the data base. At present, one cannot access the experimental spectra directly. Plans call for this in the next upgrade.

There are difficulties in predicting diastereoisomeric carbon chemical shifts. Thus, the predictions for menthone show the isopropyl methyls with one shift value. However, the program recognizes the γ-effect of substituents. As an example, the spectrum for the *anti*-isomer of menthone oxime is shown below. The experimental values are ours. The *syn*-isomer shows the appropriate chemical shift response to the changed geometry. *Cis*- and *trans*-4-*tert*-butylcyclohexanol clearly show the effect of the 1,3-diaxial interaction. However, the two methyls in 4,4dimethyl-*tert*-butylcyclohexane both appear with the same chemical shift.



Carbon No.	CHn	Chem. Shift	Cont. Units
1	C	161.13	2.3
2	CH	51.7	1.4
3	CH_2	25.9	0.9
4	CH_2	29.65	2.8
5	CH	27.36	0.7
6	CH_2	31.4	0.6
7	CH	31.47	4.2
8	CH_3	20.82	1.2
9	CH_3	20.82	1.2
10	CH_3	22	1.2

161.5
48.7
26.8
31.9
26.3
32.8
32.4
21.8
19.0
21.5

As expected, problems get worse for proton spectra, for one must not only project chemical shifts but coupling constants as well. Again working with the huge data base and molecular fragments, the ACD/HNMR program does surprising well. However, the problem of diasteriotopic proton chemical shifts and, more particularly, the effects of axial vs. equatorial proton shifts in rigid six-membered rings, pose a still unanswered problem for the programmers. For molecules where these features are not present, the predicted spectra compare favorably with the C-13 predictions. In part, the problem will be aided when direct access to the experimental data base allows one to recall the experimental spectrum. Incorporating allowances for axial-equatorial effects will be a daunting task. It should be added here that one can construct representations of spectra with the eight-spin simulation program which is part of the package.

WBS

SEARLE
Physical Methodology
Chemical Sciences Analytical
4901 Searle Parkway
Skokie, IL 60077

A Method for using the Built-in X32 AMX-500 Console Monitor with a Silicon Graphics Upgrade

(received 7/5/96)

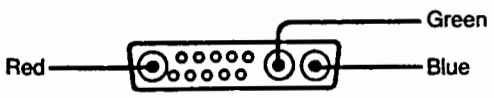
Dr. B. L. Shapiro
The NMR Newsletter
966 Elsinore Court
Palo Alto, CA 94303

Dear Barry,

We are in the process of updating our console mounted Bruker AMX-500 X32 computer with a Silicon Graphics Computer system. The normal upgrade is to an SGI Indy computer. (Our intention is to replace the Indy with an Indigo^2 as soon as we can obtain a second ethernet connection for the Indigo^2.) The upgraded system would thus normally use either the Indy standard 17-inch monitor sitting on a separate desk or installed in the console with a special custom made kit supplied by Bruker.

We were unhappy with the prospect of either using a monitor smaller than the present 19-inch monitor in the console or using a monitor on a separate desk. It appeared, however, that these were the only possibilities. Faced with this terrible choice, we began to investigate the situation in more detail.

Both the INDY and Indigo^2 are equipped with 13C3 female plugs for connecting to a monitor. The monitor which came with our Indy is a Sony GDM-17E11. Our Indigo^2 came with a Sony GDM-20D11. The Sony monitors also have 13C3 connectors. Although the literature supplied with the INDY and Indigo^2 does not appear to have a pin-out diagram for the monitor, the literature with the Sony shows the connections to be as indicated here:



Our AMX-500 console has a Mitsubishi 19–inch model C-6920 monitor. There are four BNC connectors to the Mitsubishi monitor, red, blue, green, and COMP/HD. Provision is made for the composite sync to be superimposed on the green.

We found that the AMX-500 monitor worked just as well when we disconnected the COMP/HD BNC. It thus appeared that the sync was, in fact on the green, just like the sync on the Indy and Indigo2.

We found that Black Box Corporation (phone 412-746-5500) sells cables for connecting Sun computers to third party monitors. This cable is available with the same 13C3 male connection required by the SGI on one end and the four male BNC connectors for red, blue, green, and sync required by the AMX-500 console monitor on the other end.

We found that this cable, Black Box part number EVMVDT02-0006-MM , works perfectly to drive the original 19-inch monitor with either an Indy or Indigo2 computer. (The sync cable was not connected). The size and resolution did not have to be adjusted for the Indigo2, but Mike Delk of Bruker found that the resolution had to be set to "high" on the Indy by giving the unix command: "setenv_monitor_high".

We are very happy with this arrangement. It gives us the nice in-console 19-inch monitor and does not clutter up our work space. We thought that other AMX-500 owners who are upgrading their systems might also like to know about this alternative solution.

