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trans-2-(2-Naphthyl)-3-(phenylselenyl)-tetrahydropyran

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Key indicators

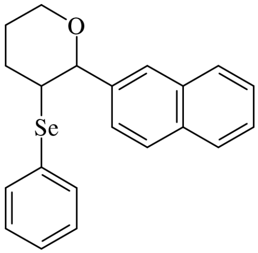
Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.004 Å
R factor = 0.026
wR factor = 0.065
Data-to-parameter ratio = 16.1

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

The crystal structure of the title compound, $C_{21}H_{20}OSe$, has established the absolute configuration and shows that the tetrahydrofuran ring adopts a chair conformation, with both the phenylselenyl and naphthyl substituents occupying equatorial positions.

Comment

As part of a programme directed towards the synthesis of aryl C-glycosides (Brimble *et al.*, 2002), selenoetherification of γ-hydroxyalkenes was a key step. Analysis of the stereochemical outcome of the selenoetherification step was important for the overall synthetic strategy. We therefore analysed the stereochemical outcome of the selenoetherification of an (*E*)-γ-hydroxyalkene and established the structure of the major selenoetherification product by X-ray crystallography. The major product obtained from selenoetherification of (*E*)-5-naphthylpent-4-en-1-ol was the *trans*-selenide, (I), arising from 6-*endo* cyclization of the transition state. The X-ray structure (Fig. 1 and Table 1) clearly shows that the tetrahydrofuran ring adopts a chair conformation, with the phenylselenyl and naphthyl substituents both occupying equatorial positions.



(I)

The selenide (I) also has a π interaction between the phenyl group and the naphthyl group. These aromatic rings are approximately parallel, with the angle between the planes being 18.8 (1)° and the average separation 3.6 Å. There are no significant intermolecular interactions.

Experimental

Phenylselenenyl chloride (180 mg, 0.94 mmol) was added to a cooled solution (195 K) of *trans*-5-naphthylpent-4-en-1-ol, (2) (100 mg, 0.47 mmol), in dichloromethane (3 ml) under nitrogen. After 4 h, the reaction was diluted using dichloromethane (7 ml) and sequentially washed with two portions of saturated sodium bicarbonate (2 × 7 ml). The aqueous fraction was then further extracted using three

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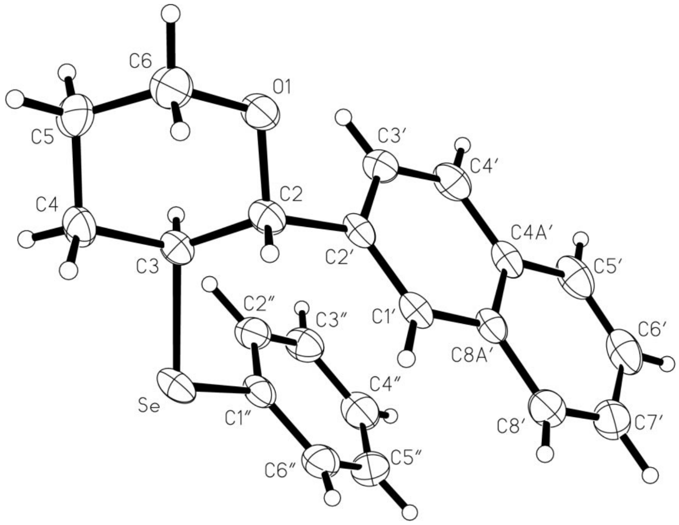

Figure 1
The structure of (I), showing 50% probability displacement ellipsoids (Siemens, 1995).

