TEXAS A&M UNIVERSITY
NMR
NEWSLETTER

No. 377
February 1990

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TABLE 1 DEUTERATED SOLVENTS

Cat. No.	Description	Formula	Min. Isotop. (%D)	Density (g/cc)	MP (°C)	BP (°C)	$-\chi_v \times 10^6$ @ (°C)
D-11	Acetic Acid-d4	CH_3COOD	99.5%	1.1[illegible]	17	118	0.551 (32)
D-120	Acetic Acid-d4 + TMS		99.5%				
D-13	Acetone-d_6	CD_3COCD_3	99.9%	0.87	-94	57	0.460 (20)
D-121	Aceonte-d_6 + 1% TMS		99.8%				
D-129							
D-14							543 (20)
D-21							0.611
D-122							
D-130							
D-28	Choloroform-d	$CDCl_3$	99.8%	1.50	-64	62	0.740 (20)
D-31	Chloroform-d + 1% TMS		99.8%				

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VARIAN

BOX/500 SHEETS

Cat. No.	Paper Size	Instrument Model	Type
WCV-100 (S-100/A)	11" X 26"	HA-100, HA-100D, HA-100D	Gridded-Two Color
WCV-60 (S-60/C)	11" X 26"	A-60, A-60A, A-60	Gridded-Two Color
WCV-XL (XL-100)	11" X 26"	XL-100	Gridded-Two Color
WCV-XLFT-100	11" X 26"	XL-100 (CW and FT)	Gridded-Two Color
WCV-XL-200	11" X 26"	XL-200, XL-300, XL-400	Gridded-Two Color
WCV-350-2	11" X 16 3/4"	EM-350	Gridded-Two Color
WCV-20 (CFT-20)	11" X 16 3/4"	CFT-20, FT-80, FT-80A	Gridded-Two Color
WC-11[illegible]	[illegible] X 17"	All Models [illegible] 16 1/2" or 16	Blank

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UER DES SCIENCES PHARMACEUTIQUES ET BIOLOGIQUES

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LABORATOIRE DE CHIMIE ORGANIQUE

DIRECTEUR : PR BERNARD P. ROQUES

(received 12/26/89)

Dear Professor Shapiro,

Without an array processor or an AMX, the evaluation of the volume of the peaks in 2D spectra is an unrewarding work. A micro-program (PICKPEAK.AU) for DISNMRP (or DISRxx) and a little Pascal program (SOMSORT.PAS) which, when combined, make the computation of the peak volumes on 2D spectra nearly automatic, are proposed here. They function correctly on our AM 3000 with a 240K memory (and no array processor) and with a NewBury Data disk .

PICKPEAK.AU: it allows peak picking on each of the lines of a 2D spectrum and puts out results in files (size about 1K each, processed in about 12 seconds). It is possible to obtain up to 999 such output files (DISRxx name limits).
SOMSORT.PAS: to compile with PASCOM (or only with slight modifications with PASCAL). Starting from all files processed, it computes the sum of integrals of all the found peaks from one line to the successive lines where this peak is to be found (with a tolerance fixed by the user for oscillations in F2 of the maximum of the peak from line to line) (processing time for one file : about 2 seconds).
One must enter in sequence:
- the rootname of output files of PICKPEAK.AU (8 characters at most),
- the number of these files,
- the number of lines (.SMX) in the 2D spectrum,
- the chemical shifts of the first and last lines in 2D spectra (with SOMSORT, those are used to compute chemical shifts with the number of lines given above; hence, the given chemical shifts must correspond to the given number of lines, but it is not necessary for these lines to be in the output files of PICKPEAK.AU),
- the deviation (in ppm) of the maximum for a peak from one line to another in order to enable recognition by SOMSORT,
- the output filename (with extension if wanted).

SOMSORT produces a sorted listing of all the peaks found as well as their "volume".
The listing fields are obtained according to the following sequence: peak chemical shift in PPM in F2, peak chemical shift in PPM in F1, volume of the peak (sum of the various integrals which constitute the peak), beginning of the peak in F2, end of the peak in F2, frequency in F2, line in which the maximum intensity of the peak occurs in F1, line of the starting peak in F1, end of the peak in F1.

If problems arise :
i) with room in the disk or in the directory (when the disk directory is not large enough to permit a great number of files to be created by PICKPEAK.AU),
or ii) with the memory available (SOMSORT can run in a 40K memory partition if the output file to be sorted contains no more than 1300 peaks. Otherwise, so as to take higher number of peaks into account, one has to change the constant np in SOMSORT and run SOMSORT in a greater memory partition),
it is possible to do the work in several steps, selecting one after another part of the spectrum and processing them sequentially.

Writing the listing requiring an unnecessary effort, anybody interested may send a floppy (and a postcard from their area) to obtain the source and executable code of these little programs.(a 3.5" disk could also be sent to obtain the source in Macintosh text file format, since a Mac is connected with our spectrometer and 3.5" disk mailing is easier too).

Ultimately but sincerely yours,

Joel POTHIER

Please credit this contribution to the account of Professor B.P.ROQUES

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```
;*****************************************************
;  PICKPEAK.AU
; puts out integrals of 2D spectrum lines
; since NMRP does not accept to number files over
;filename.999. PICKPEAK.AU does not go over the
;999th  line of the 2D spectrum. you must stock
;;previously  SR,F1,F2,MI in a parameter JOB with
;command WJ (DISRxx).
;  micro-program PICKPEAK.AU asks for:
;   1- the 2D spectrum rootname (without .SMX)
;   2- output files rootname
;   3- parameter job rootname (MI,etc..)
;   4- rootname of region file of interest
; it takes about 12 seconds fto process one ffile
1  RF #1.SMX 001 /SPECTRUM FILENAME (SMX) ?
; in place of 001, the number of the first line in
;case of processing the spectrum in successive
;parts could be put
2  RF #2.001     /OUTPUT FILES ROOTNAME ?
; but 001 must stay here because SOMSORT takes
; the "output file.001" in first
3  RF #3.001     /PARAMETER JOB FILE
4  RF #4.001     /REGION FILE NAME
5  RSR #1.SMX    ;read 2D spectrum
6  ABC  ;could be omit if spectrum is beautiful or
;replaced by another automatic baseline correction
7  PJ #3  ;read job into memory
8  SFN #4 ; region file for next command(ppid)
9  PPID #2    ;peak picking
10 IF #1.SMX  ;next 2D line
11 IF #2        ;next output file
12 LO TO 5 TIMES L5
13 EXIT
; in L5, put the number of continuous lines of interest
;n 2D,this number will be input in
;SOMSORT to manipulate the output files in
;SOMSORT, their ;rootname must have 8
;characters at most, and you must note the
;chemical shift of the first and last lines (line 1 and
;line 999 or less) that you will input in SOMSORT
;*****************************************************
;*****************************************************
Program SOMSORT;
{computes volumes from peaks integral files
found by ppid (brucker software)}
{ and output by microprogram PEAKPICK . AU
and sorts the resulting file }
{if program crashes because of lack of
memory, results are put in SOMSORT.TMP but
not ordered}
 Var
  out: TEXT;
  nout: String; {********}
 Procedure SOMINT (Var out: TEXT; Var nout:
String); {computes volumes from 2d peaks
files found by ppid (brucker software) and
output by microprogram PEAKPICK.AU }
  Type
   LINK3D = ^PIC3D;LINK2D = ^PIC2D;
   PIC3D = Record
    delta1, delta2, int, deltad, deltaf, freq,
himax, hiprev: REAL;
     exist, MAX: BOOLEAN;
    ligne, ligned, lignef: INTEGER;
    next: LINK3D;
    End;
   PIC2D = Record
    delta1, delta2, int, freq, HI: REAL;
    found: BOOLEAN;
    ligne: INTEGER;
    next: LINK2D;
     End;
  Var
   head3d, headv3: LINK3D;
   head2d, headv2: LINK2D;
   name: String[12];
   index, nsmx, nbrefich: INTEGER;
   fich: TEXT;
   mindelta, depl1, depl2: REAL;
  Procedure  INIT;{{********}
{initialization of pointer lists}
{creating output file header}
  Begin
   NEW(head3d);head3d := Nil;
   NEW(head2d);head2d := Nil;
   NEW(headv3);headv3 := Nil;
   NEW(headv2);headv2 := Nil;
   rewrite(out);
   write(out, '-----------------------------
-------------------');
   writeln(out, '---------------------------
--');
   writeln(out, 'FIRST LINE (1) ->', depl1 : 6 :
3, ' PPM; LAST LINE (', nsmx : 3, ')->', depl2
: 6 : 3, ' PPM');
   write(out, '-----------------------------
-------------------');
   writeln(out, '---------------------------
--');
   write(out, ' PPM(F2) PPM(F1)  VOLUME
START(F2) End(F2) freq(F2) ');
   writeln(out, ' LINE(F1) START  END');
   write(out, '-----------------------------
-------------------');
   writeln(out, '---------------------------
--'); End; {********}
  Procedure INSERTP (deltax, deplx, intx,
freqx, hix: REAL; ligne: INTEGER);
{INSERTP inserts a 2D peak in 2D list,
possibly takes the room in empty 2D list
headv2}
   Var
    X: LINK2D;
  Begin
   If (headv2 = Nil) Then  NEW(X)
   Else Begin
      X := headv2;headv2 := headv2^.next; End;
    X^.found := FALSE;X^.int := intx;
    X^.hi := hix;X^.delta1 := deltax;
    X^.delta2 := deplx;X^.freq := freqx;
    X^.ligne := ligne;X^.next := head2d;
head2d  := X;End; {********}
  Procedure INSERTPP (deltax, deplx, hix,
intx, freqx: REAL; ligne: INTEGER);
{INSERTPP inserts a 3D peak in 3D list,
possibly takes the room in empty 3D list
headv3}
   Var X: LINK3D;
  Begin
   If (headv3 = Nil) Then NEW(X)
   Else Begin
      X := headv3;headv3 := headv3^.next;
    End;
    X^.exist := TRUE; X^.max := FALSE;
    X^.hiprev := hix;X^.himax := hix;
    X^.int := intx;X^.freq := freqx;
    X^.delta1 := deltax;X^.delta2 := deplx;
    X^.deltad := deltax;X^.deltaf := deltax;
    X^.ligne := ligne;X^.ligned := ligne;
    X^.lignef := ligne;X^.next := head3d;
    head3d := X;
     End; {********}
  Procedure LITFIC;
{reads the files output by the microprogram
PEAKPICK.AU}
  Var
   c: char;str: String;
   car: Array[0..9] Of char;
   I, N, N1, N2, LIGNE, cent, dix, uni: INTEGER;
    UNITE: Array[1..3] Of INTEGER;
    DEPLX, FREQX, INTX, HIX, DELTAX: REAL;
  Begin
    car[0] := '0';car[1] := '1';car[2] := '2';
    car[3] := '3';car[4] := '4';car[5] := '5';
    car[6] := '6';car[7] := '7';car[8] := '8';
    car[9] := '9';
   cent := index Div 100;
   dix := (index - cent * 100) Div 10;
   uni := index - cent * 100 - dix * 10;
   name[length(name) - 3] := '.';
   name[length(name) - 2] := car[cent];
   name[length(name) - 1] := car[dix];
   name[length(name)] := car[uni];
   writeln('READING FILE -->', name);
   RESET(fich, name);
   Repeat
    read(FICH, c);
    If eoln(FICH) Then readln(fich);
   Until (c = '.');
   Repeat
    read(FICH, c);
   Until (c = 'X');
   readln(FICH, ligne);
   DEPLX := DEPL2 + (DEPL1 - DEPL2) * (1-
ligne / NSMX);
   For I := 1 To 7 Do  readln(FICH, str);
   Repeat
    If (Not (eoln(FICH))) Then  Begin
      readln(FICH, c, N1, N2, FREQX, DELTAX,
HIX, INTX);
     If INTX > 0.0 Then
      INSERTP(deltax, deplx, intx, freqx, hix,
ligne);
    End Else  readln(fich);
   Until (eof(FICH));
   CLOSE(FICH);
   writeln('MEMORY YET AVAILABLE = ',
memavail, ' WORDS');
   End; {********}
  Procedure OLDPIC;
{sets the new 2D peak p in the 3D peak set pp
if their chemical shifts are near enough}
   Var
    p, prec: LINK2D;pp, pprec: LINK3D;
    trouve: BOOLEAN;
  Begin
   pproc := head3d;pp := head3d;
   While (pp <> Nil) Do Begin
     prec := head2d;p := head2d;
     pp^.exist := FALSE;trouve := FALSE;
    While (p <> Nil) And (Not trouve) Do
     Begin { p is not at End of list 2D and is not
yet found in 3D list}
      If ABS(p^.delta1 - pp^.delta1) < mindelta
Then   Begin {p peak is in peak set pp}
        If (p^.hi < pp^.himax) Then
         pp^.max := TRUE; {a maximum is found
for peak pp which reduces}
        If (pp^.max) And (p^.hi > pp^.hiprev)
Then  Begin {peak p is a new pic near pp, so
escape to the next p}
          prec := p;p := p^.next; {next 2D peak}
        End
       Else { pp has not yet a maximum or it is
reducing, so add p to pp} Begin
         pp^.deltaf := p^.delta1;
         pp^.lignef := p^.ligne;
          pp^.hiprev := p^.hi;
```

