Table 2. *Bond lengths* (Å) *and angles* (°), *with e.s.d.'s in parentheses*

Br(1)—C(2)	1·874 (10)	N(3)—B	1·540 (8)
Br(2)—C(5)	1·876 (7)	N(4)—C(6)	1·347 (9)
Co—Cl	2·207 (3)	C(1)—C(2)	1·352 (15)
Co—N(2)	2·057 (9)	C(2)—C(3)	1·397 (13)
Co—N(4)	2·039 (6)	C(3)—C(7)	1·52 (2)
N(1)—N(2)	1·385 (11)	C(4)—C(5)	1·360 (10)
N(1)—C(1)	1·358 (12)	C(5)—C(6)	1·381 (11)
N(1)—B	1·514 (13)	C(6)—C(9)	1·510 (10)
N(2)—C(3)	1·309 (13)	C(7)—C(8)	1·513 (11)
N(3)—N(4)	1·383 (8)	C(9)—C(10)	1·544 (14)
N(3)—C(4)	1·332 (8)	C(9)—C(11)	1·525 (15)

Cl—Co—N(2)	122·7 (3)	C(1)—C(2)—C(3)	106·8 (9)
Cl—Co—N(4)	121·8 (2)	N(2)—C(3)—C(2)	108·9 (9)
Cl—Co—N(4′)	121·8 (2)	N(2)—C(3)—C(7)	122·8 (9)
N(2)—Co—N(4)	94·1 (2)	C(2)—C(3)—C(7)	128·3 (9)
N(2)—Co—N(4′)	94·1 (2)	N(3)—C(4)—C(5)	108·3 (6)
N(4)—Co—N(4′)	94·9 (3)	Br(2)—C(5)—C(4)	126·5 (6)
N(2)—N(1)—C(1)	107·5 (9)	Br(2)—C(5)—C(6)	126·3 (5)
N(2)—N(1)—B	121·9 (7)	C(4)—C(5)—C(6)	107·2 (6)
C(1)—N(1)—B	130·6 (9)	N(4)—C(6)—C(5)	108·7 (6)
Co—N(2)—N(1)	109·3 (6)	N(4)—C(6)—C(9)	122·7 (7)
Co—N(2)—C(3)	142·3 (7)	C(5)—C(6)—C(9)	128·6 (7)
N(1)—N(2)—C(3)	108·4 (8)	C(3)—C(7)—C(8)	111·6 (6)
N(4)—N(3)—C(4)	109·3 (5)	C(3)—C(7)—C(8′)	111·6 (6)
N(4)—N(3)—B	120·5 (6)	C(8)—C(7)—C(8′)	110·9 (10)
C(4)—N(3)—B	130·2 (6)	C(6)—C(9)—C(10)	109·8 (8)
Co—N(4)—N(3)	110·6 (4)	C(6)—C(9)—C(11)	112·7 (8)
Co—N(4)—C(6)	142·8 (5)	C(10)—C(9)—C(11)	110·9 (8)
N(3)—N(4)—C(6)	106·5 (6)	N(1)—B—N(3)	110·2 (5)
N(1)—C(1)—C(2)	108·4 (9)	N(1)—B—N(3′)	110·2 (5)
Br(1)—C(2)—C(1)	125·9 (7)	N(3)—B—N(3′)	109·2 (7)
Br(1)—C(2)—C(3)	127·3 (8)		

Primed atoms are related to unprimed atoms by the mirror plane: $-x, y, z$.

1·14 and 1·18), $S = 1·06$, Δ/σ (maximum) = 0·07, $\Delta\rho = 0·69$ e Å^{-3}. Positional parameters are in Table 1, bond lengths and angles in Table 2, and a view of the molecule is in Fig. 1.*

Related literature. Guggenberger, Prewitt, Meakin, Trofimenko & Jesson (1973); Trofimenko, Calabrese, Domaille & Thompson (1989); Gorell & Parkin (1990).

We thank the Natural Sciences and Engineering Research Council of Canada for financial support, and the Univ. of British Columbia Computing Centre for assistance.

