This journal is © The Royal Society of Chemistry 2007

# Molecular structures of free quinuclidine and its adducts with metal trihydrides, $MH_3$ (M = B, Al or Ga), studied by gas-phase electron diffraction, X-ray diffraction and quantum chemical calculations

Derek A. Wann,[a] Frank Blockhuys,[b] Christian Van Alsenoy,[b] Heather E. Robertson,[a] Hans-Jörg Himmel,[c] Christina Y. Tang,[d] Andrew R. Cowley,[d] Anthony J. Downs[d] and David W. H. Rankin*[a]

## Supplementary Information

**Table S1** Experimental parameters for the GED analyses of **1**–**3**.[a]

| | **1** | | **2** | | **3** | |
|---|---|---|---|---|---|---|
| Nozzle-to-film distance[b] | 93.45 | 254.87 | 203.39[c] | 264.45 | 93.71 | 261.44 |
| $T_{sample}$ | 382 | 365 | 353 | 343 | 371 | 368 |
| $T_{nozzle}$ | 398 | 393 | 363 | 358 | 383 | 378 |
| $\Delta s$ | 0.4 | 0.2 | 0.2 | 0.1 | 0.2 | 0.1 |
| $s_{min}$ | 10.0 | 2.0 | 4.0 | 2.0 | 8.0 | 2.0 |
| $sw_1$ | 12.0 | 4.0 | 6.0 | 4.0 | 10.0 | 4.0 |
| $sw_2$ | 30.4 | 13.0 | 18.0 | 6.8 | 31.0 | 12.9 |
| $s_{max}$ | 35.6 | 15.2 | 21.0 | 8.0 | 36.0 | 14.0 |
| Correlation parameter | 0.4376 | 0.4321 | 0.4653 | 0.4970 | –0.2051 | 0.4723 |
| Scale factor, $k$ | 0.687(10) | 0.739(4) | 0.462(7) | 0.455(8) | 0.588(5) | 0.591(2) |
| Electron wavelength, λ | 6.02 | 6.02 | 6.13 | 6.13 | 6.13 | 6.13 |

[a] Nozzle-to-film distances are in mm, temperatures are in K, $s$ values are in $nm^{-1}$ and electron wavelengths are in pm. Values in parentheses are standard deviations of the last digits. [b] Determined by reference to scattering patterns of benzene. [c] For **2** data were collected using a medium nozzle-to-film distance rather than the short distance used for **1** and **3**. This was necessary because thermal decomposition occurred at the higher temperature required to obtain data at the shorter distance.

This journal is © The Royal Society of Chemistry 2007

**Table S2** Interatomic distances ($r_a$ in Å), experimental and calculated amplitudes of vibration ($u_{h1}$ in Å) and curvilinear corrections ($k_{h1}$ in Å) from the SARACEN refinement of **1**. See Figure 2 for atom numbering.

| | Atom pair | $r_a$ | $u_{h1}$ (exp.) | $k_{h1}$ | $u_{h1}$ (calc.) |
|---|---|---|---|---|---|
| $u_1$ | C(2)–H′(2) | 1.109(2) | 0.086(2) | 0.004 | 0.077 |
| $u_2$ | C(2)–H(2) | 1.109(2) | 0.086(tied to $u_1$) | 0.004 | 0.077 |
| $u_3$ | C(4)–H(4) | 1.109(2) | 0.086(tied to $u_1$) | 0.004 | 0.077 |
| $u_4$ | C(3)–H(3) | 1.109(2) | 0.086(tied to $u_1$) | 0.004 | 0.077 |
| $u_5$ | C(3)–H′(3) | 1.109(2) | 0.086(tied to $u_1$) | 0.004 | 0.077 |
| $u_6$ | N(1)–C(2) | 1.463(2) | 0.040(2) | 0.001 | 0.050 |
| $u_7$ | C(3)–C(4) | 1.547(3) | 0.041(tied to $u_6$) | 0.002 | 0.052 |
| $u_8$ | C(2)–C(3) | 1.562(4) | 0.042(tied to $u_6$) | 0.001 | 0.053 |
| $u_9$ | H(2)···H′(2) | 1.771(14) | 0.123(fixed) | 0.001 | 0.123 |
| $u_{10}$ | H(3)···H′(3) | 1.777(14) | 0.123(fixed) | 0.001 | 0.123 |
| $u_{11}$ | N(1)···H(2) | 2.075(12) | 0.104(3) | –0.001 | 0.103 |
| $u_{12}$ | N(1)···H′(2) | 2.084(12) | 0.105(tied to $u_{11}$) | –0.001 | 0.104 |
| $u_{13}$ | H(3)···C(4) | 2.169(12) | 0.108(tied to $u_{11}$) | –0.001 | 0.108 |
| $u_{14}$ | C(3)···H(4) | 2.188(3) | 0.106(tied to $u_{11}$) | 0.001 | 0.106 |
| $u_{15}$ | H(3)···C(4) | 2.181(11) | 0.109(tied to $u_{11}$) | –0.001 | 0.109 |
| $u_{16}$ | C(2)···H(3) | 2.257(10) | 0.108(tied to $u_{11}$) | –0.001 | 0.108 |
| $u_{17}$ | C(2)···H′(3) | 2.198(13) | 0.108(tied to $u_{11}$) | –0.001 | 0.108 |
| $u_{18}$ | H(2)···C(3) | 2.189(14) | 0.107(tied to $u_{11}$) | 0.000 | 0.107 |
| $u_{19}$ | H′(2)···C(3) | 2.247(11) | 0.106(tied to $u_{11}$) | 0.000 | 0.106 |
| $u_{20}$ | H(2)···H(3) | 2.399(21) | 0.173(fixed) | 0.013 | 0.173 |
| $u_{21}$ | H′(4)···H′(3) | 2.399(21) | 0.174(fixed) | 0.013 | 0.174 |
| $u_{22}$ | C(2)···C(6) | 2.378(2) | 0.068(tied to $u_{27}$) | –0.003 | 0.067 |
| $u_{23}$ | H(2)···H′(7) | 2.386(21) | 0.233(fixed) | 0.014 | 0.233 |
| $u_{24}$ | H(3)···H(4) | 2.463(14) | 0.167(fixed) | 0.005 | 0.167 |
| $u_{25}$ | C(3)···C(5) | 2.511(4) | 0.073(tied to $u_{27}$) | –0.002 | 0.072 |
| $u_{26}$ | C(2)···C(4) | 2.493(2) | 0.064(tied to $u_{27}$) | –0.004 | 0.063 |
| $u_{27}$ | N(1)···C(3) | 2.508(1) | 0.064(1) | –0.004 | 0.064 |
| $u_{28}$ | C(2)···H(6) | 2.541(16) | 0.162(tied to $u_{27}$) | –0.001 | 0.161 |
| $u_{29}$ | H′(3)···H(4) | 2.544(14) | 0.167(fixed) | 0.001 | 0.167 |
| $u_{30}$ | H(3)···H′(8) | 2.550(23) | 0.252(fixed) | 0.012 | 0.252 |
| $u_{31}$ | N(1)···C(4) | 2.580(2) | 0.065(tied to $u_{27}$) | –0.002 | 0.065 |
| $u_{32}$ | C(3)···H′(8) | 2.680(17) | 0.172(tied to $u_{27}$) | –0.001 | 0.171 |
| $u_{33}$ | C(2)···H′(7) | 2.690(14) | 0.162(tied to $u_{27}$) | –0.004 | 0.161 |
| $u_{34}$ | C(2)···C(8) | 2.835(8) | 0.118(5) | –0.008 | 0.108 |
| $u_{35}$ | C(3)···H(5) | 2.824(16) | 0.185(tied to $u_{34}$) | –0.003 | 0.169 |
| $u_{36}$ | H′(2)···H(3) | 2.947(17) | 0.161(fixed) | –0.005 | 0.161 |
| $u_{37}$ | H(2)···H′(8) | 2.974(24) | 0.406(fixed) | –0.005 | 0.406 |
| $u_{38}$ | C(2)···C(5) | 2.940(6) | 0.088(tied to $u_{34}$) | –0.009 | 0.081 |
| $u_{39}$ | H(2)···H′(3) | 2.977(20) | 0.140(fixed) | –0.011 | 0.140 |
| $u_{40}$ | H(2)···C(8) | 3.062(17) | 0.277(tied to $u_{34}$) | –0.011 | 0.254 |
| $u_{41}$ | C(2)···H′(8) | 3.093(19) | 0.270(tied to $u_{34}$) | –0.011 | 0.248 |
| $u_{42}$ | H(2)···C(4) | 3.147(11) | 0.168(tied to $u_{34}$) | –0.011 | 0.155 |
| $u_{43}$ | N(1)···H′(3) | 3.159(11) | 0.163(tied to $u_{34}$) | –0.011 | 0.150 |
| $u_{44}$ | N(1)···H(3) | 3.340(9) | 0.126(5) | –0.014 | 0.119 |
| $u_{45}$ | C(2)···H(7) | 3.317(8) | 0.105(tied to $u_{44}$) | –0.012 | 0.100 |
| $u_{46}$ | C(2)···H′(6) | 3.319(8) | 0.106(tied to $u_{44}$) | –0.011 | 0.101 |

