Diindolylureas: high affinity dihydrogenphosphate receptors

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Electronic Supplementary Information

Supplementary information

Experimental Section

General remarks: All reactions were performed in oven-dried glassware under a slight positive pressure of nitrogen. 2,3-Dimethyl-7-aminoindole was synthesised by literature procedures.[1] 7-aminoindole is commercially available. ^{1}H-NMR (300 MHz) and ^{13}C NMR (75 MHz) spectra were determined on a Bruker AV300 spectrometer. Chemical shifts for ^{1}H NMR are reported in parts per million (ppm), calibrated to the residual solvent peak set, with coupling constants reported in Hertz (Hz). The following abbreviations are used for spin multiplicity: s = singlet, d = doublet, t = triplet. Chemical shifts for ^{13}C NMR are reported in ppm, relative to the central line of a septet at δ = 39.52 ppm for deuterio-dimethylsulfoxide. Infrared (IR) spectra were recorded on a Mattson Satellite (ATR). FTIR are reported in wavenumbers (cm^{-1}). Elemental analysis were performed by Medac Ltd. All solvents and starting materials were purchased from commercial sources where available. NMR titrations were performed by adding aliquots of the putative anionic guest (as the TBA) salt (0.15 M) in a solution of the receptor (0.01M) in DMSO-d_6 to a solution of the receptor (0.01M).

G.W. Bates, P.A. Gale, M.E. Light, *Chem. Commun.*, 2007, 2121.

1,3-bis (2,3-dimethyl-1*H*-indol-7-yl)urea (2) 2,3-dimethyl-7-aminoindole (0.253 g, 1.58 mmol) was dissolved in a mixture of DCM (30 mL) and a saturated aqueous solution of $NaHCO_3$ (30 mL). Triphosgene (0.47 g, 1.58

mmol) was added in portions and the reaction mixture was left stirring under nitrogen atmosphere at room temperature overnight. The organic layer was washed with water, dried over $MgSO_4$, filtered and concentrated *in vacuo*. The pure product was obtained by recrystalization from hot MeOH. The product was isolated as a white solid: yield 78% 0.21g; m.p. 259°C; ^{1}H NMR (300 MHz, DMSO-d_6): δ: 2.15 (s, 6H), 2.33 (s, 6H), 6.88 (t, J = 7.53 Hz, 2H), 7.03 (d, J = 7.53 Hz, 2H), 7.12 (d, J = 7.53 Hz, 2H), 8.44 (s, NH urea, 2H), 10.31 (s, NH indole, 2H); ^{13}C NMR (75 MHz, DMSO-d_6): δ: 8.51 (CH_3), 11.30 (CH_3), 105.58 (C), 113.08 (CH), 113.13 (CH), 118.24 (CH), 123.06 (C), 128.12 (C), 130.49 (C), 131.08 (C), 153.57 (CO); IR (film): ν = 3392 (indole NH stretching), 3247 (urea NH stretching), 1617 (urea CO stretching); LRMS (ES^-):m/z: 345 $[M-H]^-$; HRMS (ES+) $C_{21}H_{23}N_4O$ m/z/ 347.1866 Found 347.1870 (error -0.90ppm).

1,3-di(1*H*-indol-7-yl)urea (3) 7-aminoindole (0.234 g, 1.58 mmol) was dissolved in a mixture of DCM (20 mL) and a saturated aqueous solution of $NaHCO_3$ (20 mL). Triphosgene (0.47 g, 1.58 mmol) was added in portions and the reaction mixture was left stirring under nitrogen atmosphere at room temperature overnight. The organic layer was washed with water, dried over $MgSO_4$, filtered and concentrated *in vacuo*. The pure product was obtained by recrystallization from MeOH. The product was isolated as a pale grey solid: yield 50% 0.15g; m.p. 252°C; ^{1}H NMR (300 MHz, DMSO-d_6): δ: 6.44 (t, J = 2.64 Hz, 2H), 6.94 (t, J = 7.92, 2H), 7.08 (d, J = 7.14 Hz, 2H), 7.31 (d, J = 7.92, 2H), 7.34 (t, J = 2.64 Hz, 2H), 8.63 (s, NH urea, 2H), 10.77 (s, NH indole, 2H); ^{13}C NMR (75 MHz, DMSO-d_6): δ: 101.42 (CH), 113.67 (CH), 115.85 (CH), 119.00 (CH), 124.06 (C), 125.14 (CH), 129.04 (C), 129.35 (C),

153.57 (CO); IR (film): ν = 3383 (indole NH stretching), 3255 (urea NH stretching), 1620 (urea CO stretching); LRMS (ES^-):m/z: 289 $[M-H]^-$; HRMS (ES+) $C_{17}H_{15}N_4O$ m/z/ 291.1240 Found 291.1236 (error 1.52ppm).

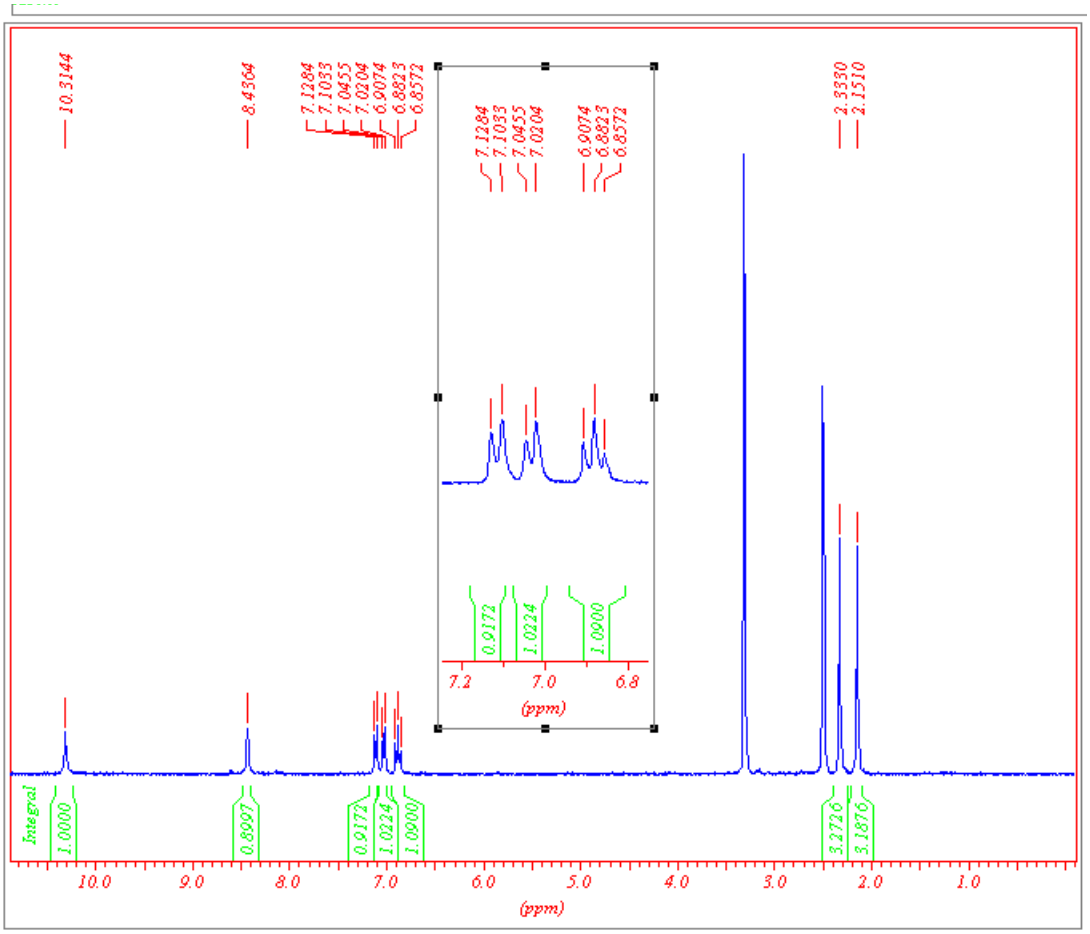


Figure S1 ^{1}H NMR spectrum of compound **2** in DMSO-d_6.