* Lists of hydrogen positions, anisotropic thermal parameters, torsion angles and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53885 (16 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

GORELL, I. B. & PARKIN, G. (1990). *Inorg. Chem.* **29**, 2452–2456.
GUGGENBERGER, L. J., PREWITT, C. T., MEAKIN, P., TROFIMENKO, S. & JESSON, J. P. (1973). *Inorg. Chem.* **12**, 508–515.
MEULENAER, J. DE & TOMPA, H. (1965). *Acta Cryst.* **19**, 1014–1018.
TROFIMENKO, S., CALABRESE, J. C., DOMAILLE, P. J. & THOMPSON, J. S. (1989). *Inorg. Chem.* **28**, 1091–1101.

Acta Cryst. (1991). C**47**, 1544–1546

Structure of an Octahedral Pyrazolylboratonickel Complex, Ni[HB(3-^{i}Pr,4-Brpz)$_3$][HB(3,5-Me$_2$pz)$_3$]*

BY MICHAEL D. OLSON, STEVEN J. RETTIG, ALAN STORR AND JAMES TROTTER

Department of Chemistry, University of British Columbia, Vancouver, BC, Canada V6T 1Z1

AND S. TROFIMENKO

Du Pont Electronics Department, Experimental Station 336/205, Wilmington, DE 19898, USA

(*Received* 22 *October* 1990; *accepted* 2 *January* 1991)

Abstract. $C_{33}H_{47}B_2Br_3N_{12}Ni$, $M_r = 931·88$, orthorhombic, *Pbca*, $a = 20·65$ (1), $b = 29·158$ (3), $c = 13·306$ (2) Å, $V = 8012$ (4) Å^3, $Z = 8$, $D_x = 1·545$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0·71069$ Å, $\mu = 34·9$ cm^{-1}, $F(000) = 3776$, $T = 294$ K, $R = 0·054$ for 1976 reflections. The molecule has octahedral coordination geometry with the Ni—N distances involving the sterically more demanding ^{i}Pr/Br ligand [2·13–2·16 (1) Å] longer than those involving the Me$_2$ ligand [2·05–2·08 (1) Å].

Experimental. Crystal dimensions 0·25 × 0·30 × 0·45 mm. Rigaku AFC6 diffractometer, monochromatized Mo $K\alpha$ radiation, lattice parameters from 25 reflections with $\theta = 10–17°$. Intensities for θ

* Tris[4-bromo-3-(2-methylethyl)pyrazolyl]hydroboratotris(3,5-dimethylpyrazolyl)hydroboratonickel.

0108-2701/91/071544-03$03.00

Table 1. *Positional (fractional) and equivalent isotropic thermal parameters* (Å^2)

$B_{eq} = (4/3)\sum_i\sum_j \beta_{ij} \mathbf{a}_i . \mathbf{a}_j$.

