CHEMISTRY
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Supporting Information

1,3-Diindolylureas and 1,3-diindolylthioureas: anion complexation studies in solution and the solid state.

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Supplementary information

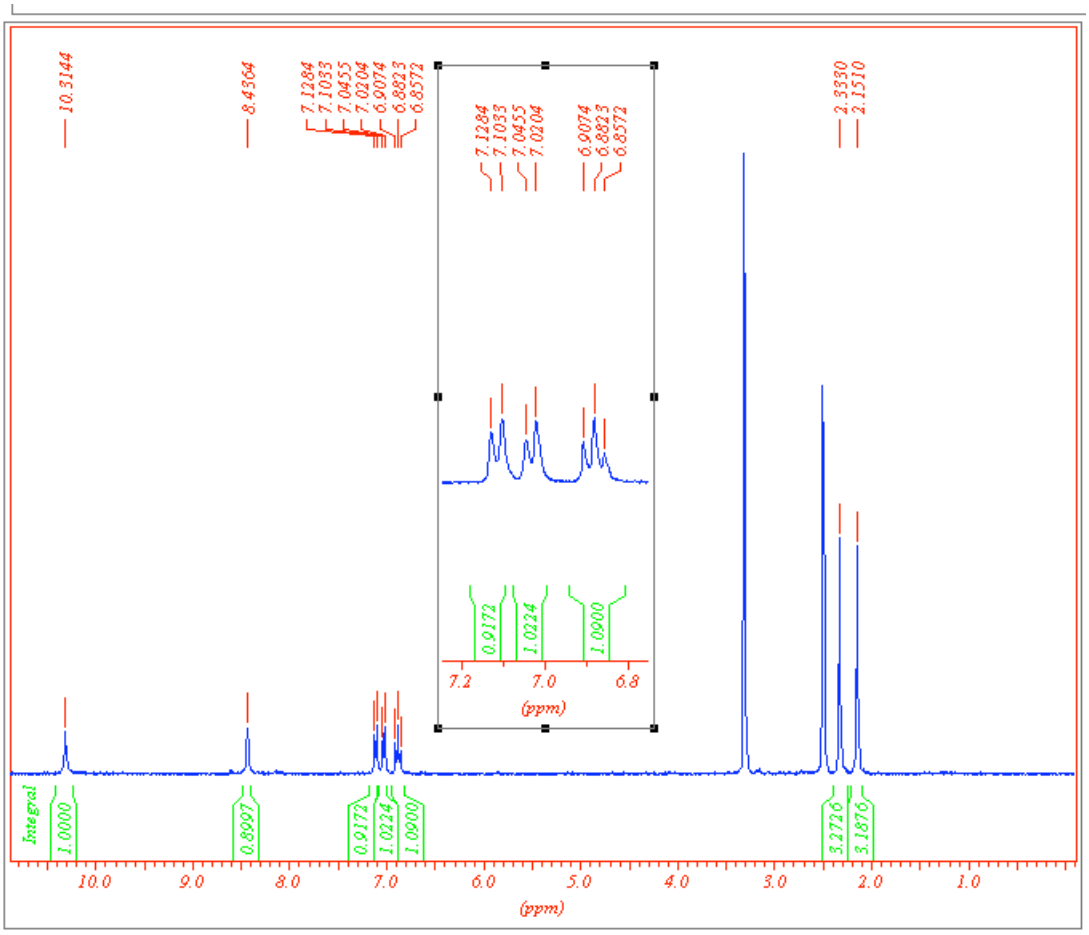


Figure S1 ^{1}H NMR spectrum of compound **1** in [D_6]DMSO.

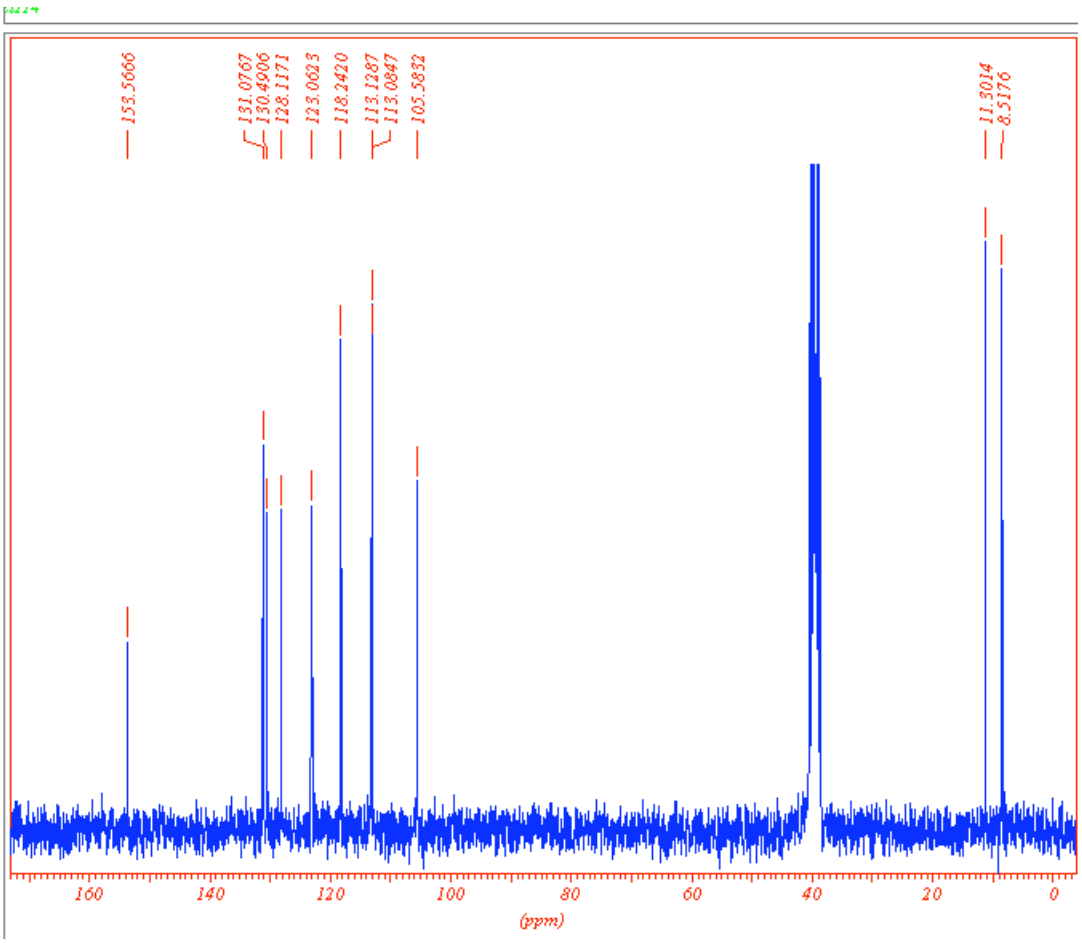


Figure S2 ^{13}C NMR spectrum of compound **1** in [D_6]DMSO.

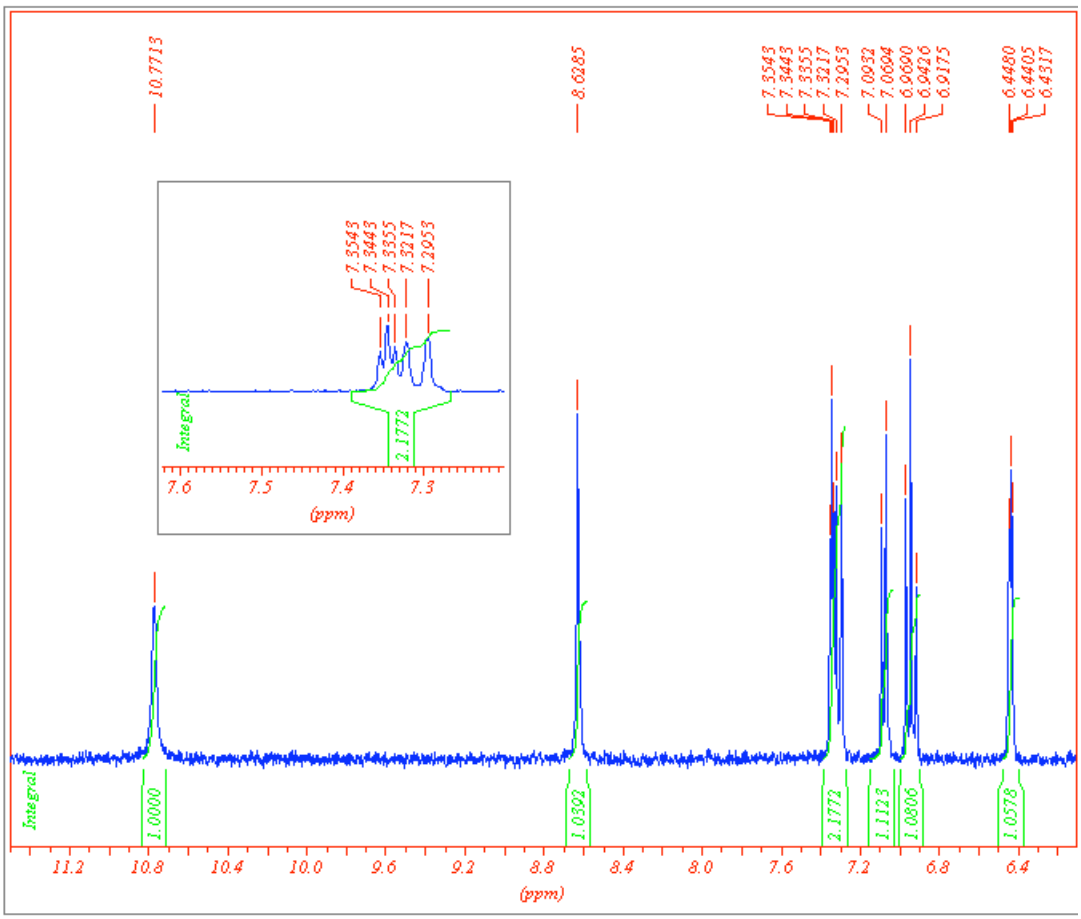


Figure S3 ^{1}H NMR spectrum of compound **2** in [D_6]DMSO.

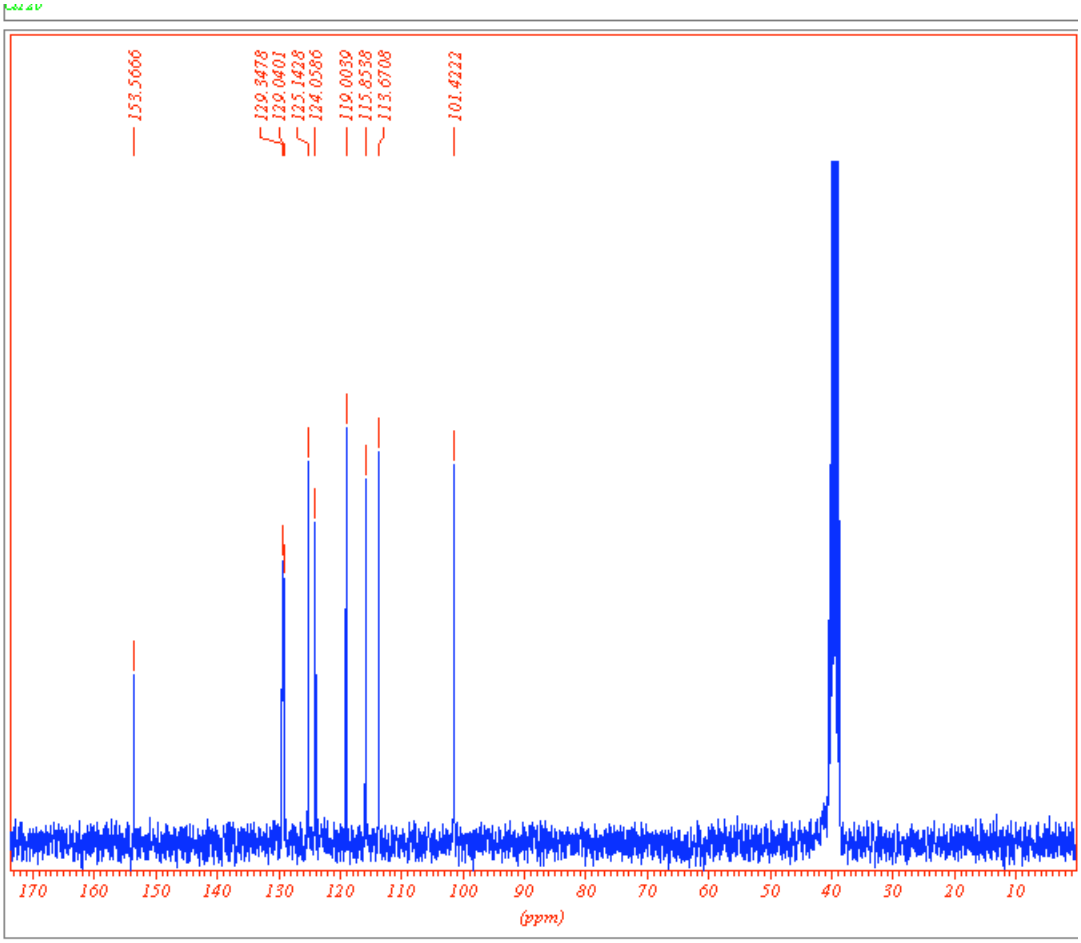


Figure S4 ^{13}C NMR spectrum of compound **2** in [D_6]DMSO.

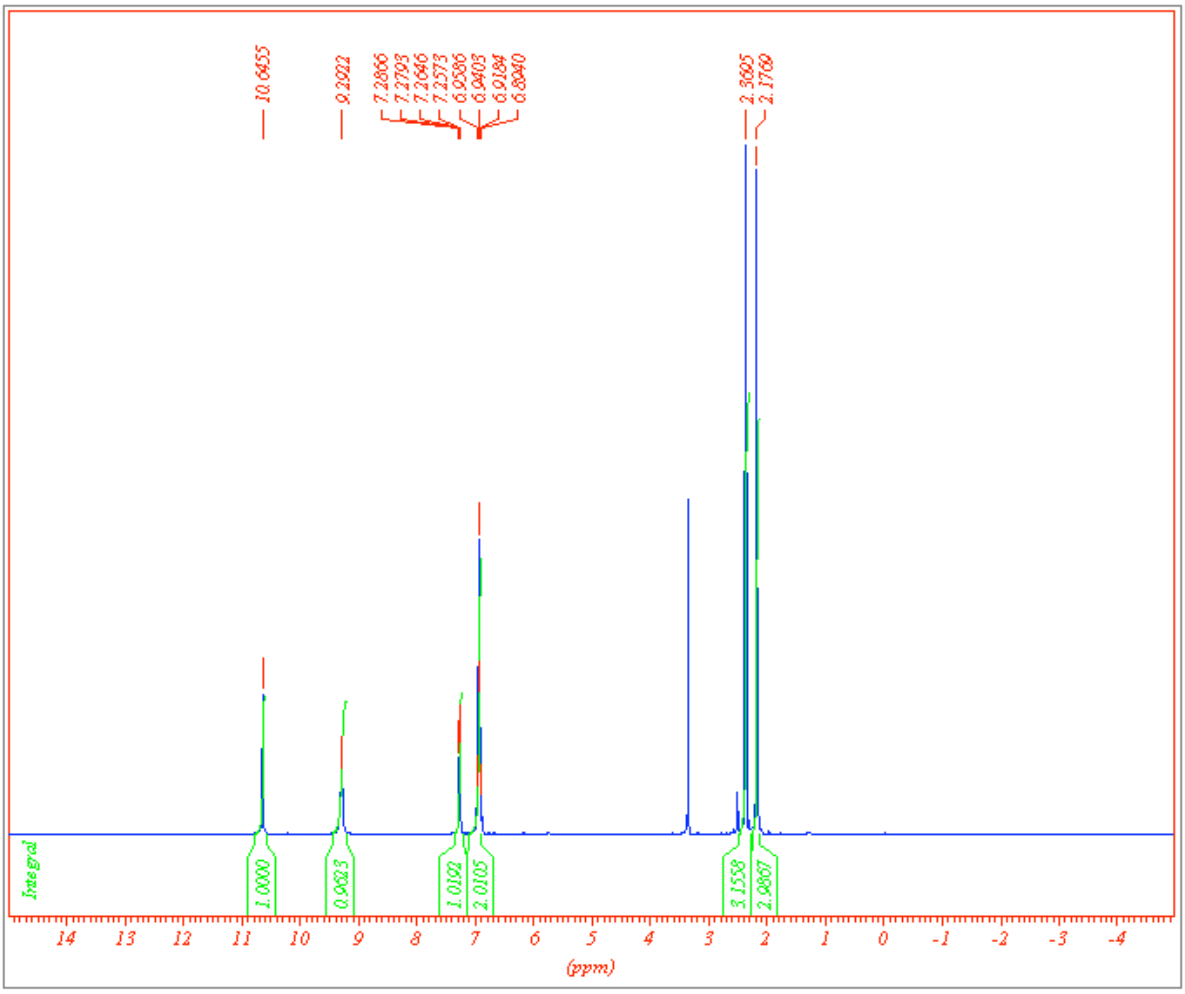


Figure S5 ^{1}H NMR spectrum of compound **3** in [D_6]DMSO.

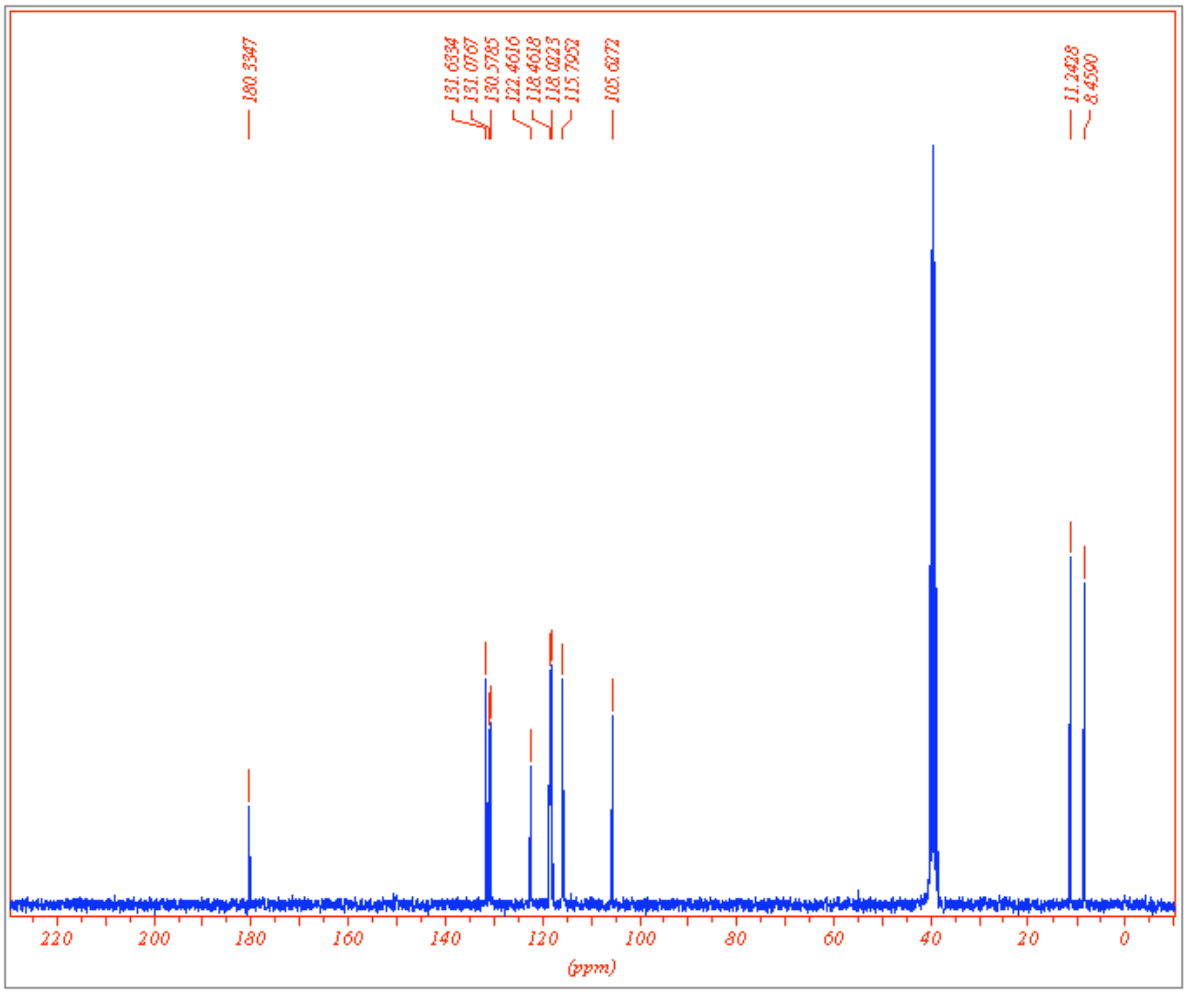


Figure S6 ^{13}C NMR spectrum of compound **3** in [D_6]DMSO.

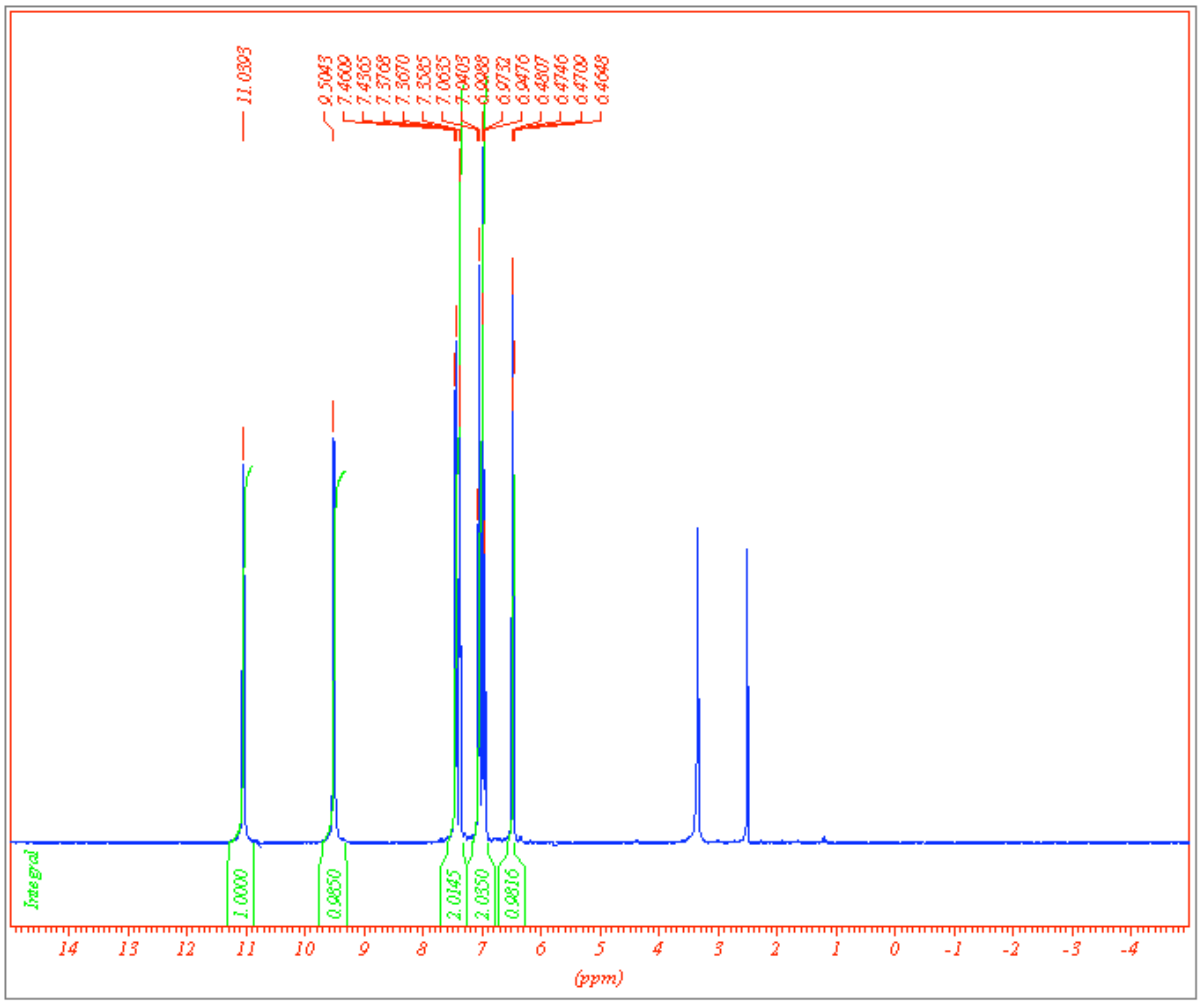


Figure S7 ^{1}H NMR spectrum of compound **4** in [D_6]DMSO.

