

C4a—C4b—C5	121.9 (2)	C4a—C10a—C10	120.3 (2)
C4a—C4b—C8a	119.8 (2)	C13—C12—S	107.45 (13)
C4b—C8a—C9	119.47 (15)	C14—C13—C12	111.3 (2)
C10—C9—C8a	120.1 (2)		

Compound (4)*Crystal data*

C ₁₈ H ₁₈ S	Mo K α radiation
$M_r = 266.38$	$\lambda = 0.71069 \text{ \AA}$
Monoclinic	Cell parameters from 20 reflections
$P2_1/c$	$\theta = 4-8^\circ$
$a = 9.010 (4) \text{ \AA}$	$\mu = 0.211 \text{ mm}^{-1}$
$b = 23.124 (8) \text{ \AA}$	$T = 193 (2) \text{ K}$
$c = 6.888 (3) \text{ \AA}$	Plate
$\beta = 97.81 (3)^\circ$	$0.3 \times 0.2 \times 0.2 \text{ mm}$
$V = 1421.8 (10) \text{ \AA}^3$	Colourless
$Z = 4$	
$D_x = 1.244 \text{ Mg m}^{-3}$	
D_m not measured	

Data collection

Rigaku AFC-7S diffractometer	$\theta_{\max} = 26.51^\circ$
$\omega/2\theta$ scans	$h = -11 \rightarrow 11$
Absorption correction: none	$k = -28 \rightarrow 28$
2851 measured reflections	$l = 0 \rightarrow 8$
2851 independent reflections	20 standard reflections
2273 reflections with $I > 2\sigma(I)$	every 100 reflections
	intensity decay: none

Refinement

Refinement on F^2	$(\Delta/\sigma)_{\max} = -0.077$
$R[F^2 > 2\sigma(F^2)] = 0.060$	$\Delta\rho_{\max} = 0.362 \text{ e \AA}^{-3}$
$wR(F^2) = 0.166$	$\Delta\rho_{\min} = -0.631 \text{ e \AA}^{-3}$
$S = 1.964$	Extinction correction: <i>SHELXL93</i>
2839 reflections	Extinction coefficient: 0.0016 (14)
227 parameters	Scattering factors from <i>International Tables for Crystallography</i> (Vol. C)
H atoms riding	
$w = 1/[\sigma^2(F_o^2) + (0.0016P)^2 + 0.1P]$	
where $P = (F_o^2 + 2F_c^2)/3$	

Table 4. *Selected geometric parameters ($\text{\AA}$, $^\circ$) for (4)*

S—C9	1.766 (3)	C8a—C9	1.459 (4)
S—C12	1.803 (3)	C9—C10	1.357 (4)
C4—C4a	1.410 (3)	C10—C10a	1.430 (4)
C4a—C4b	1.449 (4)	C12—C13	1.527 (4)
C4a—C10a	1.416 (4)	C13—C14	1.525 (4)
C4b—C5	1.418 (4)	C14—C15	1.521 (4)
C4b—C8a	1.430 (3)		
C9—S—C12	104.51 (13)	C10—C9—S	123.6 (2)
C4—C4a—C4b	123.1 (2)	C8a—C9—S	115.7 (2)
C4b—C4a—C10a	118.8 (2)	C9—C10—C10a	121.5 (2)
C4a—C4b—C5	122.5 (2)	C4a—C10a—C10	120.3 (2)
C4a—C4b—C8a	120.2 (2)	C13—C12—S	107.5 (2)
C4b—C8a—C9	118.4 (2)	C14—C13—C12	111.7 (2)
C10—C9—C8a	120.7 (2)	C15—C14—C13	112.1 (3)

Observed intensities were corrected for Lorentz and polarization effects and extinction. All non-H atoms were refined anisotropically. H atoms with isotropic U values were placed in calculated positions and refined using a riding model for (1), (2) and (4). In compound (3), additional H-atom coordinates and isotropic U values were refined.