	x	y	z	B_{eq}
Br(1)	0·21006 (8)	0·31663 (6)	0·2591 (1)	6·3 (1)
Br(2)	0·56036 (8)	0·17315 (6)	0·5862 (1)	5·4 (1)
Br(3)	0·5318 (1)	0·16041 (6)	−0·1118 (1)	7·2 (1)
Ni	0·35623 (8)	0·13183 (5)	0·2584 (1)	2·72 (8)
N(1)	0·3635 (5)	0·2328 (4)	0·251 (1)	2·6 (5)
N(2)	0·3176 (5)	0·2002 (4)	0·263 (1)	3·1 (6)
N(3)	0·4575 (5)	0·1951 (4)	0·3368 (9)	2·4 (6)
N(4)	0·4306 (6)	0·1535 (5)	0·3586 (9)	3·3 (7)
N(5)	0·4479 (6)	0·1927 (4)	0·1497 (8)	2·5 (7)
N(6)	0·4156 (6)	0·1521 (4)	0·1327 (9)	3·1 (7)
N(7)	0·3496 (6)	0·0307 (4)	0·265 (1)	3·7 (7)
N(8)	0·3930 (6)	0·0659 (4)	0·253 (1)	3·3 (6)
N(9)	0·2554 (6)	0·0696 (5)	0·183 (1)	3·6 (7)
N(10)	0·2826 (6)	0·1111 (5)	0·161 (1)	3·4 (7)
N(11)	0·2666 (6)	0·0725 (5)	0·369 (1)	3·4 (7)
N(12)	0·3008 (5)	0·1121 (5)	0·379 (1)	3·6 (7)
C(1)	0·3363 (7)	0·2750 (5)	0·249 (1)	3·4 (8)
C(2)	0·2719 (7)	0·2690 (5)	0·260 (1)	3·2 (8)
C(3)	0·2609 (7)	0·2222 (6)	0·270 (1)	3·3 (9)
C(4)	0·5007 (6)	0·2067 (5)	0·406 (1)	3·0 (8)
C(5)	0·5032 (6)	0·1723 (5)	0·474 (1)	2·2 (7)
C(6)	0·4603 (7)	0·1397 (5)	0·446 (1)	2·8 (8)
C(7)	0·4888 (7)	0·1997 (5)	0·073 (1)	3·3 (8)
C(8)	0·4829 (7)	0·1646 (6)	0·005 (1)	4 (1)
C(9)	0·4357 (7)	0·1360 (5)	0·045 (1)	2·9 (8)
C(10)	0·3820 (8)	−0·0097 (5)	0·261 (1)	3·7 (9)
C(11)	0·4447 (8)	−0·0005 (5)	0·251 (1)	4·4 (9)
C(12)	0·450 (1)	0·0475 (5)	0·244 (1)	4 (1)
C(13)	0·2108 (8)	0·0609 (6)	0·110 (1)	5 (1)
C(14)	0·2084 (8)	0·0968 (7)	0·047 (1)	6 (1)
C(15)	0·2550 (9)	0·1291 (6)	0·080 (1)	5 (1)
C(16)	0·2262 (7)	0·0675 (7)	0·447 (1)	5 (1)
C(17)	0·2339 (8)	0·1035 (7)	0·509 (1)	5 (1)
C(18)	0·2818 (8)	0·1300 (7)	0·466 (1)	5 (1)
C(19)	0·1985 (9)	0·1963 (6)	0·290 (2)	8 (1)
C(20)	0·1666 (8)	0·2118 (7)	0·389 (2)	9 (1)
C(21)	0·148 (1)	0·2034 (8)	0·208 (2)	13 (2)
C(22)	0·4484 (8)	0·0931 (5)	0·493 (1)	4 (1)
C(23)	0·508 (1)	0·0641 (5)	0·498 (1)	10 (1)
C(24)	0·4207 (8)	0·0981 (6)	0·601 (1)	8 (1)
C(25)	0·4096 (7)	0·0917 (5)	−0·004 (1)	4 (1)
C(26)	0·4606 (8)	0·0552 (5)	−0·017 (1)	6 (1)
C(27)	0·3778 (8)	0·1030 (5)	−0·105 (1)	6 (1)
C(28)	0·3466 (9)	−0·0546 (5)	0·271 (1)	7 (1)
C(29)	0·5127 (7)	0·0740 (5)	0·231 (1)	5 (1)
C(30)	0·1723 (7)	0·0175 (6)	0·111 (1)	6 (1)
C(31)	0·2724 (8)	0·1748 (6)	0·036 (1)	6 (1)
C(32)	0·1810 (8)	0·0266 (6)	0·457 (1)	7 (1)
C(33)	0·3085 (8)	0·1751 (7)	0·505 (1)	6 (1)
B(1)	0·4357 (8)	0·2214 (5)	0·242 (1)	2·9 (8)
B(2)	0·278 (1)	0·0437 (7)	0·273 (1)	4 (1)

$\leq 25°$, *hkl*: 0 to 24, 0 to 34, 0 to 15, ω–2θ scan, ω scan width $(1{\cdot}07 + 0{\cdot}35 \tan\theta)°$ at 16° min^{-1} (up to eight rescans, stationary background counts at each end of scan, scan/background time 2:1), three standard reflections showed no significant variation, Lp and absorption corrections (ψ scan, transmission factors, 0·71–1·00). 7760 unique reflections, 1976 with $I \geq 2\sigma(I)$ (only 25·5%; 38% of those in the range $\theta \leq 20°$). Structure solved by Patterson and Fourier methods, refined by full-matrix least-squares procedures, H atoms in calculated positions, not refined. Refinement on F, with $w = 1/\sigma^2(F)$, where $\sigma^2(F^2) = [S^2(C + 4B) + (0{\cdot}04F^2)^2]/\mathrm{Lp}^2$ (S = scan rate, C = scan count, B = normalized background count), scattering factors (with anomalous-dispersion corrections) from *International Tables for X-ray Crystallography* (1974, Vol. IV, pp. 99–102, 149), *TEXSAN/TEXRAY* structure analysis package