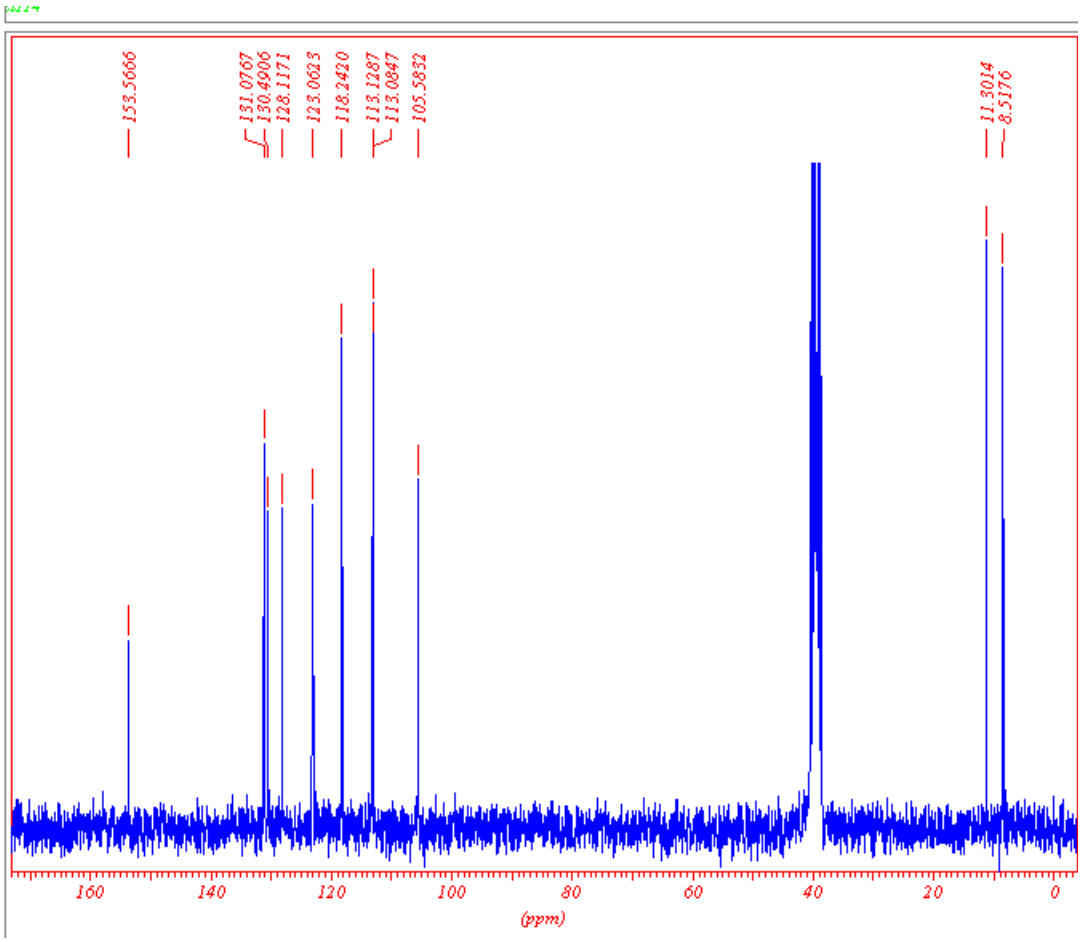


Figure S2 ^{13}C NMR spectrum of compound **2** in DMSO-d_6.

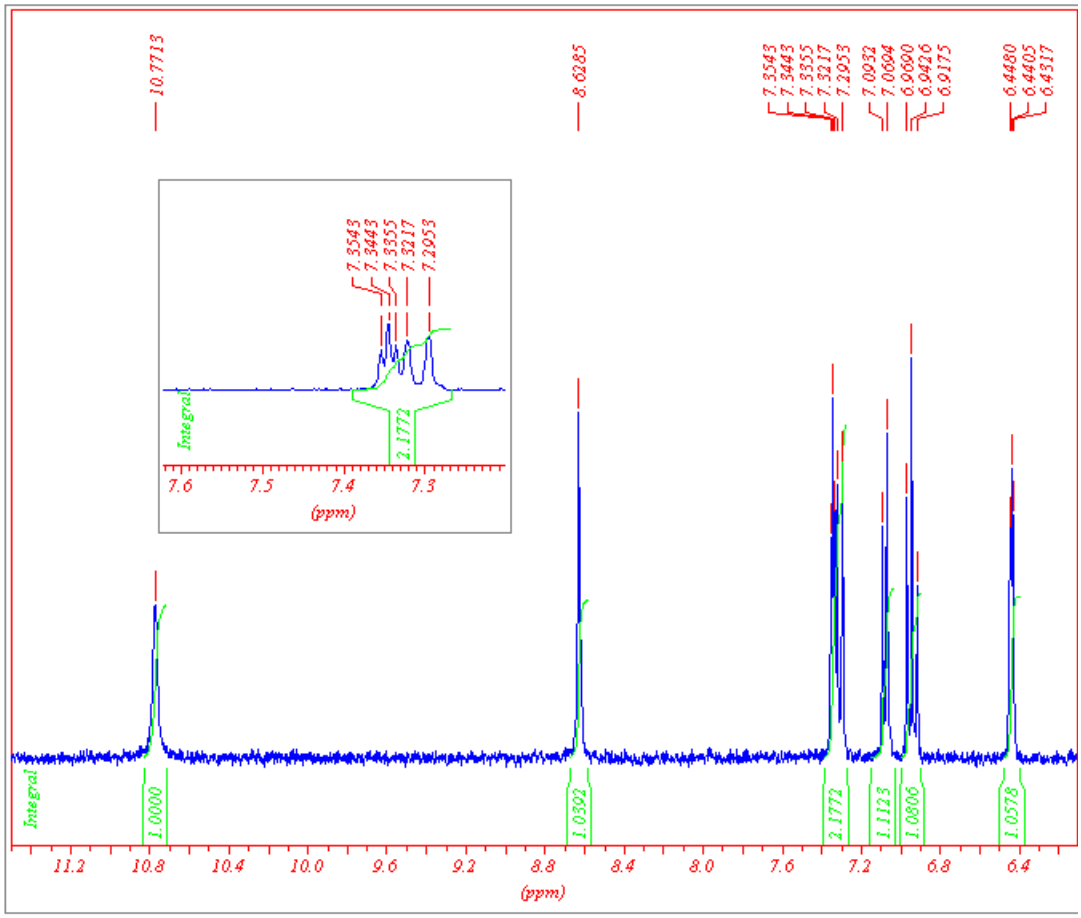


Figure S3 ^{1}H NMR spectrum of compound **3** in DMSO-d_6.

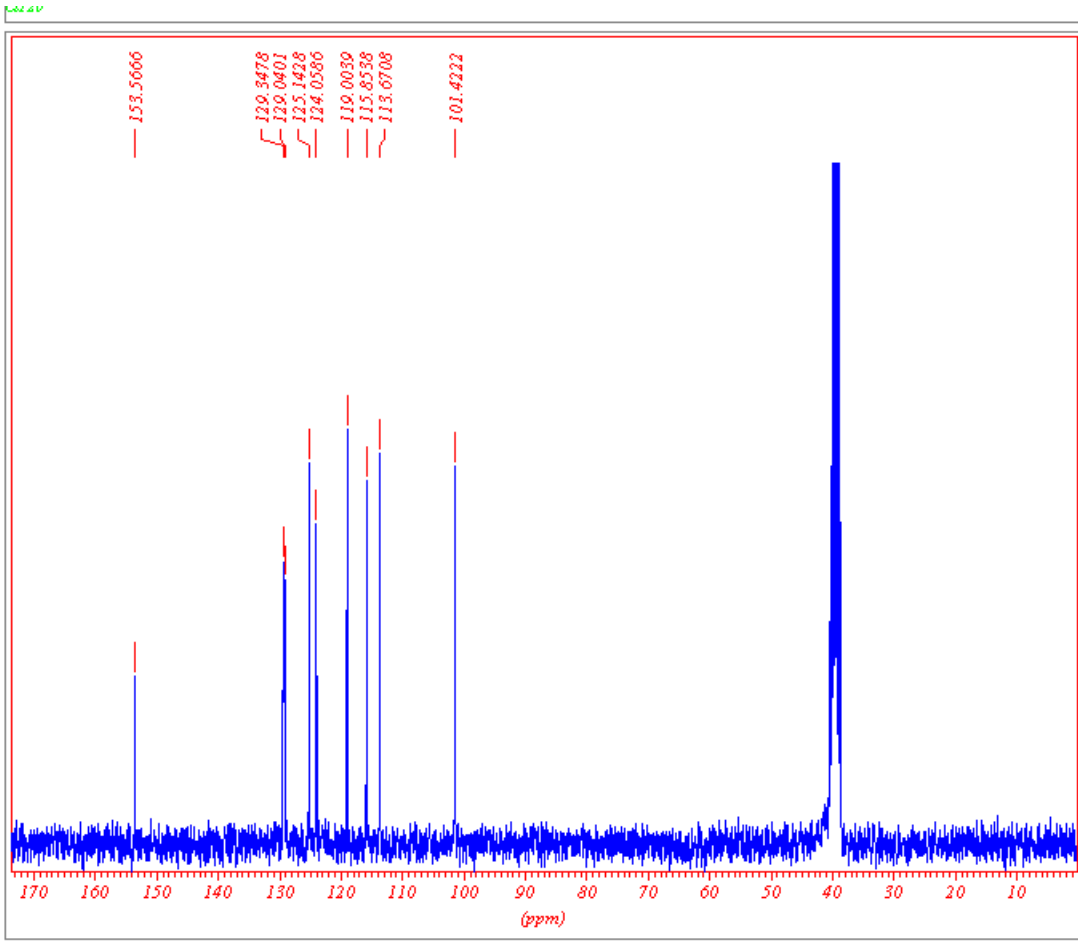


Figure S4 ^{13}C NMR spectrum of compound **3** in DMSO-d_6.