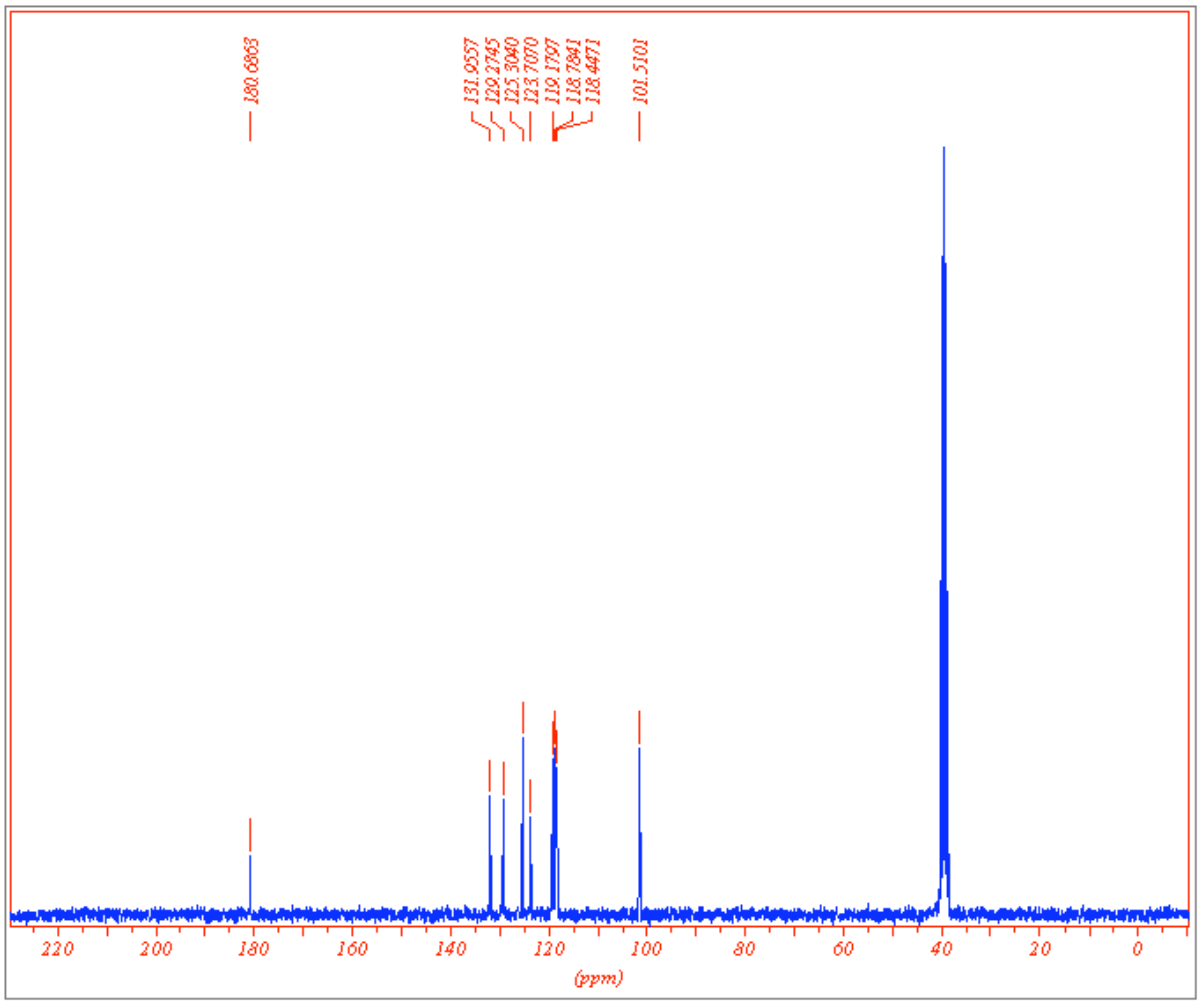


Figure S8 ^{13}C NMR spectrum of compound **4** in [D_6]DMSO.

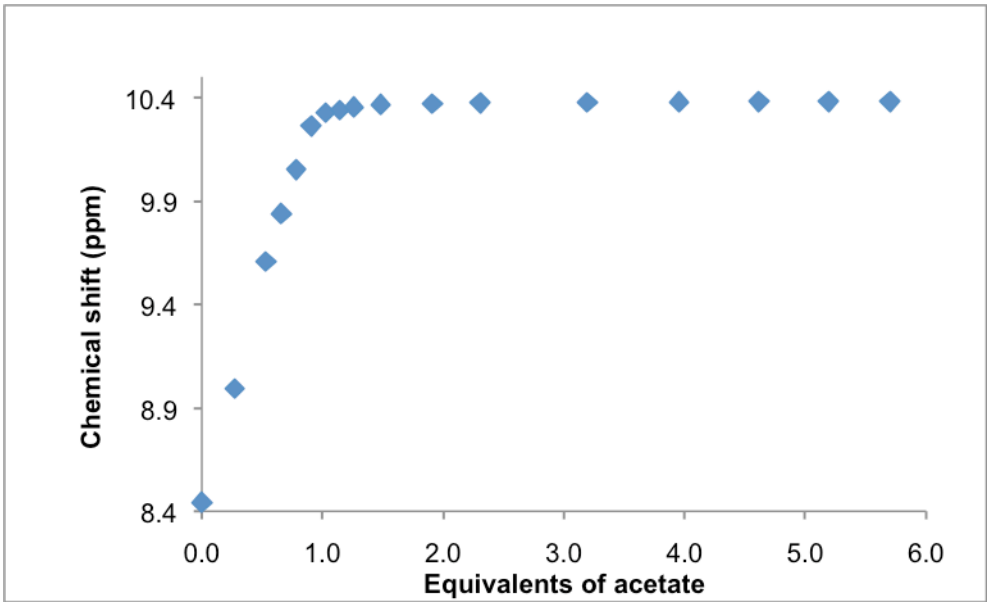


Figure S9 NMR titration of compound **1** *vs*. TBAOAc in [D_6]DMSO/H_2O 0.5%.

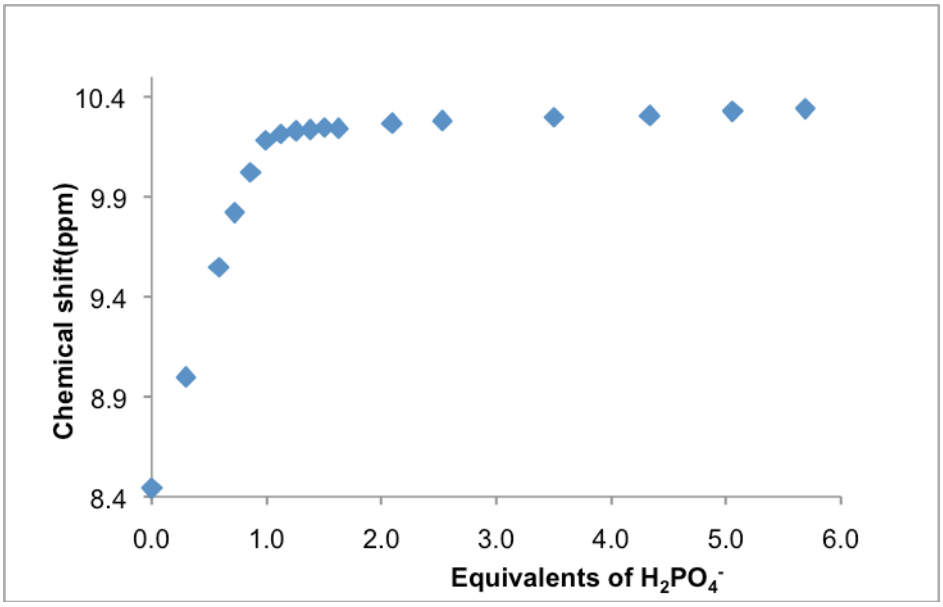
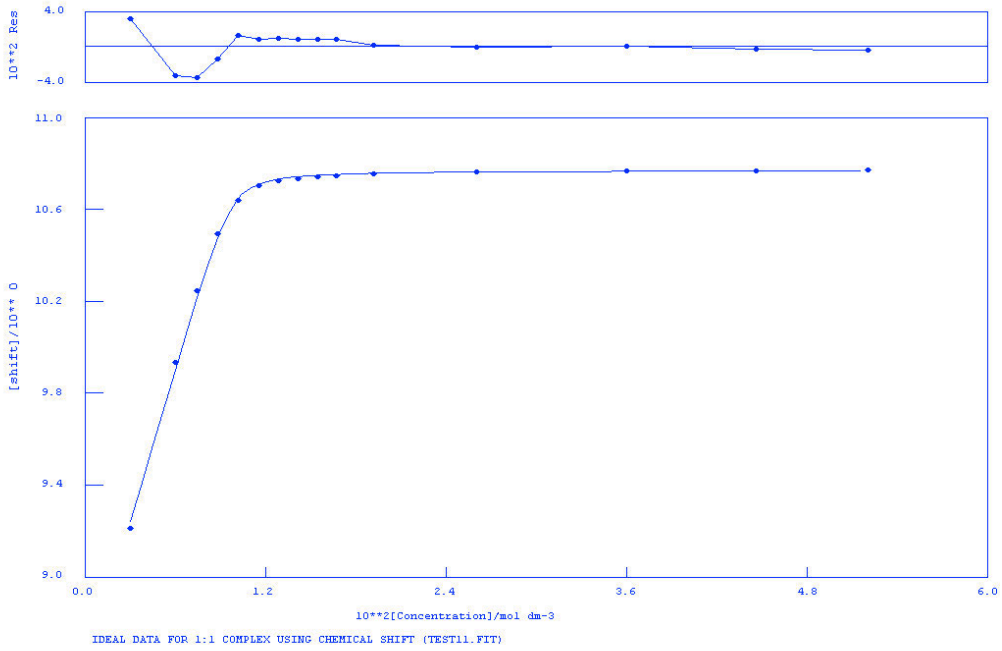


Figure S10 NMR titration of compound **1** *vs*. TBAH_2PO_4 in [D_6]DMSO/H_2O 0.5%.

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 File prepared by M. J. Hynes, October 22 2000

NO.  A   PARAMETER    DELTA     ERROR    CONDITION   DESCRIPTION
   1  1  1.70477E+04 2.000E-01 2.258E+03 1.285E+00    K1
   2  1  8.54729E+00 2.000E-01 2.291E-02 1.100E+00   SHIFT M
   3  1  1.07690E+01 1.000E+00 6.087E-03 1.370E+00    SHIFT ML

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 RESIDUALS SQUARED = 4.06E-03
 RFACTOR =     0.1561 PERCENT
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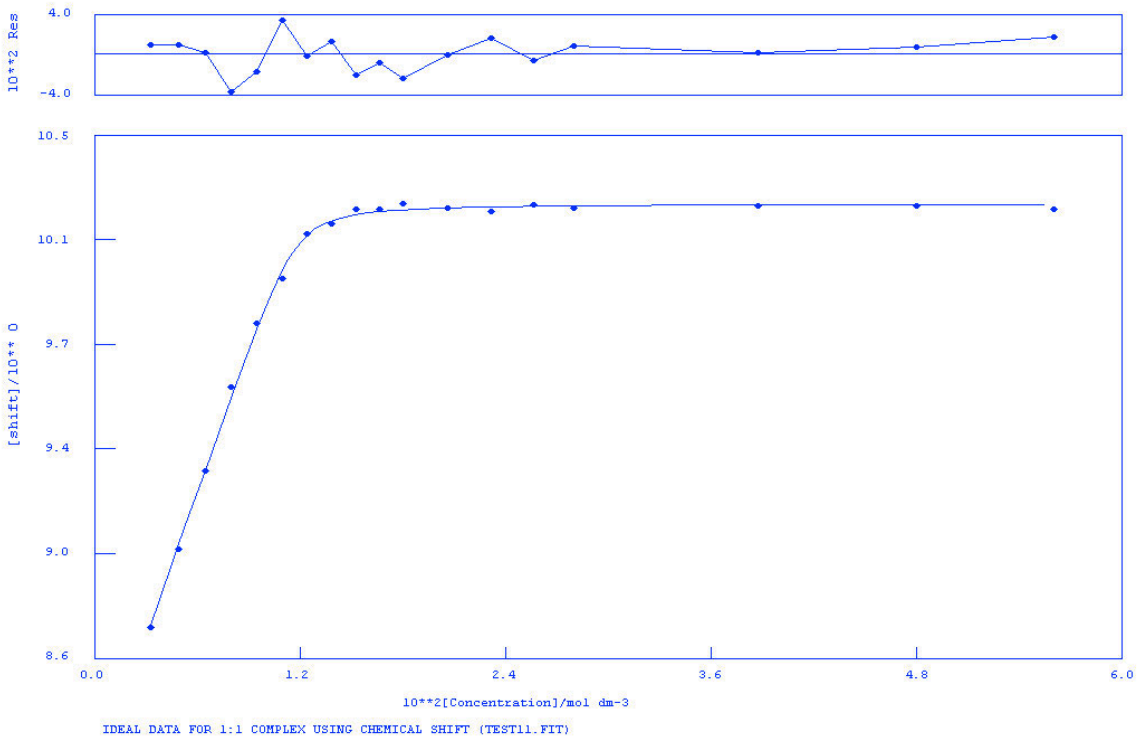
Figure S11 NMR titration of compound **1** *vs*. TBAOBz in [D_6]DMSO/H_2O 0.5%.

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 IDEAL DATA: K1 = 63.091; DELTA M = 20.0; DELTA ML = 120.0
 File prepared by M. J. Hynes, October 22 2000
NO.  A   PARAMETER    DELTA     ERROR    CONDITION   DESCRIPTION
   1  1  1.36692E+04 2.000E-01 2.018E+03 2.761E+00    K1
   2  1  8.13212E+00 2.000E-01 1.732E-02 1.145E+00   SHIFT M
   3  1  1.02507E+01 1.000E+00 7.910E-03 2.697E+00    SHIFT ML
0RMS ERROR = 1.81E-02  MAX ERROR = 3.67E-02 AT OBS.NO.  4
 RESIDUALS SQUARED = 4.90E-03
 RFACTOR =     0.1657 PERCENT
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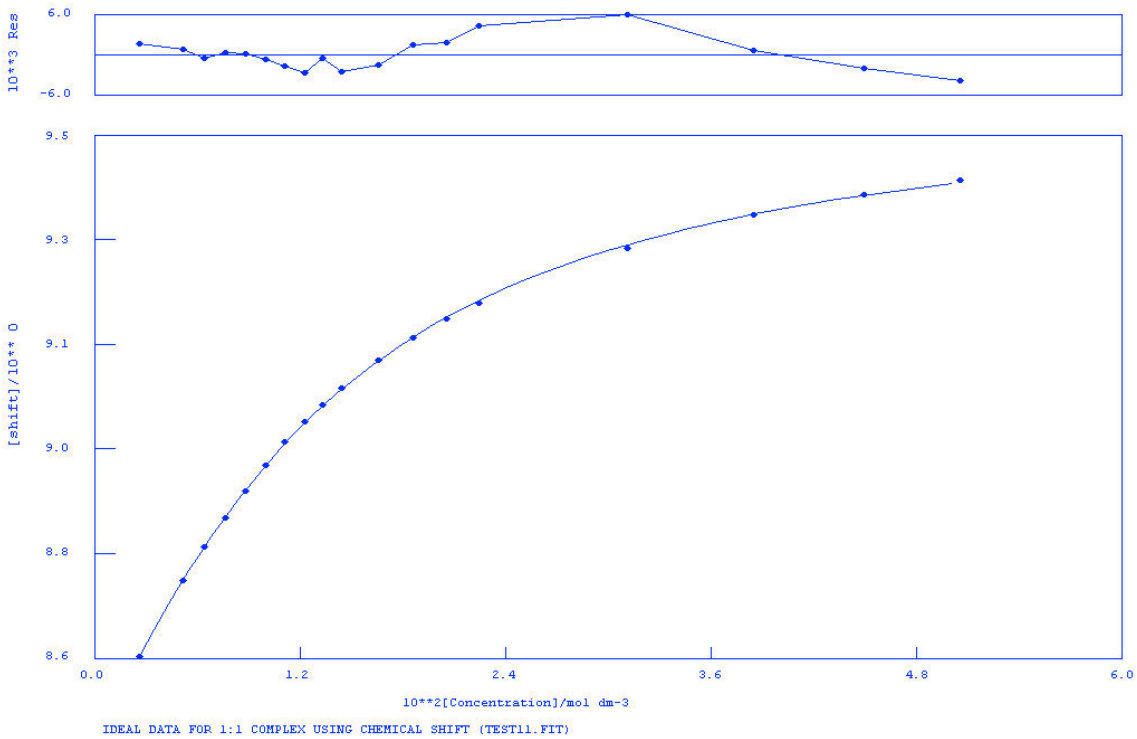
Figure S12 NMR titration of compound **1** *vs*. $TEAHCO_3$ in $[D_6]DMSO/H_2O$ 0.5%.

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 FILE: TEST11.FIT
 IDEAL DATA: K1 = 63.091; DELTA M = 20.0; DELTA ML = 120.0
 File prepared by M. J. Hynes, October 22 2000

NO.  A   PARAMETER    DELTA     ERROR    CONDITION   DESCRIPTION
   1  1  1.28260E+02 2.000E-01 1.848E+00 1.946E+01    K1
   2  1  8.44652E+00 2.000E-01 2.687E-03 5.067E+00   SHIFT M
   3  1  9.59782E+00 1.000E+00 3.484E-03 1.044E+01    SHIFT ML

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 RESIDUALS SQUARED = 1.00E-04
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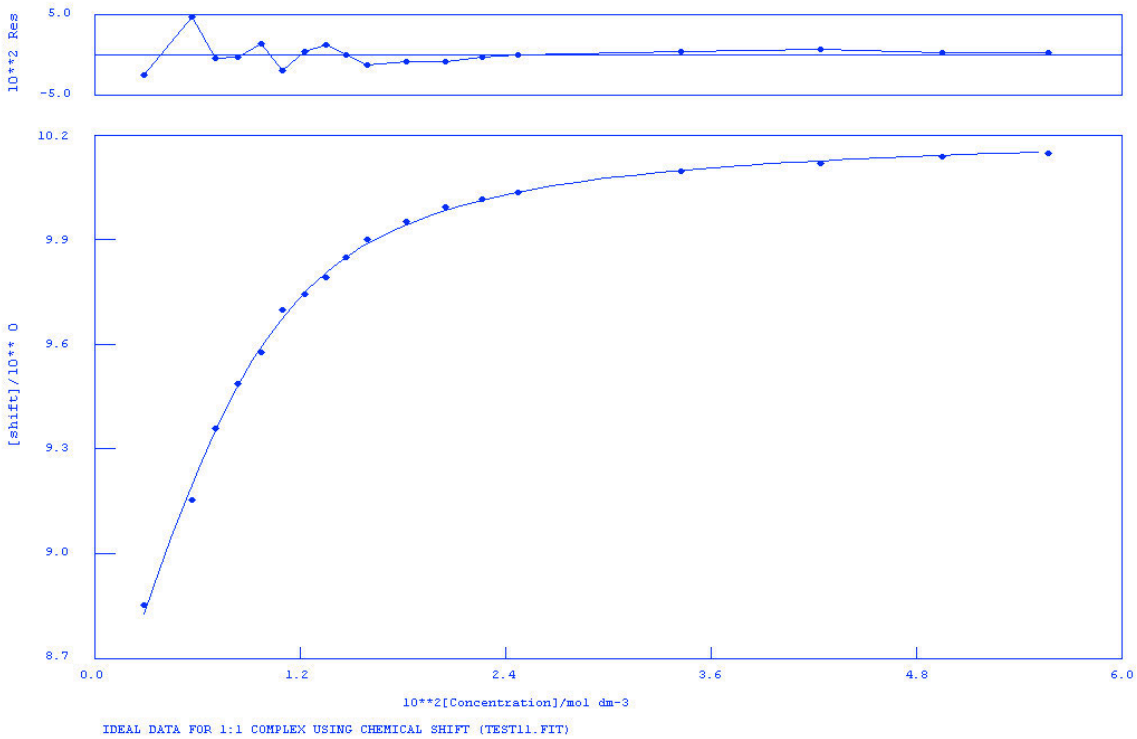
Figure S13 NMR titration of compound **1** *vs*. TBACl in [D_6]DMSO/H_2O 0.5%.

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 FILE: TEST11.FIT
 IDEAL DATA: K1 = 63.091; DELTA M = 20.0; DELTA ML = 120.0
 File prepared by M. J. Hynes, October 22 2000

NO.  A   PARAMETER    DELTA     ERROR    CONDITION   DESCRIPTION
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   2  1  8.36966E+00 2.000E-01 2.082E-02 2.759E+00   SHIFT M
   3  1  1.02193E+01 1.000E+00 1.141E-02 5.333E+00    SHIFT ML

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 RESIDUALS SQUARED = 4.03E-03
 RFACTOR =     0.1530 PERCENT
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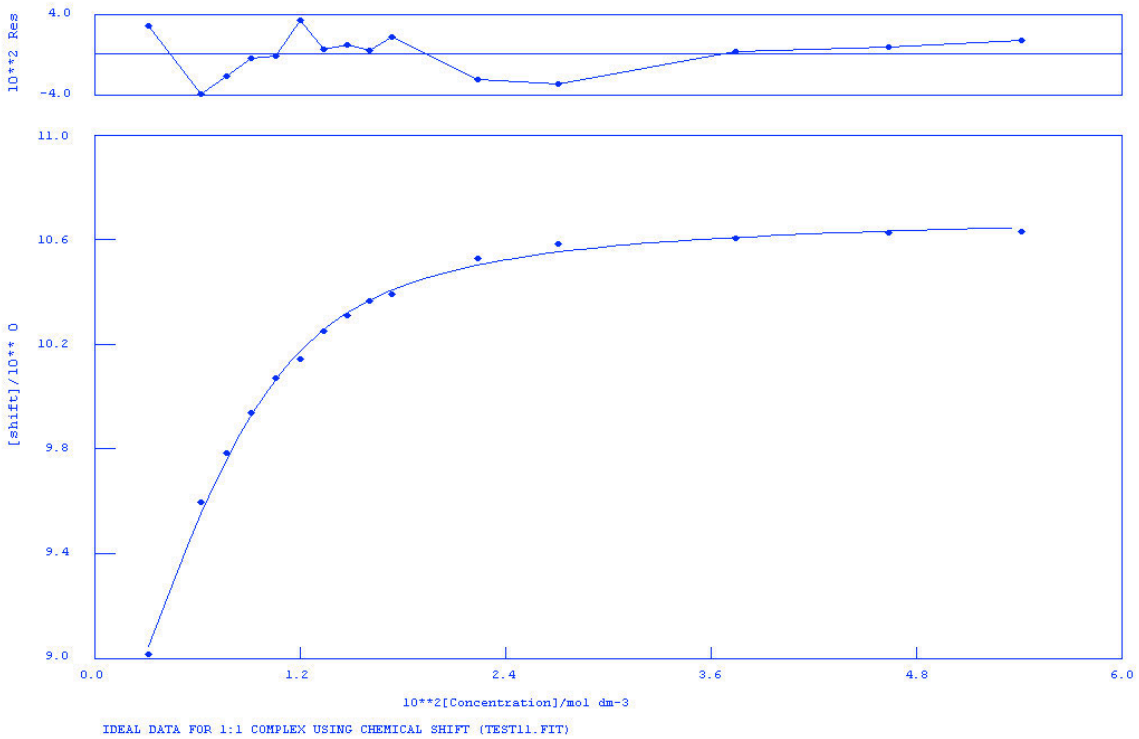
Figure S14 NMR titration of compound **1** *vs*. TBAOAc in [D_6]DMSO/H_2O 10%.

