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NMR
NEWSLETTER

No. 474

March 1998

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FORTHCOMING NMR MEETINGS

Tsukuba NMR 98, Tsukuba Science City, Japan, **March 10 - 12, 1998**. Contact: Professor Yoji Arata, Water Research Institute; +81-298-58-6183; Fax: +81-298-58-6166; e-mail: arata@wri.co.jp; http://www.wri.co.jp

Symposium on Advances in NMR Applications, Monterey, CA, **March 22, 1998**; Contact: Nalorac Corporation, 841A Arnold Dr., Martinez, CA 94553; 510-229-3501; Fax: 510-229-1651; Email: sales@nalorac.com; http://www.nalorac.com.

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

Symposium on NMR Applications in Food Science and Technology, Dallas, TX, **April 2-3, 1998**; Contact: The Fine Particle Society, 2651 East 21st St., Suite 409, Tulsa, OK 7114; 918-747-6544; Fax: 918-743-7644; Email: fineparsoc.aol.com. See Newsletter 474, 49.

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

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

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

Continued on p. 50

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Department of Physics

February 6, 1998
(received 2/17/98)

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

NMR Student Apparati

Dear Dr. Shapiro,

For many years, students joining the Conradi and R. E. Norberg NMR groups have benefitted from a portable NMR demonstrator. The device can generate up to 3 rf pulses and runs at a fixed 14.3 MHz. The magnet is an adjustable permanent magnet with 2" pole diameter and 0.3" gap. Students use it to learn about nutations from rf pulses, FIDs, spin echoes, stimulated echoes, T_1, and T_2.

More recently we acquired a Hitachi/Perkin-Elmer R-24 B60 MHz permanent magnet cw spectrometer. We built a pulsed NMR rig around it, using the original 60 MHz oscillator and receiver amplifier (narrow-band crystal filter removed). This machine demonstrates all the above mentioned principles plus shows chemical shifts and J-couplings. Further, by using the Z-shim as a gradient coil, we do 1-D imaging and even demonstrate T_1-weighted images with a 2-capillary phantom (one with $CuSO_4$ added).

The original 60 MHZ proton probe, the air-driven spinner, the shim coils and controls, the temperature regulator circuitry and heaters, and the main power supply of the R-24B were used without change. The 'aquarium' pump for the spinner air was replaced by using house air, regulated at 3 psig (metric?). A dedicated, hard-wired pulse generator for up to 3 pulses drives the rf transmitter (oscillator, 2 gates, an AR 10 watt, 40 dB gain amplifier). Even 10 watts is overkill here and one could try a cheap 1 watt module. The receiver is from the Hitachi plus another 32 dB of wideband gain for a total of ~83 dB maximum. After phase detection is the usual low-pass filter driving a storage oscilloscope (so cheap now—try Hameg of Germany or the new Tek liquid crystal scopes). For anything requiring a computer (FT), we drive a digitizer board inside a PC. The storage scopes would also make dandy digitizing front ends. A nice feature of the rig is that it runs independently of the computer, using the computer only when a FT is needed.

We would be happy to send circuit details and a laboratory write-up describing the experiments that can be done with the re-energized R-24B and the portable NMR demonstrator. [No doubt others will come up with Varian T-60's, EM-360's, etc.]. The packet will detail the rf and pulse generator portions, but the digital world changes so fast that we will not describe our data acquisition hardware or software. We are happy to bear the photocopy expenses, but we ask for a stamped and self-addressed envelope. The packet weighs 7.4 oz. In case of (our) confusion, an email address will help.

Sincerely,

Mark S. Conradi Catherine F.M. Clewett R.E. Norberg

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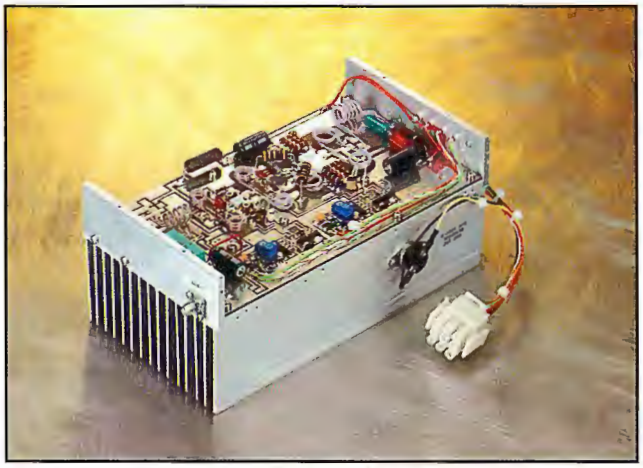

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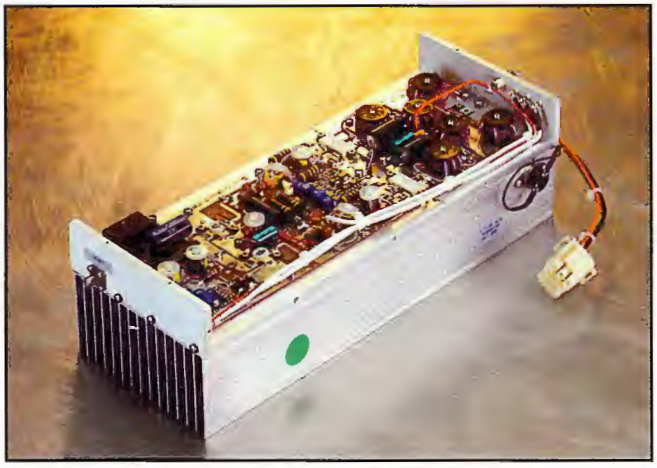

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SCIENTIFIC & MEDICAL PRODUCTS

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

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

FACILITIES

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

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

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

PRODUCTS

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

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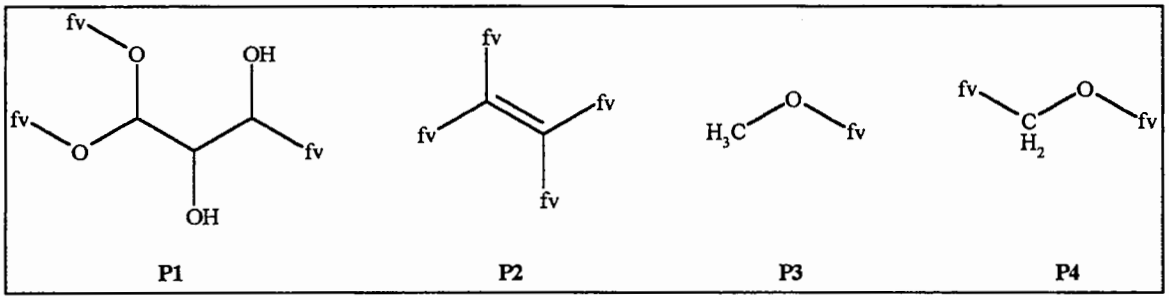
5 February 1998
(received 2/10/98)

Replacing Old CASE Programs

Dear Barry,

We have been using the DENDRAL programs in Computer Assisted Structure Elucidation (CASE) for over 20 years here at Lilly. These programs have served us well, but they are getting pretty old now and do not take advantage of all the new spectroscopic techniques that have been developed in the past two decades. In addition, they run on computers that have been "recycled" or soon will be. Thus we have spent much of 1997 looking for replacement programs.

Figure 1: Substructures Used in Generation of Structures for A201A



One of the programs we have evaluated is MolGen.[1] It seemed relevant to compare MolGen to the DENDRAL program CONGEN,[2] since these two programs appear to the user to work very similarly. Both use substructures as simple macroatoms to generate intermediate structures which are expanded in a later step in the structure elucidation process. I found a handwritten report dating from 1977 on a compound we knew at that time as A201A. For purposes of this contribution, we shall consider this problem to be the structure elucidation of a compound with the molecular formula $C_7H_{10}O_4CnRh$, where Cn and Rh were substructures elucidated by a combination of chemical degradation and spectroscopy. The substructures in Figure 1 were used as macroatoms, and the structure generation was constrained by ruling out peroxides and attachment of **P3** to **P4** (that is, substructures representing these constraints were put on BadList) and by requiring that Cn and Rh be attached to the rest of the molecule through oxygen atoms (these on GoodList). After expansion CONGEN yielded nine structures (not shown). Only one of these structures (Figure 2) was consistent with the results of ozonolysis of the compound, and these results were reported.[3] Subsequent single crystal X-ray analysis confirmed this structure and established the configurations at the optical centers.[4]

---

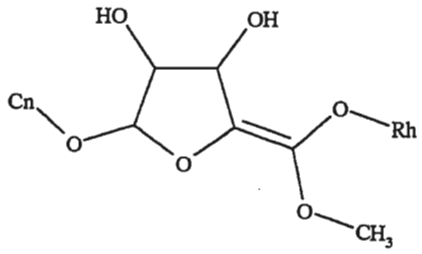
[1] Chemical Concepts GmbH, Weinheim, Germany. See Benecke, C.; Grund, R.; Hohberger, R.; Kerber, A.; Laue, R.; Wieland, Th. *Anal. Chim. Acta*, **1995**, *314*, pp. 141-147.

[2] Carhart, R.E.; Smith, D.H.; Brown, H.; Djerassi, C. *J. Am. Chem. Soc.* **1975**, *97*, 5755-5762.

[3] Kirst, H.A.; Dorman, D.E.; Occolowitz, J.L.;Szymanski, E.F.; Paschal, J.W. *Abstracts of Papers of 16th Intersci. Conf. on Antimicrob. Agents Chemother.* **1976**, #61.

[4] Kirst, H.A.; Dorman, D.E.; Occolowitz, J.L.; Jones, N.D.; Paschal, J.W., Hamill, R.L.; Szymanski, E.F. *J. Antibiotics* **1985**, *38*, 575-586.

Figure 2: Final Structure from CONGEN Generation



Reproducing this structure generation with MolGen led, to my great relief, to the same results. There were, however, some differences. For one thing, MolGen is a *lot* faster. It is impossible for me to recall now how long the CONGEN session took. For one thing, I was using the program on the Stanford University medical school computer over telephone lines. I can, however, report quite accurately how long the MolGen generation took: 0.17 sec on a 75MHz Pentium. Of course that generation was possible only after inputting all the substructures, but structure input is also faster and more natural to a chemist using the graphical interface of MolGen than it was with the keyboard input of structures required in the DENDRAL programs. Suffice it to say that MolGen is a much more usable program that CONGEN, especially since the latter program was written on a computer that probably doesn't exist anymore.

There are, of course, some problems with MolGen. One of the things that was particularly annoying about version 3, on which the above work was done, was the fact that the program was free to add protons to any substructure of the input list. For example, it might add a proton to **P4** (Figure 1) to yield either a hydroxymethyl group or a second O-methyl, even though the data clearly ruled out such possibilities. In the example above this problem was avoided by virtue of the fact that the substructures in Figure 1 account for all the protons of the molecular formula. A more recent version of MolGen (3.5) corrects this problem, and in fact provides nice ways to identify the distribution of protons and hybridization of atoms.[5] A second lucky aspect of the example above is that none of the atoms of the various macroatoms are overlapped. Such overlaps are not allowed in either CONGEN or MolGen. The DENDRAL group overcame this problem with the program GENOA.[6] The authors of MolGen have reported that they will solve this problem in a future version of MolGen.[5]

MolGen is a fine program, but it may not be the final answer to our problems. It does not automate the interpretation of data or the conversion of those data into substructures. It does not solve the problem of the ambiguous results we get from COSY and HMBC spectra. But programs that do perform those tasks are becoming available, and I have been trying some of them also. If Barry is willing to use space in this newsletter, I will continue to report my results here.

Doug Dorman
doug_dorman@lilly.com

[5] Benecke, C.; Grüner, T.; Kerber, A.; Laue, R.; Wieland, T. *Fresenius J. Anal. Chem.* **1997**, *359*, 23-32.
[6] Carhart, R.E., Smith, D.H.; Gray, N.A.B.; Nourse, J.G.; Djerassi, C., *J. Org. Chem.*, **1981**, *46*, 1708.

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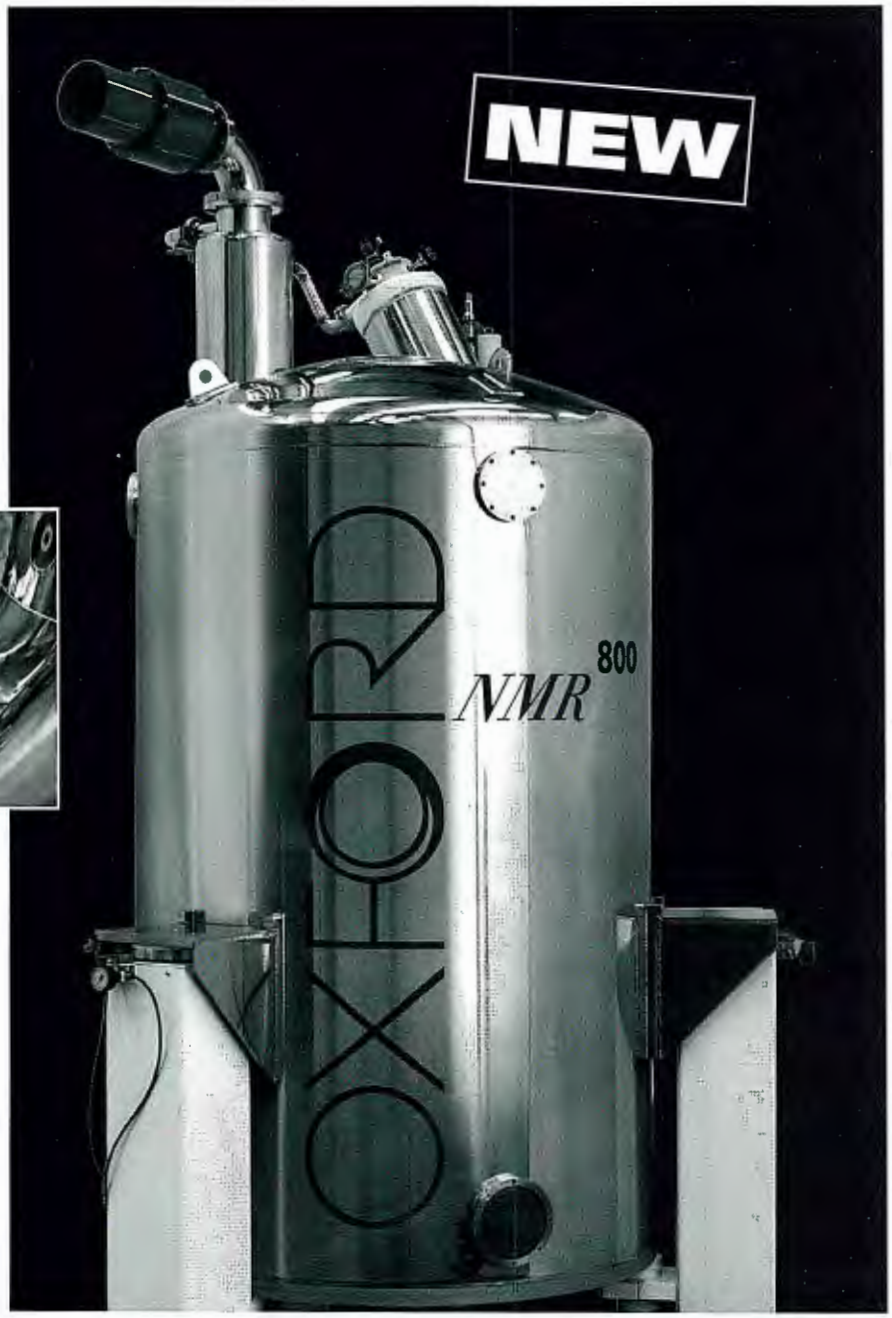