```
        pp^.int := pp^.int + p^.int;
        pp^.exist := TRUE;trouve := TRUE;
       If p^.hi > pp^.himax Then
       Begin ( p is the highest of  set pp )
         pp^.delta1 := p^.delta1;
         pp^.delta2 := p^.delta2;
          pp^.freq := p^.freq;
         pp^.ligne := p^.ligne;
          pp^.himax := p^.hi;End;
      If (p <> head2d) Then (p isn't head of
2D list)   Begin
         prec^.next := p^.next; (deletes found
peak p in 2D list)
         p^.next := headv2; (inserts it in
empty 2D-list)
          headv2 := p;p := prec^.next;End
      Else (if p is the head of 2D list) Begin
         head2d := head2d^.next; (insures that
head of 2D list is not loosed)
          p^.next := headv2;headv2 := p;
         p := head2d; prec := head2d;
       End;End;End
    Else
     Begin (p peak is not in peak set pp)
       prec := p;p := p^.next; (next 2D peak)
     End; (else) End; (While)
(all 2d peaks reviewed)
   If (Not pp^.exist) Then (if 3D-peak does not
live anymore)  Begin
    With pp^ Do   Begin
      write(out, delta1 : 8 : 3, delta2 : 8 : 3,
int : 8 : 3, deltad : 10 : 3, deltaf : 8 : 3);
      writeln(out, freq : 10 : 2, ligne : 9,
ligned : 8, lignef : 6);   End; (with)
    If (pp <> head3d) Then (if pp is not the
head of 3D list)  Begin
      pprec^.next := pp^.next; (deletes found
peak pp in 3D list)
      pp^.next := headv3; (inserts it in empty
3D-list)
       headv3 := pp; pp := pprec^.next;  End
    Else (if pp is the head of 3D list) Begin
      head3d := head3d^.next; (insures that
head of 3D list is not loosed)
       pp^.next := headv3;headv3 := pp;
      pp := head3d;pprec := head3d;  End
   End (if) Else Begin
     pprec := pp;pp := pp^.next;
   End; (else)End; (While)
 End; (begin)(********)
 Procedure NEWPIC;
(adds 2D-peaks not found at 3D-peaks list,
removes them from 2D list and adds them to
2D empty list)
  Var  temp: LINK2D;
 Begin
  While (head2d <> Nil) Do Begin (While peak
remains in 2D list, inserts it in 3D list)
   With head2d^ Do
INSERTPP(delta1, delta2, hi, int, freq, ligne);
   temp := head2d; (stores pointer to delete)
   head2d := head2d^.next; (deletion and jump
to next one)
   temp^.next := headv2; (insertion in empty
list)  headv2 := temp;
   End;End; (********)
 Procedure FINILISTE;
( at End of program, outputs the remaining of
the 3D peaks in the 3D list)
  Var temp: LINK3D;
 Begin
  While (head3d <> Nil) Do   Begin
   With head3d^ Do Begin
     write(out, delta1 : 8 : 3, delta2 : 8 : 3,
int : 8 : 3, deltad : 10 : 3, deltaf : 8 : 3);
     writeln(out, freq : 10 : 2, ligne : 9,
ligned : 8, lignef : 6);End;
   temp := head3d; (stores pointer to delete)
   head3d := head3d^.next; (deletion and jump
to next one)
   temp^.next := headv3; (insertion in empty
list)
    headv3 := temp;
  End; (While)
  writeln('----->END OF SEARCH');
  End;  (********)
 Procedure USERASK;
( gets information from user)
  Var  str_in: String[12];i: INTEGER;
 Begin
  name := '            '; (12 characters)
   NOUT := '';i := 1;
  writeln('ROOT NAME OF FILES FROM
OUTPUT OF PICKPEAK ?: (8 CHARACTERS AT
MOST)');
  readln(str_in);i := 1;
  While (i <= 8) And (i <= length(str_in)) Do
   Begin  name[i] := str_in[i]; i := i + 1;  End;
  writeln('NUMBER OF FILES
?:(MAX=999)[def  999]');
  readln(nbrefich);
  If (nbrefich > 999) Then Begin
    nbrefich := 999;
    writeln('MAXIMUM NUMBER OF FILES =
999');End;
  writeln(' NUMBER OF LINES IN THE 2D-
SPECTRUM :->');readln(nsmx);
  If (nsmx <= 0) Then nsmx := 1024;
  writeln('CHEMICAL SHIFT OF FIRST LINE (F1:
GREATEST):');readln(depl1);
  writeln('CHEMICAL SHIFT OF LAST LINE
(LINE ', nsmx : 4, ') ( F1 LOWEST ) :
');readln(depl2);
  writeln('PEAK VARIATION  ON F2 (0.05
RECOMMANDED)[def 0.05]');readln(mindelta);
  If (mindelta <= 0.0) Then mindelta := 0.05;
  writeln(' OUTPUT FILENAME? (WITH
EXTENSION IF WANTED):');readln(nout);
 End;  (********)
 Begin (SOMINT)
 INIT;USERASK;
 For index := 1 To nbrefich Do  Begin
   LITFIC;OLDPIC;NEWPIC;End;
   FINILISTE;close(out);End; (********)
 Procedure SORTINT (Var out: TEXT; nout:
String);
( sorts the file out output by SOMINT)
 Const  np = 1300; (if array too small, rise
np to a higher value)
(np correspond to the total peaks number)
 Type
  glsarray = Array[1..np] Of real;
  gliarray = Array[1..np] Of integer;
 Var
  outsort: TEXT; str: String[80];c: char;
  dim_array, i: integer;
  ppm2, ppm1, int3d, stf2, Endf2, freqf2:
glsarray;
  linef1, stf1, Endf1, indx: gliarray; (****)
 Procedure indexx (n: integer; arrin:
glsarray; Var indx: gliarray);
(from Numerical
Recipes;Press,Flannery,Teukolsky,Vetterling;
Cambridge university Press)
  Var
   l, j, ir, indxt, i: integer;q: real;
   bool: boolean;
 Begin
  bool := true;
  For j := 1 To n Do  Begin
    indx[j] := j; End; (for)
  l := (n Div 2) + 1;ir := n;
  While bool Do Begin
   If (l > 1) Then   Begin
     l := l - 1; indxt := indx[l];
     q := arrin[indxt]
   End Else Begin
     indxt := indx[ir];q := arrin[indxt];
     indx[ir] := indx[1];ir := ir - 1;
    If (ir = 1) Then
     Begin (indx[1] := indxt;)
      l := 1;bool := false; End (if)
   End; (else)
   i := l;j := l + l;
  While (j <= ir) And bool Do  Begin
    If (j < ir) Then Begin
      If (arrin[indx[j]] < arrin[indx[j + 1]])
Then
       j := j + 1; End; (if)
    If (q < arrin[indx[j]]) Then Begin
       indx[i] := indx[j];i := j;j := j + j
     End (if)  Else j := ir + 1
   End; (While)
   indx[i] := indxt
  End; (While)
 End; (begin)(********)
Begin (sortint)
 reset(out,'SOMSORT.TMP');rewrite(outsort,
nout);
 For i := 1 To 5 Do   Begin
   readln(out, str);writeln(outsort, str);End;
 i := 1;
 While (Not eof(out)) and (i<=np) Do  Begin
   readln(out, ppm2[i], ppm1[i], int3d[i],
stf2[i], Endf2[i], freqf2[i], linef1[i], stf1[i],
Endf1[i]); i := i + 1;End;
 If (i > np) Then Begin
  writeln('MORE THAN ', np : 5, ' PEAKS');
  writeln('CANNOT ORDER OUTPUT FILE);
  writeln('READ RESULTS DISORDERED IN
SOMSORT.TMP');
  close(out);close(outsort, delete);
  exit ( PROGRAM );End;
  dim_array := i - 1;
  writeln('MEMORY YET AVAILABLE = ',
memavail, ' WORDS');
  indexx(dim_array, ppm2, indx);
  For i := 1 To dim_array Do  Begin
    write(outsort, ppm2[indx[i]] : 8 : 3,
ppm1[indx[i]] : 8 : 3, int3d[indx[i]] : 8 : 3,
stf2[indx[i]] : 10 : 3);
    writeln(outsort, Endf2[indx[i]] : 8 : 3,
freqf2[indx[i]] : 10 : 2, linef1[indx[i]] : 9,
stf1[indx[i]] : 6, Endf1[indx[i]] : 6); End;
  close(outsort);End; (********)
Begin
 rewrite(out, 'somsort.tmp');
 somint(out, nout);sortint(out, nout);
 close(out, delete);
End.
```

Z•SPEC
Four Nuclei Probe

FEATURES:

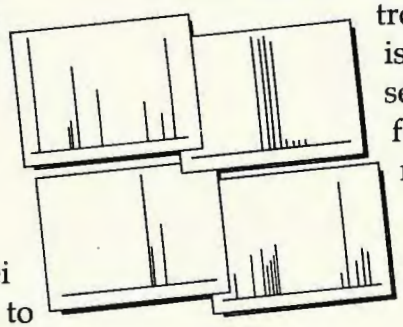
^{1}H, ^{19}F, ^{31}P and ^{13}C observe capability without retuning the probe. The four spectra shown on the back were obtained using this probe. The only thing the NMR operator does is change the observe frequency of the spectrometer. The probe contains no internal switches and thus cannot wear out from repeated observe nuclei changes.

APPLICATIONS:

The Z•SPEC Four Nuclei Probe is a great addition to any NMR lab requiring high efficiency of sample throughput. Laboratories with automatic sample changers or open access environments benefit from the increase in experimental flexibility.

TECHNICAL:

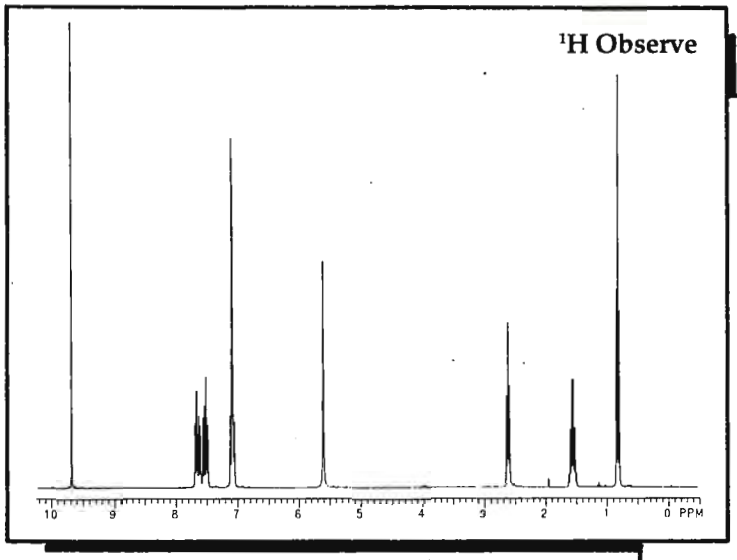
The Z•SPEC Four Nuclei Probe interfaces directly to any Varian 200, 300 or 400 MHz NMR Spectrometer. The probe is capable of observing any of the four nuclei without retuning the observe frequency or changing 1/4 wavelength cables.

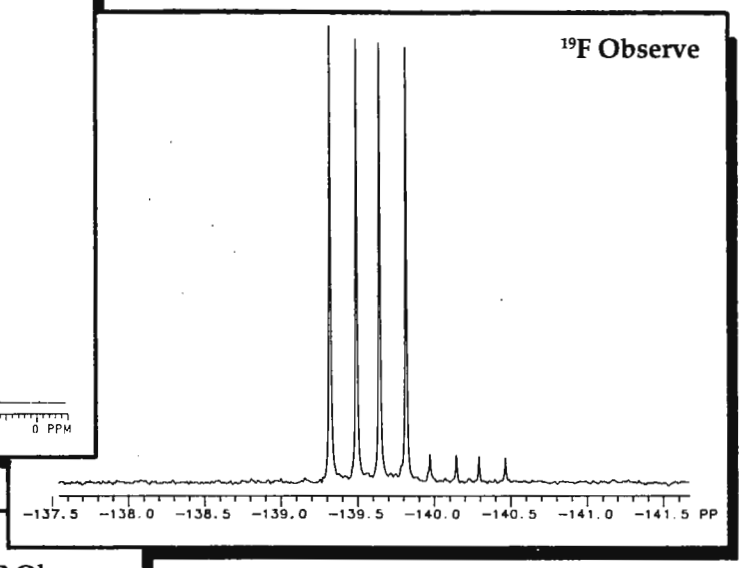


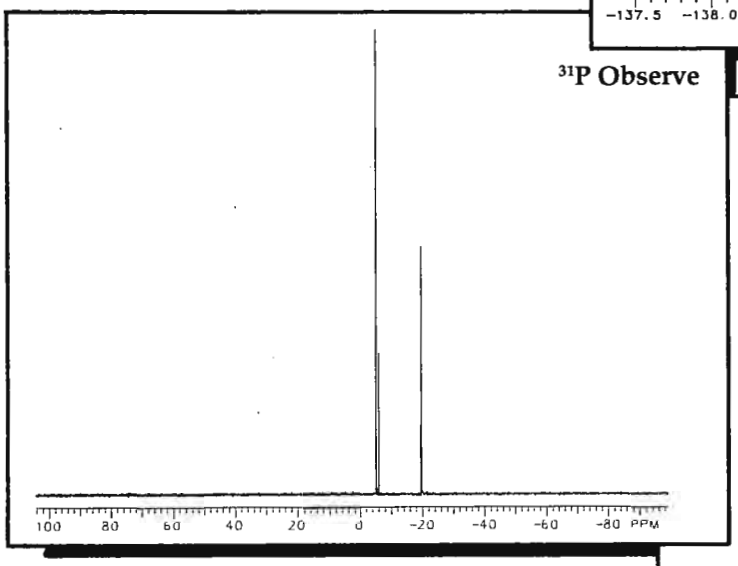
For more information, please contact Toby Zens, Manager of the Z•SPEC Products Group or visit us at ENC in the Scripps Suite.

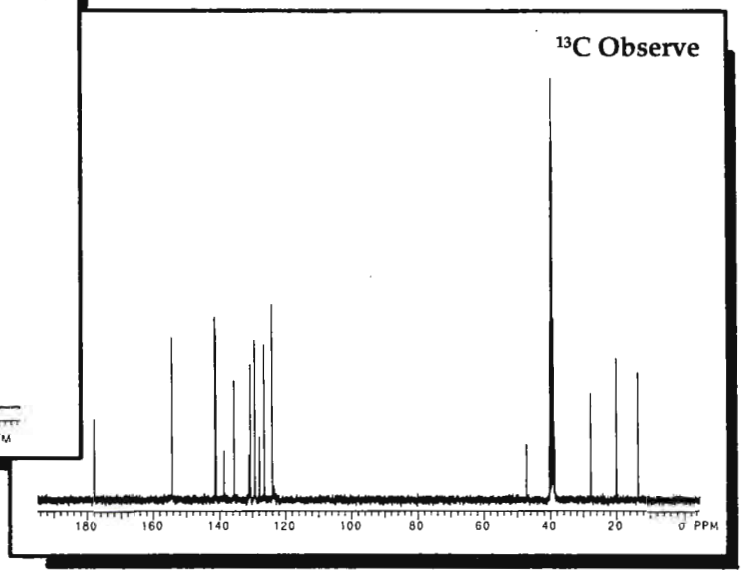
NALORAC CRYOGENICS CORPORATION
837 Arnold Drive, Suite 600, Martinez, CA 94553
Tel: (415) 229-3501 • FAX (415) 299-1651

Z•SPEC Four Nuclei Probe Spectra*









* Spectra obtained with spectrometer operating in an automatic and unattended mode.

NALORAC CRYOGENICS CORPORATION
837 Arnold Drive, Suite 600, Martinez, CA 94553
Tel: (415) 229-3501 • FAX (415) 299-1651

University of Oregon
Institute of Molecular Biology, Eugene OR 97403-1229

Identification of NOE's Between Isotopically Labeled Protons in T4 Lysozyme Using ^{15}N ω_1- and ω_2- Double-Edited NOESY Experiments

Dear Dr. Shapiro;

Dec. 18, 1989
(received 12/22/89)

We have recently assigned the backbone ^1H- and ^{15}N-NMR resonances of bacteriophage T4 lysozyme. Instrumental to this work was the use of ^{15}N ω_2-edited COSY and NOESY experiments to study selectively or uniformly ^{15}N enriched protein samples. The ω_1-edited and ω_2-edited experiments, which have been described by several research groups (Otting *et al.*, 1986 *J. Mag. Reson. 70*, 500; Griffey and Redfield, 1987 *Q. Rev. Biophys. 19*, 51; Bax and Weiss, 1987 *J. Mag. Reson. 71*, 571; McIntosh *et al.*, 1987 *J. Biomol. Struct. Dyn. 5*, 21), are two-dimensional ^1H-NMR measurements with the first or last pulse, respectively, replaced by a heteronuclear difference echo pulse sequence. This sequence selects for (or against) protons which are directly bonded to ^{13}C or ^{15}N nuclei. The final two-dimensional spectra are asymmetric, having only crosspeaks from labeled protons to all possible protons (ω_1-edited) or from all possible protons to the labeled protons (ω_2-edited). In figure 1, the downfield portion of an ω_2-edited NOESY spectrum of T4 lysozyme selectively labeled with ^{15}N-alanine is shown. Of particular interest are three sets of crosspeaks which are symmetrically disposed about the diagonal (enclosed within boxes). This pattern suggests that these crosspeaks arise from H^N_i-H^N_{i+1} interactions between labeled alanines. There are three alanine-alanine dipeptides in helices in T4 lysozyme. One approach to confirm that the NOE's are between labeled alanine amides, and not due to coincidental degeneracies, is to run this experiment without ^{15}N decoupling and identify NOE's from ^{15}N bonded protons on the basis of the splitting in ω_1 due to the large $^1J_{HN}$ coupling (Griffey and Redfield, 1985 *J. Mag. Reson. 70*, 500). An alternative approach is to use an ω_1- and ω_2-double edited NOESY experiment which will detect only interactions between two ^{15}N (or ^{13}C) labeled protons:

^1H:	90	--Δ--	180	--Δ--	t_1	90	t_{mix}	90	--Δ--	180	--Δ--	observe
$^{15}N/^{13}C$:		------	90_x 90_ϕ	------	(decouple)				------	90_x 90_ψ	------	(decouple)

(where $\Delta = (2J_{HN})^{-1}$ or slightly shorter, ϕ = x,-x,x,-x and ψ=x,x,-x,-x, and the observation is +,-,-,+, followed by standard NOESY phase cycling). The ^{15}N $\omega_{1,2}$-edited NOESY spectrum of the same labeled T4 lysozyme sample is shown in figure 2. Only three crosspeaks are observed, confirming that these arise between labeled amide protons.

The $\omega_{1,2}$-edited experiment differs from the X-filtered experiment described by Wörgötter *et al* . (1988 *J. Am. Chem. Soc. 110*, 2388) which selects for crosspeaks between protons coupled to the *same* heteronucleus. Although the signal-to-noise of this $\omega_{1,2}$-edited experiment is low due to the time required for two editing sequences, figure 2 demonstrates that interpretable spectra can be obtained in overnight runs with a practical sample of a "large" protein such as T4 lysozyme (18.7 kDa). The $\omega_{1,2}$-edited NOESY (or COSY) experiment is useful for specifically identifying interactions between ^{15}N or ^{13}C labeled protons. For example, postulated NOE's between residues in a protein or between a protein and a ligand could be tested using this experiment with the appropriate isotopically labeled samples. Also, using a triple resonance probe, NOESY and COSY spectra could be edited to detect crosspeaks between ^{13}C *and* ^{15}N labeled protons.

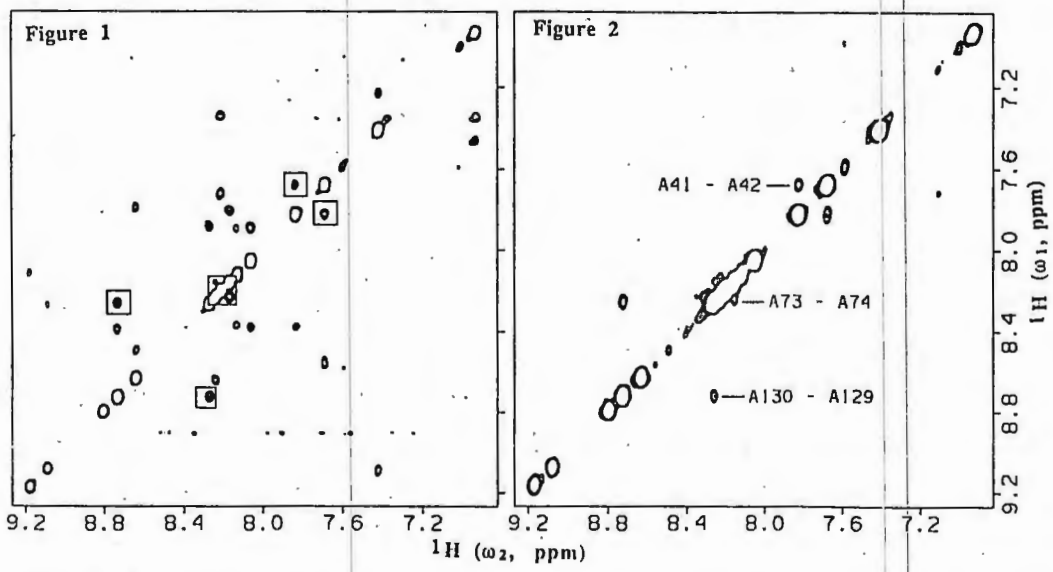


Figure 1: The downfield portion of an ^{15}N ω_2-edited NOESY spectrum of T4 lysozyme selectively labeled with ^{15}N-alanine. The sample was ca. 3 mM protein at pH 5.6 and 20°C. The spectrum was collected on a GN500 spectrometer as a 300 complex t_1 x 1024 complex t_2 data set in 18 hr. with 128 acquisitions per t_1. The mixing time was 100 msec, Δ was 4.5 msec, MLEV-64 broadband ^{15}N decoupling was used during both t_1 and t_2, and the signal from water was suppressed by selective saturation at all times except during observation. NOE crosspeaks from both labeled and unlabeled protons to the ^{15}N labeled alanine amide protons are observed.

Figure 2: The ^{15}N ω_1- and ω_2-edited NOESY spectrum of the same T4 lysozyme sample. The spectrum was recorded as a 200 complex t_1 x 1024 complex t_2 data set in 23 hr. with 256 acquisitions per t_1. The other parameters were essentially as in figure 1. Only the H^N_i-H^N_{i+1} NOE crosspeaks between ^{15}N labeled alanine amide protons (enclosed within boxes in Figure 1) are observed.

Lawrence

Lawrence McIntosh

Rich

F. W. Dahlquist

ME-182

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PHARMACEUTICALS & BIOTECHNOLOGY R&D
MEDICAL PRODUCTS DEPARTMENT

December 14, 1989
(received 12/19/89)

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

Dear Barry:

Better Productivity with 3 and 4 Nucleus Probes for XL/VXR/UNITY

Our Med Chem NMR service lab recently retired an IBM 200 MHz spectrometer that had been in use since 1982. During its life here, the instrument produced over 109,000 spectra (must be some kind of record)! In the last two years it was yielding about 24,000 proton spectra/year. With concerns about instrument age, backup, turnaround time and the ever increasing demands of a growing staff, we decided the time had come replace "old faithful".

The customer base for the Med Chem service lab is largely synthetic organic chemists who rely on NMR to confirm the products of reaction . They include 100^+ medicinal chemists, about a dozen fluorocarbon chemists and several other groups that also use our service. To satisfy the customers' needs, the new equipment had to be capable of producing at least 150 ^1H and ^{19}F spectra during daytime hours (turnaround is critical), and of automatically running ^{13}C and 2-D spectra overnight.

To accomplish this we purchased and installed two VXR-300S spectrometers during the first half of 1989. These were equipped with Sun 3/60 computers and ASM-100 sample changers (100 sample version). As expected, the startup effort was substantial, but we are very pleased with the results. The automatic operation is producing spectra that, on the average, are of higher quality than we got manually. Throughput of the system is amazing. Cycle time for a proton spectrum is on the order of 4 minutes, resulting in a total 8 a.m. - 5 p.m. capacity of over 250 samples.

Triple nucleus ($^1H/^{19}F/^{13}C$) probes for the VXRs have played a significant role in the success of the system. They totally eliminate the need for manual intervention (tuning or probe changes) when switching between ^1H and ^{19}F. This allows the sample changer to process any random mix of ^1H and ^{19}F spectra. These probes were ordered through Varian as part of the system, but they were actually built by Toby Zens of Nalorac.

The Zens probes use multiply tuned circuits that still achieve excellent signal-to-noise performance. Multiple tuning eliminates the need to mechanically switch pretuned circuits elements within the probe. Relative to Varian switchable probes that I have used, these Zens probes have better sensitivity. They also have 90° pulse widths that are shorter by 50 percent. Resolution and line shape are excellent.

Our original Zens probes were built on modified Varian probe bodies. Toby has recently designed a probe body that creates more room for electronics. The extra room has made it possible to build a $^1H/^{19}F/^{13}C/^{31}P$ "quad" probe. Although our quads haven't arrived yet, Bill Hutton at Monsanto and Tony Foris at DuPont, Jackson Labs both have them installed. I have seen data from Tony's instrument. Adding the fourth nucleus didn't compromise performance at all.

Our triple nucleus probes have significantly improved instrument productivity and the quads should be even better. From my standpoint, its hard to see how an NMR service lab can afford to be without one. Bruker has produced quads for some time. Toby Zens obviously is building them. Where are you, G.E., JEOL, Varian?

We are also developing a spectroscopic data system network that will use a Sun 4/370 computer as a file server. The unit is equipped with an optical WORM disk for archiving. I would enjoy talking with anyone interested having a similar interest.

Please credit this note to Don Bly's account.

Yours sincerely,

Mac

J. M. Read
302/695-3517

Bat-Sheva Workshop on
NEW DEVELOPMENTS AND APPLICATIONS
IN NMR AND ESR SPECTROSCOPY
Israel, October 14-24, 1990

The aim of the workshop (school) is to review the principles and applications of advanced NMR and ESR techniques with particular emphasis on recent developments in pulse techniques.

The topics to be covered are: **A.J. Shaka** (Irvine): Multiple pulse schemes in high resolution NMR, **O.W. Sørensen** (Zürich): Principles of 2D NMR spectroscopy, **A.E. Derome** (Oxford): Strategies in structure determination by high resolution NMR, **A. Pines** (Berkeley): Multiple quantum spectroscopy, **S. Vega** (Rehovot): Magic angle sample spinning NMR, **A.J. Vega** (Wilmington): NMR and NQR of non integral spin quadrupolar nuclei, **Z. Luz** (Rehovot): Structure and dynamics using spin I=1 NMR spectroscopy, **D.I. Hoult** (Bethesda): Physical principles of NMR imaging, **T.R. Brown** (Philadelphia): In-vivo localized spectroscopy, **K. Möbius** (Berlin): ESR, ENDOR and Triple resonance, **J.R. Norris** (Chicago): Pulsed Fourier transform ESR spectroscopy, **J.H. Freed** (Ithaca): Two dimensional ESR spectroscopy, **A. Schweiger** (Zürich): Multiple resonance and multiple pulse ESR spectroscopy, **L. Kevan** (Houston): Electron spin echo spectroscopy, **J. Schmidt** (Leiden): Electron spin echo spectroscopy of optically excited states.

For further information please write to:

Dr. Daniella Goldfarb, Department of Isotope Research, The Weizmann Institute of Science, 76 100 Rehovot, Israel.
Phone: 972-8-482016, Fax: 972-8-466966, Telex: 381300,
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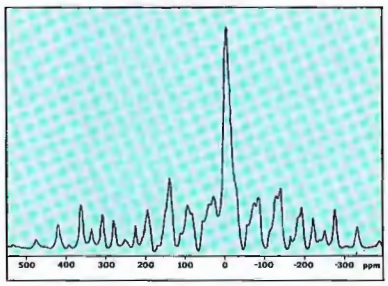

UNITY 300 MAS Spectrum of Aluminum in Sapphire.

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NMR WITH A FUTURE

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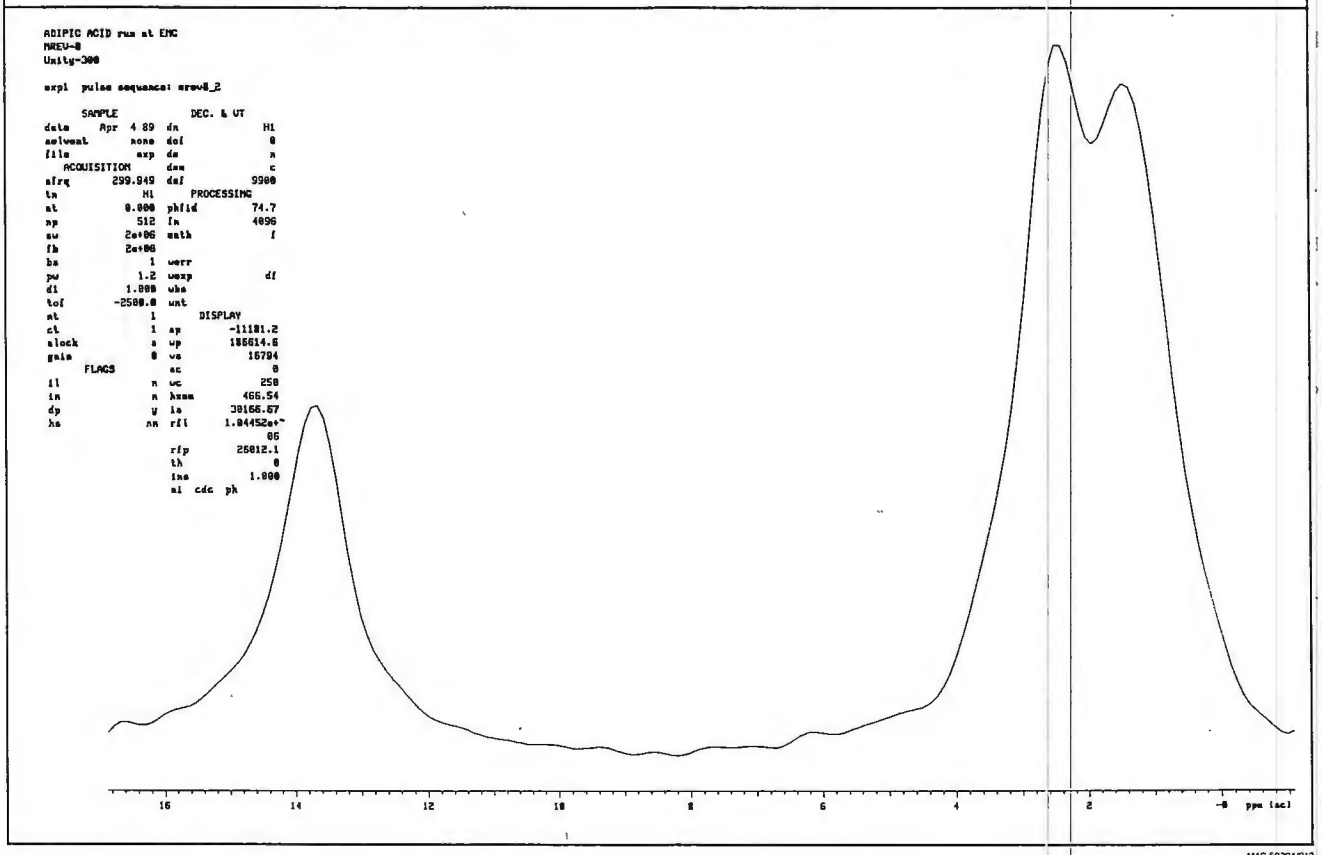
CRAMPS
ADIPIC ACID
MREV-8

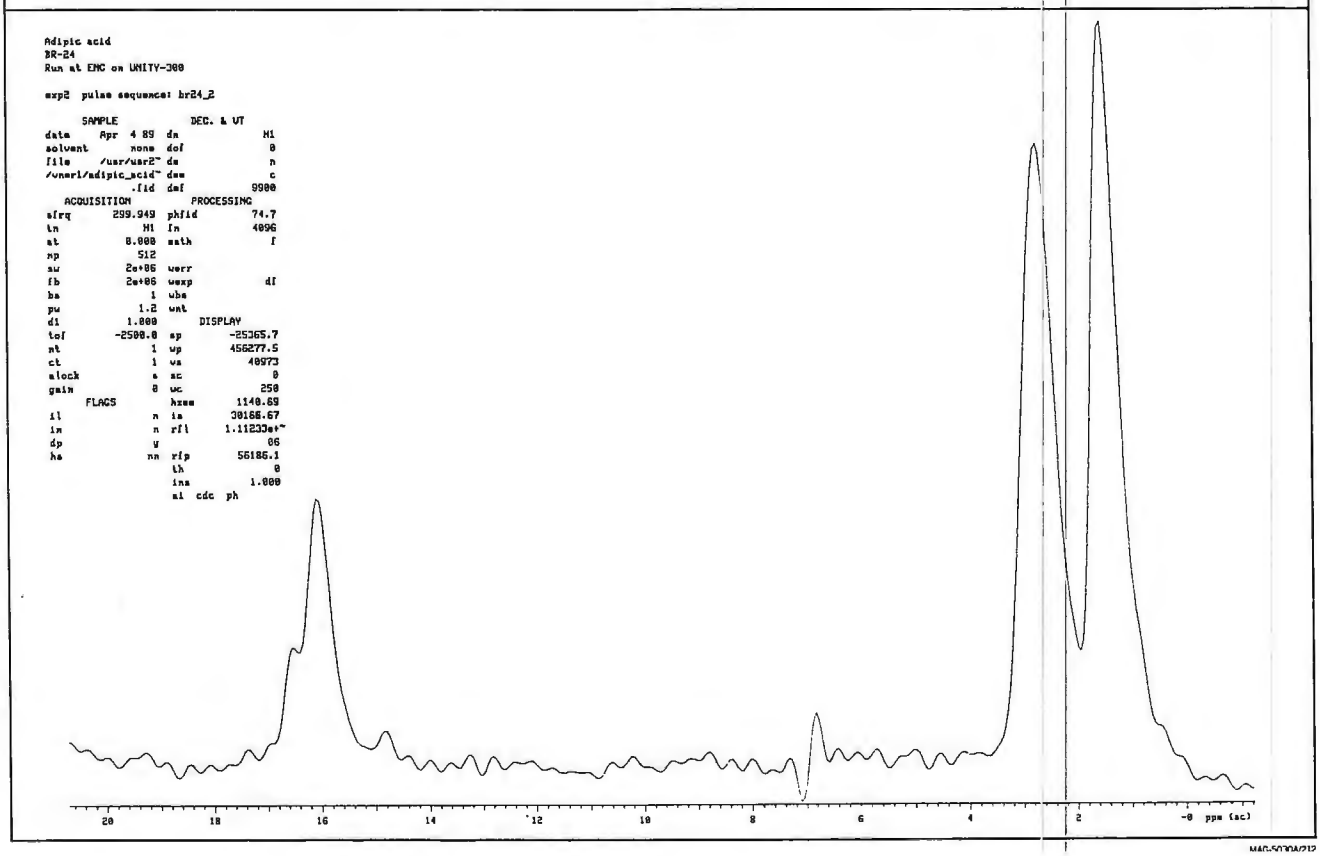
MREV-8 may be used in CRAMPS (Combined Rotation And MultiPulse Spectroscopy) as it is often not as subject to artifacts as is the BR-24 sequence. It is usually wise to run both sequences on a new sample. Adipic acid is shown here using MREV-8 on a UNITY-300.



MAG-5030A/212

CRAMPS
ADIPIC ACID
BR-24

With the same sample as that used for MREV-8, the results displayed here were acquired using BR-24. A number of differences between the two spectrums are obvious. For instance, the resolution is much higher, being of the order of 0.3 ppm full width at half height for highest field peak. There is also a rotor resonance line at about 7 ppm.



MAG-5030A/212

UNIVERSITY OF TURKU

KALEVI PIHLAJA
Professor, Dr.
Physical Chemistry

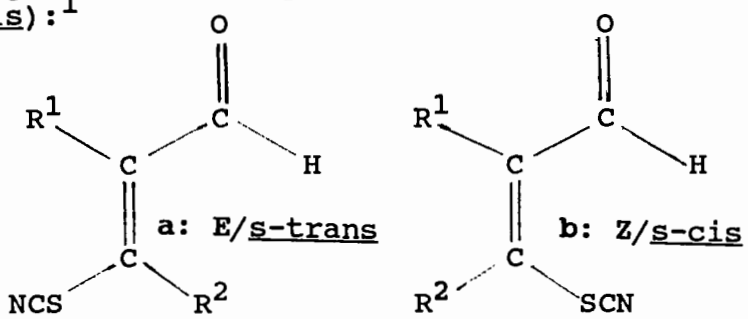
Dr. Bernard L. Shapiro
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto, CA 94303
U.S.A

Turku, 8.12.1989
(received 12/22/89)

^{17}O NMR AS A PROBE FOR THE E/Z-ISOMERISM OF β-THIOCYANATO VINYLALDEHYDES

The ^{17}O chemical shifts for some isomeric β-thiocyanato vinylaldehydes were determined and appeared to be useful in assigning their E/Z-isomerism.

E/Z configurations of some β-thiocyanato vinylaldehydes **1-4** has been studied by means of the ^{1}H NMR chemical shifts of the aldehyde protons and LIS of all protons, which also allowed the assignment of the preferred conformations (**a**: E/s-trans, **b**: Z/s-cis):[1]



R^1	R^2	
Me	Me	**1a**
$-(CH_2)_3-$		**2b**
H	Ph	**3a**
H	Ph	**3b**
Ph	Me	**4a**

The ^{17}O chemical shifts of a variety of carbonyl groups are very sensitive to both electronic[2] and steric effects.[3] Therefore we decided to record the ^{17}O NMR spectra of compounds **1-4** especially to solve the geometric isomerism (E or Z) of compound **4**, which was not succesful with the earlier approach.[1]

Clearly different ^{17}O chemical shifts (Table 1) were observed for the E/Z isomers and compounds **3** show a 14.4 ppm lowfield shift when going from the E to the Z form. Two effects can be responsible for this observation, namely the van der Waals interactions are less repulsive in the Z than in the E form[3] and/or the oxygen lone pairs of the Z form are delocalized toward the antibonding σ^* orbital of the SCN group.

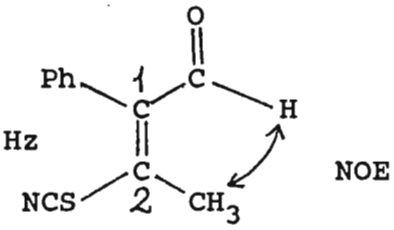
In the light of the ^{17}O chemical shifts for **1-3** there is no doubt that compound **4** (δ = 567.2 ppm) must represent the E form. This result and the s-trans-conformation[1] of **4** were confirmed by the coupled ^{13}C NMR spectrum and by the H,H-homonuclear NOE difference spectra obtained by irradiating the CHO and the methyl signals.

The remarkable NOE of the CHO-proton on the methyl protons and vice versa fix the E/s-trans stereoisomerism of **4**. The same conclusion could be drawn from the characteristic vicinal coupling constant $^{3}J_{C,H}$ = 2.5 Hz (the other ^{13}C NMR parameters for **4** are listed in Table 2).

Department of Chemistry
SF-20500 Turku, Finland

Telephone 921-6/35705
Telex 62683 tyf sf
Fax 358-21-319836

$^3J_{C,H}$ = 2.5 Hz



Probably the ^{17}O chemical shifts can be utilized as a new probe for assigning the E/Z-isomerism even if more data are needed to prove that a general phenomenon is in question.

^{17}O NMR spectra (Table 1) were recorded at 54.1 MHz on a JEOL GX-400 spectrometer at 35°C without proton decoupling with a 20 μs (= 45°) pulse, 3 Hz digital resolution, 80 ms AQ and 0.075 s repetition time. The reference was external water. Normally 200 scans were accumulated. The sample concentration in each case was ca 2.5 M in $CDCl_3$.

1. B. Schulze, R. Brämer, E. Kleinpeter and M. Mühlstädt, J. prakt. Chem. 318, 795 (1976).
2. P. Balakrishnan, A. L. Baumstark and D. W. Boykin, Org. Magn. Reson. 22, 753 (1984).
3. D. W. Boykin, R. L. Hertzler, J. K. Delphon and E. J. Eisenbraun, J. Org. Chem. 54, 1418 (1989).

Table 1. ^{17}O chemical shifts of compounds 2-4.

Compound	1E	2Z	3E	3Z	4
δ (±1 ppm)	567.6	576.2	570.1	584.5	567.2

Table 2. ^{13}C NMR parameters of 1-phenyl,2-methyl,2-thiocyanato-vinylaldehyde 4 (δ in ppm, J in Hz).

	δ/J			
CH_3	24.2	^1J 131.7		
Ph	p 128.8	^1J 168.8; ^3J 7.5	m 129.2	^1J 161.2; ^3J 7.7
	o 129.3	^1J 158.4; ^3J 7.3	i 135.0	^3J 7.4
CHO	190.6	^1J 181.6		
SCN	110.6			
C1	135.9 (m)			
C2	144.4	^1J 144.4; ^3J 2.5; ^2J 6.5		

Kalevi Pihlaja and Jorma Mattinen

Erich Kleinpeter
Sektion Chemie der
Martin-Luther-Universität
Halle-Wittenberg, GDR

Bärbel Schultze
Sektion Chemie der
Karl-Marx-Universität
Leipzig, GDR

Colorado State University

Department of Chemistry
Fort Collins, Colorado 80523

December 12, 1989
(received 12/23/89)

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

LABORATORY UPGRADE USING SUN WORKSTATIONS FOR SPECTROMETER CONTROL

Dear Barry:

For several months now, we have been using Unix-based engineering workstations for both NMR acquisition and spectrometer control, as well as spectral data reduction because of the power and relatively low cost of these computers.

In implementing this plan (figure 1), we have upgraded three ten-year old NT-type spectrometers. After replacement of the aging 1180 computers and their associated disk drives, pulse programmers and digitizers, the new spectrometer interface (figure 2) connects a Sun Microsystem workstation, a commercial pulse programmer, an acquisition processor, and two 2 MHz digitizer modules that were fabricated in-house.