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 File prepared by M. J. Hynes, October 22 2000

NO.  A   PARAMETER    DELTA     ERROR    CONDITION   DESCRIPTION
   1  1  7.36242E+02 2.000E-01 5.379E+01 7.262E+00    K1
   2  1  8.43271E+00 2.000E-01 3.009E-02 2.365E+00   SHIFT M
   3  1  1.07126E+01 1.000E+00 1.647E-02 4.969E+00    SHIFT ML

0RMS ERROR = 2.27E-02  MAX ERROR = 3.94E-02 AT OBS.NO.  2
 RESIDUALS SQUARED = 6.19E-03
 RFACTOR =     0.1992 PERCENT
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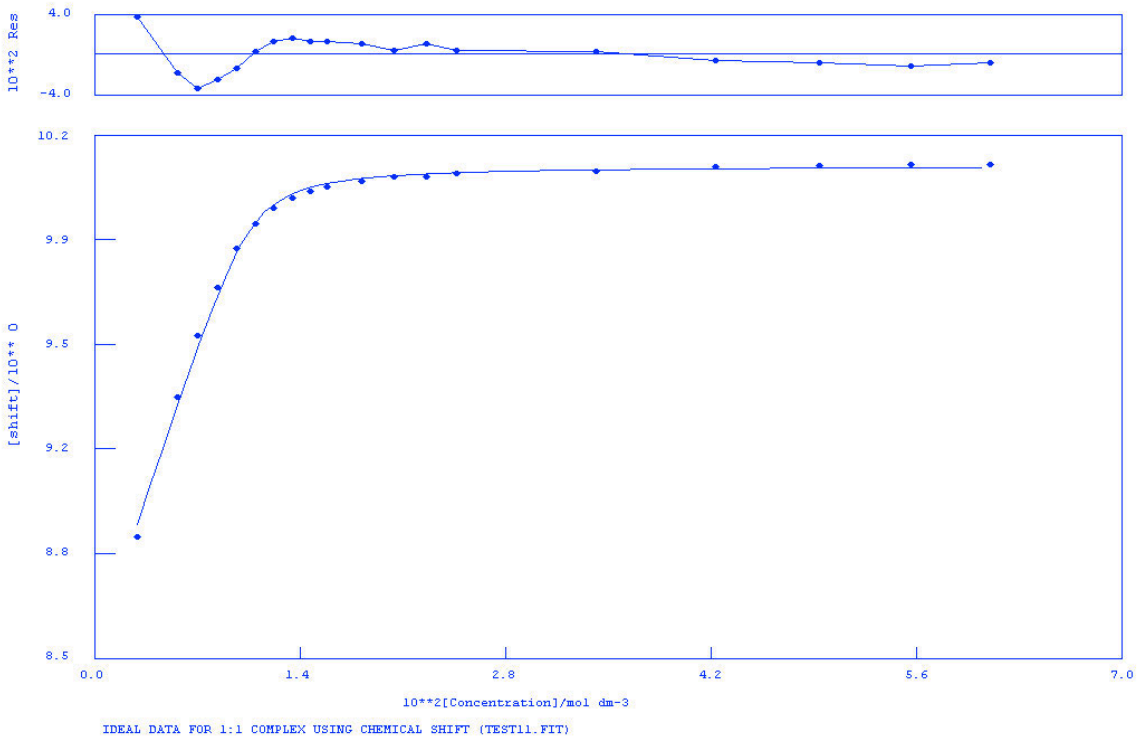
Figure S15 NMR titration of compound **1** *vs*. TBAOBz in [D_6]DMSO/H_2O 10%.

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Calculations by WinEQNMR Version 1.20 by Michael J. Hynes
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  Reaction:   M + L = ML
 FILE: TEST11.FIT
 IDEAL DATA: K1 = 63.091; DELTA M = 20.0; DELTA ML = 120.0
 File prepared by M. J. Hynes, October 22 2000

NO.  A   PARAMETER    DELTA     ERROR    CONDITION   DESCRIPTION
   1  1  4.71847E+03 2.000E-01 2.041E+02 1.516E+00    K1
   2  1  8.50810E+00 2.000E-01 1.991E-02 1.309E+00   SHIFT M
   3  1  1.00995E+01 1.000E+00 5.338E-03 1.373E+00    SHIFT ML

0RMS ERROR = 1.77E-02  MAX ERROR = 3.70E-02 AT OBS.NO.  1
 RESIDUALS SQUARED = 5.02E-03
 RFACTOR =     0.1641 PERCENT
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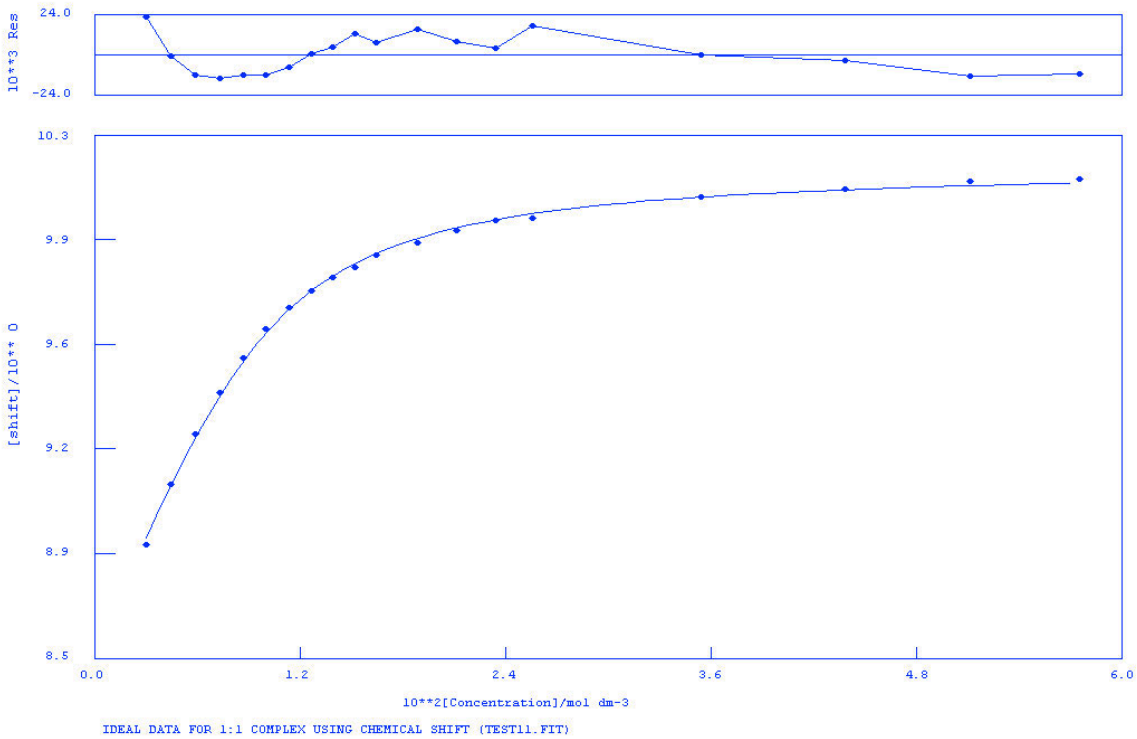
Figure S16 NMR titration of compound **1** *vs*. $TBAH_2PO_4$ in $[D_6]DMSO/H_2O$ 10%.

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  Reaction:   M + L = ML
 FILE: TEST11.FIT
 IDEAL DATA: K1 = 63.091; DELTA M = 20.0; DELTA ML = 120.0
 File prepared by M. J. Hynes, October 22 2000

NO.  A   PARAMETER    DELTA     ERROR    CONDITION   DESCRIPTION
   1  1  5.44643E+02 2.000E-01 2.416E+01 8.008E+00    K1
   2  1  8.49533E+00 2.000E-01 1.318E-02 2.509E+00   SHIFT M
   3  1  1.01960E+01 1.000E+00 8.356E-03 5.295E+00    SHIFT ML

0RMS ERROR = 1.21E-02  MAX ERROR = 2.21E-02 AT OBS.NO.  1
 RESIDUALS SQUARED = 2.35E-03
 RFACTOR =     0.1141 PERCENT
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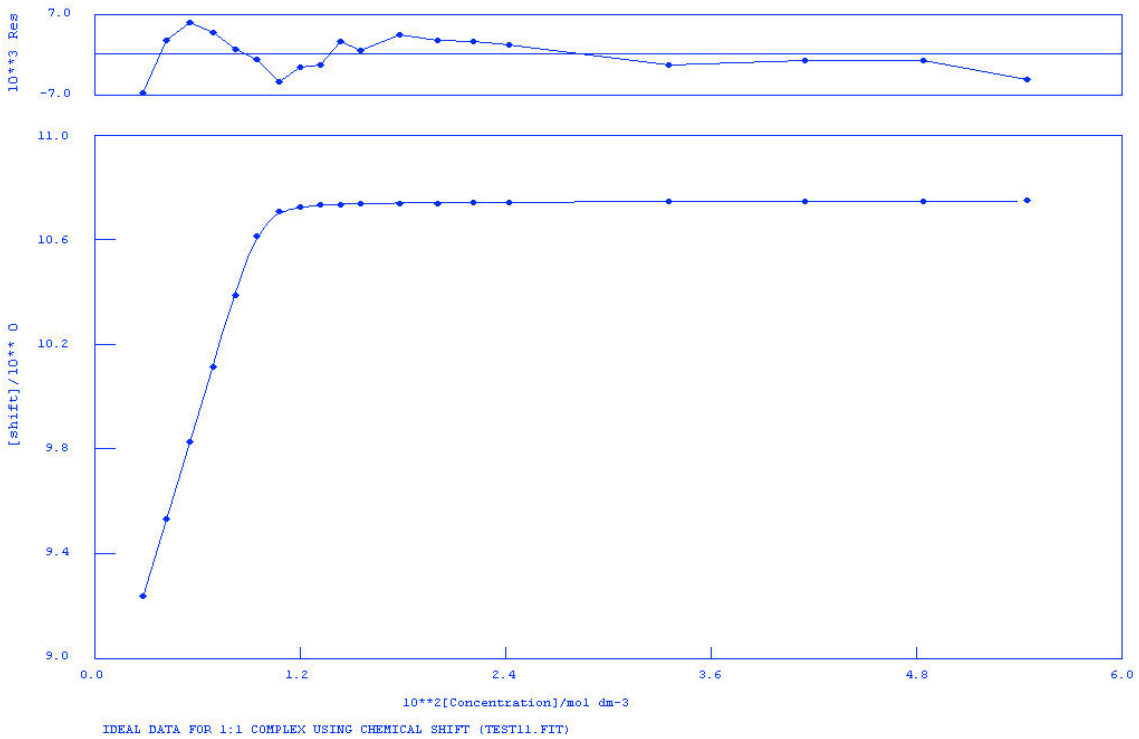
Figure S17 NMR titration of compound **1** *vs*. $TEAHCO_3$ in $[D_6]DMSO/H_2O$ 10%.

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Calculations by WinEQNMR Version 1.20 by Michael J. Hynes
Program run at 15:38:02   on 03/27/2008

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 FILE: TEST11.FIT
 IDEAL DATA: K1 = 63.091; DELTA M = 20.0; DELTA ML = 120.0
 File prepared by M. J. Hynes, October 22 2000

NO.  A   PARAMETER    DELTA     ERROR    CONDITION   DESCRIPTION
   1  1  3.60451E+04 2.000E-01 7.355E+02 1.056E+00    K1
   2  1  8.60615E+00 2.000E-01 3.361E-03 1.087E+00   SHIFT M
   3  1  1.07459E+01 1.000E+00 9.207E-04 1.131E+00    SHIFT ML

0RMS ERROR = 3.41E-03  MAX ERROR = 6.80E-03 AT OBS.NO.  1
 RESIDUALS SQUARED = 1.86E-04
 RFACTOR =     0.0298 PERCENT
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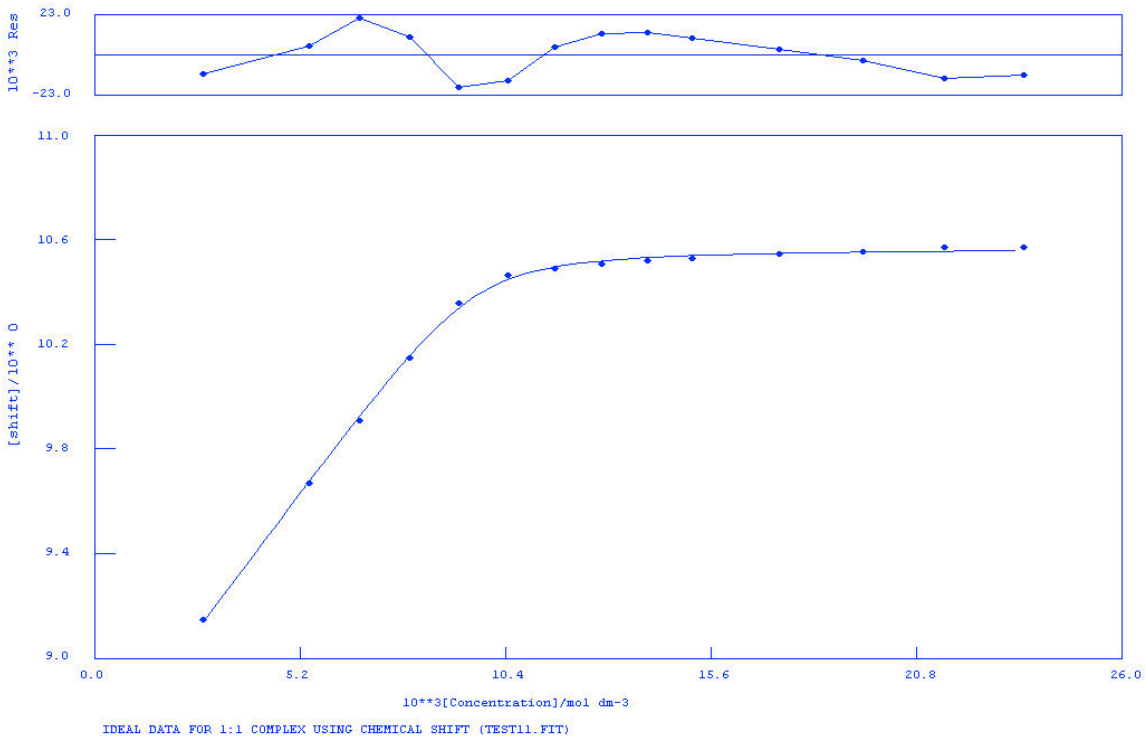
Figure S18 NMR titration of compound **2** *vs*. TBAOAc in [D_6]DMSO/H_2O 0.5%.

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Program run at 16:20:27   on 03/27/2008

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 File prepared by M. J. Hynes, October 22 2000

NO.  A   PARAMETER    DELTA     ERROR    CONDITION   DESCRIPTION
   1  1  1.06028E+04 2.000E-01 1.415E+02 1.038E+00    K1
   2  1  8.57007E+00 2.000E-01 1.545E-02 1.162E+00   SHIFT M
   3  1  1.05699E+01 1.000E+00 4.454E-03 1.196E+00    SHIFT ML

0RMS ERROR = 1.34E-02  MAX ERROR = 2.06E-02 AT OBS.NO.  3
 RESIDUALS SQUARED = 1.98E-03
 RFACTOR =     0.1157 PERCENT
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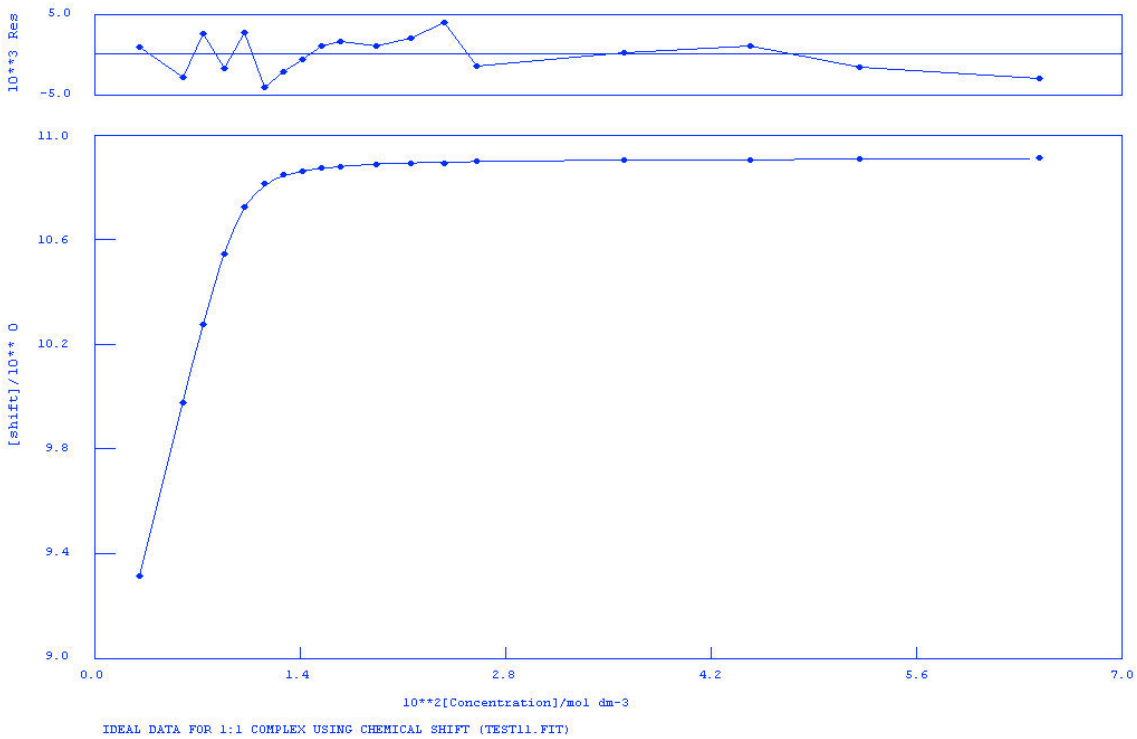
Figure S19 NMR titration of compound **2** *vs*. $TBAH_2PO_4$ in $[D_6]DMSO/H_2O$ 0.5%.

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Program run at 15:12:13   on 03/19/2008

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 FILE: TEST11.FIT
 IDEAL DATA: K1 = 63.091; DELTA M = 20.0; DELTA ML = 120.0
 File prepared by M. J. Hynes, October 22 2000

NO.  A   PARAMETER    DELTA     ERROR    CONDITION   DESCRIPTION
   1  1  1.00048E+04 2.000E-01 1.917E+02 2.147E+00    K1
   2  1  8.61681E+00 2.000E-01 2.944E-03 1.131E+00   SHIFT M
   3  1  1.09110E+01 1.000E+00 9.009E-04 2.113E+00    SHIFT ML

0RMS ERROR = 2.42E-03  MAX ERROR = 4.12E-03 AT OBS.NO.  6
 RESIDUALS SQUARED = 8.75E-05
 RFACTOR =     0.0206 PERCENT
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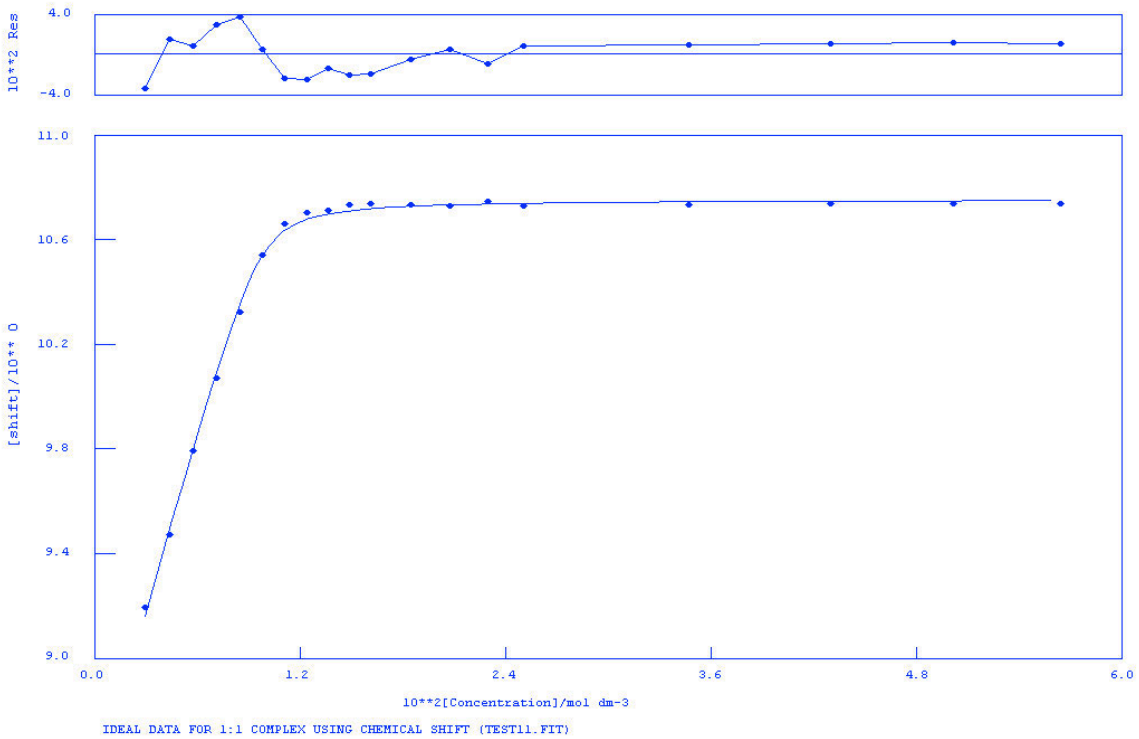
Figure S20 NMR titration of compound **2** *vs*. TBAOBz in [D_6]DMSO/H_2O 0.5%.

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Calculations by WinEQNMR Version 1.20 by Michael J. Hynes
Program run at 15:48:32   on 07/02/2008

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 FILE: TEST11.FIT
 IDEAL DATA: K1 = 63.091; DELTA M = 20.0; DELTA ML = 120.0
 File prepared by M. J. Hynes, October 22 2000

NO.  A   PARAMETER    DELTA     ERROR    CONDITION   DESCRIPTION
   1  1  9.57802E+03 2.000E-01 1.303E+03 2.112E+00    K1
   2  1  8.47565E+00 2.000E-01 2.091E-02 1.168E+00   SHIFT M
   3  1  1.07526E+01 1.000E+00 7.402E-03 2.035E+00    SHIFT ML

0RMS ERROR = 2.03E-02  MAX ERROR = 3.77E-02 AT OBS.NO.  5
 RESIDUALS SQUARED = 6.58E-03
 RFACTOR =     0.1776 PERCENT
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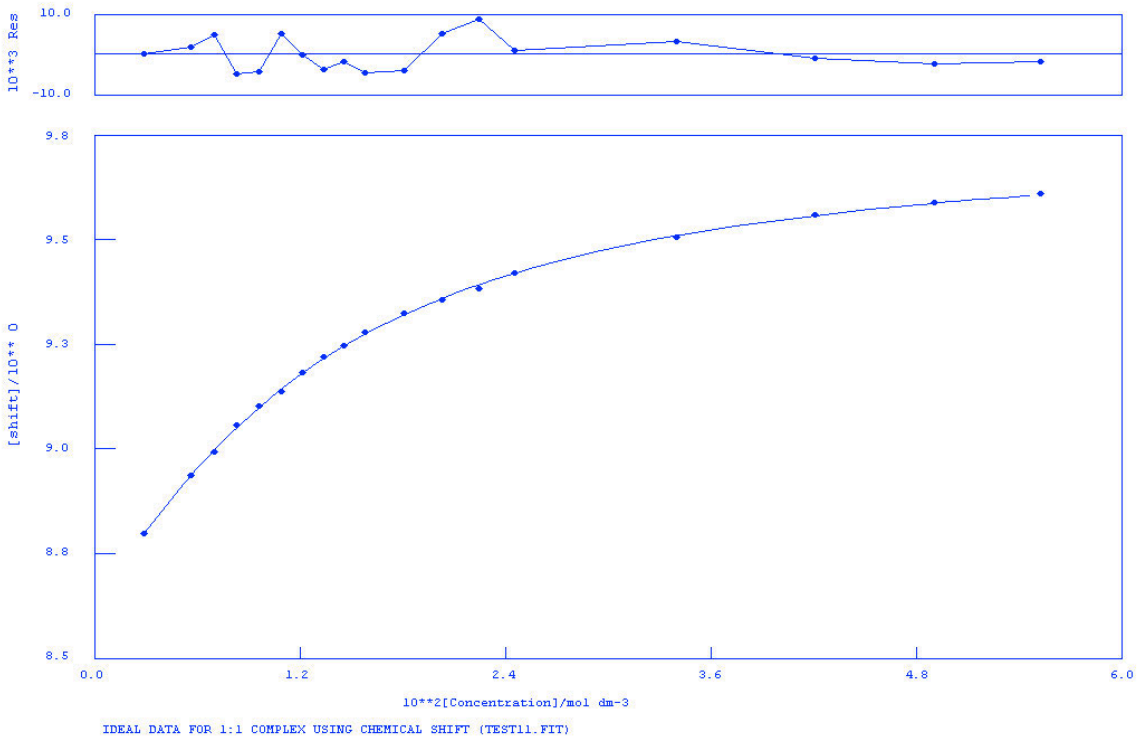
Figure S21 NMR titration of compound **2** *vs*. $TEAHCO_3$ in $[D_6]DMSO/H_2O$ 0.5%.