We also had a 5-foot cable custom made with only the required three wires (Black Box reference CBCC28546). Although this cable works, the colors are distorted. The distortion is not corrected by extending the cable length to a total of six feet.

With best regards,

Roy H. Bible, Jr.

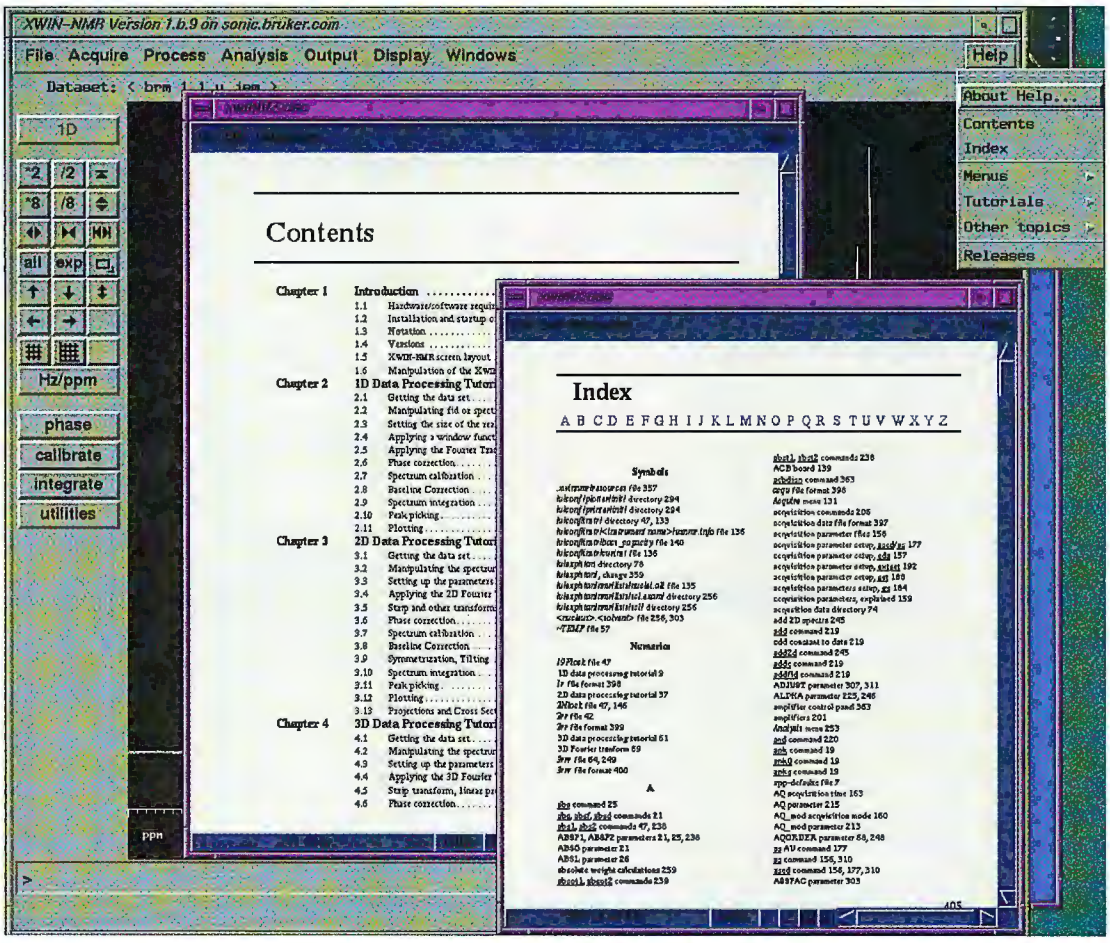
Robert W. Dykstra

The NMR ~~Re~~ evolution advances...

BRUKER

XWIN-NMR™ Software:

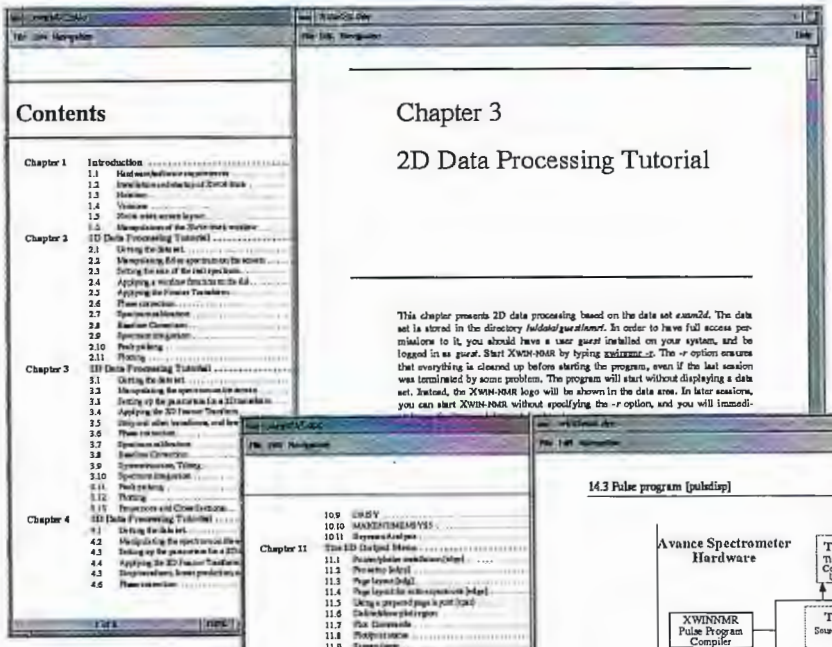
On-line help is just a click away!



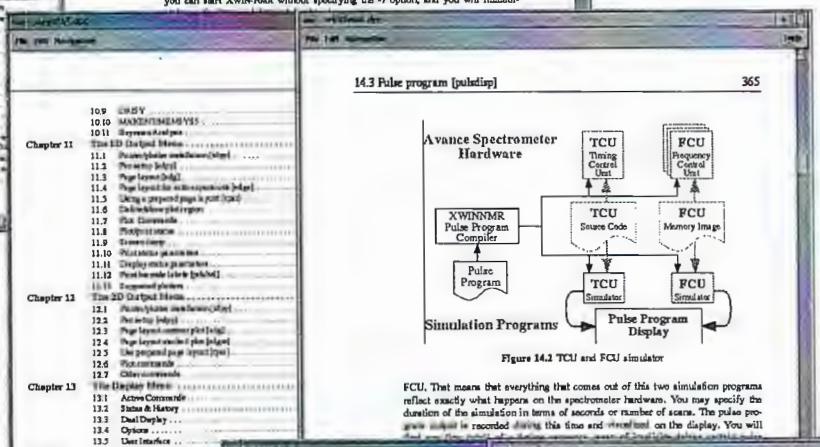
Bruker's new NMR software package, *XWIN-NMR*™, comes complete with an **on-line** manual. *XWIN-NMR*™ uses the Frame-Viewer utility to display the manual. Additionally, the table of contents and a keyword index for the manual are organized as hypertext for fast display of a desired item. Now "help" is just a click away!

BRUKER

...The NMR Revolution advances



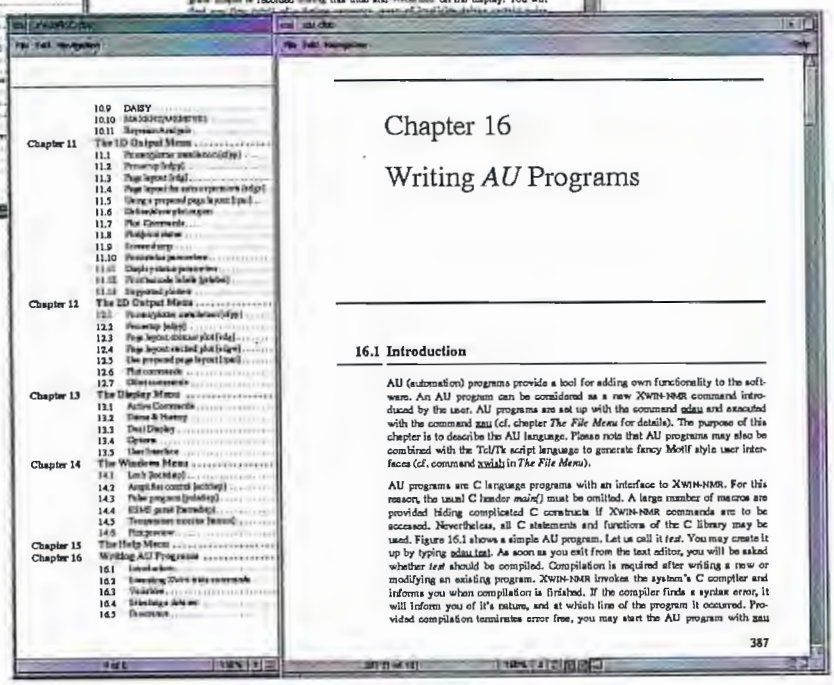
Here are some pages from the on-line manual, showing the Table of Contents that was used to find the page.



Top: Shows the tutorial help available with *XWIN-NMR*™ for 2D processing.

Middle: Explains functionality of the pulse program display software.

Bottom: Explains the fundamentals of writing and using AU programs.



