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Crystal Structure of the 1 : 2 Molecular Complex between the *cis-anti-cis* Isomer of Dicyclohexano-18-crown-6 and 3-Chloro-6-methylbenzenesulphamide

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Abstract. Reaction of the *cis-anti-cis* isomer of dicyclohexano-18-crown-6 with a mixture of 2-chloro-5-methylbenzenesulphamide and 3-chloro-6-methylbenzenesulphamide gives a crystalline product which has been characterized using X-ray crystallography. The complex (**I**) contains both isomers in a 83.0 (0.6) : 17.0 (0.6) ratio in favour of the 3-chloro-6-methyl-isomer. The joint crystallization of the crown ether with the mixture of the guest isomers might serve as a route for the partial extraction of one of them. The complex with host : guest ratio 1 : 2 crystallizes in a triclinic space group $P\bar{1}$, $a = 13.087(4)$, $b = 9.217(2)$, $c = 8.120(2)$ Å, $\alpha = 92.05(3)$, $\beta = 100.52(3)$, $\gamma = 80.64(2)°$, with $Z = 1$. The structure was refined to $R = 0.041$. Each of two equivalent guest molecules interacts with the macrocyclic ring by two NH···O hydrogen bonds, with N···O distances of 2.912(5) and 3.040(5) Å.

Supplementary Data relevant to this paper have been deposited with the British Library as Supplementary Publication No. SUP 82180 (30 pp.).

Key words. Host–guest molecular recognition, H-bonds, crown ether molecular complexes.

1. Introduction

One of the most interesting and complicated problems in host–guest chemistry is that of selectivity and molecular recognition in the process of complex formation. For metal complexes, cation selectivity depends on the correspondence between the cation diameter and the macrocyclic cavity size [1, 2]. In the case of chiral guests it is determined by specific features of host functional groups [3,

4]. Host selectivity is frequently governed by bulky substituents conjugated with the macrocyclic framework. These create additional steric hindrance and allow selective guest recognition during complexation. Diastereomers of dicyclohexano-18-crown-6 (DCH-6) are suitable examples of such hosts [5].

Previously we have shown that 2,4-dinitroaniline [6], 4-aminobenzenesulphamide [7] and 4-acetylaminobenzenesulphamide [8] form stable molecular complexes with the *cis-anti-cis* isomer of DCH-6 and do not react with the *cis-syn-cis* isomer at all. This phenomenon could be used for separating these isomers. On the other hand it would be interesting to use guest molecules with the same composition but with distinctive geometries in order to identify hosts which could separate them selectively. As such a model we have analysed the system consisting of the *cis-anti-cis*-DCH-6 isomer with a mixture of 2-chloro-5-methylbenzenesulphamide and 3-chloro-6-methylbenzenesulphamide.

The composition and structure of the title compound have been determined by X-ray diffraction.

2. Experimental

It appears that the *cis-anti-cis* isomer of DCH-6 is able to enrich the mixture of products obtained after nucleophilic substitution on the aromatic ring. The reaction sequence used was:

$$p\text{—}CH_3C_6H_4Cl \xrightarrow{ClSO_2OH} CH_3C_6H_3(SO_2Cl)Cl \xrightarrow{NH_3} CH_3C_6H_3(SO_2NH_2)Cl$$

The final product is a mixture of isomers with the sulphamide group *ortho* to the CH_3 (60%) or Cl (40%) substituents. They are well separated on 'Silufol' silica gel plates. Their development by toluidine is quite different and unequivocal. As shown below, 3-chloro-6-methylbenzenesulphamide is the main component in the complex formed with *cis-anti-cis*-DCH-6.