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Program run at 11:19:43   on 03/28/2008

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 FILE: TEST11.FIT
 IDEAL DATA: K1 = 63.091; DELTA M = 20.0; DELTA ML = 120.0
 File prepared by M. J. Hynes, October 22 2000

NO.  A   PARAMETER    DELTA     ERROR    CONDITION   DESCRIPTION
   1  1  1.28350E+02 2.000E-01 3.365E+00 2.519E+01    K1
   2  1  8.63011E+00 2.000E-01 5.070E-03 5.942E+00   SHIFT M
   3  1  9.82032E+00 1.000E+00 6.228E-03 1.309E+01    SHIFT ML

0RMS ERROR = 4.30E-03  MAX ERROR = 8.80E-03 AT OBS.NO. 13
 RESIDUALS SQUARED = 2.77E-04
 RFACTOR =     0.0422 PERCENT
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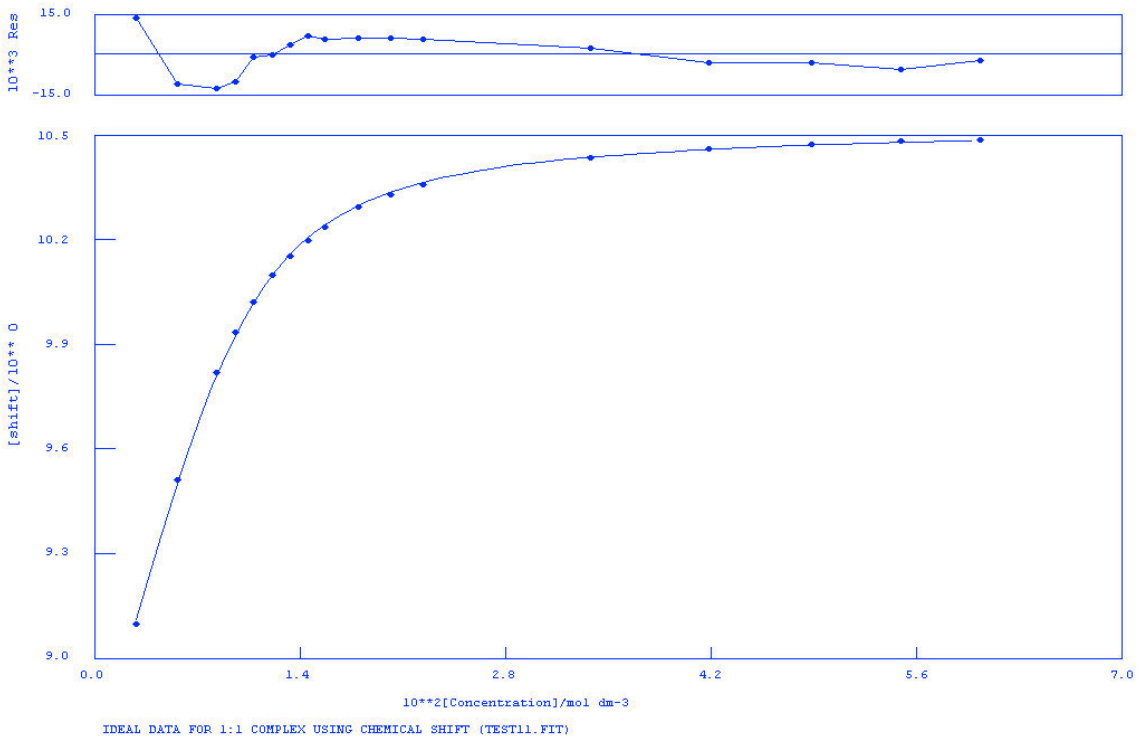
Figure S22 NMR titration of compound **2** *vs*. TBACl in [D_6]DMSO/H_2O 0.5%.

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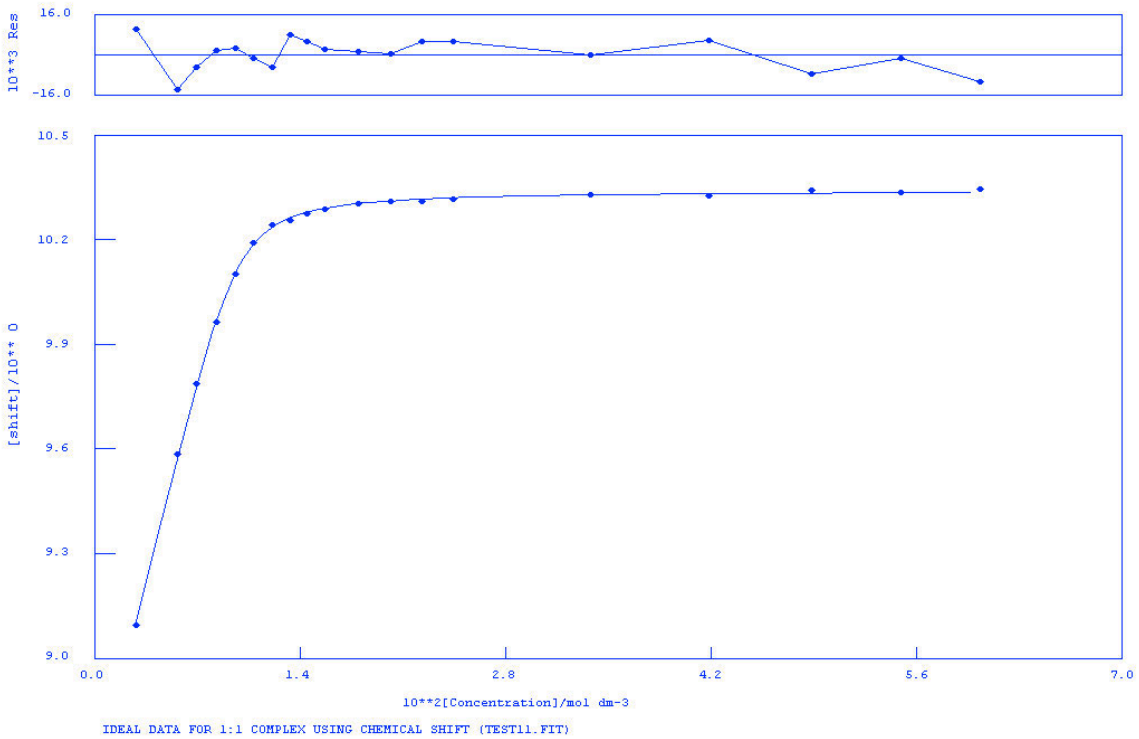
 FILE: TEST11.FIT
 IDEAL DATA: K1 = 63.091; DELTA M = 20.0; DELTA ML = 120.0
 File prepared by M. J. Hynes, October 22 2000

NO.  A   PARAMETER    DELTA     ERROR    CONDITION   DESCRIPTION
   1  1  7.74179E+02 2.000E-01 2.061E+01 5.613E+00    K1
   2  1  8.65797E+00 2.000E-01 9.369E-03 2.048E+00   SHIFT M
   3  1  1.05296E+01 1.000E+00 4.600E-03 4.007E+00    SHIFT ML

0RMS ERROR = 7.73E-03  MAX ERROR = 1.37E-02 AT OBS.NO.  1
 RESIDUALS SQUARED = 8.36E-04
 RFACTOR =     0.0691 PERCENT
```

Figure S23 NMR titration of compound **2** *vs*. TBAOAc in [D_6]DMSO/H_2O 10%.



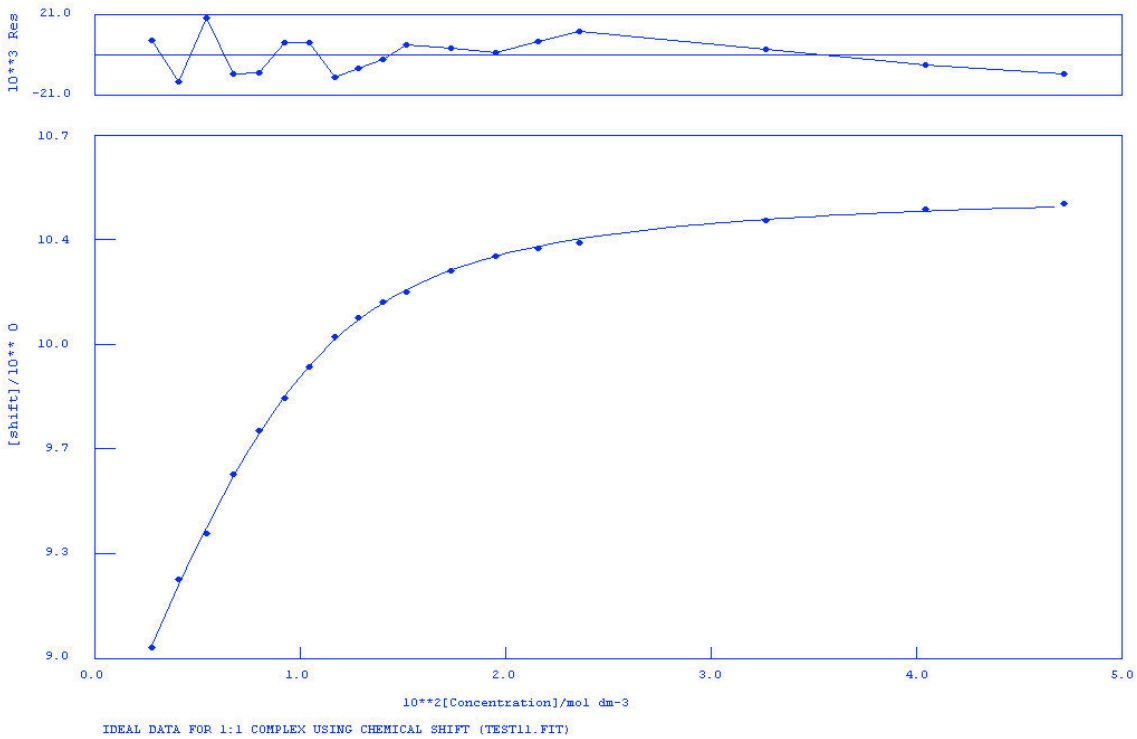

```
Calculations by WinEQNMR Version 1.20 by Michael J. Hynes
Program run at 13:39:41   on 03/29/2008

 IDEAL DATA FOR 1:1 COMPLEX USING CHEMICAL SHIFT (TEST11.FIT)
  Reaction:   M + L = ML
 FILE: TEST11.FIT
 IDEAL DATA: K1 = 63.091; DELTA M = 20.0; DELTA ML = 120.0
 File prepared by M. J. Hynes, October 22 2000

NO.  A   PARAMETER    DELTA     ERROR    CONDITION   DESCRIPTION
   1  1  5.17187E+03 2.000E-01 2.511E+02 3.079E+00    K1
   2  1  8.60225E+00 2.000E-01 7.912E-03 1.251E+00   SHIFT M
   3  1  1.03406E+01 1.000E+00 2.836E-03 2.807E+00    SHIFT ML

0RMS ERROR = 6.67E-03  MAX ERROR = 1.42E-02 AT OBS.NO.  2
 RESIDUALS SQUARED = 7.11E-04
 RFACTOR =     0.0603 PERCENT
```

Figure S24 NMR titration of compound **2** *vs*. $TBAH_2PO_4$ in $[D_6]DMSO/H_2O$ 10%.



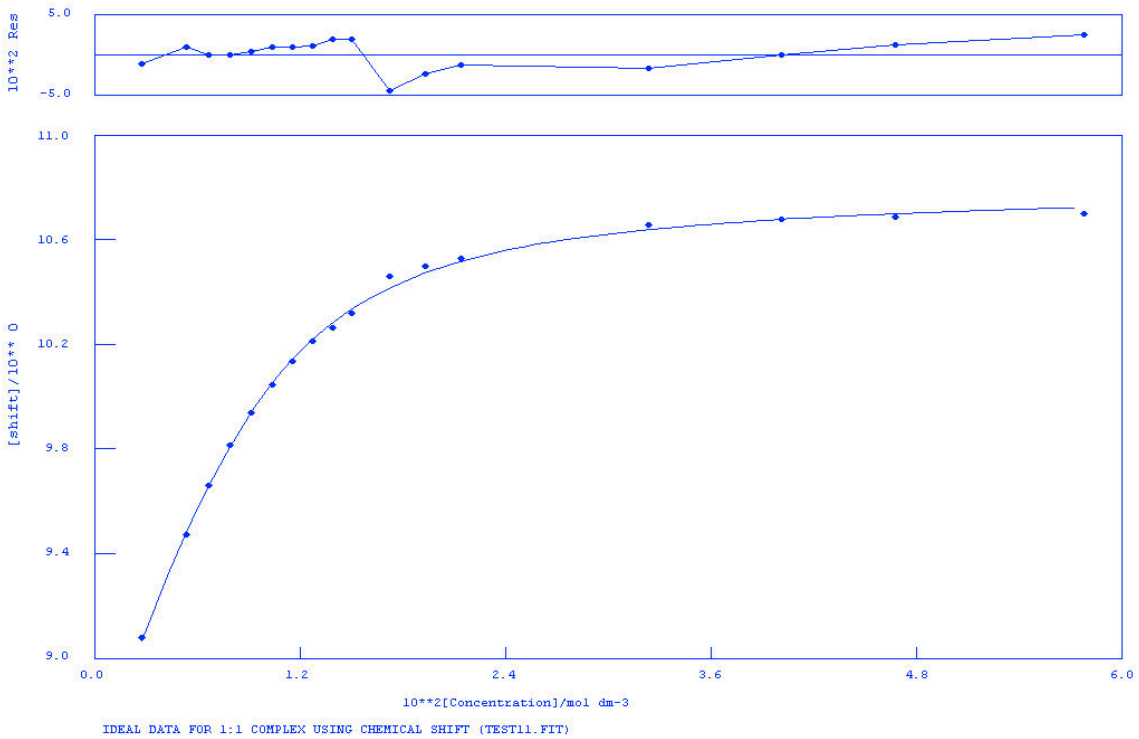

```
Calculations by WinEQNMR Version 1.20 by Michael J. Hynes
Program run at 16:27:15   on 07/03/2008

 IDEAL DATA FOR 1:1 COMPLEX USING CHEMICAL SHIFT (TEST11.FIT)
  Reaction:   M + L = ML
 FILE: TEST11.FIT
 IDEAL DATA: K1 = 63.091; DELTA M = 20.0; DELTA ML = 120.0
 File prepared by M. J. Hynes, October 22 2000

NO.  A   PARAMETER    DELTA     ERROR    CONDITION   DESCRIPTION
   1  1  6.99186E+02 2.000E-01 2.643E+01 8.334E+00    K1
   2  1  8.59973E+00 2.000E-01 9.556E-03 2.115E+00   SHIFT M
   3  1  1.05378E+01 1.000E+00 7.749E-03 6.269E+00    SHIFT ML

0RMS ERROR = 9.87E-03  MAX ERROR = 1.88E-02 AT OBS.NO.  3
 RESIDUALS SQUARED = 1.46E-03
 RFACTOR =     0.0901 PERCENT
```

Figure S25 NMR titration of compound **2** *vs*. $TEAHCO_3$ in $[D_6]DMSO/H_2O$ 10%.



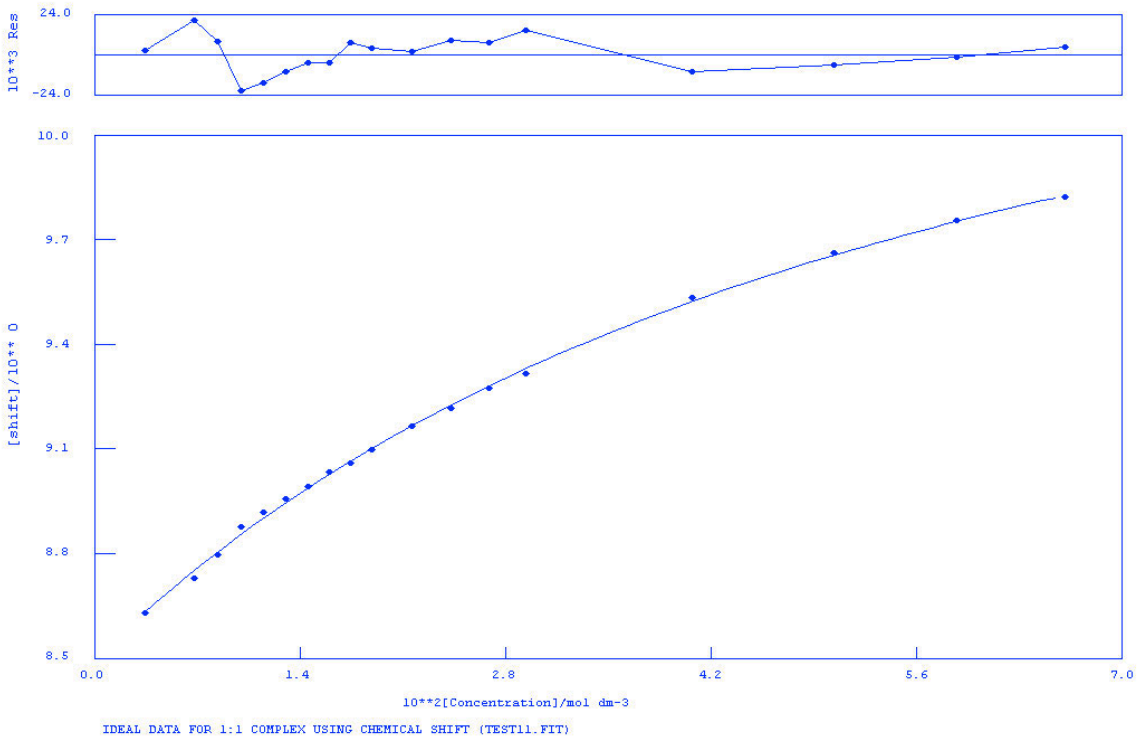

```
Calculations by WinEQNMR Version 1.20 by Michael J. Hynes
Program run at 09:20:56   on 03/31/2008

 IDEAL DATA FOR 1:1 COMPLEX USING CHEMICAL SHIFT (TEST11.FIT)
  Reaction:   M + L = ML
 FILE: TEST11.FIT
 IDEAL DATA: K1 = 63.091; DELTA M = 20.0; DELTA ML = 120.0
 File prepared by M. J. Hynes, October 22 2000

NO.  A   PARAMETER    DELTA     ERROR    CONDITION   DESCRIPTION
   1  1  5.21544E+02 2.000E-01 2.858E+01 8.400E+00    K1
   2  1  8.57897E+00 2.000E-01 2.163E-02 2.675E+00   SHIFT M
   3  1  1.08073E+01 1.000E+00 1.426E-02 5.463E+00    SHIFT ML

0RMS ERROR = 1.88E-02  MAX ERROR = 4.49E-02 AT OBS.NO. 11
 RESIDUALS SQUARED = 4.97E-03
 RFACTOR =     0.1677 PERCENT
```

Figure S26 NMR titration of compound **2** *vs*. TBAOBz in [D_6]DMSO/H_2O 10%.



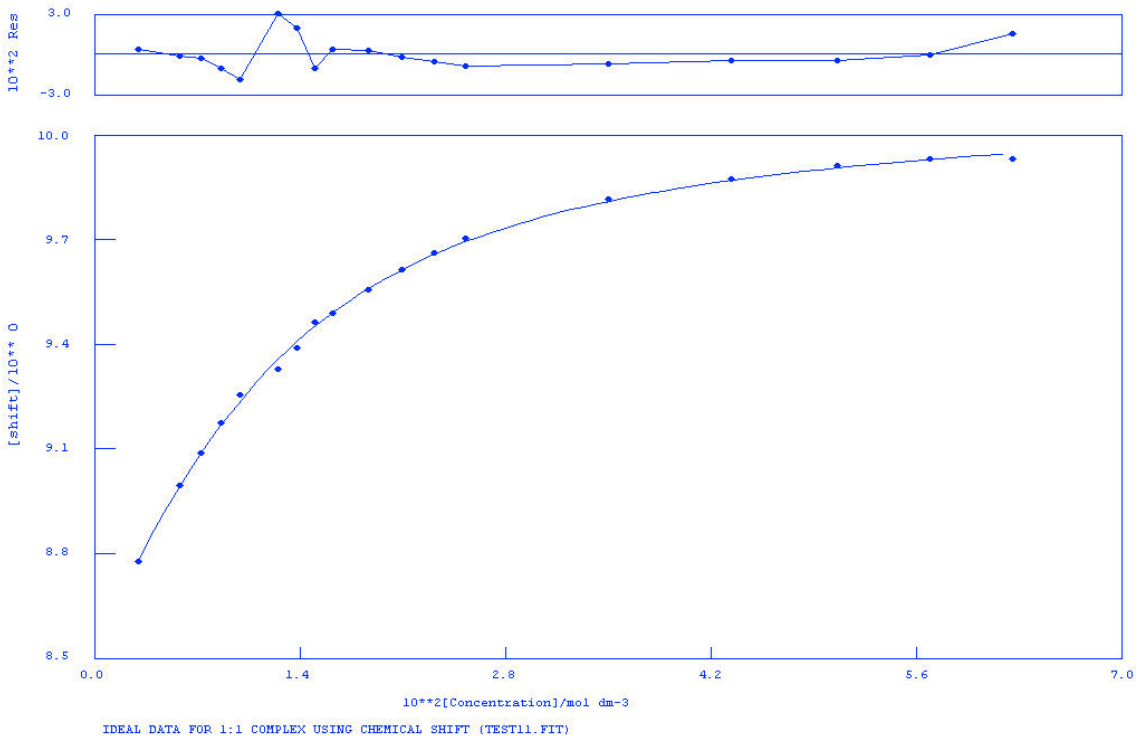

```
Calculations by WinEQNMR Version 1.20 by Michael J. Hynes
Program run at 18:34:49   on 03/30/2008

 IDEAL DATA FOR 1:1 COMPLEX USING CHEMICAL SHIFT (TEST11.FIT)
  Reaction:   M + L = ML
 FILE: TEST11.FIT
 IDEAL DATA: K1 = 63.091; DELTA M = 20.0; DELTA ML = 120.0
 File prepared by M. J. Hynes, October 22 2000

NO.  A   PARAMETER    DELTA     ERROR    CONDITION   DESCRIPTION
   1  1  2.00648E+01 2.000E-01 1.366E+00 1.110E+02    K1
   2  1  8.50001E+00 2.000E-01 1.125E-02 9.640E+00   SHIFT M
   3  1  1.09117E+01 1.000E+00 7.402E-02 7.113E+01    SHIFT ML