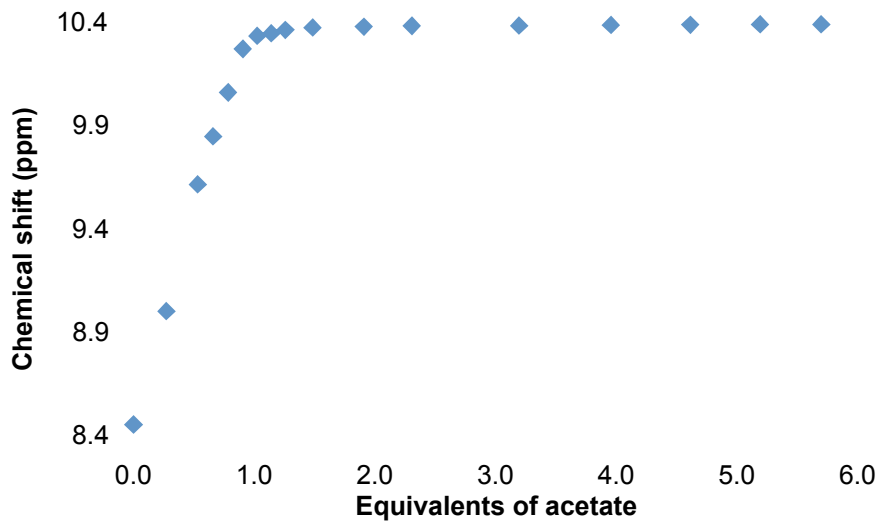


Figure S5 NMR titration of compound **2** *vs*. TBAOAc in DMSO-d_6/H_2O 0.5%.

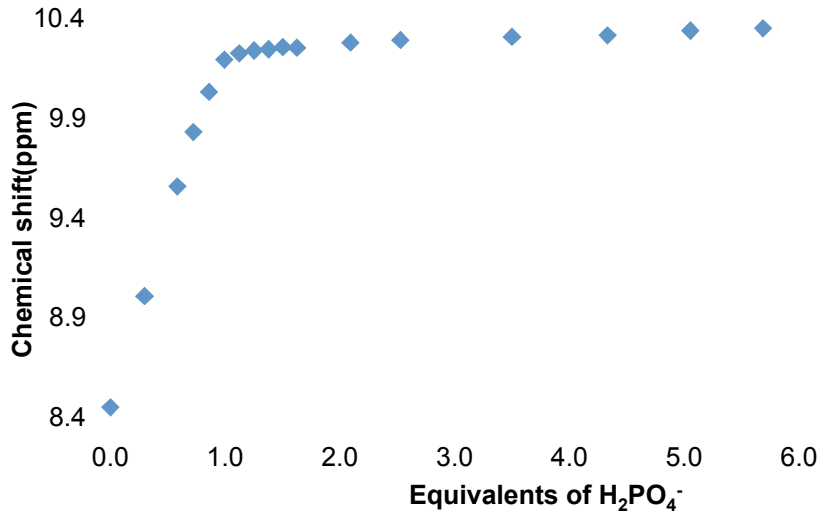
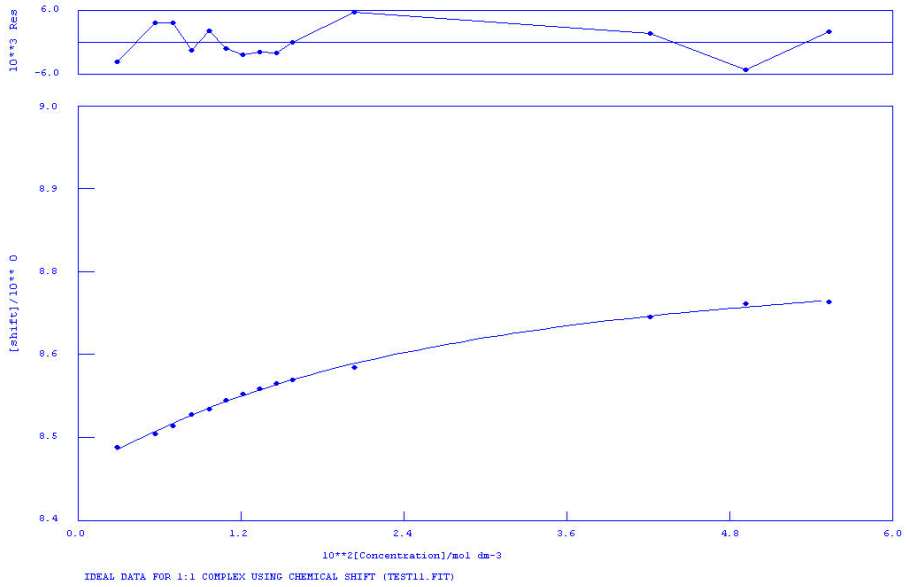


Figure S6 NMR titration of compound **2** *vs*. $TBAH_2PO_4$ in DMSO-d_6/H_2O 0.5%.



Calculations by WinEQNMR Version 1.20 by Michael J. Hynes
Program run at 15:08:42 on 04/02/2008

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

NO.	A	PARAMETER	DELTA	ERROR	CONDITION	DESCRIPTION
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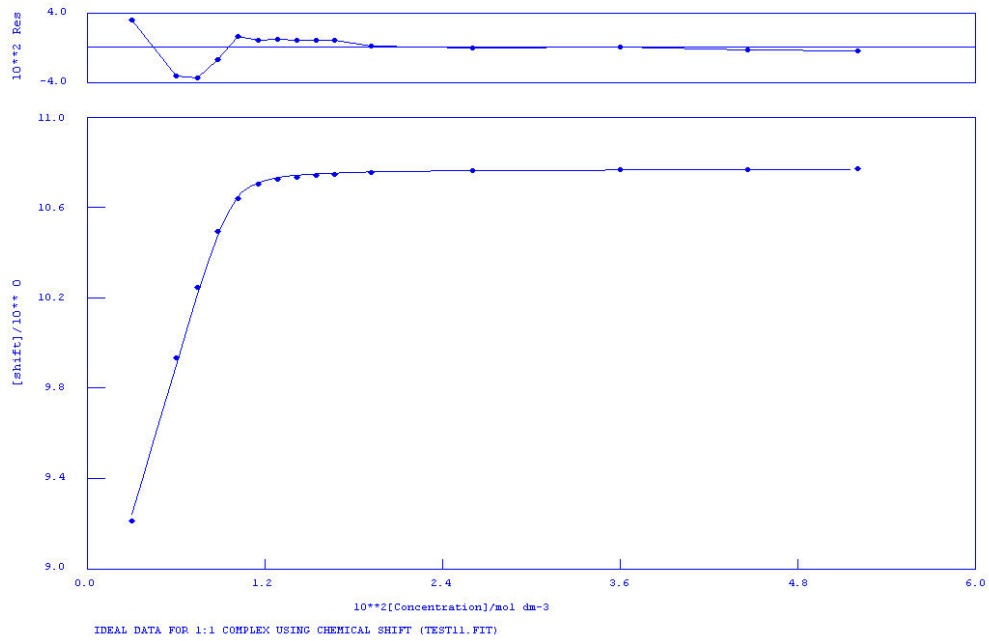
2	1	8.47001E+00	2.000E-01	3.698E-03	7.205E+00	SHIFT M
3	1	8.81874E+00	1.000E+00	1.052E-02	2.267E+01	SHIFT ML

ORMS ERROR = 3.40E-03 MAX ERROR = 5.62E-03 AT OBS.NO. 11
RESIDUALS SQUARED = 1.27E-04
RFACTOR = 0.0350 PERCENT

Figure S7 NMR titration of compound **2** *vs.* $TBAHSO_4$ in DMSO-d_6/H_2O 0.5%.