2.1. Synthesis of Complex I

The *cis-anti-cis* isomer of DCH-6 (372 mg, 1 mmol) and a mixture of 3-chloro-6-methyl- and 2-chloro-5-methylbenzenesulphamide (205.5 mg, 1 mmol, 60 and 40%, respectively, were dissolved simultaneously in 25 mL of acetone at 56 °C. After slow evaporation, crystals of complex **I** were obtained, suitable for X-ray crystallography. The yield of complex **I** was 230 mg (93%) with respect to the 3-chloro-6-methylbenzenesulphamide or 56% with respect to the general mass of both guest isomers. The chromatographic investigation was carried out using 'Silufol' plates using a methanol : chloroform mixture (1 : 8) as eluent with further developing by gaseous chlorine followed by a 1% aqueous solution of toluidine acetate. Under these conditions complex **I** is destroyed. Three spots were fixed on

the 'Silufol' plate: 3-chloro-6-methylbenzenesulphamide, DCH-6, and 2-chloro-5-methylbenzenesulphamide with R_f values of 0.41, 0.50 and 0.46 respectively. The oily residue after the spontaneous evaporation of the filtrate (m.p. 45–110 °C) consisted of a mixture of 3-chloro-6-methylbenzenesulphamide, DCH-6, and 2-chloro-5-methylbenzenesulphamide. Repeated recrystallizations of this mixture did not lead to the DCH-6 complex with 2-chloro-5-methylbenzenesulphamide or separation of the mixture. Thus, the crystalline product was considered to be the complex of DCH-6 with 3-chloro-6-methylbenzenesulphamide. Colorless, transparent crystals; soluble in methanol, ethanol, acetone; m.p. 165–166 °C. *Anal. found*, % Cl 9.30, N 3.65, S 8.10; *Calcd.* for $C_{34}H_{52}Cl_2N_2O_{10}S_2$, % Cl 9.05, N 3.57, S 8.18.

2.2. X-Ray Data Collection, Structure Determination and Refinement

The main crystallographic data are given in Table I. X-ray data were collected on a Philips PW 1100 diffractometer. Lattice parameters were obtained using 36 reflections in θ range 5.41–18.66° with a routine which repeatedly improves the angular values to reach the maximum of the peaks until the angles change by not more than 0.001°. The intensity data were collected at room temperature by ω–2θ scan technique in the range $3 < \theta < 28°$. Two standard reflections, monitored every 60 minutes, showed insignificant variations in intensity (less than 1%). Intensity data were corrected for Lorentz and polarization factors but no absorption correction was applied. The structure was solved by direct methods using the SIR92 package [9]. All calculations were performed using an IBM PS2/80 personal computer. Scattering factors were those of SHELX. The structure was refined by the full-matrix technique; isotropic refinement was carried out using SHELX76 [10] down to $R = 0.12$, while anisotropic refinement was carried out using SHELX93 [11]. At all the preceding stages (until the agreement factor $R = 0.050$) the structure was refined in the supposition that only one guest isomer, 3-chloro-6-methylbenzenesulphamide, was present. However, there were some disturbing features related to the methyl and chloro groups: the C—CH_3 bond length appeared to be abnormally long, 1.635 Å, while U_{eq} for the Cl atom was rather large: 0.087. Because the two guest isomers differ from each other merely by the interchange of a methyl group and a chloro group, which have similar van der Waals radii, there could be a small amount of 2-chloro-5-methylbenzenesulphamide in the crystal. Thus at the last stage of the refinement Cl and C (methyl) atoms were put at the fixed standard distances (C(6G)—Cl(1X) = 1.729 Å and C(3G)—C(7X) = 1.503 Å) from the proper atoms of the aromatic moiety along the bonds C(6G)—C(7G) and C(3G)—Cl(1G) and for them only the occupancy position parameters and U_{eq} (Cl(1X)) were refined. Using such an approach the ratio of the guest isomers, 3-chloro-6-methylbenzenesulphamide and 2-chloro-5-methylbenzenesulphamide, was 83.0 (0.6) : 17.0 (0.6). H-atoms were localized from a

TABLE I. Crystal data and summary of intensity data collection and structure refinement for complex (**I**).

