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Bis(*N*,*N*-diethyl-1,1-diselenocarbamato-*Se*)selenium, [(Se$_2$CNEt$_2$)$_2$Se]

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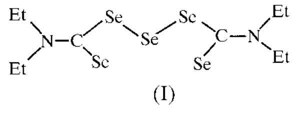
The polyseleno title compound, bis(*N*,*N*-diethylselenocarbamoyl) triselenide, [(Se$_2$CNEt$_2$)$_2$Se] or $C_{10}H_{20}N_2Se_5$, is obtained from the disproportion of sodium *N*,*N*-diethyl-1,1-diselenocarbamate. An Se atom connects two *N*,*N*-diethyl-1,1-diselenocarbamate groups with Se—Se distances in the range 2.4500 (11)–2.8601 (12) Å

Comment

In sharp contrast to many studies on the chemistry of metal complexes with 1,1-dithiolate ligands, the chemistry of metal complexes with 1,1-diselenolate ligands has received scant attention. Although the 2,2-dicyanoethylene-1,1-diselenolate and *N*,*N*-diethyl-1,1-diselenocarbamate ligands were prepared many years ago (Jensen & Henriksen, 1970; Barnard & Woodbridge, 1961), to our surprise, there are very few papers referring to such diselenolate ligands, among which the investigation was focused on the spectroscopic properties (Jensen & Krishnan, 1970), and very few crystal structures, such as bis(tetra-*n*-butylammonium) bis(2,2-dicyanoethylene-1,1-diselenolato)selenium(II) (Hummel *et al.*, 1992) and selenium bis(1-pyrrolidinecarbodiselenoate) (Esperas *et al.*, 1975), have been determined.

One of our current research interests is directed towards the understanding of transition metal complexes with seleno ligands (Hong *et al.*, 1998; Cao *et al.*, 1994). In an attempt to prepare organic ligands with polyselenide, we ran the oxidation of sodium *N*,*N*-diethyl-1,1-diselenocarbamate in MeOH and isolated an organic polyseleno compound ligand [(Se$_2$CNEt$_2$)$_2$Se], (I). The compound contains two *N*,*N*-diethyl-1,1-diselenocarbamate groups connected by an inorganic Se atom. If C1—Se1 and C6—Se3 are considered as double bonds, Se—Se—Se can be regarded as as a polyseleno center with an angle of 89.56 (4)°. The Se—Se distances in the range 2.4500 (11)–2.8601 (12) Å are comparable to those found in polyseleno compounds (Hummel *et al.*, 1992; Esperas *et al.*, 1975).



(I)

Experimental

The title compound was obtained from the disproportion of sodium *N*,*N*-diethyl-1,1-diselenocarbamate in CH_3OH. NaSe$_2$CNEt$_2$ (0.20 g, 0.75 mmol) was disolved in 30 ml of CH_3OH in air. The reaction solution turned red gradually. After stirring for 5 h, the red solution was filtered. The filtrate was kept in an icebox for one day to yield crystalline product of the title complex. Red prism crystals suitable for X-ray diffraction analysis were obtained by recrystallization from THF/CH_3OH at room temperature.

Crystal data

$C_{10}H_{20}N_2Se_5$
$M_r = 563.08$
Monoclinic, $P2_1/n$
$a = 6.7190$ (10) Å
$b = 10.029$ (2) Å
$c = 25.615$ (5) Å
$\beta = 90.65$ (3)°
$V = 1726.0$ (5) Å^3
$Z = 4$
$D_x = 2.167$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 456 reflections
$\theta = 8.34$–19.6°
$\mu = 10.595$ mm^{-1}
$T = 293$ (2) K
Prism, red
0.28 × 0.24 × 0.22 mm

Data collection

Smart CCD diffractometer
ω scans
Absorption correction: empirical empirical from equivalent reflections (*XEMP* in *SHELXTL*; Sheldrick, 1997*a*)
$T_{\rm min} = 0.0651$, $T_{\rm max} = 0.0974$
5262 measured reflections
2901 independent reflections
2205 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.040$
$\theta_{\rm max} = 25.03$°
$h = -7 \rightarrow 7$
$k = -11 \rightarrow 11$
$l = -21 \rightarrow 30$
Intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.048$
$wR(F^2) = 0.113$
$S = 0.996$
2901 reflections
155 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0564P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.74$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.99$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0076 (5)

Table 1
Selected geometric parameters (Å, °).

Se1—Se5	2.8601 (12)	Se3—Se5	2.7981 (12)
Se1—C1	1.848 (7)	Se3—C6	1.840 (7)
Se2—C1	1.898 (7)	Se4—C6	1.883 (7)
Se2—Se5	2.4847 (12)	Se4—Se5	2.4500 (11)
C1—Se2—Se5	89.6 (2)	Se1—C1—Se2	117.4 (4)
C6—Se4—Se5	89.1 (2)	N2—C6—Se3	124.1 (6)
Se4—Se5—Se2	89.56 (4)	N2—C6—Se4	119.0 (6)
N1—C1—Se2	118.5 (5)	Se3—C6—Se4	116.9 (4)

Data collection: *SMART CCD Software* (Siemens, 1994); cell refinement: *SMART CCD Software*; data reduction: *SMART CCD Software*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997*b*); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXL*97.

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