This journal is © The Royal Society of Chemistry 2007

| | | | | | |
|---|---|---|---|---|---|
| $u_{47}$ | H′(2)···C(4) | 3.337(8) | 0.127(tied to $u_{44}$) | –0.014 | 0.121 |
| $u_{48}$ | C(3)···H′(5) | 3.461(8) | 0.108(tied to $u_{44}$) | –0.012 | 0.103 |
| $u_{49}$ | C(3)···H(8) | 3.468(7) | 0.109(tied to $u_{44}$) | –0.011 | 0.103 |
| $u_{50}$ | C(2)···H(4) | 3.467(3) | 0.106(tied to $u_{44}$) | –0.010 | 0.100 |
| $u_{51}$ | H′(2)···C(5) | 3.461(16) | 0.210(tied to $u_{44}$) | –0.018 | 0.200 |
| $u_{52}$ | C(2)···H(5) | 3.475(15) | 0.205(tied to $u_{44}$) | –0.017 | 0.195 |
| $u_{53}$ | H(2)···H(6) | 3.566(19) | 0.167(fixed) | –0.016 | 0.167 |
| $u_{54}$ | H′(2)···H′(6) | 3.600(21) | 0.175(fixed) | –0.011 | 0.175 |
| $u_{55}$ | N(1)···H(4) | 3.683(2) | 0.102(tied to $u_{44}$) | –0.009 | 0.097 |
| $u_{56}$ | H′(3)···H′(5) | 3.717() | 0.178(fixed) | –0.016 | 0.178 |
| $u_{57}$ | H(3)···H(5) | 3.767(19) | 0.185(fixed) | –0.012 | 0.185 |
| $u_{58}$ | H′(2)···H(5) | 3.746(22) | 0.338(fixed) | –0.022 | 0.338 |
| $u_{59}$ | C(2)···H(8) | 3.881(8) | 0.113(5) | –0.022 | 0.115 |
| $u_{60}$ | H′(2)···C(8) | 3.890(7) | 0.114(tied to $u_{59}$) | –0.022 | 0.115 |
| $u_{61}$ | C(2)···H′(5) | 3.868(9) | 0.113(tied to $u_{59}$) | –0.021 | 0.115 |
| $u_{62}$ | H(2)···C(5) | 3.878(9) | 0.114(tied to $u_{59}$) | –0.021 | 0.115 |
| $u_{63}$ | H(2)···H(4) | 4.047(12) | 0.185(fixed) | –0.018 | 0.185 |
| $u_{64}$ | H(2)···H(8) | 4.149(18) | 0.264(fixed) | –0.029 | 0.264 |
| $u_{65}$ | H′(2)···H′(8) | 4.186(19) | 0.261(fixed) | –0.029 | 0.261 |
| $u_{66}$ | H(2)···H′(6) | 4.132(13) | 0.135(fixed) | –0.017 | 0.135 |
| $u_{67}$ | H′(2)···H(4) | 4.254(10) | 0.150(fixed) | –0.022 | 0.150 |
| $u_{68}$ | H(3)···H′(5) | 4.307(11) | 0.137(fixed) | –0.018 | 0.137 |
| $u_{69}$ | H′(2)···H′(5) | 4.469(15) | 0.186(fixed) | –0.035 | 0.186 |
| $u_{70}$ | H(2)···H(5) | 4.497(15) | 0.182(fixed) | –0.035 | 0.182 |
| $u_{71}$ | H(2)···H′(5) | 4.704(12) | 0.190(fixed) | –0.034 | 0.190 |
| $u_{72}$ | H′(2)···H(8) | 4.896(8) | 0.131(fixed) | –0.040 | 0.131 |

**Table S3** Least-squares correlation matrix (×100) from the GED refinement of **1**.[a]

| | $p_5$ | $p_8$ | $p_{11}$ | $p_{15}$ | $u_6$ | $u_{34}$ |
|---|---|---|---|---|---|---|
| $p_1$ | | | 61 | | | –55 |
| $p_2$ | | | | | 76 | |
| $p_3$ | 65 | –59 | | | –56 | |
| $p_8$ | | | | –54 | | |
| $p_{11}$ | | | | | | –83 |

[a] Only values ≥ 50% are shown.