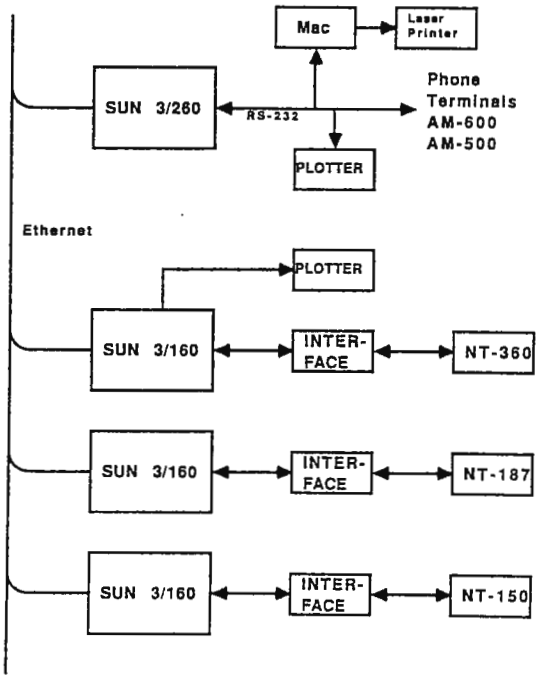


Figure 1. HARDWARE OVERVIEW

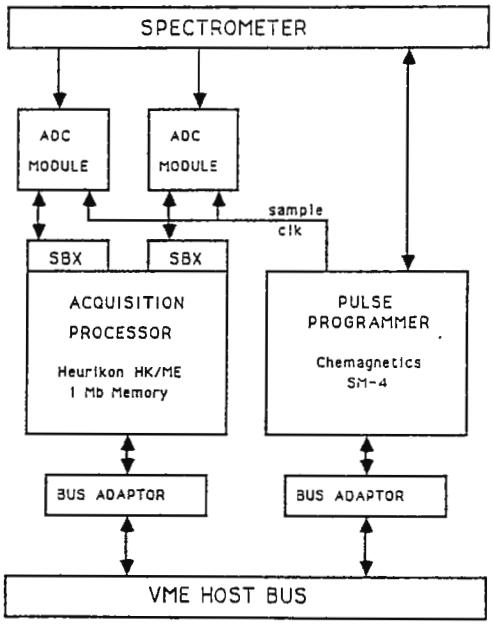


Figure 2. SPECTROMETER INTERFACE

Processing software, kindly supplied by Hare Research, was modified and linked to an acquisition module that was written in-house to allow collection of both 1D and 2D data sets. Pulse programs and parameters (Unix ASCII text files) are conveniently written and edited in the operating system's window environment and are assembled, loaded, and executed at go time under control of the software acquisition module.

This laboratory upgrade, consisting of several networked computers and spectrometers, has allowed us to make use of modern computer technology, extend our pulse programming capabilities and extend the useful lives of three spectrometers. We would be happy to provide further details to interested readers.

Sincerely,

Gary

Gary E. Maciel

Bruce

Bruce L. Hawkins

377-16

Oklahoma State University

DEPARTMENT OF CHEMISTRY
COLLEGE OF ARTS AND SCIENCES

STILLWATER, OKLAHOMA 74078-0447
PHYSICAL SCIENCES 106
405-744-5920

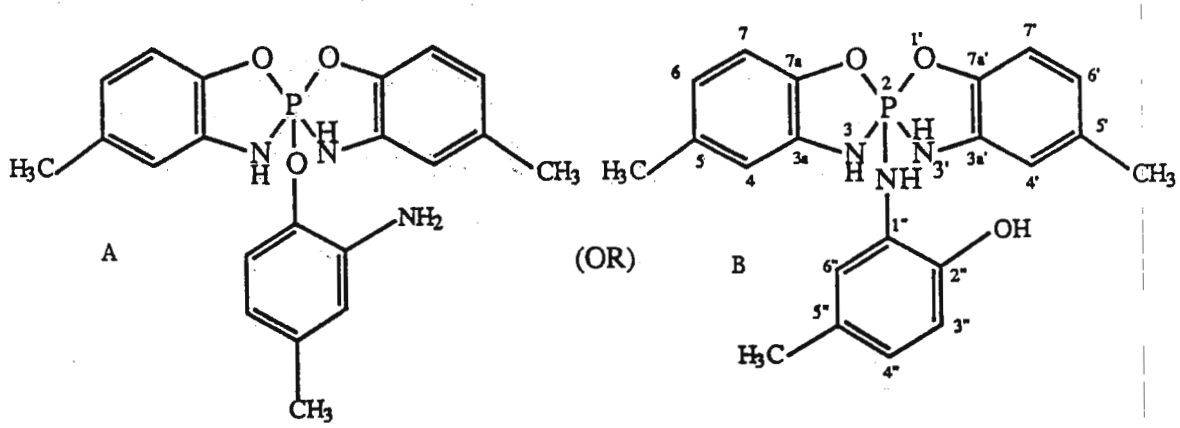
December 20, 1989
(received 12/22/89)

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

Title: Novel Pentavalent Phosphorus Heterocycles

Dear Barry:

In some recent work we have discovered some interesting and novel chemistry in collaboration with Dr. C. D. Reddy of S V University, Tirupati, India. One product isolated from a reaction mixture appeared to have "extra" peaks in the C-13 NMR spectrum. Using our XL-400 NMR spectrometer we were able to detect two P-31 signals at 11.88 and 13.12 ppm in $DCCl_3$. Consequently, although this product melts at 226-227°C and appears to be homogeneous, in solution two isomeric materials may form. We believe the solid compound to be a pentacoordinate, phosphorus-containing heterocycle of unusual structure as illustrated below. We have not been able to decide whether it is structure A or B on the basis of chemical analysis. We presume that an equilibrium may exist in solution perhaps involving both of these systems or another isomer.



If anyone has close model systems, we would be glad to discuss the chemistry. To date, very few examples of this type of pentavalent, phosphorus-containing molecules have been published and fewer have had structures confirmed. In contrast, there are a fair number of pentavalent, phosphorus-containing compounds which have five bonds from phosphorus to oxygen. However, systems with phosphorus bonded to two or more heteroatoms in related compounds are very rare. We welcome suggestions.

Sincerely yours,
Darrell
K. Darrell Berlin
Regents Professor

Fremont Magnetic Resonance
3315 Seldon Court
Fremont CA 94539
(415)623-0722
(415)623-0851 FAX

We've moved!!!

Please note the new address and telephone numbers for FMR.

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Felix / PC

NMR Data Processing Software

FMR and Hare Research announce an inexpensive alternative for NMR data processing using IBM compatible PCs. This alternative is available as either a software package for 1D or Multi-D NMR data processing utilizing Hare Research's Felix/PC(tm) software or as turnkey data stations configured and ready to process NMR data . This software features a mouse oriented, menu based, user interface. The menu software greatly increases the "user friendliness" of the processing software for the new and occasional users. The command line interface remains in place at all times for the experienced user.

Felix/PC software features:

- "Real-time" on screen phasing with mouse .
- Automatic basline selection and corrections.
- Automatic peak selection and annotation.
- Plotting to HPGL or Postscript devices.
- Efficient Matrix oriented multi-D processing.
- Contour and "Image" 2D displays.

The NMR user can use her current PC and printers/plotters with the software modules below and be processing and plotting NMR data for $1000.00. Alternatively, she can purchase one of the available turnkey system with the desired software and accessories and begin processing and plotting data immediately for as little as $6000.00.

System Requirements:

DOS 2.0 or greater.
640 K Memory
EGA or VGA display adapator.
Mouse

Highly Recommended:

12 MHz or greater 80286 or 80386 PC.
80287 or 80387 Coprocessor

PLOT

NMR Plotting Software

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Probe Qs

In the last instrumentation note, sensitivity was discussed from the aspects of coil length and filling factor. Another parameter affecting sensitivity is the probe Q. Q is defined as the frequency of the resonant circuit divided by the half power bandwidth. Many of today's probes have unloaded Qs greater than 300. If all other parameters are the same, the higher the probe Q the greater the sensitivity.

What determines the probe Q? The AC resistance of the resonant circuit. The lower the AC resistance the higher the probe Q. A very simple answer to a very complex question. The probe has two components which can limit the AC resistance: the coil and the capacitor of the resonant circuit.

The Coil

The resistance of the coil (wire) goes up as the frequency goes up by a phenomena call "Skin Effect". The "Skin Effect" is caused by the magnetic field created by the current in the wire. The magnetic field forces the electrons to move in a curved path until they hit the surface of the wire. Therefore the current is forced to flow in a smaller part of the wire. Said in another way the wire appears smaller to a high frequency current than a low frequency current. The smaller area for current flow raises the AC resistance to current flow and thereby lowers the Q of the resonant circuit. Some probe designers combat this by using foil, as opposed to wire, to increase the surface area. Other probe designs use separate coils in parallel. Since resistances in parallel are less than either individual resistance, the AC resistance of parallel coils is less than in either individual coil. Another advantage of parallel coils is that the total *inductance* of parallel coils is less than in either individual coil. Since the higher frequency probes tend to have very small capacitance values for resonance, the lower inductance makes it easier to reach higher frequencies with a given capacitance. Often the bottom value of this capacitance is limited by the stray capacitance. The paralleled inductors lower the total inductance and therefore higher capacitor values can be used.

The AC resistance of the coil is also determined by its geometry. This can be a very complex issue, but some of its basic characteristics can be simplified. If the coil has any sharp turns the electrons from the probe's current generate a magnetic field causing them to crowd to the inside edge. This crowding reduces the effective amount of conductor available to the current thereby raising its resistance and lowering Q. This effect is very similar to the "Skin Depth" effect discussed before. The electron crowding in the corners also raises the inductances per unit distance of electron flow. Therefore, to get the lowest resistance and inductance per unit of length, the degree of sharpness at all corners of the probe coil should be limited as much as possible.

The Capacitor

The AC resistance of the capacitors is usually related to the materials from which they are constructed. Air capacitors formed by two pieces of conductor in parallel have a very low resistance and therefore can have a very high Q. To obtain a given capacitance value this type of capacitor needs to be very big or have its parallel surfaces very close. Mechanical tolerances and electrical arcing under the high voltages of an RF pulse limit the use of this type of capacitor. To get the surfaces closer together and increase the capacitance of a given surface area, capacitors have a dielectric between the conductive surfaces. This dielectric introduces higher resistance in the capacitor, which lowers the Q of the resonant circuit. The material the tunable capacitors are constructed from is usually the most lossy and therefore lowers the Q the most. For this reason a compromise between the desired tuning range and the probe Q is a problem many probe designers face.

The Sample

One component which limits probe Q is the sample. The sample increases losses in the resonant circuit by inducing eddy currents in the solvent. The more conductive the sample the more the losses and the lower the probe Q. This is often seen by the longer 90° pulses with water samples than with organic solvent samples.

The losses in the sample are induced by the electric field values inside the probe. The more distributed capacitance a probe has in its resonant circuit, the lower the electric field values. Therefore a probe with distributed capacitance is less affected by the sample. Unfortunately the capacitor usually has a higher resistance than the coil. Therefore the use of distributed capacitance lowers the probes unloaded Q but can raise the loaded Q. This statement reduces to the probe designers need to compromise probe design between organic and water samples.

As was indicated before, probe design entails many details and compromises between often mutually exclusive requirements.

Kodak

January 3, 1990 (received 1/6/90)

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

Dear Dr. Shapiro:

Magneto-Optical Drives for NMR

With the increase in the number of two-dimensional and three-dimensional experiments, the amount of storage space for these experiments can easily eat up the hard disk capability on most instruments. Early this year we were considering various technologies which would provide us with fast retrieval of data. Our experience with streaming magnetic tape (141 Mbyte cartridge) and many years of 1/2-inch 9-track tape had convinced us that this was not the medium appropriate for our needs, although it is still probably the cheapest archival storage available.

The arrival of the NEXT computer brought the new magneto-optical into the limelight and so we investigated a drive distributed by Pinnacle, Inc., Ca which markets a 600-Mbyte drive based on a Sony engine. One obtains approximately 273 Mbytes of storage per side of the removable 5 1/4-inch cartridge which requires removal of the disk to access each side. Each of the disks cost $250, putting the cost per Mbyte at $.50. The drive itself cost $6,000. Our major concern with the system was that write times would be too slow for it to be used as a practical device and likewise read times would also be inefficient. However, our tests show that the write and read time is approximately 1 Mbyte/15 s. This allows transfer of a 32-Kword spectrum in about 2 s. These times are a little misleading (i.e., slower than expected) since all the read/writes are done across an ethernet network. In fact we have installed the disk on a SUN3/60 workstation and are able to remotely mount the drive from the Varian instruments and vice versa for ease of operation. Having mounted the drive from the instrument we are able to use it as though it is attached to the instrument despite the fact it is physically connected to another SUN some distance away.

Even in this mode the times are sufficiently fast to allow it to be used as a "real" interactive drive. The unit has now been fully operational for about 4 months and, despite some early glitches, the unit has performed without fail as we pour gigabytes of stunning NMR data into the little box.

Sincerely,

Richard N. Moore

Richard N. Moore
Analytical Technology Division

RNM:nar

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January 3, 1990
(received 1/5/90)

HMQC-TOCSY to Disentangle Overlapped Direct and Relayed Vicinal Proton Responses

Dear Barry,

Advances in the area of new two-dimensional NMR pulse sequences continue to appear at what seems to be an ever increasing rate. There are times, however, when rather than seeking perhaps an even more exotic solution to a problem we would do better to recall some fundamental NMR!

Heteronucleus detected relayed coherence transfer[1] (variously referred to as HC-RELAY, RCT2D, etc.) provides an excellent means of establishing the location of a given proton's vicinal neighbor(s). A problem is, however, encountered when the directly bound proton and its vicinal neighbor have very closely similar chemical shifts.[2] Before the advent of proton detected analogs of the heteronuclear relay experiment, Bolton designed a pulse sequence to circumvent this type of problem by resorting to proton double quantum heteronuclear relay.[3] Given the proton detected heteronuclear relay experiment pioneered by Lerner and Bax,[4] which we have become fond of referring to by the acronym HMQC-TOCSY after the work of Davis,[5] there is a simple solution to the problem of direct and vicinal proton resonance overlap if we recall a little of our fundamental NMR. The HMQC-TOCSY pulse sequence, shown in Figure 1, generally utilizes broadband ^{13}C-decoupling during the acquisition period as in the conventional HMQC experiment. While this eliminates the doublet character of the detected proton, a function of the one bond proton-carbon heteronuclear coupling, it is disadvantageous in that it mandates a perpetuation of the overlap of the direct and relayed responses. If we simply forego ^{13}C-decoupling during acquisition, the direct response will be a symmetrically disposed doublet centered about the chemical shift of the direct proton in question, the components of the doublet separated by the $^1J_{CH}$ scalar coupling (typically 125-200 Hz). The practical consequence of the displacement of the direct response by $\pm 1/2(^1J_{CH})$ relative to the desired vicinal relay response, which is not modulated by the one bond coupling, is to remove the overlap of the relayed vicinal proton response.

A practical illustration of this approach is provided by Figures 2-4. Figure 2 shows the ^{13}C coupled HMQC-TOCSY spectrum for a portion of a sugar (4 mg/ml) in which two carbons have protons with nearly identical chemical shifts. The conventional HMQC spectrum with ^{13}C decoupling applied in the usual fashion is shown in Figure 3. Comparision of Figure 2 with Figure 3 clearly show the relayed vicinal ^1H J connectivity between the overlapped protons which are bound to the two carbons. Note how the "center" responses at each carbon in Figure 2 are "switched" from the chemical shift responses in Figure 3. The response relayed to the vicinal proton is observed at its chemical shift without interference from the direct proton response.

In closing, a final word of caution is in order. This technique, while effective and simple, can be prone to misinterpretation if the rf of the spectrometer used for the work is prone to incomplete cancellation of the "residual" 99% ^1H-^{12}C signal. If there is any doubt, a simple check can be done using the HMQC experiment without ^{13}C decoupling. Figure 4 shows this result for our example. If the signal from the ^1H-^{12}C signal is suppressed in this spectrum then the method will work as described. This same experimental approach works well with HMQC-NOESY if the intrinsically lower signal to noise of HMQC-NOESY run at natural abundance is taken into consideration.

1. P.H. Bolton, *J. Magn. Reson.*, **48**, 336 (1982); P.H. Bolton and G. Bodenhausen, *Chem. Phys. Lett.*, **89**, 139 (1982); A. Bax, *J. Magn. Reson.*, **53**, 149 (1983).
2. M.J. Musmar, A.S. Zektzer, G.E. Martin, R.T. Gampe, Jr., M.L. Lee, M.L. Tedjamulia, R.N. Castle and R.E. Hurd, *J. Heterocyclic Chem.*, **25**, 1039 (1987).
3. P.H. Bolton, *J. Magn. Reson.*, **54**, 333 (1983).
4. L. Lerner and A. Bax, *J. Magn. Reson.*, **69**, 375 (1986).
5. D.G. Davis, *J. Magn. Reson.*, **84**, 417 (1989).

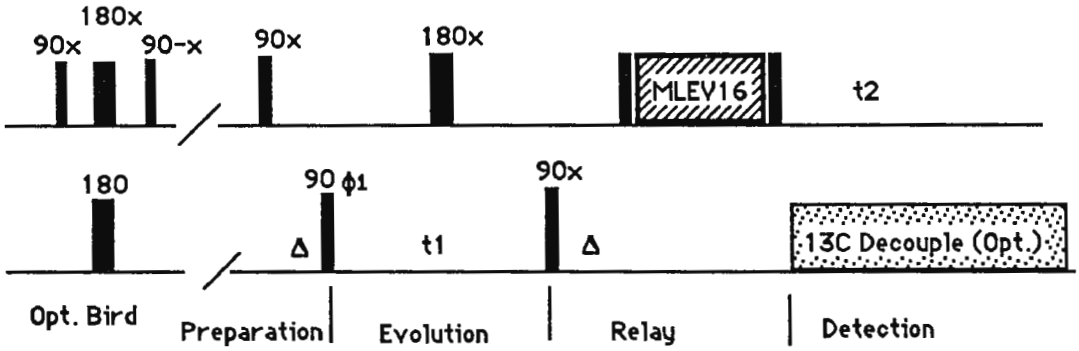

Figure 1. HMQC-TOCSY Pulsesequence

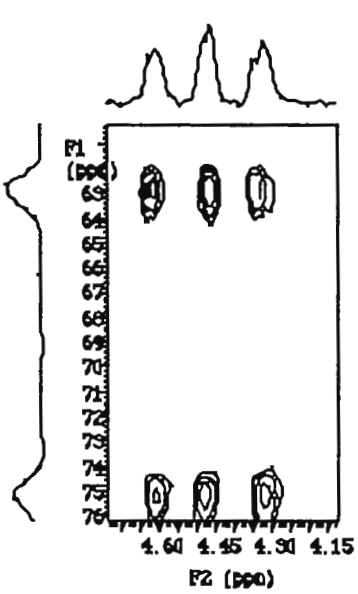

Figure 2. HMQC-TOCSY
13C coupled NT=16

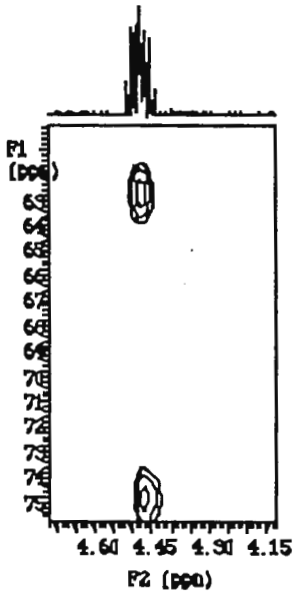

Figure 3. HMQC
13C decoupled NT=8

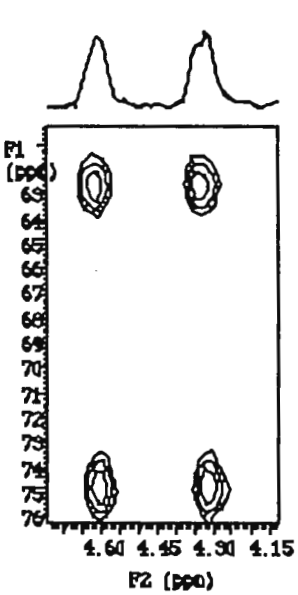

Figure 4. HMQC
13C coupled NT=16

Sincerely yours,
Ronald C. Crouch

Gary E. Martin

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Swiss Federal Institute of Technology Zurich

Institut für Molekularbiologie und Biophysik
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ETH-Hönggerberg
CH-8093 Zürich

1085

Prof. B.L. Shapiro
TAMU NMR Newsletter
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Palo Alto, CA 94303
USA

Zürich, December 19, 1989/hu
(received 12/28/89)

Using the SEU for heteronuclear decoupling on Bruker AM spectrometers.

Dear Professor Shapiro,

The DISNMR software available on Bruker AM instruments has a limited capacity for the number of pulses, pulse phases and delays that can be handled by a microprogram. Although these limits are hardly ever met with homonuclear NMR experiments, they may impose restrictions with regard to ^{1}H-detected heteronuclear experiments, where the ^{13}C or ^{15}N decoupling sequence is explicitly programmed in the microprogram. This problem can be circumvented by using the selective excitation unit (SEU) for storage of the decoupling sequence. The low power decoupling sequence is then provided by the output of the SEU rather than by the transmitter low power (TLO) channel. If necessary it may be further amplified with the use of an external broadband amplifier. For example, to program the WALTZ sequence for the SEU we generate a file containing a continuous sequence of 90-degree pulses of equal amplitude, with phases that follow desired WALTZ sequence. The number of 90-degree pulses is chosen in such a way that it covers the whole acquisition period. This file is generated by a Pascal program which requires the acquisition time and the 90-degree pulse width as the only input. (A second file which describes a single SEU output of rectangular shape is used to determine the width of the 90-degree pulse). With this setup the decoupling sequence in the microprogram can be written as a single pulse of length equal to the time of the acquisition period. Furthermore, this scheme enables the straightforward implementation of WALTZ with cycled sidebands (Shaka et al., J. Magn. Reson. **67**, 396 (1986)).

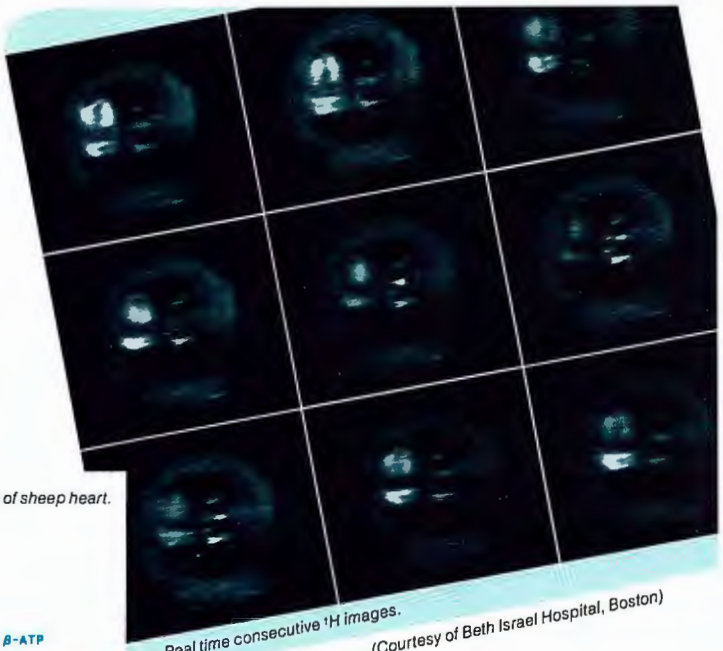
Yours sincerely,

G. Otting

Dr. G. Otting

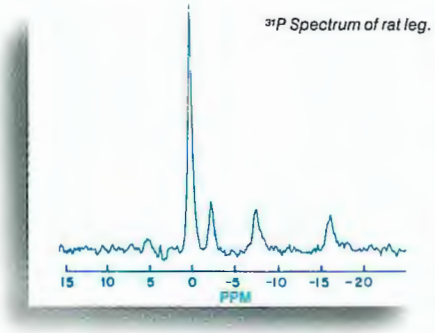
p.s.: Please credit this contribution to the subscription of Prof. K. Wüthrich.

Real Answers.

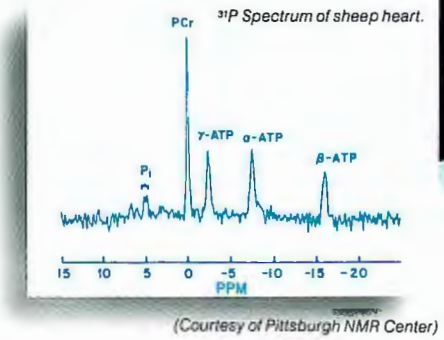


Real time consecutive ^{1}H images.

(Courtesy of Beth Israel Hospital, Boston)



^{31}P Spectrum of rat leg.



^{31}P Spectrum of sheep heart.

(Courtesy of Pittsburgh NMR Center)

S/N performance.

Unsurpassed performance in sensitivity. ^{31}P NMR spectrum obtained in a single acquisition at 81 MHz from muscle in rat leg. (1 msec adiabatic pulse) using a circular surface coil of diameter 28mm. The S/N on PCr is 60:1.

Large bore.

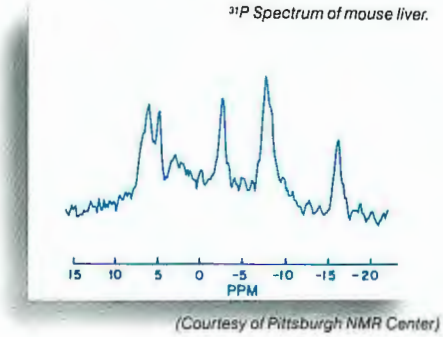
Wide access for large animal studies. ^{31}P NMR spectrum obtained at 81 MHz from heart muscle in a live sheep (64 x 90° pulses at 2 sec. intervals) using a circular surface coil of diameter 35mm.

Real time.

A series of consecutive real time images can be obtained in a single heartbeat using a fast gradient echo technique with phase reversal. Each image can be obtained in only *58 msecs*. The in-vivo images shown above illustrate the diastole-systole-diastole cardiac cycle in a mouse.

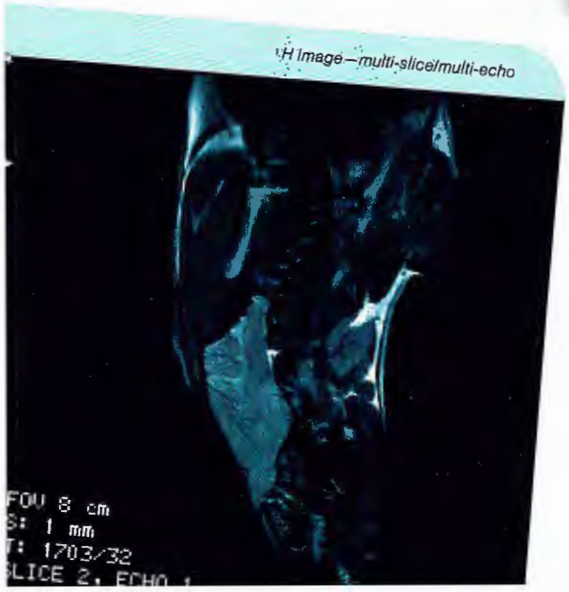
Multi-slice/Multi-echo.

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



^{31}P Spectrum of mouse liver.

(Courtesy of Pittsburgh NMR Center)



^{1}H Image—multi-slice/multi-echo

In-vivo 7.0 tesla.

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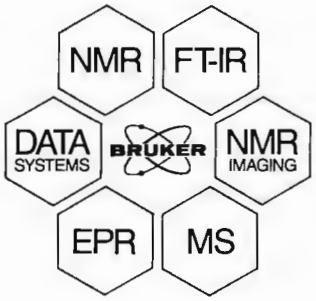

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Prof. G. C. K. Roberts
Dr. L. Y. Lian

19th Dec 1989 (received 12/26/89)
Dr.Bernard L.Shapiro
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto CA 94303
USA

Title: ^{15}N NMR of Trp Repressor

Dear Dr.Shapiro,
We have now successfully implemented many of the well-known and popular experiments which use a combination of stable-isotope labelling and proton-detected NMR spectroscopy. One of the more recent techniques is the one proposed by Norwood, Boyd and Campbell [FEBS Letts, 1989, in press] based on the ^{15}N single quantum coherence (SQC) experiment, this having the advantage of providing improved resolution in F1 by removing homonuclear scalar coupling and of prolonging the transverse relaxation time (in contrast with the multiple-quantum coherence experiment). Both these advantages are of significance in the NMR study of large proteins (M_r>20 kD)

The protein of interest to us is the DNA-binding protein, E.coli trp repressor, a dimer of total M_r approximately 25 kD, with a predominance of α-helices in its 3D structure. A uniformly ^{15}N-labelled sample of trp repressor was obtained from E.coli grown in a minimal medium using $^{15}NH_4Cl$ as the sole nitrogen source. The NMR sample comprised of 2mM protein dimer in 90% H_2O/D_2O containing 0.5M NaCl,50mM phosphate buffer, pH 5.75. To illustrate our results the spectra of the trp repressor-tryptophan (1:3) complex are shown.

The NMR spectra were recorded on a Bruker AM500 spectrometer at 310°K with the following parameters: 90°(^{1}H)=15us; 90°(^{15}N)=18us; 90°(for ^{15}N ecoupling))=80us. The GARP decoupling sequence was used; this was implemented using a BFX-5 10W linear amplifier. Water suppression was afforded using low power solvent presaturation.

Fig. (I) shows the two-dimensional ^{15}N SQC spectrum (total acquisition time=4hrs) and Fig.(II) the ^{15}N SQC-NOESY spectrum (total acquisition time = 7hrs).

Because trp repressor is a helical protein the NH resonances in the proton spectrum shows comparatively small chemical shift dispersion giving both severe spectral overlap and degeneracy. Many of the overlapping proton signals can now be resolved using the ^{15}N dimension. Fig. II shows extra cross-peaks compared with Fig. I; these cross-peaks arise from NH-NH NOE correlations.

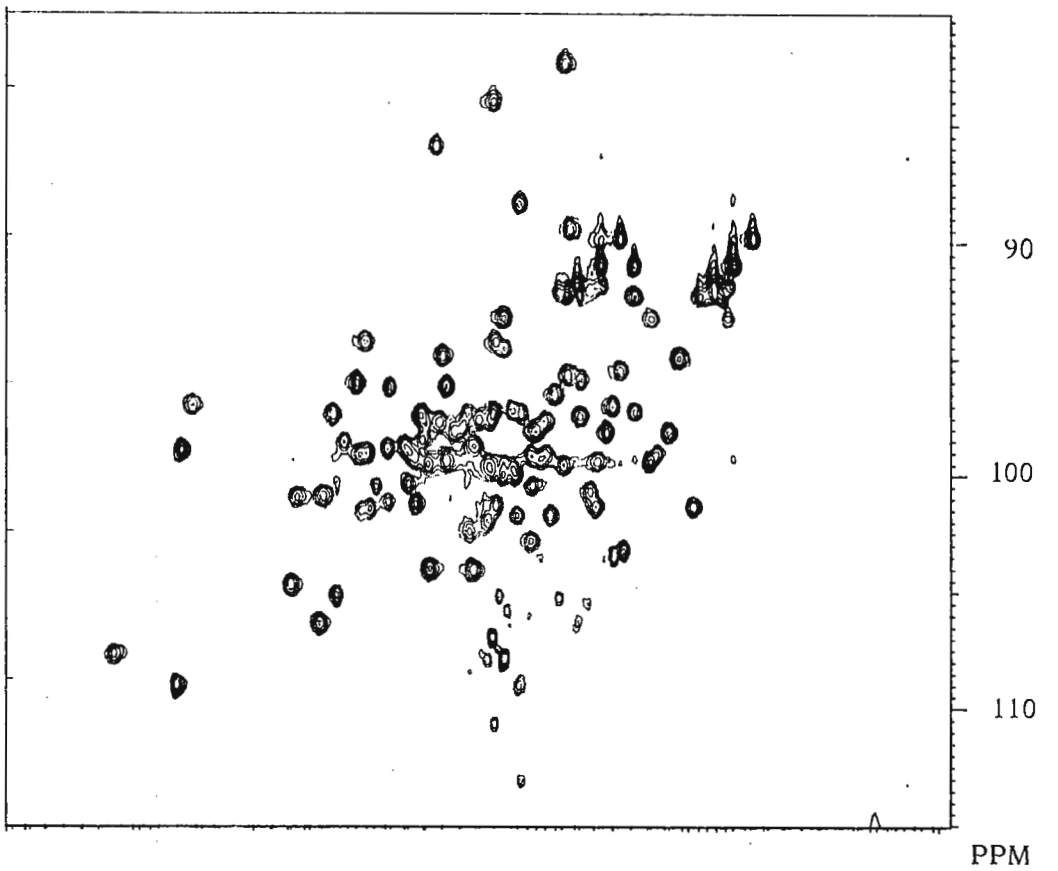
Please credit this contribution to Gordon Roberts' subscription.

Yours sincerely,

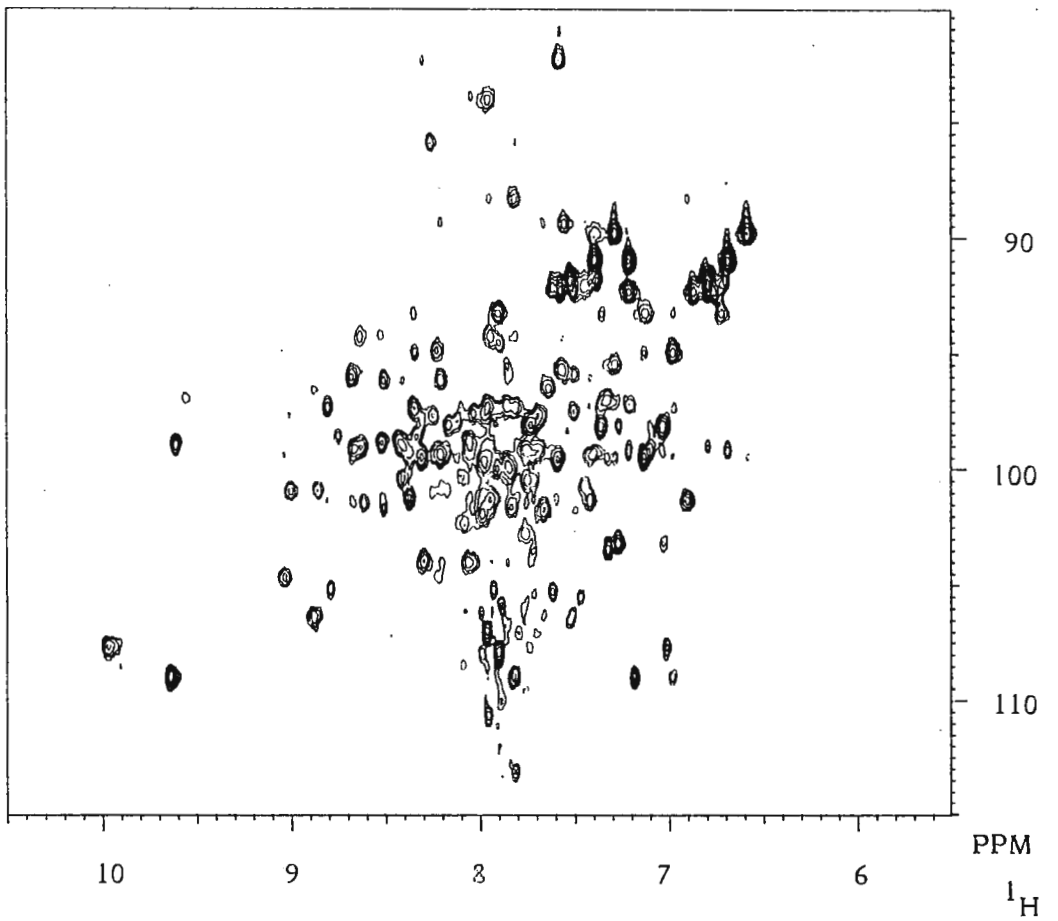
Lu-Yun Lian V.Ramesh

377-26

I



II



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Institut für organische Chemie

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Telefon 031 65 43 11

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

13. december 1989
(received 12/22/89)

DANTE IN HETERONUCLEAR EXPERIMENTS

Dear Dr. Shapiro

despite the fact that spectrometer manufacturers meanwhile offer "non-expensive" equipment to shape weak rf-pulses, selective excitation by DANTE pulsetrains seems to become popular again. Several modifications have recently been proposed to improve their selectivity and the uniformity of their excitation windows in the frequency domain. Such DANTE pulses should certainly be of considerable value not only in homonuclear, but in heteronuclear experiments as well. **To calibrate pulsewidths and to determine excitation profiles of DANTE pulses applied on the decoupler channel**, while observing a heteronucleus, a modified SPT experiment was used. The corresponding microprograms for a BRUKER AM 400 spectrometer equipped with a process controller are listed below. As an example these two pulsesequences are applied to a $CHCl_3$ sample (dopped with $Cr(acac)_3$). The corresponding results are shown in fig. 1. The low frequency proton satellite signal of the $CHCl_3$ - ^{13}C isotopomer was selectively irradiated.

DANTE PULSEWIDTH CALIBRATION