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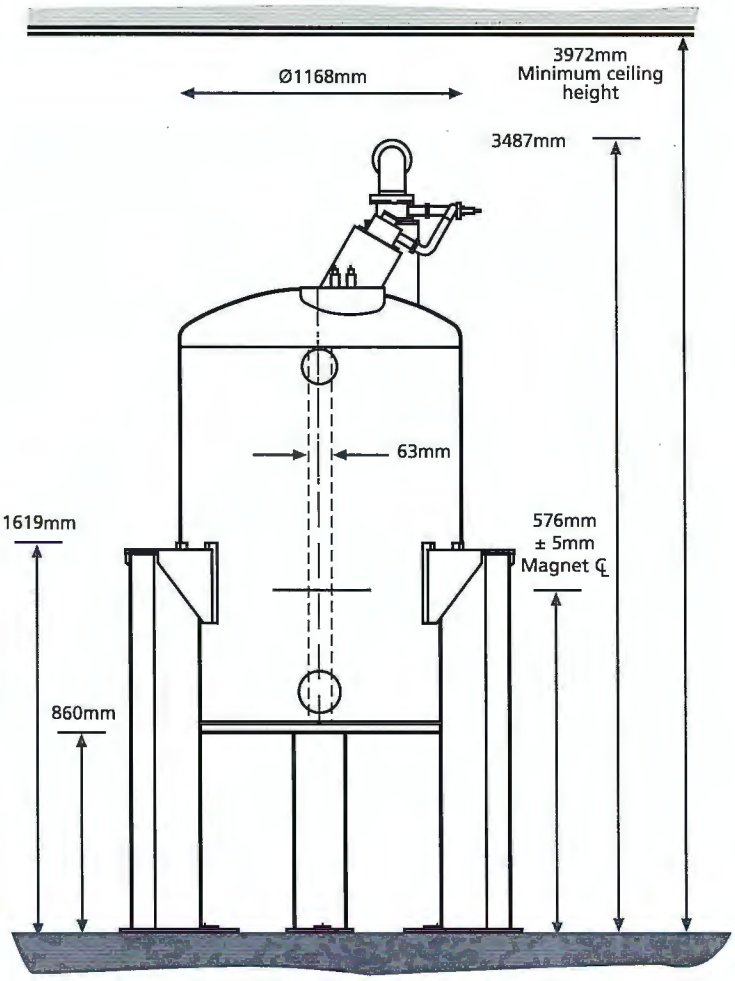

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Dr. Barry Shapiro
The NMR Newsletter
966 Elsinore Court
Palo Alto, CA 94303

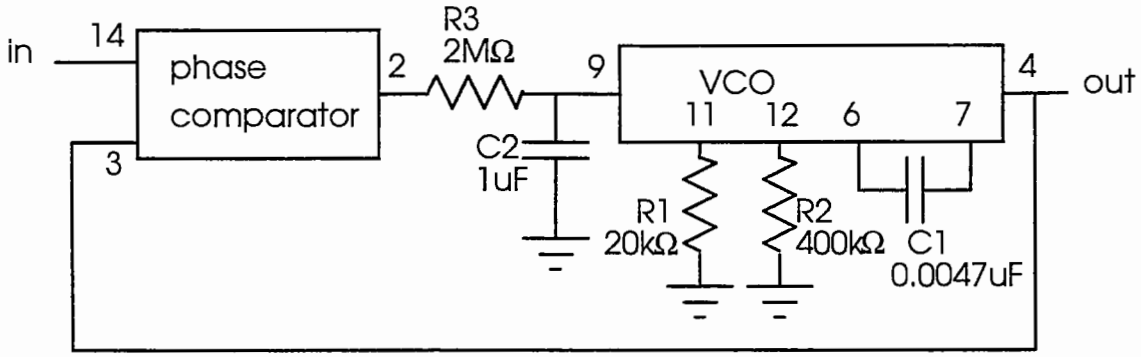
January 20, 1998
(received 1/26/98)

Taking the jitter out of spin-rate detection

Dear Barry,

Modern solid-state NMR experiments can provide detailed atomic-level structural information about a wide variety of systems, including biological macromolecules. Many such experiments rely on rotor synchronized pulses to defeat an MAS rotor's averaging of small homo- or heteronuclear dipolar couplings. The outcome of such experiments depends crucially on the ability of the spectrometer to reliably synchronize pulses to the rotor position. Most commercial systems do provide a method to actively gate the spectrometer pulse-programmer to a signal derived from the spin-rate detector, but many such signals suffer from excessive noise or jitter which may even cause problems with spin-rate control.

We have implemented a simple circuit which appears to be of significant help in cleaning up spin-rate detector signals. The circuit all but eliminates noise and significantly reduces jitter and costs under $2 in parts (plus power supply and housing). The circuit is based on a 4046 phase locked loop integrated circuit with just two capacitors and three resistors. Our circuit is based on one supplied in the Motorola 14046 technical data notes and is shown below.



The phase comparator produces an error signal which causes the voltage controlled oscillator (VCO) to match the input frequency. This output signal is a clean TTL square wave regardless of the appearance of the input signal. The minimum and maximum frequencies accessible to the VCO are controlled by R1, R2 and C1. Our values give a frequency range of approximately 1 kHz to 12 kHz. The 4046 (14046) has two different styles of phase comparators built in. We've used the "Type I" output (pin 2) because it provides better noise immunity than the "Type II" (pin 13) at the expense of locking range. A circuit with a Type II

detector will capture any input signal within the VCO range, while that with a Type I will only capture within a limited bandwidth around the middle of the range (from about 3.5 kHz - 4.5 kHz with our values). Once locked, either will follow the input to the limits of the range. The low pass filter separating the phase comparator from the VCO provides "flywheel" action, dramatically decreasing jitter in the input signal. A thorough discussion of phase locked loops may be found in [1].

As drawn here, the circuit requires an input signal which traverses the CMOS logic thresholds of ~2.75 V for the high state and ~2.25 V for the low state and does not swing more than 0.5 V beyond either supply. Any other input signal will need more processing before application of this circuit. Again, ideas in [1] (e.g. Figure 4.78) may be helpful.

[1] Horowitz, P. and Hill, W. *The Art of Electronics*, 2nd Ed. Cambridge University Press (Cambridge, UK) 1989.

Sincerely,

Carl Michal

Robert Tycko

PS Please credit this contribution to the account of E.D. Becker.

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email: kessler@artus.org.chemie.tu-muenchen.de

(received 2/4/98)

Dear Barry,

methyl group selection is a way to improve resolution in H,C-correlation spectroscopy by limiting the spectral width [1]. It has become a common strategy in deriving valuable distance constraints from the methyl groups of hydrophobic residues in proteins. Typically a DEPT module is applied to create *H*eteronuclear *Q*uadruple-*Q*uantum *C*oherence and coherence selection is achieved using either the phase cycling method or the more recent gradient selection scheme [2, 3]. The main disadvantage of the HQQC experiment is homonuclear coupling between the methyl protons and other protons. These passive couplings lead to phase distortions, decreased resolution due to additional line-broadening and invariably to a sensitivity loss proportional to the proton multiplicity of the multi-quantum coherence. It is therefore not possible to acquire high-resolution spectra, which require long indirect acquisition times t_1, with homogeneous phases using the HQQC approach. We have therefore developed an HSQC experiment with a *Q*uadruple-*Q*uantum *F*ilter (the QQF-HSQC) and coherence selection via gradients. It minimizes the detrimental effects of homonuclear proton couplings and allows for high-resolution spectra with pure phases and increased sensitivity.

The full potential of the QQF-HSQC scheme with regard to the HQQC method can be exploited in the constant-time (ct) version. The pulse sequence is depicted in **Fig. 1**. (Note that we employed magic-angle gradients [4] with $G_x = G_z\sqrt{2}$ for optimum water suppression, allowing us to increase the receiver gain from 4K to 64K). While resolution, and thus sensitivity, decreases with increasing constant time due to the evolution of proton homonuclear couplings in the HQQC method [5], this deleterious effect is completely absent in the QQF-HSQC scheme. Sensitivity in the ct-QQF-HSQC is only governed by the methyl carbons' T_2 relaxation times. These, however, are generally sufficiently long due to the rapid rotation of the methyl groups. Thus, it is practicable to set the constant time in the QQF-HSQC up to the theoretical optimum of $1/^1J_{CC}$ while such long delays are utterly precluded in the HQQC approach. **Fig. 2** shows the ct-QQF-HSQC spectrum of 0.6 mM hnps-PLA_2 acquired on a BRUKER DMX600 at 310 K with 16 scans and 88 data points in the ^{13}C dimension. The constant time was set to 24.6 ms. In order to assess the gain in sensitivity of the QQF-HSQC versus the HQQC, we recorded both experiments in the non-constant time versions and achieved an average gain in sensitivity of 40%, neatly demonstrating the expected results.

The QQF-HSQC module may easily be included into 3D experiments, particularly in its constant time version. Thus, we have run a 3D ct NOESY-QQF-HSQC, resolving many important long-range NOE's from the methyl groups. A full account of this technique will soon be published [6].

Yours sincerely

Horst Kessler Tammo Diercks Manfred Schwaiger

1. H. Kessler, P. Schmieder, M. Kurz, *J. Magn. Reson.* **85**, 400 (1989)
2. H. Kessler and P. Schmieder, *Biopolymers* **31**, 621 (1991)
3. G. L. Shaw, T. Müller, H. R. Mott, H. Oschkinat, I. D. Campbell and L. Mitschang, *J. Magn. Reson.* **124**, 479 (1997)
4. L. Mattiello, W. S. Warren, L. Mueller and B. T. Farmer II, *J. Am. Chem. Soc.* **118**, 3253-3261 (1996)
5. G. L. Shaw, T. Müller, H. R. Mott, H. Oschkinat, I. D. Campbell and L. Mitschang, *J. Magn. Reson.* **124**, 479 (1997)
6. T.Diercks, M. Schwaiger and H. Kessler, J.Magn Res. **130** (1998) in print

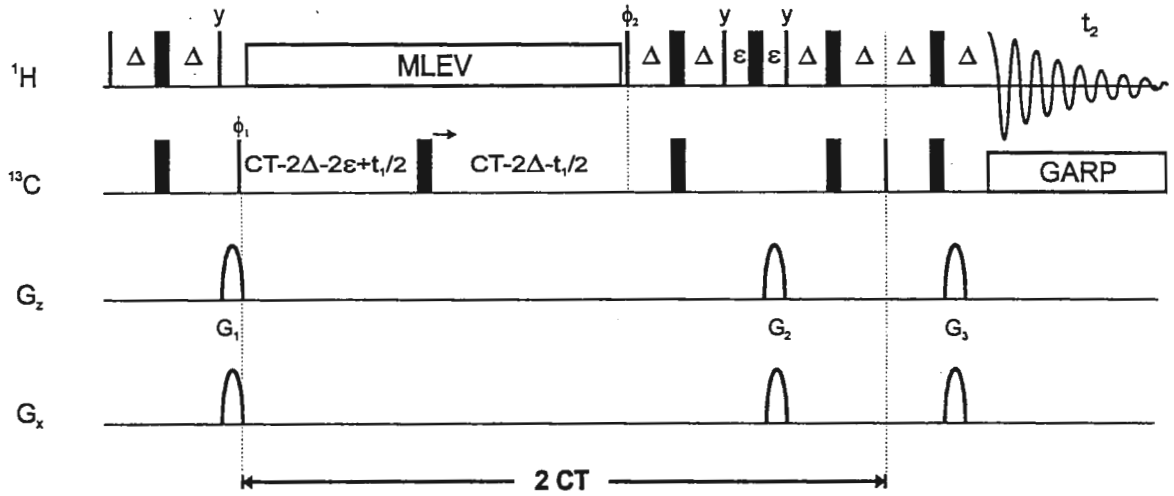

FIG. 1. Pulse sequence of the Constant Time Quadruple-Quantum Filtered HSQC (CT-QQF-HSQC) experiment with the relevant coherence-transfer pathways. Narrow and wide bars indicate 90° and 180° pulses, respectively. All pulses were applied with phase x if not otherwise indicated. The phase cycles used in this experiment were $\phi_1 = x, -x$; $\phi_2 = 2x, 2(-x)$ and $\phi_{rec} = x, 2(-x), x$. In the constant-time period the arrow indicates the direction in which the pulse is shifted as t_1 is incremented; the delays were set as follows: Δ = 2 ms, CT = 12.3 ms. Proton decoupling was achieved by a MLEV16 expansion of 180° pulses. Frequency discrimination in F_1 was achieved using the echo-antiecho procedure. Pulsed field gradients of relative amplitudes $G_1 = 20$, $G_2 = -20$, $G_3 = 65$ for p-selection and $G_1 = 20$, $G_2 = -20$ $G_3 = 55$ for the n-selection were applied for a duration of 1.0 ms, followed by 0.2 ms recovery delays. All gradient pulses were applied at an angle of 54.7° relative to the z-coordinate according to the relation $G_x = \sqrt{2} \cdot G_z$.

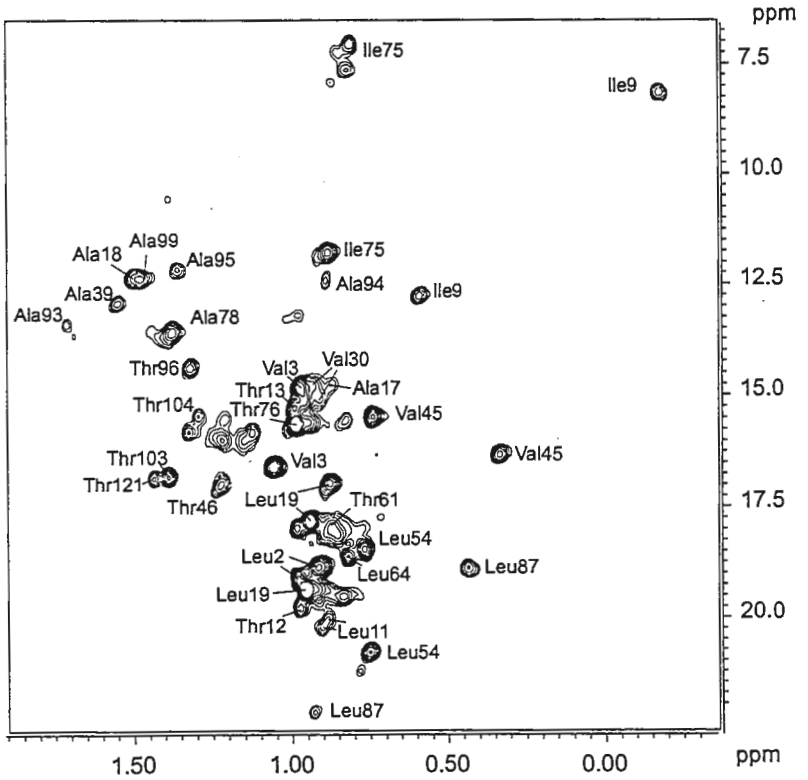

FIG. 2. CT-QQF-HSQC spectrum of 0.6mM hnps-PLA$_2$ at 600 MHz and 310 K recorded using the sequence in Fig.1. Delays and gradients were set as given in the caption to Fig.1. Carrier frequencies were set to 14.5 ppm in F_1 and to 1.0 ppm in F_2. 512 data points were acquired with a spectral width of 2400 Hz, corresponding to an acquisition time of 106.5 ms. In the indirect dimension, 88 data points were collected for a spectral width of 2700 Hz. The spectrum was sampled with 16 scans per transient and a 1.0 s recycle delay, resulting in an experiment duration of 35 minutes. Linear prediction using 24 coefficients was applied in F_1, generating 40 additional data points. Data were zero filled to 1024 x 256 points and a squared cosine-bell window function in both dimensions was applied.

