

(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, $\Delta\rho = 0.50 \text{ e \AA}^{-3}$ (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).

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* 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.

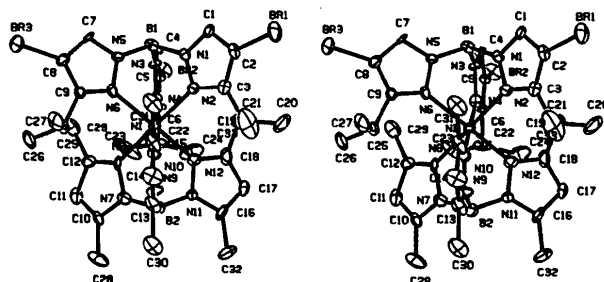


Fig. 1. Stereoview of the molecule; 50% ellipsoids for the non-H atoms.

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

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Abstract. $\text{C}_7\text{H}_7\text{BrN}_2\text{O}_2 \cdot \frac{1}{2}\text{C}_{12}\text{H}_{24}\text{O}_6$, $M_r = 347.2$, 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) Å³, $Z = 4$, $D_x = 1.46 \text{ Mg m}^{-3}$, $\lambda(\text{Mo K}\alpha) = 0.71069$ Å, $\mu = 2.77 \text{ mm}^{-1}$, $F(000) = 712$, $T = 293 \text{ K}$, 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.

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 Kristalloflex 805 diffractometer, graphite-monochromated $\text{Mo K}\alpha$ radiation, 15 reflections with $5 \leq \theta \leq 25^\circ$ used for determining lattice parameters, data collected using ω - 2θ scans up to $2\theta = 55^\circ$, 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*: $-11 \leq$

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Table 1. Fractional atomic coordinates ($\times 10^4$) and equivalent isotropic temperature factors (\AA^2) with e.s.d.'s in parentheses
$$B_{\text{eq}} = (4/3)\sum_i \sum_j \beta_{ij} a_i \cdot a_j$$

	x	y	z	B_{eq}
O(1)	-6150 (8)	-370 (3)	6355 (9)	5.59 (29)
C(2)	-7683 (13)	-111 (5)	4996 (14)	6.55 (52)
C(3)	-7250 (13)	465 (5)	4816 (13)	6.46 (47)
O(4)	-6651 (7)	777 (3)	6486 (9)	5.32 (25)
C(5)	-7960 (11)	861 (4)	6921 (14)	5.47 (46)
C(6)	-7211 (14)	1208 (4)	8606 (16)	6.26 (54)
O(7)	-5775 (9)	921 (3)	10143 (10)	5.75 (32)
C(8)	-5119 (21)	1170 (5)	11895 (23)	7.42 (83)
C(9)	-3458 (16)	902 (5)	13330 (18)	6.49 (66)
C(10)	-2646 (11)	1566 (3)	8079 (12)	3.97 (39)
C(11)	-1010 (11)	1822 (3)	9032 (12)	4.18 (40)
C(12)	-905 (13)	2332 (4)	8416 (15)	5.28 (47)
C(13)	-2350 (17)	2583 (4)	6925 (17)	6.35 (60)
C(14)	-3962 (15)	2313 (5)	5991 (15)	6.46 (55)
C(15)	-4100 (12)	1814 (4)	6582 (14)	5.07 (41)
Br(16)	1316 (2)	2685 (1)	9728 (2)	8.49 (7)
N(17)	-2904 (10)	1053 (3)	8616 (11)	4.47 (35)
C(18)	-1681 (12)	651 (4)	9386 (12)	4.27 (37)
O(19)	-134 (7)	709 (2)	9838 (8)	5.17 (28)
N(20)	-2297 (11)	181 (3)	9609 (13)	5.52 (39)
H(17)	-4023 (94)	1002 (28)	8188 (92)	7.66 (61)
H(20A)	-3395 (137)	128 (41)	9119 (140)	7.66 (61)
H(20B)	-1574 (137)	-47 (44)	9993 (146)	7.66 (61)