```
1 ZE
2 D1 S1 DO
  SP
3 (P1 PH1 D2):D
4 LO TO 3 TIMES UPR
  P3 PH2
  D3 S2 DO
  (P2 PH5):D P4 PH3
  D3 S3 DO
  GO=2 PH4 CPD
  WR DATA1
  IF DATA1
5 IUO
  LO TO 5 TIMES C
  IN=1
  EXIT

PH1=0
PH2=0 0 2 2 1 1 3 3
PH3=0 0 0 0 1 1 1 1
PH4=R0 R0 R2 R2 R1 R1 R3 R3
PH5=0
```

DANTE EXCITATION PROFILE

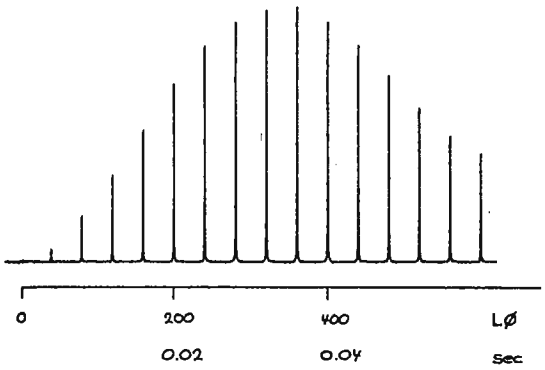
```
1 ZE
2 D1 O2
2 D1 S1 DO
  SP
3 (P1 PH1 D2):D
4 LO TO 3 TIMES UPR
  P3 PH2
  D3 S2 DO
  (P2 PH5):D P4 PH3
  D3 S2 DO
  GO=2 PH4 CPD
  WR DATA2
  IF DATA2
  IN=1
  EXIT