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Dr. Shapiro

(received 2/23/98)

Reduction of Cycling Sidebands in Broadband Proton Decoupled ^{13}C NMR[1]

The identification of degradation products in chemical and material systems using NMR continues to be an area of great interest. A major difficulty with NMR investigations is the detection and quantification of signals arising from small concentrations of degradation species in the presence of the dominating signal from native or undegraded starting material. During a series of recent ^{13}C NMR investigations involving oxidative degradation here at Sandia National Laboratories, we attempted to identify ^{13}C signals for species with concentrations <1% of the original starting material concentration. Using highly concentrated sample in hopes of observing the resulting degradation species, the appearance of cycling sidebands produced from broadband proton decoupling became the limiting factor governing the detection limits. The presence of cycling sidebands during broadband heteronuclear decoupling has been known and discussed for over a decade. The advent of asyncronous decoupling and supercycling of the composite pulse decoupling sequences has provided major reduction in the sideband intensities. The default decoupler pulse sequences utilized on commercial instrumentation had insufficient suppression for the low levels of detection we required. As an example, Fig 1 (bottom) show the ^{13}C spectra obtained using the standard Waltz-16 composite pulse decoupling sequence in asyncronous mode. The sideband modulations are clearly observable at these high manifications (x 500). For normal applications these composite pulse decoupler modulations would be insignificant and most likely not observable. For the highly concentrated samples used in our degradation investigations, the presence of these modulation sidebands becomes significant.

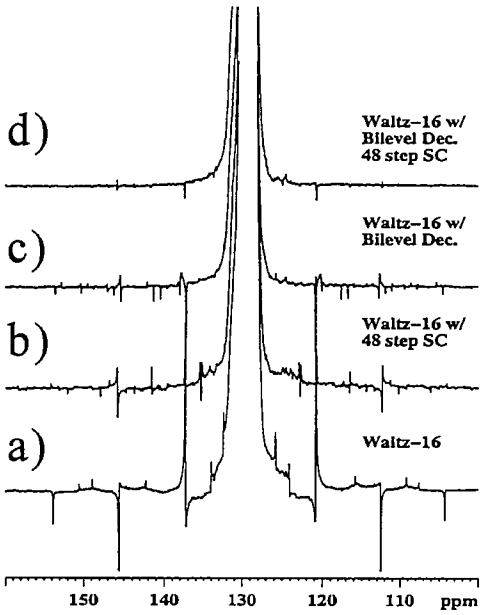

Figure 1. The effect of different decoupling schemes on the amplitude of the cycling sidebands for ^{13}C NMR using proton broadband decoupling on a 5mm BB probe: (a) Standard Waltz-16 composite pulse decoupling, composed of 96 $\pi/2$ pulses with $[Q\overline{Q}\,\overline{Q}\,Q]_n$ repeated supercycles. (b) Waltz-16 with cyclic permutation of the first 48 $\pi/2$ pulses to create 48 variations of the conventional $[Q\overline{Q}\,\overline{Q}\,Q]_n$ supercycle. (c) Bi-level decoupling using a variable high level decoupling period, τ_L=Tk/n, with k ranging from 1 to 16, using a 6.5 kHz high-level decoupling field followed by a 2.5 kHz low-level decoupling field. (d) Combination of bi-level and 48 step supercycle.

Suppression of these cycling sidebands using variation of the supercycle[2] and bi-level adiabatic decoupling[3] have been previously presented. As an example, Fig. 1b shows the suppression of cycling sidebands for a Waltz-16 decoupling sequence where the standard 96 $\pi/2$ pulse supercycle is extended by concatenating new versions of the supercycle by the cyclic permutation of the first 48 $\pi/2$ pulses to create 48 new supercycles. There is a distinct reduction in the magnitude of the cyclic sidebands (~10% of the original Waltz-16 sideband intensity). As noted by Shaka and co-workers this reduction comes at the expense of introducing numerous other

---

[1] Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin company, for the United States Department of Energy under Contract DE-AC04-94AL85000.

[2] A. J. Shaka, P. B. Barker, C. J. Bauer and R. Freeman, *J. Magn. Reson.* **67**, 396-401 (1986).

[3] E. Kupce, R. Freeman, G. Wider, K. Wüthrich, *J. Magn. Reson., Ser. A.* **122**, 81-84 (1996).

modulation frequencies at reduced amplitudes as seen in Fig. 1b. While Bruker software allows the fine control of the composite pulse decoupling sequence, present limitations include an upper limit of 12 programming loops within the composite pulse sequence. To circumvent this limitation, the 48 step supercycle permutation sequence was written "longhand" (4608 $\pi/2$ pulses), without the incorporation of programming loops.

A bi-level decoupling sequence involving a variable high-level decoupling period for suppression of cycling sidebands was also investigated, as shown in Fig. 1c. Here a variable period of high power decoupling, τ_L=Tk/n, was used prior to initiation of the regular decoupling sequence. For the spectra in Fig. 1c, n=16 and k was step from 1 to 16 with averaging of these scans. An adiabatic period of T = 2.4 ms was used in this example. It is clear that the incorporation of bi-level decoupling also reduces the amplitude of the cycling sidebands, similar to the reduction observed for the 48 step supercycle. The extent of these modulations is still not sufficient for the our desired application, so a combined bi-level decoupling, 48 step supercycle type experiment was evaluated as shown in Fig. 1d. This combined experiment afforded excellent suppression of the cycling sidebands, with no apparent loss in decoupler efficiency over the ^{1}H bandwidth. The pulse sequence used for the bi-level decoupling experiment, plus the modifications required in the first section of the composite pulse sequence in order to produce the high to low level power change for the decoupling, are given below.

Copies of the program, including the 48 step supercycle can be requested from me via e-mail at tmalam@sandia.gov. I would also like to thank Alan Deese of Bruker for helpful discussions.

(Modifications to header in Waltz-16 CPD sequence - includes 2 power level pl commands and the variable p30 pulse. To be used by the zgdc_bilev pulse sequence)

```
p30:0 pl=pl13
1 pcpd*3:180 pl=pl12
 pcpd*4:0
 pcpd*2:180
   •
   •
   •
```

;zgdc_bilev

```
;avance-version
;1D sequence with decoupling using bi-level decoupling during acquistion
;as outlined in
;Kupce, Freeman, Wider and Wuthrich, JMR, Ser A, 122, 81-84 (1996)
; Written: Alam 2/98, Sandia National Laboratories

#include <Avance.incl>
"d11=30m"
"p30=cnst1/l1"              ; high level decoupling spin lock for n*T/L1

1 ze
2 2u pl12:f2                ; set decoupling power for NOE
  d1 cpd1:f2                ; relaxation delay (CPD1)
  2u do:f2                  ; decoupler off
  2u pl13:f2                ; reset power for high level decouple pulse
  p1 ph1                    ; X nucleus pulse
  go=2 ph31 cpd2:f2         ; CPD2
  d11 ipu30
  lo to 2 times l1
  wr #0
  d11 do:f2
exit

ph1=0 2 2 0 1 3 3 1
ph31=0 2 2 0 1 3 3 1
```

Todd M. Alam

```
; total number of scans=ns*l1
;pl1 : f1 channel - power level for pulse (default)
;p1 : f1 channel -  high power pulse
;d1 : relaxation delay; 1-5 * T1
;d11: delay for disk I/O                    [30 msec]
;cpd2: decoupling sequence defined by cpdprg2
(waltz16_bilev or equivalent)
;pcpd2: f2 channel - 90 degree pulse for decoupling sequence
;cnst1=adiabatic modulation time (unitless)~ (2.4ms = 2400)
;inp30: increment of p30 pulse inp30=p30
;pl12 standard decoupling strength f2 channel for cpd/bb
;pl13 high power CW lock decoupling strength for duration
p30
;program using decoupling sequence cpd2= waltz16_bilev
```

UNIVERSITY OF VIRGINIA
DEPARTMENT OF CHEMISTRY
McCORMICK ROAD
CHARLOTTESVILLE, VIRGINIA 22901

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

January 23, 1998
(received 1/29/98)

Scalar Relaxation Contribution to Magnetic Relaxation Dispersion

Dear Dr. Shapiro,

Measurements of magnetic relaxation dispersion, MRD, have been a main interest of research in our lab. In studying the dynamics of hexafluorophosphate binding with bovine serum albumin(BSA) an interesting field dependent relaxation contribution was observed. This increase in relaxation at low field strengths in the ^{31}P dispersion plot appears to be caused by the contribution of scalar coupling(700 Hz for ^{31}P-^{19}F).

A solution of 2.55% BSA in 0.9M $NaPF_6$ was examined with a dual magnet dispersion instrument which consists of a 7.0T superconducting magnet and a variable 0-1.6T electromagnet. A rotational correlation time of 55 ns was determined by fitting the data to the heteronuclear dipole-dipole relaxation equation of fluorine and phosphorus.

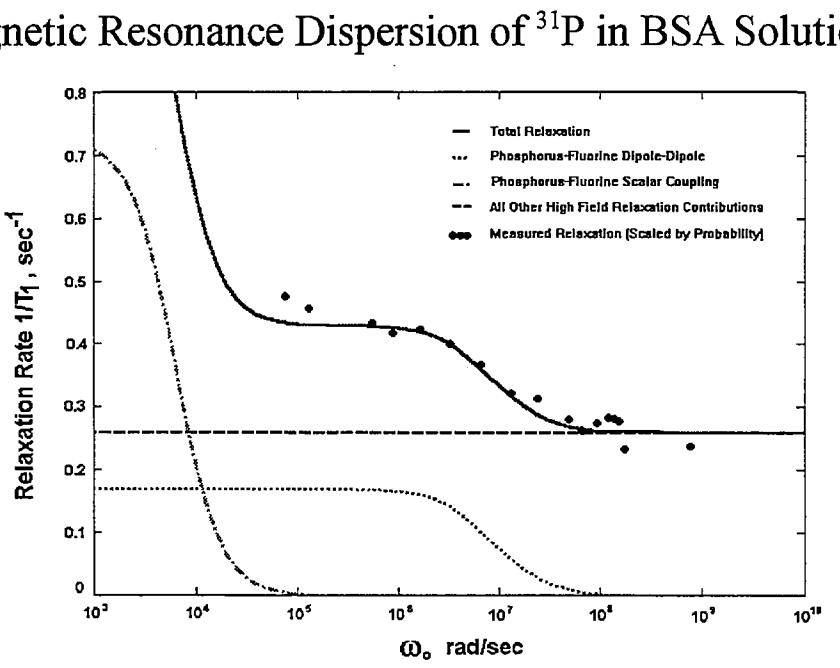

Figure 1: Spin-lattice relaxation rate as a function of magnetic field strength represented as the ^{31}P Larmor frequency for 2.55% aqueous bovine serum albumin obtained on the sample shuttle MRD.

The additional relaxation rate increase at low field may be accounted for by the large scalar coupling, often seen as contributions to transverse relaxation but more rarely to spin-lattice relaxation.

$$\frac{1}{T_1}=\frac{2}{3}A^2S(S+1)\frac{\tau_2}{1+(\omega_I-\omega_S)^2\tau_2^2}$$

The τ_2 was calculated from the data provided by the ^{19}F MRD profile and the low field increase in the rate is large at really small magnetic field strengths. These effects may be obvious in rotating frame experiments on relatively large molecules, which is clearly of interest in macromolecular dynamics studies.

Sincerely yours,

Shawn R Wagner

Robert G. Bryant

Stanford Magnetic Resonance Laboratory
STANFORD UNIVERSITY
300 Pasteur Drive, SUMC R320
Stanford, California 94305-5337

Oleg Jardetzky, M.D., Ph.D.
Professor of Molecular Pharmacology

Tel.: 650/723-6153
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February 19, 1998

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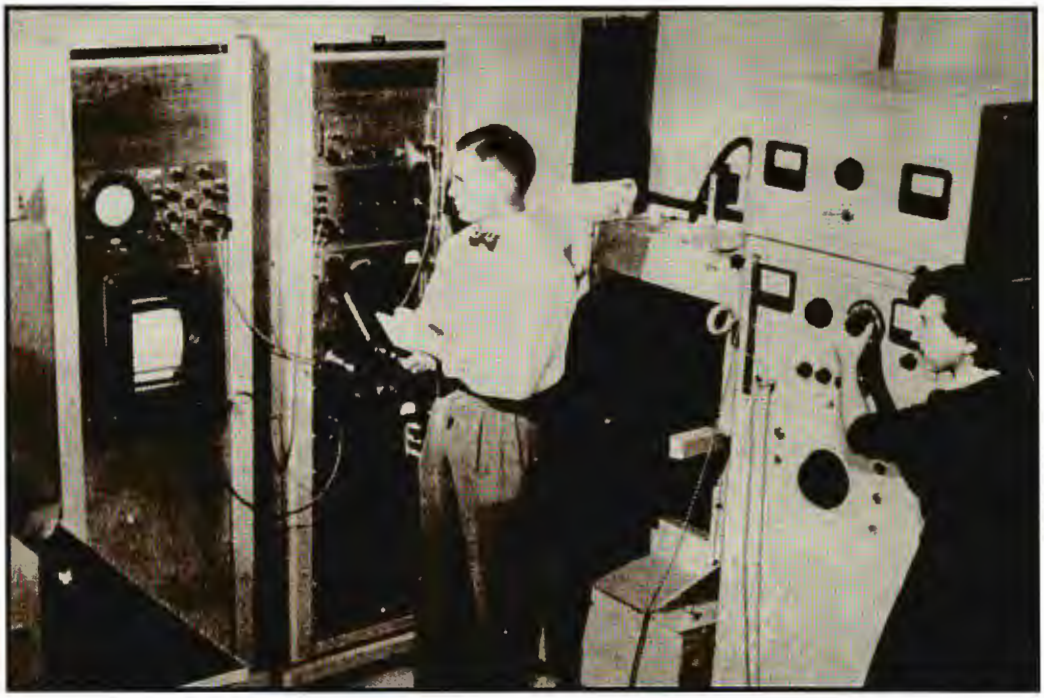

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February 20, 1998 (received 2/23/98)

Bernard L. Shapiro, Ph.D.
Editor, The NMR Newsletter
966 Elsinore Court
Palo Alto, CA 94303

The NMR Limbo
How low can you go?
Limbo lower now!

Dear Barry,

Somewhere back in time, I think it was in the 60's although I don't remember for sure, there was a song about a dance called the limbo. I've borrowed the sentiment of that old song, working under lower and lower levels, for the title of this contribution. In 1992, Ron Crouch and I, both then at Burroughs Wellcome Co., in RTP, NC, were fortunate enough to be involved with and able to report the initial development of 3 mm micro inverse NMR probes in collaboration with Toby Zens of then the Nalorac Cryogenic Corp.[1] At the time, we thought it remarkable to be able to record HMQC spectra of the small model alkaloid cryptolepine (**1**) on slightly less than 0.1 μmole of material with an over weekend acquisition. Time has moved on, and so have we. Ron is now working directly with Toby at the Nalorac Corporation while I've moved to Pharmacia & Upjohn to head up the Rapid Structure Characterization Group in Pharmaceutical Development. Our collaboration, however, hasn't ended!

Fast forward to January, 1998. Last month, we sent you a contribution describing the first results obained with the 1.7 mm prototype of a new generation Nalorac SMIDG-600-1.7 submicro inverse-detection gradient probe built by Ron and Toby. As we reported in that contribution, we were able to record a GHSQC spectrum on a 0.55 μmole sample of **1** in 25 μl of DMSO in 12 min. and a high quality GHMBC spectrum on the same sample in 1.1 hr.[2] In the proton reference spectrum recorded in 1 transient, the resonances for cryptolepine are quite prominent. At a much lower level, however, ~8% to be more precise, another set of aromatic resonances are observed consistent with two four-spin systems as shown in the top trace presented in Figure 1.