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Calculations by WinEQNMR Version 1.20 by Michael J. Hynes
Program run at 15:29:31   on 04/01/2008

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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  1.70477E+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

ORMS ERROR = 1.84E-02  MAX ERROR = 3.47E-02 AT OBS.NO.  3
 RESIDUALS SQUARED = 4.06E-03
 RFACTOR =     0.1561 PERCENT
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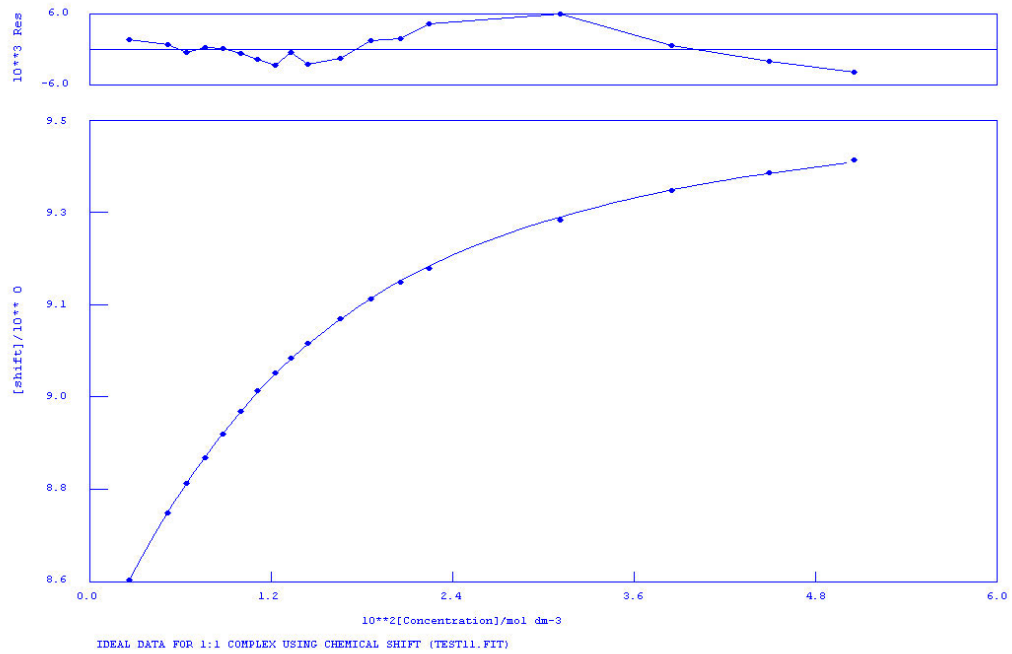
Figure S8 NMR titration of compound **2** *vs.* TBAOBz in DMSO-d_6/H_2O 0.5%.

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Calculations by WinEQNMR Version 1.20 by Michael J. Hynes
Program run at 15:40:22   on 04/01/2008

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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  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

ORMS ERROR = 2.59E-03  MAX ERROR = 5.87E-03 AT OBS.NO. 15
 RESIDUALS SQUARED = 1.00E-04
 RFACTOR =     0.0261 PERCENT
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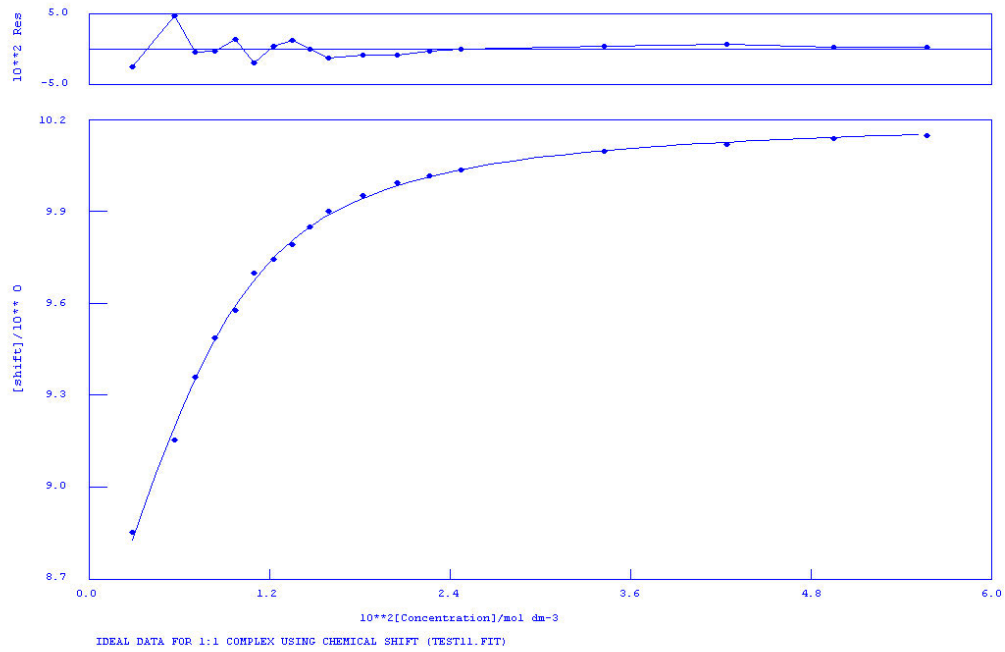
Figure S9 NMR titration of compound **2** *vs.* TBACl in DMSO-d_6/H_2O 0.5%.

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Program run at 19:14:54   on 04/02/2008

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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  5.53576E+02 2.000E-01 3.142E+01 8.459E+00     K1
   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

0RMS ERROR = 1.64E-02  MAX ERROR = 4.66E-02 AT OBS.NO.  2
 RESIDUALS SQUARED = 4.03E-03
 RFACTOR =      0.1530 PERCENT
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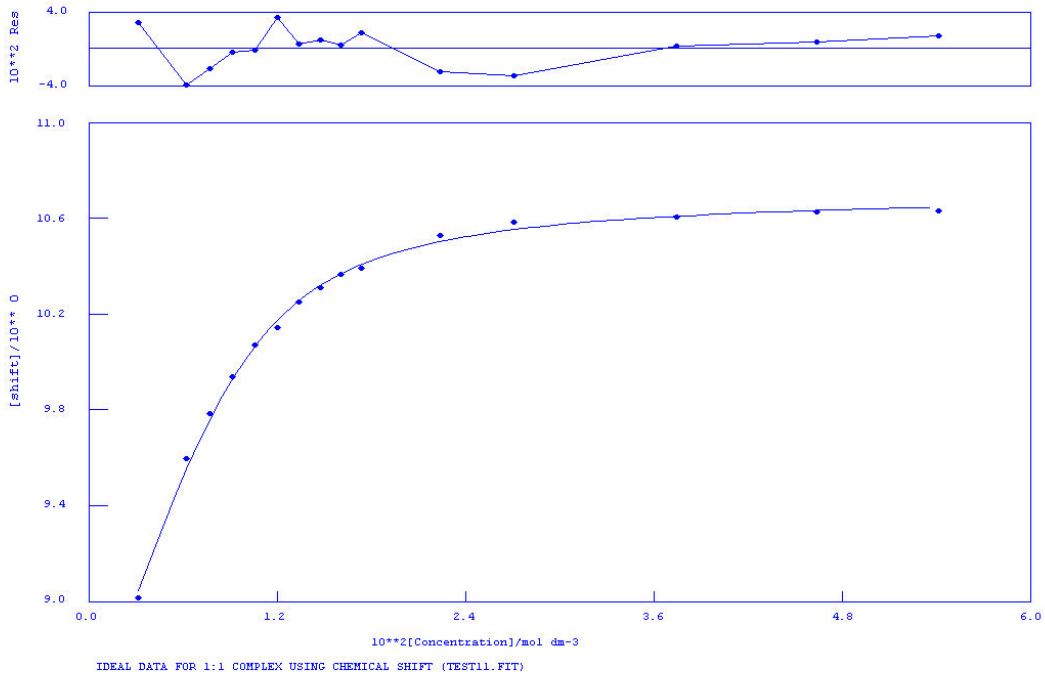
Figure S10 NMR titration of compound **2** *vs.* TBAOAc in DMSO-d_6/H_2O 10%.

