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Trimethylsulfonium methanesulfonate

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In the title compound, $C_3H_9S^+.CH_3O_3S^-$, a thermal decomposition product of dimethyl sulfoxide, both cation and anion lie on mirror planes. In the cation, the S atom lies 0.792 (2) Å out of the plane defined by the three C atoms, with S—C distances of 1.781 (2) and 1.786 (3) Å. In the anion, the S—O distances are 1.4556 (14) and 1.4646 (19) Å, and the S—C distance is 1.759 (3) Å.

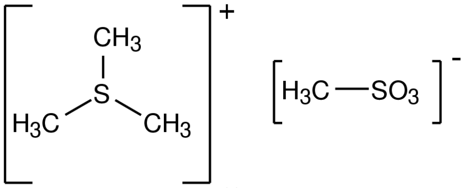
Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(S–O) = 0.002 Å
R factor = 0.039
wR factor = 0.089
Data-to-parameter ratio = 17.7

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

We have been studying the structure and mechanism of formation of colored products derived from resorcinarene macrocycles upon heating in dimethyl sulfoxide (DMSO) (Lewis *et al.*, 1997, 2000; Davis *et al.*, 1999). We isolated the title compound, (I), a decomposition product formed by prolonged heating of a solution of macrocycle in DMSO, and determined its structure to ascertain its identity. While 38 salts of the trimethylsulfonium ion and 62 salts of the methylsulfonate anion are present in the Cambridge Structural Database (December 2000, 224400 entries; Allen & Kennard, 1993), the structure of the title compound has not been previously reported. Decomposition of DMSO or its complexes to form trimethylsulfonium methanesulfonate has been previously reported as a result of heating (Banci, 1967; Arsenin *et al.*, 1988) and γ irradiation (Gutierrez *et al.*, 1977).



(I)

Both cation and anion lie across crystallographic mirrors. In the cation, the S atom lies 0.792 (2) Å out of the plane defined by the three C atoms. Geometric parameters (Table 1) are normal. Most of the H atoms are involved in C—H···O hydrogen bonding (Table 2)

Experimental

For the preparation of (I), the tetramethylresorc[4]arene 2,8,14,20-tetramethylpentacyclo[19.3.1.13,7.19,13.115,19]octacosa-1(25),3,5,-7(28),9,11,13(27),15,17,19(26),21,23-dodecaene-4,6,10,12,16,18,22,24-octol (250 mg, 0.46 mmol) was placed in a sealed tube along with 12 ml of DMSO and 3 ml of water. The mixture was heated at 493 K for 36 h. An aliquot was removed, and the solvent evaporated, yielding colorless crystals of the title compound.

Crystal data

$C_3H_9S^+ \cdot CH_3O_3S^-$
$M_r = 172.26$
Orthorhombic, *Pnma*
$a = 12.6157$ (4) Å
$b = 8.2419$ (4) Å
$c = 7.5397$ (8) Å
$V = 783.96$ (9) Å^3
$Z = 4$
$D_x = 1.459$ Mg m^{-3}

Mo *Kα* radiation
Cell parameters from 7642 reflections
$\theta = 2.5$–30.0°
$\mu = 0.62$ mm^{-1}
$T = 120$ K
Plate, colorless
0.12 × 0.10 × 0.02 mm

Data collection

KappaCCD diffractometer (with Oxford Cryosystems Cryostream cooler)
ω scans with *κ* offsets
Absorption correction: multi-scan (*HKL SCALEPACK*; Otwinowski & Minor, 1997)
$T_{\min} = 0.936$, $T_{\max} = 0.988$

7642 measured reflections
1222 independent reflections
838 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.047$
$\theta_{\max} = 30.1°$
$h = -17 \rightarrow 17$
$k = -11 \rightarrow 11$
$l = -10 \rightarrow 10$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.089$
$S = 1.02$
1222 reflections
69 parameters
Only coordinates of H atoms refined

$w = 1/[\sigma^2(F_o^2) + (0.0326P)^2 + 0.2622P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.40$ e Å^{-3}
$\Delta\rho_{\min} = -0.37$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0067 (18)

Table 1
Selected geometric parameters (Å, °).