As cryptolepine (**1**) is thought to be prone to undergo facile oxidation, which our solid sample had had plenty of time to do in the 2+ years since its isolation (initial chromatographic purity was >99%), we were interested in our ability to acquire heteronuclear chemical shift correlation data using the SMIDG-600-1.7 probe at the much lower levels necessary to be able to characterize the presumed oxidation product *in situ*. More germane to pharmaceutical development, the ability to carry out such experiments is of considerable interest for those instances when enriched feedstocks of a drug substance, such as from a mother liquor, are available containing high levels of an impurity or degradant whose characterization is necessary. The ability to acquire heteronuclear shift correlation data on an impurity present at the 8% level without isolation, even if only possible under favorable circumstances, offers obvious benefits in terms of rapid characterization.

Hence, the acquisition of a GHSQC spectrum of the 8% impurity was undertaken without isolation. After 6 hrs. of data accumulation, by scanning traces and looking for responses at the corresponding chemical shift of the 8% peaks in the proton spectrum, it was tantalizing to imagine that peaks were beginning to "grow in" that corresponded to the impurity. At 16 hrs, however, there was no question; we had in hand the data to determine the chemical shifts of the carbons directly bound to the protons of the impurity. The GHSQC spectrum of the sample of cryptolepine and the 8% impurity is shown in the contour plot in Figure 1 at 25.5 hrs when the accumulation was terminated. Resonances resulting from the major component, cryptolepine (**1**), correspond to the resonances in the proton spectrum shown in the middle trace. Resonances arising from the impurity (denoted with arrows) correspond to the small resonances in the top trace. Two of the resonances in that spectrum are partially obscured by the major resonances from the cryptolepine and are denoted by arrows in the reference spectrum. It is also interesting to note that the resonance furthest downfield in the top trace, resonating at about 9.22 ppm, which might be presumed to correspond to another H11-type proton does not give a response in the GHSQC spectrum indicating that this proton is not directly attached to carbon, but rather, is probably an indole-NH resonance.

Assuming that the impurity corresponds to an oxidation product of cryptolepine, from a chemical standpoint, the logical structure of the impurity would be cryptolepinone (**2**) identified previously by several groups.[3-5] Based on an assumed MW of 248, the 8% impurity contained in the sample being investigated corresponds to ~0.04 μmoles of material.

To further support the structure of the minor component, APCI (Atmospheric Pressure Chemical Ionization) LC/MS analysis was undertaken using a Finnigan TSQ-7000 mass spectrometer. Cryptolepine (**1**) gave a protonated molecular ion at 233 Daltons while the minor component gave a protonated molecular ion at 249 Da. Product ion spectra of the latter were obtained by CID (Collision Induced Dissociation) in the second quadrupole of the mass spectrometer using argon as a collision gas. Fragment ions at m/z = 234, 205, 104, and 77 were consistent with the proposed structure of cryptolepinone (**2**) as the oxidation product from the decomposition of cryptolepine (**1**).

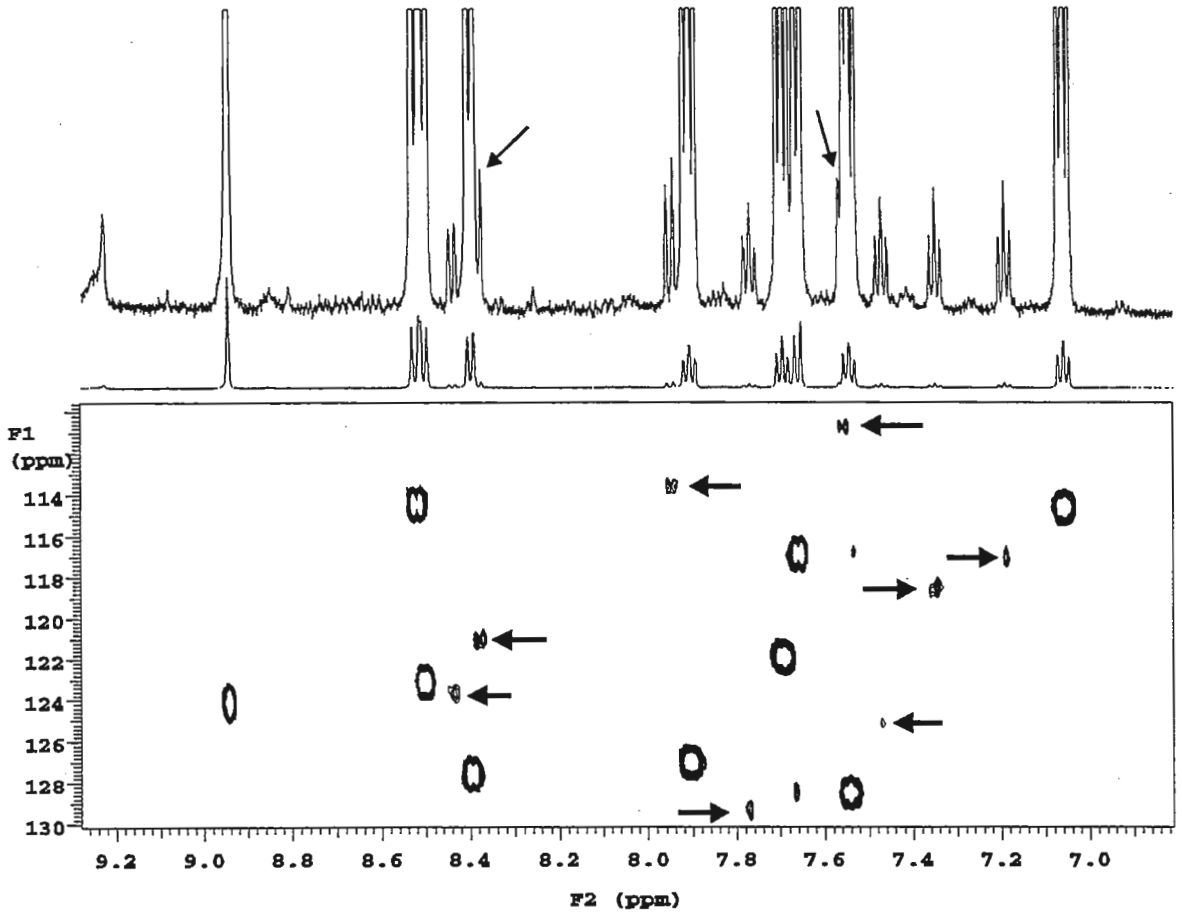
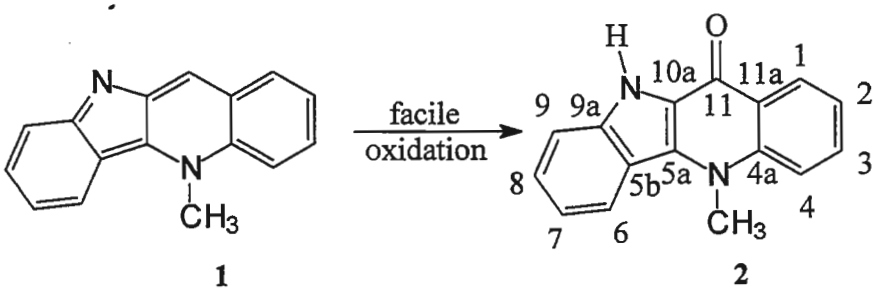


Figure 1. Top trace: ^{1}H reference spectrum of a 0.55 µmole sample of cryptolepine (**1**) dissolved in 25µl of 99.992% d_6-DMSO acquired in 256 transients using a Nalorac SMIDG-600-1.7 gradient submicro inverse-detection probe installed in a Varian Inova 600 and plotted to show the 8% impurity resonances contained in the sample. Two of the resonances, those denoted by arrows, are partially obscured by the main resonances in the spectrum due to **1**. Middle trace: ^{1}H spectrum plotted to show the cryptolepine resonances on scale. Bottom: contour plot of the GHSQC spectrum of the sample after a 25.5 hr data accumulation. The data were recorded as 2048 x 32 (2 x 32) hypercomplex files with 768 transients accumulated/t_1 increment. Resonances corresponding to the impurity are identified with arrows in the contour plot. Note the absence of a response for the singlet resonating at ~9.22 ppm, suggesting that this resonance is not directly bound to carbon and that it might correspond to the NH resonance of an indole.



1 **2**

Returning to the NMR data, proton and protonated carbon chemical shifts compared very favorably with those reported in the literature. A nearly complete set of long-range correations was also obtained on the 8% impurity from a GHMBC spectrum acquired in 56.5 hrs over a weekend. Long-range correlations were readily observable from the N-methyl group to the flanking C4a and C5a quaternary carbons whose chemical shifts were also in full agreement with reported data. From the GHMBC data, it was possible to make assignments for all of the carbon resonances of **2** with the exception of the C10a quaternary carbon between the indole NH and the carbonyl, and the carbonyl carbon itself. Although not shown, it was also possible to record a ROESY spectrum with a 600 msec mixing time in 45 min that afforded the rOe correlations from the N-methyl of **2** to the flanking aromatic protons at the 4- and 6-positions of the molecule. These data allowed the correct orientation of the four-spin systems within the molecular framework.

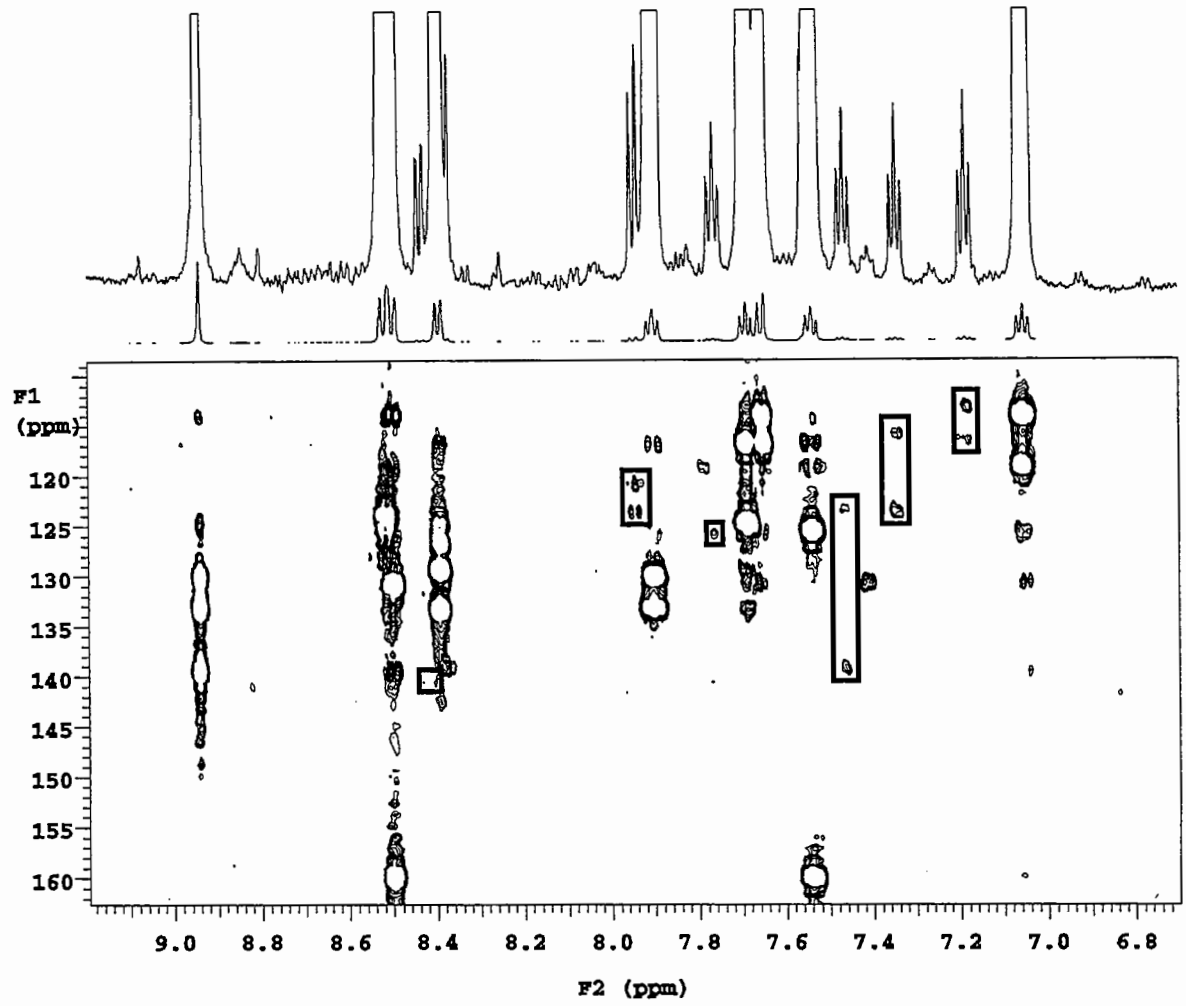


Figure 2. GHMBC spectrum recorded in 56.5 hrs on 0.55 μmole of cryptolepine and an 8% impurity (0.04 μmoles) identified as cryptolepinone (**2**) dissolved in 25 μl of d_6-DMSO, recorded using a Nalorac SMIDG-600-1.7 probe in a Varian Inova 600. The experiment was optimized for a 10 Hz long-range coupling. Data were acquired as 4K x 64 files accumulating 832 transients/t_1 increment. Responses arising from the 8% impurity are boxed. Not all responses were observed, some being obscured by the much more intense resonances of cryptolepine (**1**). Responses for the N-Methyl of the impurity (region not shown) identified the flanking C4a and C5a quaternary carbons resonating at 141.0 and 131.0 ppm, respectively.

Finally, the results of a ROESY spectrum used to assign the H4 and H6 protons of the cryptolepinone (**2**) impurity in the sample are shown in Figure 3. The data shown were recorded in ~45 min and allowed the necessary correlations (boxed responses in the spectrum) between the N-methyl group and the flanking H4 and H6 resonances to be readily assigned. These correlations established the orientation of the two four-spin systems within the molecular framework allowing the confirmation of the structure through the use of responses contained in the GHMBC spectrum shown above in Figure 2.