This journal is © The Royal Society of Chemistry 2007

**Table S4** Cartesian coordinates (in Å) for the GED refined structure ($r_{h1}$) of **1**.

| | *x* | *y* | *z* |
|---|---|---|---|
| N(1) | 0.0000 | 0.0000 | 0.0000 |
| C(2) | 0.0000 | 1.3802 | –0.5040 |
| H(2) | –0.9646 | 1.8298 | –0.1760 |
| H′(2) | 0.7940 | 1.9389 | 0.0413 |
| C(3) | 0.1478 | 1.4471 | –2.0505 |
| H(3) | –0.5922 | 2.1131 | –2.5493 |
| H′(3) | 1.1402 | 1.8542 | –2.3497 |
| C(4) | 0.0000 | 0.0000 | –2.5880 |
| H(4) | 0.0000 | 0.0000 | –3.7016 |
| C(5) | 1.1793 | –0.8516 | –2.0505 |
| H(5) | 2.1262 | –0.5437 | –2.5493 |
| H′(5) | 1.0357 | –1.9145 | –2.3497 |
| C(6) | 1.1952 | –0.6901 | –0.5040 |
| H(6) | 2.0669 | –0.0795 | –0.1760 |
| H′(6) | 1.2822 | –1.6571 | 0.0413 |
| C(7) | –1.1952 | –0.6901 | –0.5040 |
| H(7) | –1.1023 | –1.7502 | –0.1760 |
| H′(7) | –2.0762 | –0.2818 | 0.0413 |
| C(8) | –1.3271 | –0.5956 | –2.0505 |
| H(8) | –1.5339 | –1.5695 | –2.5493 |
| H′(8) | –2.1759 | 0.0603 | –2.3497 |

This journal is © The Royal Society of Chemistry 2007

**Table S5** Interatomic distances ($r_a$ in Å), experimental and calculated amplitudes of vibration ($u_{h1}$ in Å) and curvilinear corrections ($k_{h1}$ in Å) from the SARACEN refinement of **2**. See Figure 2 for atom numbering.

| | Atom pair | $r_a$ | $u_{h1}$ (exp.) | $k_{h1}$ | $u_{h1}$ (calc.) |
|---|---|---|---|---|---|
| $u_9$ | C(8)–H(8) | 1.090(4) | 0.091(tied to $u_7$) | 0.004 | 0.075 |
| $u_{10}$ | C(8)–H′(8) | 1.090(4) | 0.091(tied to $u_7$) | 0.004 | 0.075 |
| $u_1$ | C(4)–H(4) | 1.090(4) | 0.091(tied to $u_7$) | 0.004 | 0.075 |
| $u_8$ | C(7)–H(7) | 1.090(4) | 0.090(tied to $u_7$) | 0.004 | 0.074 |
| $u_7$ | C(7)–H′(7) | 1.090(4) | 0.090(4) | 0.004 | 0.074 |
| $u_{11}$ | B(9)–H(12) | 1.218(9) | 0.103(tied to $u_7$) | 0.005 | 0.085 |
| $u_{12}$ | N(1)–C(7) | 1.489(6) | 0.046(4) | 0.001 | 0.049 |
| $u_{13}$ | C(8)–C(4) | 1.536(6) | 0.047(tied to $u_{12}$) | 0.002 | 0.050 |
| $u_{14}$ | C(7)–C(8) | 1.539(6) | 0.048(tied to $u_{12}$) | 0.001 | 0.050 |
| $u_2$ | N(1)–B(9) | 1.624(9) | 0.062(tied to $u_{12}$) | 0.003 | 0.065 |
| $u_{16}$ | H(8)···H′(8) | 1.760(26) | 0.119(fixed) | 0.002 | 0.119 |
| $u_{15}$ | H′(7)···H(7) | 1.768(28) | 0.118(fixed) | 0.002 | 0.118 |
| $u_{17}$ | H(12)···H(11) | 2.030(19) | 0.132(fixed) | 0.001 | 0.132 |
| $u_{18}$ | N(1)···H(7) | 2.072(14) | 0.078(9) | 0.000 | 0.100 |
| $u_{19}$ | N(1)···H′(7) | 2.080(14) | 0.079(tied to $u_{18}$) | 0.000 | 0.100 |
| $u_{24}$ | C(7)···H′(8) | 2.107(32) | 0.081(tied to $u_{18}$) | 0.000 | 0.103 |
| $u_{25}$ | H(7)···C(8) | 2.125(29) | 0.080(tied to $u_{18}$) | 0.000 | 0.103 |
| $u_{20}$ | H′(8)···C(4) | 2.167(14) | 0.081(tied to $u_{18}$) | 0.000 | 0.104 |
| $u_{21}$ | C(8)···H(4) | 2.172(9) | 0.080(tied to $u_{18}$) | 0.002 | 0.102 |
| $u_{22}$ | H(8)···C(4) | 2.178(14) | 0.082(tied to $u_{18}$) | 0.000 | 0.105 |
| $u_{23}$ | C(7)···H(8) | 2.219(28) | 0.081(tied to $u_{18}$) | 0.000 | 0.103 |
| $u_{26}$ | H′(7)···C(8) | 2.267(30) | 0.080(tied to $u_{18}$) | 0.001 | 0.102 |
| $u_{27}$ | N(1)···H(12) | 2.278(16) | 0.090(tied to $u_{18}$) | –0.001 | 0.116 |
| $u_{28}$ | H(7)···H(8) | 2.323(37) | 0.165(fixed) | 0.018 | 0.165 |
| $u_{29}$ | H′(7)···H′(8) | 2.361(40) | 0.167(fixed) | 0.017 | 0.167 |
| $u_{30}$ | H′(7)···H(6) | 2.407(38) | 0.220(fixed) | 0.021 | 0.220 |
| $u_{32}$ | H′(7)···H(10) | 2.414(39) | 0.236(fixed) | 0.038 | 0.236 |
| $u_{31}$ | C(7)···C(2) | 2.424(12) | 0.092(tied to $u_{36}$) | –0.004 | 0.066 |
| $u_{33}$ | H(8)···H(4) | 2.458(22) | 0.160(fixed) | 0.008 | 0.160 |
| $u_{36}$ | N(1)···C(8) | 2.482(11) | 0.084(3) | –0.007 | 0.060 |
| $u_{34}$ | H(7)···H(11) | 2.487(38) | 0.231(fixed) | 0.038 | 0.231 |
| $u_{35}$ | C(8)···C(3) | 2.488(12) | 0.095(tied to $u_{36}$) | –0.004 | 0.068 |
| $u_{37}$ | C(7)···C(4) | 2.489(16) | 0.085(tied to $u_{36}$) | –0.006 | 0.061 |
| $u_{40}$ | C(7)···H(6) | 2.515(24) | 0.225(tied to $u_{36}$) | 0.000 | 0.160 |
| $u_{41}$ | C(7)···B(9) | 2.542(10) | 0.115(tied to $u_{36}$) | 0.004 | 0.082 |
| $u_3$ | N(1)···C(4) | 2.560(11) | 0.083(tied to $u_{36}$) | –0.002 | 0.059 |
| $u_{38}$ | H′(8)···H(4) | 2.566(21) | 0.163(fixed) | 0.004 | 0.163 |
| $u_{39}$ | H′(8)···H(5) | 2.566(38) | 0.234(fixed) | 0.017 | 0.234 |
| $u_{42}$ | H′(7)···B(9) | 2.583(24) | 0.212(tied to $u_{36}$) | 0.012 | 0.151 |
| $u_{43}$ | C(8)···H′(3) | 2.641(23) | 0.231(tied to $u_{36}$) | –0.001 | 0.165 |
| $u_{46}$ | C(7)···H(11) | 2.708(24) | 0.256(tied to $u_{52}$) | 0.008 | 0.176 |
| $u_{44}$ | C(7)···H′(2) | 2.722(23) | 0.233(tied to $u_{52}$) | –0.003 | 0.161 |
| $u_{45}$ | H(7)···B(9) | 2.771(26) | 0.233(tied to $u_{52}$) | 0.007 | 0.161 |
| $u_{51}$ | H′(7)···H(11) | 2.795(42) | 0.286(fixed) | 0.005 | 0.286 |
| $u_{54}$ | H(7)···H′(3) | 2.813(84) | 0.483(fixed) | –0.012 | 0.483 |
| $u_{47}$ | C(8)···H(5) | 2.815(23) | 0.239(tied to $u_{52}$) | –0.003 | 0.165 |