PH1=0
PH2=0 0 2 2 1 1 3 3
PH3=0 0 0 0 1 1 1 1
PH4=R0 R0 R2 R2 R1 R1 R3 R3
PH5=0
```

parameters:

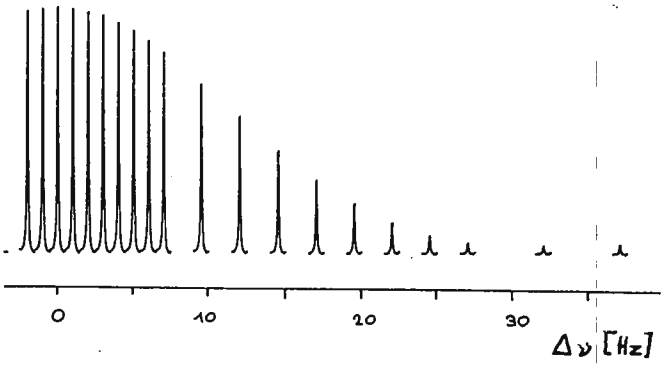
D1	3s		3s
S1	18H		18H
SP	1		1
P1	2us		2us
D2	.0001s		.0001s
L0	var.		330
P3	7.5us		7.5us
D3	.001207s		.001207s
S2	0H		0H
P2	22.4us		22.4us
P4	15us		15us
S3	17H		17H
RD, PW	0		0
DS	0		0
NS	8		8
VCLIST.001		O2 FQLIST.001	var. values
	1=40		
NE	16		22

Fig. 1a

Fig. 1b



pulsewidth calibration

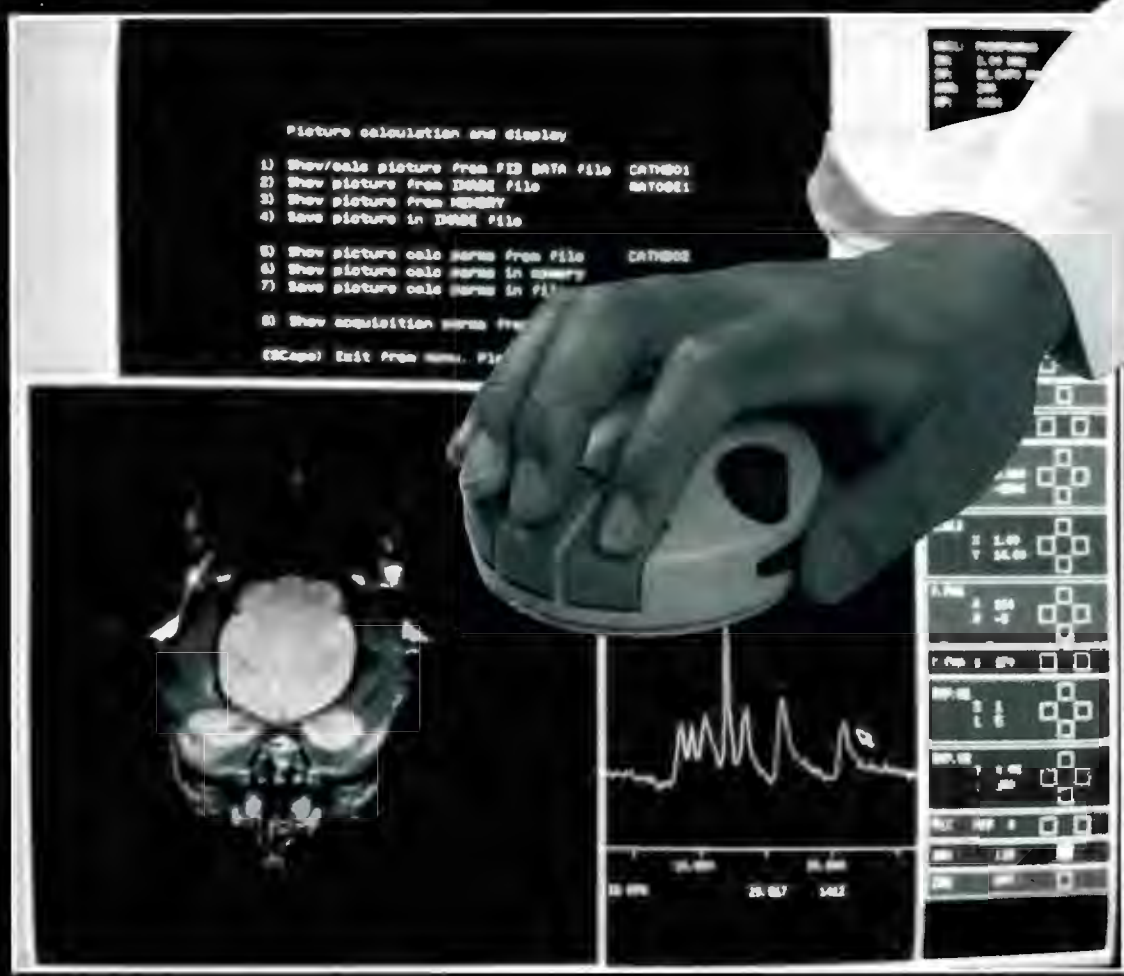


excitation profile

yours sincerely

Dr. P. Bigler

Where Research and Technology Meet...



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Phase, Variable Steps	0.1 Degree Resolution
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Power Control	0 - 100 %
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Nitrogen Evaporation (ml/hr)	400	400	400	400	450	500	400
Half Length (mm)	350	275	570	396	460	735	575
5 Gauss Line — Radial From Center (m)	3.4	3.2	4.7	3.7	5.3	6.4	5.1
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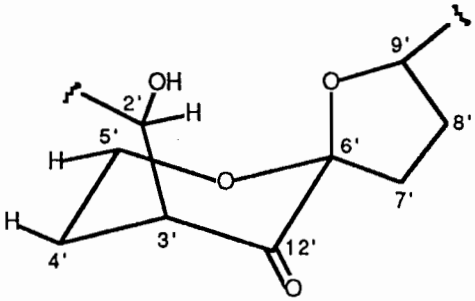
Dec. 14, 1989
(received 12/22/89)

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

Application of the FLOCK Experiment in Structural Elucidation

Dear Barry,

A recently-investigated cyclization reaction was believed to yield one of the following products:



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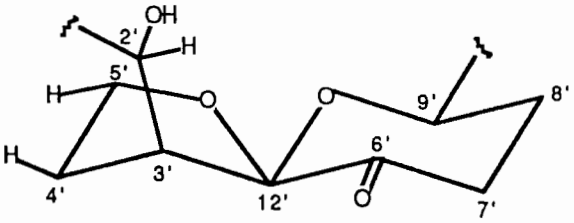

II

These two structures could, in principle, be differentiated by the FLOCK, indirect C-H correlation experiment [W. F. Reynolds *et al.*, *Magn. Reson. Chem.*, *27*, 162 (1989)], but a limit of 10-15 mg of material was believed to be pushing this experiment to its limit. A weekend experiment was conducted in which ^{1}H resolution was partially sacrificed (NI=128) for ^{13}C sensitivity (2400 transients/increment). A value of $^{n}J(CH)=7.5$ Hz was used for calculating the dephasing (Δ_1) and refocussing (Δ_2) delays and $^{1}J(CH)=140$ Hz for τ (BIRD pulses). The repetition rate was 600 ms.

After *ca.* 64 hrs. the following correlations were observed in proton slices taken at the carbonyl (200.2 ppm) and hemiketal (108.5 ppm) resonances. The former showed correlations with H-2' and H-4'eq, which, representing 3-bond C-H couplings, are possible only in I. Structure II would require that these correlations represent 4-bond, C-H couplings which are not ordinarily observed.

In addition, C-6' (hemiketal) exhibited a strong correlation with H-5'eq, indicative of a *trans*-vicinal coupling pathway in I. In structure II such a correlation would be expected to be much less intense since the $C_{12'}$-$H_{5'eq}$ dihedral angle is only *ca.* 120°.

Sincerely,

Bruce B. Jarvis

Eugene P. Mazzola

STEVENS

Department of Chemistry and Chemical Engineering
201-420-5546

Stevens Institute of Technology ♦ Castle Point on the Hudson ♦ Hoboken, New Jersey 07030

PCB's AND NMR ??

December 15, 1989
(received 12/19/89)

Dear Dr. Shapiro:

Seasons Greetings!

Recently I had the opportunity to install an Array Processor on the Aspect-3000. In the process of installation all the PCB's (printed circuit boards) had to be removed. Removal of the ADC board brought with it an interesting situation. In the vicinity of the Ultra Fast Sample and Hold module was a viscous fluid. It was cleaned of by hand and rag. Calling in Bruker revealed that it comprised of polychlorinated biphenyls (PCB's). In addition I was informed that the life of the module is 4-5 years after which leakage was normal.

Warning labels would be nice!!

On another note the EXE subroutine to the 1988 DISNMR release is a useful addition. A "LOGIN-LOGUOT" procedure using an executable file is presently being implemented including a "foolproof" sequence for persons with limnited DISNMR command knowledge.

Yours Sincerely,

Ashok Krishnaswami

Ashok Krishnaswami

INSTITUTE of CHEMICAL PHYSICS
Academy of Science USSR
ul. Kosygina - 4
Moscow, USSR

and

SPECTROSPIN AG
Industriestrasse 26
CH-8117 Fällanden
Switzerland

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

(received 12/26/89)

December 20, 1989

Correction of effects due to temperature instability in 2D NMR Spectroscopy

Dear Dr. Shapiro,

The strong temperature dependence of chemical shifts of some deuterated solvents often leads to artefacts in 2D spectra such as increased t_1-noise or badly shaped peaks. We have developed a computer program to compensate for these artefacts and the main points of the method are summarized below:

- The input for the program is the 2D matrix resulting from the Fourier transformation in the t_2 time domain. Such a spectrum shows distorted vertical strips for the signal of the substance and a straight line for solvent;
- Each row of this matrix is now shifted to get straight strips for one or several signals of interest, which are defined as reference points (as a consequence the solvent strip becomes wavy);
- The second Fourier transformation produces a 2D spectrum with correct cross peaks for the substance. The amplitude of the solvent peak proves to be significantly reduced.

This method was applied to a phase sensitive double quantum filtered COSY spectrum of a sucrose solution in D2O (5%). measured on Bruker AMX-600 spectrometer. During the course of the experiment the temperature was manualy varied in the limits of $\pm 1^{o}C$. The C-language program runs on the ASPECT X-32 computer and the processing time for moderate size 2D spectra is smaller than 5 minutes.

Sincerely,

Dmitriy Yu. Artemov
(Institute of Chemical Physics)

Heinz Rüegger
(Spectrospin AG)

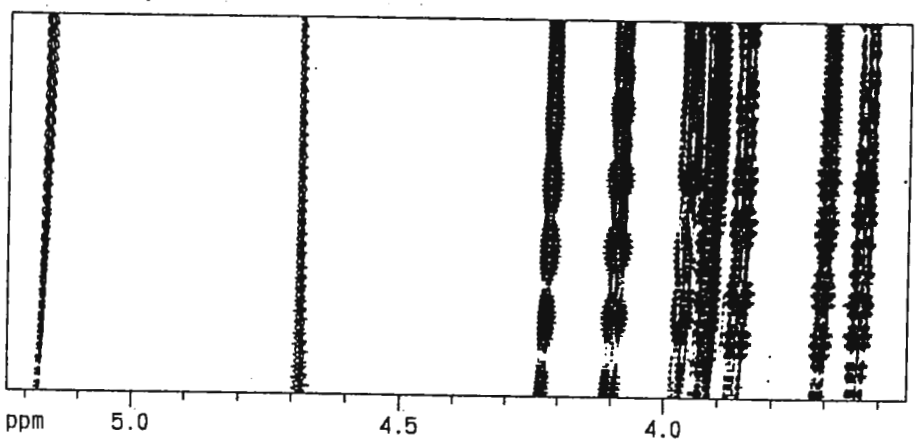

Fig.1: The effect of temperature variations produces after the Fourier transformation in $\omega 2$ wavy vertical lines at the positions of the sugar resonances and a straight line for the HDO resonance.

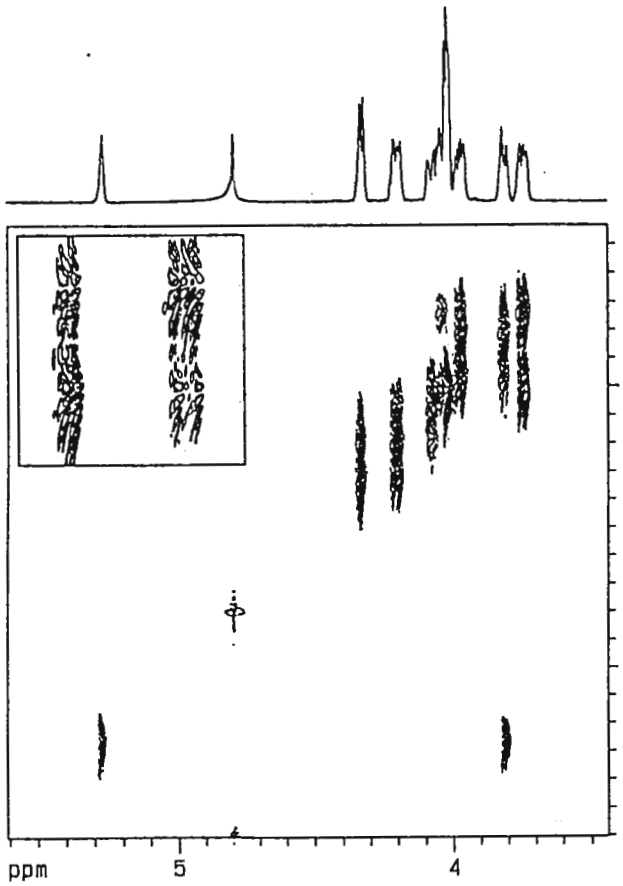

Fig. 2: Without correction, the second Fourier transformation in $\omega 1$ produces strange line shapes for the 2D peaks

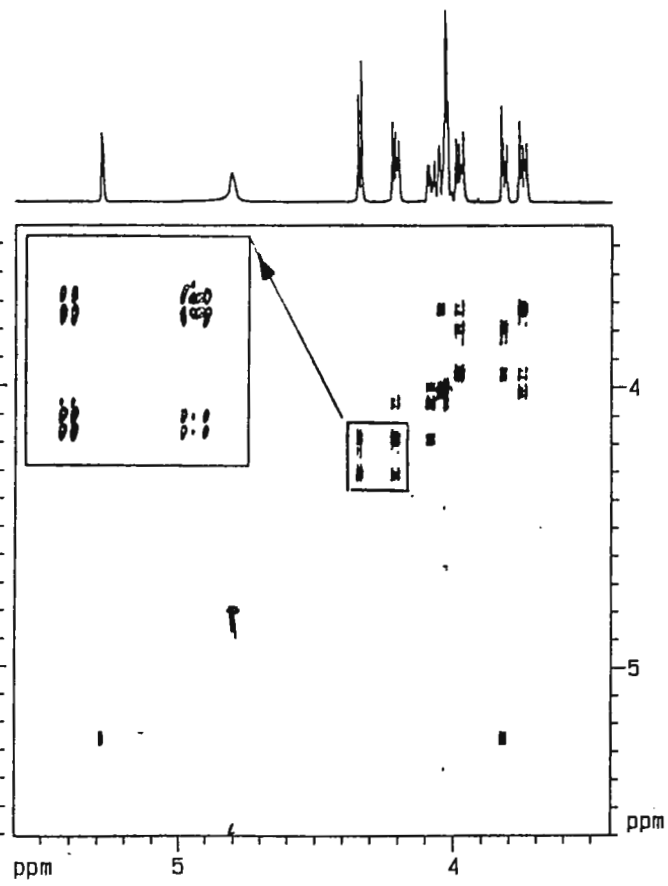

Fig. 3: With the described correction employed, correctly shaped 2D peaks result.

P.S. Please credit this contribution to Dmitriy Yu. Artemov of the Institute of Chemical Physics to initiate his subscription to the TAMU Newsletter.

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To: Professor B. L. Shapiro
Editor, TAMU Newsletter
966 Elsinore Court
Palo Alto CA 94303

Subject: 3DNMR Processing using FELIX

Dear Barry, 29. December 1989

"Everybody knows" that resolution in 2DNMR spectra of biopolymers can be a problem and that performing 3DNMR experiments is one way to introduce more resolution or more information. We've done a few 3D experiments lately and we would like to share our results and conclusions.

First, one of us (KB) has created what may be the world's largest NMR matrix: $(512)^3$ points! This may be laudable or laughable, but it can be done with existing software and hardware without difficulty. The experiment is a NOESY-NOESY (first reported by Boelens and colleagues at last year's ENC) and is useful for determining spin diffusion contributions to NOEs. The sample used was about 10mM $d(GCCTGATCAGGC)_2$ and data (250x260 experiments, 512 points, 8 scans each) were collected over seven days on a home built spectrometer. After collection, data were transferred via Ethernet to a Silicon Graphics 4D/20 for processing with FELIX software (Hare Research). Transformation required about 12 hours[1], creating a single 536 Mb matrix. Unlike the approach used by Bax *et al.*, FELIX uses a single matrix file and does not require matrix transposition or the "butterfly" data shuffling step. The matrix was compressed to 25 Mb by discarding data points lower than a user-specified S/N level.

Figure 1 shows a slice of the matrix where the total (observed) h6–h3′ NOE (horizontal plane) is only slightly larger than the indirect h6–h2′–h3′ pathway (vertical plane). The intense h6–h2′ NOE that is the spin diffusion source can also be seen. We are using this information to calculate the 2nd order contributions to NOEs in an attempt to fit back-calculated NOE buildup rates. (Neglecting indirect contributions to NOEs is a problem discussed some time ago by Ed Olejniczak and coworkers). Figure 2 is a stereo-pair and includes the region of the 3D matrix containing the aromatic and H1′ protons. The two NOESY planes are indicated by the arrows; indirect NOE contributions appear off the diagonals and a few are circled in the figure.

Figure 3 shows an expanded region of a ^{15}N correlated NOESY of the protein ubiquitin, acquired on a GE spectrometer while one of us (PW) was visiting Gerhard Wagner's lab. This experiment is similar to ones published previously and independently by Steve Fesik and Ad Bax, and is a ^{15}N-edited NOESY combined with an INEPT heterocorrelation experiment. A 512 by 256 by 128 real matrix was created from thirty separate 2D experiments (256 by 1024 points each). The narrowest dimension shown in the stereo pair is the ^{15}N dimension; the homonuclear NOESY would not have the additional resolution that this extra dimension yields. One reason we show it is how we did some rephasing after transformation. Instead of creating a 8X larger matrix for holding the FT complex pairs, we save only the reals. To rephase, the vectors in the non-acquisition dimension were subjected to a Hilbert transform to recreate the complex information, followed by rephasing (the complex part was again discarded).

Since (i) 3DNMR is pretty easy to acquire and process; (ii) much information is available that could not be easily obtained otherwise; (iii) fast computers and big hard disks are getting cheap; (iv) the data processing tools are available; we think a lot more 3D experiments will be published!

Kevin Banks Paul Weber Bob Morrison Dennis Hare

[1] It would require "only" 7.2 hours on a 4D/25.

☐ *Data Processing* ☐ *Spectral Assignment* ☐ *Structure Determination*

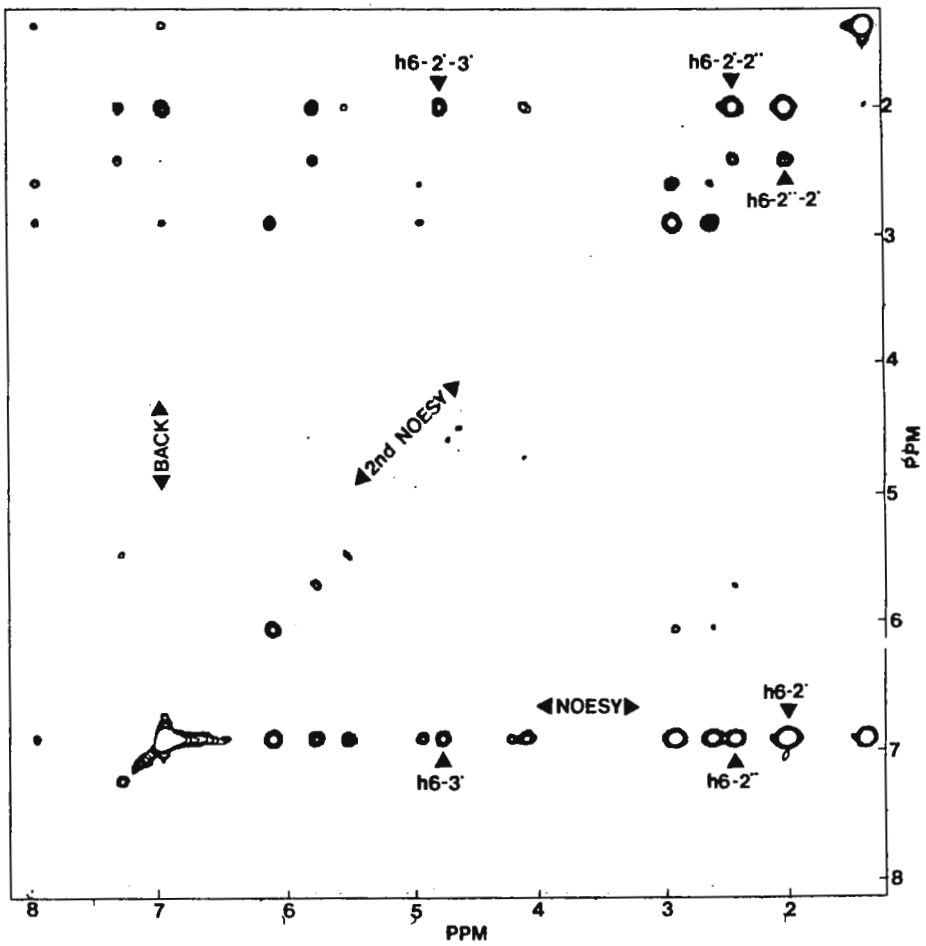


Figure 2:

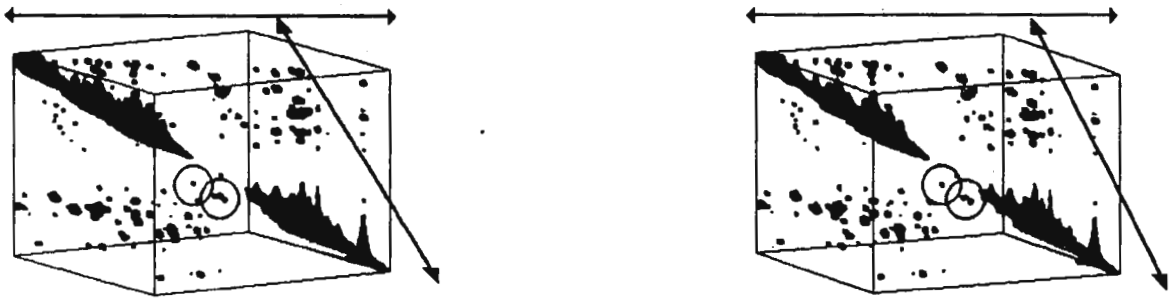
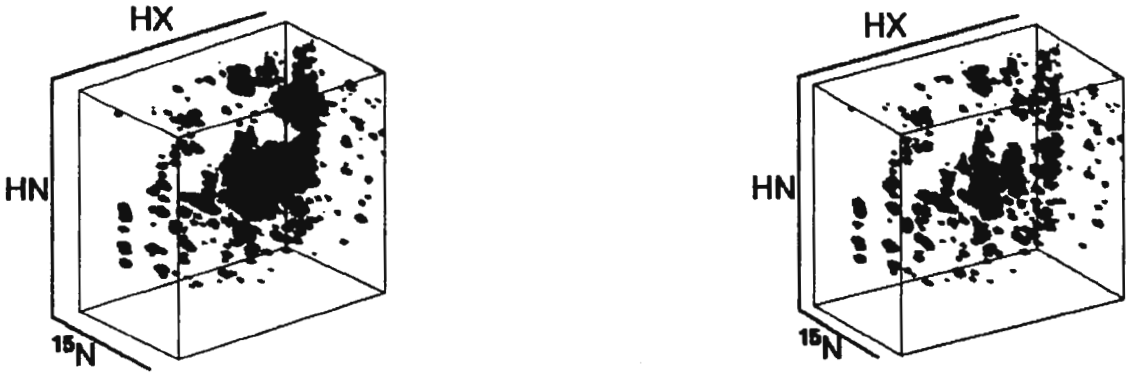


Figure 3:



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(received 12/26/89)

December 19, 1989

Dr. Bernard L. Shapiro
1989 TAMU NMR Newsletter
966 Elsinore Court
Palo Alto, California 94303

NOISE BRIDGE

The conventional way to tune and match an NMR probe is to use either a single or swept source of frequency, a bridge to compare the probe impedance against a 50 ohm reference resistor, and an oscilloscope to indicate a null in the RF or detected waveform when the probe is matched at the desired frequency. The Sweeper will always be useful for the bench development of probes.

The method used here requires no Sweeper, or much in the way of set-up procedures. A broadband noise source produces a very large RF spectrum. A bridge is used as before. The NMR Spectrometer detects a null in the noise at the specific frequency of interest. The output is audio noise, which may be heard on a loudspeaker and detected and displayed on a bargraph meter mounted on the magnet. The measuring setup is quick, requiring only the opening of the line between the preamplifier and the T/R (Transmit/Receive) switch and Probe. It is well to include the T/R switch in the measurement so that its combination with the Probe will present (the optimal) 50 ohms to the preamplifier.

The basic idea, while not new, may be new to NMR work. We first heard of it from B. Chance of the University of Pennsylvania. The 1981 ARRL Handbook (1) describes a noise bridge to be used for tuning and matching antennas. We have also seen an advertisement for a commercial antenna tuning unit made by Palomar Engineers Co.

For the noise source we use an ordinary IN751 Zener diode carrying 2 mADC followed by 40 dB of broadband amplification (see figure). The noise bandwidth is useful to more than 300 MHz. We did not use audio modulation of the noise source as suggested in the Reference because for us it did not improve the detectability of the null. Our "bridge" is a Mini-Circuits PDC-10-1 Directional Coupler. Our noise source enters the mainline of the coupler at its "out" pin 4 and is terminated by the T/R Switch and Probe at the coupler's "in" pin 1. The signal out of pin 3 represents the signal reflected back from the termination, and will be nulled for 50 ohms.

We peak detect audio noise from the spectrometer with the op amp circuit and display on the magnet-mounted bargraph meter. An optional speaker may be driven with the MOSFET circuit.

The circuit could have been a high-power, low-loss broadband directional coupler inserted permanently in the line to the preamplifier and mounted on the magnet. It would have had to be designed without ferrites. The coupler might have cost $400 commercially, and still might have had more than 0.3dB of mainline loss to add to our Noise Figure. So we sacrificed having the coupler permanently in the line and used the low power mini-circuits PDC-10-1, whose bandwidth is a surprising 0.5 to 500 MHz, thanks to its use of ferrites. It costs only $12.00, so of course that's the way we went. Hope this will be of use to others.

Sincerely, Jim James L. Engle

Reference 1: "The ARRL Handbook" (Bruce S. Hale KB1MW, Ed.) Chap. 25 p-32, American Radio Relay League, Newington, CT, 1989

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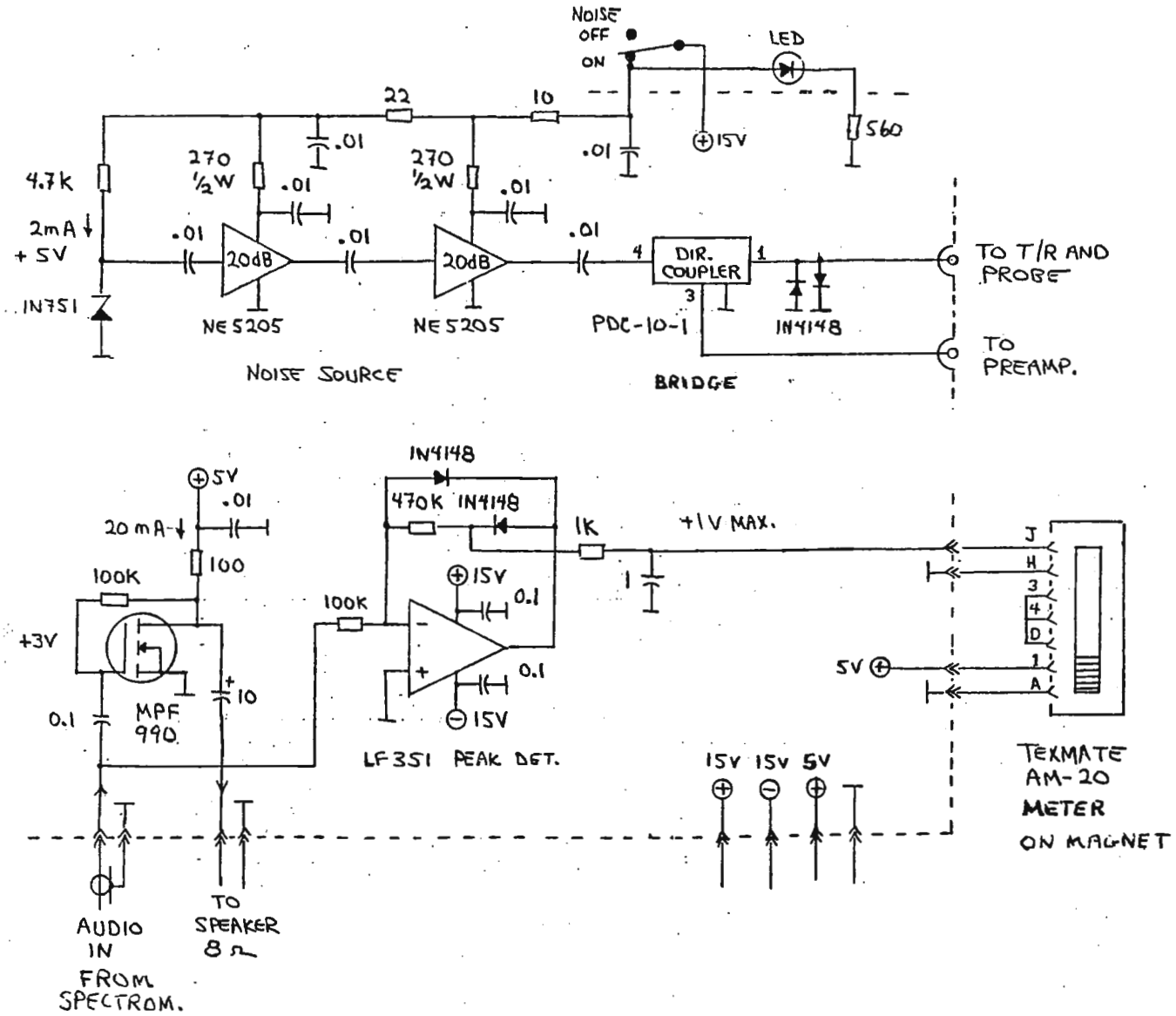

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TAMU NMR Newsletter

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1. Technical contributions should be on the *minimum* (NOTE!!) number of 8.5 x 11" (21 x 27.9 cm) pages, printed on one side only. Contributions may not exceed three pages without prior approval. Each page must have margins of at least 0.5 - 0.75" (1.3 - 2.0 cm) on all sides. Please observe these limits. Black ink for typing, drawings, etc., is essential. All drawings, figures, etc., should be mounted in place on the 8.5 x 11" pages. We do not routinely do reductions of figures or tables.

2. Foreign subscribers are reminded that regardless of the standard paper length you use, *all material* - letterhead, text, figures, addresses printed at the page bottom, *everything - must not exceed 10" (25.4 cm) from top to bottom.*

3. Significant savings of Newsletter pages and total space (and hence, subscription costs) can be made by exercising close control over the formatting and type sizes of the contributions. **Please consider the following:**

a) For those with computers, try using a smaller type font. The body of this notice is printed in 10 point type, which I believe is adequate for most purposes. Even 12 point is acceptable, I suppose, but not larger, please. Those who are computerized can also employ non-integral spacing of lines so that sub- and superscripts don't collide with the lines below or above.

b) **PLEASE** avoid excessive margins. *Instruct your secretaries to avoid normal correspondence esthetics or practices, however time-honored or 'standard'!* This page has margins on both sides of 0.6" (*ca.* 1.55 cm), which is very adequate. Margins of the same size at the top and bottom are sufficient also, but don't worry if there is more space at the end of your document, for I can often use such spaces for notices, etc.

Also, please avoid large amounts of unused space at the top of letters. Give thought to the sizes of figures, drawings, etc., and please mount these so as to use the minimum space on the page.

c) **AVOID DOUBLE SPACING BETWEEN LINES LIKE THE BLACK PLAGUE ! ! !** This is extremely wasteful of space. Even sans computer, small type and 1.5-line (if needed) spacing can be had with a little effort.

4. 'Position Available', 'Equipment Wanted', and Similar Notices. These are always welcome, without charge, but not for subscription credit, of course. Such notices will appear, however, *only* if received with these necessarily rigid constraints: a) Single spaced; b) both side margins 0.6 - 0.7" (1.5 - 1.7 cm.)- NOT WIDER; c) the minimum total height, please, but definitely no more than 4.5" (11.5 cm.) This will let me place such notices wherever a bit of space occurs.

5) Please provide short titles of all topics of your contributions, so as to ensure accuracy in the table of contents. This will also avoid titles created on the run by me, frequently without much serious or solemn thought.

Contributions which fail to meet any of these specifications may be returned for re-working.

Thank you! Your cooperation in making the Newsletter economical and tidy-looking will be appreciated, as will your efforts to keep my work to a reasonable level.

B. L. Shapiro
1 February 1990

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January 4, 1990
(received 1/11/90)

Dear Dr. Shapiro:

^{13}C MASS DEPT NMR of Whole Rape Seeds

In my last communication I discussed our experience using MASS NMR as a nondestructive method to define the unsaturate acyl distribution (oleyl and linoleyl) of triacylglycerols in a variety of our sunflower seeds. Since then, we have examined the feasibility of applying a polarization transfer technique, i.e. DEPT, to help increase sensitivity for low oil containing seeds. Rape seeds are especially difficult due to a more complex unsaturate acyl profile than sunflower seeds and a low quantity of oil (≈2mg/seed). The two rape seed varieties which we previously examined by a MASS inverse gated decoupling experiment were a high oleyl variety (Cascade) and a high erucyl variety (Bridger). We were able to identify some acyl groups (using 8 seeds) with the inverse gated technique but could not obtain adequate signal-to-noise within a practical time frame to determine the relative unsaturate quantities.

In this communication we would like to illustrate the type of sensitivity and resolution that is possible in the olefinic region using MASS DEPT on a Cascade (Figure 1a) and Bridger (Figure 1b) rape variety (4 seeds each) with 15 minutes of acquisition time.

The olefinic spectra in Figure 1 were not processed with either resolution or sensitivity enhancement. The acquisition time is 0.5 s using 32K data points and zero-filling to 128K before Fourier transformation. The DEPT spectra were obtained using a variable proton pulse of 90 deg and delay intervals (before polarization transfer and refocusing) of 3.125 ms. The decoupler 90 was calibrated for each sample using the DEPT pulse sequence.