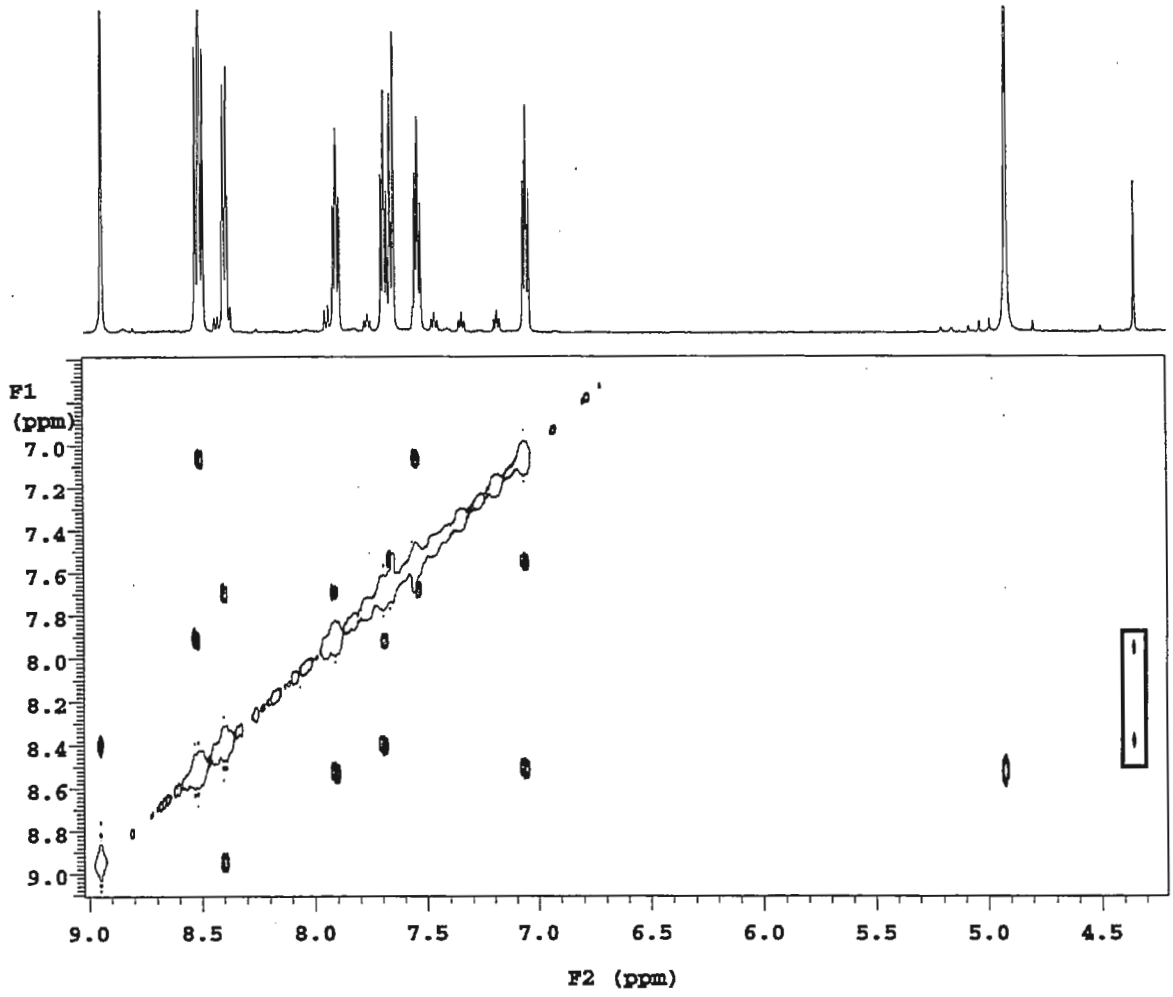


Figure 3. ROSEY spectrum of a 0.55 μmole sample of cryptolepine (**1**) containing an 8% (0.04μmole) cryptolepinone (**2**) impurity. Data were acquired as 2048 x 128 (2 x 128 hypercomplex or States-TPPI files) accumulating 8 transients/t_1 increment giving a total acquisition time of 1.5 hr. Data presented were plotted after 4 transients/t_1 incrment. Correlations are observed from the N-methyl group of cryptolepione, which resonates at 4.35 ppm, to the flanking H4 and H6 resonances resonating at 7.96 and 8.38 ppm, respectively, are denoted by the box along the right edge of the contour plot. The mixing time, 600 msec, was established from the average proton T_1 relaxation time which was 1.2 sec. The interpulse delay was set to 1.8 sec.

In conclusion, the data presented herein demonstrate more fully the possibilities of submicro gradient inverse detection under favorable circumstances. Concerted utilization of NMR and mass spectral data on a sample consisting of <0.05 μmole of a simple alkaloid are sufficient for the total characterization of the compound. The potential impact of this type of technology for the characterization of impurities and degradation products in the pharmaceutical industry, the identification of drug metabolites, in addition to the natural product application shown, is considerable. Hence the last line of the title of this contribution, "limbo lower now!" To the best of our knowledge, these spectra represent the lowest level acquisition of complete heteronuclear chemical shift correlation data to date. Complete details of this work will be reported elsewhere.

Gary E. Martin Russell H. Robins Jane E. Guido

1. R. C. Crouch and G. E. Martin, . *J. Nat. Prod.*, **55**, 1343 (1992).
2. G.E. Martin, R.C. Crouch, and A.P. Zens, "Gradient Submicro Inverse Detection --Rapid Acquisition of Inverse-Detected Heteronuclear Chemical Shift Correlation Data on Submicromole Quantities of Material," *Magn. Reson. Chem.*, submitted (1998).
3. M. H. M. Sharaf, Ph.D. Dissertation, School of Pharmacy, University of Pittsburgh, Pittsburgh, PA, 1993, pp 177-192.
4. A. Paulo, E. T. Gomes, and P. J. Houghton, *J. Nat. Prod.*, **58**, 1485 (1995).
5. M. M. Cooper, J. M. Lovell, and J. A. Joule, *Tetrahedron Lett.*, **37**, 4283 (1996).

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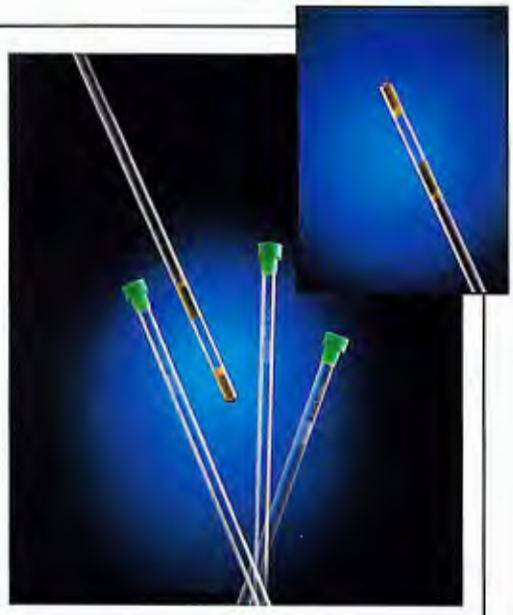

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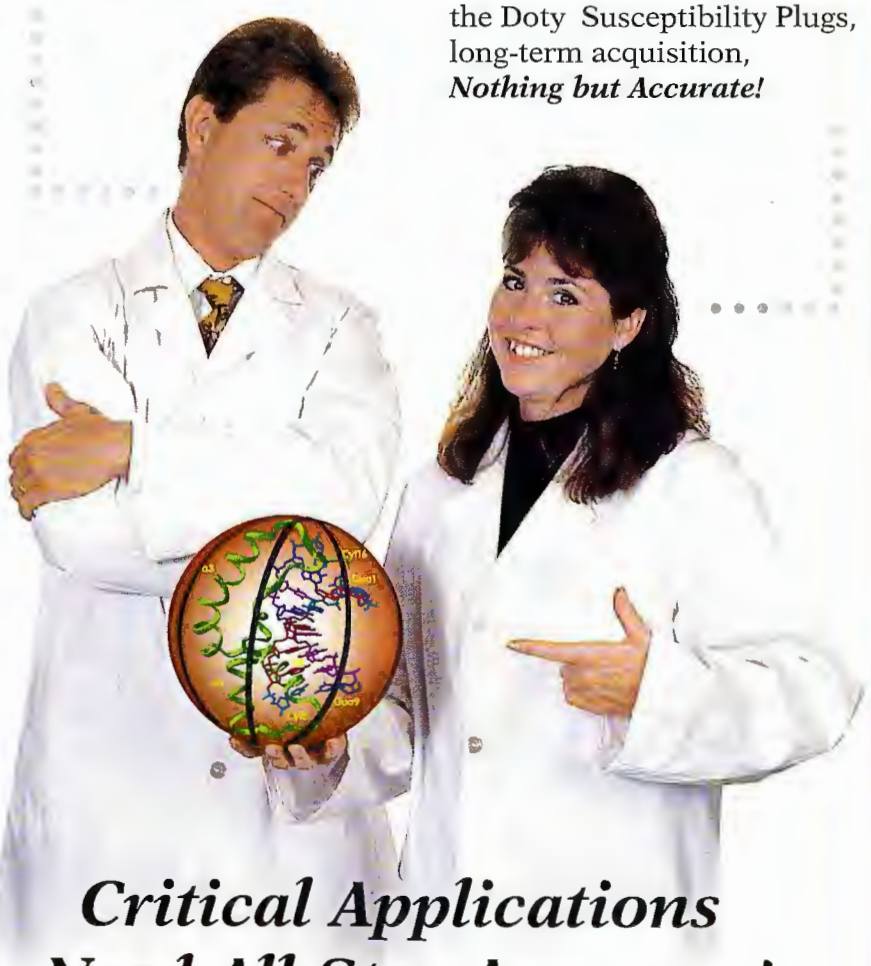

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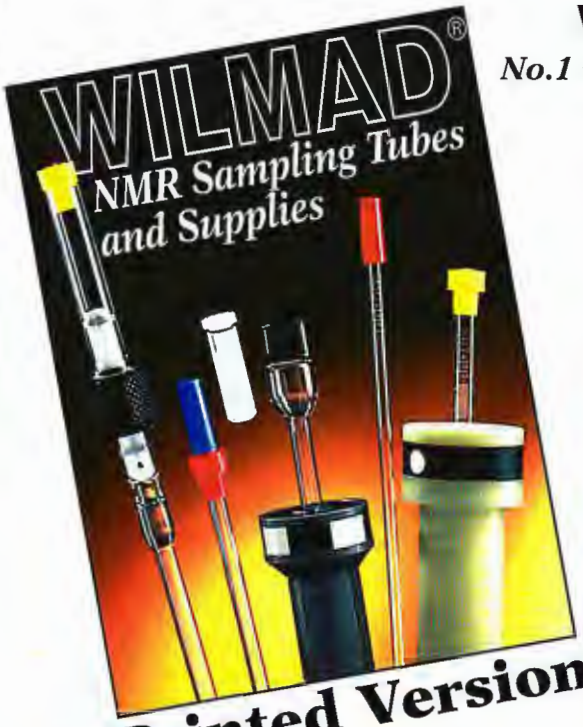

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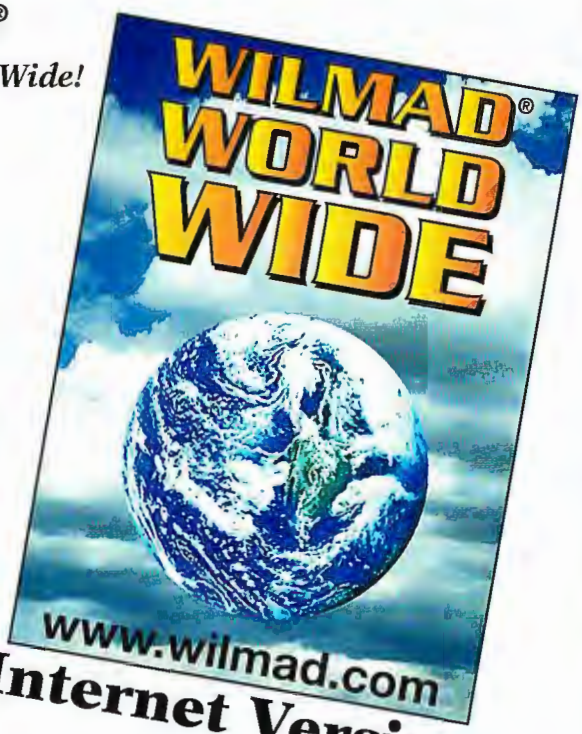

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The NMR Newsletter - Book Reviews

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

A Handbook of Nuclear Magnetic Resonance, *Second Edition*

by

Ray Freeman

Addison Wesley Longman Limited, Harlow, Essex, England, 1997; Second Edition;
ISBN 0-582-25184-2 (hbk), 344 pages; £45.00; (US price from Web booksellers, $72.75)

This is the revised edition of a book first published ten years ago. It consists of a collection of short self-contained essays (arranged alphabetically) on various aspects of solution-state NMR. These are directed towards the practical aspects of the subject, and serve to give an operator insight into what is going on in an NMR instrument, and help in getting the best out of such an instrument. The book concludes with a Index of 7-pages, a quite generous extent, in addition to the References and Cross-references which appear at the end of each essay..

The revision emphasizes to some extent the changes in what are the "hot" topics in NMR. There are the same number (59) of articles in the old and the new editions. However, the following have been deleted in the new edition: Alignment of Molecules, Gated Decoupling, Modulation and Lock-in Detection, Off-resonance Decoupling, and Shift Correlation, and have been replaced by: Coherence, Correlation Spectroscopy, Measurement of Coupling Constants Nuclear Susceptibility, and Pulsed Field Gradients. Compared with the old edition, the new one has many more references to the primary literature, and also more cross-references to other relevant articles in the book. Some articles have been completely revised (e.g. Broadband Decoupling) while others (*e.g.*, Cooley-Tukey Algorithm) stand almost unchanged in the revision.

The diagrams, mathematical equations, and other symbolic material are very clear (there are more mathematical formulae in the new edition than the first). A feature of the first edition, which is carried over into the new one, is the use of cartoons. In some cases these are apposite, but some fall wide of the mark and the drawings are not always clear.

Where is the book useful? I do not believe that it is a substitute for the operation manual of the NMR machine. It does not offer immediate solutions to any of the problems that an operator might encounter. Rather it serves as background reading, and as a stimulant to look at the mechanism of recording spectra in a new light. Within these limits the book is excellent. However, there is nothing on interpretation of spectra (there is only one chemical formula in the book!), and nothing on basic NMR theory.

To sum up, if you bought the first edition and still have it, hang on to it and buy the new one as well. If you bought the old edition and it has disappeared, buy the new edition and make a resolution to look after it. If you did not acquire the old edition, buy the new one and settle down to a very stimulating read.

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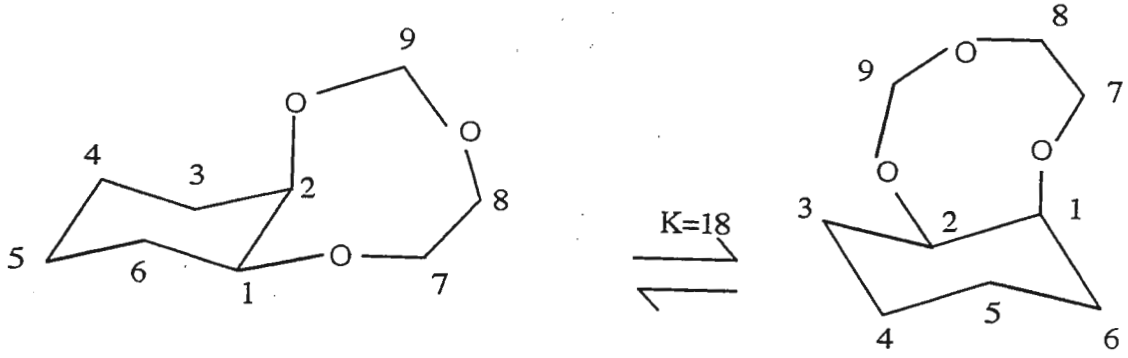
Dr. B.L. Shapiro
The NMR Newsletter
966 Elsinore Court
Palo Alto California
94303 USA

Jan. 29,1998
(received 2/17/98)

Title: ^{13}C and ^{1}H NMR Studies of Conformational Preferences in Crown Ether Analogs

Recently, we have been looking at a series of cis-1,2-cyclohexyl annelated crown ether systems in order to determine their conformational preferences[1]. The present report deals with the 8-crown-3 molecule shown below in its two conformations **1A** and **1B** which are interconvertable via cyclohexane ring inversion. Using a combination of HMBC and HMQC experiments at low temperature , we have determined that conformation **1B**, with the O-CH_2-O unit equatorial, is favoured by about 4.4 kJ/mol-ie K =17.9± 4.2. Results of molecular mechanics calculations using MM^+ are in accord, the calculated energy difference between **1A** and **1B** being 2.7 kJ/mol.