This journal is © The Royal Society of Chemistry 2007

| | | | | | |
|---|---|---|---|---|---|
| $u_{49}$ | C(7)···H(10) | 2.817(26) | 0.259(tied to $u_{52}$) | 0.005 | 0.179 |
| $u_{48}$ | C(7)···C(3) | 2.831(30) | 0.169(tied to $u_{36}$) | –0.012 | 0.121 |
| $u_{53}$ | H(7)···H′(8) | 2.864(50) | 0.208(tied to $u_{52}$) | –0.011 | 0.143 |
| $u_{52}$ | C(7)···C(5) | 2.928(21) | 0.129(8) | –0.014 | 0.089 |
| $u_{50}$ | H′(7)···H(8) | 2.932(49) | 0.161(fixed) | –0.005 | 0.161 |
| $u_{55}$ | H(7)···C(3) | 2.956(59) | 0.424(tied to $u_{52}$) | –0.017 | 0.293 |
| $u_{56}$ | C(7)···H′(3) | 3.011(56) | 0.408(tied to $u_{52}$) | –0.016 | 0.282 |
| $u_{58}$ | H(7)···C(4) | 3.068(32) | 0.234(tied to $u_{52}$) | –0.015 | 0.161 |
| $u_{57}$ | N(1)···H′(8) | 3.072(25) | 0.223(tied to $u_{52}$) | –0.015 | 0.154 |
| $u_{59}$ | H(7)···H(10) | 3.268(40) | 0.281(fixed) | –0.008 | 0.281 |
| $u_{60}$ | N(1)···H(8) | 3.308(20) | 0.124(7) | –0.018 | 0.123 |
| $u_{62}$ | C(7)···H′(6) | 3.332(15) | 0.099(tied to $u_{60}$) | –0.011 | 0.098 |
| $u_{63}$ | C(7)···H(2) | 3.332(15) | 0.098(tied to $u_{60}$) | –0.013 | 0.098 |
| $u_{61}$ | H′(7)···C(4) | 3.347(22) | 0.129(tied to $u_{60}$) | –0.018 | 0.128 |
| $u_{64}$ | C(8)···H′(5) | 3.432(14) | 0.100(tied to $u_{60}$) | –0.013 | 0.099 |
| $u_{65}$ | C(8)···H(3) | 3.442(13) | 0.100(tied to $u_{60}$) | –0.011 | 0.099 |
| $u_{67}$ | C(7)···H(4) | 3.444(18) | 0.098(tied to $u_{60}$) | –0.012 | 0.097 |
| $u_{68}$ | C(7)···H(5) | 3.459(35) | 0.231(tied to $u_{60}$) | –0.024 | 0.229 |
| $u_{66}$ | H′(7)···C(5) | 3.462(38) | 0.238(tied to $u_{60}$) | –0.025 | 0.236 |
| $u_{69}$ | C(7)···H(12) | 3.542(14) | 0.113(tied to $u_{60}$) | –0.013 | 0.112 |
| $u_{70}$ | H(7)···H(2) | 3.545(24) | 0.161(fixed) | –0.017 | 0.161 |
| $u_{71}$ | H′(7)···H′(2) | 3.587(25) | 0.166(fixed) | –0.010 | 0.166 |
| $u_{4}$ | N(1)···H(4) | 3.648(12) | 0.092(tied to $u_{60}$) | –0.008 | 0.091 |
| $u_{73}$ | H′(8)···H′(3) | 3.692(23) | 0.169(fixed) | –0.016 | 0.169 |
| $u_{75}$ | H′(7)···H(12) | 3.714(25) | 0.163(fixed) | –0.013 | 0.163 |
| $u_{72}$ | H′(7)···H(5) | 3.752(54) | 0.414(fixed) | –0.031 | 0.414 |
| $u_{74}$ | H(8)···H(3) | 3.757(24) | 0.173(fixed) | –0.011 | 0.173 |
| $u_{76}$ | H(7)···H(12) | 3.782(28) | 0.172(fixed) | –0.014 | 0.172 |
| $u_{79}$ | H(7)···C(5) | 3.812(16) | 0.147(tied to $u_{81}$) | –0.029 | 0.109 |
| $u_{80}$ | C(7)···H′(5) | 3.824(16) | 0.146(tied to $u_{81}$) | –0.028 | 0.108 |
| $u_{78}$ | C(7)···H(3) | 3.869(29) | 0.155(tied to $u_{81}$) | –0.029 | 0.115 |
| $u_{77}$ | H′(7)···C(3) | 3.871(26) | 0.153(tied to $u_{81}$) | –0.030 | 0.114 |
| $u_{81}$ | C(8)···B(9) | 3.917(11) | 0.112(6) | –0.008 | 0.083 |
| $u_{82}$ | H(7)···H(4) | 3.954(36) | 0.192(fixed) | –0.021 | 0.192 |
| $u_{84}$ | H(7)···H(3) | 4.052(61) | 0.294(fixed) | –0.039 | 0.294 |
| $u_{85}$ | H′(7)···H′(3) | 4.114(57) | 0.285(fixed) | –0.040 | 0.285 |
| $u_{83}$ | H′(7)···H(2) | 4.120(23) | 0.131(fixed) | –0.015 | 0.131 |
| $u_{88}$ | C(8)···H(11) | 4.174(28) | 0.194(tied to $u_{5}$) | –0.010 | 0.165 |
| $u_{5}$ | C(4)···B(9) | 4.182(13) | 0.092(7) | –0.003 | 0.078 |
| $u_{87}$ | C(8)···H(10) | 4.196(26) | 0.199(tied to $u_{5}$) | –0.009 | 0.169 |
| $u_{86}$ | H′(7)···H(4) | 4.252(26) | 0.156(fixed) | –0.025 | 0.156 |
| $u_{89}$ | H(8)···H′(5) | 4.289(22) | 0.132(fixed) | –0.016 | 0.132 |
| $u_{92}$ | H′(8)···H(10) | 4.388(40) | 0.263(fixed) | –0.010 | 0.263 |
| $u_{93}$ | H′(8)···B(9) | 4.391(28) | 0.214(tied to $u_{5}$) | –0.018 | 0.182 |
| $u_{90}$ | H′(7)···H′(5) | 4.424(35) | 0.208(fixed) | –0.047 | 0.208 |
| $u_{91}$ | H(7)···H(5) | 4.436(28) | 0.201(fixed) | –0.047 | 0.201 |
| $u_{97}$ | H(7)···H′(5) | 4.601(23) | 0.194(fixed) | –0.044 | 0.194 |
| $u_{94}$ | H(8)···B(9) | 4.652(23) | 0.169(tied to $u_{5}$) | –0.022 | 0.144 |
| $u_{96}$ | C(4)···H(12) | 4.656(20) | 0.166(tied to $u_{5}$) | –0.010 | 0.141 |
| $u_{95}$ | H(8)···H(11) | 4.671(37) | 0.231(fixed) | –0.016 | 0.231 |