For more information on *XWIN-NMR*™ software and our other new products, contact your local sales representative.

See us on the WEB at http://www.bruker.com

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July 12, 1996
(received 7/15/96)

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

Sorbate Induced Changes in the ^{29}Si MAS NMR Spectrum of Highly Siliceous MCM-22

Molecular sieves are microporous materials with three dimensional framework structures of tetrahedral (T) atoms, such as Si, Al, P and B that are interconnected through oxygen atoms. MCM-22 is a relatively new molecular sieve with unusual and unique structural characteristics. The framework topology of MCM-22[1] is comprised of two independent pore systems. One is defined by two dimensional, sinusoidal channels, and the other is comprised of large supercages whose inner free diameter (7.1Å) is defined by 12-membered rings with inner height of 18.2Å. The MCM-22 framework contains a very unusual T-O-T chain that passes through a modified dodecasil-1H (DOH) [$4^3 5^6 6^3$] cage, resulting in a framework T atom "buried" inside the cage and a small [4^3] cap on the top of the cage. These caps are oriented in such a way that bonding occurs through these apical atoms. The sensitivity of MAS NMR to short range ordering, local geometries, and symmetry in microcrystalline molecular sieves makes it an indispensable and complementary tool in studying subtle structural features and perturbations that are undetectable by traditional diffraction techniques.[2] In fact, recent work on MCM-22 and related materials involving NMR techniques has broadened our understanding of many of the structural intricacies of this new class of materials. In this contribution we present our recent observations on the sorbate induced changes in the ^{29}Si MAS NMR spectrum of highly siliceous MCM-22.

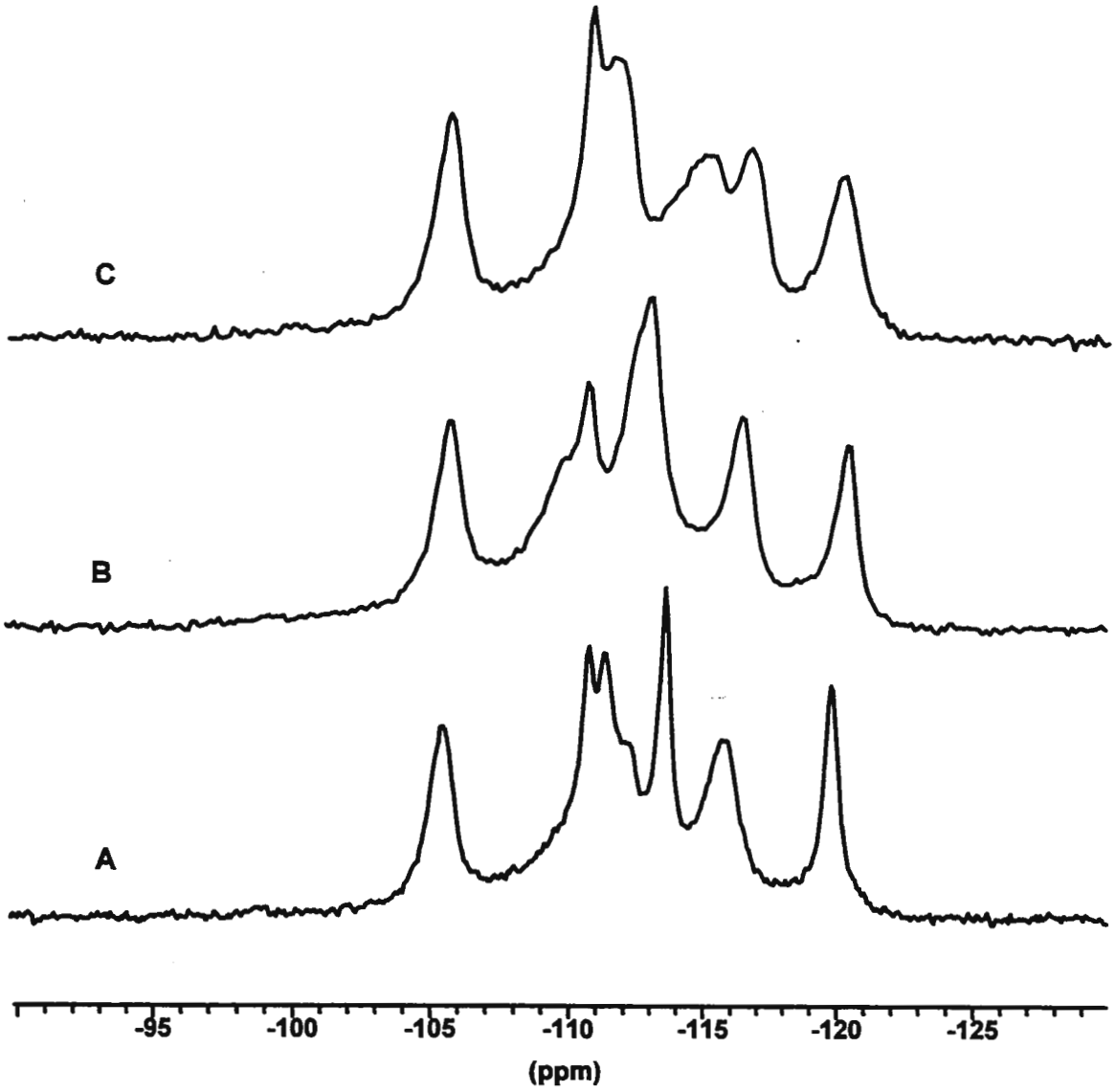
Shown in the attached figure are the ^{29}Si MAS NMR spectra of a highly siliceous MCM-22 (A) prepared by hydrothermal dealumination, and this same material after addition of 2,4-pentanedione (B) and toluene (C). These data show that addition of these organics cause very dramatic changes in the corresponding NMR spectra. The observed spectral changes are very specific in nature in that different sorbed molecules cause different and characteristic changes in the spectra. The sharpness of the spectra also indicate no loss of or change in crystallinity. These sorbate induced changes are reversible, the original spectrum (A) is obtained when the organic is removed by calcination.