Table 2. *Bond lengths* (Å) *and angles* (°), *with e.s.d.'s in parentheses*

Br(1)—C(2)	1·89 (1)	N(11)—C(16)	1·34 (2)
Br(2)—C(5)	1·90 (1)	N(11)—B(2)	1·54 (2)
Br(3)—C(8)	1·86 (1)	N(12)—C(18)	1·34 (2)
Ni—N(2)	2·15 (1)	C(1)—C(2)	1·35 (2)
Ni—N(4)	2·13 (1)	C(2)—C(3)	1·39 (2)
Ni—N(6)	2·16 (1)	C(3)—C(19)	1·52 (2)
Ni—N(8)	2·07 (1)	C(4)—C(5)	1·35 (2)
Ni—N(10)	2·08 (1)	C(5)—C(6)	1·35 (2)
Ni—N(12)	2·05 (1)	C(6)—C(22)	1·51 (2)
N(1)—N(2)	1·35 (1)	C(7)—C(8)	1·37 (2)
N(1)—C(1)	1·35 (1)	C(8)—C(9)	1·39 (2)
N(1)—B(1)	1·53 (2)	C(9)—C(25)	1·55 (2)
N(2)—C(3)	1·34 (2)	C(10)—C(11)	1·33 (2)
N(3)—N(4)	1·37 (1)	C(10)—C(28)	1·51 (2)
N(3)—C(4)	1·32 (1)	C(11)—C(12)	1·41 (2)
N(3)—B(1)	1·54 (2)	C(12)—C(29)	1·52 (2)
N(4)—C(6)	1·38 (2)	C(13)—C(14)	1·34 (2)
N(5)—N(6)	1·38 (1)	C(13)—C(30)	1·49 (2)
N(5)—C(7)	1·34 (1)	C(14)—C(15)	1·42 (2)
N(5)—B(1)	1·51 (2)	C(15)—C(31)	1·50 (2)
N(6)—C(9)	1·32 (2)	C(16)—C(17)	1·35 (2)
N(7)—N(8)	1·37 (1)	C(16)—C(32)	1·52 (2)
N(7)—C(10)	1·35 (2)	C(17)—C(18)	1·38 (2)
N(7)—B(2)	1·53 (2)	C(18)—C(33)	1·51 (2)
N(8)—C(12)	1·30 (2)	C(19)—C(20)	1·54 (2)
N(9)—N(10)	1·37 (2)	C(19)—C(21)	1·52 (2)
N(9)—C(13)	1·36 (2)	C(22)—C(23)	1·50 (2)
N(9)—B(2)	1·49 (2)	C(22)—C(24)	1·56 (2)
N(10)—C(15)	1·33 (2)	C(25)—C(26)	1·51 (2)
N(11)—N(12)	1·36 (1)	C(25)—C(27)	1·53 (2)