This journal is © The Royal Society of Chemistry 2007

| | | | | | |
|---|---|---|---|---|---|
| $u_{98}$ | C(8)···H(12) | 4.744(17) | 0.144(tied to $u_5$) | –0.022 | 0.123 |
| $u_{100}$ | H′(8)···H(11) | 4.777(39) | 0.213(fixed) | –0.025 | 0.213 |
| $u_{99}$ | H′(7)···H(3) | 4.870(24) | 0.132(fixed) | –0.052 | 0.132 |
| $u_{101}$ | H(8)···H(10) | 5.015(36) | 0.198(fixed) | –0.027 | 0.198 |
| $u_{102}$ | H′(8)···H(12) | 5.266(32) | 0.213(fixed) | –0.034 | 0.213 |
| $u_6$ | H(4)···B(9) | 5.268(14) | 0.123(tied to $u_5$) | –0.010 | 0.105 |
| $u_{103}$ | H(8)···H(12) | 5.514(27) | 0.175(fixed) | –0.038 | 0.175 |
| $u_{104}$ | H(4)···H(12) | 5.715(21) | 0.162(fixed) | –0.017 | 0.162 |

This journal is © The Royal Society of Chemistry 2007

**Table S6** Interatomic distances ($r_a$ in Å), experimental and calculated amplitudes of vibration ($u_{h1}$ in Å) and curvilinear corrections ($k_{h1}$ in Å) from the SARACEN refinement of **3**. See Figure 2 for atom numbering.

| | Atom pair | $r_a$ | $u_{h1}$ (exp.) | $k_{h1}$ | $u_{h1}$ (calc.) |
|---|---|---|---|---|---|
| $u_{10}$ | C(8)–H′(8) | 1.107(1) | 0.097(1) | 0.004 | 0.075 |
| $u_9$ | C(8)–H(8) | 1.107(1) | 0.097(tied to $u_{10}$) | 0.004 | 0.075 |
| $u_7$ | C(7)–H′(7) | 1.107(1) | 0.096(tied to $u_{10}$) | 0.004 | 0.074 |
| $u_8$ | C(7)–H(7) | 1.107(1) | 0.096(tied to $u_{10}$) | 0.004 | 0.074 |
| $u_1$ | C(4)–H(4) | 1.107(1) | 0.097(tied to $u_{10}$) | 0.004 | 0.075 |
| $u_{11}$ | N(1)–C(7) | 1.481(2) | 0.060(1) | 0.001 | 0.049 |
| $u_{12}$ | C(8)–C(4) | 1.528(2) | 0.061(tied to $u_{11}$) | 0.002 | 0.050 |
| $u_{13}$ | C(7)–C(8) | 1.535(2) | 0.062(tied to $u_{11}$) | 0.001 | 0.051 |
| $u_{14}$ | Al(9)–H(12) | 1.585(7) | 0.116(tied to $u_{11}$) | 0.004 | 0.095 |
| $u_{16}$ | H(8)···H′(8) | 1.770(20) | 0.119(fixed) | 0.003 | 0.119 |
| $u_{15}$ | H′(7)···H(7) | 1.792(18) | 0.118(fixed) | 0.003 | 0.118 |
| $u_2$ | N(1)–Al(9) | 2.033(5) | 0.082(2) | 0.003 | 0.075 |
| $u_{17}$ | N(1)···H(7) | 2.096(12) | 0.110(tied to $u_2$) | 0.001 | 0.100 |
| $u_{18}$ | N(1)···H′(7) | 2.113(12) | 0.111(tied to $u_2$) | 0.001 | 0.101 |
| $u_{24}$ | H(7)···C(8) | 2.150(20) | 0.113(tied to $u_2$) | 0.001 | 0.103 |
| $u_{19}$ | H′(8)···C(4) | 2.165(13) | 0.114(tied to $u_2$) | 0.001 | 0.104 |
| $u_{21}$ | H(8)···C(4) | 2.177(13) | 0.116(tied to $u_2$) | 0.001 | 0.105 |
| $u_{20}$ | C(8)···H(4) | 2.179(3) | 0.113(tied to $u_2$) | 0.005 | 0.103 |
| $u_{23}$ | C(7)···H′(8) | 2.181(19) | 0.113(tied to $u_2$) | 0.001 | 0.103 |
| $u_{25}$ | H′(7)···C(8) | 2.198(15) | 0.112(tied to $u_2$) | 0.002 | 0.102 |
| $u_{22}$ | C(7)···H(8) | 2.215(18) | 0.114(tied to $u_2$) | 0.001 | 0.104 |
| $u_{26}$ | H(7)···H(8) | 2.349(33) | 0.168(fixed) | 0.029 | 0.168 |
| $u_{27}$ | H′(7)···H′(8) | 2.368(28) | 0.169(fixed) | 0.029 | 0.169 |
| $u_{29}$ | C(7)···C(2) | 2.373(5) | 0.073(tied to $u_{32}$) | –0.007 | 0.067 |
| $u_{28}$ | H′(7)···H(6) | 2.438(27) | 0.228(fixed) | 0.033 | 0.228 |
| $u_{33}$ | C(7)···C(4) | 2.459(6) | 0.067(tied to $u_{32}$) | –0.011 | 0.062 |
| $u_{30}$ | H(8)···H(4) | 2.462(19) | 0.163(fixed) | 0.014 | 0.163 |
| $u_{31}$ | C(8)···C(3) | 2.474(3) | 0.076(tied to $u_{32}$) | –0.006 | 0.070 |
| $u_{32}$ | N(1)···C(8) | 2.509(5) | 0.067(4) | –0.011 | 0.062 |
| $u_{36}$ | C(7)···H(6) | 2.512(18) | 0.190(tied to $u_{32}$) | 0.000 | 0.176 |
| $u_{34}$ | H′(8)···H(4) | 2.566(19) | 0.170(fixed) | 0.009 | 0.170 |
| $u_{35}$ | H(8)···H′(3) | 2.570(30) | 0.242(fixed) | 0.026 | 0.242 |
| $u_3$ | N(1)···C(4) | 2.603(6) | 0.066(tied to $u_{32}$) | –0.002 | 0.060 |
| $u_{38}$ | C(8)···H′(3) | 2.646(19) | 0.192(tied to $u_{32}$) | –0.001 | 0.177 |
| $u_{51}$ | H(7)···H′(3) | 2.651(33) | 0.622(fixed) | –0.024 | 0.622 |
| $u_{39}$ | H(12)···H(11) | 2.699(15) | 0.157(fixed) | –0.001 | 0.157 |
| $u_{37}$ | C(7)···H′(2) | 2.725(15) | 0.211(tied to $u_{46}$) | –0.004 | 0.178 |
| $u_{43}$ | C(7)···C(3) | 2.748(10) | 0.160(tied to $u_{32}$) | –0.022 | 0.147 |
| $u_{40}$ | H′(7)···H(10) | 2.775(39) | 0.328(fixed) | 0.069 | 0.328 |
| $u_{45}$ | N(1)···H(12) | 2.780(16) | 0.188(tied to $u_{46}$) | –0.003 | 0.158 |
| $u_{42}$ | C(8)···H(5) | 2.818(18) | 0.212(tied to $u_{46}$) | –0.004 | 0.179 |
| $u_{44}$ | H′(7)···H(8) | 2.841(27) | 0.177(fixed) | –0.006 | 0.177 |
| $u_{41}$ | H(7)···H(11) | 2.857(35) | 0.323(fixed) | 0.068 | 0.323 |
| $u_{52}$ | H(7)···C(3) | 2.873(25) | 0.369(fixed) | –0.028 | 0.369 |
| $u_{47}$ | C(7)···C(5) | 2.919(4) | 0.129(tied to $u_{46}$) | –0.025 | 0.109 |
| $u_{53}$ | C(7)···H′(3) | 2.929(26) | 0.420(tied to $u_{46}$) | –0.028 | 0.353 |