For all compounds, data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1992); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN* (Molecular Structure Corporation, 1993); program(s) used to solve structure: *SHELXTL/PC* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993); molecular graphics: *SHELXTL/PC*; software used to prepare material for publication: *SHELXL93*.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: KA1230). Services for accessing these data are described at the back of the journal.

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5,8-Bis(4-methoxy-2,3,6-trimethylbenzene-sulfonyl)-1,4-dioxo-5,8-diazocane

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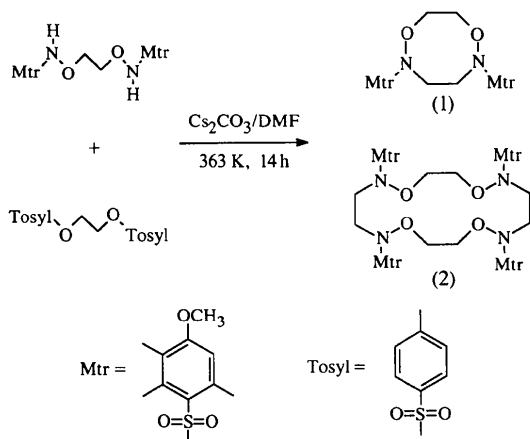
(Received 9 October 1996; accepted 6 March 1997)

Abstract

The molecular shape of the title compound, C₂₄H₃₄N₂O₈S₂, in the crystalline state is characterized by internal twofold symmetry, with the eight-membered ring adopting a crown conformation.

Comment

For the last decade, the field of macrocyclic chemistry has generated intense interest among scientists because of its wide range of applications (Alexander, 1995; Dietrich, Viout & Lehn, 1993). In our continued effort to synthesize novel macrocycles, in particular those related to oxa-macrocycles, which potentially exhibit degenerate properties when complexed with metals, we isolated, from the reaction between bis[*N*-(4-methoxy-2,3,6-benzenesulfonyl)]diaminoethan-1,2-diol and bis-(tosyl)ethan-1,2-diol in the presence of caesium carbonate in anhydrous DMF, the 1:1 adduct, (1), instead of the 2:2 adduct, (2). NMR and mass spectroscopic analysis of the product did not fully discriminate between (1) and (2). In the mass spectrum, although the main peak was observed at 543.5 m.u. ($M^+ + H$), a low-intensity peak was also recorded at 1086.5 m.u. ($2M^+ + H$). A single-crystal structure determination was therefore undertaken and has confirmed the exclusive formation of the 1:1 adduct, (1).



The molecular structure of (1) with the labelling scheme is depicted in Fig. 1. The molecule possesses approximate C_2 point symmetry, with the twofold axis running through the midpoints of the bonds C3—C4 and

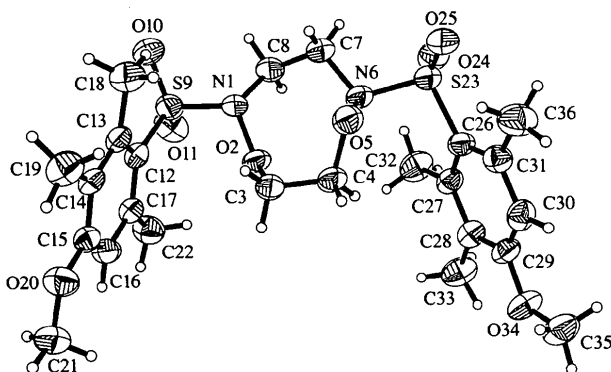


Fig. 1. View of (1) with labelling showing 50% probability displacement ellipsoids (SHELXTLPC; Sheldrick 1990). H atoms are shown as small circles of arbitrary radii.

C7—C8. Only the phenyl-ring substituent atoms C19, O20, C21, C22 and their pseudo-symmetric equivalents C33, O34, C35, C36 fail to satisfy the internal rotation symmetry. The best plane through the eight-membered ring (r.m.s. deviation 0.342 Å) makes an angle of 74.1 (1)° with the best plane through ring C12—C17 (r.m.s. deviation 0.009 Å), and of 55.9 (1)° with the best plane through ring C26—C31 (r.m.s. deviation 0.014 Å).