portions of dichloromethane (3 × 10 ml). The organic fractions were combined, washed with brine and dried over $MgSO_4$. The organic fraction was reduced under vacuum to yield a white solid. Recrystallization of the crude product from ethyl acetate–hexane (1:19) afforded the title selenide, (I) (148 mg, 86%), as opaque prisms. M.p. 354–355 K. A prism was cut to give a suitably sized sample for the diffraction analysis Analysis, found: M^+ = 368.0639. $C_{21}H_{20}OSe$ requires M = 368.0679; ν_{max} (film)/cm^{-1}: 1647, 1098, 1072, 1022, 816, 741, 692, 668; δ_H (300 MHz, $CDCl_3$, p.p.m.): 1.62–1.67 (1H, *m*, H-5_A), 1.70–1.84 (2H, *m*, H-4_A, H-5_B), 2.22–2.27 (1H, *m*, H-4_B), 3.42 (1H, *ddd*, $J_{2ax,3ax}$ = 11.0, $J_{3ax,4ax}$ = 11.0, $J_{3ax,4eq}$ = 3.9 MHz, H-3_{ax}), 3.57 (1H, *ddd*, $J_{6ax,6eq}$ = 11.6, $J_{6ax,5ax}$ = 11.6, $J_{6ax,5eq}$ = 2.2 Hz, H-6_{ax}), 4.03–4.09 (1H, *m*, H-6_{eq}), 4.38 (1H, *d*, $J_{2ax,3ax}$ = 10.4 Hz, H-2_{ax}), 6.90–7.70 (12H, *m*, ArH). δC (300 MHz, p.p.m.): 27.9 (CH_2, C-4), 32.6 (CH_2, C-5), 46.1 (CH, C-3), 68.7 (CH_2, C-6), 85.7 (CH, C-2), 125.0 (CH, Ar), 125.7 (CH, Ar), 127.0 (CH, Ar), 127.4 (CH, Ar), 127.5 (CH, Ar), 127.9 (CH, Ar), 128.0 (CH, Ar), 128.4 (CH, Ar), 133.0 (quat., Ar), 133.3 (quat., Ar), 135.5 (CH, Ar), 137.5 (quat., Ar).

Crystal data

$C_{21}H_{20}OSe$
M_r = 367.33
Orthorhombic, $P2_12_12_1$
a = 8.1766 (2) Å
b = 8.8854 (2) Å
c = 23.0888 (3) Å
V = 1677.46 (4) Å^3
Z = 4
D_x = 1.455 Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 8192 reflections
θ = 2–26°
μ = 2.24 mm^{-1}
T = 150 (2) K
Irregular fragment, colourless
0.42 × 0.36 × 0.18 mm

Data collection

Siemens SMART diffractometer
ω scans
Absorption correction: multi-scan (Blessing, 1995)
T_{min} = 0.439, T_{max} = 0.667
10162 measured reflections
3416 independent reflections

3227 reflections with $I > 2\sigma(I)$
R_{int} = 0.035
θ_{max} = 26.4°
$h = -10 \rightarrow 10$
$k = 0 \rightarrow 11$
$l = 0 \rightarrow 28$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.026
$wR(F^2)$ = 0.065
S = 1.00
3416 reflections
212 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0366P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.009
$\Delta\rho_{max}$ = 0.48 e Å^{-3}
$\Delta\rho_{min}$ = −0.48 e Å^{-3}
Absolute structure: Flack (1983), 1433 Friedel pairs
Flack parameter = 0.013 (8)

Table 1
Selected geometric parameters (Å, °).

Se—C1″	1.928 (2)	C2—C3	1.535 (3)
Se—C3	1.978 (2)	C3—C4	1.535 (3)
O1—C2	1.440 (3)	C4—C5	1.529 (4)
O1—C6	1.436 (3)	C5—C6	1.504 (4)
C2—C2′	1.516 (3)		
C1″—Se—C3	103.81 (10)	C1″—Se—C3	103.81 (10)
C2—O1—C6	111.39 (18)	C2—O1—C6	111.39 (18)
O1—C2—C2′	107.06 (17)	O1—C2—C2′	107.06 (17)
O1—C2—C3	109.40 (18)	O1—C2—C3	109.40 (18)
C2′—C2—C3	112.84 (17)	C2′—C2—C3	112.84 (17)
C2—C3—C4	110.40 (19)	C2—C3—C4	110.40 (19)
C2—C3—Se	111.73 (14)	C2—C3—Se	111.73 (14)
C4—C3—Se	105.73 (16)	C4—C3—Se	105.73 (16)
C5—C4—C3	110.2 (2)	C5—C4—C3	110.2 (2)
C6—C5—C4	110.0 (2)	C6—C5—C4	110.0 (2)
O1—C6—C5	111.9 (2)	O1—C6—C5	111.9 (2)

H atoms were clearly resolved in difference maps. They have been refined using a riding model, with U_{iso} values constrained to be 1.2 times U_{eq} of the carrier atom.

Data collection: *SMART* (Siemens, 1995); cell refinement: *SMART*; data reduction: *SAINT* (Siemens, 1995); program(s) used to solve structure: *SHELXS* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Siemens, 1995); software used to prepare material for publication: *SHELXL*97.

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