Supplementary Material (ESI) for Chemical Communications

Carboxylate complexation in 1,1'-(1,2-phenylene)bis(3-phenylurea) in solution and in the solid state

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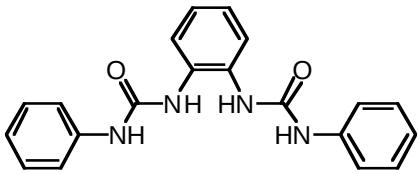
Supplementary information

1,1'-(1,2-phenylene)bis(3-phenylurea) **(3).**

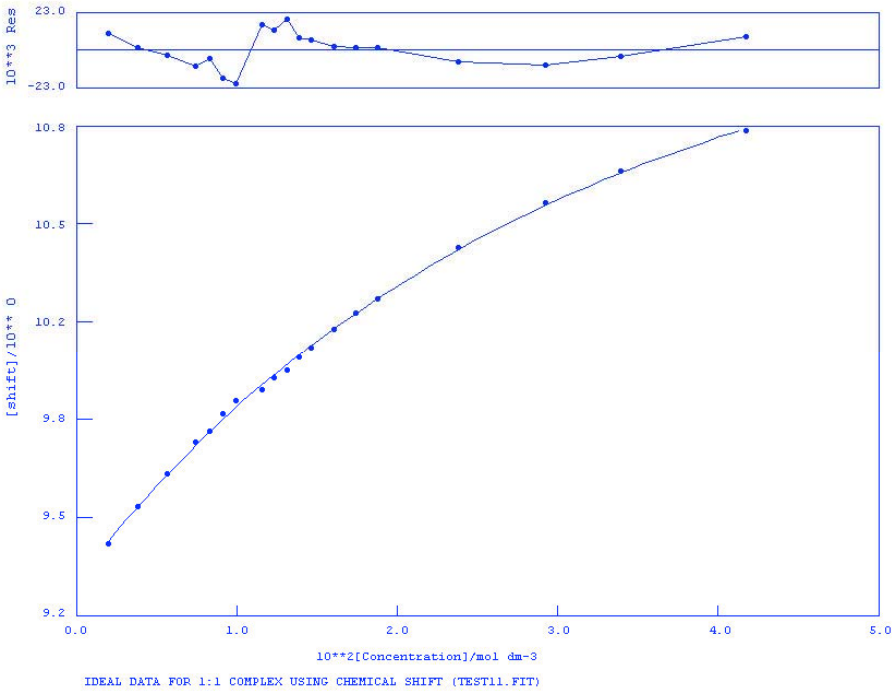


To a stirring solution of 1,2-phenylenediamine (0.68 g, 6.5 mmol) in dry dichloromethane (100 mL) was slowly added phenylisocyanate (1.36 mL, 13.0 mmol). The reaction was heated at reflux under a nitrogen atmosphere for 16 hours. The resulting white precipitate was removed via filtration and washed with petroleum ether (20 mL) followed by DCM (20 mL). White solid product dried under high vacuum. Mass of product = 2.15 g, 6.2 mmol. Yield = 96 %. ^{1}H NMR – 300 MHz (DMSO-d_6): 9.06 (s, 2H, urea N*H*), 8.06 (s, 2H, urea N*H*), 7.60 (m, 2 H, aryl C*H*), 7.45 (dd, 4H, aryl C*H*, J = 8.4 & 1.1 Hz), 7.26 (m, 4H, aryl C*H*), 7.08 (m, 2H, aryl C*H*), 6.96 (m, 2H, aryl C*H*). ^{13}C NMR - 75 MHz (DMSO-d_6): 153.2, 139.8, 131.2, 128.7, 123.96, 123.93, 121.73, and 118.14. LRMS (ES): 458.9 [M + TFA – H]$^-$, 472.8 [M + HCO_2H + 2MeCN – H]$^-$, 727.0 [2M + Cl]$^-$, 804.9 [2M + TFA – H]$^-$, 1073.7 [3M + Cl]$^-$, 1420.6 [4M + Cl]$^-$. IR (cm^{-1}): 3276, 3049, 1695, 1625, 1600, 1565, 1538, 1495, 1440, 1305 & 1239. Melting point (from DCM): 264 °C. Microanalysis: Expected C = 69.35 H = 5.24 N = 16.17. Found – C = 69.01 H = 5.28 N = 16.18.

