A new family of bis-ureidic receptors for pyrophosphate optical sensing

Claudia Caltagirone* a, Carla Bazzicalupi, b Francesco Isaia, a Mark E. Light, c Vito Lippolis, a Riccardo Montis, a Sergio Murgia, a Martina Olivari, a Giacomo Picci a

a Università degli Studi di Cagliari, Dipartimento di Scienze Chimiche e Geologiche, S.S. 554 Bivio per Sestu, Monserrato (CA), 09042, Italy.
b Università degli Studi di Firenze, Dipartimento di Chimica “Ugo Schiff”, Via della Lastruccia 3, Sesto Fiorentino (FI), 50019, Italy.
c School of Chemistry, University of Southampton, Southampton, SO17 1BJ, UK. Fax: +44 23 8059 6805; Tel: +44 23 8059 6805; E-mail: M.E.Light@soton.ac.uk.

General procedures

All reactions were performed in oven-dried glassware under a slight positive pressure of nitrogen. 1H-NMR (400 MHz, 500MHz) and 13C NMR (100 MHz, 125MHz) spectra were determined on a Varian INOVA-400 spectrometer, and Varian INOVA-500 spectrometer. Chemical shifts for 1H 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, m = multiplet . Chemical shifts for 13C 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 NICOLET 5700 FT-IR spectrophotometer and reported in wavenumbers (cm⁻¹). Microanalytical data were obtained using a Fisons EA CHNS-O instrument (T = 1000 °C). Fluorescence spectra were recorded on a Cary Eclypse spectrofluorimeter. All solvents and starting materials were purchased from commercial sources where available. Proton NMR titrations were performed by adding aliquots of the putative anionic guest (as the TBA salt, 0.075 M) in a solution of the receptor (0.005M) in DMSO-d6/0.5% water to a solution of the receptor (0.005M).

The synthesis of pyridine-2,6-diyldimethanaminium chloride has already been reported in literature.1

Synthesis of 1,1'-(1,3-phenylenebis(methylene))bis(3-phenylurea) L\textsuperscript{1}

A solution of phenyl isocyanate (0.3595g, 3.02mmol) in DCM (10 ml) was added dropwise to the solution of phenyl-1,3-dimethylamine (0.222g, 1.63 mmol) in DCM (20 ml) and was left stirring at ambient temperature under a N\textsubscript{2} atmosphere for 24h. The precipitate was collected by filtration, washed with diethyl ether and dried under reduced pressure to give the desired product as a white solid.

Yield: 80.14\% (g 0.49g, 1 mmol); M.p.:222-225°C; \textsuperscript{1}H NMR(400 MHz, DMSO-\textit{d}\textsubscript{6}, 298K) δ\textsuperscript{H}:4.25 (d, J=5.6 Hz, 4 H); 6.56 (t, J=5.6 Hz, NH, 2H); 6.84 (t, J=7.6 Hz, ArH, 2H); 7.13-7.20(m, ArH, 7H); 7.26 (t, J=7.6 Hz,ArH, 1H); 7.35 (d, J=8 Hz, 4H); 8.49(s, NH, 2H); \textsuperscript{13}C-NMR(100 MHz, DMSO-\textit{d}\textsubscript{6}, 298 K) δ\textsuperscript{C} 42.77; δ\textsubscript{ArH} 117.7, 121.1, 125.6, 126.0, 128.3, 128.7, 140.5; δ\textsuperscript{CO} 155.21

Synthesis of 1,1'-(1,3-phenylenebis(methylene))bis(3-(2-nitrophenyl)urea) L\textsuperscript{2}

A solution of 2-nitrophenyl isocyanate (0.4825 g, 2.94 mmol) in 10 ml of DCM was added dropwise to the solution of phenyl-1,3-dimethylamine (0.235 g, 1.47 mmol) in 20 ml of DCM and was stirred at room temperature under a N\textsubscript{2} atmosphere for 24h. The yellow solid formed was isolated by filtration, washed with diethyl ether and dried under vacuum.

Yield: 77.64\% (0.53g, 1.1mmol); M.p.:236-240°C; \textsuperscript{1}H NMR(400 MHz, DMSO-\textit{d}\textsubscript{6}, 298K) δ\textsuperscript{H}:4.32 (d, J=6 Hz, 4 H); 7.12 (t, J=8, ArH, 2H); 7.21 (d, J=7.2, ArH, 2H); 7.27 (s, ArH, 1H); 7.33 (t, J=7.2, ArH, 1H); 7.62 (t, J=8.4, ArH,2H); 8.00-8.06 (m, ArH, NH, 4H); 8.32 (d, J=8.4, 2H); 9.43 (s, NH, 2H); \textsuperscript{13}C-NMR (100MHz, DMSO-\textit{d}\textsubscript{6} , 298 K) δ\textsuperscript{C} 42.94; δ\textsubscript{ArC} 121.4, 122.4, 125.3, 125.9, 126.2, 128.4, 134.9,135.8,136.8, 139.8; δ\textsuperscript{CO} 154.28.

Synthesis of 1,1'-(1,3-phenylenebis(methylene))bis(3-(naphthalen-1-yl)urea) L\textsuperscript{3}

1-naphthyl isocyanate (0.5084 g 3.00 mmol) dissolved in DCM (10 ml) was added dropwise to a solution of phenyl-1,3-dimethylamine (0.233g, 1.63mmol) in 20 ml of DCM. The mixture of
reaction was allowed to stir for 24h under a N₂ atmosphere at room temperature. The resulting white precipitate was removed by filtration, washed with diethyl ether and dried under vacuum.

Yield: 67.22% (0.52 g, 1.1 mmol); M.p.: 245-248°C; ¹H NMR(500 MHz, DMSO-d₆, 298K) δCH 4.38 (d, J=6 Hz, 4 H); δArH 7.04 (t, J=6 Hz, NH, 2H); 7.33-7.42 (m, ArH, 4H); 7.48-7.58 (m, ArH, 6H); 7.88 (d, J=7.5 Hz, ArH, 2H); 8.01 (d, J=8 Hz, ArH, 2H); 8.08 (d, J=8 Hz, ArH, 2H); 8.59 (s, NH, 2H).

Synthesis of 1,1'-(pyridine-2,6-diylbis(methylene))bis(3-phenylurea) L₄

A solution of phenyl isocyanate (0.170 g; 1.42 mmol) in DCM (10 ml) was added dropwise to the suspension of pyridine-2,6-diyldimethanamine dihydrochloride (0.150g, 0.714 mmol) and TEA (1 ml) in DCM (20ml). The reaction mixture was refluxed for 24h under a N₂ atmosphere and the precipitate thus obtained was filtered off, washed with H₂O and dried over vacuum to give the product as a white solid.

Yield 53% (0.180 g; 0.378mmol) ; M.p. 225-226°C; ¹H NMR(500 MHz, DMSO-d₆, 298K) δCH 4.40 (d, J=5 Hz, 4 H); δArH 6.73 (t, J=6 Hz, NH, 2H); 6.90 (t, 7Hz, ArH, 2H); 7.21-7.24 (m, ArH, 6H); 7.40 (d, J=8, ArH, 4H); 7.75 (t, J=8, ArH, 1H); 8.72 (s, NH, 2H); ¹³C-NMR(125 MHz, DMSO-d₆ , 298 K) δC 45.17; δArC 118.19, 119.52, 121.61, 129.11, 137.84, 140.9, 155.7, δCO 159.04.

Synthesis of 1,1'-(pyridine-2,6-diylbis(methylene))bis(3-(2nitrophenyl)urea) L₅

A solution of 2-nitrophenyl isocyanate (0.468 g; 2.85 mmol) in 15 ml of DCM was added dropwise to a stirred suspension of pyridine-2,6-diyldimethanamine dihydrochloride and TEA (1 ml) in DCM (20ml). The reaction was refluxed under N₂ atmosphere overnight. The resulting precipitate was then filtered, washed with water and then with MeOH, dried under reduced pressure and isolated as a yellow solid.