Formula	$C_{20}H_{36}O_6 \cdot 2(C_7H_8ClNO_2S)$
M.W.	783.8
Crystal system	Triclinic
Space group	$P\bar{1}$
a (Å)	13.087 (4)
b (Å)	9.217 (2)
c (Å)	8.120 (2)
α (deg)	92.05 (3)
β (deg)	100.53 (3)
γ (deg)	80.64 (2)
V (Å^3)	950.2 (3)
Z	1
D_x (Mg/m^3)	1.370
Crystal form	Prism
Crystal size (mm)	0.3 ×0.5 × 0.9
Diffractometer	Philips PW 1100
Radiation	MoK_α (λ = 0.71069 Å)
μ (cm^{-1})	3.4
T (K)	293
Number of reflections	
Measured	4179
Observed	2029
Condition for obs.	$(I) > 2\sigma(I)$
No. of variables	326
GOF	1.248
$(\Delta/\sigma)_{max}$	0.116
$\Delta\rho_{min}$ (e/Å^3)	0.244
$\Delta\rho_{max}$ (e/Å^3)	−0.231
$R(hkl)$ (w = 1)	0.041

difference Fourier map, excluding those of the methyl group, which were put in calculated positions, all H-atoms were refined isotropically. The final difference Fourier map does not show any residual peaks having significant intensity. The final agreement factor was R = 0.041 for 2029 independent reflections with unit weights. The further description and all the calculated parameters refer to the complex of DCH-6 with 3-chloro-6-methylbenzenesulphamide. Final positional parameters are listed in Table II, bond distances, valence and torsion angles in Table III. The figures were produced using the CRYSRULER [12] and ORTEP [13] packages. The atomic numbering scheme in the molecules forming **I** is given in Figure 1.

TABLE II. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters (Å^2 $\times$ 10^3) for 1. U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

Atom	x/a	y/b	z/c	U(eq)
Cl (1G)	5542 (2)	8499 (2)	5958 (3)	76 (1)
S (1G)	8148 (1)	3312 (1)	5991 (1)	30 (1)
O (1G)	8698 (2)	2464 (3)	7420 (4)	45 (1)
O (2G)	7799 (3)	2557 (3)	4485 (4)	47 (1)
N (1G)	8878 (3)	4403 (4)	5562 (6)	35 (1)
C (1G)	6993 (3)	4354 (5)	6569 (5)	29 (1)
C (2G)	6743 (4)	5827 (5)	6155 (6)	34 (1)
C (3G)	5825 (4)	6652 (5)	6489 (6)	43 (1)
C (4G)	5141 (4)	6014 (7)	7200 (7)	57 (2)
C (5G)	5401 (4)	4545 (7)	7631 (7)	55 (2)
C (6G)	6321 (4)	3674 (5)	7322 (6)	40 (1)
C (7G)	6538 (6)	2047 (9)	7825 (11)	74 (5)
O (1)	8746 (2)	5406 (3)	2159 (3)	35 (1)
C (2)	8793 (4)	4318 (5)	870 (6)	40 (1)
C (3)	9419 (4)	2910 (5)	1580 (6)	37 (1)
O (4)	10490 (2)	3082 (3)	2158 (4)	37 (1)
C (5)	11148 (4)	1734 (5)	2733 (6)	43 (1)
C (6)	11072 (5)	1302 (5)	4455 (6)	44 (1)
O (7)	11562 (2)	2258 (3)	5662 (3)	33 (1)
C (8)	11408 (4)	1994 (4)	7316 (5)	31 (1)
C (9)	8182 (4)	6815 (4)	1553 (6)	31 (1)
C (10)	7003 (4)	6921 (6)	1415 (7)	45 (1)
C (11)	6428 (5)	8412 (7)	791 (8)	61 (2)
C (12)	6839 (5)	9638 (6)	1885 (8)	56 (2)
C (13)	8021 (4)	9501 (5)	2007 (7)	42 (1)
Cl (1X)	6611	1851	7921	61 (5)
C (7X)	5579	8253	6030	72