N(2)—Ni—N(4)	88·4 (5)	C(1)—C(2)—C(3)	107 (1)
N(2)—Ni—N(6)	88·9 (5)	N(2)—C(3)—C(2)	109 (1)
N(2)—Ni—N(8)	179·7 (7)	N(2)—C(3)—C(19)	121 (1)
N(2)—Ni—N(10)	91·0 (5)	C(2)—C(3)—C(19)	130 (1)
N(2)—Ni—N(12)	91·6 (5)	N(3)—C(4)—C(5)	108 (1)
N(4)—Ni—N(6)	89·7 (4)	Br(2)—C(5)—C(4)	123 (1)
N(4)—Ni—N(8)	91·8 (5)	Br(2)—C(5)—C(6)	129 (1)
N(4)—Ni—N(10)	179·3 (5)	C(4)—C(5)—C(6)	108 (1)
N(4)—Ni—N(12)	89·8 (5)	N(4)—C(6)—C(5)	108 (1)
N(6)—Ni—N(8)	91·1 (5)	N(4)—C(6)—C(22)	122 (1)
N(6)—Ni—N(10)	90·8 (5)	C(5)—C(6)—C(22)	129 (1)
N(6)—Ni—N(12)	179·2 (4)	N(5)—C(7)—C(8)	109 (1)
N(8)—Ni—N(10)	88·7 (5)	Br(3)—C(8)—C(7)	124 (1)
N(8)—Ni—N(12)	88·3 (5)	Br(3)—C(8)—C(9)	131 (1)
N(10)—Ni—N(12)	89·7 (5)	C(7)—C(8)—C(9)	105 (1)
N(2)—N(1)—C(1)	111 (1)	N(6)—C(9)—C(8)	110 (1)
N(2)—N(1)—B(1)	123 (1)	N(6)—C(9)—C(25)	124 (2)
C(1)—N(1)—B(1)	127 (1)	C(8)—C(9)—C(25)	126 (1)
Ni—N(2)—N(1)	112·8 (8)	N(7)—C(10)—C(11)	108 (1)
Ni—N(2)—C(3)	141 (1)	N(7)—C(10)—C(28)	121 (1)
N(1)—N(2)—C(3)	107 (1)	C(11)—C(10)—C(28)	131 (1)
N(4)—N(3)—C(4)	111 (1)	C(10)—C(11)—C(12)	106 (1)
N(4)—N(3)—B(1)	120 (1)	N(8)—C(12)—C(11)	109 (1)
C(4)—N(3)—B(1)	129 (1)	N(8)—C(12)—C(29)	125 (1)
Ni—N(4)—N(3)	115 (1)	C(11)—C(12)—C(29)	126 (2)
Ni—N(4)—C(6)	140 (1)	N(9)—C(13)—C(14)	109 (2)
N(3)—N(4)—C(6)	105 (1)	N(9)—C(13)—C(30)	121 (2)
N(6)—N(5)—C(7)	108 (1)	C(14)—C(13)—C(30)	130 (2)
N(6)—N(5)—B(1)	122 (1)	C(13)—C(14)—C(15)	107 (1)
C(7)—N(5)—B(1)	130 (1)	N(10)—C(15)—C(14)	106 (2)
Ni—N(6)—N(5)	113 (1)	N(10)—C(15)—C(31)	125 (2)
Ni—N(6)—C(9)	140 (1)	C(14)—C(15)—C(31)	129 (2)
N(5)—N(6)—C(9)	107 (1)	N(11)—C(16)—C(17)	109 (2)
N(8)—N(7)—C(10)	109 (1)	N(11)—C(16)—C(32)	123 (2)
N(8)—N(7)—B(2)	117 (1)	C(17)—C(16)—C(32)	129 (2)
C(10)—N(7)—B(2)	134 (1)	C(16)—C(17)—C(18)	105 (1)
Ni—N(8)—N(7)	117 (1)	N(12)—C(18)—C(17)	111 (2)
Ni—N(8)—C(12)	136 (1)	N(12)—C(18)—C(33)	122 (2)
N(7)—N(8)—C(12)	107 (1)	C(17)—C(18)—C(33)	128 (2)
N(10)—N(9)—C(13)	107 (1)	C(3)—C(19)—C(20)	112 (2)
N(10)—N(9)—B(2)	119 (1)	C(3)—C(19)—C(21)	113 (2)
C(13)—N(9)—B(2)	134 (2)	C(20)—C(19)—C(21)	107 (1)
Ni—N(10)—N(9)	115 (1)	C(6)—C(22)—C(23)	113 (1)
Ni—N(10)—C(15)	135 (1)	C(6)—C(22)—C(24)	111 (1)
N(9)—N(10)—C(15)	110 (1)	C(23)—C(22)—C(24)	108 (1)
N(12)—N(11)—C(16)	110 (1)	C(9)—C(25)—C(26)	113 (1)
N(12)—N(11)—B(2)	117 (1)	C(9)—C(25)—C(27)	110 (1)
C(16)—N(11)—B(2)	132 (2)	C(26)—C(25)—C(27)	111 (1)
Ni—N(12)—N(11)	117 (1)	N(1)—B(1)—N(3)	109 (1)
Ni—N(12)—C(18)	137 (1)	N(1)—B(1)—N(5)	110 (1)
N(11)—N(12)—C(18)	137 (1)	N(3)—B(1)—N(5)	110 (1)
N(1)—C(1)—C(2)	107 (1)	N(7)—B(2)—N(9)	111 (1)
Br(1)—C(2)—C(1)	125 (1)	N(7)—B(2)—N(11)	110 (1)
Br(1)—C(2)—C(3)	128 (1)	N(9)—B(2)—N(11)	110 (1)

(Molecular Structure Corporation, 1985). Final $R = 0.054$, $wR = 0.048$ for 460 parameters, 1976 reflections with $I \geq 2\sigma(I)$, $S = 1.37$, Δ/σ (maximum) = 0.09, $\Delta\rho = 0.50$ e Å^{-3} (near Br). Positional parameters are in Table 1, bond lengths and angles in Table 2, and a view of the molecule is in Fig. 1.*

Related literature. Calabrese, Domaille, Thompson & Trofimenko (1990); Olson, Rettig, Storr, Trotter & Trofimenko (1991).