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Calculations by WinEQNMR Version 1.20 by Michael J. Hynes
Program run at 11:50:10   on 04/02/2008

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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  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

ORMS 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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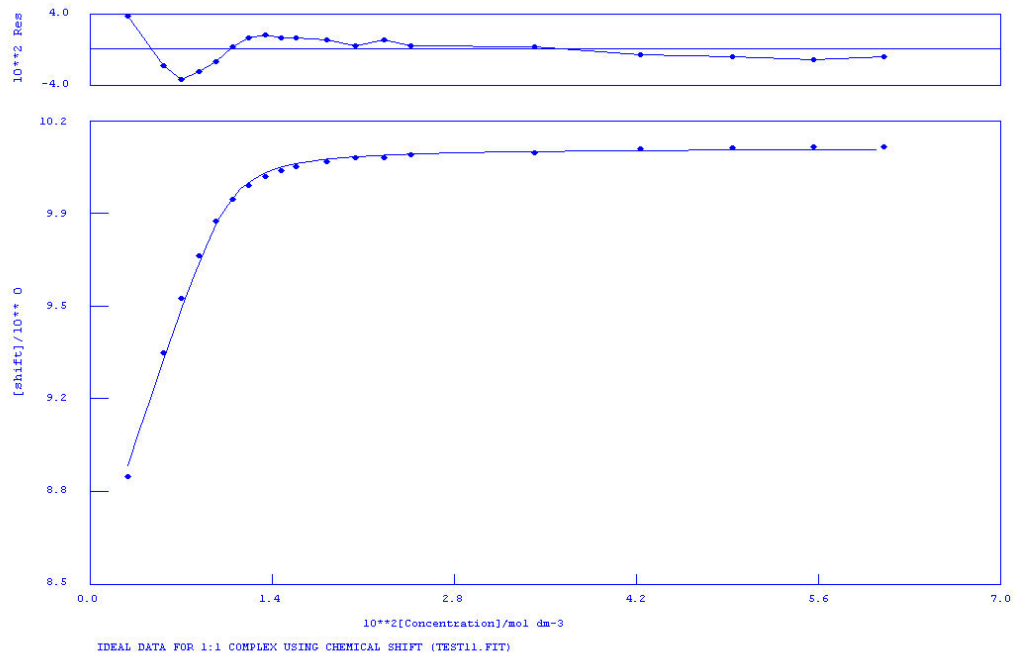
Figure S11 NMR titration of compound **2** *vs.* TBAOBz in DMSO-d_6/H_2O 10%.

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Calculations by WinEQNMR Version 1.20 by Michael J. Hynes
Program run at 12:03:03   on 04/02/2008

 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  4.71847E+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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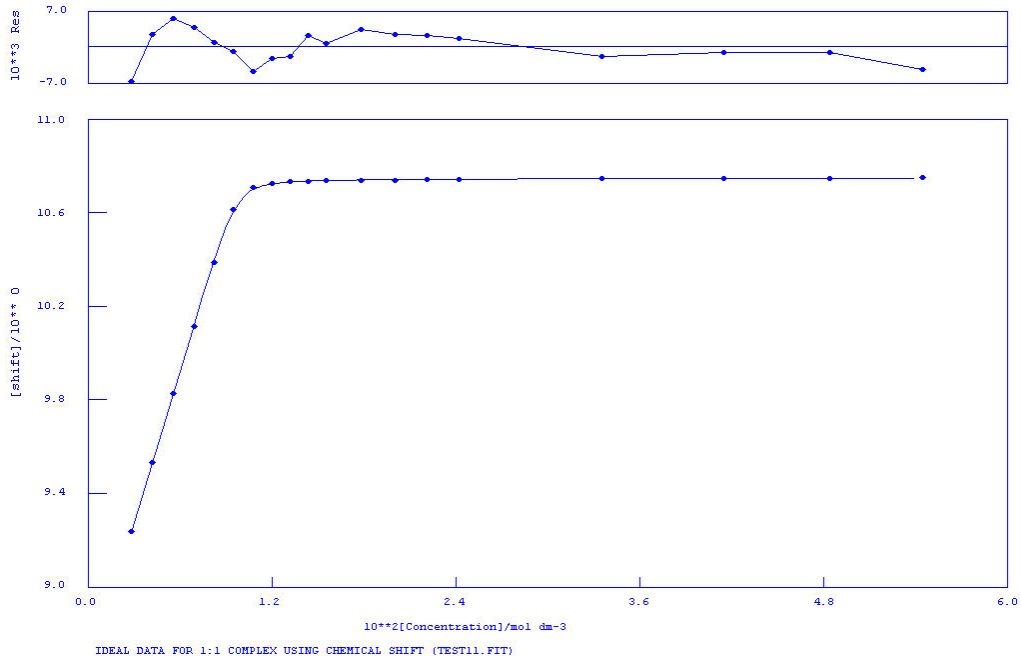
Figure S12 NMR titration of compound **2** *vs.* $TBAH_2PO_4$ in DMSO-d_6/H_2O 10%.

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

 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  3.60451E+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

ORMS 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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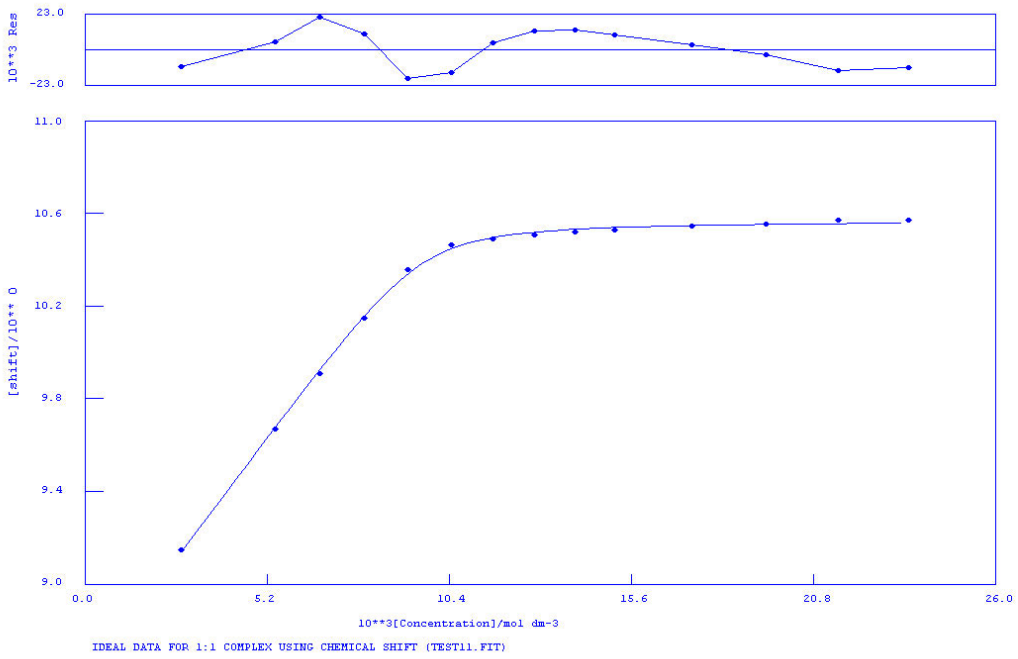
Figure S13 NMR titration of compound **3** *vs.* TBAOAc in DMSO-d_6/H_2O 0.5%.