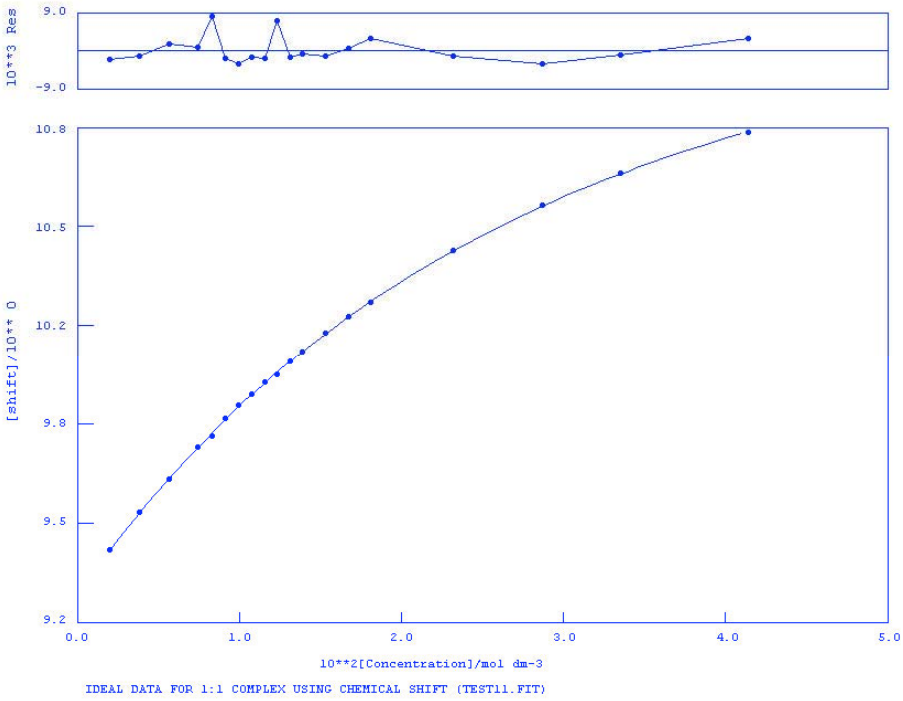
^{1}H NMR TITRATION CURVES.

***N*-(2-Acetylamino-phenyl)-acetamide (1).**

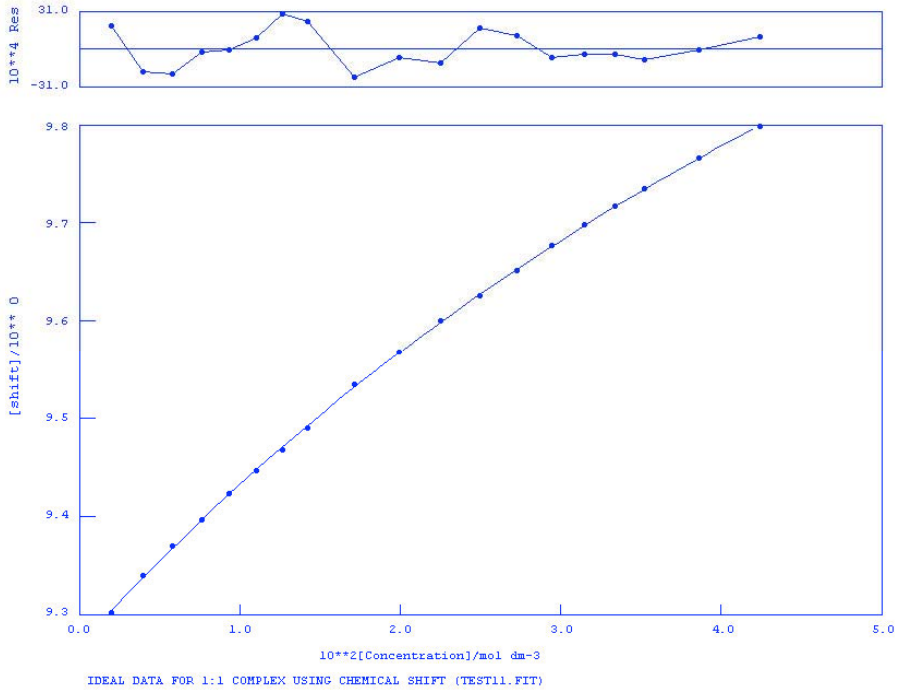
TBA.Acetate in DMSO-d_6 / 0.5% H_2O.