The resolution shown in Figure 1 begins to approach that obtainable with high-resolution solutions NMR. We are able to identify not only each olefinic carbon pertaining to 18:1(O=oleyl), 18:2(L=linoleyl) and 18:3(Ln=linolenyl) but also the O_9, O_{10}, L_{10}, Ln_9 and Ln_{10} positional carbons (1,3- and 2-glycerol attachment). This permits a total determination of both the 18:1, 18:2, 18:3, 20:1(eicosenoyl) +22:1(erucyl) acyl profile as well as the 18:1, 18:2, 18:3 1,3-,2-acyl distribution. The positional carbons of the high oleyl rape seeds indicate that approximately half of the 18:2 and 18:3 acyl groups are in the 1,3-position and half are in the 2-position compared to a more random distribution (67% 1,3-acyl and 33% 2-acyl) of the 18:1 group. This is consistent with the acyl positional distribution which we have observed of extracted high oleyl rape oil. In contrast to the high oleyl Cascade seeds is a MASS DEPT spectrum of high erucyl rape seeds (Figure 1b). Obviously the 1,3-,2-acyl distribution of the Bridger seeds is quite different than that observed for the Cascade seeds with the majority of the 18:1, 18:2, and 18:3 attached to the 2-glycerol position. This is a result of the preferential substitution of 22:1 for 18:n (n=1,2,3) groups in the 1,3-glycerol position.

Our preliminary results also indicate favorable quantitative results between the MASS DEPT and ^{1}H MASS techniques as illustrated in Table 1.

Please credit this contribution to Horton Dunn's (Lubrizol Corp.) account

Sincerely,

K F Wollenberg

K. F. Wollenberg

Table 1: Acyl distribution of whole rape seeds derived from MASS DEPT and ^{1}H MASS experiments.

Seed	18:1	18:2	18:3	20:1+22:1	18:1+20:1+22:1	MASS
Cascade	60.1	21.7	12.1	6.0	66.1	DEPT
4 seeds	---[1]	19.7	12.1	---[1]	68.7	^{1}H
Cascade	54.6	25.1	13.1	7.2	61.8	DEPT
1 seed	---[1]	23.3	14.3	---[1]	62.3	^{1}H
Cascade	61.3	18.5	11.8	8.2	69.5	DEPT
5 seeds	---[1]	23.1	10.4	---[1]	66.4	^{1}H
Bridger	16.2	13.2	9.4	61.1	77.3	DEPT
4 seeds	---[1]	15.2	8.6	---[1]	76.2	^{1}H
Bridger	11.0	13.5	11.2	64.2	75.2	DEPT
1 seed	---[1]	15.3	11.9	---[1]	72.7	^{1}H

1) can not differentiate monoene distribution in ^{1}H spectrum

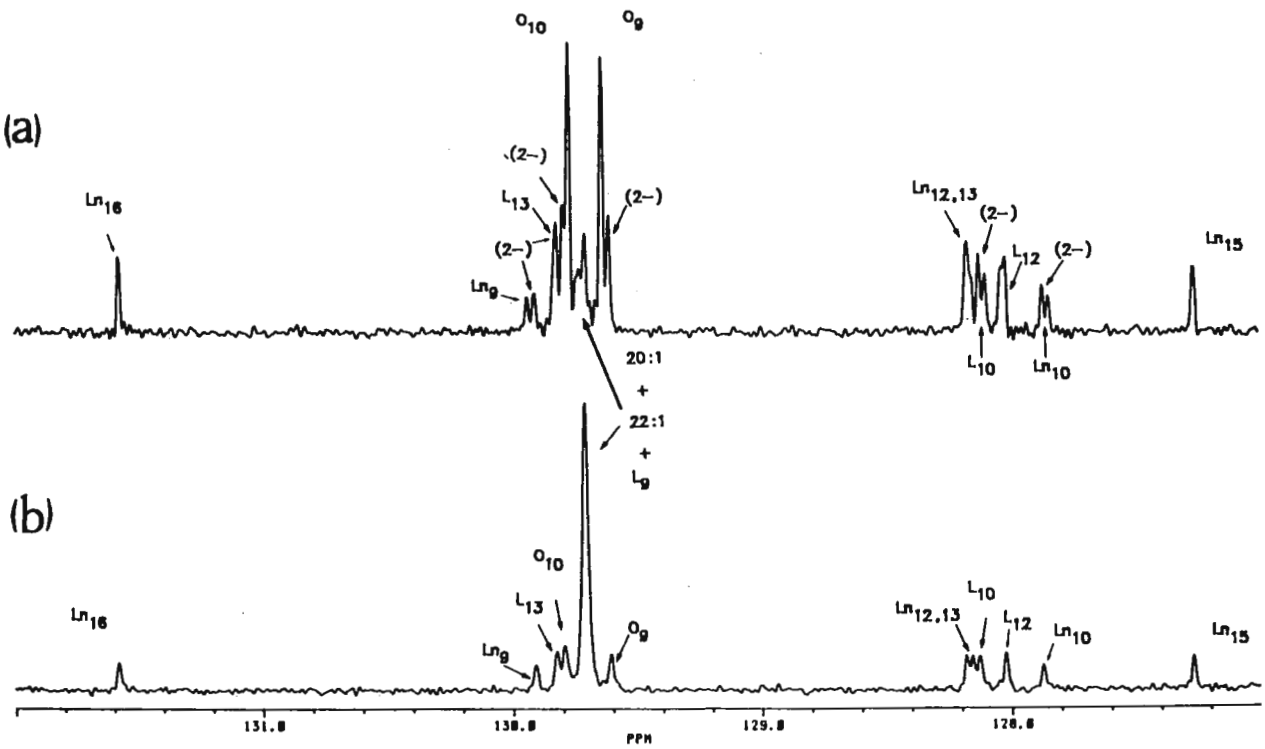

Figure 1: 100.6 MHz ^{13}C MASS DEPT spectra (variable H pulse = 90 deg) of a) high oleyl Cascade rape seeds(4 seeds) where only the 2-acyl group of each 1,3-,2-acyl peak pair is defined b) high erucyl Bridger rape seeds (4 seeds) with L_9, O_9, L_{10}, and L_{12} exhibiting exclusively 2-glycerol substitution. The spectra are referenced to α $O-CH_2$ = 61.9 ppm. Each peak is defined as O = oleyl, L = linoleyl, Ln = linolenyl with the subscript corresponding the carbon chain position. 20:1(eicosenoyl), 22:1 (erucyl), and L_9 (2-glycerol) position are not resolved as indicated in both spectra. Not all peaks appear at the same relative chemical shift as observed in solutions spectra of the corresponding oil.

McMASTER UNIVERSITY
Department of Chemistry
1280 Main Street West, Hamilton, Ontario L8S 4M1
Telephone: (416) 525-9140
FAXMAIL (416) 528-5030

(received 12/30/89)

Dec. 18, 1989

ERROR ESTIMATION IN T_1 MEASUREMENTS

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

Dear Barry,

After reading a couple of excellent new books on statistics, and that ugly word – chemometrics – we have been looking again at the inversion–recovery T1 experiment. The books in question are "Chemometrics: a textbook", by Massart, Vanderginste, Deming, Michotte and Kaufmann (Elsevier, 1988) and "Numerical Recipes" by Press, Flannery, Teukolsky and Vetterling (Cambridge University Press 1986). Both of them discuss quite well the non–linear least squares methods for fitting inversion–recovery data to the equation $M(t) = a - b\,e^{-rt}$ and point out that the errors in the parameters a, b and r can be defined many ways. These errors are often underestimated by commercial software, so we wrote a little program to actually calculate the sum–of–squares surface in parameter space near the best values of the parameters. The best values define the minimum of this surface, of course, and the contours of the surface are the most brutally honest measure of the errors in the parameters such as T1. The 95% confidence contour is calculated from the F statistic, and then the projection of this contour onto the parameter axis gives the error in the parameter. This is illustrated below for some deuterium T1 data on DMSO. This is a contour in the plane of the b and r parameters, and inclination of the contour shows the strong correlation between these parameters. The projection of the contour gives a 95% error limit in T1 of +/– 2.2%, compared to the 1.5% that would be calculated from two standard deviations. This is typical of what we have found: the error is actually about half again larger than the standard deviation would have you believe.

b \ r	0.576	0.579	0.581	0.583	0.585	0.587	0.590	0.592	0.594	0.596	0.598
1.786	1.70	1.60	1.62	1.76	2.02	2.38	2.87	3.46	4.16	4.97	5.89
1.789	1.68	1.51	1.46	1.53	1.71	2.01	2.43	2.95	3.59	4.34	5.19
1.793	1.71	1.47	1.35	1.35	1.47	1.70	2.05	2.51	3.08	3.76	4.55
1.796	1.80	1.49	1.30	1.23	1.28	1.45	1.73	2.12	2.63	3.24	3.97
1.800	1.95	1.57	1.31	1.17	1.16	1.25	1.47	1.79	2.23	2.78	3.44
1.803	2.16	1.71	1.38	1.18	1.09	1.12	1.26	1.52	1.90	2.38	2.98
1.807	2.43	1.91	1.51	1.24	1.08	1.04	1.12	1.31	1.62	2.04	2.57
1.810	2.77	2.17	1.70	1.35	1.13	1.02	1.03	1.15	1.40	1.75	2.21
1.814	3.16	2.49	1.95	1.53	1.24	1.06	1.00	1.06	1.23	1.52	1.92
1.817	3.61	2.87	2.26	1.77	1.40	1.16	1.03	1.02	1.13	1.34	1.68
1.821	4.12	3.31	2.63	2.07	1.63	1.31	1.12	1.04	1.08	1.23	1.50
1.824	4.69	3.81	3.05	2.42	1.91	1.53	1.26	1.12	1.09	1.17	1.37
1.828	5.32	4.37	3.54	2.84	2.26	1.80	1.47	1.25	1.15	1.17	1.30
1.831	6.01	4.98	4.08	3.31	2.66	2.13	1.73	1.44	1.28	1.23	1.30
1.835	6.77	5.66	4.69	3.84	3.12	2.52	2.05	1.70	1.46	1.34	1.34
1.839	7.58	6.40	5.35	4.43	3.64	2.97	2.43	2.01	1.70	1.52	1.45
1.842	8.45	7.20	6.08	5.08	4.22	3.48	2.87	2.37	2.00	1.75	1.61

Yours truly,

Alex D. Bain
Associate Professor
of Chemistry
BAIN@MCMASTER.CA

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Direct Dial

January 3, 1989
(received 1/8/90)

Maintaining Moderately Low Temperatures on a VXR-500

Dear Barry:

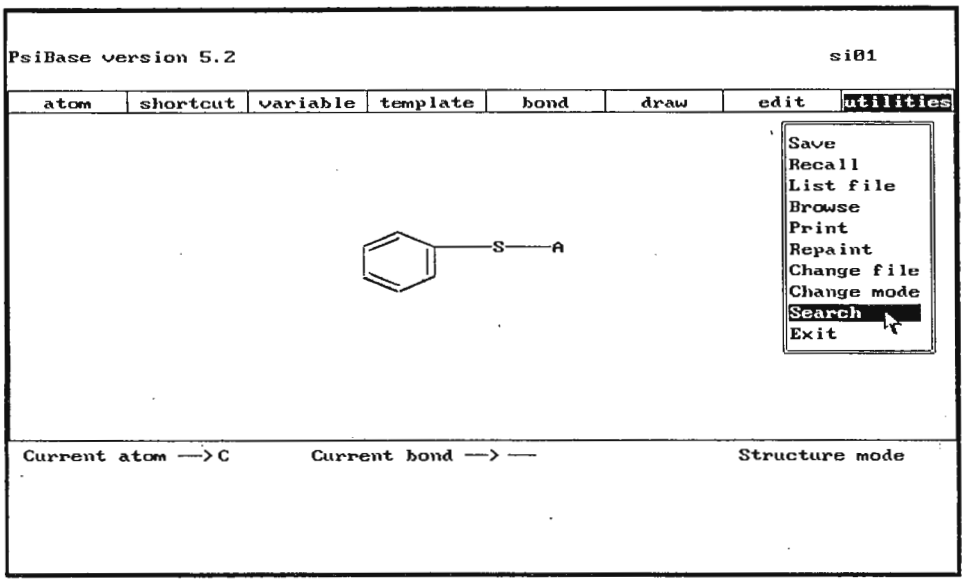
In the absence of some form of temperature control, our VXR-500 spectrometer runs at about 26.4° and above. We typically perform experiments at 20-25°. We have rigged up a simple, maintenance-free device to deliver cooled nitrogen to the probe. A couple of new owners of Varian instruments have inquired about our set-up, so we thought there might be enough general interest to warrant describing it here.

The cooling chamber is a 7 liter dewar with a clamp lid (Lab-Line Instruments), which is filled with 50:50 aqueous diethyl glycol. Three holes have been drilled into the lid. Into one hole is inserted a cold finger (Cole Palmer Immersion Cooler VLT-60A); the other two serve as entry and exit ports for a coil of copper tubing, through which the nitrogen travels. The connection between the dewar and the probe is made via 5 feet of 5/8" insulated rubber tubing. Within this tubing is a T valve, to permit bypass of the cooling chamber if desired.

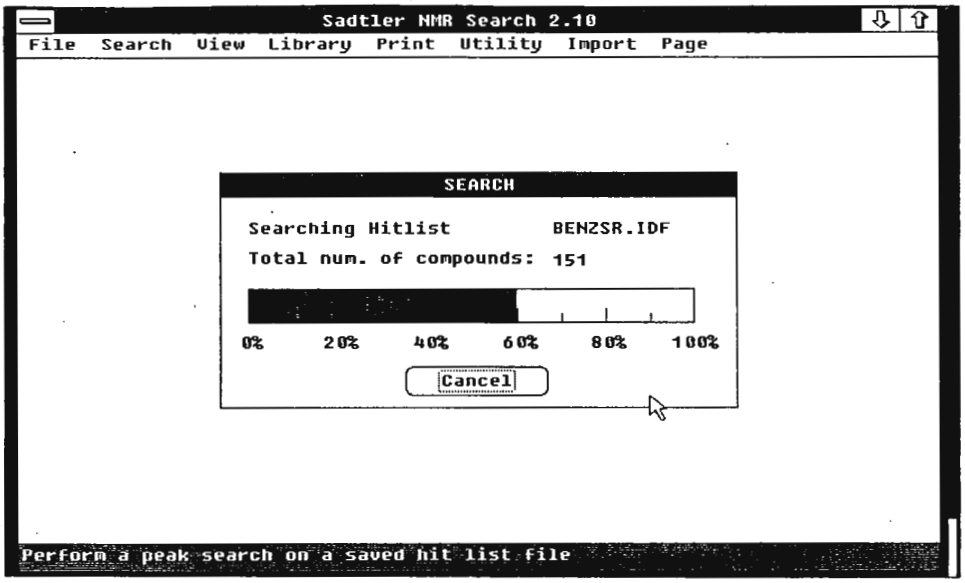
This apparatus has run unattended for almost a year, without a significant build-up of ice.

Sincerely yours,

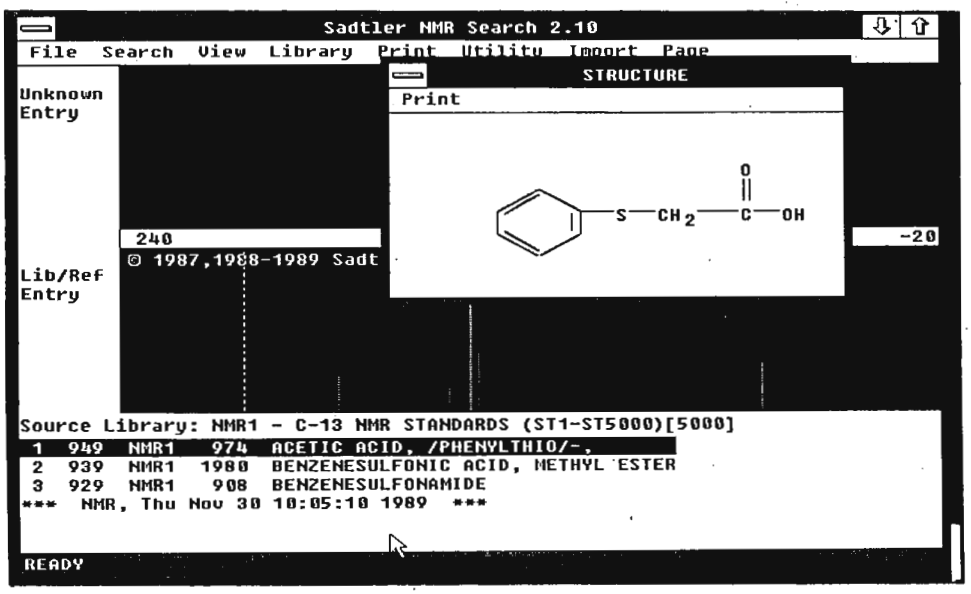
David N. Greeley, David C. Fry, and Ross G. Pitcher
Department of Physical Chemistry



Step 1: Search for all thiophenol derivatives.



Step 2: Search for those derivatives that match your spectrum.



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December 14, 1989 (received 12/22/89)

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

Title: Homonuclear Editing

Dear Barry,

The information available from the application of NMR to in vivo situations is often limited by the problem of severe spectral overlap. Several methods have been recently proposed to address this issue. Typically these methods are limited either by observing a single compound at a time[1], or are somewhat time consuming[2] (i.e., 2D COSY) and thus are mostly limited to observing steady-state metabolism, or are fairly complicated requiring additional hardware[3]. In many situations, all that is required to edit the spectra is appropriate suppression of the unwanted peak(s). The best example of this is solvent suppression in ^{1}H NMR. In this light, we propose an editing scheme which consists of selective suppression of a group of scalar coupled spins via a homonuclear pulse sequence. In this way resonances under the suppressed region can be revealed.

One pulse sequence which can provide effective suppression is based on the standard decoupler calibration scheme popularized by Bax[4]. In that experiment a simple AX system is examined. The ^{13}C nuclide is given a 90° pulse and is allowed to evolve into a "J-ordered" state at a time τ later. At this time a 90° pulse is applied to the protons and the carbon signal is immediately acquired. When $\tau = 1/2J$, all the carbon signal is effectively converted into multiple quantum transitions and is not detected. The homonuclear editing version of this is:

$$90_H - 1/2J - 90_{sel} - AQ,$$

where the initial 90° pulse is hard and the second 90° pulse is semi-selective.

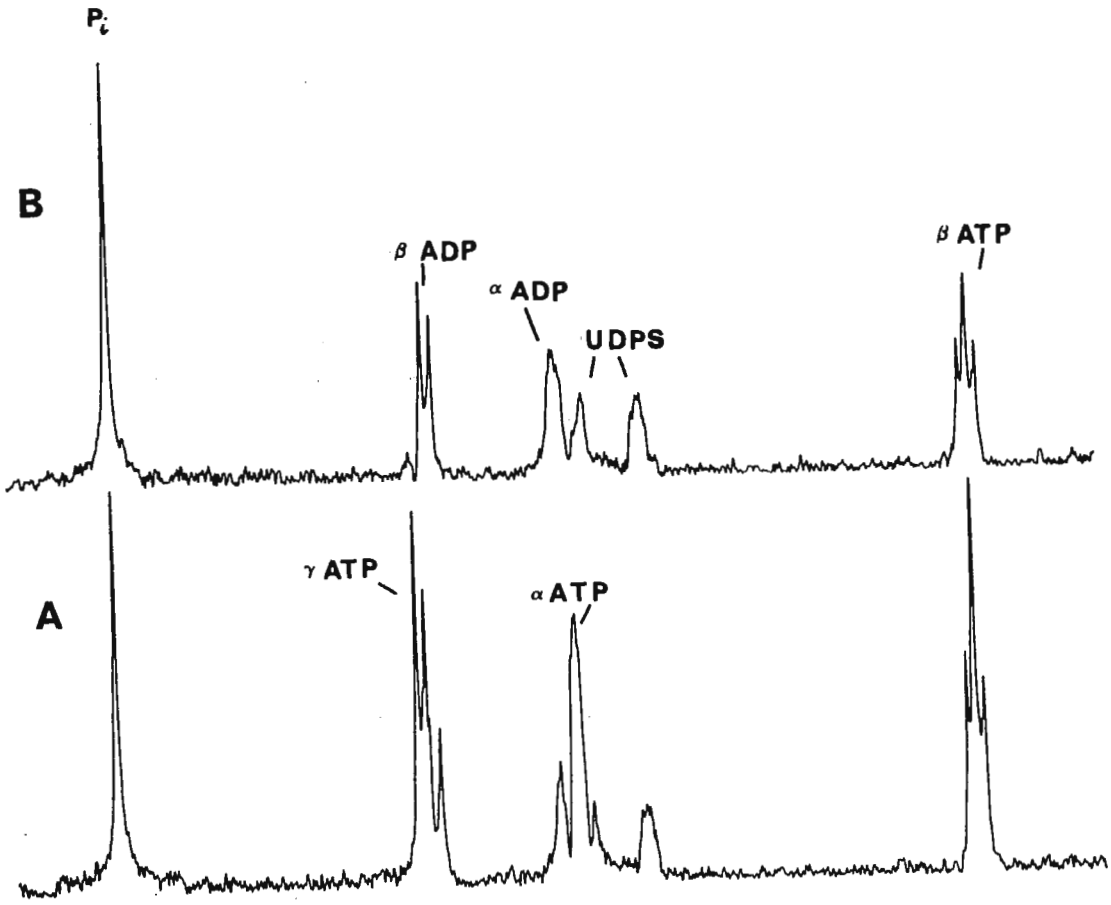
To test this sequence, we applied it to a microsphere containing ATP, ADP, UDP sugars, and P_i on GE 4.7T CSI system. Data were collected using a home-built Helmholtz coil. The selective pulse was a 3 ms single-lobed sinc function. Because of the delay (25 ms) between the hard 90 and the acquisition these spectra are presented in magnitude mode. Software controlled attenuation switching was used to change the power for the hard and selective pulses. In practice we found that insertion of a hard 180° pulse during the 1/2J period (plus exorcyling and cylcopsing) refocused chemical shifts and improved sensitivity. The results of this experiment are shown in the figure where A) was acquired with the selective 90° pulse power set to zero and B) was acquired with the selective 90° pulse set to 35% and positioned on the β resonance of ATP. A 2.5 Hz linebroadening was applied and both spectra were similarly scaled. Note that this very effectively suppressed the α

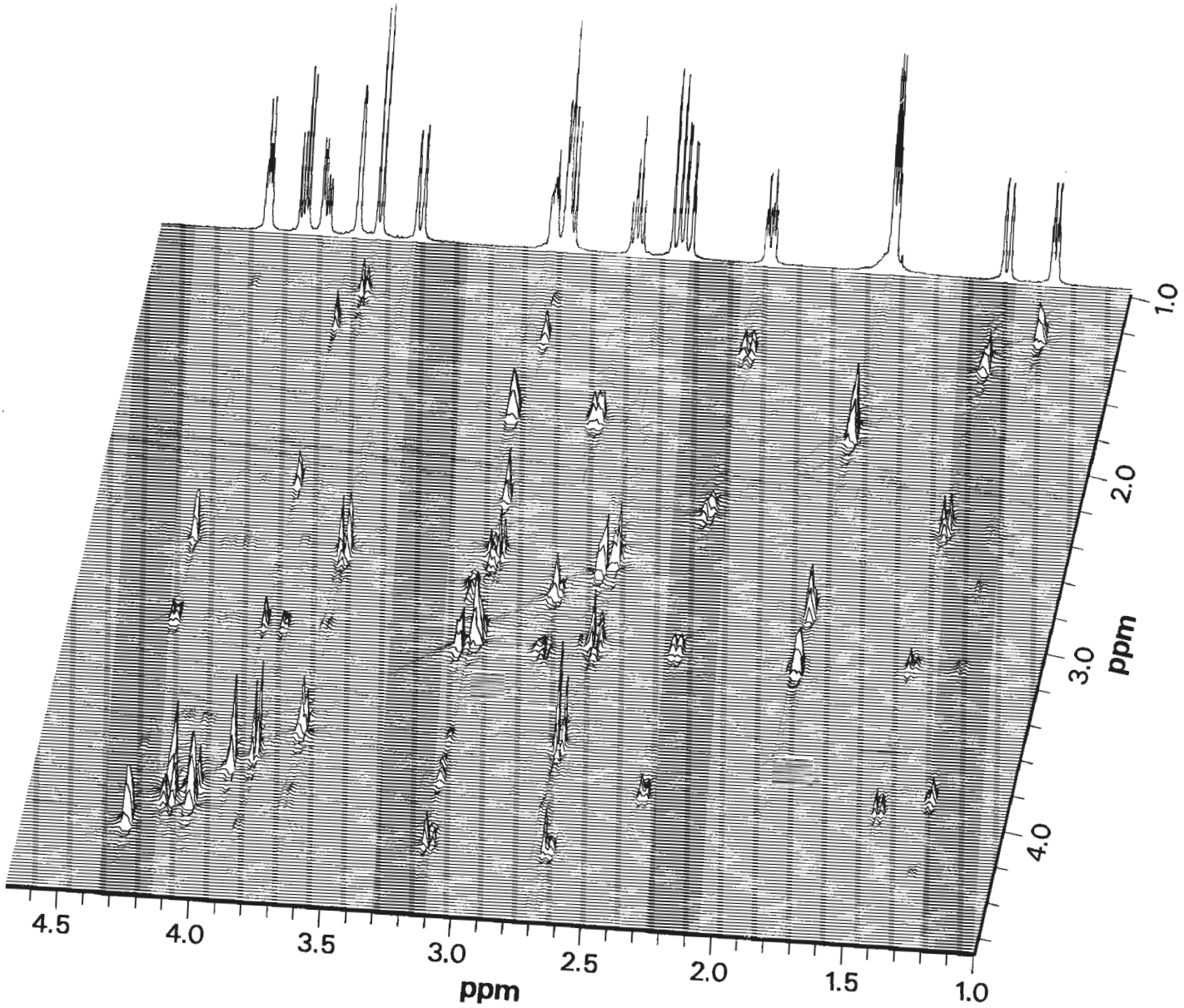
and γ resonances of ATP and allows straightforward observation of the resonances of ADP and the UDP sugars.

Sincerely,

Bruce A. Berkowitz Robert E. London

1) D.L.Rothman,K.L.Behar,H.P.Hetherington, and R.G.Shulman, Proc. Natl. Acad. Sci. USA 81, 6330 (1983).
2) B.A.Berkowitz,S.D.Wolff, and R.S.Balaban, J. Magn. Reson. 79, 547 (1988).
3) K.M.Brindle,M.B.Smith,B.Rajagopalan, and G.K. Radda, J. Magn. Reson. 61, 559 (1985).
4) A.Bax, J. Magn. Reson. 52, 76 (1983).





HOHAHA of Strychnine on an Omega 600

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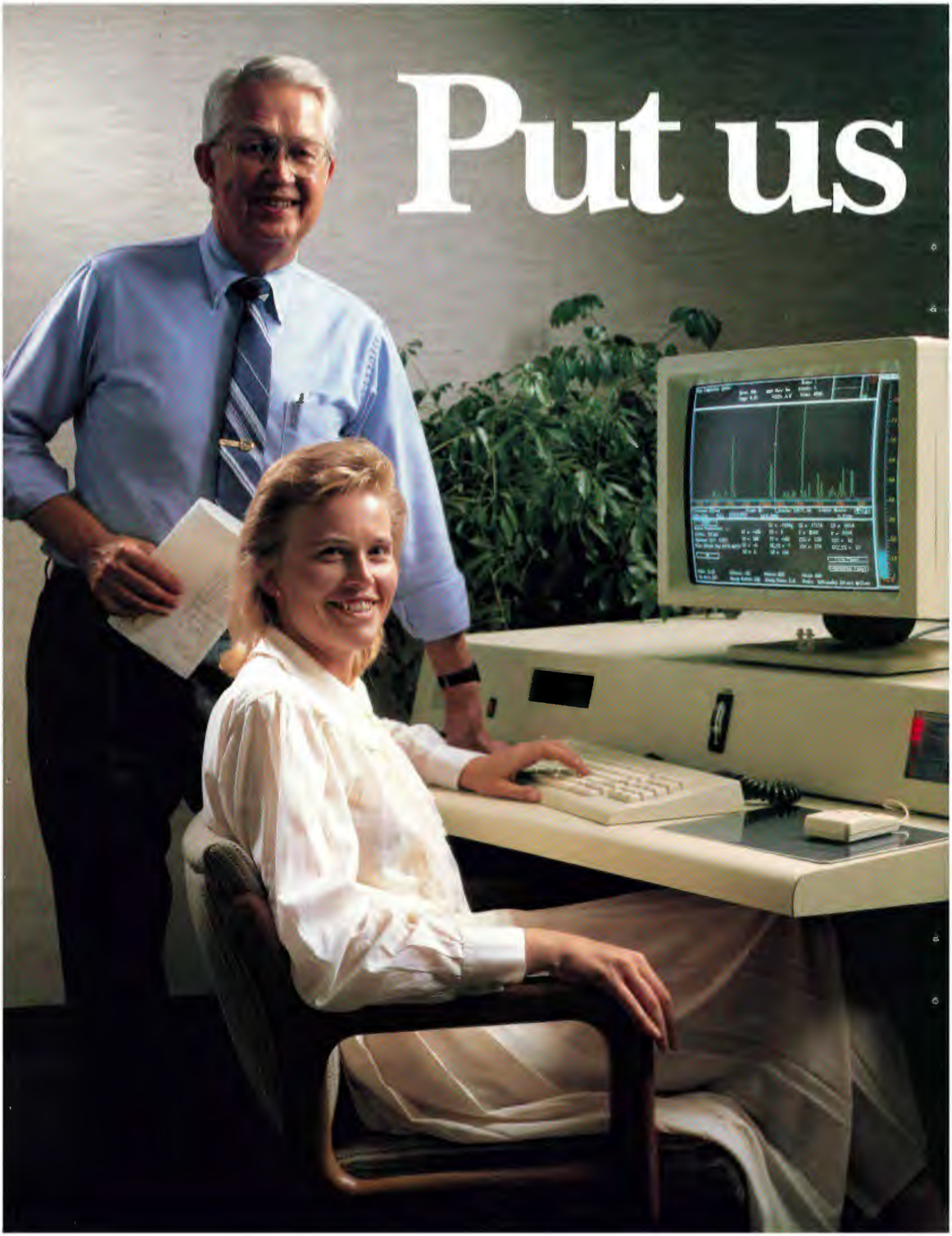
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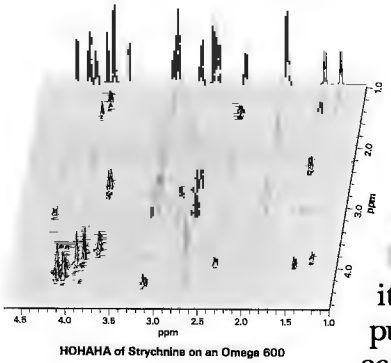

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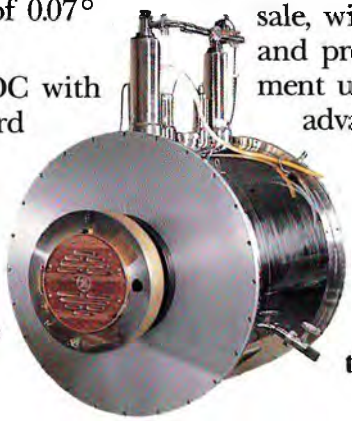

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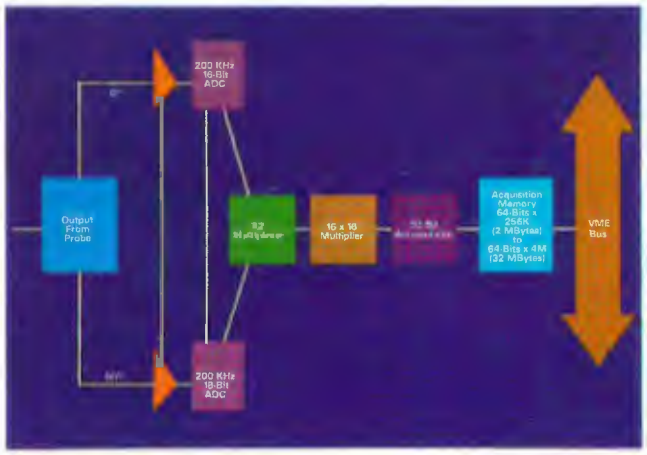

Fig. 1

The Alpha HDR digitizer.

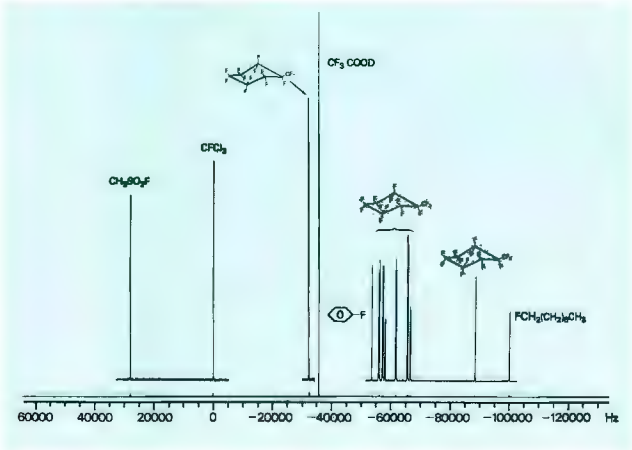

Fig. 2

200 KHz spectral width ^{19}F spectrum acquired on a GN-500 Omega System. Note the extremely flat baseline obtained with the Alpha HDR.

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DEPARTMENT OF CHEMISTRY DAVIS, CALIFORNIA 95616

January 2, 1990
(received 1/4/90)

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

The Application of NOE to Paramagnetic Iron-Sulfur Proteins

Dear Barry:

The use of the nuclear Overhauser enhancement (NOE) effect in structure elucidation of proteins using one- and two-dimensional NMR techniques is well known. The method has also been applied successfully to paramagnetic forms of a variety of hemoproteins. However, its usefulness has not been demonstrated in the case of iron-sulfur proteins. The strongly relaxed hyperfine shifted resonances in different iron-sulfur clusters arise primarily from cysteine amino acid protons. The identification or assignment of these protons to a particular cysteine residue, or at least determining the β-CH_2 geminal pair of protons, is crucial in obtaining structural information on redox states. The application of NOE methodology to this class of paramagnetic proteins was hampered by the presence of broad and efficiently relaxed proton resonances coupled with the air-sensitive nature of these samples.

We have been able to demonstrate the usefulness of NOE to various cluster types in this class of proteins of molecular weight ranges 8 - 50 kDa without interference of spin-diffusion. The determination of a β-CH_2 geminal proton pair can be easily accomplished even for a low-molecular weight protein and with nuclear spin-lattice relaxation times in 3 - 10 ms range. It appears that the method will have wide applicability for various iron-sulfur proteins.

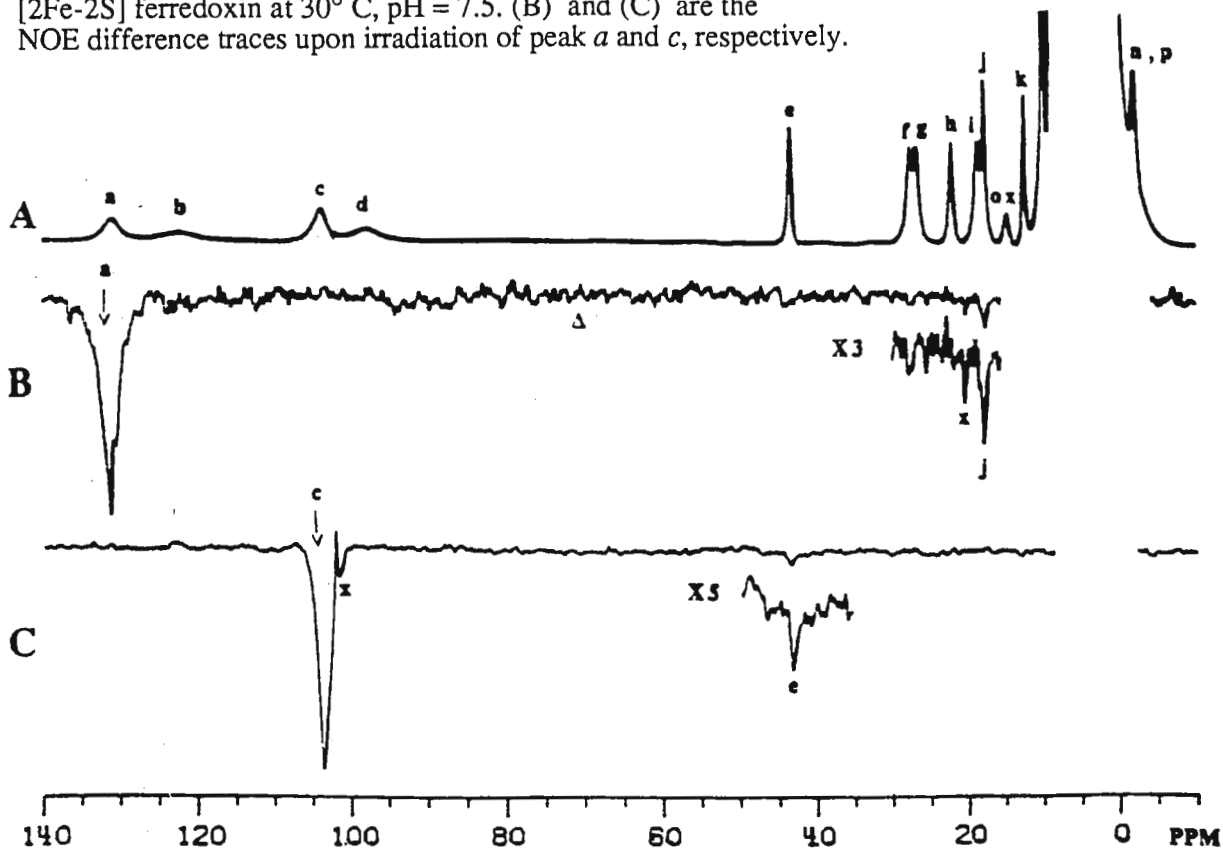
In collaboration with Lucia Banci and Ivano Bertini of the University of Florence, we have assigned all hyperfine-shifted signals to individual cysteines in the sequence, and identified the iron bound to Cys 41 and 46 as the one that accepts the odd electron in the reduced form of a 2Fe-2S alga ferredoxin. Figure 1 (B) and (C) show typical 1D NOE difference traces. The NOEs are small and the dynamic range problems severe. Nevertheless, the NOE a→j and c→e (trace (B) and (C), respectively) identifies the vicinal protons (j and e) of two cysteines coordinated to Fe(III).

Sincerely,

L. B. Dugad
Postgraduate Research Associate

Gerd N. La Mar
Professor of Chemistry

Figure 1: (A) 360 MHz ^{1}H NMR spectrum of reduced [2Fe-2S] ferredoxin at 30° C, pH = 7.5. (B) and (C) are the NOE difference traces upon irradiation of peak *a* and *c*, respectively.



Position Available: **Manager, NMR Core Facility**

Applications are invited for the position of manager of the NMR Core Facility at the University of Alabama at Birmingham . Job duties include overseeing day-to-day operation, routine maintenance, software update, implementation of new NMR techniques, assisting users etc. Basic knowledge in NMR instrumentation and 2D-NMR spectroscopy is a definite asset. The Facility consists of Bruker NMR spectrometers operating at 600 MHz (AM-600), 400 MHz (WH-400, upgraded) and 200 MHz (CXP-200,upgraded), and a 4.7 T 15 cm horizontal bore imaging/spectroscopy magnet system. The NMR Facility is also equipped with a Sun 4/110 work station and microVAX II and VAX 11/750 computers. Excellent opportunities exist for collaborative work in some of the ongoing research projects. Salary is commensurate with experience and educational background. Send a resume and arrange for three letters of recommendation to reach: Dr. N. Rama Krishna, Director, NMR Core Facility, CHSB-B31, Comprehensive Cancer Center, University of Alabama at Birmingham, Birmingham, AL 35294. EO/AAE.

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Dear Dr Shapiro

^{13}C-^{19}F SOLID STATE NMR

Recently we have done some initial work on ^{13}C-^{19}F single pulse and cross polarisation MAS experiments using a Bruker ^{13}C-^{19}F DBMAS probe on an MSL200. We have looked at a variety of PTFE (Fluon) type samples and fluorinated samples such as perfluorobenzophenone.

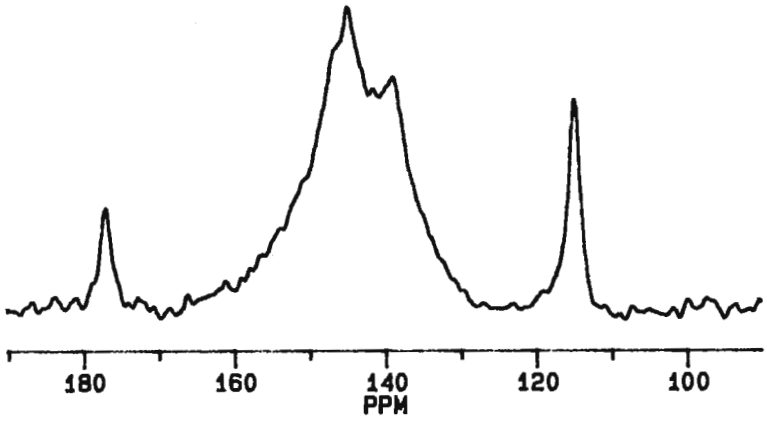
We were able to use decoupling fields of 100 kHz with single pulse high power decoupling sequences without arcing problems and a decoupling field of 85 kHz for a satisfactory CP match with no arcing problems. However, using a sample of n-$C_{24}F_{50}$ (Aldrich) experiment showed that the effective decoupling field was not sufficient to remove the dipolar broadening for a CF_2 resonance (estimated at 67 kHz) but could be gainfully used to narrow the linewidth of the CF_3 resonance (estimated at 43 kHz).

With PTFE type samples we found spinning samples at <2 kHz gave a better CP match than at higher spinning rates, as with adamantane in ^{13}C-^{1}H CPMAS, indicating that molecular dynamic processes are present.

Though more work of a practical nature needs to be done, we can obtain spectra of adequate resolution as illustrated by the spectrum below of perfluorobenzophenone which shows the carbonyl resonance at a chemical shift of 176.9 ppm, the quaternary phenyl carbons at a chemical shift of 114.7 ppm

and inequivalent CF resonances between chemical shifts of approximately 130 and 160 ppm. The spectrum was referenced using C_6F_6. A comparison with a spectrum of protonated benzophenone showed similar detail is resolved although the analogous carbon resonances are at different chemical shifts.

13C CPMAS/FLIP PERFLUOROBENZOPHENONE



Please credit this contribution to Alan Bunn.

Yours sincerely

Richard Jennings Peter.

RICHARD JENNINGS, PETER JACKSON
Spectroscopy, Surface Science & Simulation Group
Wilton Materials Research Centre

Postdoctoral Position Available

I will have a postdoctoral position available starting April 1, 1990; the initial appointment will be for one year. Anyone interested in the development of new techniques in high resolution NMR spectroscopy, particularly for applications in biochemical studies, is encouraged to apply. The facilities available include AMX-600, AMX-500, AM-500 and MSL-300 spectrometers and a Cray-XMP supercomputer, Convex C1 and C240 mini-supercomputers and Sun and SGI workstations. Please send a resume and the names of two references to:

Dr. Mark Rance
Dept. of Molecular Biology
Research Institute of Scripps Clinic
10666 North Torrey Pines Road
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January 5th, 1990.
(received 1/11/90)

TAMU NMR Newsletter
966 Elsinore Court
Palo Alto, CA94303
Dear Dr. Barry Shapiro

Methods for ^{31}P Spectroscopic Imaging

We would like to report on our latest developments in Spectroscopic Imaging (SI) of phosphorus metabolites in the human brain. Previously we have reported data obtained using the 'FID' acquisition method (1), which encounters the well-known problem of the missing sampled data points as the phase encoding gradients are applied during the initial part of the FID. We have previously implemented a linear prediction algorithm to correct for the baseline distortions produced, which worked for data of high S/N. However, as we aim to obtain spectroscopic images with as high a spatial resolution as possible, the S/N of our data is relatively poor. In this situation many of the alternatives to the Fourier transform which are less sensitive to missing time data points, as well as automatic baseline correction techniques, fail to operate reliably.