One of the factors which appears to be destabilizing **1A**, is a transannular 1,4 H···H interaction, between protons on C2 and C7. Furthermore, it is interesting to note that there is a 10.1 ppm ^{13}C chemical shift difference at C2 in the two conformers, ie 67.88 in **1A** and 77.99 in **1B**. As expected, C2 in **1A**, bearing the axial oxygen, is shielded relative to its counterpart in **1B**. However, the additional repulsive intraction between the equatorial proton at C2 in **1A** and one of the C7 protons, may induce a further upfield shift at the C2 site. Normally, the observed shielding difference between cyclohexyl methine carbons bearing axial and equatorial oxygenated substituents is 5-7 ppm.



1A **1B**

[1] G.W. Buchanan, M. Gerzain and K. Bourque. Magn. Res. Chem. 35, 287 (1997).

G.W. Buchanan, Professor and Chairman

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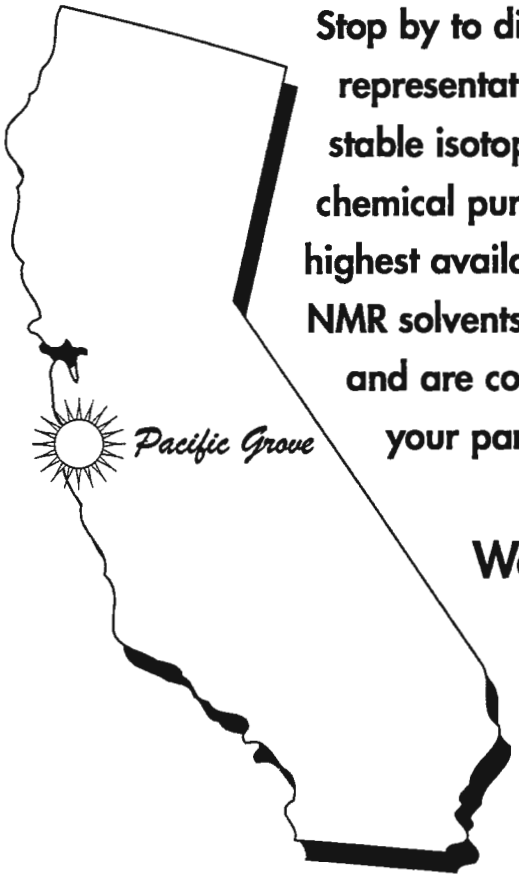

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James Prestegard, University of Georgia

STRATEGIES FOR STUDYING MEMBRANE PROTEINS BY NMR
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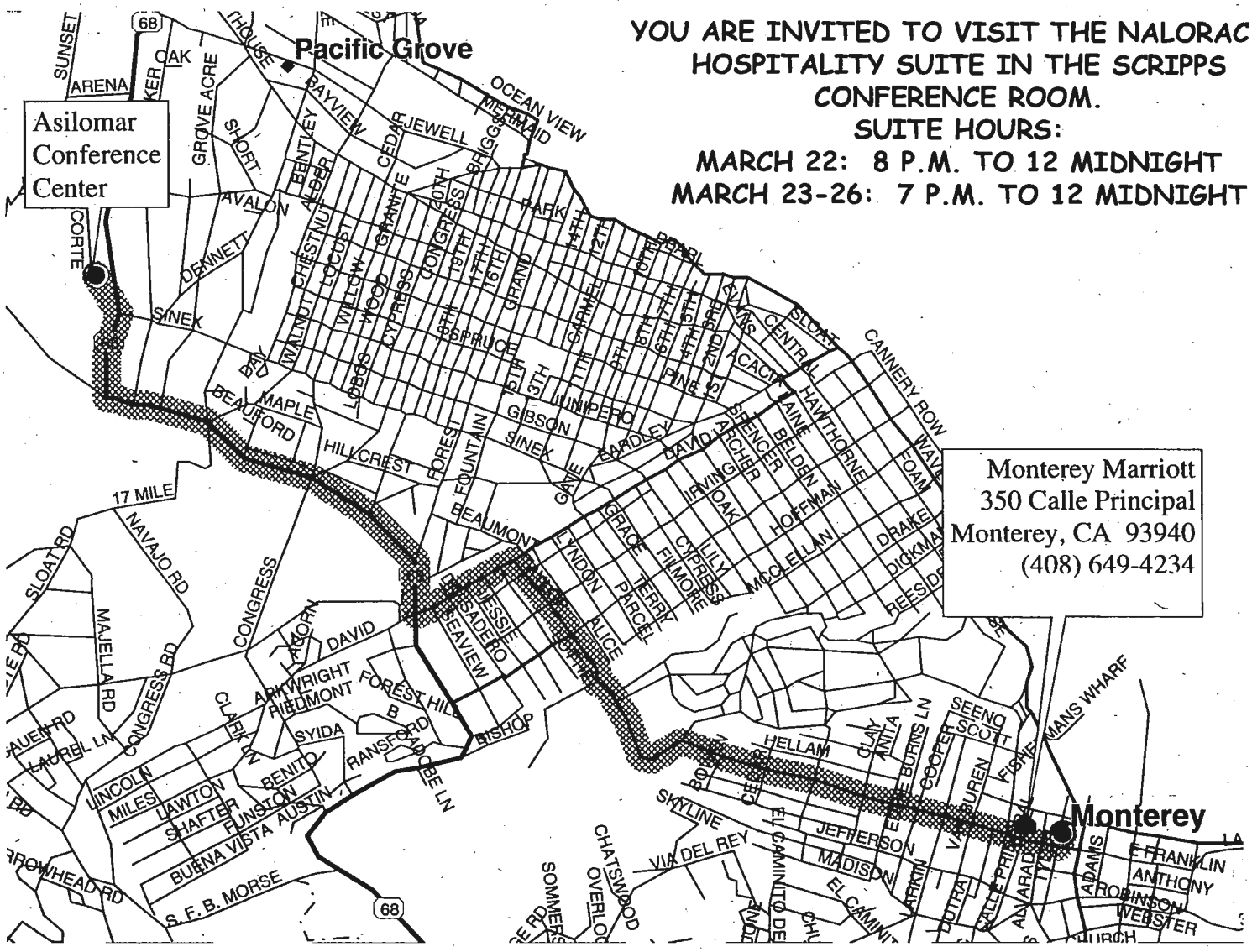

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QUEEN MARY
AND WESTFIELD COLLEGE
UNIVERSITY OF LONDON

29th January 1998
(received 2/9/98)

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

Dear Barry,

Gallium Citrate Revisited

Recently a colleague, Paul O'Brien at Imperial College, determined[1] the X-ray structure of a gallium citrate complex, $(NH_4)_3[Ga(C_6H_5O_7)_2].4H_2O$. The anion is centrosymmetric and the molecule is achiral with one molecule as the asymmetric unit. Each citrate ligand binds to gallium through the hydroxide oxgen, an oxygen of one of the $CH_2CO_2^-$ groups and an oxygen of the remaining CO_2^- group, leaving a pendant CH_2CO_2H group. The high frequency region of the ^{13}C CP/MAS spectrum is shown in Figure 1, and of the expected 6 carbonyl resonances, five are observed at 173.9, 179.4, 180.7, 185.6 and 189.9 d, with that at 179.4 d being *ca.* double the intensity of the other four.

There has been considerable work published on the solution state ^1H, ^{13}C and ^{71}Ga spectra of gallium(III) in the presence of citrate[2,3], but there are still unanswered questions concerning the speciation. Therefore we decided to try to add to this using a higher field spectrometer (AMX-600). In solution it is clear that there is partial dissociation of the 2:1 complex, and in the pH range 2 to 7 we observed a new ^{71}Ga signal at *ca.* 27 d (*vs.* 1 M gallium nitrate solution) with $Dn_{½}$ *ca.* 10 kHz which we assign to complexed gallium - either as a 1:1 and/or 2:1 species.

In the pH region 5 to 7 the ^1H and ^{13}C spectra are dominated by two sets of signals due to free and complexed citrate in slow exchange (there is a characteristic difference in the $^2J_{AB}$ coupling in the ^1H spectrum between free and complexed citrate). The relative proportions of free and complexed ligand vary with concentration in just the way predicted by using the stability constants for the stepwise dissociation of the 2:1 species estimated by Chang *et al.*[3] ($K_1 = 48 \pm 1$ M^{-1}, $K_2 = 13 \pm 1$ M^{-1} at pH 5.8). We tend to believe that the set of resonances due to the complexed citrate is a superposition of both 1:1 and 2:1 species, although we have no hard evidence for that - we have some ongoing experiments here to clarify this. The carbonyl ^{13}C signals for the complexed citrate are at 186.5 and 179.5 d, in the intensity ratio 1:2. The average shift for the two highest frequency signals in the CP/MAS spectrum is 187.8 δ and the average for the other carboxyl signals in the solid state is 178.4 δ. These solid state averages are very close to the solution chemical shifts and indicate that in solution the citrate is bound in a similar manner to the solid state.

We are working on two ideas here. The first is that in solution the two $CH_2CO_2^-$ carboxyl carbons of each bound citrate give just one signal. If one carboxyl is bound to citrate and the other pendant, then they must be in fast *intra*molecular exchange. However the fact that we see separate signals for free and bound citrate indicates a slow process for the *inter*molecular exchange. Our interpretation is that the binding of citrate to gallium through the carboxyl carbons is much weaker than the binding through the hydroxide oxygen. Therefore the hydroxide oxygen acts as an anchor for the ligand at gallium, while the carboxyl-gallium bonds are broken and reformed much more rapidly. In support of this is the fact that the X-ray structure[1] shows the two hydroxide bond lengths (1.890 and 1.900 Å) to be significantly shorter than the four carboxyl oxygen-gallium lengths (1.976 to 2.054 Å).

Best wishes.

Yours sincerely,

Geoff Hawkes

Dr. G.E. Hawkes

[1] P. O'Brien, H. Salacinski and M. Motevalli, *J. Am. Chem. Soc.*, **119**, 12695, (1997).
[2] J.D. Glickson, T.P. Pitner, J. Webb and R.A. Gams, *J. Am. Chem. Soc.*, **97**, 1679, (1975).
[3] C.H.F. Chang, T.P. Pitner, R.E. Lenkinski and J.D. Glickson, *J. Am. Chem. Soc.*, **99**, 5858, (1997).

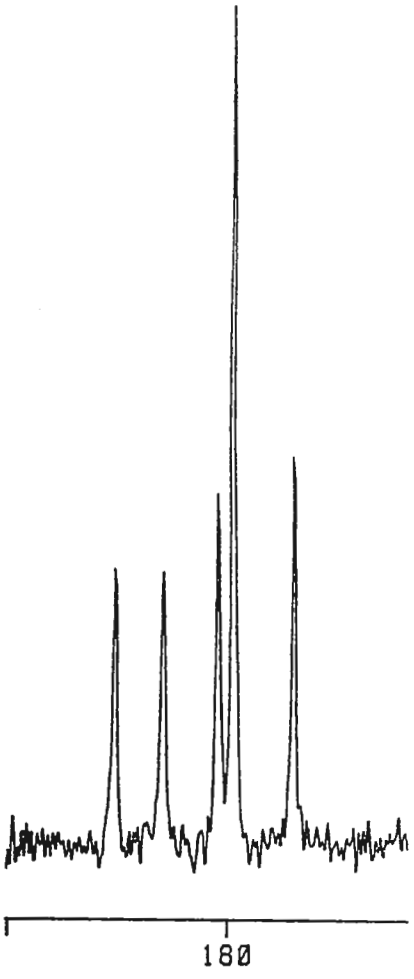
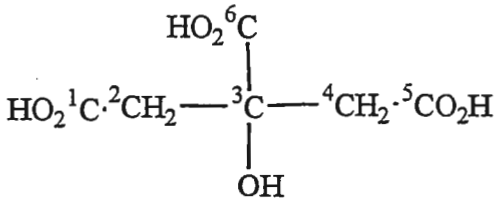


Figure 1. 75.5 MHz ^{13}C CP/MAS spectrum (MSL-300) of $(NH_4)_3[Ga(C_6H_5O_7)_2].4H_2O$, 4mm o.d. rotor, MAS at 8483 Hz, 1812 scans.



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JCAMP - The Standard (!) NMR Data Format

February 05,1998
(received 2/12/98)

Dear Barry,

In a recent contribution to the NMR Newsletter we read about difficulties colleagues had in obtaining current information on existing NMR standards. We may take that as a serious indicator that even in times of lightspeed mail and networks there is still no substitute for a good conversation on a conference or for institutions like the NMR Newsletter.

However, because of lack of information a lot of assumptions and implications got worded in that contribution to that need to be set straight.

Lack of information

I carried out a simple search for the term 'JCAMP' on several search engines on the Internet. All of the references used in my letter were found, however, not by all search engines.

Search engine	# hits	comments
Altavista	582	relevant hits within the top 10
Hotbot	1042	relevant hits within the top 10
Infoseek	301	a good 'related topics' section came up, too
Lycos	100+	./.
Yahoo	4	all four hits were **totally irrelevant**

I consider this the clue to the earlier paper we all saw*: It doesn't mean much if your favorite search engine doesn't find JCAMP!*

A bit of JCAMP's background

As implied by the abbreviation, the original JCAMP (Joint Committee on Atomic and Molecular Physical Data) committee was a group of people that worked on the technical details and publication of JCAMP. Bob McDonald (`http://members.aol.com/rmcdjcamp/index.htm`) had the honor to be the first chairman of this committee. Now, the argument that vendors don't like a comparison of their data is probably a bit off target. Most of Bruker's processing programs can read other parties datasets since quite a while (and frankly, I always thought NUTS could do that as well). Thus, if customers really wanted to compare, they simply would buy a copy of WINNMR or use XWIN-NMR on their instruments and go do it. S/he wouldn't have to wait for JCAMP to materialize.

A brief history of JCAMP, including all literature references, can be found here: `http://wwwchem.uwimona.edu.jm:1104/software/jcmpsum.html`

Meanwhile the original JCAMP committee got promoted to an IUPAC sub-committee, chaired by Tony Davies (`http://www.isas-dortmund.de/projects/jcamp/index.html`).

Talking about politics of standards

Way back in September 1990 (!) the "Industrial NMR User's Meeting" (INUM) was held in a small village at the Lago Maggiore in Italy, featuring a discussion about NMR data standards (sic!). All of the major vendors sent high-ranking representatives and promised to implement JCAMP as soon as possible. However, someone seems to have royally messed up the implementation priorities for our competitors, because despite all promises Bruker is still the only vendor who has fully implemented the capability to read, write and email their FID and spectra in JCAMP format at the time of this writing.

Why so little happened elsewhere since September 1990 is everybody's guess.

Bruker colleagues helped to create the standard back in 1986 (!) and all the UXNMR and XWIN-NMR programs in the field have JCAMP capability long since. Also our WIN-NMR software on the PCs can readily interact with JCAMP files or attachments to emails sent from our instruments. The fact that a format similar to JCAMP became native for our parameter files was just a side effect of this move. The commands '`tojdx`' and '`fromjdx`' do the conversion of both, parameters and data, into a JCAMP file. Both are explained in every manual and online help.

A Word on Binary vs. ASCII

With storage cost rapidly approaching the mark of 1 cent per megabyte the argument of actual space occupancy for a spectrum is becoming more and more obsolete. But people suggesting that binary format just *had* to be more compact than 'ASCII' have been around all along the prior decade of JCAMP-discussions.

However, the contrary is more likely.