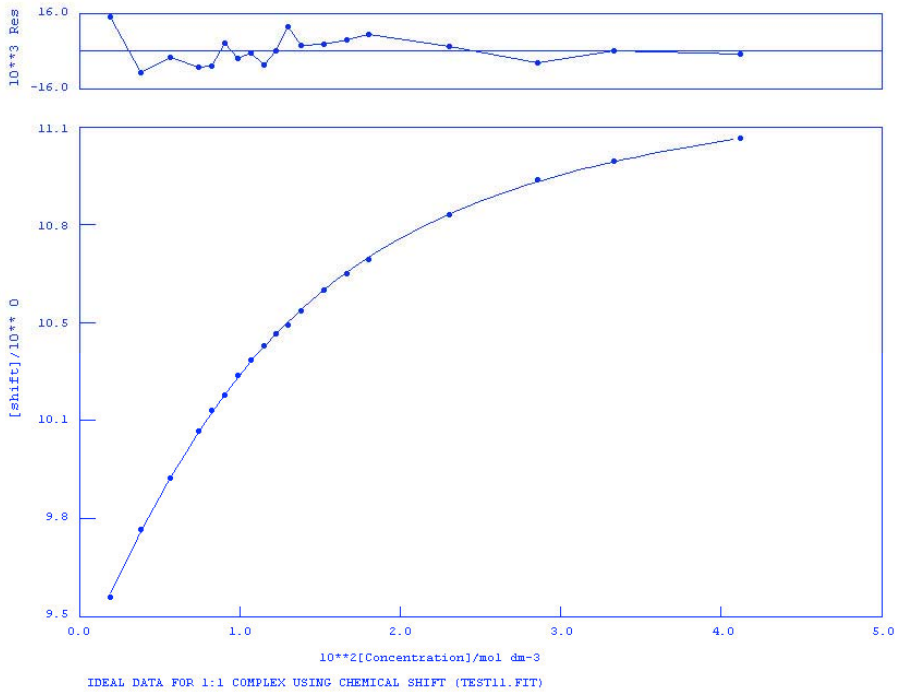
TBA.Benzoate in DMSO-d_6 / 0.5% H_2O.



TBA.Chloride in DMSO-d_6 / 0.5% H_2O.