3. Results and Discussion

3.1. Host–Guest Binding Mode

Figure 1 shows the structure of the host : guest molecular complex between the *cis-anti-cis*-DCH-6 isomer and 3-chloro-6-methyl-benzenesulphamide. The complex has a stoichiometry of 1 : 2. The host molecule, an analogue of 18-crown-6, hindered by two cyclohexyl substituents, lies on an inversion centre. Thus, on both equal faces of the cavity the same guest molecules interact in an identical manner. Host–guest interactions involve both H-atoms of the sulphanmide group. The parameters of the two H-bonds are: N(1G)···O(1) 2.912 (5), H(1N)···O(1) 2.01 Å, angle

TABLE III. Bond lengths (Å) and angles (deg) for **1**.

Atoms	*d*, Å	Atoms	*d*, Å
Cl (1G)—C (3G)	1.735 (5)	O (1)—C (9)	1.441 (5)
S (1G)—O (2G)	1.424 (3)	C (2)—C (3)	1.491 (7)
S (1G)—O (1G)	1.432 (3)	C (3)—O (4)	1.427 (5)
S (1G)—N (1G)	1.582 (4)	O (4)—C (5)	1.432 (5)
S (1G)—C (1G)	1.784 (4)	C (5)—C (6)	1.492 (7)
C (1G)—C (2G)	1.383 (6)	C (6)—O (7)	1.429 (5)
C (1G)—C (6G)	1.394 (6)	O (7)—C (8)	1.429 (5)
C (2G)—C (3G)	1.379 (6)	C (8)—C (9)#	1.509 (6)
C (3G)—C (4G)	1.366 (7)	C (8)—C (13)#	1.526 (6)
C (3G)—C (7X)	1.503 (5)		
C (4G)—C (5G)	1.383 (8)	C (9)—C (10)	1.514 (7)
C (5G)—C (6G)	1.392 (7)	C (10)—C (11)	1.512 (8)
C (6G)—C (7G)	1.537 (9)	C (11)—C (12)	1.519 (9)
C (6G)—Cl (1X)	1.729 (5)	C (12)—C (13)	1.516 (8)
O (1)—C (2)	1.426 (5)		
Atoms	deg.	Atoms	deg.
O (2G)—S (1G)—O (1G)	118.6 (2)	C (2)—O (1)—C (9)	113.1 (3)
O (2G)—S (1G)—N (1G)	107.4 (2)	O (1)—C (2)—C (3)	109.9 (4)
O (1G)—S (1G)—N (1G)	108.4 (2)	O (4)—C (3)—C (2)	110.3 (4)
O (2G)—S (1G)—C (1G)	105.9 (2)	C (3)—O (4)—C (5)	112.9 (4)
O (1G)—S (1G)—C (1G)	107.2 (2)	O (4)—C (5)—C (6)	114.4 (4)
N (1G)—S (1G)—C (1G)	109.1 (2)	O (7)—C (6)—C (5)	109.8 (4)
C (2G)—C (1G)—C (6G)	120.7 (4)	C (6)—O (7)—C (8)	111.9 (3)
C (2G)—C (1G)—S (1G)	118.6 (3)	O (7)—C (8)—C (9)#	107.8 (3)
C (6G)—C (1G)—S (1G)	120.6 (3)	O (7)—C (8)—C (13)#	112.8 (4)
C (3G)—C (2G)—C (1G)	120.5 (4)	C (9)#—C (8)—C (13)#	108.9 (4)
C (4G)—C (3G)—C (2G)	120.3 (5)	O (1)—C (9)—C (8)#	109.0 (3)
C (4G)—C (3G)—C (7X)	120.6 (4)	O (1)—C (9)—C (10)	112.5 (4)
C (2G)—C (3G)—C (7X)	119.1 (4)	C (8)—C (9)—C (10)	111.1 (4)
C (4G)—C (3G)—Cl (1G)	120.6 (4)	C (11)—C (10)—C (9)	111.6 (5)
C (2G)—C (3G)—Cl (1G)	119.1 (4)	C (10)—C (11)—C (12)	111.1 (5)
C (3G)—C (4G)—C (5G)	119.0 (5)	C (13)—C (12)—C (11)	110.1 (5)
C (4G)—C (5G)—C (6G)	122.5 (5)	C (12)—C (13)—C (8)#	111.9 (4)
C (5G)—C (6G)—C (1G)	117.0 (4)		
C (5G)—C (6G)—C (7G)	118.9 (5)		
C (1G)—C (6G)—C (7G)	124.1 (5)		
C (5G)—C (6G)—Cl (1X)	120.3 (4)		
C (1G)—C (6G)—Cl (1X)	122.7 (4)		
Atoms			
C (9)—O (1)—C (2)—C (3)	178.0		
O (1)—C (2)—C (3)—O (4)	−65.5		
C (2)—C (3)—O (4)—C (5)	−175.5		
C (3)—O (4)—C (5)—C (6)	−79.2		
O (4)—C (5)—C (6)—O (7)	−71.4		
C (5)—C (6)—O (7)—C (8)	173.5		
C(6)—O (7)—C (8)—C (9)#	−172.2		
O (7)—C (8)—C (9)#—O (1)#	59.2		