This journal is © The Royal Society of Chemistry 2007

| | | | | | |
|---|---|---|---|---|---|
| $u_{46}$ | C(7)···Al(9) | 2.930(3) | 0.124(2) | 0.007 | 0.105 |
| $u_{49}$ | H′(7)···Al(9) | 2.955(19) | 0.221(tied to $u_{46}$) | 0.023 | 0.186 |
| $u_{48}$ | H(7)···H′(8) | 2.961(20) | 0.157(fixed) | –0.015 | 0.157 |
| $u_{55}$ | H(7)···C(4) | 3.041(21) | 0.221(tied to $u_{46}$) | –0.023 | 0.186 |
| $u_{54}$ | N(1)···H′(8) | 3.123(22) | 0.209(tied to $u_{46}$) | –0.022 | 0.176 |
| $u_{50}$ | H(7)···Al(9) | 3.140(22) | 0.240(tied to $u_{46}$) | 0.013 | 0.202 |
| $u_{56}$ | C(7)···H(11) | 3.171(27) | 0.303(tied to $u_{46}$) | 0.013 | 0.255 |
| $u_{62}$ | H′(7)···H(11) | 3.263(41) | 0.395(fixed) | 0.007 | 0.395 |
| $u_{57}$ | C(7)···H(10) | 3.263(28) | 0.232(tied to $u_{58}$) | 0.009 | 0.255 |
| $u_{59}$ | H′(7)···C(4) | 3.309(11) | 0.134(tied to $u_{58}$) | –0.028 | 0.148 |
| $u_{60}$ | C(7)···H(2) | 3.313(8) | 0.090(tied to $u_{58}$) | –0.018 | 0.099 |
| $u_{61}$ | C(7)···H′(6) | 3.330(8) | 0.090(tied to $u_{58}$) | –0.015 | 0.099 |
| $u_{58}$ | N(1)···H(8) | 3.350(15) | 0.128(5) | –0.027 | 0.141 |
| $u_{65}$ | C(8)···H′(5) | 3.429(9) | 0.091(tied to $u_{58}$) | –0.017 | 0.100 |
| $u_{67}$ | C(7)···H(4) | 3.432(6) | 0.090(tied to $u_{58}$) | –0.016 | 0.099 |
| $u_{66}$ | C(8)···H(3) | 3.437(9) | 0.091(tied to $u_{58}$) | –0.015 | 0.100 |
| $u_{63}$ | H′(7)···C(5) | 3.476(15) | 0.278(tied to $u_{58}$) | –0.042 | 0.306 |
| $u_{64}$ | C(7)···H(5) | 3.484(18) | 0.266(tied to $u_{58}$) | –0.040 | 0.293 |
| $u_{68}$ | H(7)···H(2) | 3.564(19) | 0.166(fixed) | –0.022 | 0.166 |
| $u_{69}$ | H′(7)···H′(2) | 3.648(20) | 0.170(fixed) | –0.012 | 0.170 |
| $u_{72}$ | H′(8)···H′(3) | 3.696(20) | 0.174(fixed) | –0.021 | 0.174 |
| $u_{70}$ | H(7)···H(10) | 3.699(39) | 0.389(fixed) | –0.013 | 0.389 |
| $u_{4}$ | N(1)···H(4) | 3.708(6) | 0.091(tied to $u_{76}$) | –0.008 | 0.092 |
| $u_{73}$ | H(8)···H(3) | 3.757(22) | 0.178(fixed) | –0.013 | 0.178 |
| $u_{71}$ | H′(7)···H(5) | 3.766(24) | 0.545(fixed) | –0.053 | 0.545 |
| $u_{74}$ | C(7)···H(3) | 3.804(11) | 0.123(tied to $u_{76}$) | –0.046 | 0.124 |
| $u_{76}$ | H(7)···C(5) | 3.805(14) | 0.112(5) | –0.047 | 0.113 |
| $u_{75}$ | H′(7)···C(3) | 3.809(9) | 0.122(tied to $u_{76}$) | –0.048 | 0.123 |
| $u_{77}$ | C(7)···H′(5) | 3.822(12) | 0.111(tied to $u_{76}$) | –0.046 | 0.112 |
| $u_{78}$ | H(7)···H(4) | 3.929(23) | 0.218(fixed) | –0.030 | 0.218 |
| $u_{81}$ | H(7)···H(3) | 3.948(26) | 0.360(fixed) | –0.063 | 0.360 |
| $u_{83}$ | H′(7)···H′(3) | 4.020(27) | 0.347(fixed) | –0.065 | 0.347 |
| $u_{79}$ | C(7)···H(12) | 4.078(14) | 0.145(tied to $u_{76}$) | –0.023 | 0.146 |
| $u_{80}$ | H′(7)···H(2) | 4.168(17) | 0.131(fixed) | –0.015 | 0.131 |
| $u_{82}$ | H′(7)···H(4) | 4.217(13) | 0.176(fixed) | –0.037 | 0.176 |
| $u_{84}$ | H(8)···H′(5) | 4.295(19) | 0.133(fixed) | –0.017 | 0.133 |
| $u_{85}$ | H′(7)···H(12) | 4.295(22) | 0.197(fixed) | –0.022 | 0.197 |
| $u_{87}$ | C(8)···Al(9) | 4.328(4) | 0.161(4) | –0.016 | 0.101 |
| $u_{86}$ | H(7)···H(12) | 4.349(26) | 0.210(fixed) | –0.025 | 0.210 |
| $u_{89}$ | H(7)···H(5) | 4.455(19) | 0.246(fixed) | –0.075 | 0.246 |
| $u_{88}$ | H′(7)···H′(5) | 4.457(17) | 0.259(fixed) | –0.075 | 0.259 |
| $u_{90}$ | C(8)···H(10) | 4.582(26) | 0.273(tied to $u_{5}$) | –0.014 | 0.248 |
| $u_{92}$ | H(7)···H′(5) | 4.585(17) | 0.221(fixed) | –0.070 | 0.221 |
| $u_{5}$ | C(4)···Al(9) | 4.631(6) | 0.096(4) | –0.007 | 0.088 |
| $u_{91}$ | C(8)···H(11) | 4.640(26) | 0.267(tied to $u_{5}$) | –0.017 | 0.243 |
| $u_{94}$ | H′(8)···H(10) | 4.720(40) | 0.364(fixed) | –0.010 | 0.364 |
| $u_{95}$ | H′(8)···Al(9) | 4.808(25) | 0.242(tied to $u_{5}$) | –0.030 | 0.220 |
| $u_{93}$ | H′(7)···H(3) | 4.827(10) | 0.146(fixed) | –0.080 | 0.146 |
| $u_{96}$ | H(8)···Al(9) | 5.063(18) | 0.192(tied to $u_{5}$) | –0.036 | 0.175 |
| $u_{97}$ | H(8)···H(11) | 5.080(34) | 0.332(fixed) | –0.022 | 0.332 |