The atoms of the eight-membered ring are displaced alternately above and below the best plane through the eight atoms. This crown conformation is energetically the most favourable, with the C3—C4 and C7—C8 bonds having *gauche* conformations [torsion angles O2—C3—C4—O5 61.9 (5) and N6—C7—C8—N1 64.7 (5)°] and the sulfonyl groups placed equatorially. According to the Cremer & Pople (1975) treatment, the eight-membered ring (numbered in sequence N1, O2, ...) can be described by the puckering coordinates $q(2)$, $q(3)$, $q(4)$, $\varphi(2)$ and $\varphi(3)$, which are 0.477 (4), 0.020 (4) and -0.843 (4) Å and 230.3 (5) and 122 (11)°, respectively. Both N atoms are sp^3 hybridized, with the sum of the angles around the N atom being 326 (N1) and 329° (N6).

The crystal packing shows the stacking of the aromatic rings and short intermolecular contacts (2.450 Å) between atoms O10 and H21C of molecules related by an inversion centre.

Experimental

Diaminoethane-1,2-diol dihydrochloride, prepared as previously described (Shirayev, Moiseev & Kong Thoo Lin, 1997) (0.25 g, 0.0015 mol) was dissolved in anhydrous pyridine (15 ml). To this solution was added 4-methoxy-2,3,6-trimethylbenzenesulfonyl chloride (0.79 g, 0.0032 mol). After 2 h the solvent was removed and the solid residue was dissolved in chloroform and washed with bicarbonate solution. Evaporation of the solvent gave a solid which was recrystallized from acetonitrile to give bis(*N*-Mtr)diaminoethan-1,2-diol as colourless crystals (42%). To a solution of the ethan-1,2-diol (15.5 g, 0.25 mol) and triethylamine (71 ml, 0.5 mol) in dichloromethane (400 ml) was added tosyl chloride (95 g, 0.5 mol). After 5 h, at room temperature a precipitate formed. It was filtered off and the filtrate was washed with bicarbonate solution. After drying and evaporation of the dichloromethane layer, the crude product was recrystallized from ethanol to afford pure bis(tosyl)ethan-1,2-diol as colourless crystals (66%). A mixture of bis(*N*-Mtr)diaminoethan-1,2-diol (0.25 g, 0.00048 mol), bis(tosyl)ethan-1,2-diol (0.179 g, 0.00048 mmol) and Cs_2CO_3 (0.358 g, 0.0011 mmol) in anhydrous DMF (30 ml) was heated at 363 K for 14 h in a pressure bottle. The residue left after removal of solvent was dissolved in water and extracted three times with chloroform. After drying and evaporation of the solvent, the crude product was recrystallized from ethanol/acetonitrile to give (1) as colourless crystals (24%, m.p. 481–483 K). FAB MS (VGZAB-E NOBA matrix): m/z = 543.5 ($M^+ + H$). 1H NMR ($CDCl_3$, 90 MHz, Jeol EX90, δ p.p.m.): 2.08 (3H singlet, CH_3), 2.53 (3H singlet, CH_3), 2.62

(3H singlet, CH₃), 3.28 (2H singlet, CH₂), 3.71 (2H singlet, CH₂), 3.79 (3H singlet, OCH₃), 6.54 (1H singlet, aromatic proton).

