Crystal structure of *N*1-[2-(phenylseleninyl)cyclohexyl]benzamide, $C_{19}H_{21}NO_2Se$

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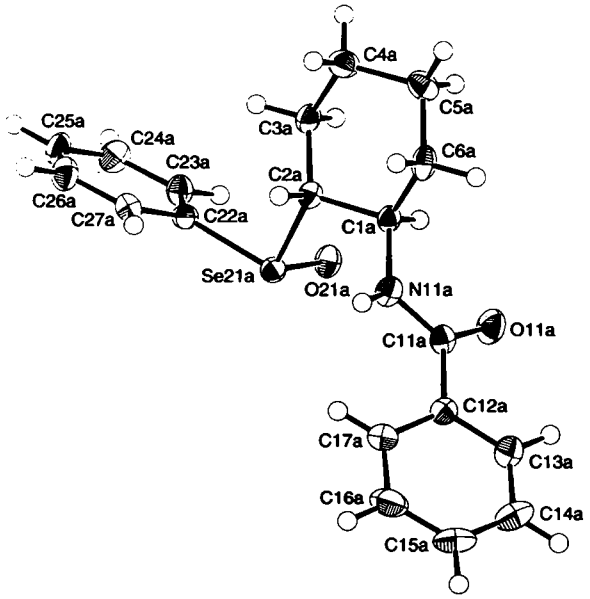

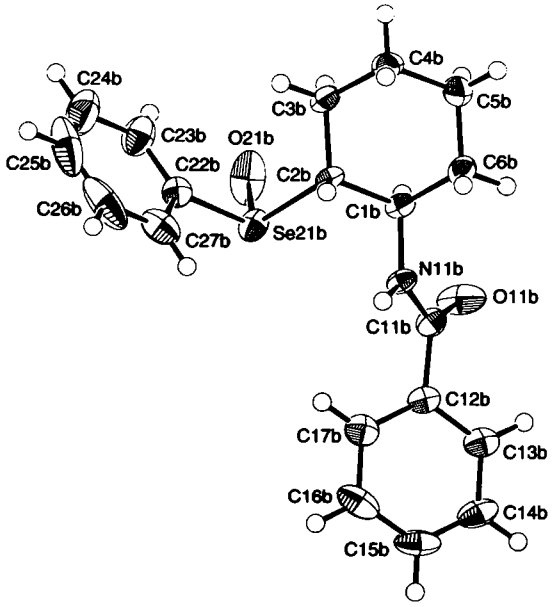

Abstract

$C_{19}H_{21}NO_2Se$, monoclinic, $P12_1/c1$ (No. 14), $a = 9.972(1)$ Å, $b = 29.548(3)$ Å, $c = 11.830(2)$ Å, $\beta = 105.25(1)°$, $V = 3363.0$ Å^3, $Z = 8$, $R_{gt}(F) = 0.048$, $wR_{ref}(F^2) = 0.127$, $T = 173$ K.

Source of material

β-Benzamidocyclohexyl phenyl selenide, prepared according to [1,2] from cyclohexene, was oxidised with *meta*-chloroperbenzoic acid (1.1 equiv.) in dichloromethane at RT for 1 h. The mixture was further diluted with dichloromethane, washed with 10 % aqueous sodium hydroxide and saturated aqueous sodium chloride. The organic layer was dried over $MgSO_4$ and concentrated in vacuo to give the title compound as a white solid in 96 % yield. Recrystallisation by vapour diffusion of ethyl acetate into a methanol solution of the compound gave colourless needles, mp 410 K – 411.5 K. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.17 (br s, 1H, NH), 7.90 (d, J 6.6 Hz, 2H, Ar), 7.54 – 7.59 (m, 2H, Ar), 7.43 – 7.54 (m, 6H, Ar), 3.53 – 3.66 (m, 2H, CHN, CHSe) 2.25 (d, J 12.3 Hz, 1H), 1.74 – 1.81 (m, 4H), 1.43 – 1.54 (m, 1H), 1.31 (t, J 10.5 Hz, 2H). Upon standing in deuterochloroform solution, inversion at selenium occurred to give an equilibrium mixture of four stereoisomers; one enantiomeric pair, as characterised in the present crystal- lographic study, and the other pair, with inversion at selenium, exhibited the following diagnostic resonances in the ^{1}H NMR spectrum: δ 4.06 (dt, δ 4.5, 11.7 Hz, 1H, CHN) and 3.07 (dt, 3.0, 11.7 Hz, CHSe).

Experimental details

The C-bound H atoms were placed in their geometrically calculated positions and included in the final refinement in the riding model approximation. The N-bound H atoms were located from a difference map calculated towards the end of the refinement.