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 Calculations by WinEQNMR Version 1.20 by Michael J. Hynes
Program run at 16:20:27   on 03/27/2008

 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  1.06028E+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

ORMS 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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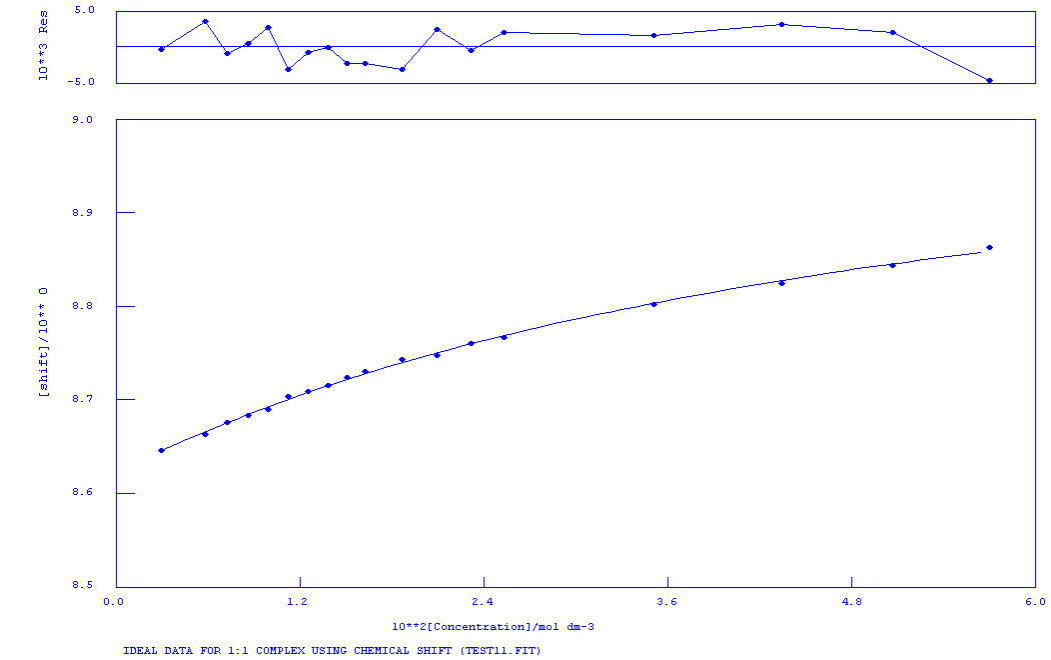
Figure S14 NMR titration of compound **3** *vs.* $TBAH_2PO_4$ in DMSO-d_6/H_2O 0.5%.



Calculations by WinEQNMR Version 1.20 by Michael J. Hynes
Program run at 20:15:40 on 03/31/2008

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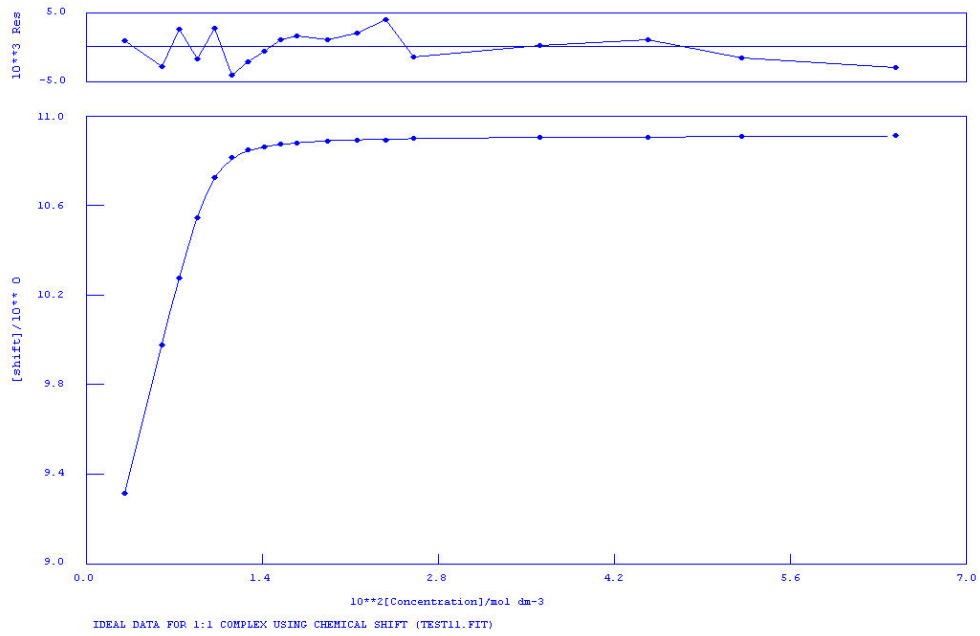
Reaction: M + L = ML
FILE: TEST11.FIT
IDEAL DATA: K1 = 63.091; DELTA M = 20.0; DELTA ML = 120.0
File prepared by M. J. Hynes, October 22 2000

NO.	A	PARAMETER	DELTA	ERROR	CONDITION	DESCRIPTION
1	1	2.30003E+01	2.000E-01	2.026E+00	1.114E+02	K1
2	1	8.62306E+00	2.000E-01	2.327E-03	8.160E+00	SHIFT M
3	1	9.05698E+00	1.000E+00	1.780E-02	7.582E+01	SHIFT ML

0RMS ERROR = 2.59E-03 MAX ERROR = 4.64E-03 AT OBS.NO. 18
RESIDUALS SQUARED = 1.00E-04
RFACTOR = 0.0270 PERCENT

Figure S15 NMR titration of compound **3** *vs.* $TBAHSO_4$ in DMSO-d_6/H_2O 0.5%.

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Calculations by WinEQNMR Version 1.20 by Michael J. Hynes
Program run at 15:12:13   on 03/19/2008

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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  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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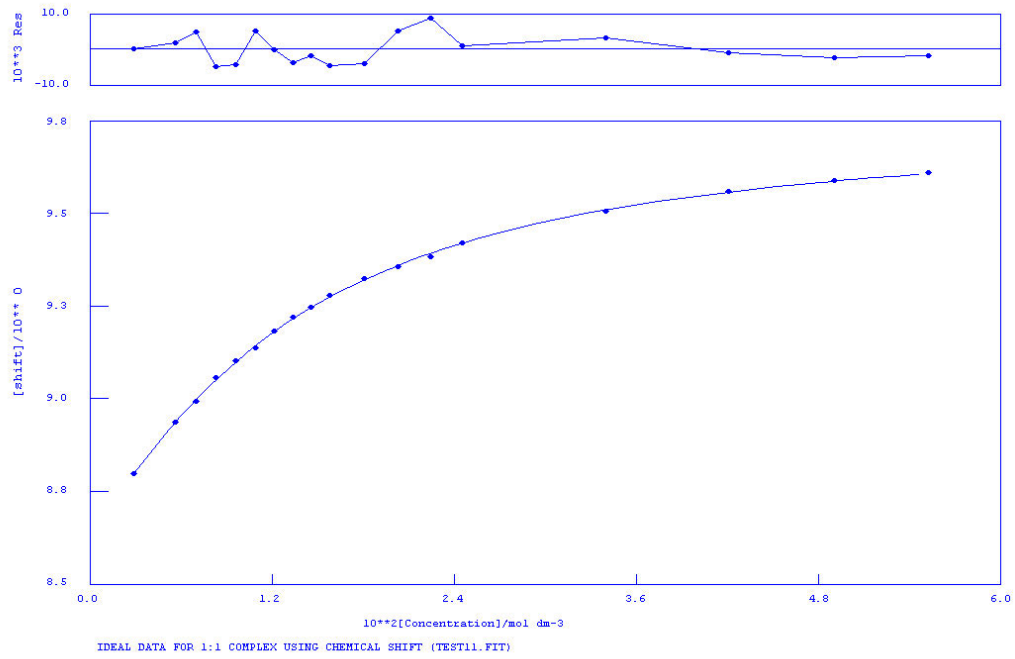
Figure S16 NMR titration of compound **3** *vs.* TBAOBz in DMSO-d_6/H_2O 0.5%.

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Calculations by WinEQNMR Version 1.20 by Michael J. Hynes
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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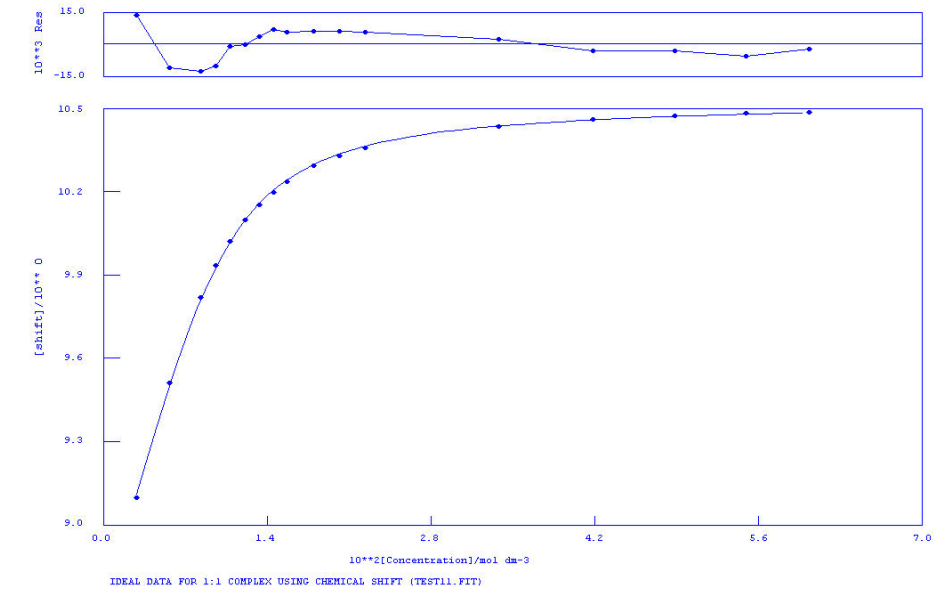
Figure S17 NMR titration of compound **3** *vs.* TBACl in DMSO-d_6/H_2O 0.5%.