Symmetry transformations used to generate equivalent atoms: # $-x+2, -y+1, -z+1$.

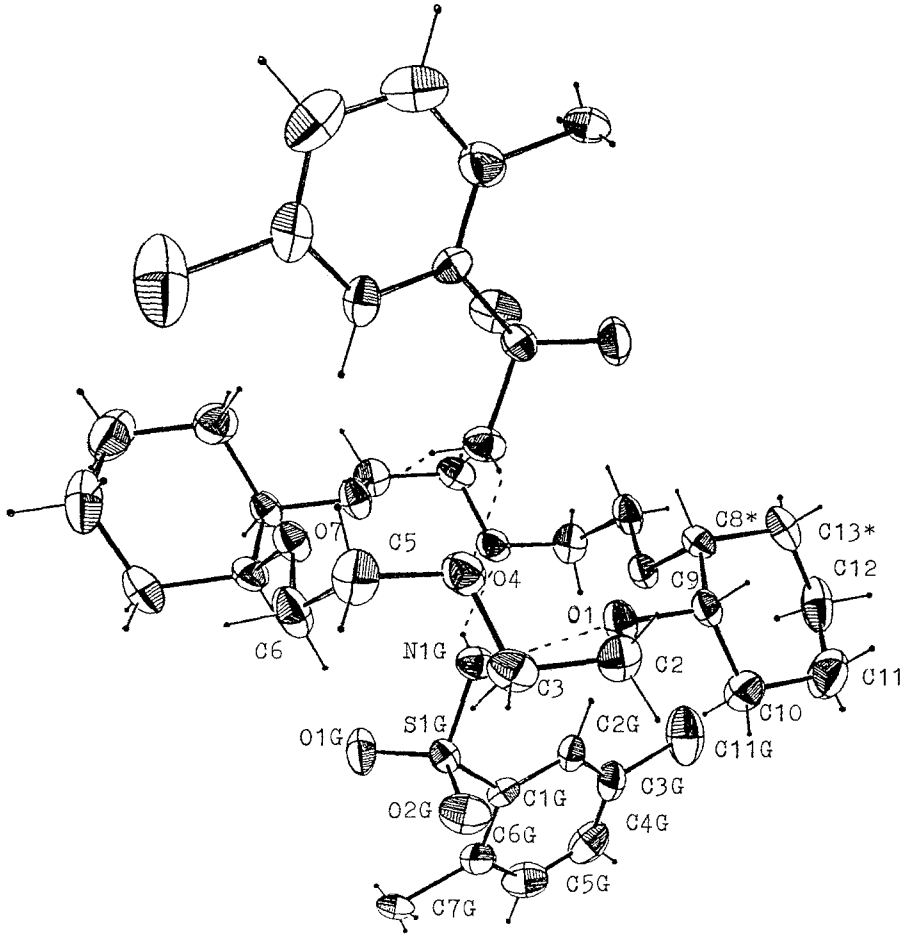

Fig. 1. ORTEP diagram of complex **I**. The atoms are represented with 50% probability ellipsoids. H-bonds are shown as dotted lines.