0RMS ERROR = 1.14E-02  MAX ERROR = 2.14E-02 AT OBS.NO.  4
 RESIDUALS SQUARED = 1.95E-03
 RFACTOR =     0.1136 PERCENT
```

Figure S27 NMR titration of compound **2** *vs*. TBAOAc in [D_6]DMSO/H_2O 25%.



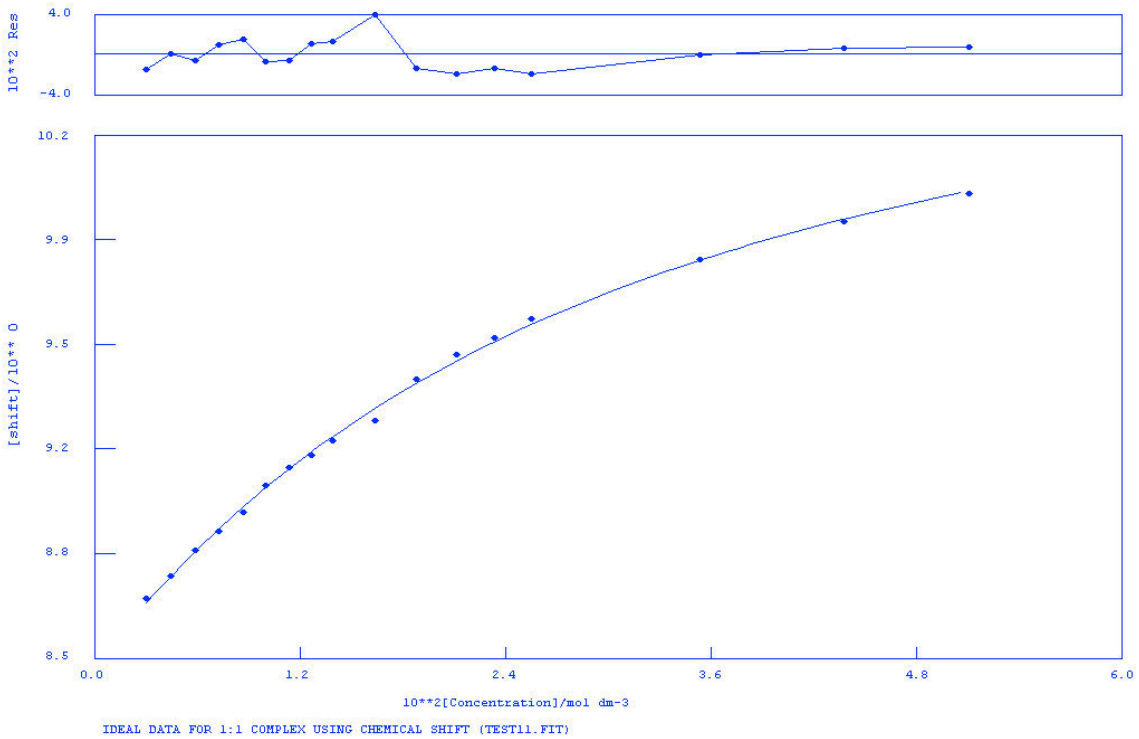

```
Calculations by WinEQNMR Version 1.20 by Michael J. Hynes
Program run at 18:10:59   on 03/30/2008

 IDEAL DATA FOR 1:1 COMPLEX USING CHEMICAL SHIFT (TEST11.FIT)
  Reaction:   M + L = ML
 FILE: TEST11.FIT
 IDEAL DATA: K1 = 63.091; DELTA M = 20.0; DELTA ML = 120.0
 File prepared by M. J. Hynes, October 22 2000

NO.  A   PARAMETER    DELTA     ERROR    CONDITION   DESCRIPTION
   1  1  1.60426E+02 2.000E-01 7.513E+00 1.567E+01    K1
   2  1  8.52240E+00 2.000E-01 1.439E-02 4.598E+00   SHIFT M
   3  1  1.01134E+01 1.000E+00 1.304E-02 8.133E+00    SHIFT ML

0RMS ERROR = 1.26E-02  MAX ERROR = 3.07E-02 AT OBS.NO.  6
 RESIDUALS SQUARED = 2.37E-03
 RFACTOR =     0.1206 PERCENT
```

Figure S28 NMR titration of compound **2** *vs*. $TBAH_2PO_4$ in $[D_6]DMSO/H_2O$ 25%.



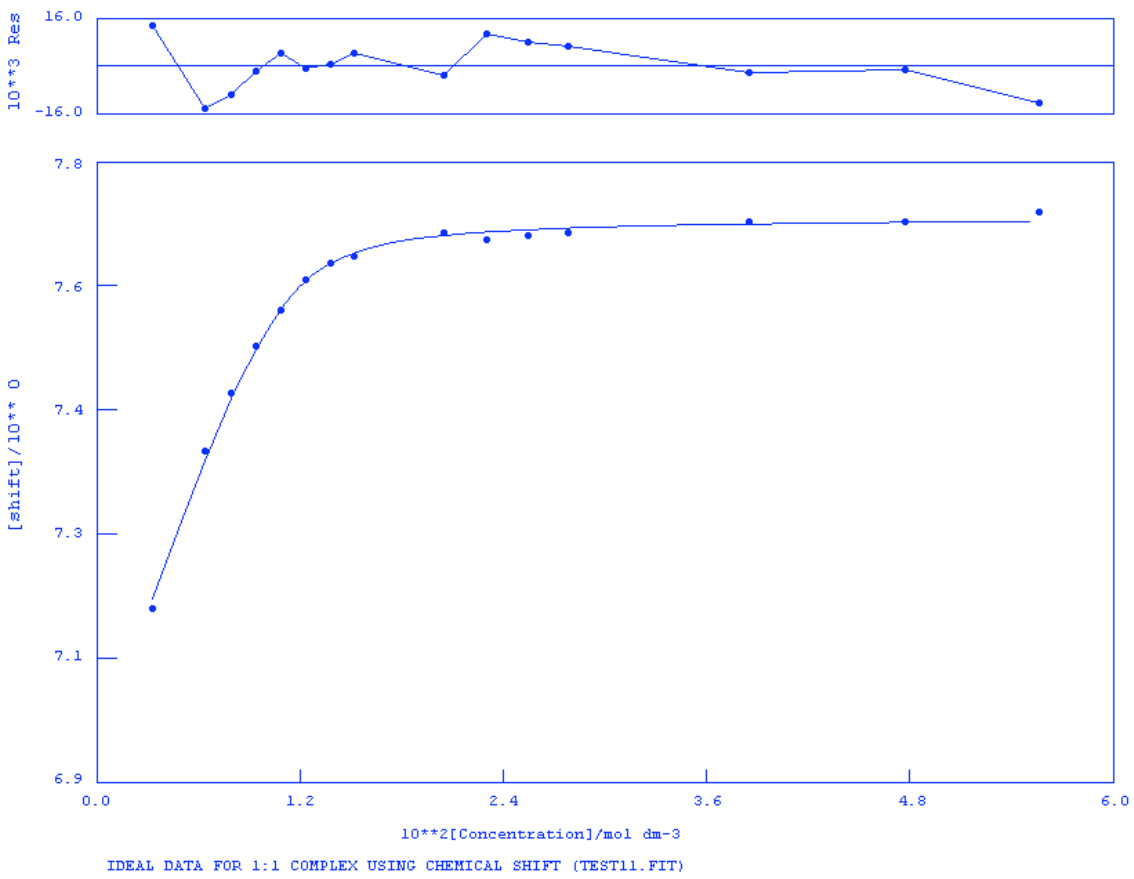

```
Calculations by WinEQNMR Version 1.20 by Michael J. Hynes
Program run at 16:43:39   on 07/03/2008

 IDEAL DATA FOR 1:1 COMPLEX USING CHEMICAL SHIFT (TEST11.FIT)
  Reaction:   M + L = ML
 FILE: TEST11.FIT
 IDEAL DATA: K1 = 63.091; DELTA M = 20.0; DELTA ML = 120.0
 File prepared by M. J. Hynes, October 22 2000

NO.  A   PARAMETER    DELTA     ERROR    CONDITION   DESCRIPTION
   1  1  4.24578E+01 2.000E-01 3.300E+00 6.734E+01    K1
   2  1  8.48036E+00 2.000E-01 1.566E-02 7.252E+00   SHIFT M
   3  1  1.08273E+01 1.000E+00 6.805E-02 4.260E+01    SHIFT ML

0RMS ERROR = 1.63E-02  MAX ERROR = 3.91E-02 AT OBS.NO. 10
 RESIDUALS SQUARED = 3.71E-03
 RFACTOR =     0.1591 PERCENT
```

Figure S29 NMR titration of compound **2** *vs*. $TEAHCO_3$ in $[D_6]DMSO/H_2O$ 25%.



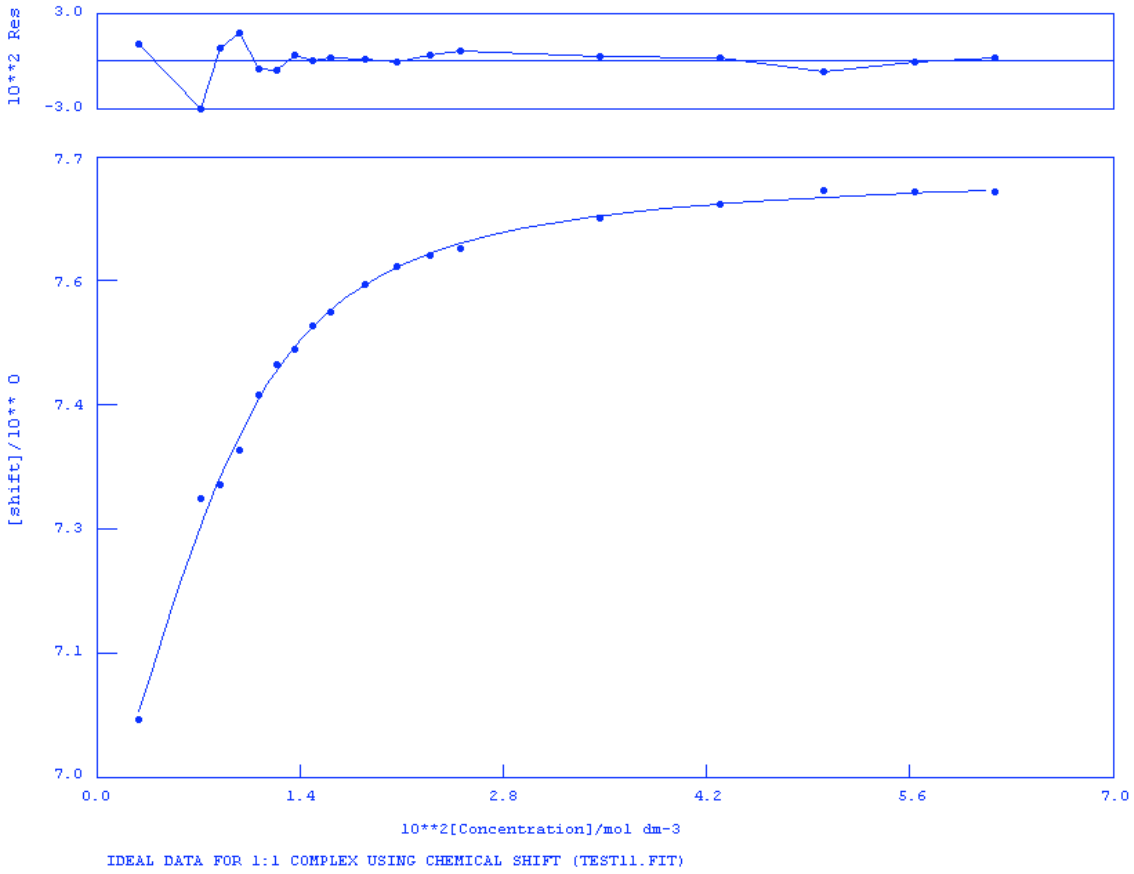

```
Calculations by WinEQNMR Version 1.20 by Michael J. Hynes
Program run at 11:48:31   on 06/05/2008

 IDEAL DATA FOR 1:1 COMPLEX USING CHEMICAL SHIFT (TEST11.FIT)
  Reaction:   M + L = ML
 FILE: TEST11.FIT
 IDEAL DATA: K1 = 63.091; DELTA M = 20.0; DELTA ML = 120.0
 File prepared by M. J. Hynes, October 22 2000

NO.  A   PARAMETER    DELTA     ERROR    CONDITION   DESCRIPTION
   1  1  2.82794E+03 2.000E-01 1.462E+02 3.882E+00    K1
   2  1  6.94586E+00 2.000E-01 1.805E-02 4.033E+00   SHIFT M
   3  1  7.71848E+00 1.000E+00 2.878E-03 1.217E+00    SHIFT ML

0RMS ERROR = 8.77E-03  MAX ERROR = 1.47E-02 AT OBS.NO.  2
 RESIDUALS SQUARED = 9.22E-04
 RFACTOR =     0.1032 PERCENT
```

Figure S30 NMR titration of compound **3** *vs*. TBAOAc in [D_6]DMSO/H_2O 0.5%.



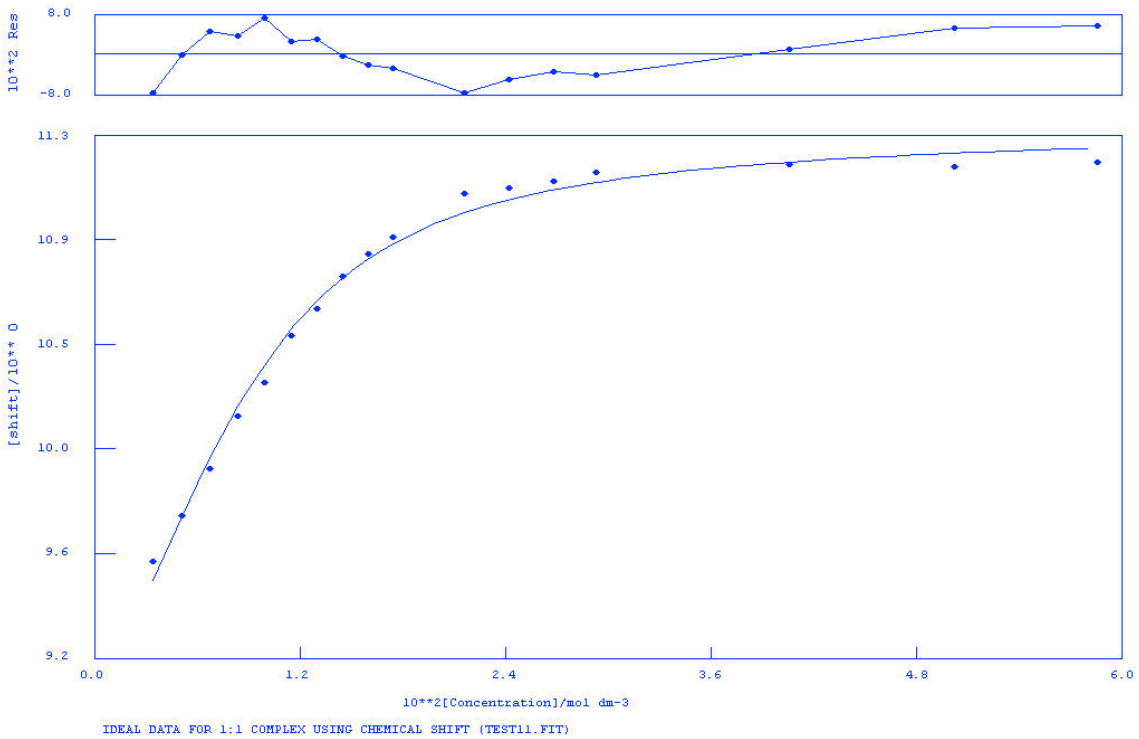

```
Calculations by WinEQNMR Version 1.20 by Michael J. Hynes
Program run at 11:08:27   on 06/05/2008

 IDEAL DATA FOR 1:1 COMPLEX USING CHEMICAL SHIFT (TEST11.FIT)
  Reaction:   M + L = ML
 FILE: TEST11.FIT
 IDEAL DATA: K1 = 63.091; DELTA M = 20.0; DELTA ML = 120.0
 File prepared by M. J. Hynes, October 22 2000

NO.  A   PARAMETER    DELTA     ERROR    CONDITION   DESCRIPTION
   1  1  5.13893E+02 2.000E-01 4.125E+01 7.478E+00    K1
   2  1  6.90291E+00 2.000E-01 1.264E-02 2.563E+00   SHIFT M
   3  1  7.69037E+00 1.000E+00 6.772E-03 4.853E+00    SHIFT ML

0RMS ERROR = 1.03E-02  MAX ERROR = 3.08E-02 AT OBS.NO.  2
 RESIDUALS SQUARED = 1.58E-03
 RFACTOR =     0.1247 PERCENT
```

Figure S31 NMR titration of compound **3** *vs*. TBAOBz in [D_6]DMSO/H_2O 0.5%.



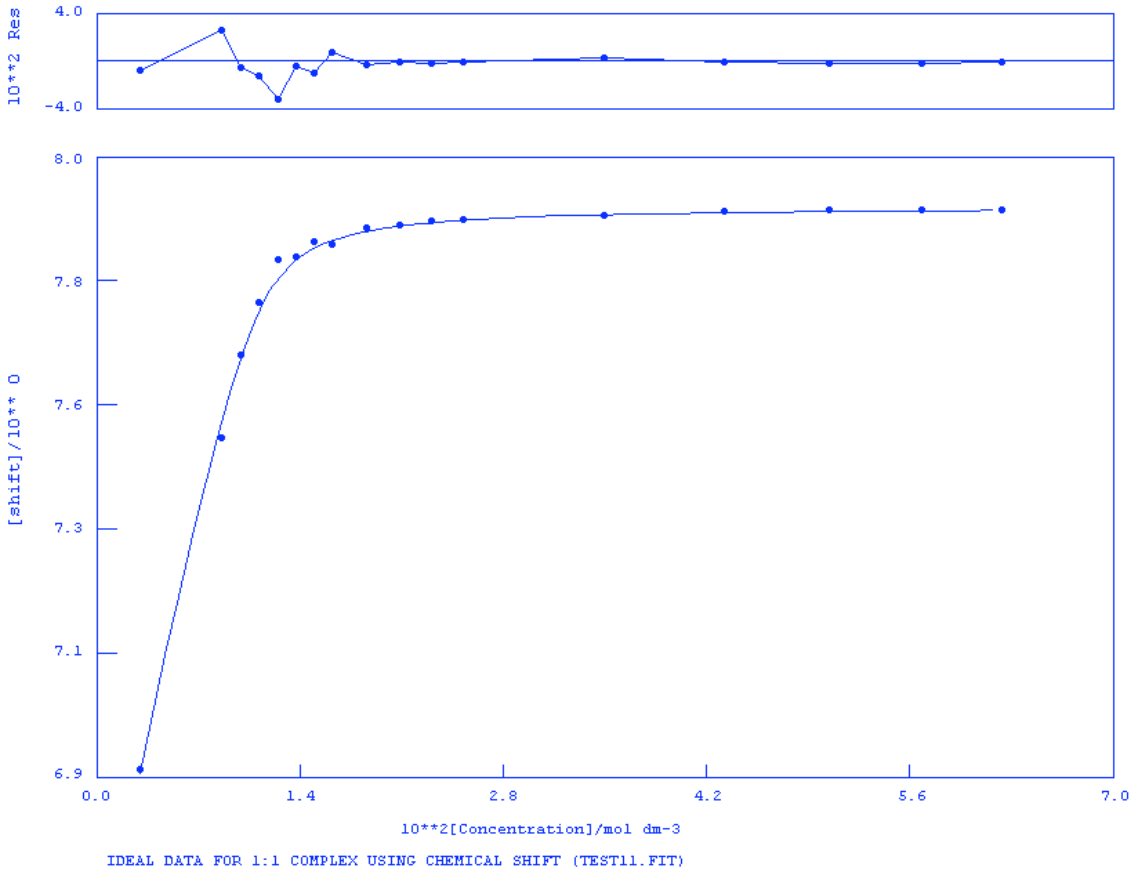

```
Calculations by WinEQNMR Version 1.20 by Michael J. Hynes
Program run at 16:06:36   on 06/26/2008

 IDEAL DATA FOR 1:1 COMPLEX USING CHEMICAL SHIFT (TEST11.FIT)
  Reaction:   M + L = ML
 FILE: TEST11.FIT
 IDEAL DATA: K1 = 63.091; DELTA M = 20.0; DELTA ML = 120.0
 File prepared by M. J. Hynes, October 22 2000

NO.  A   PARAMETER    DELTA     ERROR    CONDITION   DESCRIPTION
   1  1  4.76696E+02 2.000E-01 6.759E+01 9.041E+00    K1
   2  1  8.91273E+00 2.000E-01 5.511E-02 2.556E+00   SHIFT M
   3  1  1.13481E+01 1.000E+00 3.979E-02 6.067E+00    SHIFT ML

0RMS ERROR = 4.99E-02  MAX ERROR = 7.71E-02 AT OBS.NO. 11
 RESIDUALS SQUARED = 3.49E-02
 RFACTOR =     0.4242 PERCENT
```

Figure S32 NMR titration of compound **3** *vs*. $TEAHCO_3$ in $[D_6]DMSO/H_2O$ 0.5%.



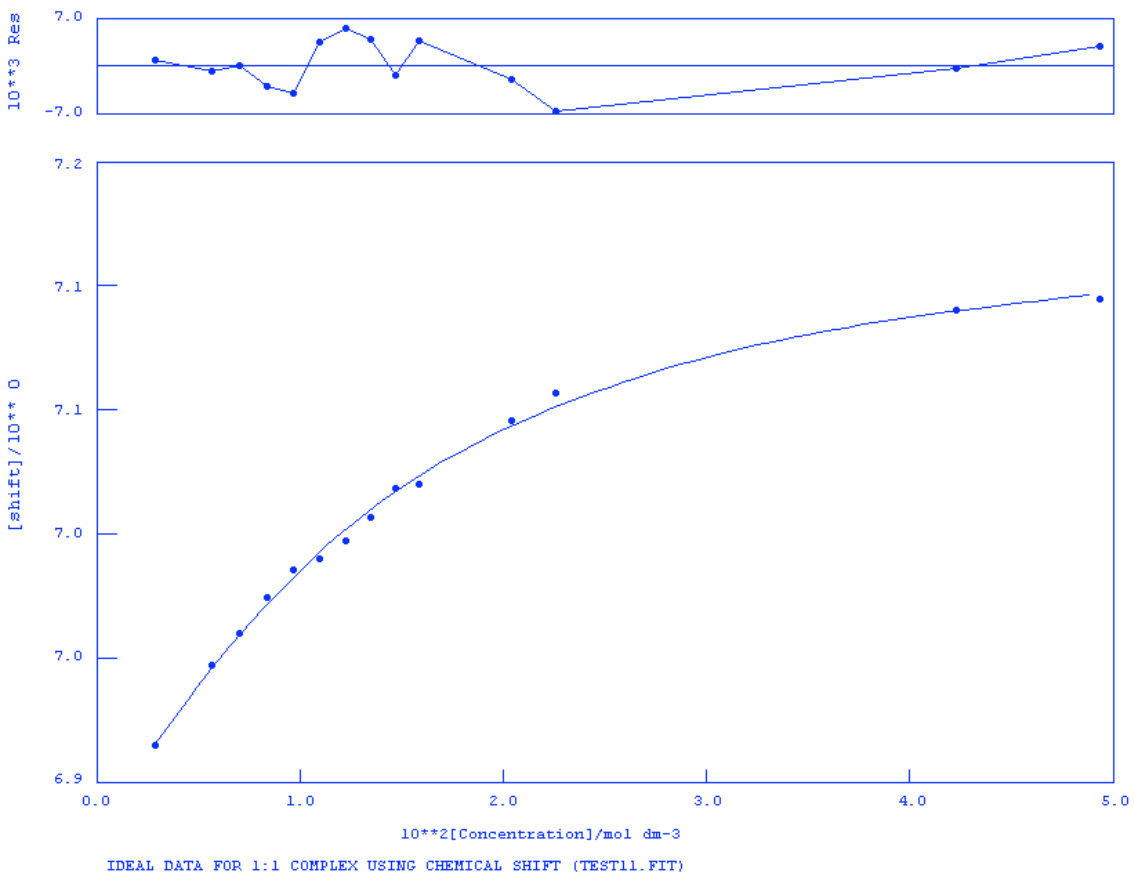

```
Calculations by WinEQNMR Version 1.20 by Michael J. Hynes
Program run at 10:51:16   on 06/05/2008

 IDEAL DATA FOR 1:1 COMPLEX USING CHEMICAL SHIFT (TEST11.FIT)
  Reaction:   M + L = ML
 FILE: TEST11.FIT
 IDEAL DATA: K1 = 63.091; DELTA M = 20.0; DELTA ML = 120.0
 File prepared by M. J. Hynes, October 22 2000