Yield: 51% (0.340g, 0.73 mmol); M.p. 234°C; ¹H NMR(500 MHz, DMSO-d₆, 298K) δCH 4.41 (d, J=6 Hz, 4 H); δArH 7.14 (t, J=7 Hz, ArH, 2H); 7.25 (d, J=7.5Hz, ArH, 2H); 7.64 (t, J=7 ArH, 2H); 7.78 (t, J=8, ArH, 1H); 8.03-8.10 (m, 4H); 8.27 (d, J=8.5, ArH, 2H) 9.50 (s, NH, 2H) ¹³C-NMR(125 MHz, DMSO-d₆ , 298 K) δC 44.91; δArC 119.22, 121.60, 122.20, 125.24, 134.82, 135.53, 137.25, 137.47, 154.41; δCO 158.25

Synthesis of 1,1'-(pyridine-2,6-diylbis(methylene))bis(3-(naphthalen1-yl)urea) L₆

A solution of 1-naphthyl isocyanate (0.320 g; 1.90 mmol) in DCM (15 ml) was added dropwise to a
stirred suspension of pyridine-2,6-diylidimethanamine dihydrochloride (0.200 g; 0.95 mmol) and TEA (1 ml) in DCM (15 ml). After refluxing under N₂ atmosphere overnight the white solid was isolated by filtration, washed with water and dried under reduced pressure. Yield 59.7% (0.270 g; 0.57 mmol); M.p. >250°C; ¹H NMR(500 MHz, DMSO-d₆, 298 K) δCH 4.5 (d, J=6 Hz, 4 H); δArH 7.20 (t, J=6 NH, 2H); 7.30 (d, J=8Hz, ArH , 2H); 7.43 (t, J=7.5 ArH, 2H); 7.49-7.59 (m, ArH, 6H); 7.80 (t, J=7.5, ArH, 1H); 7.90 (d, J=8, ArH, 2H); 8.00 (d, J=8, ArH, 2H); 8.14 (d, J=8, ArH, 2H); 8.77 (s, NH, 2H); ¹³C-NMR(125 MHz, DMSO-d₆ , 298 K) δC 44.89, δArC 116.77, 119.14, 121.45, 122.24, 125.39, 125.71, 125.86, 128.28, 133.69, 135.03, 137.48, 155.65, δCO 158.61.

Calculations by winEQNMR Version 1.20 by Michael J. Hynes
Program run at 12:50:07 on 12/30/2011

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  1.19274E+02 2.000E-01 3.364E+00 5.740E+01  K₁
2  1  8.51926E+00 2.000E-01 9.384E-03 8.464E+00  SHIFT M
3  1  1.15161E+01 1.000E+00 2.614E-02 3.230E+01  SHIFT ML

RMS ERROR = 8.03E-03 MAX ERROR = 1.73E-02 AT OBS.NO.  1
RESIDUALS SQUARED = 8.39E-04
RFACTOR = 0.0744 PERCENT
**Figure S1** $^1$H-NMR titration of $L^1$ with TBAAcO in DMSO-$d_6$.

![Graph showing $^1$H-NMR titration of $L^1$ with TBAAcO in DMSO-$d_6$.]

Calculations by WinEQNMR Version 1.20 by Michael J. Hynes
Program run at 14:59:54 on 01/04/2012

**IDEAL DATA FOR 1:1 COMPLEX USING CHEMICAL SHIFT (TEST11.FIT)**

Reaction: $M + L = ML$

FILE: TEST11.FIT

IDEAL DATA: $K_1 = 63.091$; DELTA M = 20.0; DELTA ML = 120.0

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

<table>
<thead>
<tr>
<th>NO.</th>
<th>A</th>
<th>PARAMETER</th>
<th>DELTA</th>
<th>ERROR</th>
<th>CONDITION</th>
<th>DESCRIPTION</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1</td>
<td>1.08941E+02</td>
<td>2.000E-01</td>
<td>5.928E+00</td>
<td>4.741E+01</td>
<td>$K_1$</td>
</tr>
<tr>
<td>2</td>
<td>1</td>
<td>8.62882E+00</td>
<td>2.000E-01</td>
<td>1.547E-02</td>
<td>7.546E+00</td>
<td>SHIFT M</td>
</tr>
<tr>
<td>3</td>
<td>1</td>
<td>1.10901E+01</td>
<td>1.000E+00</td>
<td>4.079E-02</td>
<td>2.653E+01</td>
<td>SHIFT ML</td>
</tr>
</tbody>
</table>

$\text{ORMS ERROR} = 1.45E-02 \quad \text{MAX ERROR} = 3.11E-02 \text{ AT OBS.NO. 1}$

$\text{RESIDUALS SQUARED} = 2.93E-03$

$\text{RFACOR} = 0.1363 \text{ PERCENT}$

**Figure S2** $^1$H-NMR titration of $L^1$ with TBABzO in DMSO-$d_6$.
Calculations by WinEQNMR Version 1.20 by Michael J. Hynes  
Program run at 11:06:06 on 12/29/2011  

**IDEAL DATA FOR 1:1 COMPLEX USING CHEMICAL SHIFT (TEST11.FIT)**

**Reaction:** \( \text{M} + \text{L} = \text{ML} \)  
**FILE:** TEST11.FIT  
**IDEAL DATA:** \( K_1 = 63.091; \) \( \Delta \text{M} = 20.0; \) \( \Delta \text{ML} = 120.0 \)  
**File prepared by M. J. Hynes, October 22 2000**

<table>
<thead>
<tr>
<th>NO.</th>
<th>A</th>
<th>PARAMETER</th>
<th>DELTA</th>
<th>ERROR</th>
<th>CONDITION</th>
<th>DESCRIPTION</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1</td>
<td>4.83740E+02</td>
<td>2.000E-01</td>
<td>2.235E+01</td>
<td>1.596E+01</td>
<td>( K_1 )</td>
</tr>
<tr>
<td>2</td>
<td>1</td>
<td>8.40857E+00</td>
<td>2.000E-01</td>
<td>1.770E-02</td>
<td>3.921E+00</td>
<td>( \text{SHIFT M} )</td>
</tr>
<tr>
<td>3</td>
<td>1</td>
<td>1.07129E+01</td>
<td>1.000E+00</td>
<td>1.774E-02</td>
<td>9.549E+00</td>
<td>( \text{SHIFT ML} )</td>
</tr>
</tbody>
</table>

**RMS ERROR = 1.51E-02**  
**MAX ERROR = 2.38E-02 AT OBS.NO. 12**  
**RESIDUALS SQUARED = 2.97E-03**  
**RFACTOR = 0.1380 PERCENT**

**Figure S3** \(^1\text{H-NMR titration of L}^1 \) with TBAH\(_2\text{PO}_4\) in DMSO-\(d_6\).
Calculations by WinEQNMR Version 1.20 by Michael J. Hynes
Program run at 12:59:10   on 02/08/2012

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

<table>
<thead>
<tr>
<th>NO.</th>
<th>A</th>
<th>PARAMETER</th>
<th>DELTA</th>
<th>ERROR</th>
<th>CONDITION</th>
<th>DESCRIPTION</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1</td>
<td>5.48167E+03</td>
<td>2.000E-01</td>
<td>1.185E+03</td>
<td>1.181E+01</td>
<td>K1</td>
</tr>
<tr>
<td>2</td>
<td>1</td>
<td>8.27172E+00</td>
<td>2.000E-01</td>
<td>7.242E-02</td>
<td>2.434E+00</td>
<td>SHIFT M</td>
</tr>
<tr>
<td>3</td>
<td>1</td>
<td>1.07303E+01</td>
<td>1.000E+00</td>
<td>3.290E-02</td>
<td>9.007E+00</td>
<td>SHIFT ML</td>
</tr>
</tbody>
</table>

\[
\text{RMS ERROR} = 3.11E-02 \quad \text{MAX ERROR} = 5.49E-02 \text{ AT OBS.NO. 6}
\]

\[
\text{RESIDUALS SQUARED} = 7.74E-03
\]

\[
\text{RFACOR} = 0.2562 \text{ PERCENT}
\]