Calculations by WinEQNMR Version 1.20 by Michael J. Hynes
Program run at 13:52:15 on 03/29/2008

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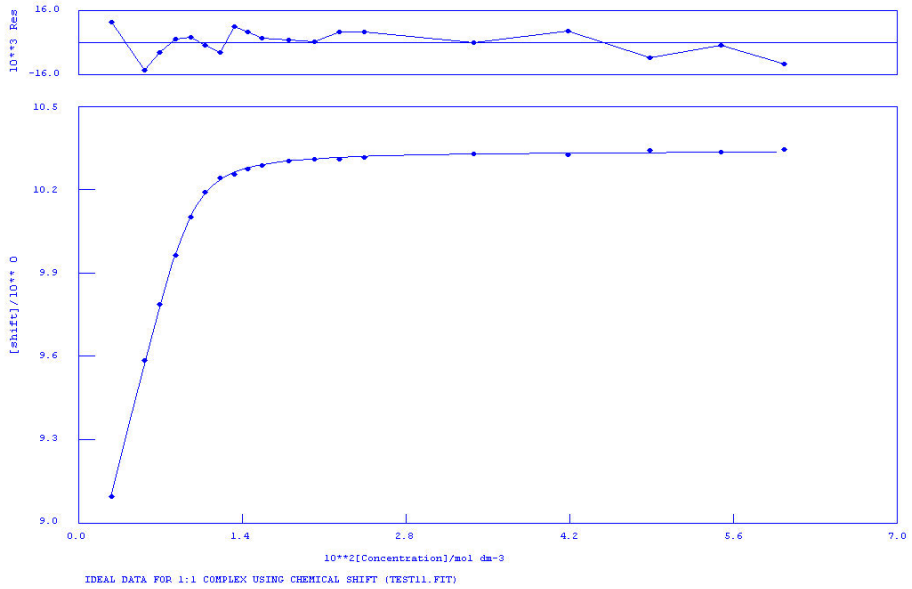
Reaction: M + L = ML
FILE: TEST11.FIT
IDEAL DATA: K1 = 63.091; DELTA M = 20.0; DELTA ML = 120.0
File prepared by M. J. Hynes, October 22 2000

NO.	A	PARAMETER	DELTA	ERROR	CONDITION	DESCRIPTION
1	1	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

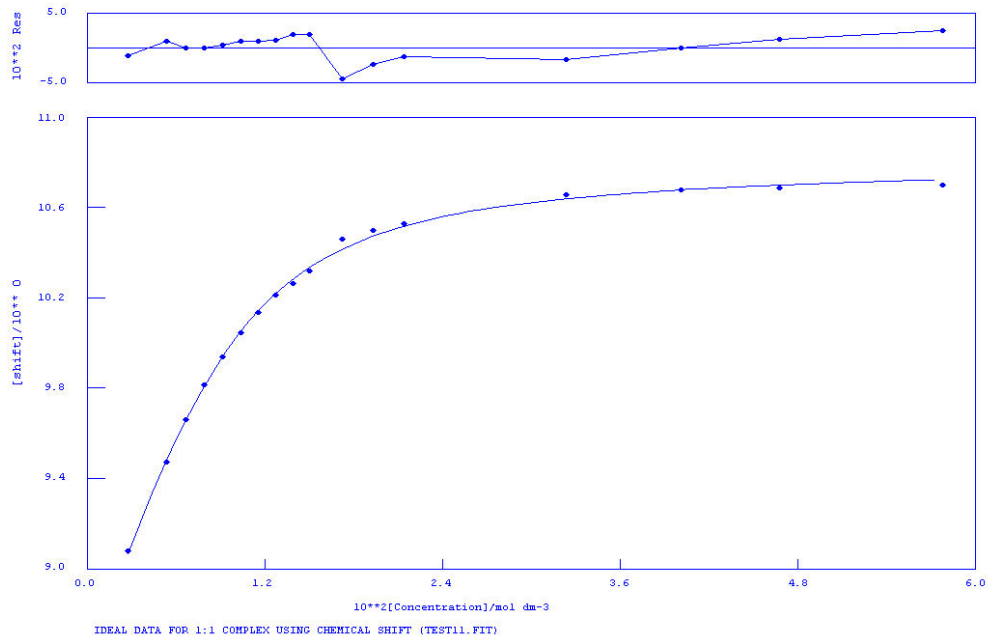
0RMS ERROR = 7.73E-03 MAX ERROR = 1.37E-02 AT OBS.NO. 1
RESIDUALS SQUARED = 8.36E-04
RFACTOR = 0.0691 PERCENT

Figure S18 NMR titration of compound **3** *vs.* TBAOAc in DMSO-d_6/H_2O 10%.



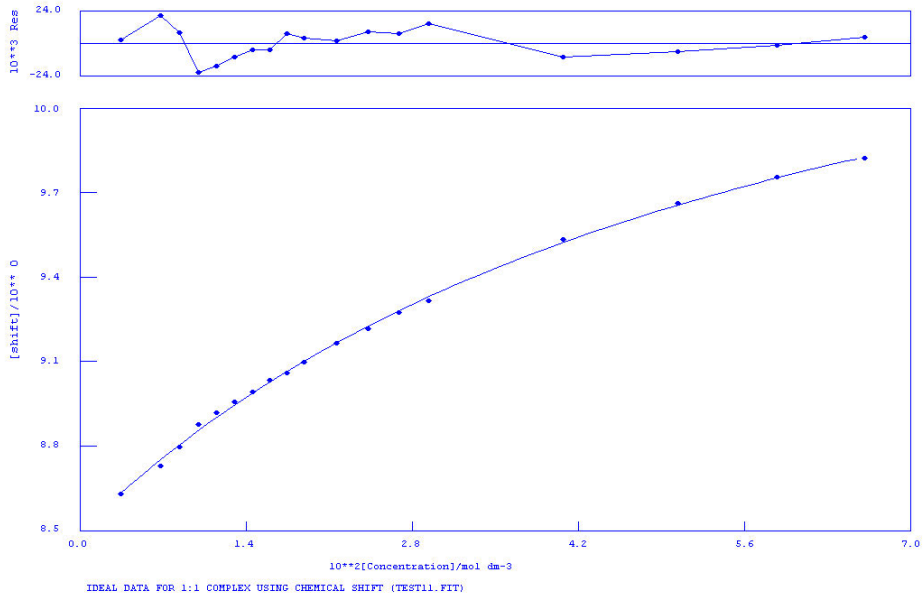

```
Calculations by WinEQNMR Version 1.20 by Michael J. Hynes
Program run at 13:39:41   on 03/29/2008

 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 S19 NMR titration of compound **3** *vs.* $TBAH_2PO_4$ in DMSO-d_6/H_2O 10%.



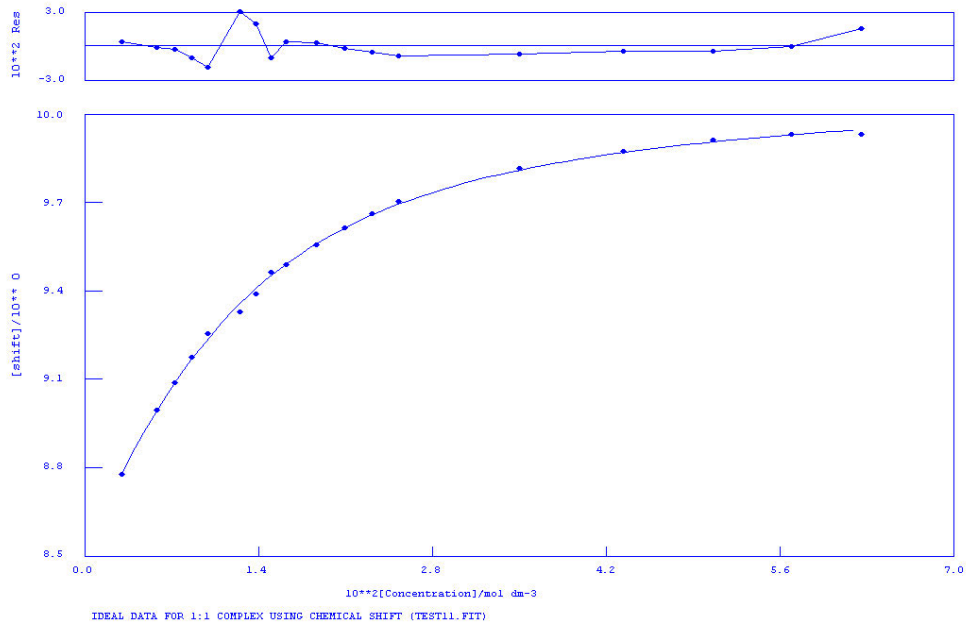