NO.  A   PARAMETER    DELTA     ERROR    CONDITION   DESCRIPTION
   1  1  3.83053E+03 2.000E-01 2.194E+02 1.237E+00    K1
   2  1  6.55005E+00 2.000E-01 1.617E-02 1.151E+00   SHIFT M
   3  1  7.91079E+00 1.000E+00 3.796E-03 1.283E+00    SHIFT ML

ORMS ERROR = 1.26E-02  MAX ERROR = 3.25E-02 AT OBS.NO.  5
 RESIDUALS SQUARED = 2.24E-03
 RFACTOR =     0.1474 PERCENT
```

Figure S33 NMR titration of compound **3** *vs*. $TBAH_2PO_4$ in $[D_6]DMSO/H_2O$ 0.5%.



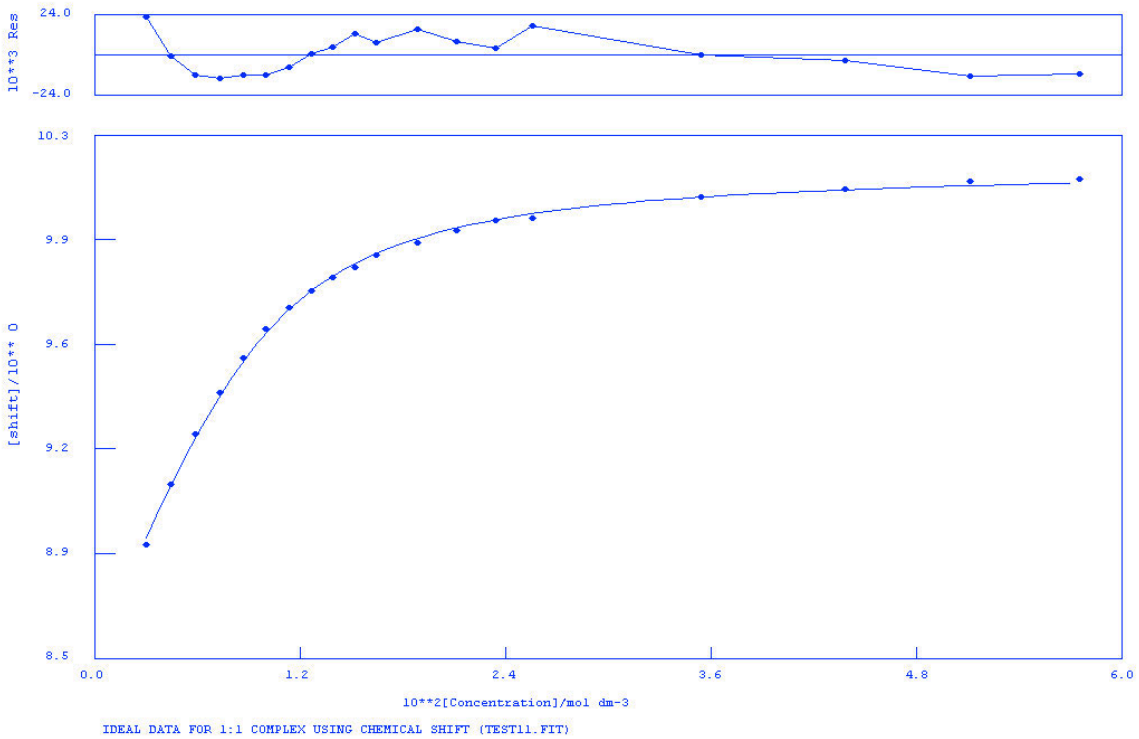

```
Program run at 11:34:02   on 06/05/2008

 IDEAL DATA FOR 1:1 COMPLEX USING CHEMICAL SHIFT (TEST11.FIT)
  Reaction:   M + L = ML
 FILE: TEST11.FIT
 IDEAL DATA: K1 = 63.091; DELTA M = 20.0; DELTA ML = 120.0
 File prepared by M. J. Hynes, October 22 2000

NO.  A   PARAMETER    DELTA     ERROR    CONDITION   DESCRIPTION
   1  1  1.28327E+02 2.000E-01 1.244E+01 2.397E+01    K1
   2  1  6.87329E+00 2.000E-01 4.134E-03 4.853E+00   SHIFT M
   3  1  7.18638E+00 1.000E+00 7.218E-03 1.429E+01    SHIFT ML

0RMS ERROR = 3.79E-03  MAX ERROR = 6.81E-03 AT OBS.NO. 12
 RESIDUALS SQUARED = 1.58E-04
 RFACTOR =     0.0478 PERCENT
```

Figure S34 NMR titration of compound **3** *vs*. TBACl in [D_6]DMSO/H_2O 0.5%.



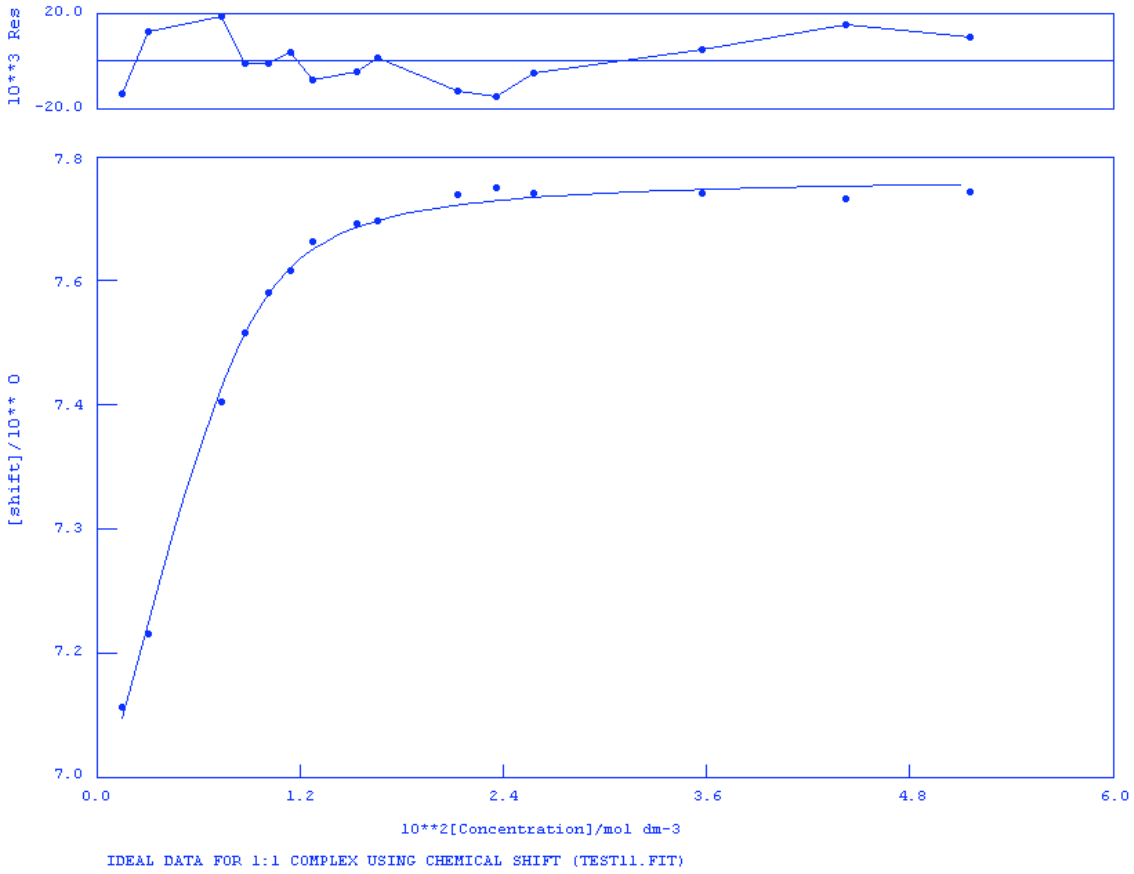

```
Calculations by WinEQNMR Version 1.20 by Michael J. Hynes
Program run at 19:47:48   on 06/26/2008

 IDEAL DATA FOR 1:1 COMPLEX USING CHEMICAL SHIFT (TEST11.FIT)
  Reaction:   M + L = ML
 FILE: TEST11.FIT
 IDEAL DATA: K1 = 63.091; DELTA M = 20.0; DELTA ML = 120.0
 File prepared by M. J. Hynes, October 22 2000

NO.  A   PARAMETER    DELTA     ERROR    CONDITION   DESCRIPTION
   1  1  5.44643E+02 2.000E-01 2.416E+01 8.008E+00    K1
   2  1  8.49533E+00 2.000E-01 1.318E-02 2.509E+00   SHIFT M
   3  1  1.01960E+01 1.000E+00 8.356E-03 5.295E+00    SHIFT ML

0RMS ERROR = 1.21E-02  MAX ERROR = 2.21E-02 AT OBS.NO.  1
 RESIDUALS SQUARED = 2.35E-03
 RFACTOR =     0.1141 PERCENT
```

Figure S35 NMR titration of compound **3** *vs*. $TEAHCO_3$ in $[D_6]DMSO/H_2O$ 0.5%.



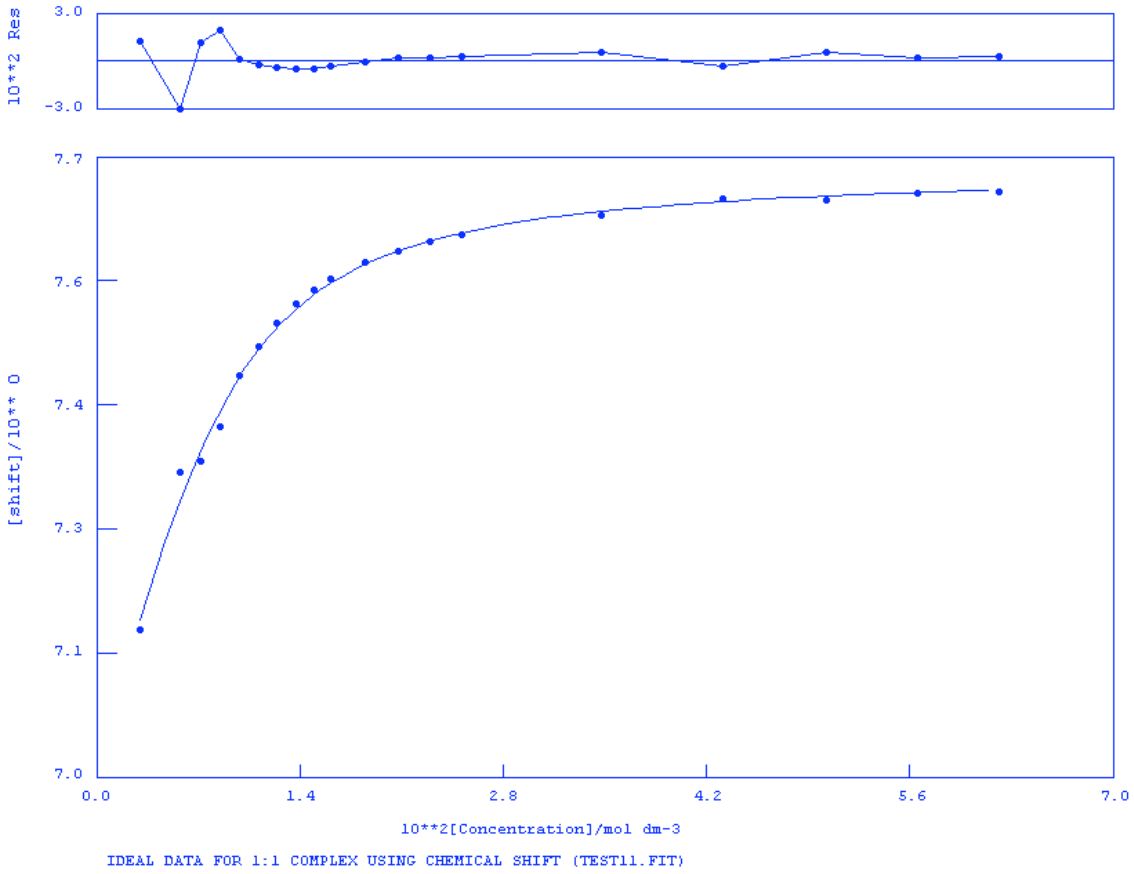

```
Calculations by WinEQNMR Version 1.20 by Michael J. Hynes
Program run at 15:48:43   on 06/09/2008

 IDEAL DATA FOR 1:1 COMPLEX USING CHEMICAL SHIFT (TEST11.FIT)
  Reaction:   M + L = ML
 FILE: TEST11.FIT
 IDEAL DATA: K1 = 63.091; DELTA M = 20.0; DELTA ML = 120.0
 File prepared by M. J. Hynes, October 22 2000

NO.  A   PARAMETER    DELTA     ERROR    CONDITION   DESCRIPTION
   1  1  1.62402E+03 2.000E-01 1.970E+02 4.053E+00    K1
   2  1  6.94904E+00 2.000E-01 1.073E-02 1.302E+00   SHIFT M
   3  1  7.72691E+00 1.000E+00 6.581E-03 3.614E+00    SHIFT ML

0RMS ERROR = 1.13E-02  MAX ERROR = 1.80E-02 AT OBS.NO.  3
 RESIDUALS SQUARED = 1.54E-03
 RFACTOR =     0.1335 PERCENT
```

Figure S36 NMR titration of compound **4** *vs*. TBAOAc in [D_6]DMSO/H_2O 0.5%.



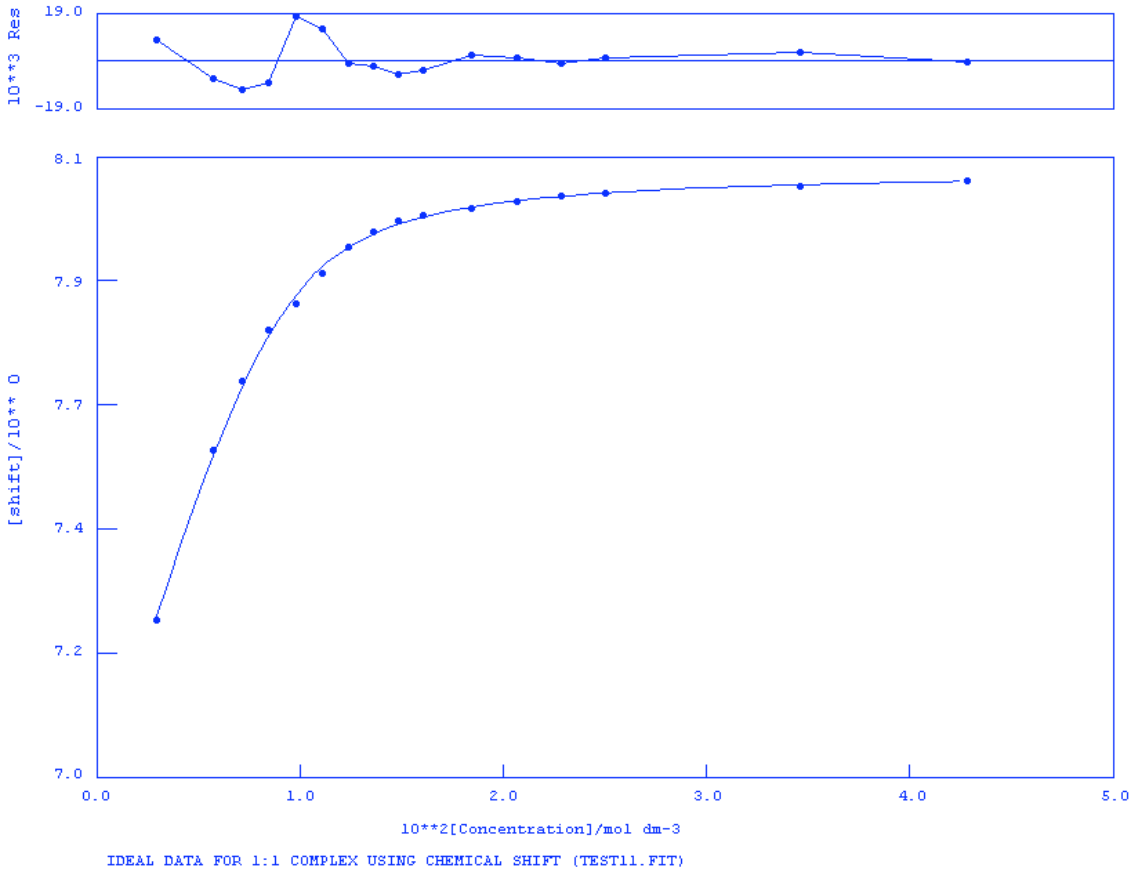

```
Calculations by WinEQNMR Version 1.20 by Michael J. Hynes
Program run at 16:48:58   on 06/03/2008

 IDEAL DATA FOR 1:1 COMPLEX USING CHEMICAL SHIFT (TEST11.FIT)
  Reaction:   M + L = ML
 FILE: TEST11.FIT
 IDEAL DATA: K1 = 63.091; DELTA M = 20.0; DELTA ML = 120.0
 File prepared by M. J. Hynes, October 22 2000

NO.  A   PARAMETER    DELTA     ERROR    CONDITION   DESCRIPTION
   1  1  4.76840E+02 2.000E-01 3.625E+01 6.774E+00    K1
   2  1  7.00902E+00 2.000E-01 1.388E-02 3.073E+00   SHIFT M
   3  1  7.68736E+00 1.000E+00 5.912E-03 3.719E+00    SHIFT ML

0RMS ERROR = 1.05E-02  MAX ERROR = 3.06E-02 AT OBS.NO.  2
 RESIDUALS SQUARED = 1.76E-03
 RFACTOR =     0.1279 PERCENT
```

Figure S37 NMR titration of compound **4** *vs*. TBAOBz in [D6]DMSO/H_2O 0.5%.



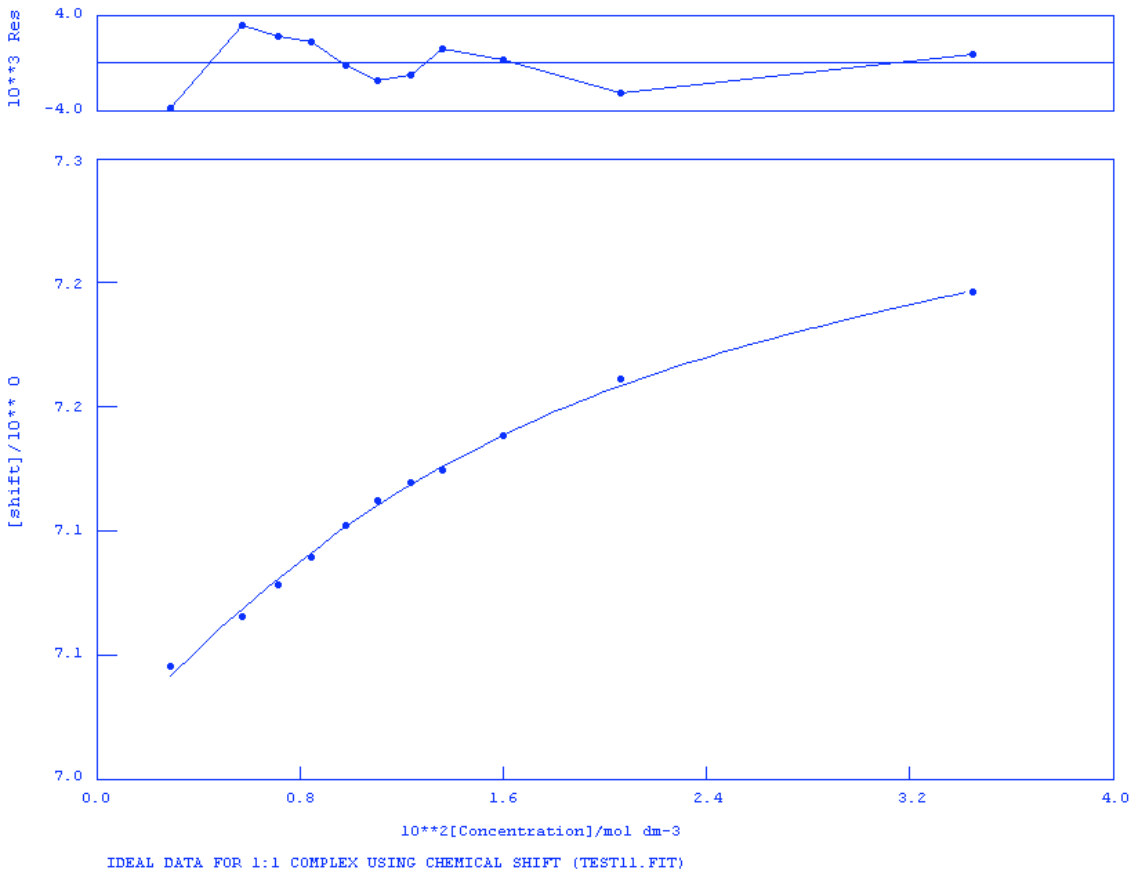

```
Calculations by WinEQNMR Version 1.20 by Michael J. Hynes
Program run at 16:32:46   on 06/03/2008

 IDEAL DATA FOR 1:1 COMPLEX USING CHEMICAL SHIFT (TEST11.FIT)
  Reaction:   M + L = ML
 FILE: TEST11.FIT
 IDEAL DATA: K1 = 63.091; DELTA M = 20.0; DELTA ML = 120.0
 File prepared by M. J. Hynes, October 22 2000

NO.  A   PARAMETER    DELTA     ERROR    CONDITION   DESCRIPTION
   1  1  1.62518E+03 2.000E-01 1.065E+02 6.079E+00    K1
   2  1  6.95471E+00 2.000E-01 1.087E-02 1.788E+00   SHIFT M
   3  1  8.07554E+00 1.000E+00 5.184E-03 4.746E+00    SHIFT ML

0RMS ERROR = 8.09E-03  MAX ERROR = 1.71E-02 AT OBS.NO.  5
 RESIDUALS SQUARED = 8.51E-04
 RFACTOR =     0.0925 PERCENT
```

Figure S38 NMR titration of compound **4** *vs*. $TBAH_2PO_4$ in $[D_6]DMSO/H_2O$ 0.5%.