N(1G)···H(1N)···O(1) 164°; N(1G)···O(4)* 3.040 (5), H(2N)···O(4)* 2.27 Å, angle N(1G)—H(2N)···O(4)* 156° (* symmetry operation $2 - x$, $1 - y$, $1 - z$) (Figure 1). Host oxygen atoms divided by the oxyethylene fragment participate in these contacts. This is a simple mode of coordination for the equal-faced 18-membered crown ethers. The N-atom of the sulphamide group is 1.73 Å from the host average plane defined by the six oxygen atoms. The dihedral angle between the same plane of the crown ether and the guest aromatic moiety is 77.3° and defines the mutual arrangement of the two components in the complex.

The structure of the *cis-anti-cis-* DCH-6 complex with 2-methylbenzenesulphamide has been determined previously [14] by X-ray crystallography. The geometric parameters are similar to the analogous ones discussed here. The N···O

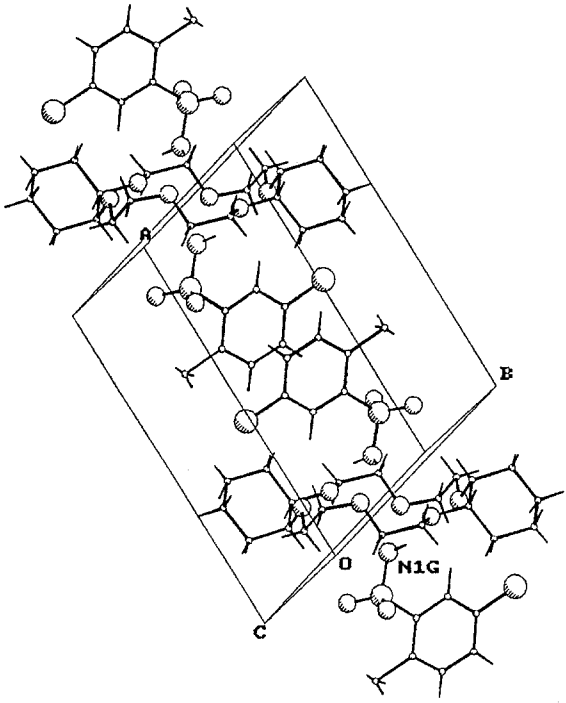

Fig. 2. Crystal packing of the complex in the unit cell.

distances are 2.910 and 3.082 Å. The N-atom of the sulphamide group is 1.74 Å from the host average plane defined by the six oxygen atoms.

Analysis of the intermolecular distances involving Cl and Me-group atoms shows that they do not participate in C—H· · ·Cl and C—H· · ·O non-bonded interactions. Note, however, the short Cl· · ·Cl contact of 3.211 Å between guest molecules belonging to different complexes and related by the symmetry operation $1 - x$, $2 - y$, $1 - z$. This value is in range for Ar—Cl· · ·Cl—Ar contacts described in refs. [15, 16]. All other intermolecular distances equal or exceed the sum of the van der Waals radii. The crystal packing for complex **I** is shown in Figure 2.

3.2. Host Conformation and Geometry

cis-anti-cis-DCH-6 has a di-angular conformation [17] with the corner fragments fixed at the C5 (C5*) atoms. All C—C bonds are in *gauche* conformation with torsion angles in the range 59.2–71.4°. Two of the C—O bonds related by the inversion center also adopt *gauche* conformations, with a torsion angle of −79.2°, all the other C—O bonds being in *anti* conformations with torsion angles in the range 152.8–178.0°. The various torsion angles in the macrocyclic framework are given in Table III. Host oxygen atoms are coplanar within ±0.217 Å. The transannular O· · ·O distances are O (1)· · ·O (1)* 5.152, O (4)· · ·O (4)* 5.913, O (7)* 5.957 Å. Bond distances and angles in the host molecule have the following average