**Figure S4** $^1$H-NMR titration of L$^1$ with (TBA)$_3$HPpi in DMSO-$d_6$. 
Calculations by WinEQNMR Version 1.20 by Michael J. Hynes  
Program run at 10:40:22 on 01/27/2012

IDEAL DATA FOR 1:1 COMPLEX USING CHEMICAL SHIFT (TEST11.FIT)  
Reaction: \( M + L = ML \)  
FILE: TEST11.FIT  
IDEAL DATA: \( K_1 = 63.091; \Delta M = 20.0; \Delta ML = 120.0 \)  
File prepared by M. J. Hynes, October 22 2000

<table>
<thead>
<tr>
<th>NO.</th>
<th>A</th>
<th>PARAMETER</th>
<th>DELTA</th>
<th>ERROR</th>
<th>CONDITION</th>
<th>DESCRIPTION</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1</td>
<td>4.34068E+01</td>
<td>2.000E-01</td>
<td>1.224E+00</td>
<td>1.688E+02</td>
<td>( K_1 )</td>
</tr>
<tr>
<td>2</td>
<td>1</td>
<td>8.51709E+00</td>
<td>2.000E-01</td>
<td>2.894E-03</td>
<td>9.815E+00</td>
<td>( \Delta M )</td>
</tr>
<tr>
<td>3</td>
<td>1</td>
<td>1.06512E+01</td>
<td>1.000E+00</td>
<td>2.964E-02</td>
<td>1.179E+02</td>
<td>( \Delta ML )</td>
</tr>
</tbody>
</table>

\( \Omega \text{RMS ERROR} = 2.87E-03 \)  \( \text{MAX ERROR} = 5.48E-03 \) AT OBS.NO. 14  
\( \text{RFACCTOR} = 1.07E-04 \)  \( \text{SQUARED} = 0.0287 \) PERCENT

**Figure S5** \(^1\text{H-NMR} \) titration of \( L^1 \) with \((\text{TBA})_2\text{Glu}\) in DMSO-\( d_6 \).
Calculations by WinEQNMR Version 1.20 by Michael J. Hynes
Program run at 10:50:01 on 01/27/2012

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 1.12687E+01 2.000E-01 3.822E+00 9.005E+02 3.822E+00 9.005E+02 K1
2 1 8.49895E+00 2.000E-01 1.054E-03 7.199E+00 8.114E+00 8.025E+02 SHIFT M
3 1 8.81263E+00 1.000E+00 1.054E-03 7.199E+00 8.114E+00 8.025E+02 SHIFT ML

0RMS ERROR = 1.06E-03 MAX ERROR = 1.22E-03 AT OBS.NO. 4
RESIDUALS SQUARED = 6.74E-06
RFACTOR = 0.0101 PERCENT

Figure S6 $^1$H-NMR titration of L$^1$ with (TBA)$_2$Mal in DMSO-$d_6$. 
Calculations by WinEQNMR Version 1.20 by Michael J. Hynes
Program run at 15:23:03 on 12/16/2011

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

<table>
<thead>
<tr>
<th>NO.</th>
<th>A</th>
<th>PARAMETER</th>
<th>DELTA</th>
<th>ERROR</th>
<th>CONDITION</th>
<th>DESCRIPTION</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1</td>
<td>8.83907E+01</td>
<td>2.000E-01</td>
<td>2.582E+00</td>
<td>6.315E+01</td>
<td>K1</td>
</tr>
<tr>
<td>2</td>
<td>1</td>
<td>9.45328E+00</td>
<td>2.000E-01</td>
<td>7.234E-03</td>
<td>8.584E+00</td>
<td>SHIFT M</td>
</tr>
<tr>
<td>3</td>
<td>1</td>
<td>1.19201E+01</td>
<td>1.000E+00</td>
<td>2.498E-02</td>
<td>3.644E+01</td>
<td>SHIFT ML</td>
</tr>
</tbody>
</table>

| RMS ERROR | 6.52E-03 | MAX ERROR | 1.21E-02 AT OBS.NO. 1 |
| RESIDUALS SQUARED | 5.53E-04 |
| RFACTOR | 0.0568 PERCENT |

Figure S7 $^1$H-NMR titration of $L^2$ with TBAAcO in DMSO-$d_6$. 

Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry
This journal is © The Royal Society of Chemistry 2013
Calculations by WinEQNMR Version 1.20 by Michael J. Hynes
Program run at 15:52:48   on 12/16/2011

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

<table>
<thead>
<tr>
<th>NO.</th>
<th>A</th>
<th>PARAMETER</th>
<th>DELTA</th>
<th>ERROR</th>
<th>CONDITION</th>
<th>DESCRIPTION</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1</td>
<td>6.47549E+01</td>
<td>2.000E-01</td>
<td>6.441E+00</td>
<td>7.977E+01</td>
<td>K1</td>
</tr>
<tr>
<td>2</td>
<td>1</td>
<td>9.39505E+00</td>
<td>2.000E-01</td>
<td>1.697E+00</td>
<td>8.514E+00</td>
<td>SHIFT M</td>
</tr>
<tr>
<td>3</td>
<td>1</td>
<td>1.14835E+01</td>
<td>1.000E+00</td>
<td>8.118E+00</td>
<td>4.923E+01</td>
<td>SHIFT ML</td>
</tr>
</tbody>
</table>

ORMS ERROR = 1.69E-02  MAX ERROR = 4.48E-02 AT OBS.NO.  1
RESIDUALS SQUARED = 3.98E-03
RFACTOR = 0.1521 PERCENT

**Figure S8** ¹H-NMR titration of L² with TBABzO in DMSO-d₆.
Calculations by WinEQNMR Version 1.20 by Michael J. Hynes
Program run at 16:15:07 on 12/16/2011

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

<table>
<thead>
<tr>
<th>NO.</th>
<th>A</th>
<th>PARAMETER</th>
<th>DELTA</th>
<th>ERROR</th>
<th>CONDITION</th>
<th>DESCRIPTION</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1</td>
<td>1.80508E+02</td>
<td>2.000E-01</td>
<td>9.117E+00</td>
<td>2.953E+01</td>
<td>K1</td>
</tr>
<tr>
<td>2</td>
<td>1</td>
<td>8.00609E+00</td>
<td>2.000E-01</td>
<td>6.519E-03</td>
<td>5.914E+00</td>
<td>SHIFT M</td>
</tr>
<tr>
<td>3</td>
<td>1</td>
<td>8.92857E+00</td>
<td>1.000E+00</td>
<td>1.136E-02</td>
<td>1.626E+01</td>
<td>SHIFT ML</td>
</tr>
</tbody>
</table>

ORMS ERROR = 6.08E-03  MAX ERROR = 1.23E-02 AT OBS.NO. 14
RESIDUALS SQUARED = 5.18E-04
RFACTOR = 0.0653 PERCENT

**Figure S9** $^1$H-NMR titration of $L^2$ with TBAH$_2$PO$_4$ in DMSO-$d_6$. 
Calculations by WinEQNMR Version 1.20 by Michael J. Hynes
Program run at 11:40:12 on 01/27/2012

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

<table>
<thead>
<tr>
<th>NO.</th>
<th>A</th>
<th>PARAMETER</th>
<th>DELTA</th>
<th>ERROR</th>
<th>CONDITION</th>
<th>DESCRIPTION</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1</td>
<td>1.04984E+01</td>
<td>2.000E-01</td>
<td>1.028E+00</td>
<td>8.119E+02</td>
<td>K1</td>
</tr>
<tr>
<td>2</td>
<td>1</td>
<td>9.42859E+00</td>
<td>2.000E-01</td>
<td>2.719E-03</td>
<td>7.974E+00</td>
<td>SHIFT M</td>
</tr>
<tr>
<td>3</td>
<td>1</td>
<td>1.20573E+01</td>
<td>1.000E+00</td>
<td>1.932E-01</td>
<td>7.114E+02</td>
<td>SHIFT ML</td>
</tr>
</tbody>
</table>