The observed spectral changes are thought to be due to a modification or a perturbation of the lattice structure of MCM-22 similar to what has been previously reported for ZSM-5. In the case of ZSM-5 addition of varying amounts of different organics to highly siliceous ZSM-5 can induce a monoclinic to orthorhombic phase transition. However, in the present case of MCM-22 it is postulated that the presence of the organics is not inducing a phase transition but rather is causing some perturbations of the local geometry at specific T sites in the unit cell while leaving others unaffected. Details of the spectral interpretation, its impact on our understanding of the structure of MCM-22, and how it further supports the presence of the modified DOH cage in its framework structure will be published in the near future.

Best regards,

Gordon Kennedy

Gordon J. Kennedy



^{29}Si MAS NMR spectra of highly siliceous MCM-22 (A), and this same sample after saturation with 2,4-pentanedione (B), and toluene (C). ^{29}Si MAS NMR spectra were obtained on a Bruker AM-500 spectrometer at 99.35 MHz with 4.5 kHz spinning speeds, 60° excitation pulses, and a 60s recycle time. Chemical shifts are referenced to TMS.

References:

1 M. E. Leonowicz, J. A. Lawton, S. L. Lawton, and M. K. Rubin, *Science*, 264 (1994) 1910.

2 G. Engelhardt and D. Michel, *High Resolution Solid State NMR of Silicates and Zeolites*; John Wiley and Sons: New York, 1987.

Dr. B. L. Shapiro
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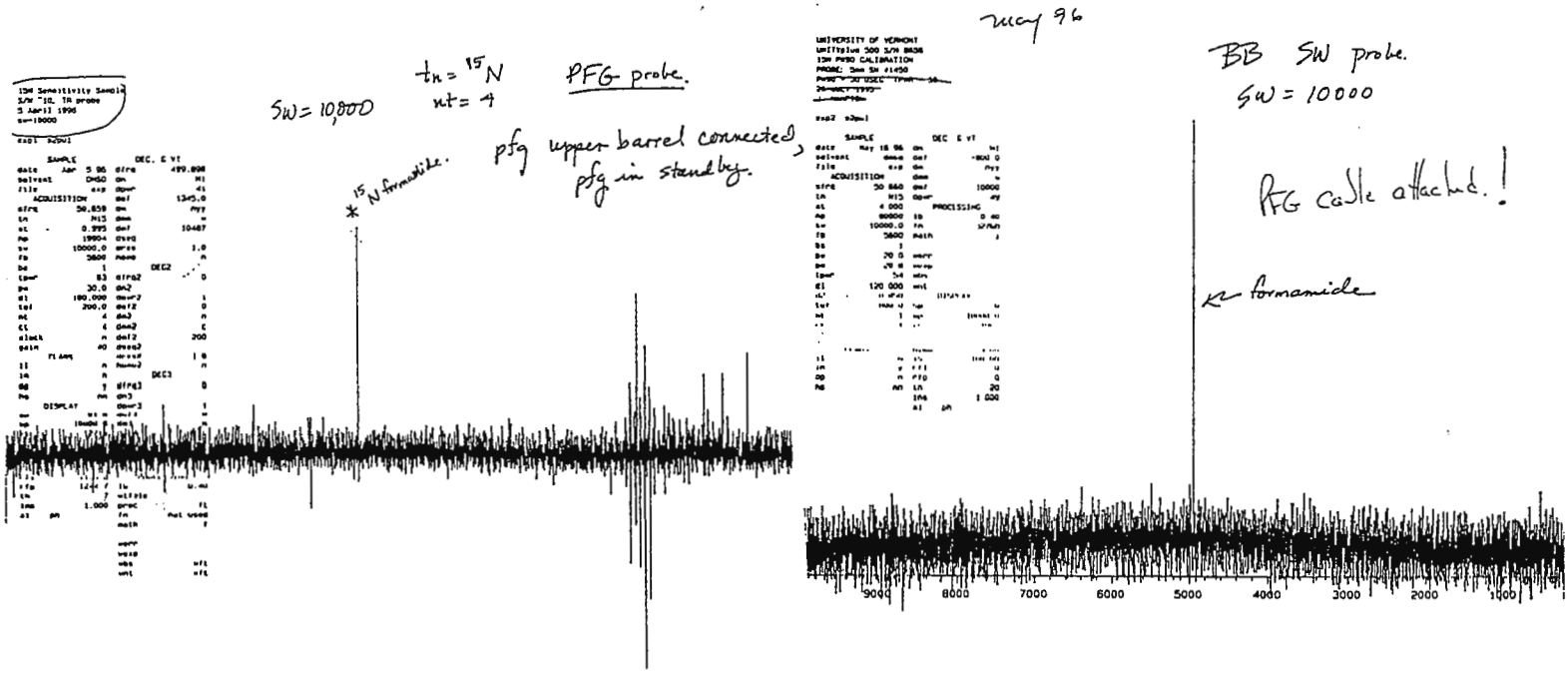
July 23, 1996
(received 7/27/96)