```
Calculations by WinEQNMR Version 1.20 by Michael J. Hynes
Program run at 16:09:47   on 06/05/2008

 IDEAL DATA FOR 1:1 COMPLEX USING CHEMICAL SHIFT (TEST11.FIT)
  Reaction:   M + L = ML
 FILE: TEST11.FIT
 IDEAL DATA: K1 = 63.091; DELTA M = 20.0; DELTA ML = 120.0
 File prepared by M. J. Hynes, October 22 2000

NO.  A   PARAMETER    DELTA     ERROR    CONDITION   DESCRIPTION
   1  1  7.43907E+01 2.000E-01 5.838E+00 2.080E+01    K1
   2  1  7.00869E+00 2.000E-01 2.168E-03 3.876E+00   SHIFT M
   3  1  7.28516E+00 1.000E+00 7.204E-03 1.661E+01    SHIFT ML

0RMS ERROR = 2.35E-03  MAX ERROR = 3.88E-03 AT OBS.NO.  1
 RESIDUALS SQUARED = 4.43E-05
 RFACTOR =     0.0282 PERCENT
```

Figure S39 NMR titration of compound **4** *vs*. TBACl in [D_6]DMSO/H_2O 0.5%.

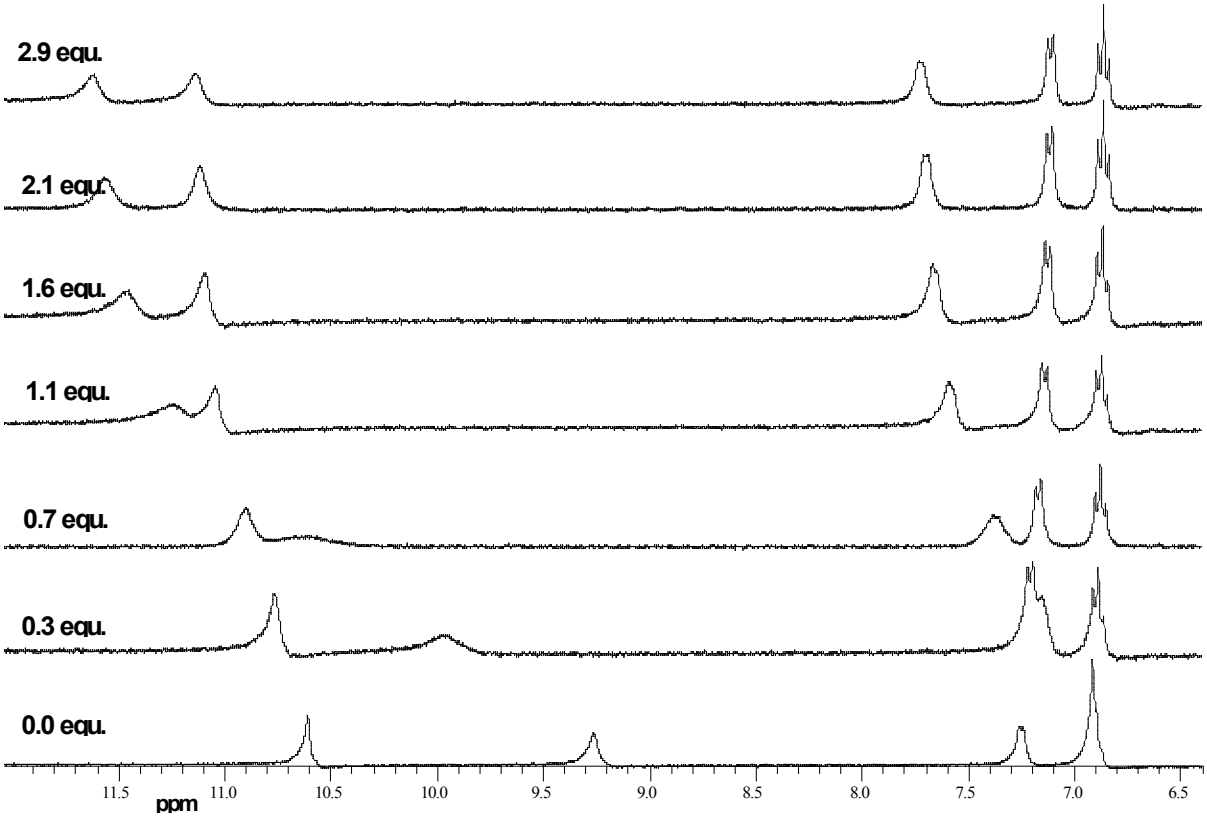


Figure S40 NMR Stack plot of compound **3** *vs* TBAOAc in [D_6]DMSO/H_2O 0.5%.

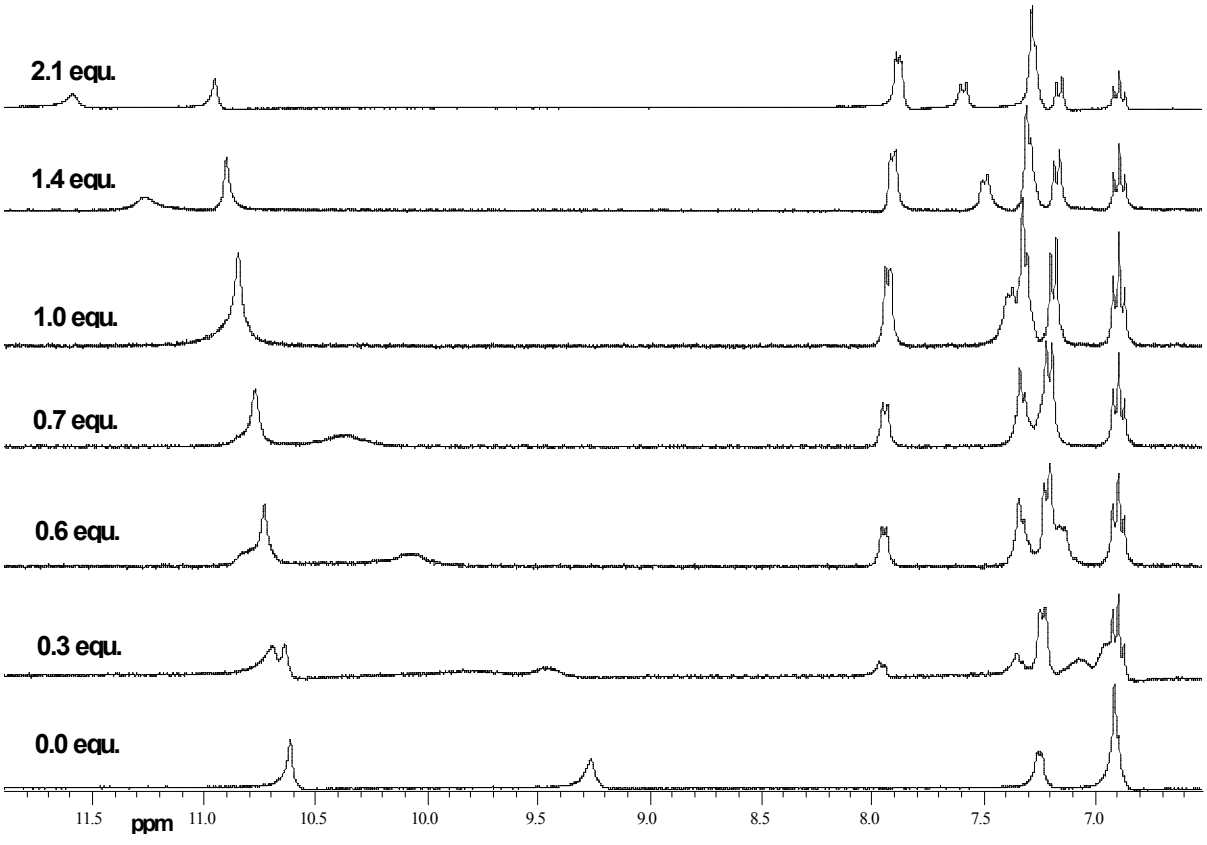


Figure S41 NMR Stack plot of compound **3** *vs* TBAOBz in [D_6]DMSO/H_2O 0.5%.

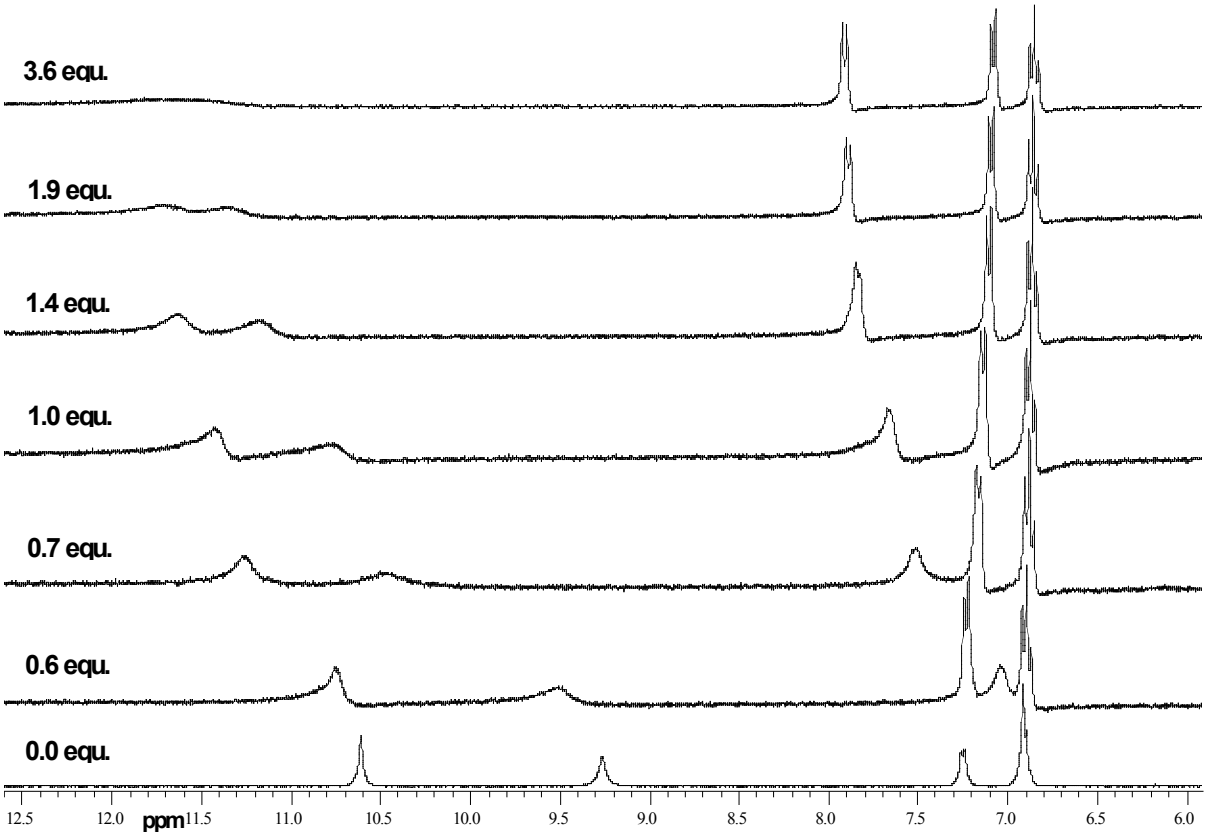


Figure S42 NMR Stack plot of compound **3** *vs* $TBAH_2PO_4$ in $[D_6]DMSO/H_2O$ 0.5%.

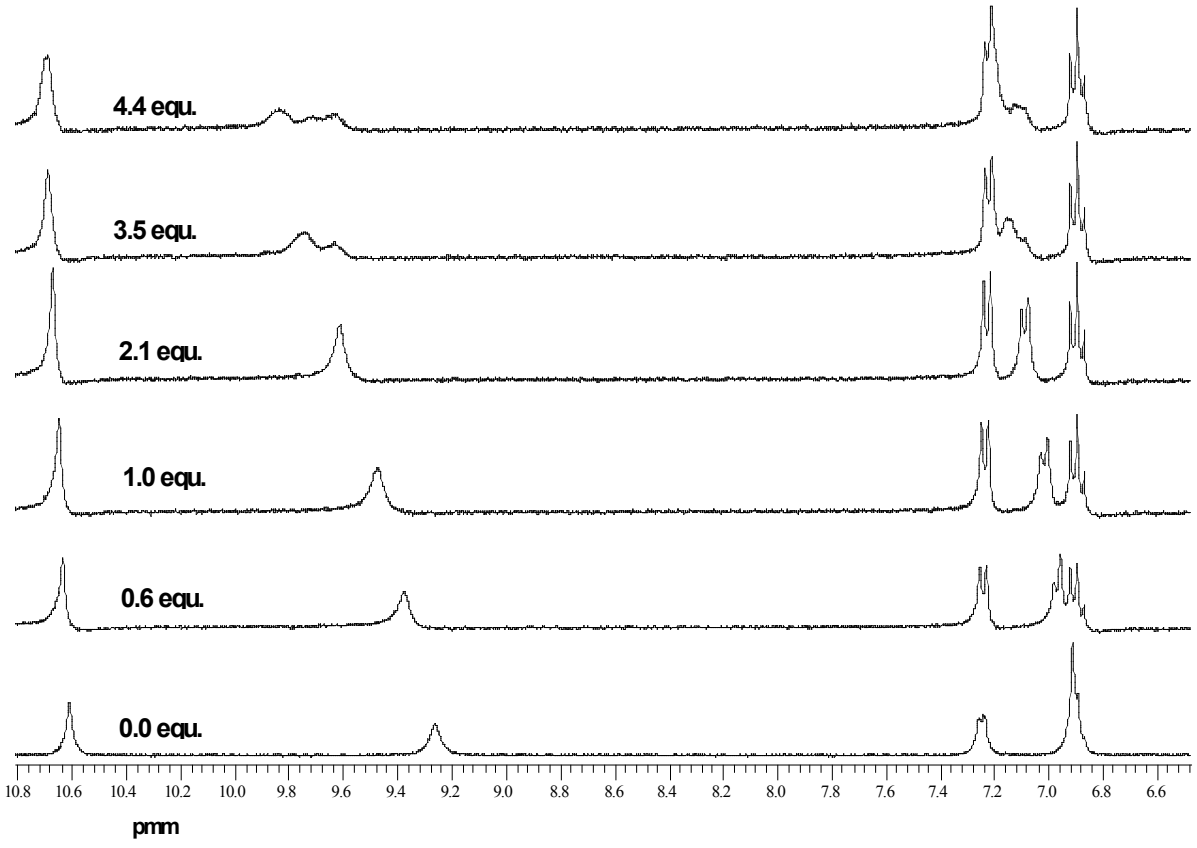


Figure S43 NMR Stack plot of compound **3** *vs* TBACl in $[D_6]DMSO/H_2O$ 0.5%.

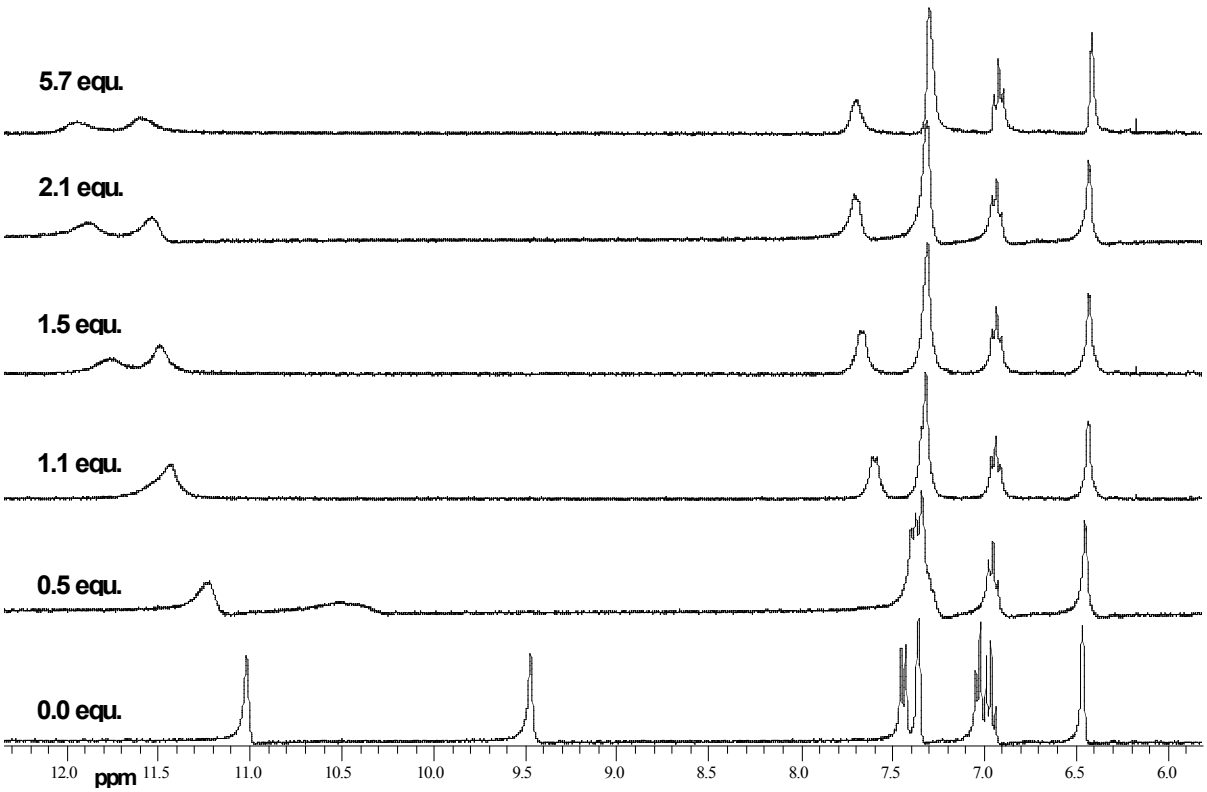


Figure S44 NMR Stack plot of compound **4** *vs* TBAAcO in [D_6]DMSO/H_2O 0.5%.

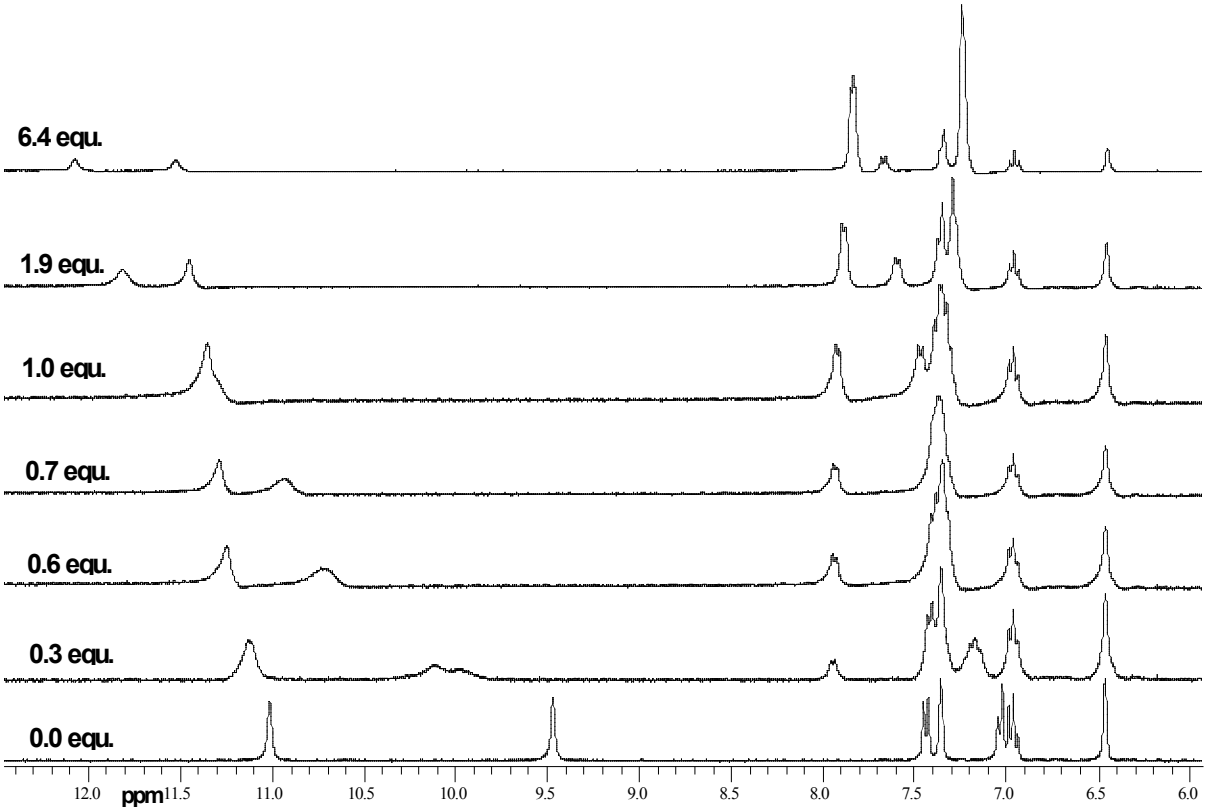


Figure S45 NMR Stack plot of compound **4** *vs* TBABzO in [D_6]DMSO/H_2O 0.5%.

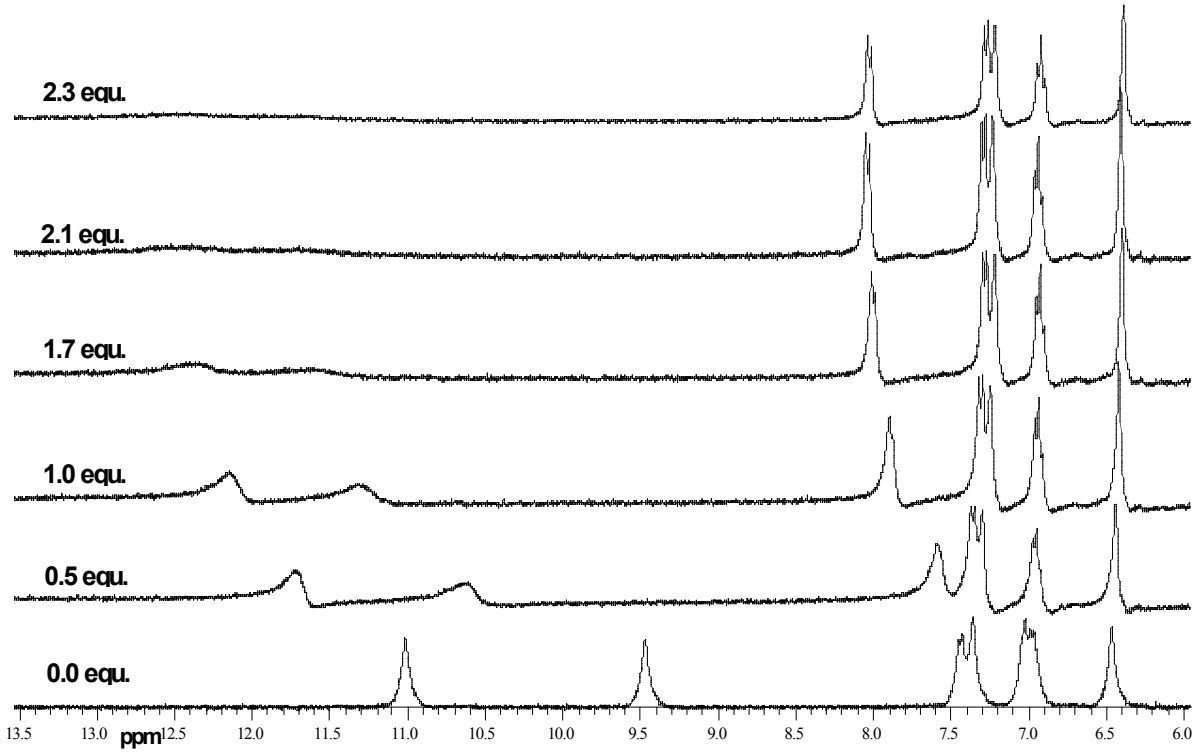


Figure S46 NMR Stack plot of compound **4** *vs* $TBAH_2PO_4$ in $[D_6]DMSO/H_2O$ 0.5%.

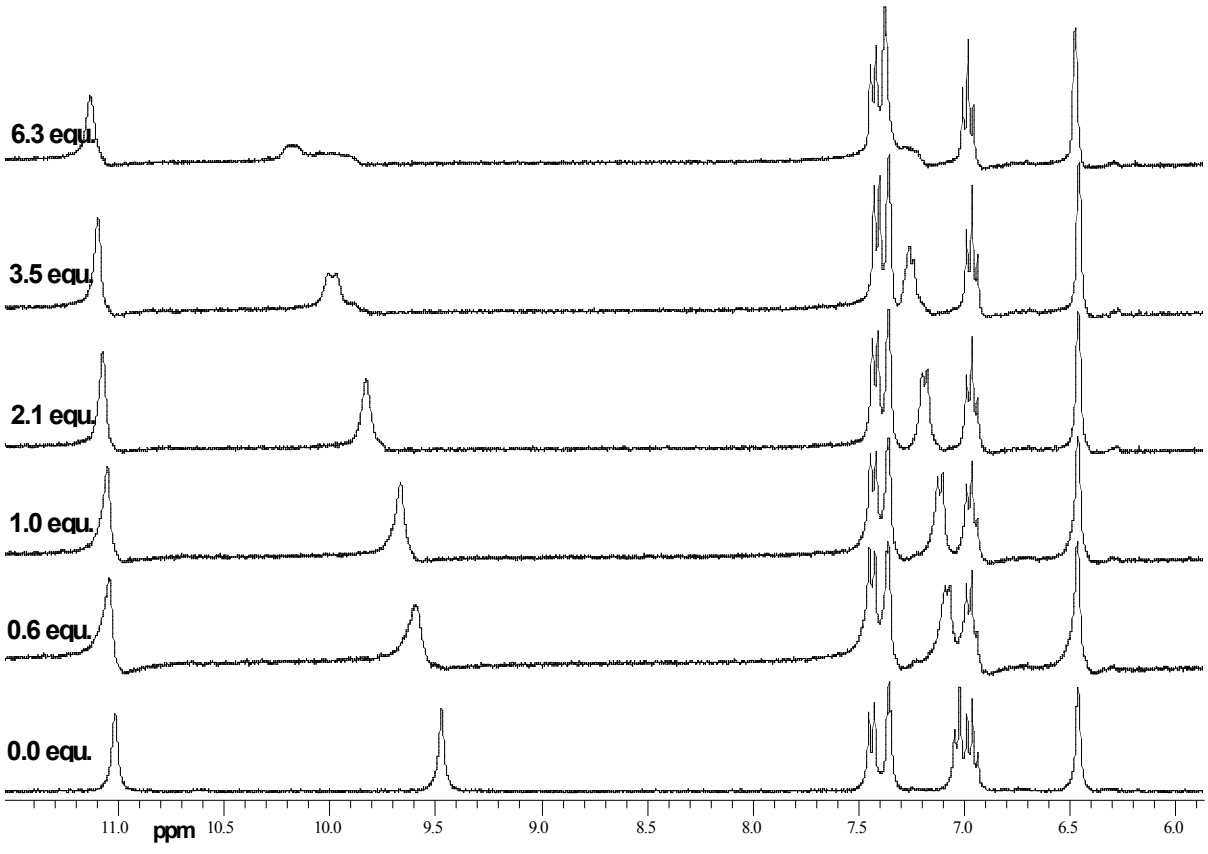


Figure S47 NMR Stack plot of compound **4** *vs* TBACl in $[D_6]DMSO/H_2O$ 0.5%.

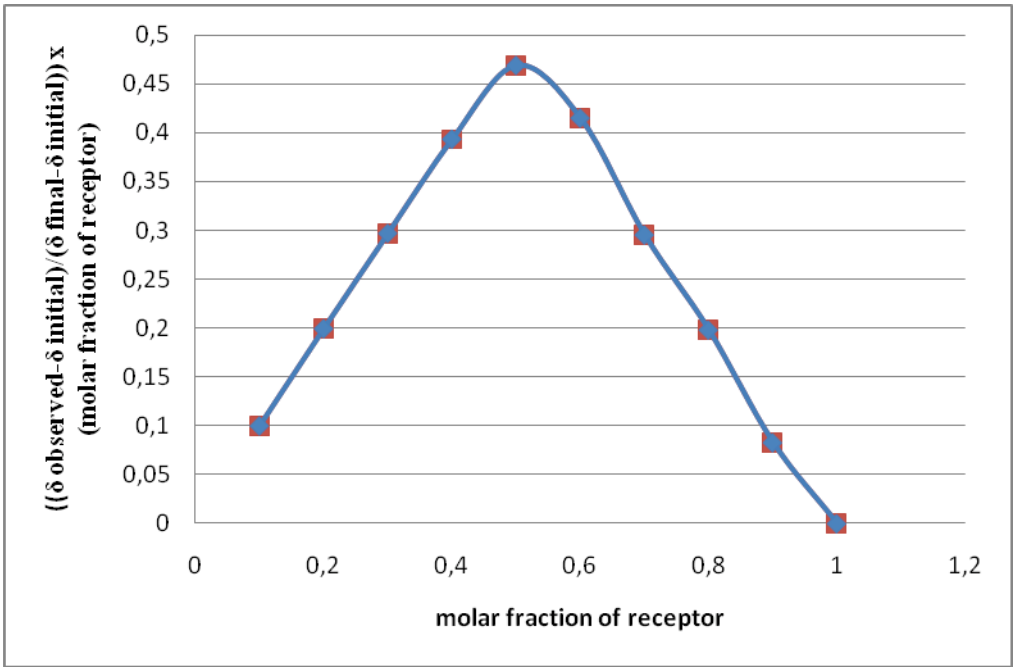


Figure S48 Job plot of compound **1** *vs* $TBAH_2PO_4$ in $[D_6]DMSO/H_2O$ 0.5%.

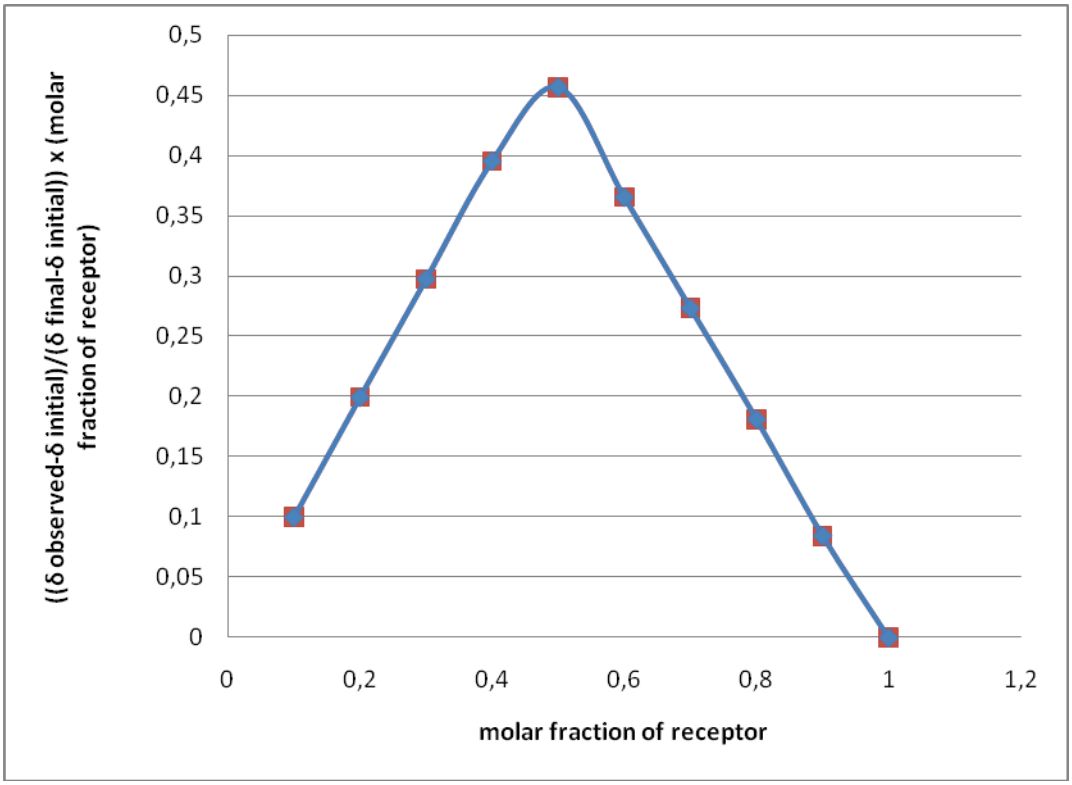


Figure S49 Job plot of compound **2** *vs* $TBAH_2PO_4$ in $[D_6]DMSO/H_2O$ 0.5%.

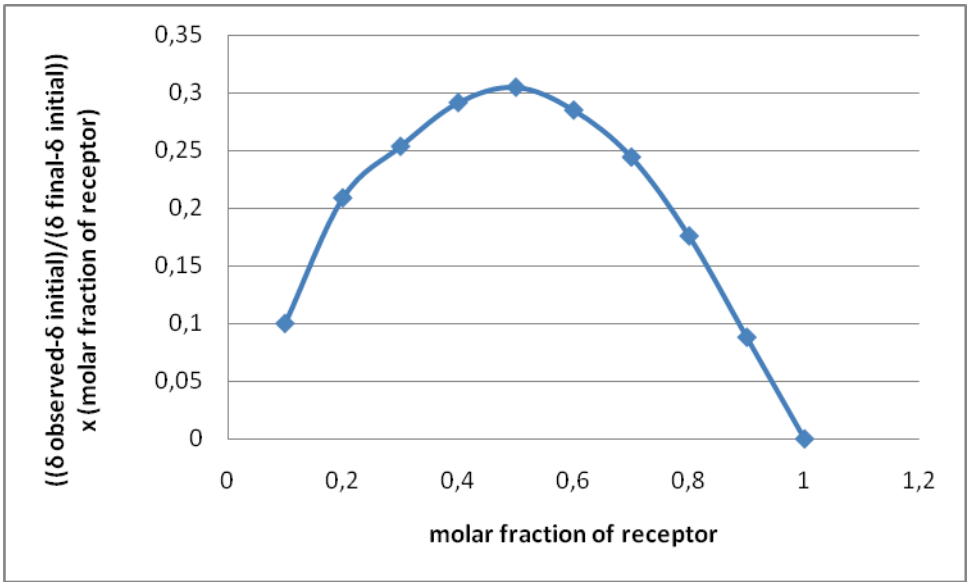


Figure S50 Job plot of compound **2** *vs* $TBAH_2PO_4$ in $[D_6]DMSO/H_2O$ 25%.