RMS ERROR = 3.13E-03 MAX ERROR = 5.92E-03 AT OBS.NO. 4
RESIDUALS SQUARED = 9.79E-05
RFACTOR = 0.0283 PERCENT

Figure S10 1H-NMR titration of L2 with (TBA)2Glu in DMSO-d6.
Calculations by WinEQNMR Version 1.20 by Michael J. Hynes
Program run at 11:49:43 on 01/27/2012

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

<table>
<thead>
<tr>
<th>NO.</th>
<th>A</th>
<th>PARAMETER</th>
<th>DELTA</th>
<th>ERROR</th>
<th>CONDITION</th>
<th>DESCRIPTION</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1</td>
<td>7.58123E+00</td>
<td>2.000E-01</td>
<td>7.427E-01</td>
<td>9.636E+00</td>
<td>K1</td>
</tr>
<tr>
<td>2</td>
<td>1</td>
<td>9.42298E+00</td>
<td>2.000E-01</td>
<td>2.983E-04</td>
<td>2.867E+00</td>
<td>SHIFT M</td>
</tr>
<tr>
<td>3</td>
<td>1</td>
<td>9.49195E+00</td>
<td>1.000E+00</td>
<td>6.581E-03</td>
<td>1.229E+01</td>
<td>SHIFT ML</td>
</tr>
</tbody>
</table>

\textit{ORMS ERROR} = 4.91E-04 \textit{MAX ERROR} = 7.30E-04 AT OBS.NO. 9

\textit{RESIDUALS SQUARED} = 1.44E-06

\textit{RFACtor} = 0.0042 PERCENT

\textbf{Figure S11} \textsuperscript{1}H-NMR titration of L\textsuperscript{2} with con (TBA)\textsubscript{2}Mal in DMSO-\textit{d}_6.
Calculations by WinEQNMR Version 1.20 by Michael J. Hynes
Program run at 12:08:55 on 01/05/2012

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

<table>
<thead>
<tr>
<th>NO.</th>
<th>A</th>
<th>PARAMETER</th>
<th>DELTA</th>
<th>ERROR</th>
<th>CONDITION</th>
<th>DESCRIPTION</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1</td>
<td>1.36759E+02</td>
<td>2.000E+01</td>
<td>6.352E+00</td>
<td>3.705E+01</td>
<td>K1</td>
</tr>
<tr>
<td>2</td>
<td>1</td>
<td>8.64369E+00</td>
<td>2.000E+01</td>
<td>1.102E-02</td>
<td>7.098E+00</td>
<td>SHIFT M</td>
</tr>
<tr>
<td>3</td>
<td>1</td>
<td>1.03948E+01</td>
<td>1.000E+00</td>
<td>2.188E-02</td>
<td>1.974E+01</td>
<td>SHIFT ML</td>
</tr>
</tbody>
</table>

ORMS ERROR = 9.92E-03  MAX ERROR = 1.91E-02 AT OBS.NO. 1
RESIDUALS SQUARED = 1.38E-03
RFACOR = 0.0954 PERCENT

Figure S12 $^1$H-NMR titration of L$^1$ with TBAAcO in DMSO-$d_6$. 
Calculations by WinEQNMR Version 1.20 by Michael J. Hynes
Program run at 12:14:41 on 01/05/2012

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

<table>
<thead>
<tr>
<th>NO.</th>
<th>A</th>
<th>PARAMETER</th>
<th>DELTA</th>
<th>ERROR</th>
<th>CONDITION</th>
<th>DESCRIPTION</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1</td>
<td>7.83725E+01</td>
<td>2.000E-01</td>
<td>2.803E+00</td>
<td>6.018E+01</td>
<td>K1</td>
</tr>
<tr>
<td>2</td>
<td>1</td>
<td>8.60057E+00</td>
<td>2.000E-01</td>
<td>6.112E-03</td>
<td>7.970E+00</td>
<td>SHIFT M</td>
</tr>
<tr>
<td>3</td>
<td>1</td>
<td>1.03937E+01</td>
<td>1.000E+00</td>
<td>2.247E-02</td>
<td>3.531E+01</td>
<td>SHIFT ML</td>
</tr>
</tbody>
</table>

ORMS ERROR = 6.02E-03 MAX ERROR = 1.20E-02 AT OBS.NO. 1
RESIDUALS SQUARED = 5.07E-04
RFACCTOR = 0.0592 PERCENT

**Figure S13** ¹H-NMR titration of L¹ with TBABzO in DMSO-d₆.
Calculations by WinEQNMR Version 1.20 by Michael J. Hynes
Program run at 15:30:21 on 01/04/2012

IDEAL DATA FOR 1:1 COMPLEX USING CHEMICAL SHIFT (TEST11.FIT)

Reaction: M + L = ML
FILE: TEST11.FIT

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

NO.  A  PARAMETER  DELTA  ERROR  CONDITION  DESCRIPTION
1  1  4.35285E+02 2.000E-01 1.209E+01 1.759E+01  K1
2  1  8.50083E+00 2.000E-01 8.853E-03 4.244E+00  SHIFT M
3  1  1.04192E+01 1.000E+00 9.400E-03 1.028E+01  SHIFT ML

0RMS ERROR = 7.52E-03  MAX ERROR = 1.86E-02 AT OBS.NO. 15
RESIDUALS SQUARED = 7.34E-04
RFACTOR = 0.0700 PERCENT

**Figure S14** $^1$H-NMR titration of $L^1$ with TBAH$_2$PO$_4$ in DMSO-$d_6$. 
Calculations by WinEQNMR Version 1.20 by Michael J. Hynes
Program run at 15:22:35   on 01/04/2012

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

<table>
<thead>
<tr>
<th>NO.</th>
<th>A</th>
<th>PARAMETER</th>
<th>DELTA</th>
<th>ERROR</th>
<th>CONDITION</th>
<th>DESCRIPTION</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1</td>
<td>1.60759E+04</td>
<td>2.000E-01</td>
<td>2.052E+03</td>
<td>1.981E+00</td>
<td>K1</td>
</tr>
<tr>
<td>2</td>
<td>1</td>
<td>7.41311E+00</td>
<td>2.000E-01</td>
<td>1.563E-02</td>
<td>1.362E+00</td>
<td>SHIFT M</td>
</tr>
<tr>
<td>3</td>
<td>1</td>
<td>8.16089E+00</td>
<td>1.000E+00</td>
<td>3.130E-03</td>
<td>1.759E+00</td>
<td>SHIFT ML</td>
</tr>
</tbody>
</table>

ORMS ERROR = 8.15E-03  MAX ERROR = 1.16E-02 AT OBS.NO. 2
RESIDUALS SQUARED = 7.31E-04
RFACCTOR = 0.0892 PERCENT

Figure S15 $^1$H-NMR titration of L$^1$ with (TBA)$_3$HPi in DMSO-$d_6$. 
Calculations by WinEQNMR Version 1.20 by Michael J. Hynes  
Program run at 13:56:31 on 01/23/2012

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

<table>
<thead>
<tr>
<th>NO.</th>
<th>A</th>
<th>PARAMETER</th>
<th>DELTA</th>
<th>ERROR</th>
<th>CONDITION</th>
<th>DESCRIPTION</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1</td>
<td>1.98147E+01</td>
<td>2.000E-01</td>
<td>1.999E-01</td>
<td>2.863E+02</td>
<td>K1</td>
</tr>
<tr>
<td>2</td>
<td>1</td>
<td>8.54750E+00</td>
<td>2.000E-01</td>
<td>5.498E-04</td>
<td>9.004E+00</td>
<td>SHIFT M</td>
</tr>
<tr>
<td>3</td>
<td>1</td>
<td>1.05114E+01</td>
<td>1.000E+00</td>
<td>1.200E-02</td>
<td>2.226E+02</td>
<td>SHIFT ML</td>
</tr>
</tbody>
</table>

$$\text{ORMS ERROR} = 6.37E-04 \quad \text{MAX ERROR} = 1.11E-03 \text{ AT OBS.NO. 16}$$  
$$\text{RESIDUALS SQUARED} = 5.67E-06$$  
$$\text{RFACCTOR} = 0.0065 \ \text{PERCENT}$$

