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The tris(dimethoxyethane) adduct of strontium iodide

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Key indicators

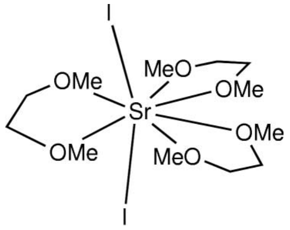
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.013 Å
R factor = 0.037
wR factor = 0.092
Data-to-parameter ratio = 21.5

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

Strontium iodide was prepared from strontium metal and ammonium iodide in liquid ammonia and crystallized as the dimethoxyethane adduct, namely tris(1,2-dimethoxyethane-$\kappa^2 O,O'$)diiodostrontium(II), $[SrI_2(C_4H_{10}O_2)_3]$. High purity and crystallinity, solubility, and ease of preparation are characteristics of this anhydrous starting material.

Comment

The reaction of strontium metal with a solution of ammonium iodide in liquid ammonia followed the published procedure for the synthesis of ytterbium diiodide (Tilley *et al.*, 1990). The single-crystal structure of the title tris(dimethoxyethane) complex, (I), obtained from dimethoxyethane (DME) solution, shows a bent I—Sr—I unit with an angle of 158.9 (2)°.



(I)

The six O donor atoms of the three DME ligands are arranged in a puckered chair-like fashion around the Sr ion, with Sr—O distances between 2.631 (4) and 2.656 (4) Å. The average Sr—O distance of 2.644 Å is slightly longer than that found for seven-coordinated $[SrI_2(THF)_5]$ (2.567 Å; THF is tetrahydrofuran; Ruhlandt-Senge *et al.*, 1995) and significantly longer than the average distance of 2.44 Å found in $[SrI_2\{O{=}P(NMe_2)_3\}_4]$ (Barr *et al.*, 1989).

The bite angles of the DME ligands vary from 60.22 (18) to 63.37 (14)° (average 61.58°). The O—Sr—O angles between adjacent O donor sites of different DME ligands are in the range 67.3 (2)–74.1 (2)°. The sum of the six O—Sr—O angles is 398.45° and this reflects the significant deviation of the O atoms (between 1.082 and 0.945 Å) from the calculated least-squares plane through the O atoms. The same coordination number and a similar coordination geometry have been found for $[SrI_2(bipy)_3]$ (bipy is 2,2′-bipyridine; Skelton *et al.*, 1996).

Experimental

Strontium metal (0.88 g, 10.0 mmol) was dissolved in a refluxing solution of ammonium iodide (2.90 g, 20.0 mmol) in liquid ammonia (100 ml). The solvent was allowed to evaporate and the residue was dissolved in boiling dimethoxyethane. The extract was filtered and allowed to cool slowly to 293 K. Colourless crystals of (I) up to several millimeters in size were obtained in 4.67 g yield (7.64 mmol; 76.4%). Analysis for $C_{12}H_{30}O_6I_2Sr$ (611.79), calculated: C 23.56, H 4.94%; found: C 23.10, H 4.77%.

Crystal data

$[SrI_2(C_4H_{10}O_2)_3]$
M_r = 611.78
Monoclinic, $P2_1$
a = 8.5222 (8) Å
b = 10.6245 (7) Å
c = 12.3742 (12) Å
β = 93.697 (11)°
V = 1118.08 (17) Å^3
Z = 2

D_x = 1.817 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 8000 reflections
θ = 2.4–25.9°
μ = 5.19 mm^{-1}
T = 293 (2) K
Block, colourless
0.54 × 0.36 × 0.33 mm

Data collection

Stoe IPDS diffractometer
φ scans
Absorption correction: analytical (*ABST* in *PLATON*; Spek, 2003)
$T_{\min}$ = 0.083, $T_{\max}$ = 0.227
15 530 measured reflections
4209 independent reflections

3735 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.091
$\theta_{\max}$ = 25.7°
$h = -10 \rightarrow 10$
$k = -12 \rightarrow 12$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.092
S = 1.05
4209 reflections
196 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.045P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max}$ = 0.013
$\Delta\rho_{\max}$ = 0.61 e Å^{-3}
$\Delta\rho_{\min}$ = −0.98 e Å^{-3}
Absolute structure: Flack (1983), with 2236 Friedel pairs
Flack parameter = 0.074 (9)

H atoms were included in calculated positions, with C—H = 0.97 Å (methylene) and 0.96 Å (methyl), and were included in the refinement in the riding-model approximation, with $U_{\rm iso}$ = 1.2 times (or 1.5 times for methyl) $U_{\rm eq}$(C).

Data collection: *EXPOSE* in *IPDS* (Stoe & Cie, 1997); cell refinement: *CELL* in *IPDS* (Stoe & Cie, 1997); data reduction: *INTEGRATE* in *IPDS* (Stoe & Cie, 1997); program(s) used to solve structure: *SIR*97 (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *SHELXTL*.

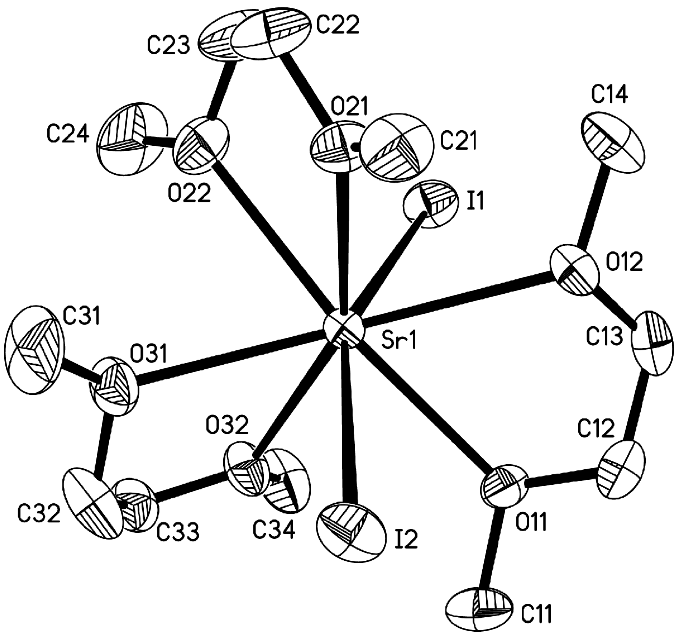


Figure 1
A view of (I) with the atomic numbering scheme. Displacement ellipsoids are drawn at the 40% probability level. H atoms have been omitted.

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