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Acta Cryst. (1999). C**55**, 1861–1862

Absolute configuration of 2,4,6-tri-*tert*-butyldiphenylselenonium-*N*-toluene-4′-sulfonimide

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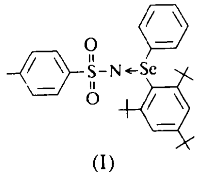
(*Received 5 May 1999; accepted 29 June 1999*)

Abstract

The title compound, *N*-[phenyl(2,4,6-tri-*tert*-butylphenyl)selenonio]-4-toluenesulfonamidate, $C_{31}H_{41}NO_2SSe$, is an optically pure enantiomer which was separated by an optically active column. The absolute configuration of the selenium atom was determined as the *S* configuration.

Comment

In the course of our studies on the optical resolution of selenonium imides using optically active columns (Taka *et al.*, 1999), we found that the racemic title compound, (I), can be separated into optically pure enantiomers. To determine the absolute configuration of the selenium atom of the (−)-enantiomer, (I*a*), X-ray analysis was carried out.



(I)

Compound (I) contains a tricoordinate Se atom (Fig. 1) with an imide N atom, a phenyl group and a 2,4,6-tri-*tert*-butylphenyl group. Thus, the Se atom is a chiral centre, with a tetrahedral configuration including lone-paired electrons. The absolute configuration of the Se atom could be significantly determined as *S* by the structure refinement using Bijvoet-pair reflections. The degree of pyramidalization of the Se atom is less pronounced than that of the Se atom in *N*-phenylsulfonyl-*Se*,*Se*-diphenylselenimide, (II) (Furmanova *et al.*, 1984) and the S atom in *N*-*p*-tolylsulfonyl-*S*,*S*-diphenylsulfimide, (III) (Kálmán *et al.*, 1971). The sums of the bond angles at Se or S in (I*a*), (II) and (III) are 310.3 (3), 295.9 (12) and 308.5 (7)°, respectively.

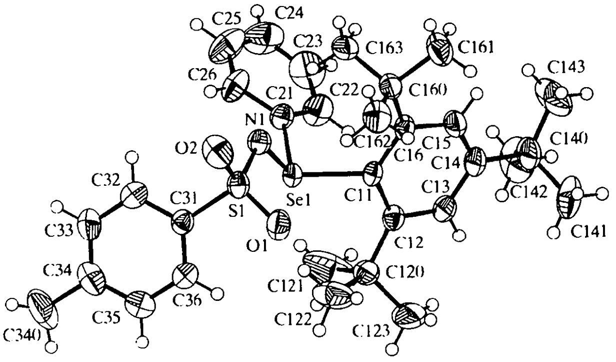


Fig. 1. The molecular structure of (I*a*) showing 50% probability displacement ellipsoids. H atoms are drawn as spheres with an arbitrary radius.

Although the length of the Se1—C21 bond corresponds with those found in (II) [1.93 (1)–1.95 (1) Å], the length of the Se1—C11 bond is significantly longer than those of Se1—C21 in (I*a*) and Se—C in (II). The deviation from planarity of C11 is more pronounced than those of C21 and C31. The sums of the bond angles at C11, C21 and C31 are 357.8 (4), 359.7 (6) and 360.0 (6)°, respectively. The less distinct pyramidal nature of the Se atom and the skewed bonds of C11 can be ascribed to the steric hindrance of two *tert*-butyl groups. No special intermolecular interactions except for van der Waals contacts were found in the crystal structure of (I*a*).

Experimental

The preparation and optical resolution of the (−)-enantiomer of (I) will be described in our related paper (Taka *et al.*, 1999). The crystals of (I*a*) used in the X-ray measurement were obtained by the slow evaporation of an acetonitrile solution in the dark.