**Figure S16** $^1$H-NMR titration of L$^3$ with (TBA)$_2$Glu in DMSO-$d_6$.  

Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry  
This journal is © The Royal Society of Chemistry 2013
Calculations by WinEQNMR Version 1.20 by Michael J. Hynes
Program run at 14:07:16 on 01/23/2012

IDEAL DATA FOR 1:1 COMPLEX USING CHEMICAL SHIFT (TEST11.FIT)
Reaction: \[ M + L = ML \]
FILE: TEST11.FIT
IDEAL DATA: \( K_1 = 63.091 \); \( \Delta M = 20.0 \); \( \Delta ML = 120.0 \)
File prepared by M. J. Hynes, October 22 2000

<table>
<thead>
<tr>
<th>NO.</th>
<th>A</th>
<th>PARAMETER</th>
<th>DELTA</th>
<th>ERROR</th>
<th>CONDITION</th>
<th>DESCRIPTION</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1</td>
<td>9.79880E+00</td>
<td>2.000E-01</td>
<td>1.173E+00</td>
<td>5.838E+01</td>
<td>( K_1 )</td>
</tr>
<tr>
<td>2</td>
<td>1</td>
<td>8.58775E+00</td>
<td>2.000E-01</td>
<td>3.937E-04</td>
<td>2.958E+00</td>
<td>SHIFT M</td>
</tr>
<tr>
<td>3</td>
<td>1</td>
<td>8.72901E+00</td>
<td>1.000E+00</td>
<td>1.238E-02</td>
<td>5.177E+01</td>
<td>SHIFT ML</td>
</tr>
</tbody>
</table>

\( \text{ORMS ERROR} = 7.17E-04 \) \( \text{MAX ERROR} = 1.30E-03 \) AT OBS.NO. 12
RESIDUALS SQUARED = 4.63E-06
RFACCTOR = 0.0072 PERCENT

**Figure S17** \(^1\)H-NMR titration of \( L^1 \) with \((\text{TBA})_2\text{Mal in DMSO-}d_6\).
Calculations by WinEQNMR Version 1.20 by Michael J. Hynes
Program run at 15:47:29   on 06/06/2012

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

<table>
<thead>
<tr>
<th>NO.</th>
<th>A</th>
<th>PARAMETER</th>
<th>DELTA</th>
<th>ERROR</th>
<th>CONDITION</th>
<th>DESCRIPTION</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1</td>
<td>1.97486E+02</td>
<td>2.000E-01</td>
<td>1.029E+01</td>
<td>2.566E+01</td>
<td>K1</td>
</tr>
<tr>
<td>2</td>
<td>1</td>
<td>8.80900E+00</td>
<td>2.000E-01</td>
<td>2.233E-02</td>
<td>6.163E+00</td>
<td>SHIFT M</td>
</tr>
<tr>
<td>3</td>
<td>1</td>
<td>1.14063E+01</td>
<td>1.000E+00</td>
<td>2.981E-02</td>
<td>1.301E+01</td>
<td>SHIFT ML</td>
</tr>
</tbody>
</table>

RMS ERROR = 1.86E-02   MAX ERROR = 3.52E-02 AT OBS.NO. 1
RESIDUALS SQUARED = 4.49E-03
RFACCTOR = 0.1644 PERCENT

Figure S18 $^1$H-NMR titration of L$^+$ with TBAAcO in DMSO-$d_6$.  

Calculations by WinEQNMR Version 1.20 by Michael J. Hynes
Program run at 16:08:11 on 06/06/2012

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

<table>
<thead>
<tr>
<th>NO.</th>
<th>A</th>
<th>PARAMETER</th>
<th>DELTA</th>
<th>ERROR</th>
<th>CONDITION</th>
<th>DESCRIPTION</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1</td>
<td>1.06438E+02</td>
<td>2.000E-01</td>
<td>3.285E+00</td>
<td>4.759E+01</td>
<td>K1</td>
</tr>
<tr>
<td>2</td>
<td>1</td>
<td>8.75456E+00</td>
<td>2.000E-01</td>
<td>8.965E-03</td>
<td>7.752E+00</td>
<td>SHIFT M</td>
</tr>
<tr>
<td>3</td>
<td>1</td>
<td>1.12730E+01</td>
<td>1.000E+00</td>
<td>2.354E-02</td>
<td>2.626E+01</td>
<td>SHIFT ML</td>
</tr>
</tbody>
</table>

0 RMS ERROR = 8.32E-03 MAX ERROR = 1.59E-02 AT OBS.NO. 1
RESIDUALS SQUARED = 9.68E-04
RFACCTOR = 0.0772 PERCENT

Figure S19 $^1$H-NMR titration of L$^4$ with TBABzO in DMSO-$d_6$. 
Calculations by WinEQNMR Version 1.20 by Michael J. Hynes
Program run at 15:59:57 on 06/06/2012

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 6.98292E+02 2.000E-01 1.614E+01 1.074E+01 K1
2 1 8.76393E+00 2.000E-01 7.809E-03 3.386E+00 SHIFT M
3 1 1.04988E+01 1.000E+00 5.360E-03 6.271E+00 SHIFT ML

ORMS ERROR = 6.38E-03 MAX ERROR = 1.28E-02 AT OBS.NO. 1
RESIDUALS SQUARED = 5.70E-04
RFACTOR = 0.0581 PERCENT

**Figure S20** $^1$H-NMR titration of L$^4$ with TBAH$_2$PO$_4$ in DMSO-$d_6$. 

Calculations by WinEQNMR Version 1.20 by Michael J. Hynes
Program run at 16:21:44   on 06/06/2012

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

<table>
<thead>
<tr>
<th>NO.</th>
<th>A</th>
<th>PARAMETER</th>
<th>DELTA</th>
<th>ERROR</th>
<th>CONDITION</th>
<th>DESCRIPTION</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1</td>
<td>7.71037E+01</td>
<td>2.000E-01</td>
<td>1.590E+00</td>
<td>6.830E+01</td>
<td>K1</td>
</tr>
<tr>
<td>2</td>
<td>1</td>
<td>8.73194E+00</td>
<td>2.000E-01</td>
<td>3.174E-03</td>
<td>6.310E+00</td>
<td>SHIFT M</td>
</tr>
<tr>
<td>3</td>
<td>1</td>
<td>1.05761E+01</td>
<td>1.000E+00</td>
<td>1.407E-02</td>
<td>4.574E+01</td>
<td>SHIFT ML</td>
</tr>
</tbody>
</table>

0RMS ERROR = 3.02E-03  MAX ERROR = 5.99E-03 AT OBS.NO. 10
RESIDUALS SQUARED = 9.10E-05
RFACCTOR = 0.0281 PERCENT

Figure S21 $^1$H-NMR titration of L$^4$ with (TBA)$_2$Glu in DMSO-$d_6$. 
Calculations by WinEQNMR Version 1.20 by Michael J. Hynes
Program run at 14:48:56 on 06/06/2012

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

<table>
<thead>
<tr>
<th>NO.</th>
<th>A</th>
<th>PARAMETER</th>
<th>DELTA</th>
<th>ERROR</th>
<th>CONDITION</th>
<th>DESCRIPTION</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1</td>
<td>1.21073E+02</td>
<td>2.000E-01</td>
<td>4.592E+00</td>
<td>4.259E+01</td>
<td>K1</td>
</tr>
<tr>
<td>2</td>
<td>1</td>
<td>9.56785E+00</td>
<td>2.000E-01</td>
<td>1.224E-02</td>
<td>7.395E+00</td>
<td>SHIFT M</td>
</tr>
<tr>
<td>3</td>
<td>1</td>
<td>1.21602E+01</td>
<td>1.000E+00</td>
<td>2.839E-02</td>
<td>2.325E+01</td>
<td>SHIFT ML</td>
</tr>
</tbody>
</table>

0RMS ERROR = 1.12E-02 MAX ERROR = 2.33E-02 AT OBS.NO. 1
RESIDUALS SQUARED = 1.77E-03
RFACTOR = 0.0954 PERCENT

Figure S22 $^1$H-NMR titration of $L^5$ with TBAAcO in DMSO-$d_6$. 