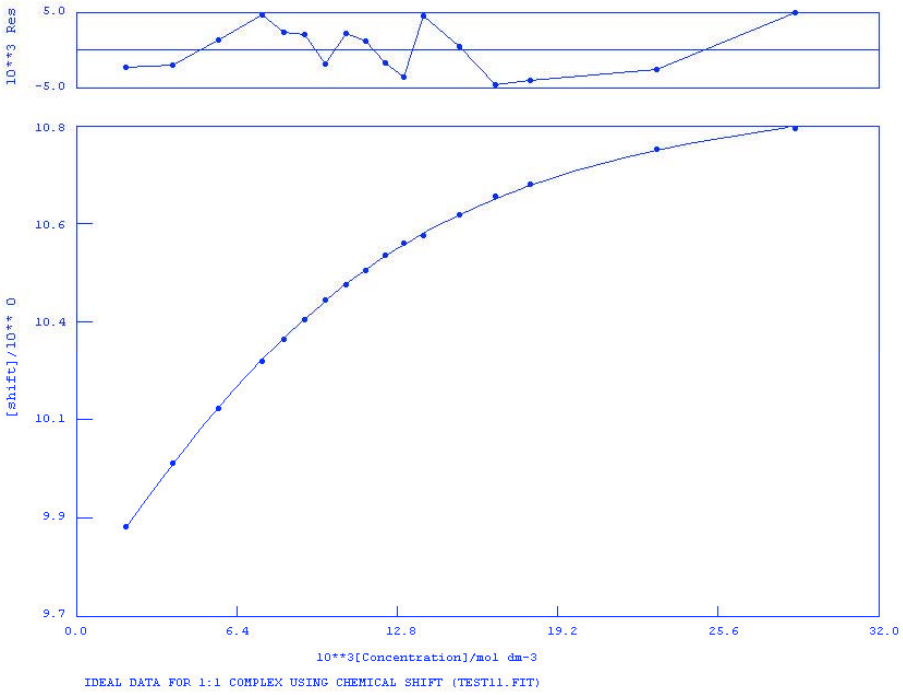


TBA.Dihydrogen Phosphate in DMSO-d_6 / 0.5% H_2O.

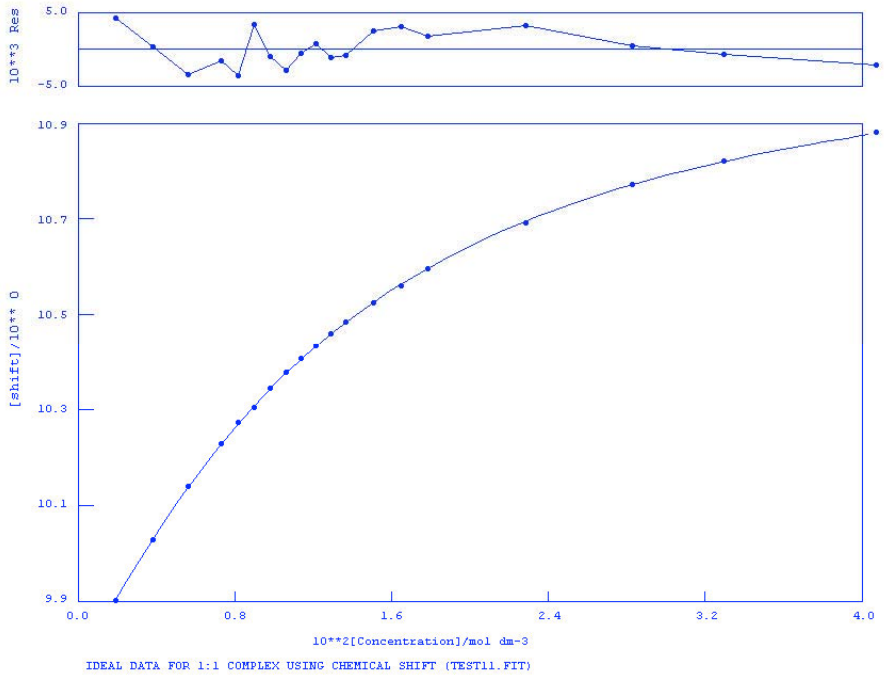


***N,N*,-(1,2-phenylene)-*bis*-(1H-pyrrole-2-caboxamide) (2).**

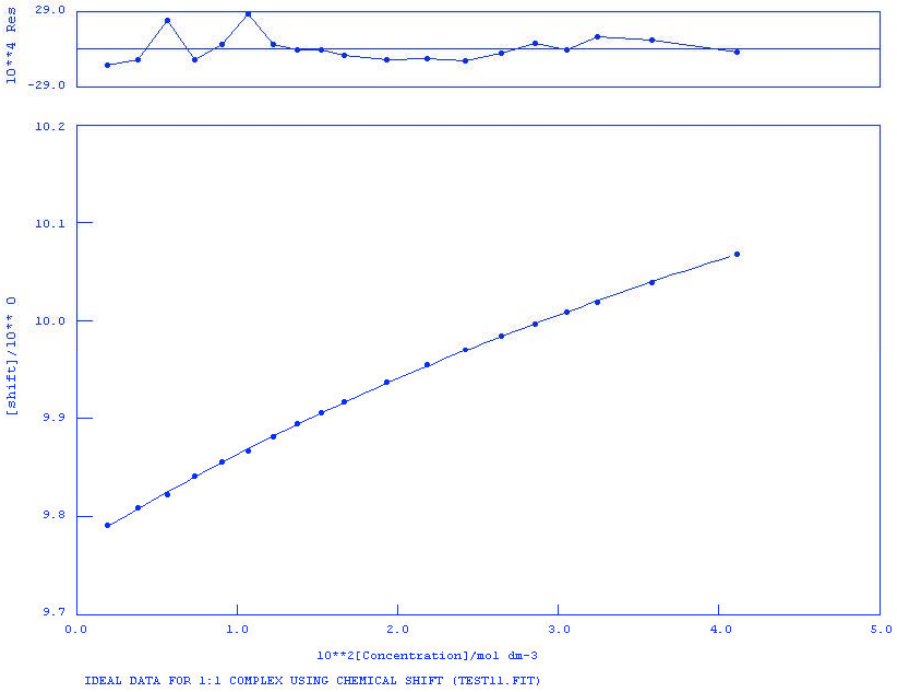
TBA.Acetate in DMSO-d_6 / 0.5% H_2O.