Crystal data

C₂₄H₃₄N₂O₈S₂

$M_r = 542.65$

Triclinic

$P\bar{1}$

$a = 9.079 (1) \text{ \AA}$

$b = 10.188 (2) \text{ \AA}$

$c = 14.920 (2) \text{ \AA}$

$\alpha = 74.49 (1)^\circ$

$\beta = 80.00 (1)^\circ$

$\gamma = 83.13 (1)^\circ$

$V = 1305.7 (3) \text{ \AA}^3$

$Z = 2$

$D_x = 1.380 \text{ Mg m}^{-3}$

D_m not measured

Cu $K\alpha$ radiation

$\lambda = 1.54178 \text{ \AA}$

Cell parameters from 20 reflections

$\theta = 11\text{--}19^\circ$

$\mu = 2.282 \text{ mm}^{-1}$

$T = 289 (2) \text{ K}$

Block

$0.40 \times 0.35 \times 0.20 \text{ mm}$

White

Data collection

Siemens P4-PC diffractometer

$2\theta/\omega$ scans

Absorption correction: none

3455 measured reflections

2684 independent reflections

2479 reflections with

$I > 2\sigma(I)$

$R_{\text{int}} = 0.039$

$\theta_{\text{max}} = 50.44^\circ$

$h = -1 \rightarrow 8$

$k = -9 \rightarrow 9$

$l = -14 \rightarrow 14$

3 standard reflections

every 97 reflections

intensity decay: 5%

Refinement

Refinement on F^2

$R[F^2 > 2\sigma(F^2)] = 0.058$

$wR(F^2) = 0.165$

$S = 1.061$

2682 reflections

326 parameters

H atoms riding with $U(\text{H}) = 1.2U_{\text{eq}}(\text{C})$

$w = 1/[\sigma^2(F_o^2) + (0.0979P)^2 + 1.6198P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} = 0.001$

$\Delta\rho_{\text{max}} = 0.33 \text{ e \AA}^{-3}$

$\Delta\rho_{\text{min}} = -0.35 \text{ e \AA}^{-3}$

Extinction correction:

$SHELXL93$ (Sheldrick, 1993)

Extinction coefficient: 0.0012 (5)

Scattering factors from *International Tables for Crystallography* (Vol. C)

Rapid decomposition of the crystal led to premature termination of the data collection ($\theta_{\text{max}} = 50^\circ$) and precluded measurement of ψ -scan data for empirical absorption.

Data collection and data reduction: *XSCANS* (Siemens, 1992). Program used to solve structure: *SHELXTLPC* (Sheldrick, 1990). Program used to refine structure and to prepare material for publication: *SHELXL93* (Sheldrick, 1993) and *SHELXTLPC*. Program used to search for internal symmetry: *BUNYIP* in *Xtal3.4* (Hall, King & Stewart, 1995). Molecular graphics: *SHELXTLPC*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: MU1298). Services for accessing these data are described at the back of the journal.

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Two Pairs of Stereoisomers of Dimethyl 2-(3,4,5-Trimethoxyphenyl)tetrahydrofuran-3,4-dicarboxylate

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Abstract

Two racemic pairs of stereoisomers of the title compound, C₁₇H₂₂O₈, were characterized by single-crystal

Table 1. Selected geometric parameters ($\text{\AA}$, $^\circ$)

N1—O2	1.445 (4)	N6—S23	1.689 (4)
N1—C8	1.456 (5)	C7—C8	1.495 (6)
N1—S9	1.708 (4)	S9—O11	1.421 (3)
O2—C3	1.428 (5)	S9—O10	1.441 (3)
C3—C4	1.501 (6)	S9—C12	1.763 (4)
C4—O5	1.439 (5)	S23—O25	1.420 (4)
O5—N6	1.440 (4)	S23—O24	1.430 (4)
N6—C7	1.482 (6)	S23—C26	1.776 (4)
O2—N1—C8	108.0 (3)	O11—S9—O10	117.1 (2)
O2—N1—S9	105.7 (2)	O11—S9—N1	111.16 (19)
C8—N1—S9	112.6 (3)	O10—S9—N1	103.58 (18)
C3—O2—N1	110.3 (3)	O11—S9—C12	111.2 (2)
O2—C3—C4	112.7 (3)	O10—S9—C12	112.1 (2)
O5—C4—C3	112.5 (3)	N1—S9—C12	100.04 (18)
N6—O5—C4	110.6 (3)	O25—S23—O24	117.4 (2)
O5—N6—C7	109.0 (3)	O25—S23—N6	110.89 (19)
O5—N6—S23	106.2 (2)	O24—S23—N6	104.1 (2)
C7—N6—S23	113.4 (3)	O25—S23—C26	111.1 (2)
N6—C7—C8	112.5 (4)	O24—S23—C26	111.4 (2)
N1—C8—C7	112.6 (4)	N6—S23—C26	100.38 (18)