JCAMP's DIFDUP format is *a lot smarter* than just printing up an FID as real or integers numbers in a row. Although entirely in printable ASCII, an average proton FID has about 80% of its original size when stored in JCAMP format. That includes all the parameters that are usually stored outside the binary FID files used as the 100% reference here.

Furthermore, those who can afford to sacrifice platform independence are free to apply their favorite file-zipper for an extra (up to) 30% or so, compression on those JCAMP files. We have taken the liberty to provide a few dozen spectra in JCAMP format on the WINNMR HomePage for fellow spectroscopists to check out (`http://www.bruker.com/nmr/software/winhome` ;The links given in this letter are also available from there).

An increasing number of Bruker customers have chosen JCAMP format for long-term archiving *precisely because* its totally printable ASCII. And their point is easy to understand after you had to convert, say, 100,000 NMR spectra stored over the last decade in a binary format suitable for a suddenly doomed hardware platform. Trust me, disk space is the least of their concerns.

Headed towards ENC...

Chances are that JCAMP already is the 'de facto' standard in NMR. However, it seemed to have gotten there unnoticed by parts of the NMR community.

We shall all be very grateful that Virginia and Woody had the courage to step forward and send their letter here a few weeks ago: It was this letter that helped convince the ENC committee to schedule a discussion on NMR standards in Asilomar later this year in a last-minute effort.

As NMR evolves over time there are always new issues that need to be addressed and worked into the existing version of JCAMP. And I am convinced IUPAC's Working Party on Spectroscopic Data Standards (JCAMP-DX) will welcome any suggestions spectroscopists have to make.

My very best regards,

Dr. Michael Grzonka
Software Support Manager

TU Delft

Faculty of Applied Physics

Delft University of Technology

Delft, January 23, 1998.

Dear Professor Shapiro,

Improved quantification of FAIR perfusion measurements .

Non-invasive perfusion imaging can be done using pulsed spin labelling techniques on tissue water, with labelling and detection within the same slice (1,2). A perfusion map could be calculated (3) from the difference in T_1 maps of the non-slice (ns) selective and the slice selective (ss) labelled data set (T_1 difference method). A problem is the imprecision in the perfusion rate due to the high imprecisions in the obtained T_1 maps. To improve this and increase the accuracy, here a method is introduced in which the perfusion rate f is estimated directly from the (ns) and (ss) measured magnitude data sets, using a maximum likelihood (ML) estimator which uses both data sets simultaneously. Also the sample point distribution can be optimized in advance with respect to the Cramer Rao (CR) lower bound, resulting in optimized sample schemes.
The models describing the magnitude data sets for varying sample points t_n are given by:

$$A_{ns}(n) = \left\| M_0 + (M_b^{ns} - M_0)e^{-t_n/T_1} \right\|$$

$$A_{ss}(n) = \left\| M_0 + (M_b^{ss} - M_0)e^{-t_n(1/T_1 + f/\lambda)} \right\|$$

which are just magnitude relaxation curves. The 5 parameters to be estimated are given by the vector vector β: $\beta = (M_0, T_1, M_b^{ns}, f, M_b^{ss})^T$. M_0 and T_1 are present in both model functions. The ML estimator for this combined model is given by:

$$\left(\frac{\partial \vec{A}_{ns}}{\partial \beta}\right)^T \left\{C_{ns}\vec{M}_{ns} - \vec{A}_{ns}\right\} + \left(\frac{\partial \vec{A}_{ss}}{\partial \beta}\right)^T \left\{C_{ss}\vec{M}_{ss} - \vec{A}_{ss}\right\} = 0$$

where $\vec{M}$ is a vector of measured magnitude data points and **C** a correction matrix to account for the Rician noise distribution (4) in the magnitude data set. Also the CR lower bound could be calculated and could therefore be optimized with respect to the sample point distribution.
Simulations were done using CR optimized sample schemes. Using the 5 parameter fit a 32% decrease in imprecision of the perfusion rate could be obtained, compared with the T_1 difference method. First in-vivo experiments on rat brain on a 6.3T home built spectrometer showed that using the 5 parameter fit the perfusion rate was less sensitive to systematic errors due to pulsating flow in the measurements.
The above introduced combined ML estimator has an improved imprecision compared with T_1 difference method. The calculated optimal sample schemes contained a decreased number of sample points with long recovery time, compared with the optimal sample scheme for the T_1 difference method, resulting in a decreased measurement time. Also in advance a tradeoff could be made between the obtained imprecisions and the total measurement times.

References:

1. Kim S.G. et al Magn. Res. Med **34**:293, 1995. 2. Kwong K.K. et al Magn. Res. Med **34**: 878, 1995. 3. Schwarzbauer C. et al Magn. Res. Med **35**: 540, 1996. 4. Gudbjartsson H. et al Magn. Res. Med **34**: 910, 1995.

Ole Karlsen

Wim Bovée

NMR SPECTROSCOPIST -- UNIVERSITY OF DELAWARE

The Department of Chemistry and Biochemistry of the University of Delaware seeks an NMR Spectroscopist to operate and maintain the magnetic resonance facility, including spectrometer maintenance, training of users in the proper operation of instruments, determining user competence, scheduling usage of spectrometers, performing experiments for researchers in the department not having appropriate expertise and for researchers outside the department, performing collaborative research and maintaining professional knowledge of all aspects of magnetic resonance. A Ph.D. in chemistry, biochemistry or some allied field with specialization in NMR spectroscopy is preferred. The facility consists of two Bruker liquids instruments (proton frequency of 250 MHZ), a Bruker DRX-400 multinuclear NMR spectrometer, a Bruker MSL-300 solids spectrometer, a Chemagnetics M100S solids spectrometer and a Bruker ER-200 D-SRC ESR spectrometer. Applicants should submit a curriculum vitae (which includes a complete description of experience with NMR spectroscopy) and transcripts and arrange to have three letters of recommendation sent to: Professor Dennis H. Evans, Department of Chemistry and Biochemistry, University of Delaware, Newark, DE 19716-2522, postmarked before April 30, 1998. Women and minorities are especially encouraged to apply. The University of Delaware is an Equal Opportunity Employer.

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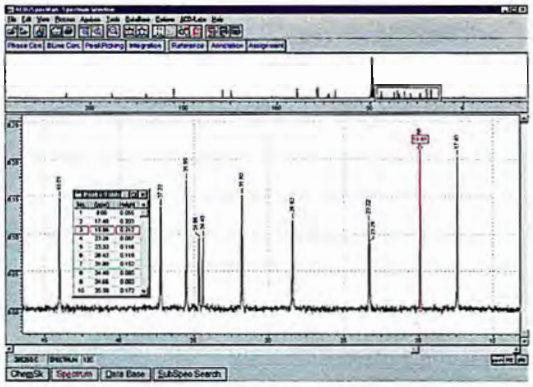

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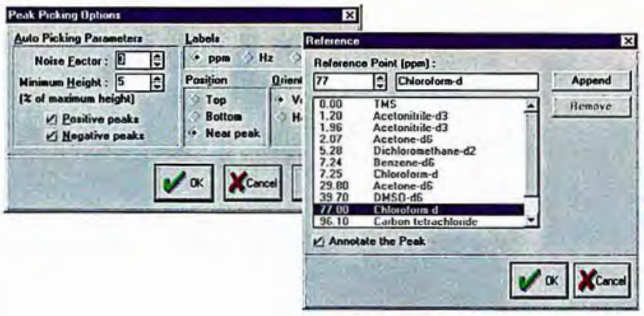

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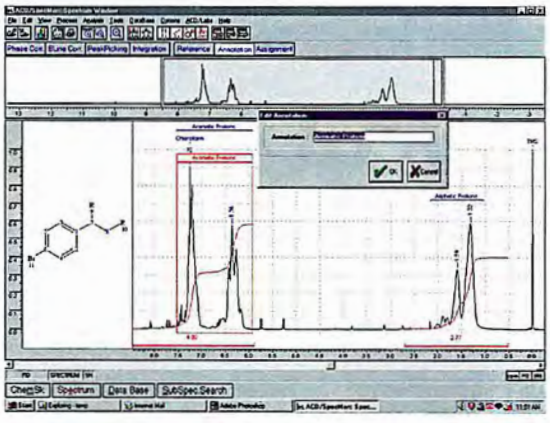

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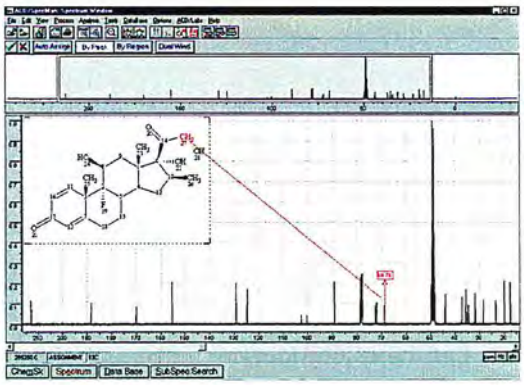

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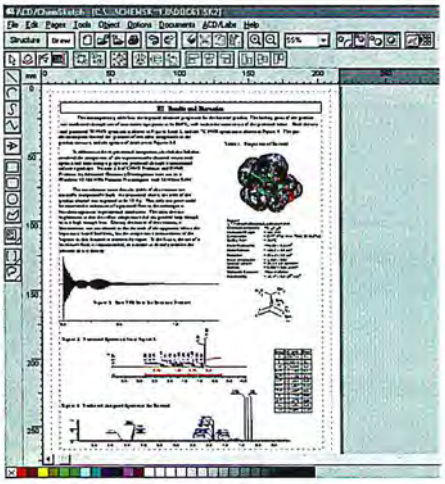

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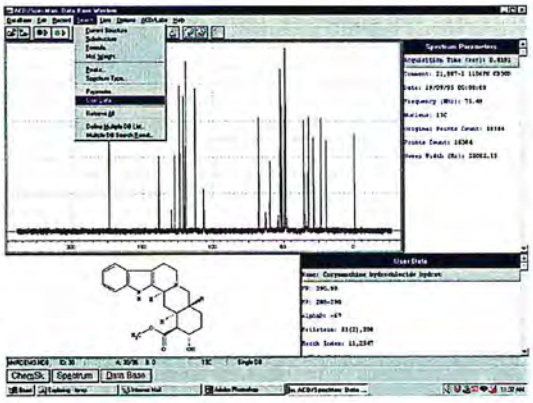

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Dr. Bernard L. Shapiro
The NMR Newsletter
966 Elsinore Court
PALO ALTO, CA 94303
USA

Vienna, February 12, 1998
(received 2/18/98)

UPGRADING OF OUTDATED NMR EQUIPMENT

Dear Dr. Shapiro,

in connection with the purchase of a 600 MHz NMR spectrometer we were encountered with the question of how to deal with our old equipment (250 MHz from 1980 and 400 MHz from 1989) in the context of generating a uniform user interface on all machines. In particular, it was not quite clear if upgrading or complete replacement would be the method of choice, especially with respect to the 250 MHz open-shop spectrometer. Finally, we were convinced by the arguments of the manufacturer (technical) and the sponsor (financial) to decide upon upgrading - with a somewhat uneasy feeling, though. In this contribution, I want to talk about the fate of our 250 MHz NMR spectrometer in the course of the eighteen years of its existence.

When the above mentioned spectrometer was purchased as a Bruker WM 250 in 1980, it was a front-end tool for multinuclear research and among the very first supercons installed in Austria. It was operated solely by a small group of NMR specialists at this time, among them me as a youngster then, and performed exceedingly well for about ten years.

With the acquisition of a spectrometer of the next generation (AM 400 WB) in 1989, we could no longer resist to the synthetic chemists' claim to provide open access to the low-field device. It soon became evident that operating a spectrometer designed in the late seventies was not as easy as imagined by the average organic chemist, and problems started to accumulate. In 1992 we finally succeeded in raising a fund for an exchange of the console, and at the same time we were able to convince the manufacturer to perform a service of the magnet whose helium consumption had risen considerably over the last years and required cold pumping at about half-year intervals. Exchanging seals (according to the manufacturers statement, especially the large o-ring at the bottom plate of the magnet was prone to cause trouble in older systems) and connecting the magnet to an AC 250 F console resulted in a very stable configuration, restricted to proton and carbon NMR now, but exceedingly well suited for open-shop work for undergraduates and staff (about 40-50 people mainly interested in black-box operation). It should be mentioned in this context that the helium hold time of the magnet exceeded the initial specs from 1980 after the above treatment and has been doing so until today.

However, there was one thing which - not very surprisingly - did not change upon the modernization: the quality of the spectra. The magnet had never been easy to shim, and in spite of stored homogeneity parameters (which, by the way, were sometimes overwritten by accident with user-created files), the ever increasing number of users did not improve on this fact. Therefore, when a new decision had to be made a year ago (leave as is / upgrade / exchange as a whole), we were not convinced that a further upgrading (now with a DPX console) would be very useful. Nonwithstanding our doubts, we were forced to accept this version due to the reasons mentioned at the beginning (the option "leave as is" was soon ruled out because of the difficulties expected from a different user interface - the 250 MHz spectrometer which was connected to an Aspect X32 by a fiber-optic link and, therefore, via ethernet to to the rest of the world during its AC-period would have been actually degraded to a stand-alone device this way).

Amazingly, the situation was completely different this time. The combination of new features like digital lock, digital filters, and digital quadrature detection in combination with last generation low-noise electronic components (preamplifiers etc.) actually led to high-quality spectra not distinguishable from those recorded with the 400 MHz spectrometer at the first glance. The significant improvement of the signal-to-noise ratio (using the same probe as before!) led to considerably shorter measurement times and thus dramatically improved the turnover rate, a point having been requested by the user community from the very beginning of the open-shop policy, and the rebellious magnet was tamed to a gentle pet by the new shim system. Together with the much easier and faster handling of the produced data on an up-to-date workstation, even hard-core syntheticists now occasionally try to measure their own spectra, and to their surprise they frequently succeed. It is needless to state in this context, of course, that the generation of routine 2D spectra (DQF-COSY, HSQC, NOESY) by routine users now at least really has become routine work. What should be explicitely mentioned, however, is the time needed for the whole upgrading process: two days from the shutdown of the old console to the completed acceptance tests for the new one and the beginning of the first retraining courses for the staff!

The upgrading of the 400 MHz spectrometer (AM 400 WB → DRX 400) taking place shortly afterwards led to similar results, but this time this was already what we expected - it is amazing how fast you get acquainted to positive experiences! The only new aspect in this connection was the sample changer which turned out to be handled much easier by now.

As a conclusion, it might be stated that upgrading NMR spectrometers is a real and much cheaper alternative to the purchase of new ones, especially if the magnet is doing well. However, even troubles with older magnets can be overcome easily using modern materials and techniques, and it obviously depends on the customer to persuade the manufacturer to include a magnet service in the deal. Perhaps this story might be of help for someone being confronted with the same problem as we were and having to come to a decision.

Please credit this contribution to the account of Dr. Hanspeter Kählig.