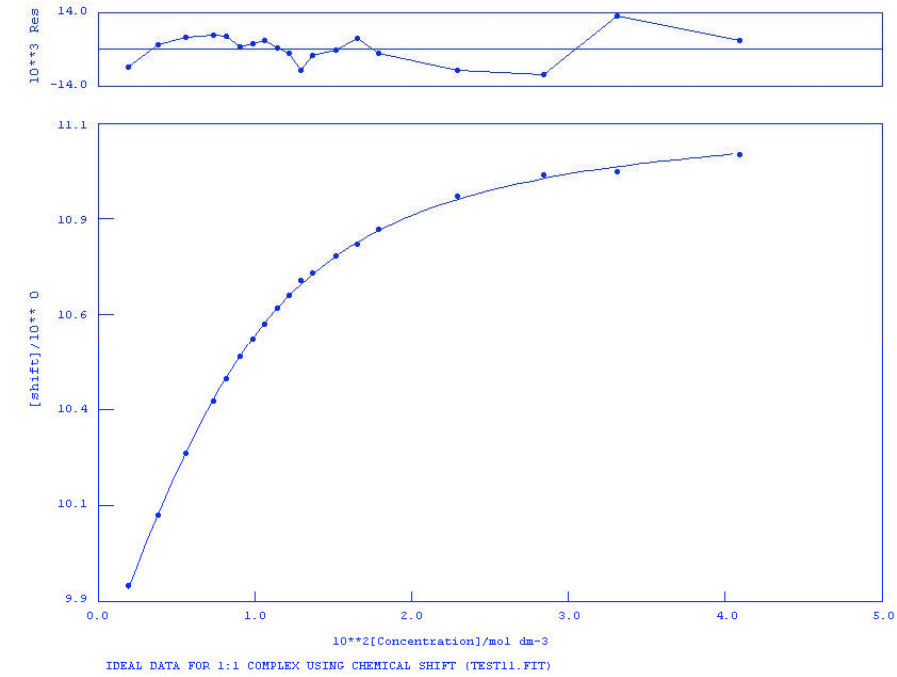
TBA.Benzoate in DMSO-d_6 / 0.5% H_2O.



TBA.Chloride in DMSO-d_6 / 0.5% H_2O.