Recently we have implemented a short spin echo acquisition which allows all time data points to be sampled. In order to reduce the T_2 losses encountered in the spin echo sequence, the phase encoding gradients are applied in a bipolar fashion on either side of the 180° refocussing pulse, and the signal is sampled from the peak of the echo only. We use a homemade 10" birdcage design head coil, operating at 34 MHz for phosphorus, and our SI sequence obtains volumetric data. Typically we obtain 12x12x12 phase encodings and 256 sample points in the spectral dimension. Following some smoothing of the data during processing, we estimate the final spatial resolution to be approximately 3 cm in each dimension.

The figure shows two spectra obtained in the brains of normal volunteers, corresponding to a 25 cc volume, obtained using a) the spin echo method and b) the FID method, along with a diagram of the pulse and gradient timings used. Some of our observations from using the spin echo method are:

1. The spin echo method successfully eliminates the rolling baseline problem which plagues the FID acquisition method, and the quality of the resultant spectra is significantly improved.

2. By using the bipolar gradient application, we are able to obtain a spin echo time of 2.7 msec on our Philips Gyroscan whole-body imaging system. This is set by the gradient slew rate limitations on a system which has the capability for 0.3 G/cm with a switching time of 1 msec. The TE could certainly be shortened on systems which have stronger gradient capability, and especially with the use of self-shielded gradients.

3. The additional T_2 losses encountered appear to be small. In particular we note that the broad PDE resonance is still clearly observed, which suggests that this resonance is composed of multiple resonances with a T_2 value longer than that which would be derived from a linewidth measurement.

The relative amplitudes of all lines compare well with spectra obtained using ISIS localization.

4. We have been limited by rf power capabilities to typical 180° pulse lengths of 360μs. This results in signal losses from off resonance effects, as well as necessitating the use of an EXORCYCLE sequence to eliminate unwanted transverse magnetizations.

5. The minimum total acquisition time is set by the number of phase encodings and the need for EXORCYCLE. To keep this time within reason, we use TR = 350 msec and an excitation pulse angle of >90°, which following the refocussing pulse results in an effective excitation angle of <90°.

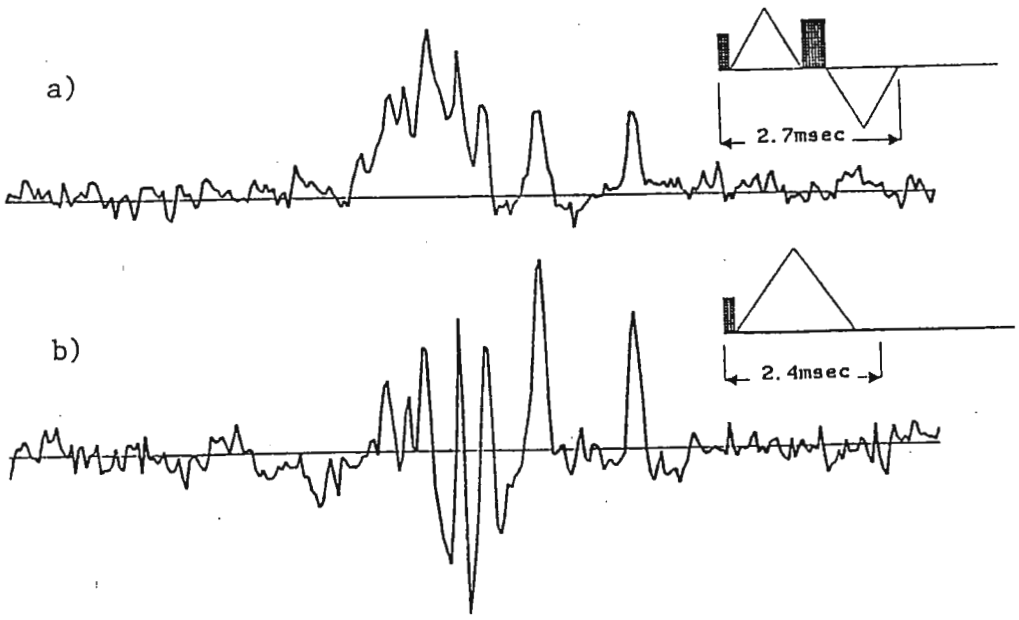
6. We have developed a SI display package which runs on a graphics workstation (VAXstation) which allows display of spectra or images. The ability to rapidly review of the large data sets produced has greatly enhanced our clinical SI capabilities. Images obtained using this package are to be shown in an upcoming paper (2).

(1) D.B. Twieg, D.J. Meyerhoff, B. Hubesch, K. Roth, D. Sappey-Marinier *et. al., Magn. Reson. Med.* 12.291(1989).
(2) A.A. Maudsley, D.B. Twieg, D. Sappey-Marinier, B. Hubesch, J.W. Hugg, G.B. Matson and M.W. Weiner. *Magn. Reson. Med.* In press (1990).

sincerely,

Andrew A. Maudsley, Ph.D.
Don B. Twieg, Ph.D.
Gerald B. Matson, Ph.D.
Michael W. Weiner, M.D.

^{31}P Spectroscopic Imaging
Comparison of Spin Echo and FID Acquisition



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Villeurbanne, January 4, 1990

Dr Bernard SHAPIRO
TAMU NMR Newsletter
966 Elsinore Court
PALO ALTO CA 94303

Dear Professor Shapiro,

I have pleasure to inform you that the fourth meeting of the "Groupe Thématique, Magnétisme Nucléaire et Biologie" which is sponsored by the french Biological Chemistry Society, will be held on 23 to 26 October 1990 in Eveux a small village, 25 km North-West to Lyon. In this village, Le Corbusier built a wonderful monastery at the begining of the sixties. The convent is also devoted to meetings and assemblies. So we plan to take this opportunnity to gather about 130 participants who are interested by recent progress of biological NMR. This year, topics are large molecules structure determination and contribution of NMR to metabolism knowledge.

On behalf of the organizing committee, I would ask you if the first announcement (in French) could be proposed to the readers of TAMU NMR Newsletter.

Thank you.

Sincerely yours,

Prof. André BRIGUET

SOCIETE DE CHIMIE BIOLOGIQUE

GROUPE THEMATIQUE
MAGNETISME NUCLEAIRE ET BIOLOGIE

La quatrième réunion du groupe thématique "Magnétisme Nucléaire et Biologie" (responsable : P. COZZONE) aura lieu à EVEUX (Rhône), du mardi 23 octobre au soir au vendredi 26 octobre après-midi 1990. Cette réunion fait suite à celles de Carry-le-Rouet (1982), Autrans (1984) et Ajaccio (1988). Elle doit permettre de faire le point sur les derniers développements concernant les applications biologiques de la RMN et sera intitulée :

"PROGRES METHODOLOGIQUES DE LA RMN EN BIOLOGIE : STRUCTURES MACROMOLECULAIRES ET METABOLISME"

Les sujets abordés concerneront principalement :
(1) Les méthodes RMN récentes et leurs applications pour l'élucidation de la structure tridimensionnelle des macromolécules d'intérêt biologique
(2) Les progrès dans l'exploitation du signal RMN : traitement, attribution, quantification
(3) Contribution de la RMN à l'étude du métabolisme cellulaire et tissulaire

La réunion comportera des exposés présentés par des conférenciers invités ainsi que des communications orales et par voie d'affiche. Les résumés des travaux présentés seront publiés dans "Regards sur la Biochimie".

COMITE SCIENTIFIQUE :
Paul CANIONI, Patrick COZZONE, Michel DECORPS, Eric GUITTET, Jean-Marc LHOSTE, Marius PTAK et André BRIGUET.

COMITE LOCAL D'ORGANISATION :
André BRIGUET, Gabriel BAVEREL, Jean Claude DUPLAN, Bernard FENET et François PENIN.

Pour tout renseignement :

A. BRIGUET Tél : 72 44 82 67

INFORMATIONS GENERALES

Le colloque "Progrès Méthodologiques de la RMN en Biologie" aura lieu à EVEUX (près de l'ARBRESLE, Rhône) situé à 20 km au Nord-Ouest de LYON. La réunion se tiendra au couvent dominicain de la Tourette. L'accueil des participants s'effectuera mardi 23 octobre dans la soirée. Un service de cars assurera le transport des participants depuis la gare de la Part-Dieu. Des indications détaillées seront communiquées au moment de l'inscription définitive. L'hébergement est prévu sur place en pension complète du mardi soir 23 octobre au vendredi 26 octobre après-midi et se fera au tarif suivant :

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Cet arrangement forfaitaire n'est pas fractionnable. Les participants qui ne désirent pas assister à la totalité de la réunion ou qui ne souhaitent pas bénéficier de la pension complète devront prendre en charge leur propre hébergement.

Les frais d'inscription s'élèvent à 450 F pour les membres de la Société de Chimie Biologique et à 550 F pour les non-membres. Le demi-tarif s'appliquera pour les étudiants et les chercheurs non statutaires. Un nombre limité de bourses pourra être attribué à des étudiants.

Des titres de réduction sur les transports SNCF seront adressés aux congressistes en temps utile.

Les personnes intéressées sont invitées à retourner la fiche de préinscription ci-jointe et leurs suggestions sur le programme scientifique avant le :

1er MARS 1990

Une seconde circulaire contenant les éléments du programme scientifique, les indications pour la rédaction des résumés, les détails d'organisation et une fiche d'inscription définitive sera envoyée. La fiche d'inscription définitive devra être retournée avec le résumé des communications et le montant des frais d'inscription et d'hébergement avant le :

31 JUILLET 1990

Toute correspondance doit être adressée à :
Prof. André BRIGUET
Laboratoire de Résonance Magnétique Nucléaire
Université Claude Bernard - Lyon 1
43, Bd du 11 Novembre 1918
69622 - VILLEURBANNE - Cedex

Lawrence Livermore National Laboratory

Dr. Bernard L. Shapiro
TAMU NMR News Letter
966 Elsinore Court
Palo Alto, CA 94303

January 12, 1990
(received 1/13/90)

^{13}C-CPMAS Studies of a DNA-Protamine Complex

Dear Barry:

Our interest in using CPMAS techniques to study polymer systems continues. This time I thought I would describe some recent studies on a DNA-Protamine complex.

Protamine is a small basic protein that binds to and coats the entire surface of DNA, neutralizing the phosphodiester backbone and packaging it inside the nuclei of human and other mammalian sperm. These proteins are used to inactivate the genes in each sperm and prepare them for transport into the oocyte where they can be reactivated as needed during the course of normal embryonic development. Sperm of the bull (Bos tarus) contain only a single type of protamine, P1. While modeling and x-ray fiber diffraction studies suggest that the polyarginine-rich core of the protamine molecule binds to the minor groove of DNA, the actual location of the DNA binding region within the protamine molecule has only been hypothesized and the site of protamine binding has not been confirmed.

Solid state ^{13}C CPMAS techniques are being used to study the nucleoprotein complex due to its limited solubility. CPMAS, $T_{1rho}(C)$, $T_1(C)$, T_{CH}, $T_{1rho}(H)$, and interrupted decoupling measurements have been made on arginine, polyarginine, calf thymus DNA, and the natural DNA-protamine complex isolated from bull sperm nuclei. Studies are in progress on isolated bull protamine-P1. CPMAS relaxation measurements were made at a maximum rf field strength of 60 KHz for both proton and carbon nuclei. $T_{1rho}(C)$ values for individual carbon peaks for the DNA-Protamine-1 complex are approx. a factor of 5 less than for DNA itself, 8 ms compared to 37 ms. T_{CH} measurements require a two component fit for high field peaks and a one component fit for low field peaks for both the DNA- Protamine-1 complex as well as for DNA. Values are between 2 and 3 times smaller for the DNA-Protamine-1 complex than for DNA, 0.4 ms compared to 1.6 ms for the low field peaks and 6 us to 24 us and 150 us to 300 us for the high field peaks. We are in the process of sorting out the data in terms of a complex between protamine and DNA. The CPMAS measurements were made on a MSL-300 spectrometer using 7mm rotors at a spinning rate of about 4KHz.

Our MSL-300 continues to work well. We recently obtained a high speed MAS probe and plan to report on its use next time. We have had a problem with the printer supplied with the MSL, however. The printer works well when the amount of material to be printed is a page or less, however when a directory listing, for example, is requested the paper advance mechanism invariably fails and destroys the print out. We have temporarily solved this problem by using a PC printer with a buffer. If anyone knows a solution to this problem we would appreciate hearing about it.

Sincerely,

Ray Ward

Raymond L. Ward James A. Happe Rod Balhorn

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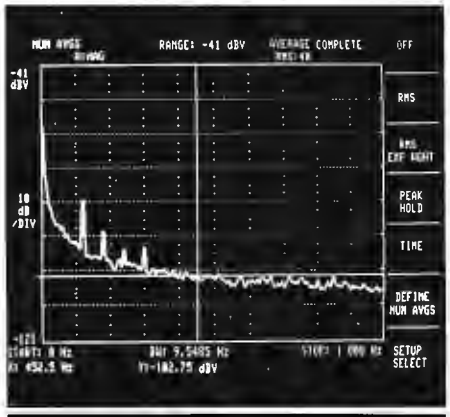

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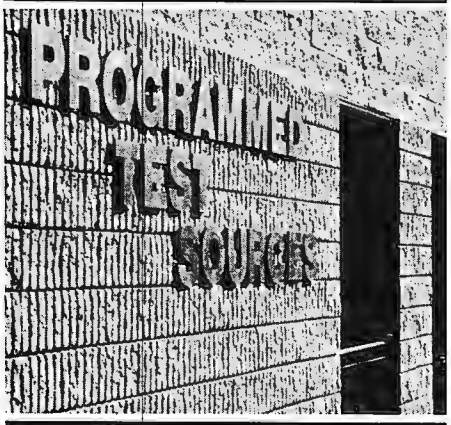

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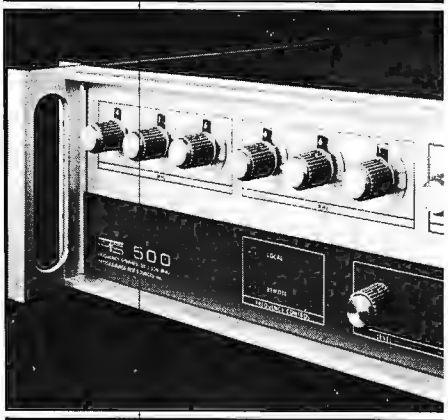

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January 2, 1990
(received 1/12/90)

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

Re: "Mental Relaxation"

Dear Barry,

The decay rate of sodium double-quantum (DQ) coherence[1,2] was measured in the rat brain after death. For a uniform sample, this should be independent of the DQ preparation time. For a non-uniform sample, this may not be the case: Short preparation times may preferentially excite DQ coherence in strongly-relaxed spins, which have large DQ relaxation rates. Conversely, long preparation times may preferentially excite DQ coherence in less strongly relaxed spins, which have smaller DQ relaxation rates. Figure 1 shows that there is ample evidence for this effect in the rat brain (figure 1A), where the DQ relaxation rate was six times greater for a preparation time of 1 msec than for a preparation time of 14 msec, unlike the case of a uniform agarose gel (figure 1B; DQ relaxation independent of DQ preparation time). This spectroscopic evidence of non-uniformity may be related to physiological compartmentalization.

A more detailed description of this work forms part of a manuscript [3].

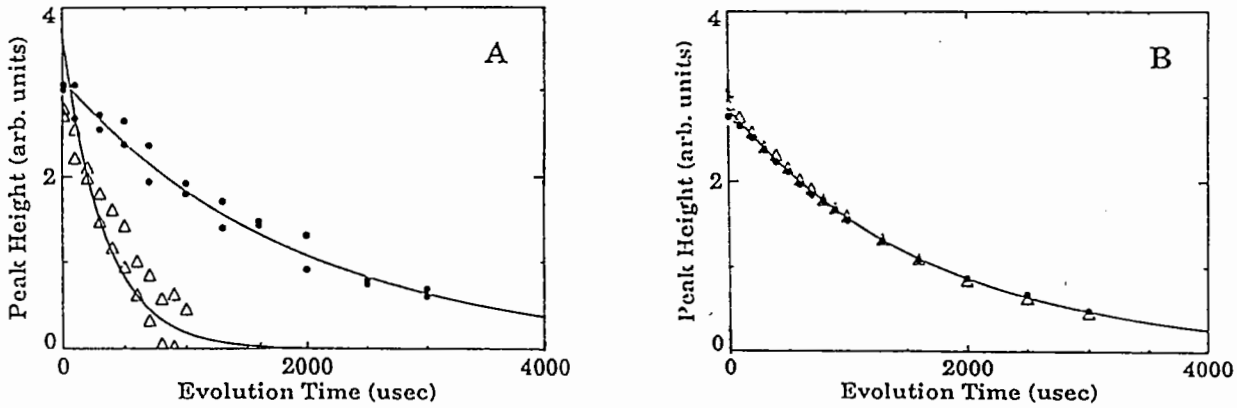

Figure 1. Dependence of the peak height of double-quantum filtered sodium spectra on evolution time, at two different preparation times. A, rat brain in situ after death; B, 10% agarose gel made with 2M NaCl solution. Open triangles, preparation time = 1 msec; closed circles, preparation time = 14 msec.

Best regards,

James Pekar
BEIB, DRS

Robbe C. Lyon
LMMB, NIAAA

Chrit T.W. Moonen
BEIB, DRS

Alan C. McLaughlin
LMMB, NIAAA

---

[1] J Pekar and JS Leigh, Jr., J. Magn. Reson. 69:582 (1986).

[2] G Jacard, S Wimperis, and G Bodenhausen, J. Chem. Phys. 85:6282 (1986).

[3] RC Lyon, J Pekar, CTW Moonen, and AC McLaughlin, Magn. Reson. Med., submitted (1989).

377-70

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Dr. Bernard L. SHAPIRO
TAMU NMR Newsletter
966 Elsinore Court

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N./Réf. 89 12 795 MB/MW
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Wissembourg, le December 13th, 1989

Dear Barry,

Selective transfers can be done using soft pulses in sequences, such as 1D COSY (1) or 1D INADEQUATE (2). Up to now, these experiments have been applied respectively to proton and carbon NMR and it was tempting to test their use at low frequency (16.67 MHz for 183W on an AM 400).

In this purpose, we have run, at 16.67 MHz, a 1D COSY and a SELINADEQUATE sequence on a sample of $Na_7PW_{11}O_{39}$ - 1 M/D_2O. The assignment of the 183W spectrum of this compound (figure 1) was done (3) on a WM 400, some years ago, with a 2D COSY and a 2D INADEQUATE, each of them requiring 30 hours.

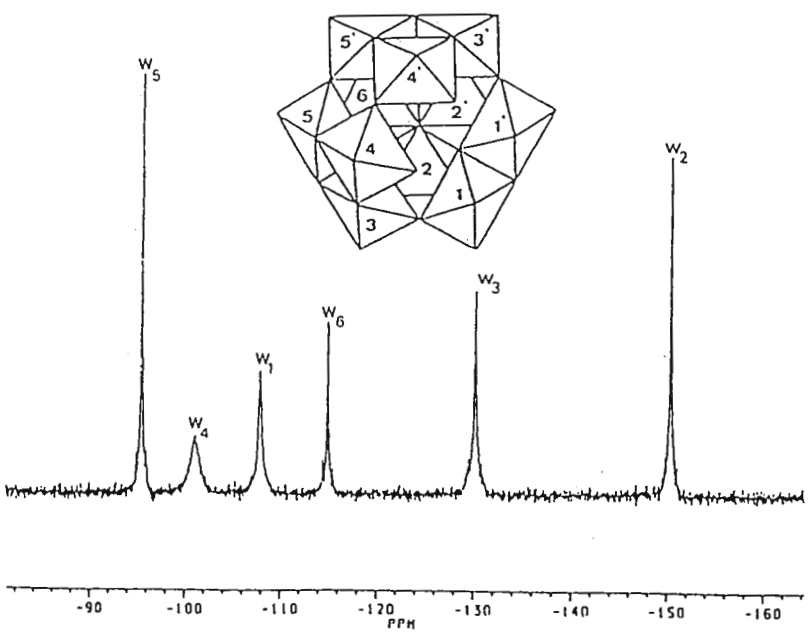
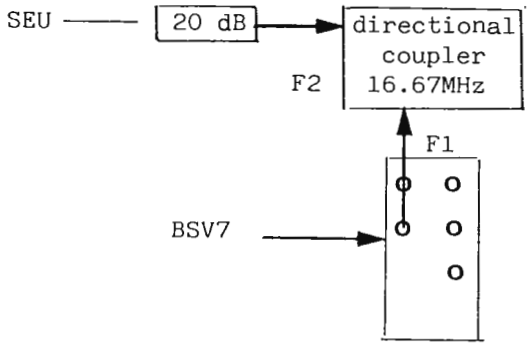


Figure 1 : 1D ^{183}W spectrum

The tungsten spectra were recorded, at 333K, with an AM 400 equipped with a 10mm probe covering a frequency range from 9MHz to 27MHz and a selective excitation unit. The shaped pulse was a gaussian pulse with 512 points, 1 % of truncation and 20 ms of duration. An external attenuation of 20 dB was used between the selective excitation unit and the directional coupler.



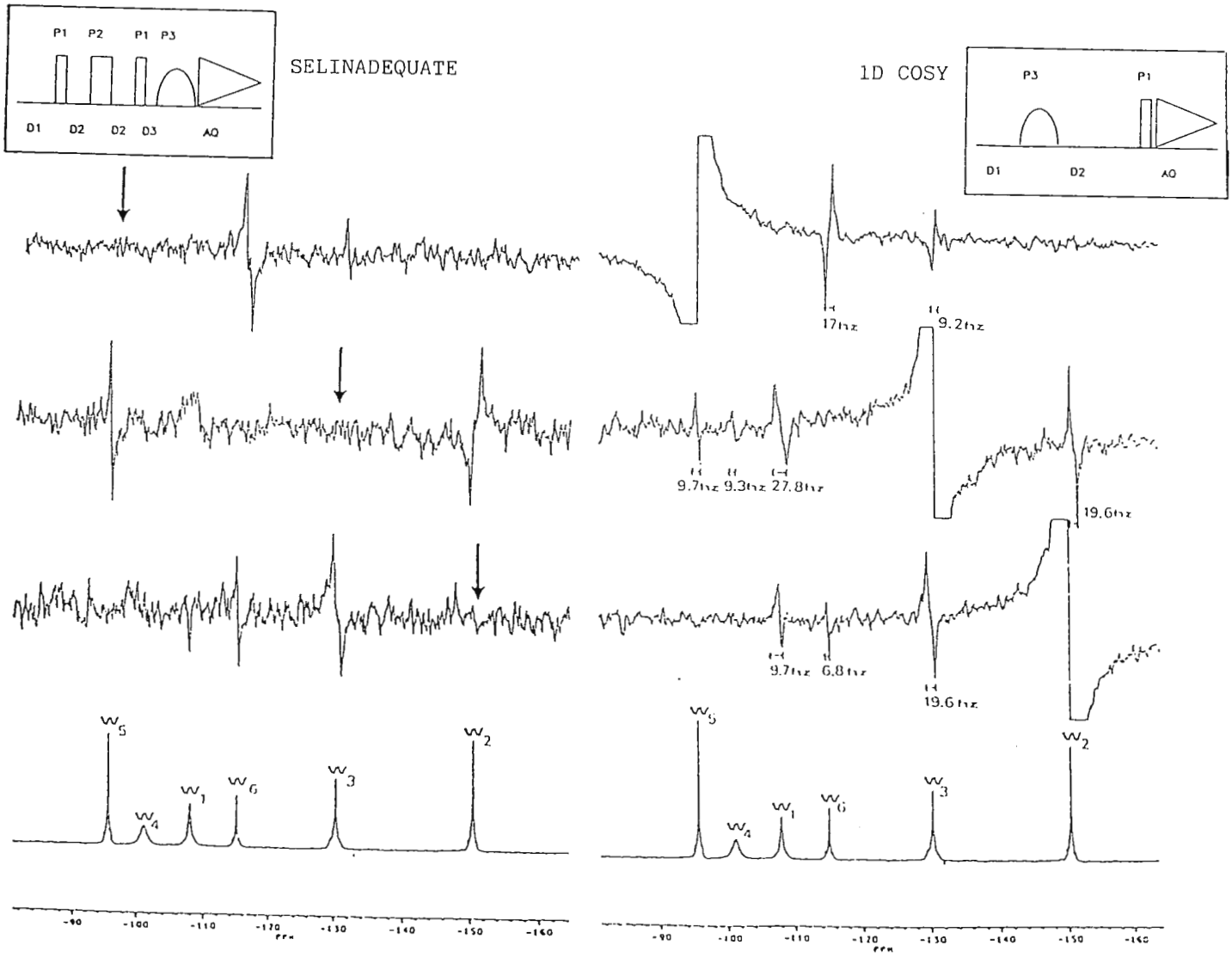


Figure 2 : SELINADEQUATE (P1 = 41 μs, P2 = 82 μs, P3 = 20 ms, D1 + AQ = 1.3s, D2 = 12.5 ms, NS = 15040) et COSY1D (P3 = 20 ms, P1 = 41 μs, D1 + AQ = 1.3s, D2 = 15 ms, NS = 15 000).

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Figure 2 represents the 1D COSY and the SELINADEQUATE with three different frequencies of the soft pulse. The fit between the infomations of couplings and J coupling constants given by the two 2D experiments and the two 1D selective experiments is good. More, from the comparison between the two experimental times (17 hours for each of 1D selective experiments versus 30 hours for each of 2D correlations), it is clear that the selective excitation unit for assignments in heterconuclear NMR is an excellent solution.

M. BOURDONNEAU

Bourdonneau

C. BREVARD

Happy X'mas
christian

Bibliography
1. H. KESSLER, H. OSCHKINAT and C. GRIESINGER, J. Magn. Reson. 70 106 (1986).
2. S. BERGER, Angew. Chem. 9, 1196 (1988).
3. C. BREVARD, R. SCHIMPF, G. TOURNE and C.M. TOURNE, J. Am. Chem. Soc. 105, 7060 (1983).

National Research Council Canada
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Division of Biological Sciences
Division des sciences biologiques

Ottawa, Canada
K1A 0R6

January 9, 1990 (received 1/17/90)

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

Dear Barry,

RE: Actively Switched High Power Transcoupler

For many experiments involving shaped pulses it is necessary to use an actively gated transmit/receive switch (transcoupler) in order to eliminate the nonlinearities inherent in passive diode transcouplers (Figure 1).

The circuit shown in Figure 1 works well for hard pulses but is non-linear at low power levels. One solution is to use high power PIN diode switches, however, diodes capable of handling up to a kilowatt over the NMR frequency range, which also exhibit low capacitance, fast recovery and low attenuation, can be difficult to obtain. Our solution, which was developed by Paul Morris of Morris Instruments Inc. is reproduced here with his permission, and shown in Figure 2.

During the transmitter pulse the TTL gate signal is high which causes a D.C. bias current of about 1 Amp to flow through CR2 and CR4. This allows linear transmission of shaped pulses up to about 25 watts. Above this level the diodes CR1 and CR3 also conduct allowing the transmitted amplitude to increase up to the limit of the diodes (which is about a kilowatt depending on the pulse duration). Some non-linearity is expected in the transition region where CR1 and CR3 begin to turn on but the deviation is on the order of the diode turn-on voltage which is very small (~ 0.6 V) compared to the transmitter amplitude (about 100 V p-p in the transition region). In actual testing we have not seen any distortion which we could attribute to the transcoupler. For operation at high frequency (200 MHz) it was necessary to add the inductor L1 and D.C. blocking capacitor C1 across CR1 and CR2 in order to tune out the capacitance of the diodes to improve the isolation of the circuit.

Yours sincerely,

Tony Wessel — T. Wessel
P. Kozlowski — P. Kozlowski
John — J.K. Saunders
Dan — I.C.P. Smith

Canada

FIGURE 1

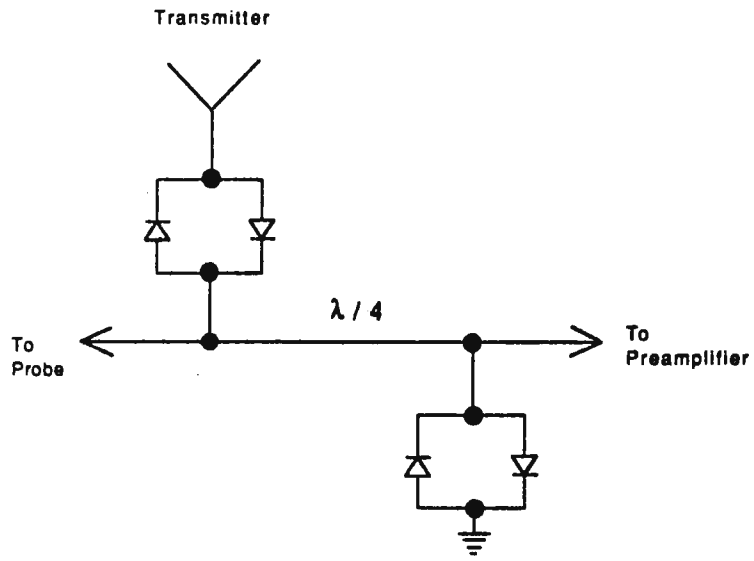
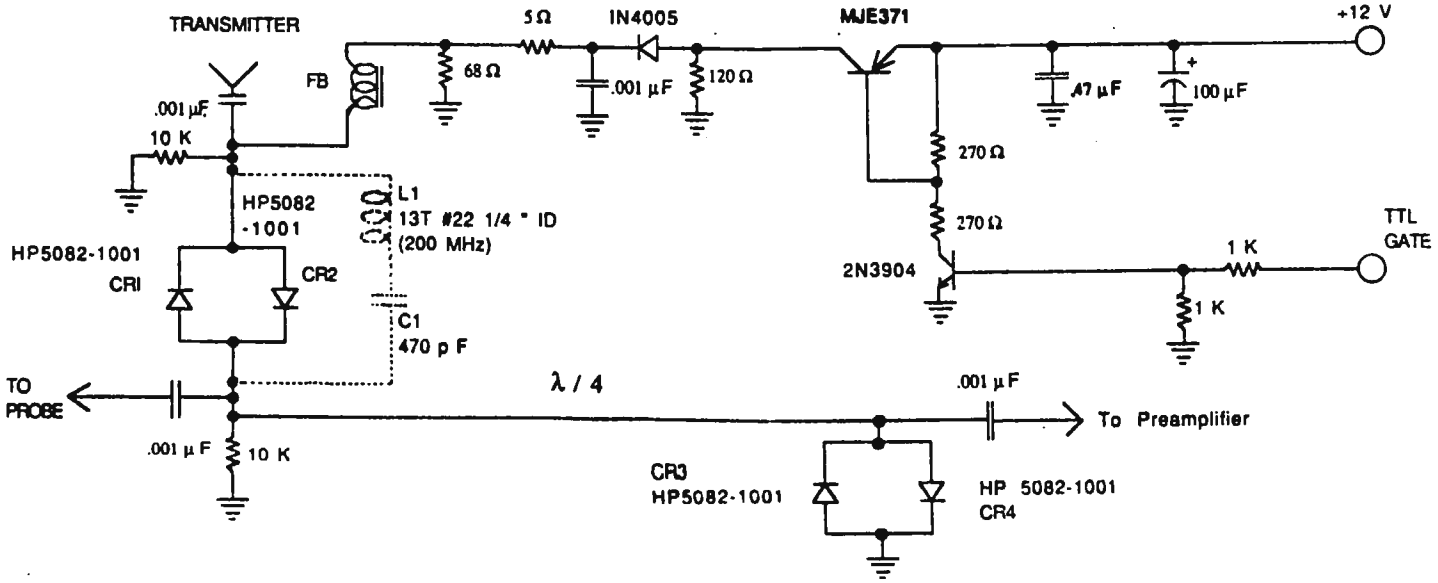


FIGURE 2



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Professor Bernard L. Shapiro
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto, CA 94303

January 9, 1990
(received 1/18/90)

"T_1 Values compiled for Phosphorus Metabolites in Langendorff Perfused Rat Heart"

Dear Professor Shapiro:

Our recent in-vitro P-31 NMR studies on perfused rat hearts have necessitated the compilation of T_1 values of the phosphate metabolites. In searching the literature for this information, we found that only some T_1 values were published for phosphorus resonances of perfused rat hearts at a variety of different field strengths. Therefore we wish to report a complete list of relaxation time measurements of phosphorus metabolites in perfused rat heart which were obtained in our laboratory.