This journal is © The Royal Society of Chemistry 2007

| | | | | | |
|---|---|---|---|---|---|
| $u_{98}$ | C(4)···H(12) | 5.141(20) | 0.220(tied to $u_5$) | –0.015 | 0.201 |
| $u_{99}$ | C(8)···H(12) | 5.269(17) | 0.181(tied to $u_5$) | –0.040 | 0.165 |
| $u_{100}$ | H′(8)···H(11) | 5.292(36) | 0.285(fixed) | –0.042 | 0.285 |
| $u_{101}$ | H(8)···H(10) | 5.391(34) | 0.278(fixed) | –0.043 | 0.278 |
| $u_6$ | H(4)···Al(9) | 5.733(6) | 0.123(tied to $u_5$) | –0.015 | 0.112 |
| $u_{102}$ | H′(8)···H(12) | 5.783(32) | 0.275(fixed) | –0.059 | 0.275 |
| $u_{103}$ | H(8)···H(12) | 6.069(23) | 0.229(fixed) | –0.067 | 0.229 |
| $u_{104}$ | H(4)···H(12) | 6.202(21) | 0.221(fixed) | –0.024 | 0.221 |

**Table S7** Least-squares correlation matrix (×100) from the GED refinement of **2**.[a]

| | $p_2$ | $p_4$ | $p_{13}$ | $p_{19}$ | $u_{14}$ | $u_{30}$ | $u_{50}$ | $u_{57}$ | $u_{79}$ | $k_1$ | $k_2$ |
|---|---|---|---|---|---|---|---|---|---|---|---|
| $p_1$ | 76 | | | | | | | | | | |
| $p_3$ | | –91 | | | | | | | | | |
| $p_5$ | | | | | 62 | | | | | | |
| $p_7$ | | | –71 | –63 | | | | | | | |
| $p_{19}$ | | | | | | –68 | | 65 | | | |
| $u_{14}$ | | | | | | | 61 | | | | 60 |
| $u_{50}$ | | | | | | | | | | 53 | 59 |
| $u_{57}$ | | | | | | | | | 50 | | |
| $k_1$ | | | | | | | | | | | 53 |

[a] Only values ≥ 50% are shown; $k_1$ and $k_2$ are scale factors.

**Table S8** Cartesian coordinates (in Å) for the GED-refined structure ($r_{h1}$) of **2**.

| | *x* | *y* | *z* |
|---|---|---|---|
| N(1) | 0.0000 | 0.0000 | 0.0000 |
| C(2) | 1.2155 | –0.7017 | –0.4979 |
| H(2) | 1.0756 | –1.7575 | –0.2484 |
| H′(2) | 2.0572 | –0.3175 | 0.0854 |
| C(3) | 1.3003 | –0.6205 | –2.0322 |
| H(3) | 1.5026 | –1.5794 | –2.5181 |
| H′(3) | 2.1451 | 0.0422 | –2.2406 |
| C(4) | 0.0000 | 0.0000 | –2.5644 |
| H(4) | 0.0000 | 0.0000 | –3.6582 |
| C(5) | –1.1875 | –0.8158 | –2.0322 |
| H(5) | –2.1190 | –0.5116 | –2.5181 |
| H′(5) | –1.0360 | –1.8788 | –2.2406 |
| C(6) | –1.2155 | –0.7017 | –0.4979 |
| H(6) | –2.0598 | –0.0528 | –0.2484 |
| H′(6) | –1.3036 | –1.6228 | 0.0854 |
| C(7) | 0.0000 | 1.4035 | –0.4979 |
| H(7) | 0.9842 | 1.8102 | –0.2484 |
| H′(7) | –0.7536 | 1.9403 | 0.0854 |
| C(8) | –0.1127 | 1.4363 | –2.0322 |
| H(8) | 0.6165 | 2.0909 | –2.5181 |
| H′(8) | –1.1091 | 1.8366 | –2.2406 |
| B(9) | 0.0000 | 0.0000 | 1.6226 |
| H(10) | –1.0764 | 0.4736 | 1.9562 |
| H(11) | 0.9484 | 0.6954 | 1.9562 |
| H(12) | 0.1281 | –1.1690 | 1.9562 |

This journal is © The Royal Society of Chemistry 2007

**Table S9** Least-squares correlation matrix (×100) from the GED refinement of **3**.[a]

| | $p_3$ | $p_4$ | $p_7$ | $p_{19}$ | $u_1$ | $u_{29}$ | $u_{47}$ | $u_{59}$ | $u_{73}$ | $u_{90}$ | $k_2$ |
|---|---|---|---|---|---|---|---|---|---|---|---|
| $p_1$ | | –50 | –80 | 66 | 76 | | 70 | 62 | | | |
| $p_2$ | –90 | | | | | | | | | | |
| $p_4$ | | | | | | 74 | | | | | |
| $p_6$ | | | | | | | | | | | 55 |
| $p_7$ | | | | –62 | –67 | | –95 | –77 | | | |
| $p_{10}$ | | | | | | | | | –56 | | |
| $p_{19}$ | | | | | 58 | | 60 | 70 | | | |
| $u_1$ | | | | | | | 63 | 57 | | | |
| $u_6$ | | | | | | | | | | 52 | |
| $u_{12}$ | | | | | | | | | | | 50 |
| $u_{29}$ | | | | | | | | | | | 52 |
| $u_{47}$ | | | | | | | | 76 | | | |

[a] Only values ≥ 50% are shown; $k_2$ is a scale factor.