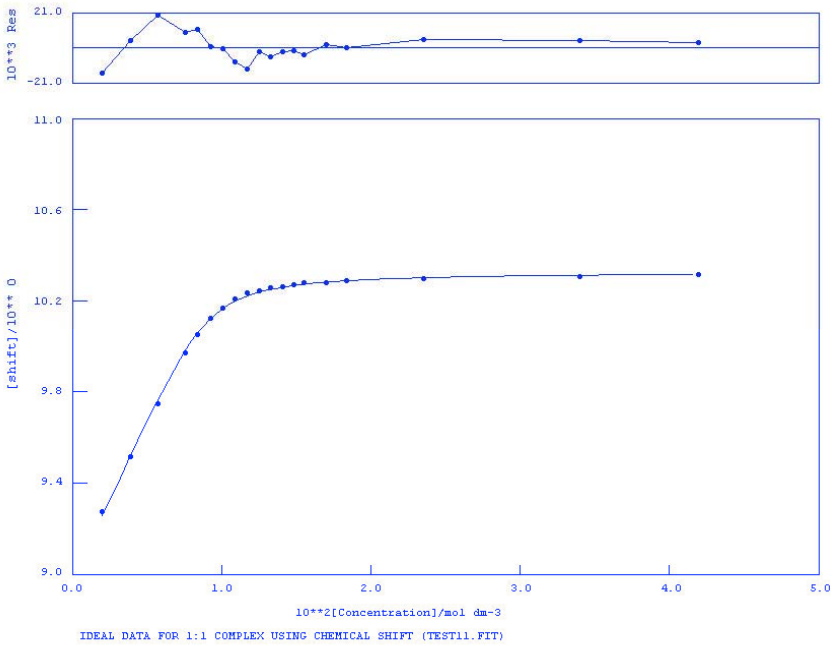


TBA.Dihydrogen Phosphate in DMSO-d_6 / 0.5% H_2O.

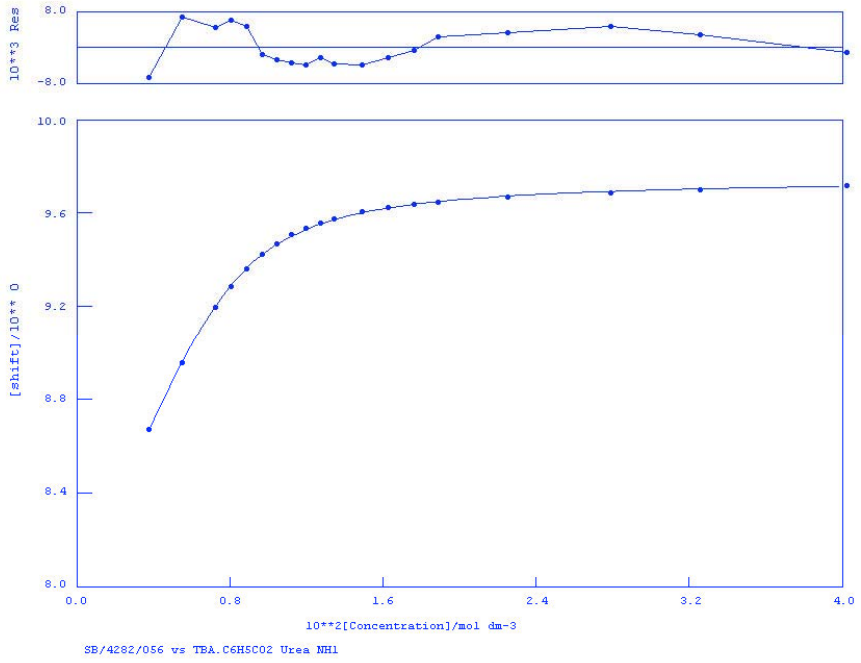


1,1'-(1,2-phenylene)bis(3-phenylurea) (3).

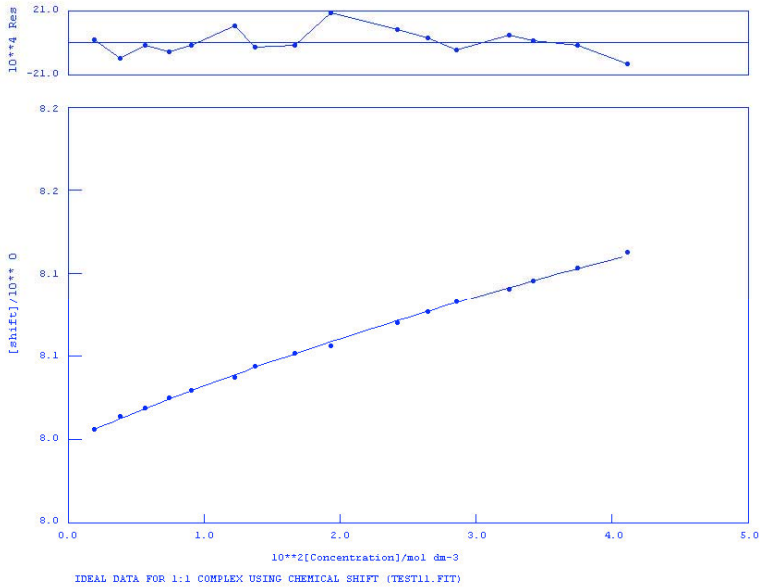
TBA.Acetate in DMSO-d_6 / 0.5% H_2O.