Broadband Lessons

Dear Professor Shapiro,

Our instrument has been up for a year now, and I would like to relate some of the problems we have encountered on the way to a happy, productive facility. As it turns out, our UNITY PLUS (soon to be INOVA, I hope) 500 worked like a charm from the outset with all the multichannel multinuclear indirect detect PFG pulse sequences we could throw at it. However, if we tried to run a routine ^{13}C or ^{15}N observe we always recorded nearly zero S/N. After going through broadband (BB) preamps as though they were bits of popcorn, it was finally determined that the software was switching a relay during tuning such that the broadband transmitter signal was being fed directly into the preamp. The easy work around for this is that we keep the BB transmitter cable disconnected except when we actually run a BB observe experiment. Along the way to discovering this software problem, which we hope will be cured in the next release, our BB probe lost the ability to lock and had to be completely rebuilt. During the time the BB probe was in the shop we used our PFG triple resonance probe for BB observe spectra and we noticed large "birds" in the ^{15}N observe spectra that made these data unusable (see below). Some determined sleuthing traced these signals to the cable connecting the PFG module to the upper barrel of the magnet. Thinking "AH HA!" we related to Varian that the problem would have to be fixed. But as it turns out, the return of our BB probe showed that probes without the PFG option do not pick up these problem birds. At least not to any large degree (see below). The moral of the story is "use the correct equipment for the job".



Best Regards,

Barbara A. Lyons

Jessica Dion

The NMR Newsletter - Book Reviews

Book Review Editor: **William B. Smith**, Texas Christian University, Fort Worth, TX 76129

Nuclear Magnetic Resonance

by

P. J. Hore

Oxford Science Publications (Oxford Univ. Press), Walton Street, Oxford OX2 6DP, England,
Oxford University Press Inc., New York, NY, USA.
1995. ISBN 0-19-855682-9 (Paperback). 90 pages. £5.99 ($9.95)

This slim volume is number 32 in the series Oxford Chemistry Primers (edited by R. G. Compton, S. G. Davies, and J. Evans) which "are designed to provide clear and concise introductions to a wide range of topics that may be encountered by chemistry students as they progress from the fresh*man (sic)* stage to graduation". The price of these books has been kept low by their being generously sponsored by Zeneca.

There are six chapters:- 1 Introduction; 2 Chemical shifts; 3 Spin-spin coupling; 4 Chemical exchange; 5 Spin relaxation; 6 Experimental methods; plus a bibliography and an adequate 2-page index. Literature references are given for the examples.

This an excellent book and should go a long way in meeting the needs of students (and others) who require more than the superficial accounts of NMR provided in general textbooks, but less than the treatment of specialized monographs. It concentrates on the principles and theory of NMR rather than the practice. Examples are given to illustrate particular points but there are neither worked examples of structural elucidation nor problems for solution. Most of the book is devoted to solution-state NMR, but solid-state spectra are discussed briefly, and while proton and carbon spectra provide most of the examples, other nuclei are given more than a mention. The discussion of exchange phenomena and the nuclear Overhauser effect are very good for a book of this size.