**Table S10** Cartesian coordinates (in Å) for the GED-refined structure ($r_{h1}$) of **3**.

| | *x* | *y* | *z* |
|---|---|---|---|
| N(1) | 0.0000 | 0.0000 | 0.0000 |
| C(2) | 1.1908 | –0.6875 | –0.5527 |
| H(2) | 1.0809 | –1.7706 | –0.3307 |
| H′(2) | 2.0932 | –0.3187 | –0.0196 |
| C(3) | 1.3314 | –0.5304 | –2.0744 |
| H(3) | 1.6033 | –1.4793 | –2.5845 |
| H′(3) | 2.1420 | 0.1908 | –2.3140 |
| C(4) | 0.0000 | 0.0000 | –2.6063 |
| H(4) | 0.0000 | 0.0000 | –3.7174 |
| C(5) | –1.1250 | –0.8878 | –2.0744 |
| H(5) | –2.0828 | –0.6488 | –2.5845 |
| H′(5) | –0.9057 | –1.9504 | –2.3140 |
| C(6) | –1.1908 | –0.6875 | –0.5527 |
| H(6) | –2.0739 | –0.0507 | –0.3307 |
| H′(6) | –1.3226 | –1.6534 | –0.0196 |
| C(7) | 0.0000 | 1.3750 | –0.5527 |
| H(7) | 0.9930 | 1.8214 | –0.3307 |
| H′(7) | –0.7706 | 1.9721 | –0.0196 |
| C(8) | –0.2064 | 1.4182 | –2.0744 |
| H(8) | 0.4795 | 2.1281 | –2.5845 |
| H′(8) | –1.2362 | 1.7596 | –2.3140 |
| Al(9) | 0.0000 | 0.0000 | 2.0330 |
| H(10) | –1.4080 | 0.6804 | 2.3168 |
| H(11) | 1.2932 | 0.8792 | 2.3168 |
| H(12) | 0.1148 | –1.5595 | 2.3168 |

This journal is © The Royal Society of Chemistry 2007

**Table S11** Atomic coordinates and equivalent isotropic thermal parameters (Å$^2$) for non-hydrogen atoms in the crystal structure of **2**. See Figure 2 for atom numbering.

| Atom | *x* | *y* | *z* | $U_{equiv}$ |
|---|---|---|---|---|
| B(9) | 0.5879(2) | 0.0879(2) | 0.4121(2) | 0.0420 |
| N(1) | 0.68782(14) | 0.18782(14) | 0.31218(14) | 0.0256 |
| C(2) | 0.67891(19) | 0.34236(19) | 0.35833(18) | 0.0323 |
| C(3) | 0.7903(2) | 0.43414(18) | 0.2794(2) | 0.0360 |
| C(4) | 0.84827(19) | 0.34827(19) | 0.15173(19) | 0.0340 |

**Table S12** Atomic coordinates and isotropic thermal parameters (Å$^2$) for the hydrogen atoms in the crystal structure of **2**. See Figure 2 for atom numbering.

| Atom | *x* | *y* | *z* | $U_{iso}$ |
|---|---|---|---|---|
| H(10) | 0.597(3) | –0.025(3) | 0.362(2) | 0.058(7) |
| H(2) | 0.6969 | 0.3485 | 0.4643 | 0.0388 |
| H′(2) | 0.5805 | 0.3800 | 0.3365 | 0.0388 |
| H(3) | 0.8710 | 0.4586 | 0.3462 | 0.0432 |
| H′(3) | 0.7442 | 0.5248 | 0.2441 | 0.0432 |
| H(4) | 0.9104 | 0.4104 | 0.0896 | 0.0408 |

**Table S13** Anisotropic thermal parameters (Å$^2$) for the crystal structure of **2**. See Figure 2 for atom numbering.

| Atom | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{23}$ | $U_{13}$ | $U_{12}$ |
|---|---|---|---|---|---|---|
| B(9) | 0.0420(10) | 0.0420(10) | 0.0420(10) | 0.0084(10) | 0.0084(10) | –0.0084(10) |
| N(1) | 0.0256(5) | 0.0256(5) | 0.0256(5) | –0.0023(6) | –0.0023(6) | 0.0023(6) |
| C(2) | 0.0366(9) | 0.0288(9) | 0.0315(9) | –0.0049(7) | 0.0032(7) | 0.0039(8) |
| C(3) | 0.0420(11) | 0.0257(9) | 0.0402(10) | –0.0022(7) | –0.0025(9) | –0.0016(8) |
| C(4) | 0.0340(7) | 0.0340(7) | 0.0340(7) | 0.0034(8) | 0.0034(8) | –0.0034(8) |

This journal is © The Royal Society of Chemistry 2007

**Figure S1** Experimental and difference (experimental – theoretical) molecular-scattering intensity curves for **1**.

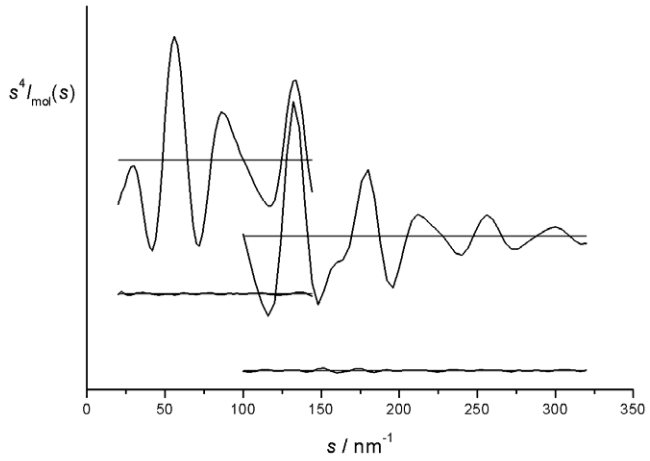
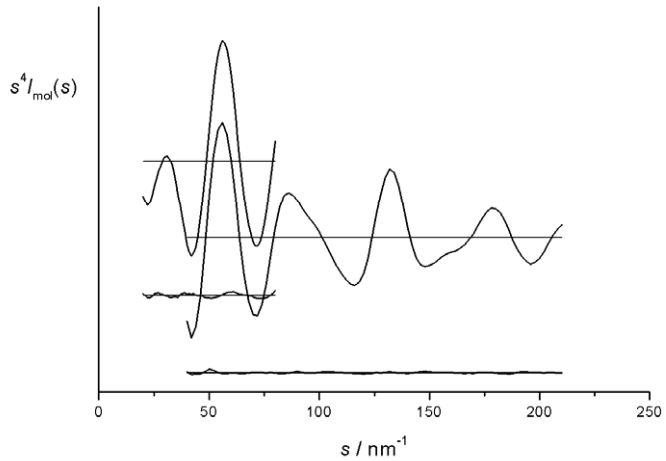



**Figure S2** Experimental and difference (experimental – theoretical) molecular-scattering intensity curves for **2**.



This journal is © The Royal Society of Chemistry 2007

**Figure S3** Experimental and difference (experimental – theoretical) molecular-scattering intensity curves for **3**.