$h \leq 9$, $0 \leq k \leq 32$, $0 \leq l \leq 10$. Structure solved by direct methods (*MULTAN*11/84; Main, Germain & Woolfson, 1984) and refined by the full-matrix least-squares technique (*SHELX*76; Sheldrick, 1976). Final $R = 0.067$, $wR = 0.061$, $\sum w(\Delta F)^2$ minimized, $w = 6.2991/[\sigma^2(F) + 0.00074F^2]$, 191 parameters refined, $S = 1.62$, final accuracy of the refinement is limited by the poor quality of the crystals, $(\Delta/\sigma)_{\text{max}}$ (for non-H atoms) = 0.62, maximum and minimum electron densities in final difference synthesis 0.44 and -0.47 e \AA^{-3} . All non-H atoms refined with anisotropic thermal parameters; H included using riding model (C—H 1.08 Å) with a fixed overall isotropic temperature factor $U = 0.097 \text{ \AA}^2$, except those bonded to N atoms, which were found from ΔF synthesis and refined isotropically. Atomic scattering factors as incorporated in *SHELX*76; for torsion angles and other geometrical calculations the programs *PARST* (Nardelli, 1983) and *XANADU* (Roberts & Sheldrick, 1975) were used. Atomic coordinates and equivalent isotropic temperature factors are listed in Table 1; selected bond distances, angles and torsion angles are given in Table 2.* Atom numbering follows that shown in Fig. 1.

Related literature. The structure of the title compound was studied as part of an investigation into

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and bond lengths, angles and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53862 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. Bond distances, bond angles, selected torsion angles and hydrogen-bonding geometry ($^\circ$, Å)

C(2)—O(1)	1.389 (11)	C(3)—C(2)	1.516 (14)
O(4)—C(3)	1.435 (11)	C(5)—O(4)	1.411 (9)
C(6)—C(5)	1.480 (12)	O(7)—C(6)	1.436 (10)
C(8)—O(7)	1.406 (14)	C(9)—C(8)	1.482 (17)
O(1*)—C(9)	1.433 (11)	C(11)—C(10)	1.392 (10)
C(15)—C(10)	1.383 (11)	N(17)—C(10)	1.414 (10)
C(12)—C(11)	1.393 (12)	C(13)—C(12)	1.382 (14)
Br(16)—C(12)	1.896 (9)	C(14)—C(13)	1.393 (14)
C(15)—C(14)	1.368 (12)	C(18)—N(17)	1.365 (11)
O(19)—C(18)	1.229 (9)	N(20)—C(18)	1.348 (11)
N(17)—H(17)	0.872 (67)	N(20)—H(20A)	0.847 (101)
N(20)—H(20B)	0.789 (104)		
C(2)—O(1)—C(9*)	110.5 (7)	O(1)—C(2)—C(3)	109.3 (8)
C(2)—C(3)—O(4)	113.5 (5)	C(3)—O(4)—C(5)	113.7 (7)
O(4)—C(5)—C(6)	108.6 (7)	C(5)—C(6)—O(7)	107.7 (8)
C(6)—O(7)—C(8)	113.5 (9)	O(7)—C(8)—C(9)	109.5 (9)
C(8)—C(9)—O(1*)	109.3 (7)	C(11)—C(10)—C(15)	120.6 (8)
C(11)—C(10)—N(17)	122.0 (8)	C(15)—C(10)—N(17)	117.5 (8)
C(10)—C(11)—C(12)	117.4 (8)	C(11)—C(12)—C(13)	122.5 (9)
C(11)—C(12)—Br(16)	117.6 (8)	C(13)—C(12)—Br(16)	120.0 (8)
C(12)—C(13)—C(14)	118.6 (9)	C(13)—C(14)—C(15)	120.0 (10)
C(10)—C(15)—C(14)	121.0 (9)	C(10)—N(17)—C(18)	126.2 (7)
N(17)—C(18)—O(19)	122.9 (9)	N(17)—C(18)—N(20)	115.0 (8)
O(19)—C(18)—N(20)	122.2 (9)	C(10)—N(17)—H(17)	110 (5)
H(17)—N(17)—C(18)	123 (5)	C(18)—N(20)—H(20A)	121 (7)
C(18)—N(20)—H(20B)	112 (8)	H(20A)—N(20)—H(20B)	125 (11)
C(8*)—C(9*)—O(1)—C(2)	179.2 (11)	O(4)—C(5)—C(6)—O(7)	-63.4 (11)
C(9*)—O(1)—C(2)—C(3)	175.2 (10)	C(5)—C(6)—O(7)—C(8)	-170.7 (10)
O(1)—C(2)—C(3)—O(4)	-64.1 (12)	C(6)—O(7)—C(8)—C(9)	-170.0 (11)
C(2)—C(3)—O(4)—C(5)	-66.6 (12)	O(7)—C(8)—C(9)—O(1*)	-68.0 (15)
C(3)—O(4)—C(5)—C(6)	-177.3 (9)		

	$d(\text{N}\cdots\text{O})$	$d(\text{H}\cdots\text{O})$	$\angle \text{N—H}\cdots\text{O}$
N(17)—H(17) \cdots O(4)	2.91 (1)	2.07 (7)	162 (7)
N(20)—H(20A) \cdots O(1)	3.34 (1)	2.63 (9)	143 (11)

* Atoms belonging to the molecular complex at symmetry position $-x, -y, -z$ with regard to the respective atoms.