Yours sincerely

Hermann Kalchhauser

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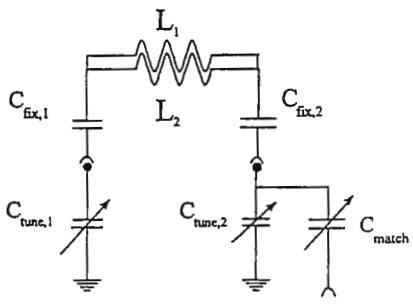
Date: February 4, 1998
(received 2/10/98)

Improved tuning range and B_1-Homogeneity of micro-imaging sample coils

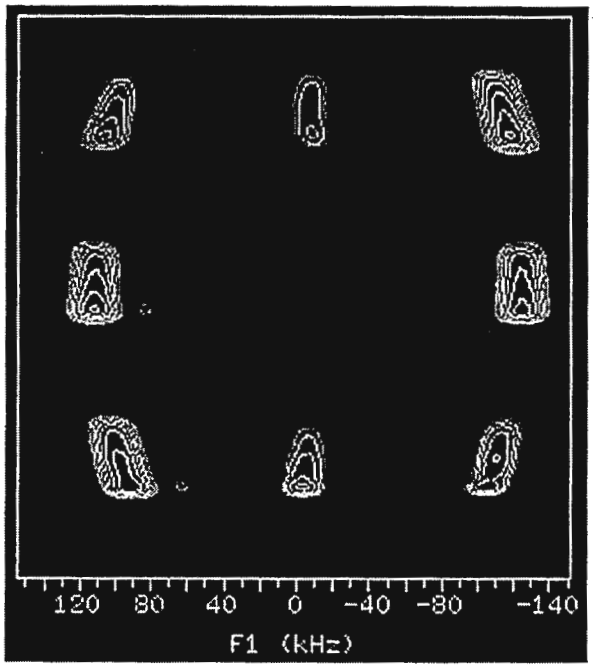
Dear Barry,
while constructing probes for our home-made micro-imaging equipment, we faced the problem of limited tuning range of the probe circuit, in particular for coils covering larger sample volumes and/or for lossy samples. One approach to work around this problem is to decrease the inductance of the sample coil while keeping the number of turns constant. This can be achieved by putting two equal coils parallel, i.e. winding two wires so that they interpenetrates each other. This halves the inductance, resulting in an improved tuning range and - as a byproduct - in a better homogeneity of the B_1- field. The price to pay is a decrease of pulse power by 50%, however, for most imaging experiments, this is acceptable. The drawing below displays the single tuned probe circuit used in our experiments. Putting two tuning capacitors makes the tuning procedure more convenient. The probe is designed to plug and unplug sample coils featuring different geometries and resonance frequencies for different applications easily. The table below compares parameters of probe circuits using single and double wired coil, respectively. Note in particular the considerably improved tuning range of the latters.

coil diameter	sample length	no. of turns	av. pulse width @ 200W	tuning range / MHz
6.5mm	15mm	8	2.7µs	380 .. 420
6.5mm	15mm	2x4	5.3µs	205 .. 430
11mm	15mm	2x4	6.0µs	375 .. 445

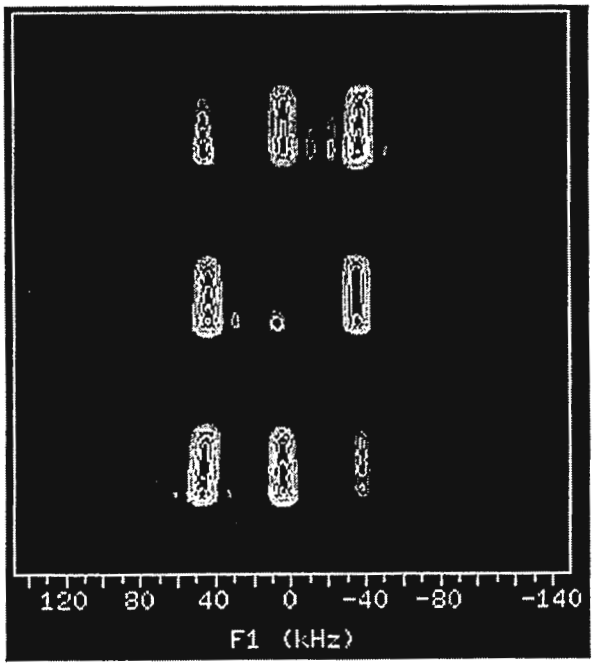


To visualize the improved B_1-homogeneity, we placed three similar rubber pieces in the sample volume and performed a spatially resolved B_1-check by the sequence HE(t_1)-[acquisition+gradient]. HE(t_1) means a Hahn-Echo where the length of the 1st pulse is incremented in a 2D manner. The gradient along the coil axis provides the spatial resolution. After 2D-FT, one can directly read the strength of the B_1-field in frequency unit vs. the spatial position along the coil axis from the 2D spectrum. The improved homogeneity as well as the loss in pulse power for the double wired coil is obvious. The peaks at F1=0 are dc-artefacts.

Currently, we are perfoming computer optimizations of the coil geometry to improve the homogeneity even more.



Single wired coil



Double wired coil

Best regards

Uwe Heuert Detlef Reichert Horst Schneider

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SYMPOSIUM ON NMR APPLICATIONS IN FOOD SCIENCE AND TECHNOLOGY

Organizing Committee: I.C. Baianu, Chairman, University of Illinois, Urbana-Champaign (UIUC), 217-244-6630; H.N. Cheng, Hercules; P. Chinachoti, University of Massachusetts; R. Clarkson, UIUC; R. Ruan, University of Minnesota; G. Turner, Spectral Data Services

THURSDAY, APRIL 2, 1998

SOLID-STATE NMR TECHNIQUES/APPLICATIONS TO FOODS & PROCESSING & FINE PARTICLES I

8:00 A.M. - 10:00 A.M. **Spectrum**

Session Chair: G. Turner, Spectral Data Services, Inc., Urbana, IL

Some Recent Studies of the Silica Surface
T. Tao, H. Lock and G.E. Maciel, Texas A&M U., College Station, TX

CP-MAS and FT-IR Studies of Fatty Acid Adsorption on Magnesium Silicate Particles
E.G. Perkins, I.C. Baianu, UIUC, Urbana, IL and T. Yates, The Dallas Group of America, Jeffersonville, IN

High-Resolution, Solid-State NMR of Silicates and Zeolites
G. Turner, Spectral Data Services, Urbana, IL

Two-Dimensional and Double-Resonance Silicon-29 NMR Spectroscopy of Minerals
J. Shore, South Dakota State Univ., Brookings, SD

SOLID-STATE NMR TECHNIQUES/APPLICATIONS TO FOODS & PROCESSING & FINE PARTICLES II

10:30 A.M. - 12:30 P.M. **Spectrum**

Session Chair: G. Turner, Spectral Data Services, Inc., Urbana, IL

To Be Announced
J. Bass, PQ Corp., Valley Forge, PA

To Be Announced
C. Little, Cabot Corp.,

To Be Announced
R.J. Kirkpatrick, Univ. of Illinois, Urbana, IL

APPLICATIONS OF MAGNETIC RELAXATION TECHNIQUES IN FOODS & BIOMEDICAL SYSTEMS

1:30 P.M. - 3:30 P.M. **Spectrum**

Session Chair: R. Clarkson, Univ. of Illinois, Urbana, IL

Recent Advances in Spin Relaxation Studies of Biopolymers
R. Clarkson, UIUC, Urbana, IL

NMR Relaxation Studies of Myosin Activity in Concentrated Electrolyte Solutions
A. M.-Gutierrez, Univ. of Texas, Dallas, TX and I.C. Baianu, UIUC, Urbana, IL

NMR Relaxation Studies of Selected Food Systems
P. Cornillon, Purdue Univ., W. Lafayette, IN

NMR APPLICATIONS IN FOODS AND Q.C.

4:00 P.M. - 6:00 P.M. **Spectrum**

Session Chair: I.C. Baianu, Univ. of Illinois, Urbana-Champaign, IL

NMR of Casein Hydration and Activity in Solutions with Ions
A. M.-Gutierrez, University of Texas, Dallas, TX

To Be Announced
T.F. Kumosinski, USDA, Philadelphia, PA

NMR and Rheological Studies of Soy Protein Interactions with Water and Electrolytes in Relation fo Food Processing
T.C. Wei, Natl. Food Res. Inst., TAIWAN

NMR Studies of Myofibrillar Proteins in Relation to Surimi Food Analogues
J.R. Lee, Natl. Res. Inst., Seoul, KOREA

FRIDAY, APRIL 3, 1998

NMR OF SILICON POLYMERS

8:00 A.M. - 10:00 A.M. **Spectrum**

Session Chair: H.N. Cheng, Hercules Inc., Wilmington, DE

Si-29 MASS, NMR and Temperature Programmed Desorption Studies of Silicagels
D. Costescu, C. Costescu, I.C. Baianu, UIUC, IL and T. Yates, The Dallas Group of America, Jeffersonville, IN

High-Resolution NMR and FT-IR of Silicon Polymers in Solution
G. Turner, Spectral Data Services, Urbana, IL and J. Bass, PQ Corp., Valley Forge, PA

Synthesis and Characterization of Optically Active Silicon-Containing Polymers
Y. Kawakami, Japan Adv. Inst. of Sci. and Tech., JAPAN

FINE PARTICLES IN FOOD PROCESSING

10:30 A.M. - 12:30 P.M. **Spectrum**

Session Chair: I.C. Baianu, UIUC, Urbana-Champaign, IL

Modelling and Monte Carlo Simulation of Cross-linked Polymers
H.N. Cheng, P.J. Cowan, and L.J. Kasehagen, Hercules, Inc., Wilmington, DE

Electron Microscopy and Monte Carlo Studies of Silica Gel Surface Structure
D. Costescu and I. Baianu, UIUC, Urbana-Champaign, IL and T. Yates, The Dallas Group of America, Jeffersonville, IN

Determination of Pore Size Distribution in Silica Polymer Particles
C. Carlos, Quantachrome Corp.,

NMR Studies of Potato Starch Hydration and Heat Processing of Potatoes from Selected Cultivars
P.I. Yakubu, Protein Technologies Inc., St. Louis, MO and E. Ozu, UIUC, Urbana, IL

NMR OF FOOD BIOPOLYMERS AND GLASS TRANSITION

1:30 P.M. - 3:30 P.M. **Spectrum**

Chairman: P. Chinachoti, Univ. of Massachusetts, Amhurst, MA

NMR and DSC of Glass Transition in Hydrated Starch Systems and Doughs
P. Chinachoti, Univ. of Massachusetts, Amhurst, MA

Deuterium and C-13 CP-MASS NMR of Hydrated Potato Starch
P.I. Yakubu, Protein Technologies Inc., St. Louis, MO and I.C. Baianu, UIUC, Urbana, IL

MRI and NMR Relaxation Studies of Food Systems
S. Schmidt, UIUC, Urbana, IL

MRI OF FOOD SYSTEMS

4:00 P.M. - 6:00 P.M. **Spectrum**

Chairman: R. Ruan, Univ. of Minnesota, St. Paul, MN

MRI of Food Doughs during Processing
R. Ruan, University of Minnesota, St. Paul, MN

MRI of Corn Drying
B. Litchfield, UIUC, Urbana, IL

NMR Studies of Water Distribution and Solid Matrix Changes in Selected Cultivars of Pumpkin during their Development
E. Ozu, and I. Baianu, UIUC, Urbana, IL

This symposium is sponsored by the Fine Particle Society and will take place at Hotel Inter-Continental, Dallas, Texas on April 2-3, 1998. There is a discount in registration fees for those also registered in the National ACS Meeting in Dallas on March 29-April 2, 1998. The discount registration fee is $150 for one day and $250 for two days. (If you are not registered in the ACS meeting, the fees are $350 for Fine Particle Society members and $410 for non-members if you register before March 10, 1998.; $50 extra after that.)

You may register by providing your name, affiliation, address, phone/fax numbers, and send check or money order payable to THE FINE PARTICLE SOCIETY, in U.S. dollars and drawn on U.S. bank, net of all bank charges, to The Fine Particle Society, 2651 East 21st Street, Suite 409, Tulsa, OK 74114. (Sorry, credit card is not accepted.) Full meeting program (including short courses and exhibition) and hotel reservation form are available from the Fine Particle Society at above address. Phone: 918-747-6544, FAX: 918-743-7644, email: fineparsoc@aol.com.

Forthcoming NMR Meetings, continued from page 1:

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

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

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

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

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

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

40th ENC (Experimental NMR Conference), Clarion Plaza Hotel, Orlando, Florida, **February 28 - March 4, 1999**;immediately preceding Pittcon in Orlando; Contact: ENC, 1201 Don Diego Avenue, Santa Fe, NM 87505; (505) 989-4573; Fax: (505) 989-1073; Email: enc@enc-conference.org.

Additional listings of meetings, etc., are invited.

E-mail Addresses Wanted

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

BLS
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Deadline Dates

No. 475 (Apr.)	27 Mar. 1998
No. 476 (May)	24 Apr. 1998
No. 477 (Jun.)	22 May 1998
No. 478 (July)	26 June 1998
No. 479 (Aug.)	24 July 1998

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

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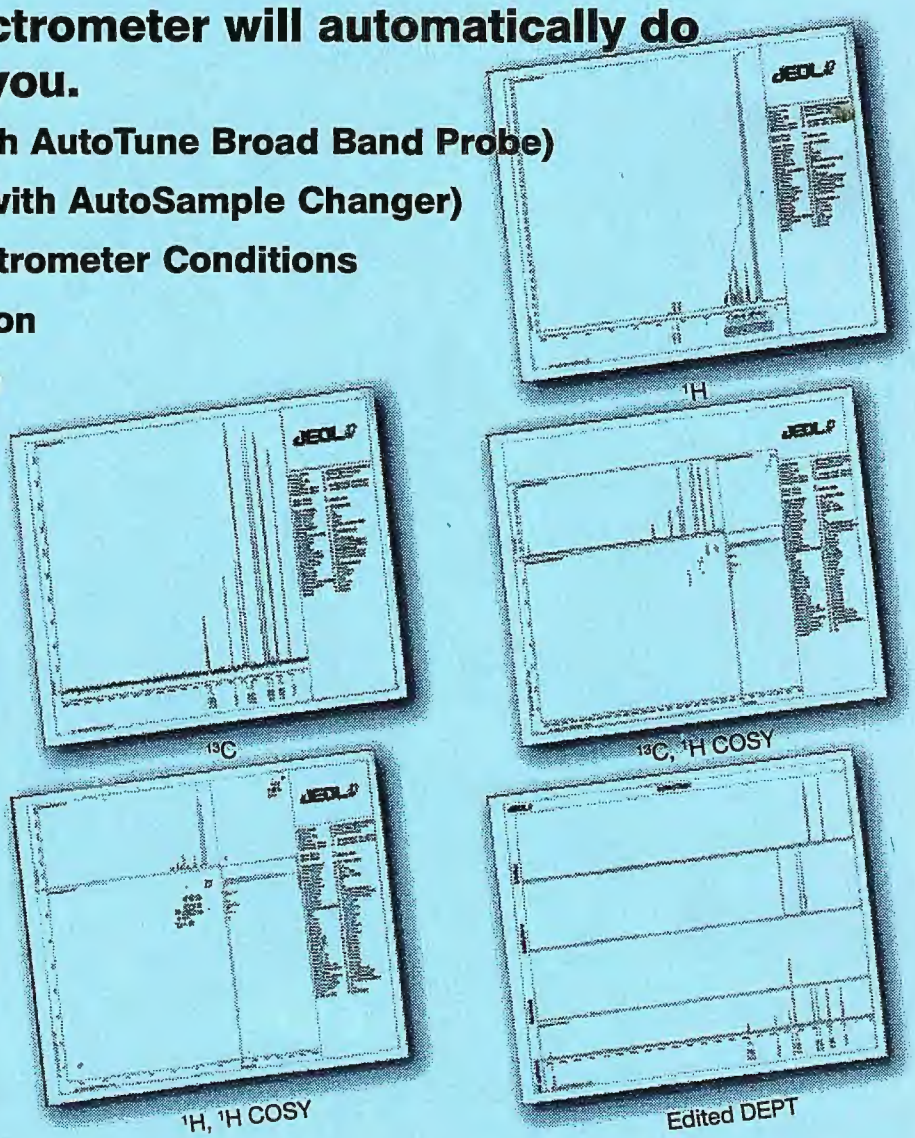

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