S1—O1	1.4646 (19)	S2—C2	1.786 (3)
S1—O2	1.4556 (14)	S2—C3	1.781 (2)
S1—C1	1.759 (3)		
O2—S1—O1	112.20 (7)	O2—S1—C1	106.11 (8)
O2$^{\rm i}$—S1—O2	113.45 (12)	C3—S2—C3$^{\rm ii}$	101.31 (15)
O1—S1—C1	106.11 (13)	C3—S2—C2	102.00 (10)

Symmetry codes: (i) $x, \frac{1}{2} - y, z$; (ii) $x, \frac{3}{2} - y, z$.

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1*B*···O2$^{\rm i}$	0.95 (2)	2.54 (2)	3.463 (2)	163.5 (18)
C2—H2*A*···O1$^{\rm ii}$	1.06 (3)	2.41 (3)	3.382 (4)	152 (2)
C3—H3*A*···O1$^{\rm ii}$	0.97 (2)	2.48 (2)	3.388 (3)	156.0 (19)
C3—H3*B*···O2$^{\rm iii}$	0.95 (2)	2.44 (2)	3.279 (3)	147.3 (19)
C3—H3*C*···O1	0.92 (3)	2.41 (3)	3.280 (2)	157.1 (19)

Symmetry codes: (i) $\frac{3}{2} - x, -y, z - \frac{1}{2}$; (ii) $1 - x, \frac{1}{2} + y, -z$; (iii) $1 - x, \frac{1}{2} + y, 1 - z$.

The coordinates of H atoms were refined, while their isotropic displacement parameters were assigned as $U_{\rm iso} = 1.5U_{\rm eq}$ of the attached atom.

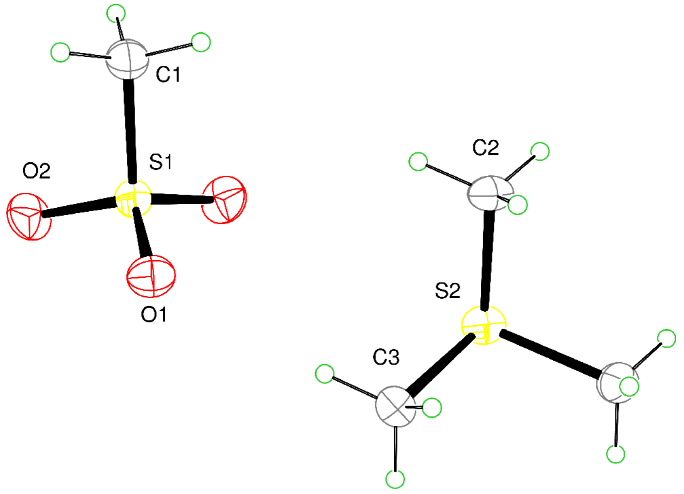


Figure 1
The atom-numbering scheme for (I) with ellipsoids at the 50% probability level.

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *DENZO* and *SCALEPACK*; data reduction: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SIR* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 (Farrugia, 1997); software used to prepare material for publication: *SHELXL*97.

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References

Allen, F. H. & Kennard, O. (1993). *Chem. Des. Autom. News*, **8**, 1, 31–37.
Altomare, A., Cascarano, G., Giacovazzo, C. & Guagliardi, A. (1994). *J. Appl. Cryst.* **27**, 435.
Arsenin, K. I., Malinko, L. A., Sheka, I. A., Pishchai, I. Y., Tikhonova, R. V., Kolotio, L. V. & Antishko, A. N. (1988). *Zh. Obshch. Khim.* **58**, 2102–2109.
Banci, F. (1967). *Ann. Chim.* **57**, 549–554.
Davis, C. J., Lewis, P. T., McCarroll, M. E., Reed, M. W., Cueto, R. & Strongin, R. M. (1999). *Org. Lett.* **1**, 331–334.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Gutierrez, M. C., Barrra, R., Sanz, H. & Parellada, R. (1977). *An. Quim.* **73**, 520–526.
Lewis, P. T., Davis, C. J., Cabell, L. A., He, M., Read, M. W., McCarroll, M. E. & Strongin, R. M. (2000). *Org. Lett.* **2**, 589–592.
Lewis, P. T., Davis, C. J., Saraiva, M. C., Treleavan, D., McCarley, T. & Strongin, R. M. (1997). *J. Org. Chem.* **62**, 6110–6111.
Nonius (2000). *COLLECT*. Nonius BV, Delft, The Netherlands.
Otwinowski, Z. & Minor, W. (1997). *Methods Enzymol.* **276**, 307–326.
Sheldrick, G. (1997). *SHELXL*97. University of Göttingen, Germany.

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