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2,2,5,5-Tetramethyl-1,6-diphenyl-7,8-diselenabicyclo[4.1.1]octane 7,8-dioxide

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.014 Å
R factor = 0.058
wR factor = 0.235
Data-to-parameter ratio = 16.8

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

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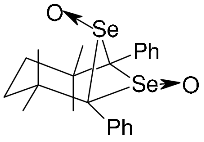
In the title compound, $C_{22}H_{26}O_2Se_2$, there are two selenoxide groups, and the two Se—O bonds point in opposite directions. The Se_2C_2 ring is not planar, having a dihedral angle between the two triangular C_2Se planes of 152.9 (3)°.

Comment

In our recent investigations of the oxidation of 1,3-diselenetane, we studied the reaction of 2,2,5,5-tetramethyl-1,6-diphenyl-7,8-diselenabicyclo[4.1.1]octane (Ishii *et al.*, 1992) with *m*-chloroperbenzoic acid. The title compound, (I), is one of the products in this reaction and an X-ray crystallographic analysis was undertaken to study its stereochemistry.



(I)

The molecular structure of (I) is shown in Fig. 1, and confirms the presence of two selenoxide groups. The bond lengths of the four Se—C bonds vary between 2.007 (8) and 2.051 (8) Å (Table 1). This is a little longer than the normal Se—C bond length (1.98–2.00 Å). The two Se—O bonds point in opposite directions (Table 1) and their bond lengths are 1.652 (7) and 1.651 (6) Å. This is compatible with the normal Se—O length. The other bond lengths in (I) are also within normal ranges (Allen *et al.*, 1987).

In the Se_2C_2 ring, the dihedral angle between the C1—Se1—C6 and C1—Se2—C6 planes is 152.9 (3)°. This is in

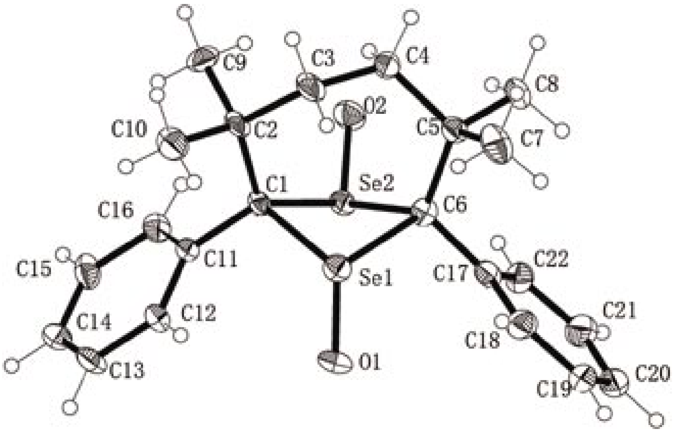


Figure 1
The structure of compound (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme. Dashed lines denote intermolecular C—H···O interactions.

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contrast to the approximately square-planar arrangement found for the Se_2C_2 ring in 1,3-diselenetanes (Eeker *et al.*, 1990; Adrien *et al.*, 1989).

The distance between the two Se atoms is 2.87 (2) Å, which is too long to be considered as a bond. In fact, it is possible to find several Se—Se bonds whose bond lengths are close to 2.87 (2) Å, but these bonds only exist in inorganic compounds (Shantha Nandana *et al.*, 1990; Dana & Andrews, 1996).

Experimental

A solution of 2,2,5,5-tetramethyl-1,6-diphenyl-7,8-diselenabicyclo[4.1.1]octane (179.9 mg, 0.40 mmol) in dichloromethane was added to a solution of *m*-chloroperbenzoic acid (96 mg, 0.56 mmol) in dichloromethane. The mixture was stirred for 15 min at room temperature. After the solvent had been removed under vacuum, the residue was separated on a chromatographic column, using an acetone–hexane (1:2 *v*/*v*) mixture as eluant. Single crystals suitable for X-ray crystallographic analysis were prepared by slow evaporation of a solution in dichloromethane–hexane (1:5 *v*/*v*).

Crystal data

$C_{22}H_{26}O_2Se_2$
$M_r = 480.35$
Monoclinic, $P2_1/n$
$a = 9.1930\ (18)$ Å
$b = 17.752\ (4)$ Å
$c = 12.779\ (3)$ Å
$\beta = 103.43\ (3)^\circ$
$V = 2028.4\ (7)$ Å^3
$Z = 4$
$D_x = 1.573$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 10–13^\circ$
$\mu = 3.66$ mm^{-1}
$T = 293\ (2)$ K
Block, colourless
0.4 × 0.3 × 0.3 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{min} = 0.279$, $T_{max} = 0.334$
4203 measured reflections
3951 independent reflections
2764 reflections with $I > 2\sigma(I)$
$R_{int} = 0.065$
$\theta_{max} = 26.0^\circ$
$h = 0 \rightarrow 10$
$k = 0 \rightarrow 21$
$l = -15 \rightarrow 15$
3 standard reflections every 200 reflections intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.058$
$wR(F^2) = 0.235$
$S = 1.14$
3951 reflections
235 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.14P)^2 + 4P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.007$
$\Delta\rho_{max} = 1.05$ e Å^{-3}
$\Delta\rho_{min} = -1.07$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Se1—C6	2.051 (8)	Se2—C6	2.007 (8)
Se2—O2	1.651 (6)	Se2—C1	2.023 (8)
O1—Se1—C1	106.1 (4)	O2—Se2—C6	112.3 (3)
O1—Se1—C6	103.2 (4)	O2—Se2—C1	113.7 (3)

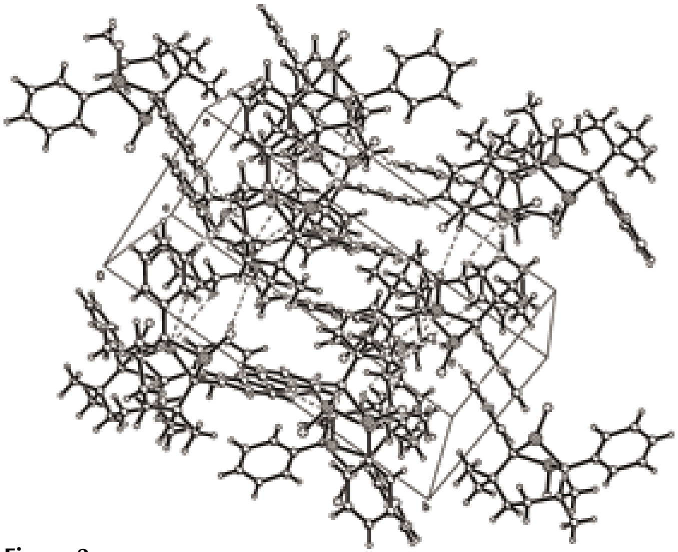

Figure 2
A packing diagram of compound (I).

The H atoms were included in calculated positions and treated as riding atoms [C—H distances are 0.93 Å for CH and 0.97 Å for CH_2 groups, with $U_{iso}(H) = 1.2U_{eq}(C)$, and C—H = 0.96 Å for methyl groups, with $U_{iso}(H) = 1.5U_{eq}(C)$].

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989); cell refinement: *CAD*-4 *Software*; data reduction: *XCAD*4 (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

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