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Hydrogen bonding in *N,N,N',N'*-tetramethylethylenediammonium bis(benzeneselenolate)

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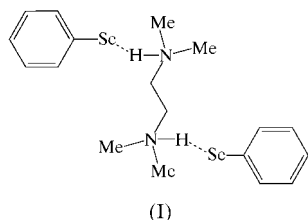
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The crystal structure of the title compound, $[\text{Me}_2\text{NHC}_2\text{H}_4\text{NHMe}_2][\text{SePh}]_2$ or $\text{C}_6\text{H}_{18}\text{N}_2^{2+}\cdot 2\text{C}_6\text{H}_5\text{Se}^-$, reveals hydrogen bonding between the benzeneselenolate anions and the tetramethylethylenediammonium cations. The asymmetric unit contains one formula unit of the title compound. The two $\text{Se}\cdots\text{H}$ distances are 2.22 (4) and 2.34 (4) Å.

Comment

During our studies on the preparation of $[\text{M}(\text{ER})_6]^{Z-}$ ($E = \text{S}, \text{Se}$), we also examined the reaction of $[\text{ZrMe}_6]^{2-}$ with benzeneselenol which afforded, in addition to the target compound, a white crystalline material. A selected crystal proved it to be the title compound, (I). A rational route to $[\text{Me}_2\text{NHC}_2\text{H}_4\text{NHMe}_2][\text{SePh}]_2$ was developed.



Experimental

Trimethylsilylphenylselenide (2.293 g, 10 mmol) was added to absolute ethanol (20 ml). After 10 min, the clear solution was treated with

N,N,N',N'-tetramethylethylenediamine (0.75 ml, 0.558 g, 5 mmol) at room temperature to give the product in 88.9% yield (m.p. 385–386 K).

Crystal data

$\text{C}_6\text{H}_{18}\text{N}_2^{2+}\cdot 2\text{C}_6\text{H}_5\text{Se}^-$
 $M_r = 430.36$
Monoclinic, $P2_1/a$
 $a = 11.542$ (4) Å
 $b = 11.246$ (5) Å
 $c = 15.641$ (5) Å
 $\beta = 105.43$ (3)°
 $V = 1957$ (2) Å³
 $Z = 4$

$D_x = 1.46$ Mg m⁻³
Mo $K\alpha$ radiation
Cell parameters from 10 reflections
 $\theta = 10\text{--}14^\circ$
 $\mu = 3.778$ mm⁻¹
 $T = 294$ (2) K
Plate, colourless
0.25 × 0.15 × 0.05 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
 θ/θ scans
Absorption correction: empirical via ψ scans (North *et al.*, 1968)
 $T_{\text{min}} = 0.381$, $T_{\text{max}} = 0.828$
4396 measured reflections
3845 independent reflections
2768 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.071$
 $\theta_{\text{max}} = 26^\circ$
 $h = 0 \rightarrow 14$
 $k = 0 \rightarrow 13$
 $l = -19 \rightarrow 18$
3 standard reflections
frequency: 50 min
intensity decay: 6.90%

Refinement

Refinement on F^2
 $R(F) = 0.035$
 $wR(F^2) = 0.085$
 $S = 1.006$
3845 reflections
311 parameters

All H-atom parameters refined
 $w = 1/[\sigma^2(F_o^2) + (0.0517P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.56$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.71$ e Å⁻³

Backgrounds were obtained from analysis of the scan profile (Blessing *et al.*, 1974). Refined distances involving H atoms were as follows: N–H 0.92 (4) and 0.99 (4) Å; C–H 0.82 (4)–1.06 (4) Å.

Data collection: *CAD-4 Operations Manual* (Enraf–Nonius, 1977); cell refinement: *CAD-4 Operations Manual*; data reduction: *PROCESS* in *MolEN* (Fair, 1990); program(s) used to solve structure: *MULTAN80* (Main *et al.*, 1980); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); software used to prepare material for publication: *CIF VAX* in *MolEN*.

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