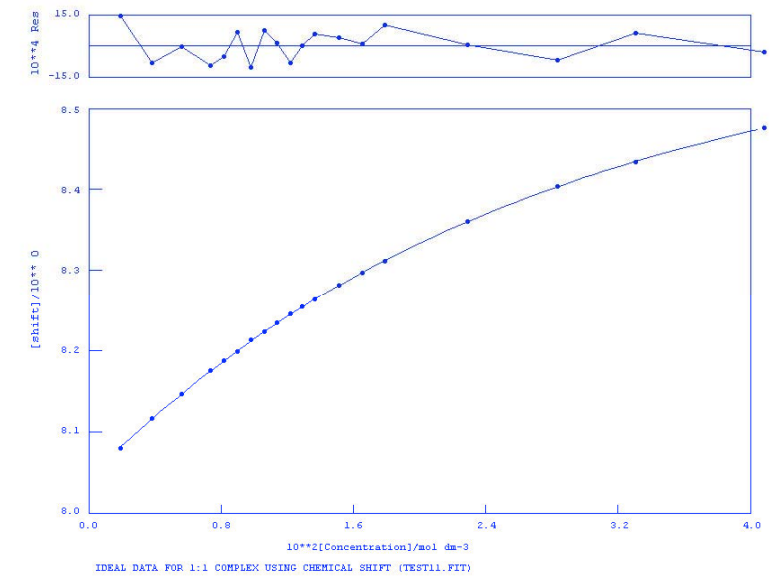
TBA.Benzoate in DMSO-d_6 / 0.5% H_2O.



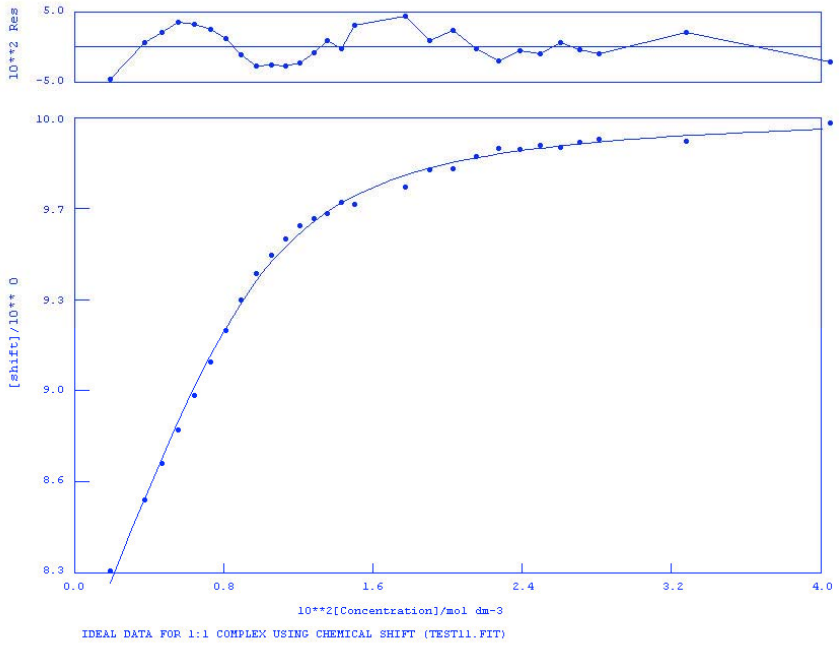
TBA.Bromide in DMSO-d_6 / 0.5% H_2O.



TBA.Chloride in DMSO-d_6 / 0.5% H_2O.



TBA.Dihydrogen Phosphate in DMSO-d_6 / 0.5% H_2O.



TBA.Hydrogen Sulphate in DMSO-d_6 / 0.5% H_2O.