TABLE IV. Anisotropic displacement parameters ($Å^2 \times 10^3$) for **1**. The anisotropic displacement factor exponent takes the form: $-2\pi^2 (h^2 a^{*2} U_{11} + \cdots + 2hka^* b^* U_{12})$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Cl (1G)	85 (1)	46 (1)	81 (1)	14 (1)	10 (1)	27 (1)
S (1G)	35 (1)	25 (1)	30 (1)	−1 (1)	8 (1)	−5 (1)
O (1G)	44 (2)	41 (2)	46 (2)	19 (2)	11 (2)	7 (2)
O (2G)	58 (2)	43 (2)	44 (2)	−19 (2)	14 (2)	−20 (2)
N (1G)	38 (2)	40 (2)	29 (2)	−1 (2)	9 (2)	−15 (2)
C (1G)	23 (2)	33 (2)	29 (2)	−2 (2)	1 (2)	−6 (2)
C (2G)	35 (3)	34 (3)	31 (3)	2 (2)	4 (2)	−5 (2)
C (3G)	42 (3)	38 (3)	44 (3)	1 (2)	1 (2)	6 (2)
C (4G)	33 (3)	71 (4)	63 (4)	−9 (3)	10 (3)	4 (3)
C (5G)	39 (3)	69 (4)	64 (4)	−6 (3)	20 (3)	−19 (3)
C (6G)	37 (3)	42 (3)	41 (3)	5 (2)	3 (2)	−13 (2)
C (7G)	65 (6)	39 (4)	135 (10)	19 (4)	41 (5)	−28 (3)
O (1)	52 (2)	25 (2)	26 (2)	−1 (1)	1 (1)	−5 (1)
C (2)	49 (3)	38 (3)	30 (3)	−8 (2)	0 (2)	−9 (2)
C (3)	47 (3)	32 (3)	34 (3)	−9 (2)	7 (2)	−14 (2)
O (4)	44 (2)	32 (2)	34 (2)	1 (1)	3 (2)	−10 (1)
C (5)	54 (4)	37 (3)	35 (3)	−9 (2)	4 (2)	1 (2)
C (6)	68 (4)	23 (2)	36 (3)	0 (2)	−2 (3)	−8 (2)
O (7)	42 (2)	28 (2)	27 (2)	1 (1)	3 (1)	−8 (1)
C (8)	34 (3)	28 (2)	32 (2)	3 (2)	11 (2)	−7 (2)
C (9)	39 (3)	29 (2)	25 (2)	6 (2)	6 (2)	−4 (2)
C (10)	41 (3)	52 (3)	40 (3)	1 (3)	−1 (2)	13 (2)
C (11)	41 (4)	76 (4)	57 (4)	15 (3)	0 (3)	5 (3)
C (12)	61 (4)	49 (3)	50 (4)	18 (3)	15 (3)	23 (3)
C (13)	62 (4)	31 (3)	37 (3)	6 (2)	16 (3)	−6 (2)

values: C(sp^3)—O 1.431, C(sp^3)—C(sp^3) 1.497 Å, endocyclic angles C—O—C 112.9, C—C—O 110.2°. The cyclohexyl substituents have a chair conformation with the average C—C bond distance equal to 1.519 Å and C—C—C angle 110.8° (Table III).

3.3. Guest Geometry

The geometric characteristics of the guest molecule are similar to those for related aromatic molecules. The average C—C distance is 1.388 Å, the C—C—C endocyclic angles are in the range 117.0–122.5° and depend on the substituents bonded to the aromatic moiety.

The torsion angles in the guest molecule around the S—C bond are similar to those given in ref. [14]. The torsion angles N(1G)—S(1G)—C(1G)—C(2G) and

N(1G)—S(1G)—C(1G)—C(6G) have the values 17.0 and $-167.2°$ in this guest molecule; the corresponding values for 2-methylbenzenesulphamide are -11.9 and $+169.9°$. According to [18] this is an unusual conformation for benzenesulphamide derivatives, since the N—S—C(1)—C (x) angles more frequently fall in the range $-75 \pm 20°$, which is the most heavily populated range found in a search of the Cambridge Crystal Data File.