Calculations by WinEQNMR Version 1.20 by Michael J. Hynes
Program run at 15:03:17 on 06/06/2012

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

<table>
<thead>
<tr>
<th>NO.</th>
<th>A</th>
<th>PARAMETER</th>
<th>DELTA</th>
<th>ERROR</th>
<th>CONDITION</th>
<th>DESCRIPTION</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1</td>
<td>6.03580E+01</td>
<td>2.000E-01</td>
<td>3.150E+00</td>
<td>9.171E+01</td>
<td>K1</td>
</tr>
<tr>
<td>2</td>
<td>1</td>
<td>9.55363E+00</td>
<td>2.000E-01</td>
<td>9.247E-03</td>
<td>8.442E+00</td>
<td>SHIFT M</td>
</tr>
<tr>
<td>3</td>
<td>1</td>
<td>1.19574E+01</td>
<td>1.000E+00</td>
<td>4.975E-02</td>
<td>5.886E+01</td>
<td>SHIFT ML</td>
</tr>
</tbody>
</table>

RMSE ERROR = 8.78E-03  MAX ERROR = 1.72E-02 AT OBS.NO. 1
RESIDUALS SQUARED = 9.25E-04
RFACTOR = 0.0762 PERCENT

**Figure S23** $^1$H-NMR titration of L$^5$ with TBABzO in DMSO-$d_6$. 

Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry
This journal is © The Royal Society of Chemistry 2013
Calculations by WinEQNMR Version 1.20 by Michael J. Hynes
Program run at 14:36:47 on 06/06/2012

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

<table>
<thead>
<tr>
<th>NO.</th>
<th>A</th>
<th>PARAMETER</th>
<th>DELTA</th>
<th>ERROR</th>
<th>CONDITION</th>
<th>DESCRIPTION</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1</td>
<td>1.66748E+02</td>
<td>2.000E-01</td>
<td>1.195E+01</td>
<td>2.979E+01</td>
<td>K1</td>
</tr>
<tr>
<td>2</td>
<td>1</td>
<td>9.59614E+00</td>
<td>2.000E-01</td>
<td>1.690E-02</td>
<td>7.748E+00</td>
<td>SHIFT M</td>
</tr>
<tr>
<td>3</td>
<td>1</td>
<td>1.10940E+01</td>
<td>1.000E+00</td>
<td>2.570E-02</td>
<td>1.365E+01</td>
<td>SHIFT ML</td>
</tr>
</tbody>
</table>

$\text{ORMS ERROR} = 1.35E-02$ $\text{MAX ERROR} = 2.25E-02$ AT OBS.NO. 3
$\text{RESIDUALS SQUARED} = 2.18E-03$
$\text{RFACCTOR} = 0.1170$ PERCENT

**Figure S24** $^1\text{H-NMR}$ titration of L$^5$ with TBAH$_2$PO$_4$ in DMSO-$d_6$. 
Calculations by WinEQNMR Version 1.20 by Michael J. Hynes
Program run at 15:26:34 on 06/06/2012

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

<table>
<thead>
<tr>
<th>NO.</th>
<th>A</th>
<th>PARAMETER</th>
<th>DELTA</th>
<th>ERROR</th>
<th>CONDITION</th>
<th>DESCRIPTION</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1</td>
<td>1.52949E+01</td>
<td>2.000E-01</td>
<td>4.882E-01</td>
<td>4.931E+02</td>
<td>K1</td>
</tr>
<tr>
<td>2</td>
<td>1</td>
<td>9.50565E+00</td>
<td>2.000E-01</td>
<td>1.295E-03</td>
<td>8.319E+00</td>
<td>SHIFT M</td>
</tr>
<tr>
<td>3</td>
<td>1</td>
<td>1.19438E+01</td>
<td>1.000E+00</td>
<td>5.325E-02</td>
<td>4.130E+02</td>
<td>SHIFT ML</td>
</tr>
</tbody>
</table>

ORMS ERROR = 1.53E-03  MAX ERROR = 3.52E-03 AT OBS.NO. 14
RESIDUALS SQUARED = 2.80E-05
RFACCTOR = 0.0139 PERCENT

**Figure S25** $^1$H-NMR titration of L$^5$ with (TBA)$_2$Glu in DMSO-$d_6$. 
Calculations by WinEQNMR Version 1.20 by Michael J. Hynes
Program run at 11:44:14 on 02/08/2012

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

<table>
<thead>
<tr>
<th>NO.</th>
<th>A</th>
<th>PARAMETER</th>
<th>DELTA</th>
<th>ERROR</th>
<th>CONDITION</th>
<th>DESCRIPTION</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1</td>
<td>2.99470E+02</td>
<td>2.000E-01</td>
<td>1.765E+01</td>
<td>1.905E+01</td>
<td>K1</td>
</tr>
<tr>
<td>2</td>
<td>1</td>
<td>8.79504E+00</td>
<td>2.000E-01</td>
<td>1.701E-02</td>
<td>4.785E+00</td>
<td>SHIFT M</td>
</tr>
<tr>
<td>3</td>
<td>1</td>
<td>1.05260E+01</td>
<td>1.000E+00</td>
<td>1.959E-02</td>
<td>1.045E+01</td>
<td>SHIFT ML</td>
</tr>
</tbody>
</table>

0RMS ERROR = 1.52E-02; MAX ERROR = 2.90E-02 AT OBS.NO. 6
RESIDUALS SQUARED = 3.22E-03
RFACOR = 0.1405 PERCENT

Figure S26 $^1$H-NMR titration of L$^6$ with TBAAcO in DMSO-$d_6$. 
Calculations by WinEQNMR Version 1.20 by Michael J. Hynes
Program run at 17:06:57  on 03/01/2012

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

<table>
<thead>
<tr>
<th>NO.</th>
<th>A</th>
<th>PARAMETER</th>
<th>DELTA</th>
<th>ERROR</th>
<th>CONDITION</th>
<th>DESCRIPTION</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1</td>
<td>8.79467E+01</td>
<td>2.000E-01</td>
<td>1.500E+00</td>
<td>5.130E+01</td>
<td>K1</td>
</tr>
<tr>
<td>2</td>
<td>1</td>
<td>8.77283E+00</td>
<td>2.000E-01</td>
<td>3.254E-03</td>
<td>7.445E+00</td>
<td>SHIFT M</td>
</tr>
<tr>
<td>3</td>
<td>1</td>
<td>1.06041E+01</td>
<td>1.000E+00</td>
<td>1.029E-02</td>
<td>2.960E+01</td>
<td>SHIFT ML</td>
</tr>
</tbody>
</table>

RMS ERROR = 3.12E-03  MAX ERROR = 5.76E-03 AT OBS.NO. 1
RESIDUALS SQUARED = 1.26E-04
RFACCTOR =  0.0297 PERCENT

Figure S27 $^1$H-NMR titration of L$^6$ with TBABzO in DMSO-$d_6$. 