Crystal data

$C_{31}H_{41}NO_2SSe$
$M_r = 570.67$

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å

Monoclinic
$P2_1$
$a = 10.8176\,(18)$ Å
$b = 9.7701\,(13)$ Å
$c = 15.242\,(3)$ Å
$\beta = 108.832\,(18)^\circ$
$V = 1524.6\,(4)$ Å^3
$Z = 2$
$D_x = 1.243$ Mg m^{-3}
D_m not measured

Cell parameters from 25 reflections
$\theta = 12.6$–20.1°
$\mu = 1.326$ mm^{-1}
$T = 293\,(2)$ K
Prismatic
0.65 × 0.32 × 0.20 mm
Colourless

Data collection

Rigaku AFC-5*R* diffractometer
$2\theta/\omega$ scans
Absorption correction: numerical (Coppens *et al.*, 1965)
$T_{\min} = 0.48$, $T_{\max} = 0.78$
7255 measured reflections
3707 independent reflections (plus 3294 Friedel-related reflections)

5804 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.027$
$\theta_{\max} = 27.49^\circ$
$h = -14 \rightarrow 13$
$k = -12 \rightarrow 12$
$l = 0 \rightarrow 19$
3 standard reflections every 100 reflections intensity decay: 13.2%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.105$
$S = 1.033$
7001 reflections
366 parameters
H atoms constrained
$w = 1/[\sigma^2(F_o^2) + (0.062P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.008$

$\Delta\rho_{\max} = 0.48$ e Å^{-3}
$\Delta\rho_{\min} = -0.64$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)
Absolute structure: Flack (1983)
Flack parameter = $-0.008\,(9)$

Table 1. *Selected geometric parameters (Å, °)*

Se1—N1	1.791 (3)	S1—N1	1.607 (3)
Se1—C21	1.932 (4)	S1—C31	1.769 (4)
Se1—C11	1.985 (3)		
N1—Se1—C21	96.63 (16)	C12—C11—C16	122.6 (3)
N1—Se1—C11	116.66 (14)	C12—C11—Se1	110.6 (2)
C21—Se1—C11	96.95 (15)	C16—C11—Se1	124.6 (2)
S1—N1—Se1	112.55 (17)		

The data collection covered a hemisphere of reciprocal space to include the Bijvoet pairs. The average intensity of the standard reflections decayed by 13.2%, and the colour of the specimen changed from colourless to red after the intensity measurement. Measured intensities were corrected by polynomial fitting of the variation of standards, although the chemical reason for this phenomenon was not clear. All H atoms were placed in geometrically calculated positions and refined using a riding model because of the large thermal displacement of the *tert*-butyl groups. Although C121, C123 and C143 had large anisotropic parameters, no disorder treatment could be applied. The final Flack parameter (Flack, 1983) was $-0.008\,(9)$, while the refinement of the inverted structure gave a value of 1.01 (2).

Data collection: *Rigaku/AFC Diffractometer Control Software* (Rigaku, 1990). Cell refinement: *Rigaku/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1992). Program(s) used to solve structure: *DIRDIF*92 (Beurskens *et al.*, 1992). Program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997). Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *SHELXL*97.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: OA1092). Services for accessing these data are described at the back of the journal.

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Tetracyclohexyloxaquaterene†

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(*Received 29 March 1999; accepted 2 July 1999*)

Abstract

The title compound, $C_{40}H_{48}O_4$, has twofold crystallographic symmetry and exhibits a square-like conformation, with furan rings positioned alternately up–down–up–down, nearly orthogonal to the mean plane of the macrocyclic ring. This is the most orthogonal position of the furan rings towards the mean plane of the macrocyclic ring among known compounds of this type.

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† Systematic name: 21,22,23,24-tetraoxapentacyclo[16.2.1.1^{3,6}.1^{8,11}.1^{13,16}]tetracosa-3,5,8,10,13,15,18,20-octaene-2,7,12,17-tetraspirocyclohexane.

Acta Crystallographica Section C
ISSN 0108-2701 © 1999