The crystal data analysis confirms that full selective complexation with only one guest isomer does not occur. The similar topology of 3-chloro-6-methylbenzenesulphamide and 2-chloro-5-methylbenzenesulphamide results in the enrichment of the first isomer by the complex. This phenomenon is connected with the packing conditions rather than with the host-guest interaction peculiarities.

References

1. M. Hiraoka: *Kraun-soedineniya. Ih svoiatva i primenenie.* Moscow, Nauka, p. 363 (1986). M. Hiraoka: *Crown Compounds. Their Characteristics and Applications,* Kodansha Ltd., Amsterdam-Oxford-New York.
2. N.P. Strel'tsova, A.A. Varnek, A.S. Glebov and V.K. Bel'skii: in *Problems of Crystal Chemistry* (Russian), A collection of papers, Moscow, Nauka, p. 47 (1989).
3. D.J. Cram: *Chemica Scripta* **28**, 263 (1988).
4. J.S. Bradshaw, P. Huszthy, T. Wang, Ch. Zhu, A.Y. Nazarenko and R.M. Izatt: *Supramol. Chem.* **1**, 267 (1993).
5. Yu.A. Simonov, M.S. Fonari, A.A. Dvorkin and T.J. Malinowski: in *Problems of Modern Crystallography* (Russian), A collection of papers, Moscow, Nauka, p. 221 (1992).
6. E.V. Ganin, V.F. Makarov, S.A. Kotlyar, N.G. Luk'yanenko, M.S. Fonari, A.A. Dvorkin and Yu.A. Simonov: *Khim. Geterocycl. Soed.* (Russian) **9**, 1190 (1988).
7. A.A. Dvorkin, M.S. Fonari, S.T. Malinowski, E.V. Ganin, Yu.A. Simonov, V.F. Makarov, S.A. Kotlyar and N.G. Luk'yanenko: *Zh. Strukt. Khim.* (Russian) **30**, 96 (1989).
8. Yu.A. Simonov, E.V. Ganin, M.S. Fonari, A.A. Dvorkin and S.A. Kotlyar: *Zh. Strukt. Khim.* (Russian) **31**, 106 (1990).
9. A. Altomare, G. Cascarano, C. Giacovazzo, A. Guagliardi, M.C. Burla, G. Polidori and M. Camalli: *SIR92 Program for Crystal Structure Determination.* Personal communication.
10. G.M. Sheldrick: *SHELX76, Program for Crystal Structure Determination,* University of Cambridge, England (1976).
11. G.M. Sheldrick: *SHELXL-93, Program for Crystal Structure Determination,* University of Göttingen, Germany (1993).
12. C. Rizzoli, V. Sangermano, G. Calestaniand and G.D. Andreetti: 'CRYSRULER, an Integrated System of Computer Programs for Crystal Structure Analysis on a Personal Computer', *J. Appl. Crystallogr.* **20**, 436 (1987).
13. C.K. Johnson: ORTEP II. Report ORNL-5138. Oak Ridge National Lab. Tennessee, U.S.A. (1976).
14. Y.A. Simonov, M.S. Fonari, E.V. Ganin, A.A. Dvorkin, G.S. Musienko, E.N. Krylov and T.I. Malinowski: *Zh. Strukt. Khim.* (Russian) **33**, 138 (1992).
15. N. Ramasubbu, R. Parthasarathy and P. Murray-Rust: *J. Am. Chem. Soc.* **108**, 4308 (1986).
16. B.S. Goud, S.S. Pathaneni and G.R. Desiraju: *Acta Crystallogr.* **C49**, 1107 (1993).
17. J. Dale: *Acta Chem. Scand.* **27**, 1115 (1973).
18. A. Kalman, M. Czugler and G. Argay: *Acta Crystallogr.* **B37**, 868 (1981).