Calculations by WinEQNMR Version 1.20 by Michael J. Hynes
Program run at 12:10:49 on 02/08/2012

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

<table>
<thead>
<tr>
<th>NO.</th>
<th>A</th>
<th>PARAMETER</th>
<th>DELTA</th>
<th>ERROR</th>
<th>CONDITION</th>
<th>DESCRIPTION</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1</td>
<td>2.73005E+02</td>
<td>2.000E-01</td>
<td>1.018E+01</td>
<td>2.087E+01</td>
<td>K1</td>
</tr>
<tr>
<td>2</td>
<td>1</td>
<td>8.78206E+00</td>
<td>2.000E-01</td>
<td>9.409E-03</td>
<td>5.442E+00</td>
<td>SHIFT M</td>
</tr>
<tr>
<td>3</td>
<td>1</td>
<td>1.02701E+01</td>
<td>1.000E+00</td>
<td>1.072E-02</td>
<td>1.076E+01</td>
<td>SHIFT ML</td>
</tr>
</tbody>
</table>

0RMS ERROR = 8.03E-03 MAX ERROR = 1.69E-02 AT OBS.NO. 2
RESIDUALS SQUARED = 9.02E-04
RFACCTOR = 0.0757 PERCENT

**Figure S28** $^1$H-NMR titration of L$^6$ with TBAH$_2$PO$_4$ in DMSO-$d_6$. 
Calculations by WinEQNMR Version 1.20 by Michael J. Hynes
Program run at 16:06:56 on 03/01/2012

IDEAL DATA FOR 1:1 COMPLEX USING CHEMICAL SHIFT (TEST11.FIT)
Reaction: \( M + L = ML \)
FILE: TEST11.FIT
IDEAL DATA: \( K_1 = 63.091; \ \delta M = 20.0; \ \delta ML = 120.0 \)
File prepared by M. J. Hynes, October 22 2000

<table>
<thead>
<tr>
<th>NO.</th>
<th>A</th>
<th>PARAMETER</th>
<th>DELTA</th>
<th>ERROR</th>
<th>CONDITION</th>
<th>DESCRIPTION</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1</td>
<td>3.02974E+01</td>
<td>2.000E-01</td>
<td>5.495E-01</td>
<td>1.614E+02</td>
<td>( K_1 )</td>
</tr>
<tr>
<td>2</td>
<td>1</td>
<td>8.77531E+00</td>
<td>2.000E-01</td>
<td>1.252E-03</td>
<td>8.871E+00</td>
<td>SHIFT M</td>
</tr>
<tr>
<td>3</td>
<td>1</td>
<td>1.03904E+01</td>
<td>1.000E+00</td>
<td>1.555E-02</td>
<td>1.151E+02</td>
<td>SHIFT ML</td>
</tr>
</tbody>
</table>

ORMS ERROR = 1.40E-03  MAX ERROR = 2.43E-03 AT OBS.NO. 3
RESIDUALS SQUARED = 2.74E-05
RFACOR = 0.0139 PERCENT

Figure S29: \(^1\)H-NMR titration of \( L^6 \) with (TBA)\(_2\)Glu in DMSO-\( d_6 \).
Calculations by WinEQNMR Version 1.20 by Michael J. Hynes
Program run at 13:22:08 on 07/25/2012

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

<table>
<thead>
<tr>
<th>NO.</th>
<th>A</th>
<th>PARAMETER</th>
<th>DELTA</th>
<th>ERROR</th>
<th>CONDITION</th>
<th>DESCRIPTION</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1</td>
<td>1.47071E+01</td>
<td>2.000E-01</td>
<td>1.452E+00</td>
<td>2.970E+02</td>
<td>K1</td>
</tr>
<tr>
<td>2</td>
<td>1</td>
<td>8.57506E+00</td>
<td>2.000E-01</td>
<td>1.292E-03</td>
<td>5.168E+00</td>
<td>SHIFT M</td>
</tr>
<tr>
<td>3</td>
<td>1</td>
<td>9.37610E+00</td>
<td>1.000E+00</td>
<td>5.472E-02</td>
<td>2.557E+02</td>
<td>SHIFT ML</td>
</tr>
</tbody>
</table>

0 RMS ERROR = 1.42E-03 MAX ERROR = 1.65E-03 AT OBS.NO. 3
RESIDUALS SQUARED = 1.01E-05
RFACCTOR = 0.0129 PERCENT

**Figure S30** $^1$H-NMR titration of L$^3$ with TBAAcO in DMSO-d$_6$/0.5%H$_2$O.
Calculations by WinEQNMR Version 1.20 by Michael J. Hynes
Program run at 13:29:07 on 07/25/2012

IDEAL DATA FOR 1:1 COMPLEX USING CHEMICAL SHIFT (TEST11.FIT)
Reaction: \( M + L = ML \)
FILE: TEST11.FIT
IDEAL DATA: \( K_1 = 63.091; \) \( \Delta M = 20.0; \) \( \Delta ML = 120.0 \)
File prepared by M. J. Hynes, October 22 2000

<table>
<thead>
<tr>
<th>NO.</th>
<th>A</th>
<th>PARAMETER</th>
<th>DELTA</th>
<th>ERROR</th>
<th>CONDITION</th>
<th>DESCRIPTION</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1</td>
<td>1.14006E+01</td>
<td>2.000E-01</td>
<td>1.607E+00</td>
<td>5.616E+02</td>
<td>( K_1 )</td>
</tr>
<tr>
<td>2</td>
<td>1</td>
<td>8.57504E+00</td>
<td>2.000E-01</td>
<td>2.008E-03</td>
<td>7.313E+00</td>
<td>\text{SHIFT M}</td>
</tr>
<tr>
<td>3</td>
<td>1</td>
<td>9.71930E+00</td>
<td>1.000E+00</td>
<td>1.117E-01</td>
<td>4.838E+02</td>
<td>\text{SHIFT ML}</td>
</tr>
</tbody>
</table>

\( \text{ORMS ERROR} = 1.99E-03 \) \( \text{MAX ERROR} = 2.45E-03 \) AT OBS.NO. 1
\( \text{RESIDUALS SQUARED} = 2.39E-05 \)
\( \text{RFACTOR} = 0.0187 \) \text{PERCENT}

**Figure S31** \( ^1 \text{H-NMR} \) titration of \( L^3 \) with TBABz0 in DMSO-\( d_6/0.5\%H_2O \).
Calculations by WinEQNMR Version 1.20 by Michael J. Hynes
Program run at 13:34:19 on 07/25/20

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

<table>
<thead>
<tr>
<th>NO.</th>
<th>A</th>
<th>PARAMETER</th>
<th>DELTA</th>
<th>ERROR</th>
<th>CONDITION</th>
<th>DESCRIPTION</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1</td>
<td>2.07454E+01</td>
<td>2.000E-01</td>
<td>4.325E+00</td>
<td>2.344E+02</td>
<td>K1</td>
</tr>
<tr>
<td>2</td>
<td>1</td>
<td>8.56248E+00</td>
<td>2.000E-01</td>
<td>7.751E-03</td>
<td>7.191E+00</td>
<td>SHIFT M</td>
</tr>
<tr>
<td>3</td>
<td>1</td>
<td>1.00055E+01</td>
<td>1.000E+00</td>
<td>1.895E-01</td>
<td>1.860E+02</td>
<td>SHIFT ML</td>
</tr>
</tbody>
</table>

0RMS ERROR = 7.80E-03  MAX ERROR = 1.02E-02 AT OBS.NO. 5
RESIDUALS SQUARED = 4.26E-04
RFACCTOR = 0.0743 PERCENT

**Figure S32** $^1$H-NMR titration of $L^3$ with TBAH$_2$PO$_4$ in DMSO-$d_6$/0.5%H$_2$O.
Calculations by WinEQNMR Version 1.20 by Michael J. Hynes
Program run at 17:52:28 on 06/21/2012

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

<table>
<thead>
<tr>
<th>NO.</th>
<th>A</th>
<th>PARAMETER</th>
<th>DELTA</th>
<th>ERROR</th>
<th>CONDITION</th>
<th>DESCRIPTION</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1</td>
<td>5.69754E+02</td>
<td>2.000E-01</td>
<td>3.132E+01</td>
<td>1.634E+01</td>
<td>K1</td>
</tr>
<tr>
<td>2</td>
<td>1</td>
<td>7.43388E+00</td>
<td>2.000E-01</td>
<td>8.033E-03</td>
<td>5.719E+00</td>
<td>SHIFT M</td>
</tr>
<tr>
<td>3</td>
<td>1</td>
<td>8.01066E+00</td>
<td>1.000E+00</td>
<td>3.742E-03</td>
<td>7.258E+00</td>
<td>SHIFT ML</td>
</tr>
</tbody>
</table>