The book assumes at least a grounding in quantum chemistry, and while the mathematics is kept to a minimum, the relevant equations are included. I have only one criticism; the treatment of spin-spin coupling concentrates on first-order spectra. The justification is, I suppose, that with modern high field instruments nearly everything is first order; but this is not quite so, and a mention of (for example) AA'BB' spectra would have been useful.

The book can be safely recommended for purchase by students without the feeling that they are being condemned to a life of starvation or sobriety.

Peter Bladon
Gallowhill House
Larch Avenue, Lenzie
Glasgow G66 4HX
Scotland.

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(received 7/29/96)

July 18, 1996

ANSIG - Assignment of NMR Spectra by Interactive Graphics

Dear Barry:

DU PONT
MERCK

Research & Development

In the NMR structure determination of biological macromolecules we generate an obscene amount of data in the form of multiple 2D, 3D and 4D datasets. While there are several processing and display engines available (Felix, Nuts, Azara, nmrPipe/nmrDraw etc.) to transform the time domain data into frequency information, there are major deficiencies in further analyzing these processed data. It may come as a surprise to those not actively involved with this discipline to learn that there are no suitable packages from the large software vendors. Rather, individual laboratories have written their own software to solve specific problems. We were very fortunate to be involved with the early stages of ANSIG[1] (Assignment of NMR Spectra by Interactive Graphics) written by Per Kraulis (then at the University of Cambridge, and now at Pharmacia-Upjohn in Stockholm). We have now used this software in several projects[1,2] and I wholeheartedly endorse it as a powerful tool in bridging the gap between processed data and deriving the distance constraints used in 3D structure calculations.

ANSIG is written in FORTRAN 77 (that this dinosaur can understand!) and uses standard Silicon Graphics GL calls. It's power stems from efficient and flexible viewing of multiple windows of 2D planes from 2D, 3D and 4D matrices, coupled with a powerful internal programming language which allows tailoring of tasks to suit different types of experiments. This is something that the vendors have clearly not understood; with different physics governing the spectra of a bewildering array of experiments, a programmable interface is needed to logically deal with multiple nuclei, multiple types of correlation, and to move smoothly between data from different experiments. The (binary) electronic database containing positions, intensities, assignments, symmetry and connectivity information is extremely well trapped for inconsistent entry and is manipulated only within the program. Nevertheless an exportable ASCII mode is also available to allow transfer of information to other applications. Space limits me from further extolling the virtues but further information is available at two Web sites for the interested reader:

http://nirvana.bioc.cam.ac.uk/ at the University of Cambridge and
http://nmrsgi1.ncifcrf.gov/ansig/doc/ansig.html at the mirror site at NCI.

Please credit this contribution to the account of Chris Roe.

Best regards,

Peter

Peter Domaille

1.P. J. Kraulis, P. J. Domaille, S. L. Campbell-Burk, T. Van Aken, E. D. Laue *Biochemistry* **33**, 3515-3531 (1994); P. J. Kraulis *J. Magn. Reson.* **84**, 627-633 (1989).
2. T. M. Handel and P. J. Domaille *Biochemistry* **35**, 6569-6584 (1996).

50 Years of NMR

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James S. Hyde, Biophysics Research Institute, Medical College of Wisconsin
Oleg Jardetzky, Stanford Magnetic Resonance Lab, Stanford
Albert Macovski, Magnetic Resonance Systems Research Lab, Stanford
Harden M. McConnell, Department of Chemistry, Stanford
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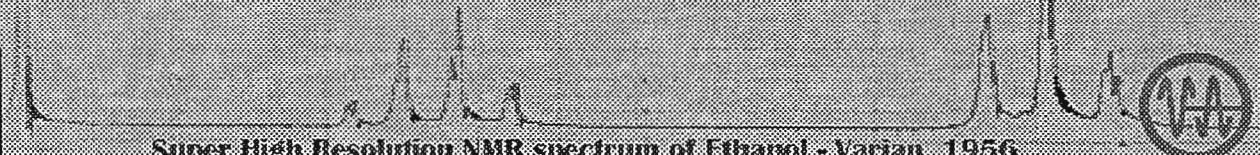
For more information contact Robin Holbrook.

Tel: 415/723-6270, Fax: 415/723-2253, Email: holbrook@camis.stanford.edu

http://cmgm.stanford.edu/SMRL/50.html



Super High Resolution NMR spectrum of Ethanol - Varian, 1956

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The position is grant funded for two years and is available immediately with a salary of $28K. Experience in solid state NMR is required, and some polymer knowledge is desirable.