The T_1 values reported below (Table 1) were obtained using a Varian XL-400 vertical wide bore NMR spectrometer with a 20 mm probe tuned to ^{31}P at 161.9 MHz. Isolated rat hearts were perfused in Langendorff mode with Kreb's Henseleit buffer. An intraventricular balloon was used for measurement of the hemodynamic function of the heart. The perfused hearts were immersed in buffer in a 20 mm NMR tube and excess buffer was constantly pumped out.

Shimming was performed on the FID of water and line broadening of 18 Hz was applied prior to Fourier transformation. The T_1 relaxation times were determined with the inversion recovery method. Spectra were obtained using a 57 μs (90°) pulse width, a sweep width of 6711.4 Hz, and relaxation delays ranging from 0.0195 to 20 seconds. A total of 56 transients were collected per spectrum. Representative spectra from the T_1 array are given in Figure 1.

Table 1. P-31 NMR T_1 Relaxation Time Data for Langendorff Perfused Rat Heart

Resonance	T_1 Relaxation Time (sec) (±SEM)
P_i (intracellular)	2.13 ± 0.11
PCr	2.06 ± 0.05
αATP	0.81 ± 0.02
βATP	1.17 ± 0.05
γATP	1.25 ± 0.04
P_i (extracellular)	4.28 ± 0.14

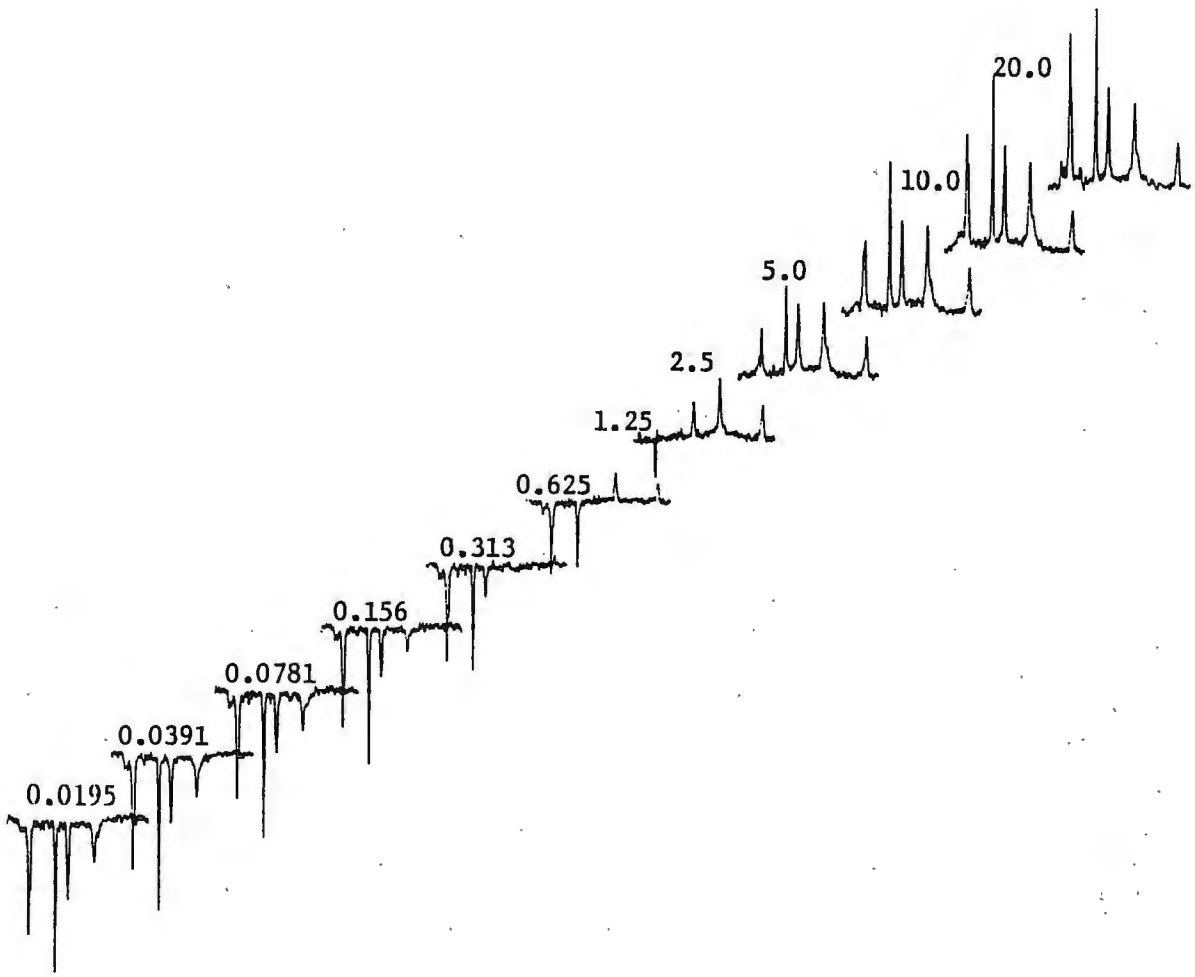

Figure 1. T_1 array: P-31 NMR spectra of Langendorff Perfused Rat Heart with respective relaxation delays (sec).

Since relaxation times are required to correct for changes in integrated intensities which occur during rapid scanning, we feel this information will be of value to other investigators engaged in similar studies with 400 MHz NMR spectrometers.

Sincerely,

Nina C. Gonnella

Nina C. Gonnella

G S Sandhu

Gulzar Sandhu

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Tulane

Department of Chemistry
Tulane University
New Orleans, Louisiana 70118
(504) 865-5573

January 11, 1990
(received 1/18/90)

Dear Barry,

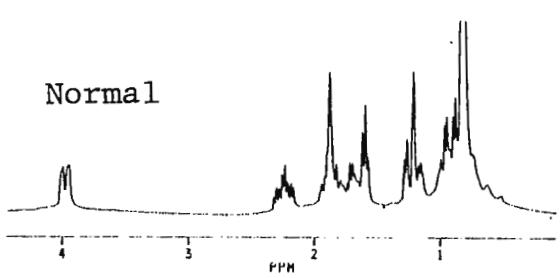
Title: A **More** selective pulse for poor folks

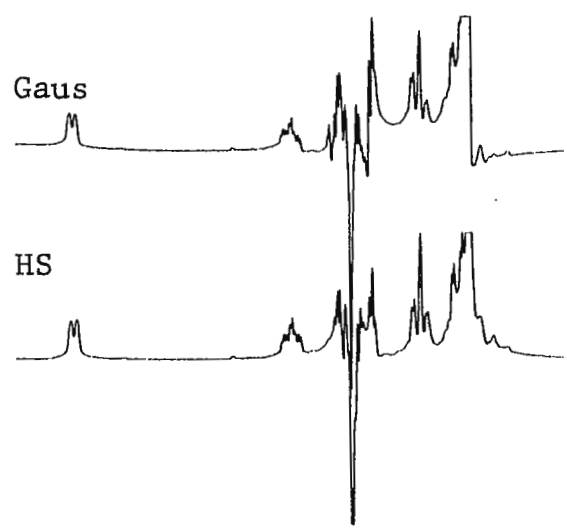
We have been using pseudo-shaped selective pulses that are actually shaped composite pulses. Until recently we have had good success with a shaped Gaussian pulse, but now we have found something even better: a composite hyperbolic secant pulse (HS). This pulse has the amplitude modulation similar to the gaussian, but with phase modulation as well. The result is extremely rectangular excitation. The width of the pulse can be controlled to some extent by changing the delay between the composite pulses (and changing the total pulse time). We have been able to implement this pulse in a 64 component version on our Bruker AC-200 which is equipped with a process controller, but the 128 component version will not fit into the controller's memory.

One limitation of the HS pulse is that it is only useful for a 180° inversion pulse, and not other angles. To test its effectiveness we created an experiment where our sample (borneol) was irradiated with the HS pulse from the decoupler, and then a hard 90° pulse from the transmitter, (keeping the frequency sources for the two components coherent.)

As you can see from the results below, the HS pulse definitely has fewer effects outside its proscribed width. The Gaussian pulse creates phase distortions up to 60 Hz from the irradiation frequency, while the only noticeable effects of the HS pulse appears to create only amplitude distortions. In the experiments below we used 1 ms delays between pulses for both experiments. We are now planning to incorporate this pulse into other experiments.

Because this "pulse" is so long (nearly one whole precious page) I have decided to distribute the actual microprogram via INTERNET/BITNET or mail. If the recipient has managed to link his or her Bruker to their mail computer then it will also be possible to avoid typing errors by downloading directly.





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AW JL CA

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ANALYTICAL AND ENVIRONMENTAL RESEARCH

January 12, 1990
(received 1/14/90)

Earthquake Preparedness

Dear Barry,

Many of us in California have taken some action to prevent damage during the earthquakes which are known to occur in this vicinity. In this laboratory, we have attached our superconducting magnets to the floor using three to five Z shaped brackets which prevents the magnet base from moving more than 5 mm in any direction. The brackets are secured to the floor using 3/8"x1" bolts in lead anchors and mounted with 3-5 mm clearance between the bracket and the magnet base thus preventing floor vibrations being transmitted to the magnet. These brackets are best installed before the magnet is energized. On October 17, 1989 at 1704, we had the opportunity to test the reliability and effectiveness of these restraints during a Richter Magnitude 7.1 earthquake with an epicenter less than thirty miles away. The accelerations in the laboratory were sufficient to chip the paint where the brackets made contact with the magnet bases during the shaking and cause the three bolts that connect the Dewar to the magnet base of the AM-500 to be pulled out of the aluminum alloy Dewar bottom. The magnet rotated off the base axis about 15° coming to rest about 20 mm off center. I believe that these restraints prevented more serious damage and certainly kept the Dewars from overturning. The WM-300 magnet, having a lower center of gravity and somewhat stronger connections between the Dewar and base, was undamaged.

During the shaking, the sample changer on the AM-500 was observed to move through an arc of more than 30° and afterward was more then 30 mm out of alignment. The spectrometer consoles also moved significantly; the WM-300 console lifted enough to come to rest on top of the cables connecting the console to the magnet. The CDC disk in the WM-300 console suffered a head crash and we now have that disk platter mounted in a memorial frame. Prior to the installation of our AM-500, we had the WM-300 magnet connected to the ceiling by ropes. Our engineering staff suggested that the floor brackets would be sufficient and less expensive. The brackets are quite unobtrusive and have prevented a recurrence of the Tour Guides telling visitors that the magnets are suspended from the ceiling because the floors are not strong enough to carry the weight.

After the quake, Tim Kelly of the San Jose Bruker Office and I were able to recenter the AM500 Dewar on its base by using a wooden lever and without discharging the magnet. We then installed 30 mm (thread length) bolts in each of the holes for connecting the magnet to the base replacing the original 25 mm long bolts. Users in regions subject to earthquakes should ensure that all of the bolts are installed, of sufficient length to provide adequate strength and torqued to specifications. I also recommend the use of bronze or other non-magnetic tools for working near energized magnets.

Both Bruker and Varian generously offered their support in our putting the NMR laboratory back into full operation and I would especially like to thank Dr. Mark O'Neil-Johnson and Tim Kelly of Bruker and Dr. Jim Shoolery of Varian for their assistance.

Sincerely yours,

Michael L. Maddox

UNIVERSITY OF CALIFORNIA, SANTA BARBARA

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DEPARTMENT OF CHEMISTRY SANTA BARBARA, CALIFORNIA 93106

12 January 1990 (received 1/19/90)

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

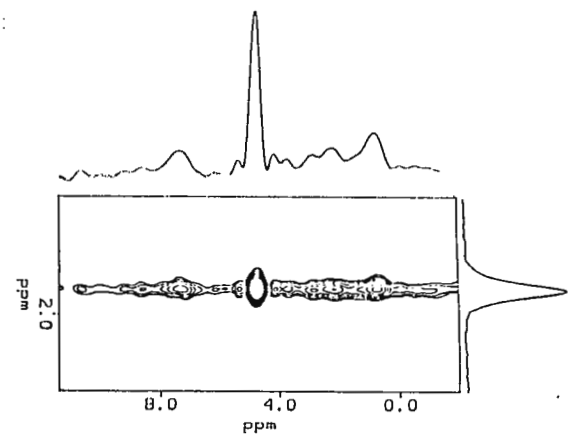
Dear Barry,

TWO-DIMENSIONAL TRITIUM-PROTON NOEs IN PROTEINS

Tritium nmr spectra from isotopically labeled materials are useful for addressing many questions in the small molecule context but there have been few examples of tritium nmr studies with protein systems. We have found that observation of tritium signals from a 25 kDal protein specifically labeled with tritium is remarkably facile and have used two-dimensional $^3H\{^1H\}$ Overhauser effects to examine the local environment of the tritium-containing reporter group.

The enzyme chymotrypsin reacts stoichiometrically with tosyl fluoride to give an inactive protein. In the present work tritiated tosyl fluoride was prepared by catalytic reduction of 4-(dichloromethyl)benzenesulfonyl fluoride with 10% Pd on carbon and 100% T_2 gas. Treatment of chymotrypsin with this material produced inactive enzyme which, after extensive dialysis to replace the reaction medium with a deuterated solvent mixture, was examined by tritium nmr at 320 MHz.

Two-dimensional $^3H\{^1H\}$ NOESY spectra with phase sensitivity in both dimensions were obtained using a standard pulse sequence. A number of cross peaks were observed and their relative intensities as a function of mixing time were examined. The Figure to the right shows some of our results. The mixing time dependences observed indicate that the tritons of the labeled protein are relaxed by direct dipolar interactions with protons having shifts of 2.21, 7.09, 3.72, and 7.78 ppm. The first two groups of protons reside on the tosyl group and correspond to the residual ^1H in the tosyl methyl group and the protons of the tosyl aromatic ring, respectively. Residual protons in the solvent also contribute appreciably to relaxation of the tritons as the intensity of the cross peak at 4.76 ppm varied with the isotopic composition of the solvent. We are attempting to account for these and related observations of [^3H]-tosylchymotrypsin by the construction of a theoretical model of the structure and dynamics in the vicinity of the tosyl group in the modified enzyme.



Phase-sensitive 3H{1H} 2D-NOE spectrum of [3H]-tosylchymotrypsin obtained at 320 MHz and 25° with a mixing time of 600 ms. The protein concentration was 1 mM and the estimated total tritium concentration was 1.4 mM. Data for 64 t1 increments (416 scans each) were processed with FTNMR. Projections to the proton and tritium axes are shown; the proton shift axis appears horizontally.

Sincerely,

Tom

T. M. O'Connell P. G. Williams* J. T. Gerig

*National Tritium Labeling Facility, Lawrence Berkeley Laboratory

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January 17, 1990
(received 1/20/90)

Physikalisches Institut, Morgenstelle, D-7400 Tübingen

Prof. Dr. Bernhard L. Shapiro
Editor TAMU NMR Newsletter
966 Elsinore Court
Palo Alto, California 94303, U.S.A.

Localised Proton NMR Spectroscopy with a 1.5 T Whole Body Imager

Dear Barry,

we have proceeded in volume selective proton NMR spectroscopy with a 1.5 T whole body imager using CODEX and double spin echo sequences. One of our studies had the aim to examine the short- and long-term stability of the methods and the imager. For this we observed the multiplet of the CH_2-signal of ethanol and found the splitting given in the figure. The resolution is better than $3 \cdot 10^{-8}$ and is obviously sufficient for the requirements of high resolution localised in vivo spectroscopy. Almost the same resolution and position of the pattern were observed for individual runs lasting seconds or some minutes in the course of hours.

Sincerely

Otto Lutz Wulf-Ingo Jung Karin Müller Markus Pfeffer

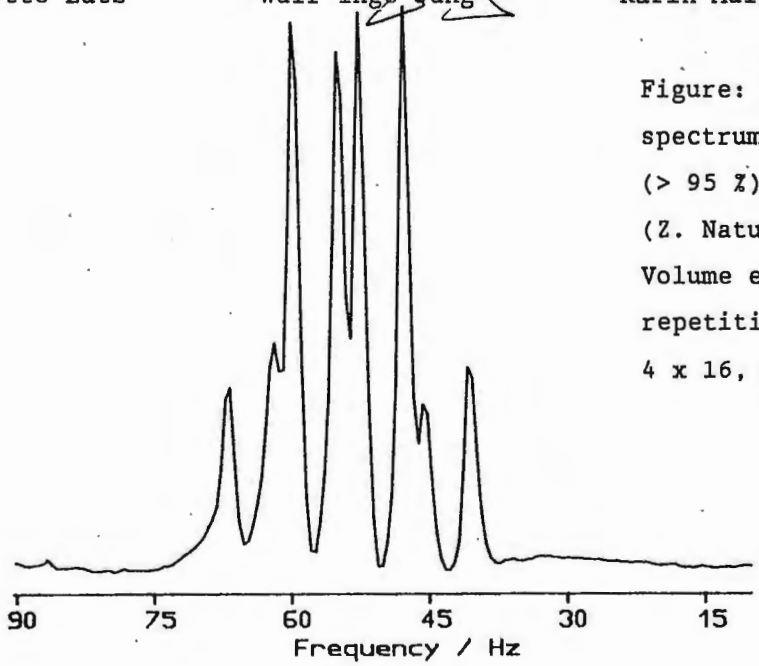

Figure: CH_2-part of the ^1H NMR spectrum from a 1 l bottle of ethanol (> 95 %). Volume selection by CODEX (Z. Naturforsch. 43a, 909 (1988)). Volume element: (13x13x13) mm^3, repetition time: 12 s, acquisitions: 4 x 16, measuring time: 13 min.

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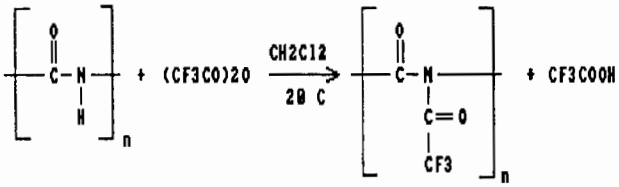
Dr. B.L. Shapiro
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto, CA 94303
U.S.A.

December 4, 1989
(received 12/19/89)

Solution NMR of Triflouroacetylated NYLONS

Dear Dr. Shapiro

Polyamides (NYLONS) are very important synthetic polymers. The solution NMR analysis of these materials is complicated by the fact that they are insoluble in the common deuterated NMR solvents. Common NYLON solvents include formic acid, m-cresol, and hexaflouroisopropanol.We have been employing the method of triflouroacetylation[1] (equation 1) to solubilize nylon polymers for compositional analysis by NMR.



Samples are prepared by mixing approx. 100mg of polymer with 0.5ml of triflouroacetic anhydride in 2.0ml of CH_2Cl_2 in a dry closed vial. The mixture is allowed to stand overnight to solubilize the polymer. After the polymer is solubilized the excess TFAA along with the CH_2Cl_2 as well as triflouroacetic acid (a product of the triflouroacetylation reaction) are removed by passing a stream of dry nitrogen through the vial. The resulting triflouroacetylated polymer is redissolved in CD_2Cl_2.

The ^{13}C and ^{1}H NMR spectra of a triflouroacetylated NYLON 6/66 copolymer are shown in Figure 1. The spectra were acquired on a Bruker AM-400 spectrometer. Part A is the ^{13}C NMR spectrum of the NYLON copolymer. The peaks can be assigned as indicated in the figure. The resonances due to the triflouroacetyl group can be identified by their distinctive couplings to the ^{19}F nuclei. We are currently evaluating the quantitative reliability of the spectra acquired using this method for use as a tool for measuring NYLON composition.

Yours Sincerely

Mike McKinnon

Michael McKinnon

1. E.Jacobi, H. Schuttenberg, R.C. Schultz Makromol.Chem.,rapid Commun., 1, 397 (1980)

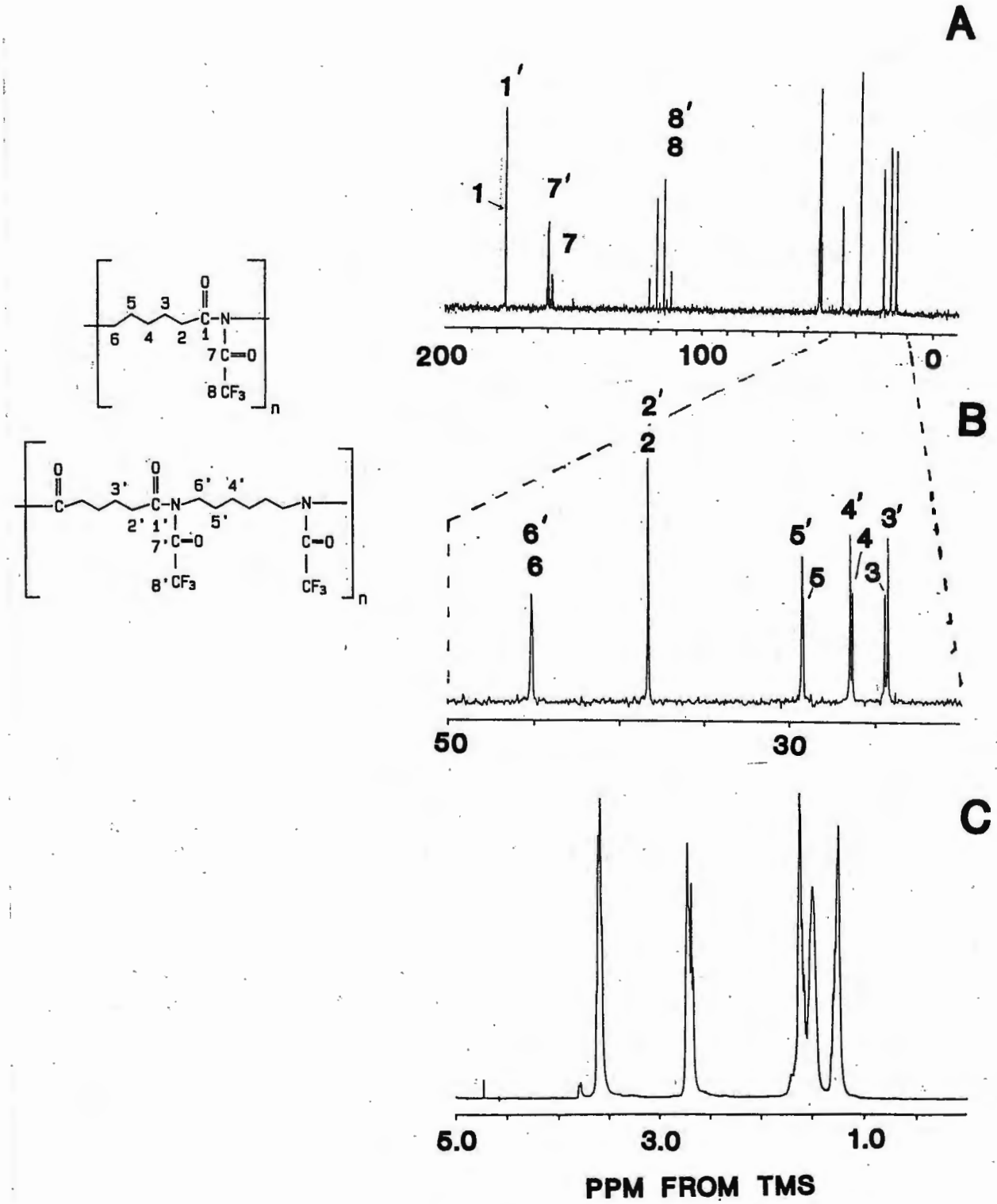


FIGURE 1. A) ^{13}C solution NMR spectrum of Triflouroacetylated NYLON 6/66 copolymer (20 wt% in CD_2Cl_2). B) Expansion of the 20-50 PPM region of spectrum in A). C) ^{1}H NMR spectrum of sample A).

Continued from page 1:

FORTHCOMING NMR MEETINGS

Workshop "NMR and Structure of Biomolecules", **March 4-9, 1990**; Gainesville, Florida; Contact: W. S. Brey, Univ. of Florida, - see Newsletter 375, 68.

Workshop on In Vivo Magnetic Resonance Spectroscopy III, **March 29 - April 1, 1990**, San Francisco, CA.; Contact: Dr. M. W. Weiner or Dr. G. B. Matson, Magnetic Resonance Unit, Veterans Administration Medical Center, 4150 Clement Street (11D), San Francisco, CA 94121; (415) 750-2146.

31st ENC (Experimental NMR Conference), **April 1-5, 1990**; Asilomar Conference Center, Pacific Grove, California; Contact: ENC, 750 Audubon, East Lansing, MI 48823; (517) 332-3667; Attendance: 1,200.

Frontiers of Polymer Characterization by NMR Spectroscopy, Symposium at the American Chemical Society National Meeting, **April 22-27, 1990**, Boston, Mass.; See Newsletter 372, 54.

10th European Experimental NMR Conference, **May 28 - June 1, 1990**; Veldhoven, The Netherlands. Contact: M. J. A. de Bie: see Newsletter 376, 26.

Workshop of Special Topics in Medical Magnetic Resonance, sponsored by the Society of Magnetic Resonance in Medicine and the National Research Council of Canada, **July 23-27, 1990**; Whistler Mountain, BC, Canada. Contact: L. Forget - see Newsletter 374, 46.

Expanding Frontiers in Polypeptide and Protein Structural Research, sponsored by the National Research Council of Canada, **July 23-27, 1990**; Whistler Mountain, BC, Canada. Contact: L. Forget - see Newsletter 374, 46.

Tenth International Biophysics Conference, sponsored by the International Union of Pure and Applied Biophysics and the National Research Council of Canada, **July 29 - August 3, 1990**; Vancouver, BC, Canada. Contact: L. Forget - see Newsletter 374, 46.

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

Additional listings of meetings, etc., are invited.

All Newsletter Correspondence

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DEADLINE DATES

No. 379 (April)---------- 16 March 1990
No. 380 (May)---------- 20 April 1990
No. 381 (June) ---------- 18 May 1990
No. 382 (July) ----------20 June 1990

TEXAS A&M UNIVERSITY NMR NEWSLETTER

No. 377
February 1990

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CSI 2T Applications

Shielded Gradients: Theory and Design

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

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

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

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

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

Fig. 1a.

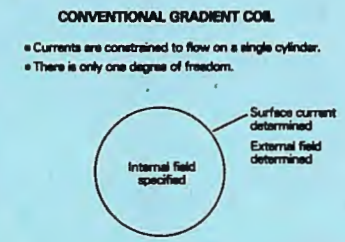


Fig. 1b.

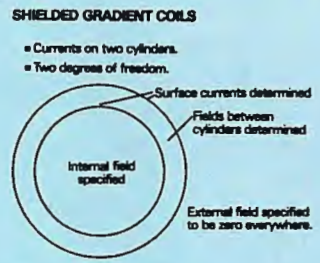


Fig. 1a—Design problem for unshielded gradients. The field inside the winding is specified to be a linear gradient and the current pattern on the cylinder is determined. Fig. 1b—Design arrangement for shielded gradients. The field inside the inner cylinder is specified to be a linear gradient and the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

Fig. 2.

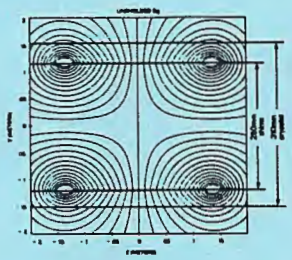
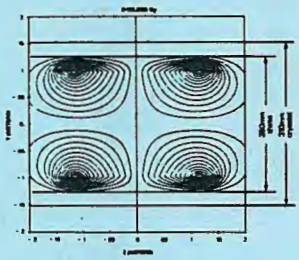


Fig. 3.



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

Fig. 4a.

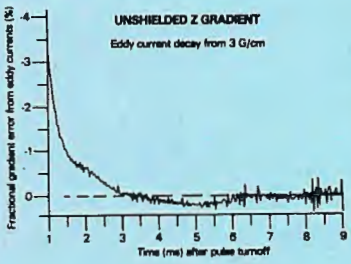


Fig. 4b.

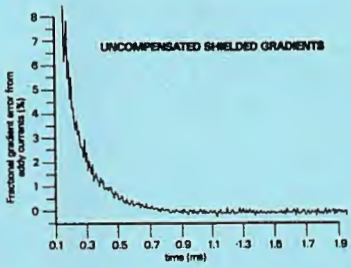
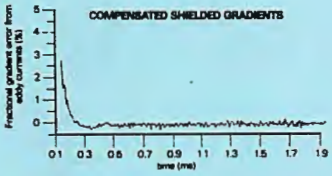


Fig. 4c.



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

GE NMR Instruments

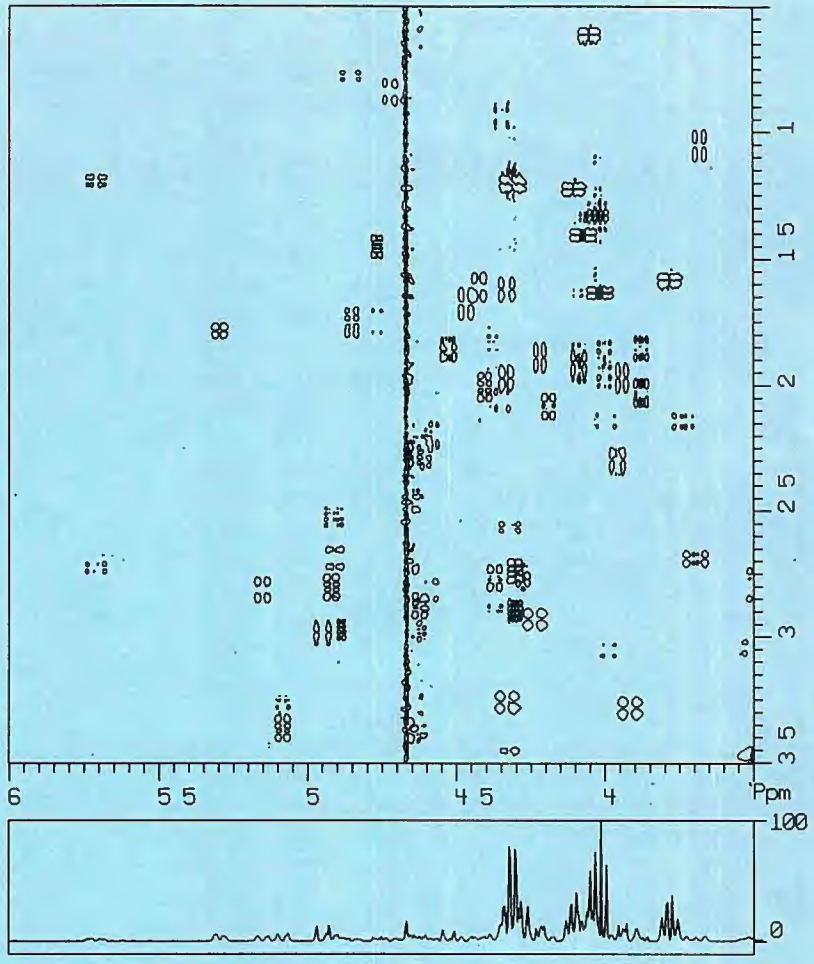
VPLX

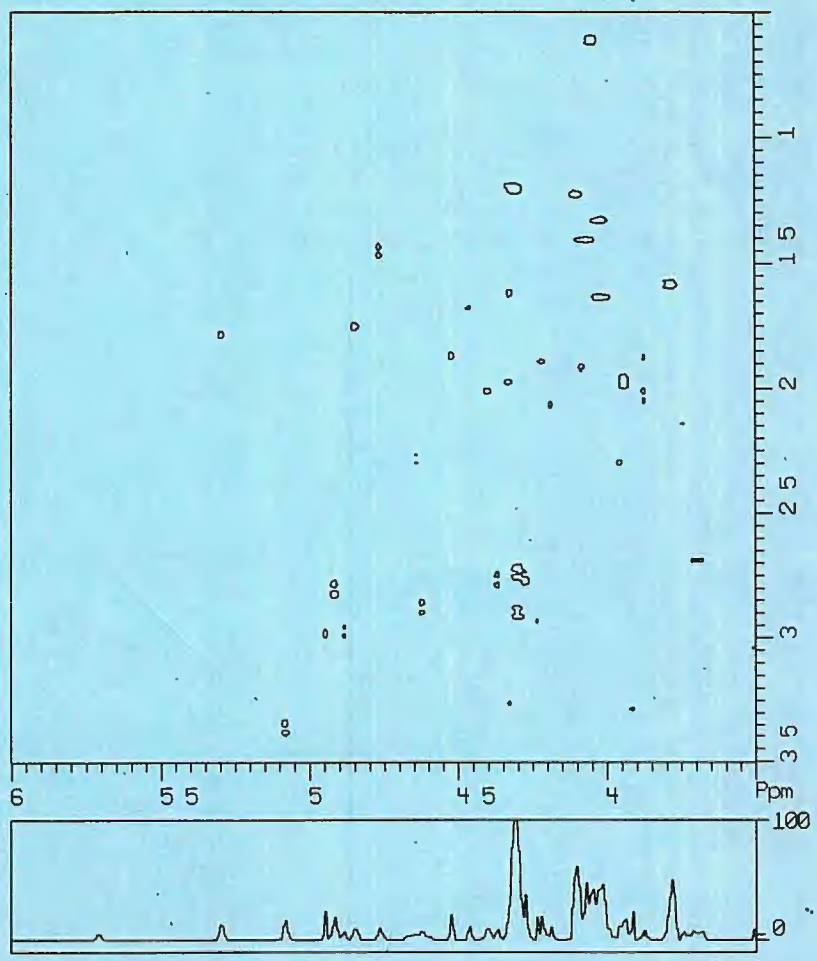
A GSX UPGRADE

JEOL USA introduces the VPLX data processing package, our latest upgrade to the GX and GSX NMR spectrometers. When used with the latest network options of Multi-PLEXUS, VPLX provides the power and speed of a VAX™ and eliminates the need to learn a new set of software commands.

In addition to allowing for off-line processing, VPLX offers advanced functionality such as MEM/LPZ and Symmetry Filtering. The top data shows the normal NH to alpha region in a double quantum filtered COSY of BPTI in water. This matrix was produced on a GSX-400, processed on VPLX, and printed on a laser printer. The bottom data is identical to the first with the exception that a symmetry filter has been applied to the matrix. This symmetry filter discriminates on the basis of the known phase relationship of true COSY peaks. Each of the COSY peaks that passes through the filter is reduced to a centroid representation.** This filtering allows for the rapid elimination of spurious cross peaks and is the first step necessary for computer based spectral interpretation.

For more information, contact JEOL.





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**JC Hoch, S Hengyi, M Kjaer, S Ludvigsen, and FM Pousen, "Symmetry Recognition Applied to Two-Dimensional NMR Data", Carlsberg Res., Commun., Vol. 52, p.111, (1987).