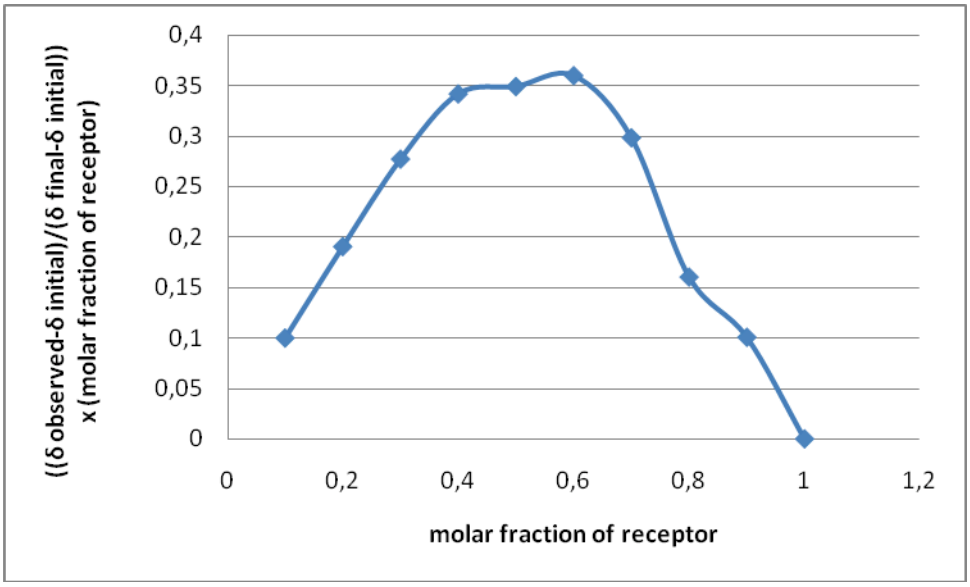


Figure S51 Job plot of compound **2** *vs* $TEAHCO_3$ in $[D_6]DMSO/H_2O$ 10%.

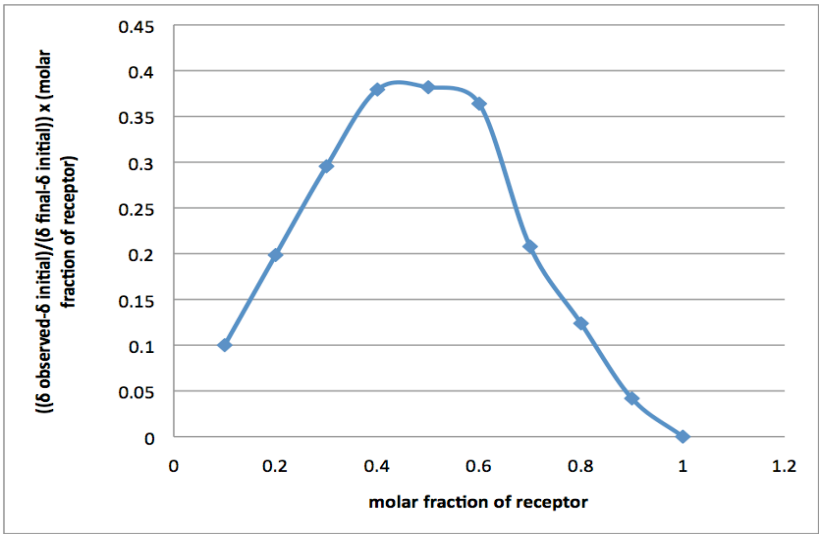


Figure S52 Job plot of compound **3** with TBA H_2PO_4 in [D6]DMSO

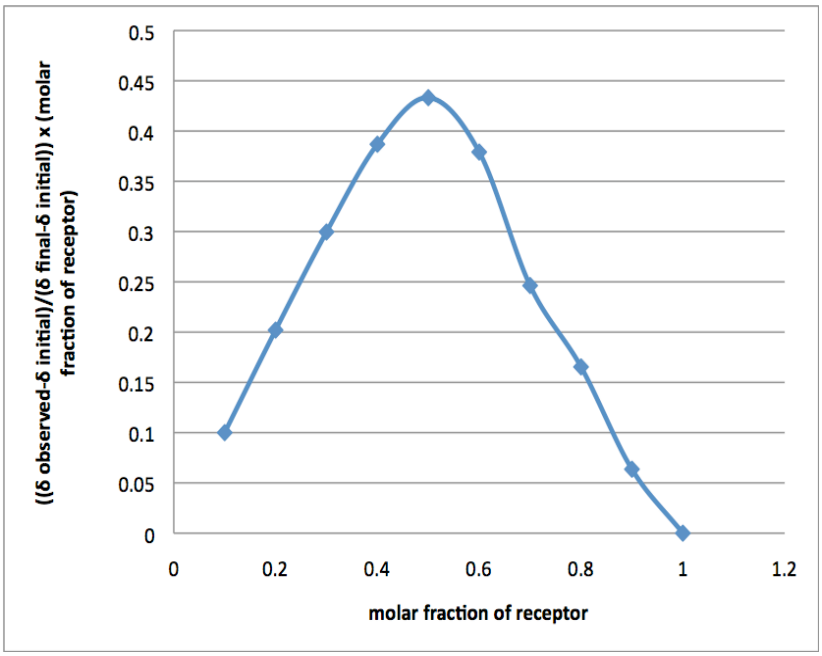


Figure S53 Job plot of compound **4** with TBA H_2PO_4 in [D6]DMSO

Isothiocyanate intermediates were very reactive and crude material was used in the synthesis of compounds **3** and **4** directly after preparation. However some characterisation data is available for the isothiocyanate intermediates: (isothiocyanate intermediate for compound **3**) Yield 62%; ^{1}H NMR (300 MHz, DMSO-d_6): δ:2.15 (s, CH_3 ,3H), 2.33 (s, CH_3 ,3H), 6.96 (m, 1H), 7.13 (d, 1H), 7.39 (d, 1H), 11.32 (s, NH indole, 2H); ^{13}C NMR (75 MHz, DMSO-d_6): δ: 8.3 (CH_3), 11.1 (CH_3), 106.5 (C), 112.6 (C), 117.7 (CH), 118.5 (CH), 118.6 (CH), 129.6 (C), 130.9 (C), 132.6 (C), 133.4 (CS) LRMS (ES^-):m/z: 201 $[M-H]^-$

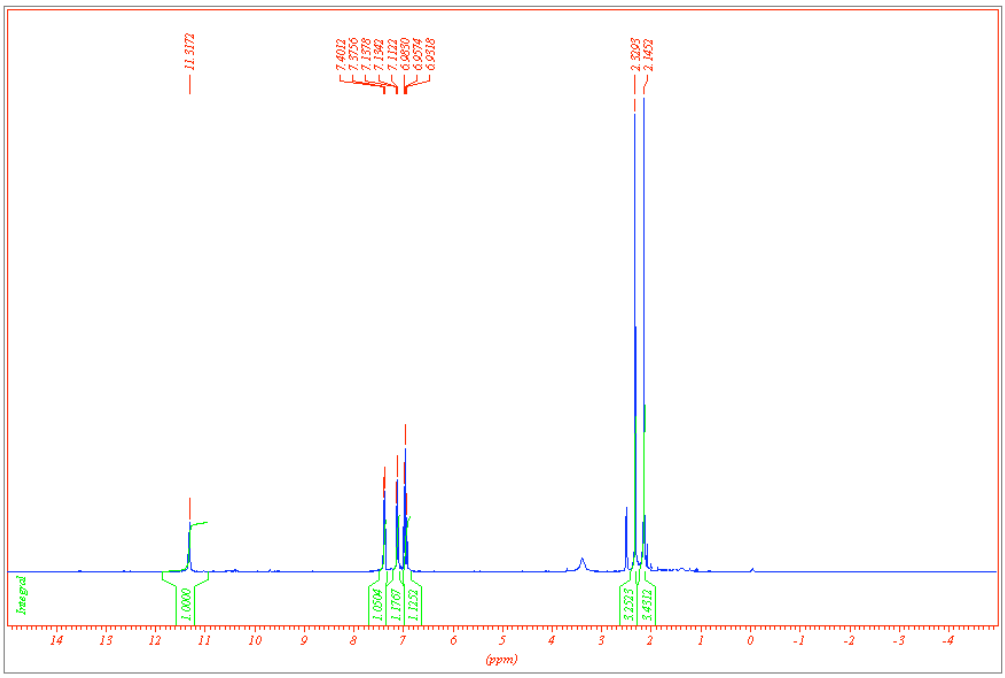


Figure S54 ^{1}H NMR of isothiocyanate intermediate used to synthesise compound **3**.

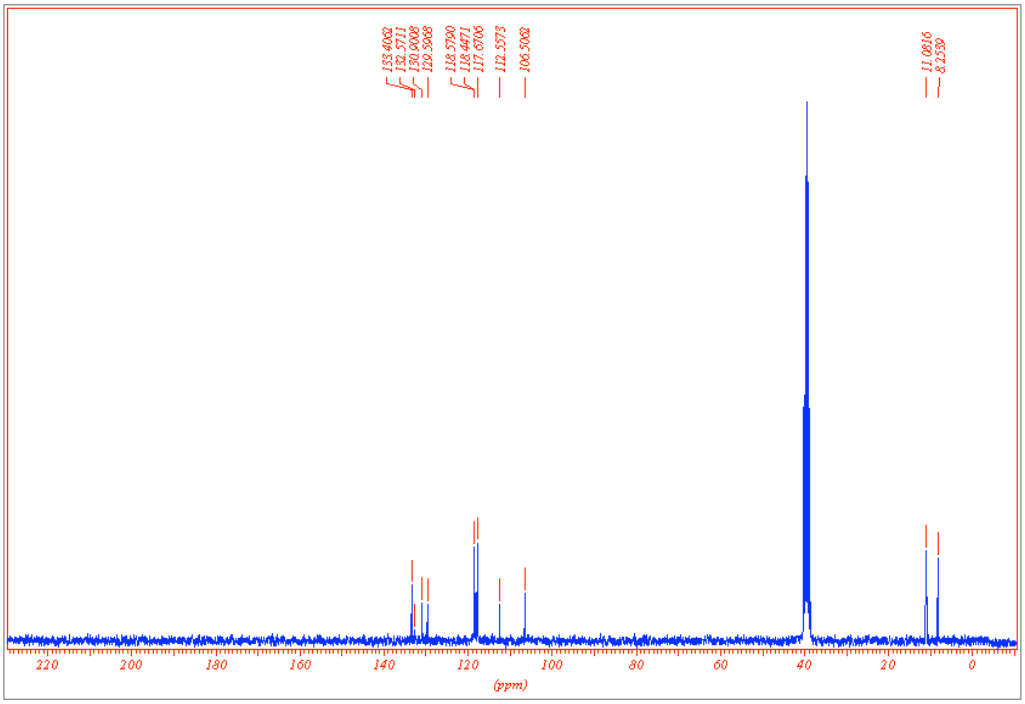


Figure S55 ^{13}C NMR of isothiocyanate intermediate used to synthesise compound **3**.

Isothiocyanate intermediates were very reactive and crude material was used in the synthesis of compounds **3** and **4** directly after preparation. However some characterisation data is available for the isothiocyanate intermediates: (isothiocyanate intermediate for compound **4**) Yield 79 %; ^{1}H NMR (300 MHz, DMSO-d_6): δ: 6.55 (s, 1H), 7.02 (m, 1H), 7.22 (d, 1H), 7.42 (d, 1H), 7.58 (d, 2H), 11.78 (s, NH indole, 2H); ^{13}C NMR (75 MHz, DMSO-d_6): δ: 102.4 (CH), 113.9 (C), 119.4 (CH), 119.6 (CH), 120.4 (CH), 126.8 (CH), 129.8 (C), 130.3 (C), 133.5 (CS); LRMS (ES$^-$):m/z: 173 [M-H]$^-$

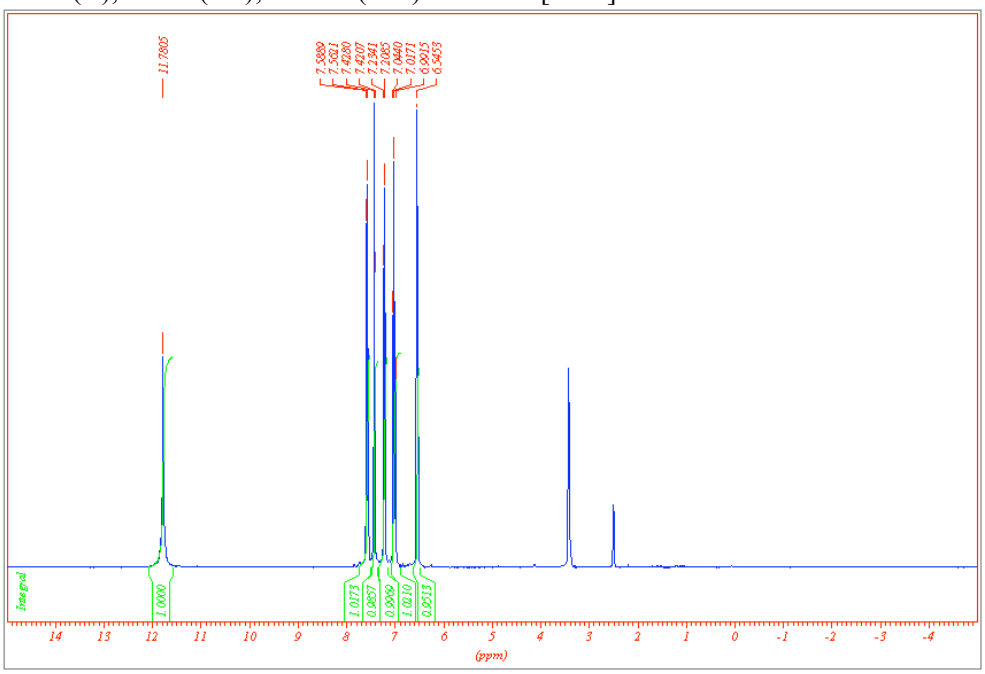


Figure S56 ^{1}H NMR of isothiocyanate intermediate used to synthesise compound **4**.

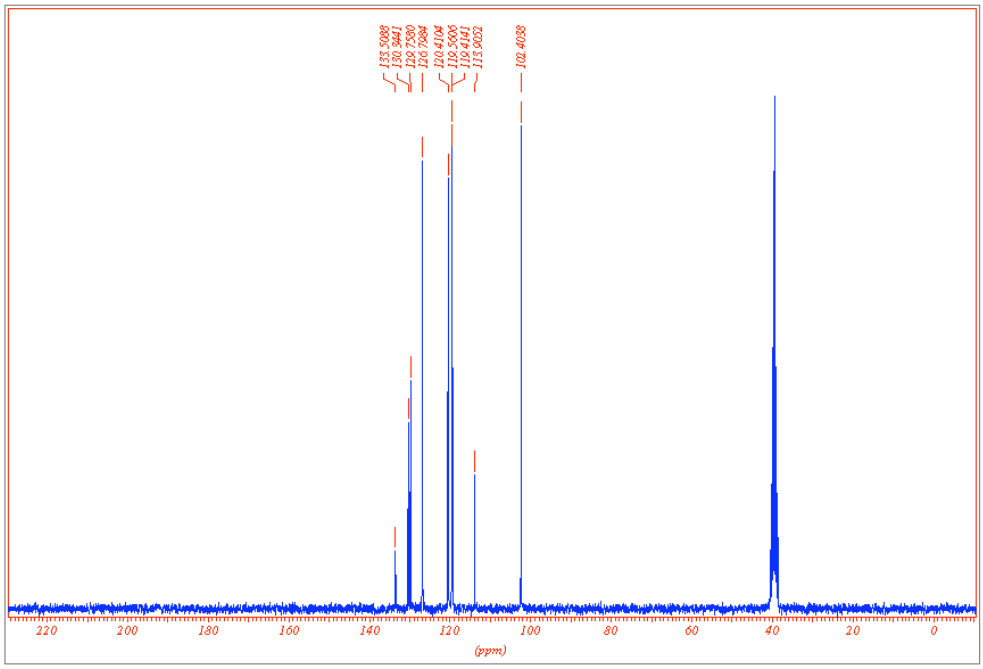


Figure S57 ^{13}C NMR of isothiocyanate intermediate used to synthesise compound **4**.