ORMS ERROR = 3.64E-03  MAX ERROR = 7.45E-03 AT OBS.NO. 4
RESIDUALS SQUARED = 1.19E-04
RFACTOR = 0.0401 PERCENT

Figure S33 ¹H-NMR titration of L³ with (TBA)₃HPpi in DMSO-d₆/0.5%H₂O.
Calculations by WinEQNMR Version 1.20 by Michael J. Hynes
Program run at 17:41:55 on 06/06/2012

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

<table>
<thead>
<tr>
<th>NO.</th>
<th>A</th>
<th>PARAMETER</th>
<th>DELTA</th>
<th>ERROR</th>
<th>CONDITION</th>
<th>DESCRIPTION</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1</td>
<td>3.68944E+02</td>
<td>2.000E-01</td>
<td>3.693E+01</td>
<td>1.359E+01</td>
<td>K1</td>
</tr>
<tr>
<td>2</td>
<td>1</td>
<td>8.62850E+00</td>
<td>2.000E-01</td>
<td>6.646E-03</td>
<td>3.673E+00</td>
<td>SHIFT M</td>
</tr>
<tr>
<td>3</td>
<td>1</td>
<td>8.99647E+00</td>
<td>1.000E+00</td>
<td>6.268E-03</td>
<td>7.884E+00</td>
<td>SHIFT ML</td>
</tr>
</tbody>
</table>

\[
\text{ORMS ERROR} = 5.57E-03 \quad \text{MAX ERROR} = 1.08E-02 \text{ AT OBS.NO. 1}
\]

\[
\text{RESIDUALS SQUARED} = 3.41E-04
\]

\[
\text{RFACCTOR} = 0.0557 \text{ PERCENT}
\]

**Figure S34** ¹H-NMR titration of L³ with NaAMP in DMSO-\(d_6\).
Calculations by WinEQNMR Version 1.20 by Michael J. Hynes
Program run at 18:02:50 on 06/21/2012

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

<table>
<thead>
<tr>
<th>NO.</th>
<th>A</th>
<th>PARAMETER</th>
<th>DELTA ERROR</th>
<th>CONDITION</th>
<th>DESCRIPTION</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1</td>
<td>3.53294E+01</td>
<td>2.000E-01</td>
<td>5.083E+00</td>
<td>1.465E+02</td>
</tr>
<tr>
<td>2</td>
<td>1</td>
<td>8.62097E+00</td>
<td>2.000E-01</td>
<td>3.994E-03</td>
<td>1.175E+01</td>
</tr>
<tr>
<td>3</td>
<td>1</td>
<td>9.00015E+00</td>
<td>1.000E+00</td>
<td>2.445E-02</td>
<td>9.371E+01</td>
</tr>
</tbody>
</table>

\[ \text{ORMS ERROR} = 2.88E-03 \quad \text{MAX ERROR} = 5.89E-03 \quad \text{AT OBS.NO. 1} \]
\[ \text{RESIDUALS SQUARED} = 7.47E-05 \]
\[ \text{RFACTOR} = 0.0286 \quad \text{PERCENT} \]

**Figure S35** $^1$H-NMR titration of L$^3$ with NaAMP in DMSO-$d_6$/0.5%H$_2$O.
Figure S35 Emission spectra of L^3 (1.50 \cdot 10^{-4} \text{ M}) (pink line) and of L^3 in the presence of 1 equivalent of TBAOH (blue line) in DMSO.

Figure S36 Emission spectra of L^6 (8.5 \cdot 10^{-5} \text{ M}) (pink line) and of L^6 in the presence of 1 equivalent of TBAOH (blue line) in DMSO.
Figure S37 Changes in the intensity emission of L₃ (1.50·10⁻⁴ M) at 500 nm after addition of 10 equivalents of HPpi³⁻ and 20 equivalents of the other anions in DMSO.
Table S1. Crystal data and structure refinement details.

<table>
<thead>
<tr>
<th>Identification code</th>
<th>2012acc0042 (MAR94_4 BENZOATO)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Empirical formula</td>
<td>C_{75}H_{107}N_{7}O_{6} C_{29}H_{52}N_{7}O_{4}, 2(C_{16}H_{36}N), 2(C_{7}H_{5}O_{2})</td>
</tr>
<tr>
<td>Formula weight</td>
<td>1202.68</td>
</tr>
<tr>
<td>Temperature</td>
<td>100(2) K</td>
</tr>
<tr>
<td>Wavelength</td>
<td>0.71075 Å</td>
</tr>
<tr>
<td>Crystal system</td>
<td>Monoclinic</td>
</tr>
<tr>
<td>Space group</td>
<td>P2_1/n</td>
</tr>
<tr>
<td>Unit cell dimensions</td>
<td>a = 8.623(3) Å, b = 41.598(13) Å, c = 19.413(6) Å</td>
</tr>
<tr>
<td>Volume</td>
<td>6846(4) Å³</td>
</tr>
<tr>
<td>Density (calculated)</td>
<td>1.167 Mg / m³</td>
</tr>
<tr>
<td>Absorption coefficient</td>
<td>0.074 mm⁻¹</td>
</tr>
<tr>
<td>F(000)</td>
<td>2616</td>
</tr>
<tr>
<td>Crystal</td>
<td>Rod; Colourless</td>
</tr>
<tr>
<td>Crystal size</td>
<td>0.22 × 0.04 × 0.02 mm³</td>
</tr>
<tr>
<td>θ range for data collection</td>
<td>2.94 – 25.03°</td>
</tr>
<tr>
<td>Index ranges</td>
<td>–10 ≤ h ≤ 9, –49 ≤ k ≤ 44, –13 ≤ l ≤ 23</td>
</tr>
<tr>
<td>Reflections collected</td>
<td>26420</td>
</tr>
<tr>
<td>Independent reflections</td>
<td>12037 [R_int = 0.0798]</td>
</tr>
<tr>
<td>Completeness to θ = 25.03°</td>
<td>99.5 %</td>
</tr>
<tr>
<td>Absorption correction</td>
<td>Semi–empirical from equivalents</td>
</tr>
<tr>
<td>Max. and min. transmission</td>
<td>0.9985 and 0.9840</td>
</tr>
<tr>
<td>Refinement method</td>
<td>Full-matrix least-squares on F²</td>
</tr>
<tr>
<td>Data / restraints / parameters</td>
<td>12037 / 0 / 801</td>
</tr>
<tr>
<td>Goodness-of-fit on F²</td>
<td>1.196</td>
</tr>
<tr>
<td>Final R indices [F² &gt; 2σ(F²)]</td>
<td>R1 = 0.1018, wR2 = 0.1504</td>
</tr>
<tr>
<td>R indices (all data)</td>
<td>R1 = 0.1606, wR2 = 0.1762</td>
</tr>
<tr>
<td>Largest diff. peak and hole</td>
<td>0.384 and –0.249 e Å⁻³</td>
</tr>
</tbody>
</table>


Table S2. Hydrogen bonds [Å and °].

<table>
<thead>
<tr>
<th>D–H···A</th>
<th>d(D–H)</th>
<th>d(H···A)</th>
<th>d(D···A)</th>
<th>θ(DHA)</th>
</tr>
</thead>
<tbody>
<tr>
<td>N1–H901···O3</td>
<td>0.88</td>
<td>1.94</td>
<td>2.816(4)</td>
<td>171.9</td>
</tr>
<tr>
<td>N2–H902···O4</td>
<td>0.88</td>
<td>1.95</td>
<td>2.809(4)</td>
<td>166.1</td>
</tr>
<tr>
<td>N4–H904···O5</td>
<td>0.88</td>
<td>1.96</td>
<td>2.831(4)</td>
<td>170.1</td>
</tr>
<tr>
<td>N5–H905···O6</td>
<td>0.88</td>
<td>1.91</td>
<td>2.760(4)</td>
<td>161.6</td>
</tr>
</tbody>
</table>