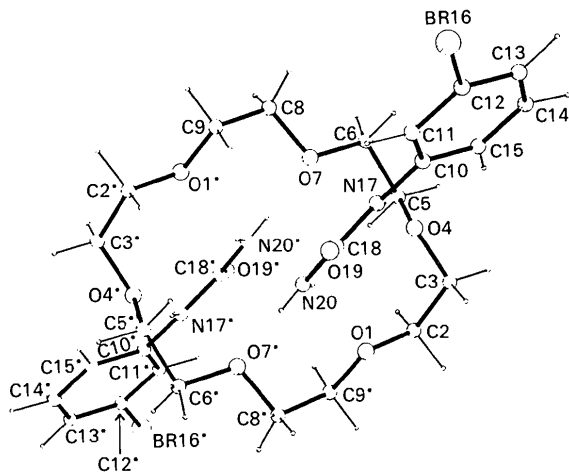


Fig. 1. Perspective view of the complex with the atom-numbering scheme (*PLUTO*; Motherwell & Clegg, 1978).

the crystalline complexes of a macrocyclic polyether (18-crown-6) with phenylurea derivatives (Weiler, 1985). The preparation and the structure of the complex is similar to that of the chlorine-containing analogue (Nastopoulos & Weiler, 1988).

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Structure of 2-*N,N*-Dimethylamino-5-hexadecyl-1,3-dithiolium-4-thiolate

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Abstract. C₂₁H₃₉NS₃, $M_r = 401.7$, triclinic, $P\bar{1}$, $a = 5.7017$ (6), $b = 9.404$ (1), $c = 22.104$ (4) Å, $\alpha = 91.45$ (1), $\beta = 93.17$ (1), $\gamma = 104.17$ (1)°, $V = 1146.5$ (3) Å³, $Z = 2$, $D_x = 1.1636$ (3) g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 3.15$ cm⁻¹, $F(000) = 220$, $T = 293$ K, final $R = 0.065$ for 1183 unique observed reflections. The molecule is almost planar and the bond angles of the long aliphatic chain indicate no strain. The packing is dominated by electrostatic interactions between the heterocyclic part of the molecules, no close contacts or hydrogen bonds are observed.

Experimental. The title compound was prepared according to Jørgensen, Lerstrup & Bechgaard (1991). 2-Bromooctadecanoic acid was reacted with sodium *N,N*-dimethyldithiocarbamate to obtain 1-carboxyheptadecyl-*N,N*-dimethyldithiocarbamate ester. The ester was then treated with acetic acid anhydride, triethylamine and carbon disulfide to obtain the title compound. X-ray intensity data were collected on an Enraf–Nonius CAD-4 diffractometer using ω - 2θ scan, and corrected for Lorentz, polarization and absorption effects. The structure was solved using direct methods for locating the non-H atoms. H atoms of the CH₂ groups were fixed at geometrically calculated positions and the CH₃ groups were considered as rigid bodies, all C—H distances were fixed at 1.08 Å. Least-squares

refinement based on F , minimizing the sum of $w(\Delta F)^2$, was performed with anisotropic S, C, and N atoms and isotropic H atoms with the constraints given above. The H atoms were refined with one common isotropic temperature factor for all CH₂ H atoms and the H atoms of each of the three CH₃ groups had one common isotropic temperature factor. Scattering factors and anomalous-dispersion correction parameters were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). Further experimental details are summarized in Table 1. Atomic coordinates and equivalent isotropic temperature factors of the non-H atoms are listed in Table 2, bond distances and angles are given in Table 3. The molecular geometry together with the atomic labelling used in the present work is shown in Fig. 1, and the packing within the unit cell is depicted in Fig. 2.* Programs used were *SHELX76* (Sheldrick, 1976) for structure solution and refinement, *DISTAN* (Lundgren, 1985) for calculation of bond lengths and angles, *ORTEPII* (Johnson, 1976) and *PLUTO* (Motherwell & Clegg, 1978) for illustrations and the *CONVERT* utility programs (Hjorth, 1990) to facilitate interfacing of the different programs.

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53761 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.