```
Calculations by WinEQNMR Version 1.20 by Michael J. Hynes
Program run at 09:20:56   on 03/31/2008

 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 S20 NMR titration of compound **3** *vs.* TBAOBz in DMSO-d_6/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 S21 NMR titration of compound **3** *vs.* TBAOAc in DMSO-d_6/H_2O 25%.



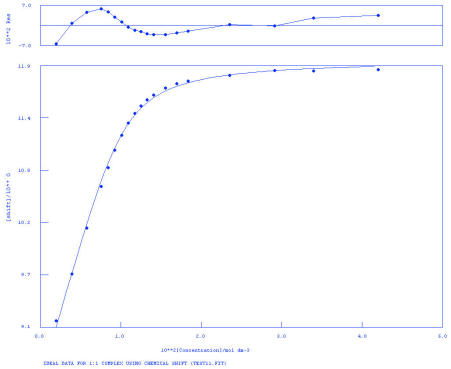

```
Calculations by WinEQNMR Version 1.20 by Michael J. Hynes
Program run at 18:10:59   on 03/30/2008

 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

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

Figure S22 NMR titration of compound **3** *vs.* $TBAH_2PO_4$ in DMSO-d_6/H_2O 25%.

Acetate.

K_a = 1261

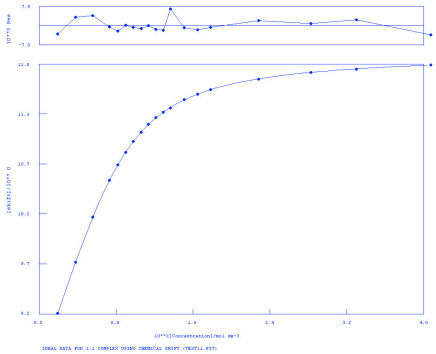
Error = 7.4 %



Benzoate.

K_a = 674

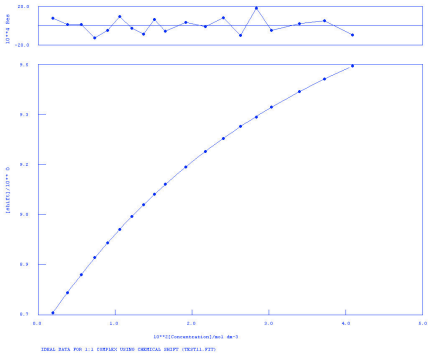
Error = 0.5 %



Chloride.

K_a = 31

Error = 0.8 %



Dihydrogen Phosphate.

K_a = 523

Error = 3.1 %

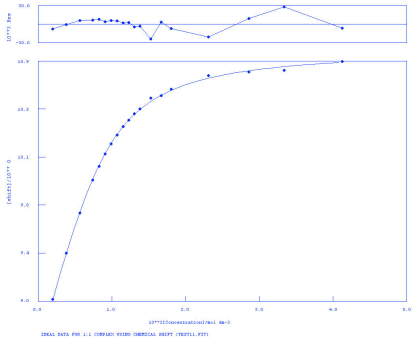


Figure S23 NMR titrations of 1,3-diphenylurea *vs.* various anions in DMSO-d_6/H_2O 0.5%.

Table S1 Output from Spartan ’06 for DFT calculation on dihydrogen phosphate complex of compound **3**. Dihydrogen phosphate was placed near the receptor and the structure minimised using AM1. The AM1 structure was then subjected to a DFT minimization (without symmetry constraints) at the 6-31G* level of theory.

Dihydrogen phosphate complex

MacSPARTAN '06 Quantum Mechanics Program: (x86/Darwin) build 129cv3

Job type: Geometry optimization.

Method: RB3LYP

Basis set: 6-31G(D)

Number of shells: 141

Number of basis functions: 441

Multiplicity: 1

SCF model:

A restricted hybrid HF-DFT SCF calculation will be performed using Pulay DIIS + Geometric Direct Minimization

Optimization:

Step	Energy	Max Grad.	Max Dist.
1	-1594.1530439	0.044981	0.125345
2	-1594.1659397	0.015414	0.105096
3	-1594.1699231	0.009119	0.114535
4	-1594.1714013	0.006636	0.109438
5	-1594.1722508	0.006413	0.081237
6	-1594.1725963	0.006074	0.128203
7	-1594.1728285	0.002557	0.111756
8	-1594.1730204	0.002360	0.167580
9	-1594.1732708	0.002190	0.187406
10	-1594.1735715	0.001991	0.200676
11	-1594.1738958	0.001791	0.203147
12	-1594.1742294	0.001672	0.200622
13	-1594.1745457	0.001642	0.200113
14	-1594.1748372	0.001549	0.190419
15	-1594.1750977	0.001710	0.190484
16	-1594.1753110	0.002072	0.164706
17	-1594.1754654	0.001749	0.159274
18	-1594.1755301	0.001465	0.075183

19 -1594.1755508 0.000820 0.079223

20 -1594.1755645 0.000956 0.049137

21 -1594.1755723 0.000541 0.036628

22 -1594.1755765 0.000406 0.013934

23 -1594.1755805 0.000334 0.010747

24 -1594.1755827 0.000196 0.017219

25 -1594.1755843 0.000201 0.006915

26 -1594.1755850 0.000137 0.008312

Program Wall Time: 6:29:33.0

Reason for exit: Successful completion

Quantum Calculation CPU Time : 5:51:10.1

Quantum Calculation Wall Time: 6:29:37.1

SPARTAN PROPERTIES PACKAGE: MAC/P4 build 129c

Reason for exit: Successful completion

Properties CPU Time : 4.12

Properties Wall Time: 4.34

molecule M0001 terminated normally End- molecule "M0001" Wed Apr 16 17:54:42 2008

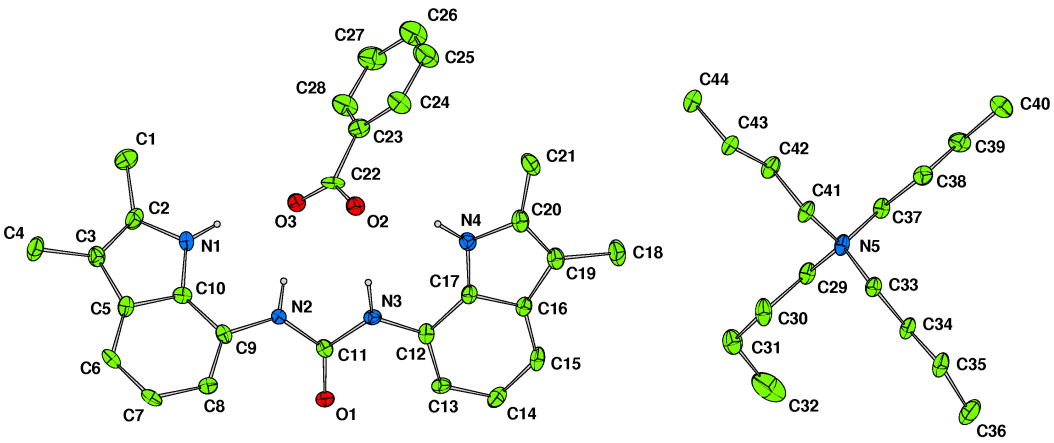


Figure S24 Thermal ellipsoid plot of the tetrabutylammonium benzoate complex of compound **2**. Thermal ellipsoids drawn at 30% probability level.