We thank the Natural Sciences and Engineering Research Council of Canada for financial support.

* Lists of hydrogen positions, anisotropic thermal parameters, torsion angles, intermolecular distances, mean planes and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53886 (65 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

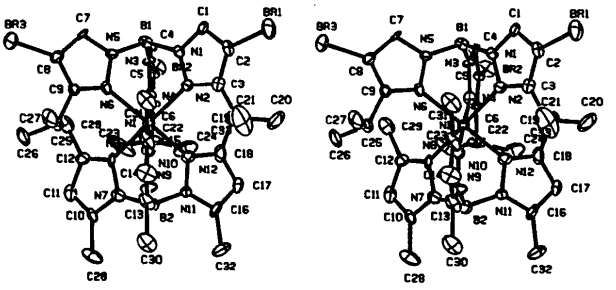

Fig. 1. Stereoview of the molecule; 50% ellipsoids for the non-H atoms.

References

CALABRESE, J. C., DOMAILLE, P. J., THOMPSON, J. S. & TROFIMENKO, S. (1990). *Inorg. Chem.* **29**, 4429–4437.

Molecular Structure Corporation (1985). *TEXSAN. TEXRAY Structure Analysis Package*. MSC, 3200A Research Forest Drive, The Woodlands, TX77381, USA.

OLSON, M. D., RETTIG, S. J., STORR, A., TROTTER, J. & TROFIMENKO, S. (1991). *Acta Cryst.* C**47**, 1543–1544.

Acta Cryst. (1991). C**47**, 1546–1548

1:2 Complex of 1,4,7,10,13,16-Hexaoxacyclooctadecane (18-Crown-6) with *N*-*m*-Bromophenylurea

BY V. NASTOPOULOS*

Department of Chemistry, University of Patras, Gr-261 10 *Patras, Greece*

AND G. GERMAIN AND J. WEILER

Unité de Chimie Physique Moléculaire et de Cristallographie, Université de Louvain, Place L. Pasteur 1, *B*-1348 *Louvain-la-Neuve, Belgium*

(*Received* 11 *April* 1990; *accepted* 21 *December* 1990)

Abstract. $C_7H_7BrN_2O.\frac{1}{2}C_{12}H_{24}O_6$, $M_r = 347.2$, monoclinic, $P2_1/c$, $a = 8.910$ (3), $b = 24.924$ (8), $c = 8.391$ (4) Å, $\beta = 122.00$ (1)°, $V = 1580.3$ (10) Å^3, $Z = 4$, $D_x = 1.46$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 2.77$ mm^{-1}, $F(000) = 712$, $T = 293$ K, final $R = 0.067$ for 1229 observed reflections. The host 18-crown-6 molecule lies about a crystallographic centre of symmetry. This centre relates the two guest bromophenylurea molecules which lie on either side of the host molecule almost perpendicular to the macrocyclic ring. Each guest molecule is hydrogen bonded to two adjacent O atoms of the crown ether by means of one H atom from each amino group. The complex adopts the biangular conformation of the macrocycle and is evidence for the formation of a discrete 1:2 crown ether–urea molecular complex.

* Author to whom correspondence should be addressed.

Experimental. Crystals were grown from a methanol/ethyl acetate solution at 253 K, melting point 386–387 K. The possibility that the structure can be described in terms of a *C*-centred orthorhombic cell was excluded by transforming the intensity data to the orthorhombic indexing and observing that *mmm* intensity symmetry was not obeyed. Precession photographs were used to verify the space group and to obtain approximate cell constants. Crystal size 0.25 × 0.30 × 0.40 mm, Siemens Kristaloflex 805 diffractometer, graphite-monochromated Mo $K\alpha$ radiation, 15 reflections with $5 \leq \theta \leq 25°$ used for determining lattice parameters, data collected using ω–2θ scans up to $2\theta = 55°$, one standard reflection monitored after every 50 measurements showed no significant deviation from its mean intensity, 3609 unique reflections measured of which 1229 with $I > 2.5\sigma(I)$ were used in refinement, range of *hkl*: $-11 \leq$

0108-2701/91/071546-03$03.00