Paul T. Inglefield Dept. of Chemistry, Clark University, 950 Main Street
Worcester, MA 01610-1477, USA

Phone: 508/793-7753; Fax: 508/793-8861; email: pinglefield@vax.clarku.edu

Position Available

Director of the Chemistry Department Instrumentation Laboratory,
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Requirements: Experience in modern multidimensional NMR spectroscopy, with a Ph.D. in Chemistry preferred. Ability to interact with students and faculty with diverse needs and interests. Interested candidates should submit two copies of a résumé, the names of four individuals who would be prepared to provide letters of reference on our request and cover letter to: Mr. Ken Hewitt, MIT Personnel Office, Room E19-230, 77 Massachusetts Avenue, Cambridge, MA 02139-4307.

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B. L. Shapiro
1 August 1996

Address all Newsletter correspondence to:

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No. 457(Oct.)	27 Sept. 1996
No. 458 (Nov.)	25 Oct. 1996
No. 459 (Dec.)	22 Nov. 1996
No. 460 (Jan.)	20 Dec. 1996

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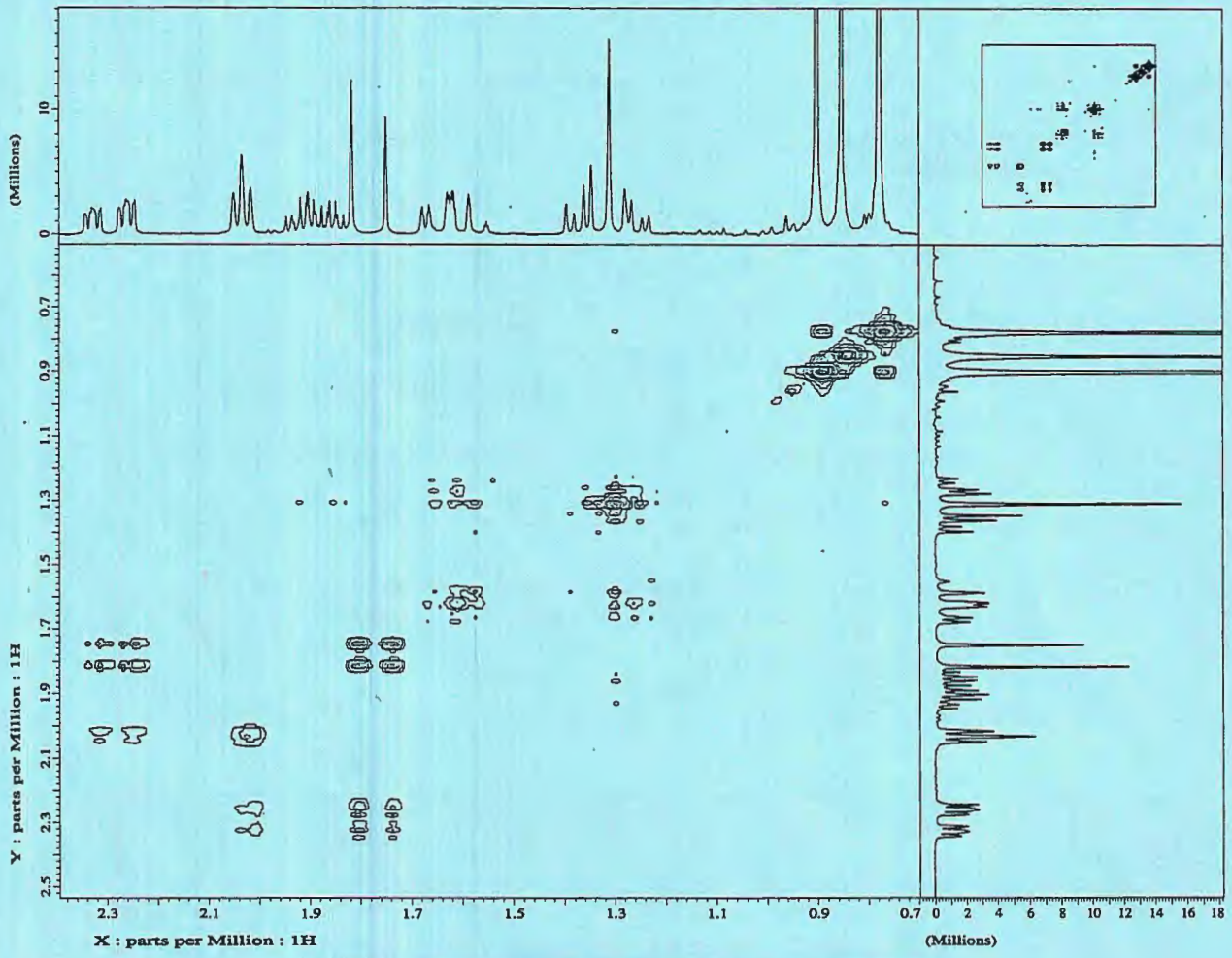

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