Discussion

Two independent molecules comprise the crystallographic asymmetric unit. The major difference between the molecules is found in the relative orientation of the phenylseleninyl and amido groups with respect to the cyclohexyl ring. Thus, there is a twist of about 13.5° about the Se21—C22 bond as seen in the magnitudes of the C2–Se21–C22–C23 torsion angles of 112.4(4)° and 98.7(4)° for molecules a and b, respectively. A greater twist is noted for C11—C12 bond: the respective N11–C11–C12–C13 torsion angles are −162.2(4)° and −140.0(5)°. Each cyclohexyl ring adopts a chair conformation with the non-H substituents occupying equatorial positions in each case. The chirality at each of the C1 and C2 centres in the figure is *R*; the unit cell contains equal amounts of both enantiomers. Hydrogen bonding interactions involving the amido-H and the seleninyl-O atoms stabilise the crystal structure. Thus, N11a—H11a is 1.91 Å from O21b with *d*(H11a···O21b) of 2.788(6) Å and an angle of 159° subtended at H11a; symmetry operation: $-x, -y, -z$. Similarly, N11b—H11b is 1.99 Å from O21a with *d*(H11b···O21a) 2.859(5) Å and angle 159° at H11b; symmetry operation: $-1-x, -y, -z$. The hydrogen bonding described links molecules into chains aligned along the *a*-axis.

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Table 1. Data collection and handling.

Crystal:	colourless block , size 0.24 x 0.37 x 0.52 mm
Wavelength:	Mo K_α radiation (0.71069 Å)
μ:	22.43 cm^{-1}
Diffractometer, scan mode:	Rigaku AFC7R, ω/2θ
$2\theta_{max}$:	55°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	8318, 7721
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 4200
$N(param)_{refined}$:	416
Programs:	DIRDIF92 [3], SHELXL-97 [4], teXsan [5], DIFABS [6], PLATON [7]

Table 2. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	x	y	z	U_{iso}
H(1B)	4e	-0.4464	0.1918	0.0296	0.040(2)
H(1A)	4e	-0.0635	-0.1607	0.0499	0.040
H(2A)	4e	0.1366	-0.1556	-0.0859	0.040
H(2B)	4e	-0.6953	0.1377	-0.0305	0.040
H(3A)	4e	-0.0101	-0.2031	-0.2238	0.040
H(3B)	4e	-0.1186	-0.2061	-0.1453	0.040
H(3C)	4e	-0.5347	0.1653	-0.1868	0.040
H(3D)	4e	-0.6703	0.1338	-0.2217	0.040
H(4C)	4e	-0.8105	0.1941	-0.1890	0.040
H(4D)	4e	-0.7275	0.2101	-0.2810	0.040
H(4A)	4e	0.1584	-0.2422	-0.0806	0.040
H(4B)	4e	0.0193	-0.2719	-0.1144	0.040
H(5A)	4e	-0.0396	-0.2482	0.0572	0.040
H(5B)	4e	0.1138	-0.2694	0.0936	0.040
H(5C)	4e	-0.7166	0.2667	-0.1347	0.040
H(5D)	4e	-0.5639	0.2491	-0.1323	0.040
H(6C)	4e	-0.5844	0.2521	0.0608	0.040
H(6D)	4e	-0.7209	0.2211	0.0267	0.040
H(6A)	4e	0.2138	-0.1967	0.1136	0.040
H(6B)	4e	0.1067	-0.1995	0.1933	0.040
H(13A)	4e	0.0001	-0.0561	0.3671	0.032(2)
H(13B)	4e	-0.3459	0.2203	0.4565	0.032
H(14B)	4e	-0.3317	0.1911	0.6417	0.032
H(14A)	4e	0.1135	0.0050	0.4754	0.032
H(15B)	4e	-0.3763	0.1161	0.6644	0.032
H(15A)	4e	0.2874	0.0427	0.4170	0.032
H(16B)	4e	-0.4386	0.0682	0.5016	0.032
H(16A)	4e	0.3402	0.0226	0.2439	0.032
H(17A)	4e	0.2298	-0.0394	0.1345	0.032
H(17B)	4e	-0.4646	0.0977	0.3127	0.032
H(23A)	4e	-0.2838	-0.1368	-0.3625	0.032
H(23B)	4e	-0.4980	0.0538	-0.2286	0.032
H(24B)	4e	-0.6408	0.0006	-0.3503	0.032
H(24A)	4e	-0.2934	-0.1515	-0.5586	0.032
H(25A)	4e	-0.0944	-0.1476	-0.6235	0.032
H(25B)	4e	-0.8129	-0.0347	-0.2867	0.032
H(26B)	4e	-0.8506	-0.0172	-0.1078	0.032
H(26A)	4e	0.1176	-0.1289	-0.4946	0.032
H(27B)	4e	-0.7113	0.0362	0.0133	0.032
H(27A)	4e	0.1301	-0.1135	-0.2985	0.032
H(11A)	4e	0.1799	-0.1071	0.1046	0.032
H(11B)	4e	-0.5917	0.1520	0.1761	0.032

Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Se(21A)	4e	-0.07142(5)	-0.10599(1)	-0.15579(4)	0.0251(2)	0.0234(2)	0.0206(2)	-0.0022(2)	0.0053(2)	-0.0014(2)
Se(21B)	4e	-0.47700(5)	0.09361(2)	0.00068(4)	0.0255(3)	0.0401(3)	0.0341(3)	0.0037(2)	0.0013(2)	-0.0086(2)
O(11A)	4e	-0.0752(3)	-0.1090(1)	0.1982(3)	0.036(2)	0.047(2)	0.039(2)	-0.012(2)	0.021(2)	-0.009(2)
O(11B)	4e	-0.3276(4)	0.2094(1)	0.2344(3)	0.042(2)	0.082(3)	0.034(2)	-0.037(2)	-0.003(2)	0.007(2)
O(21A)	4e	-0.2326(3)	-0.1168(1)	-0.1467(3)	0.024(2)	0.038(2)	0.026(2)	0.001(1)	0.008(1)	0.001(1)
O(21B)	4e	-0.3548(4)	0.1047(1)	-0.0673(4)	0.031(2)	0.057(3)	0.105(4)	-0.012(2)	0.037(2)	-0.026(2)
N(11A)	4e	0.0907(4)	-0.1162(1)	0.1014(3)	0.022(2)	0.035(2)	0.023(2)	-0.005(2)	0.005(2)	-0.005(2)
N(11B)	4e	-0.5239(4)	0.1699(1)	0.1603(3)	0.024(2)	0.036(2)	0.021(2)	-0.006(2)	0.008(2)	-0.002(2)
C(1B)	4e	-0.5405(5)	0.1847(2)	0.0400(4)	0.024(2)	0.035(3)	0.022(2)	-0.007(2)	0.007(2)	-0.002(2)
C(1A)	4e	0.0356(5)	-0.1584(1)	0.0471(3)	0.024(2)	0.027(2)	0.017(2)	-0.006(2)	0.005(2)	-0.003(2)
C(2A)	4e	0.0384(4)	-0.1594(1)	-0.0817(3)	0.019(2)	0.025(2)	0.018(2)	0.003(2)	0.003(2)	0.001(2)
C(2B)	4e	-0.6033(4)	0.1459(1)	-0.0437(4)	0.018(2)	0.034(3)	0.017(2)	-0.001(2)	0.004(2)	-0.002(2)
C(3A)	4e	-0.0189(5)	-0.2033(1)	-0.1424(4)	0.036(3)	0.031(3)	0.017(2)	0.002(2)	0.007(2)	-0.000(2)
C(3B)	4e	-0.6256(5)	0.1592(2)	-0.1708(4)	0.037(3)	0.037(3)	0.023(2)	-0.005(2)	0.011(2)	-0.005(2)
C(4B)	4e	-0.7170(5)	0.2011(2)	-0.1986(4)	0.044(3)	0.036(3)	0.025(3)	-0.008(2)	0.003(2)	0.002(2)
C(4A)	4e	0.0606(5)	-0.2435(2)	-0.0764(4)	0.042(3)	0.026(3)	0.029(3)	0.001(2)	0.005(2)	0.001(2)
C(5A)	4e	0.0572(5)	-0.2438(2)	0.0524(4)	0.034(3)	0.028(3)	0.031(3)	0.002(2)	0.003(2)	0.008(2)
C(5B)	4e	-0.6536(6)	0.2402(2)	-0.1176(4)	0.053(4)	0.031(3)	0.034(3)	-0.004(2)	0.002(3)	0.000(2)
C(6B)	4e	-0.6300(5)	0.2268(2)	0.0102(4)	0.036(3)	0.034(3)	0.030(3)	-0.001(2)	0.004(2)	-0.006(2)
C(6A)	4e	0.1142(5)	-0.1993(2)	0.1115(4)	0.030(3)	0.044(3)	0.016(2)	-0.001(2)	0.003(2)	0.002(2)
C(11A)	4e	0.0325(5)	-0.0954(2)	0.1781(4)	0.021(2)	0.030(3)	0.017(2)	-0.004(2)	0.000(2)	0.001(2)
C(11B)	4e	-0.4155(5)	0.1829(2)	0.2476(4)	0.025(3)	0.037(3)	0.029(3)	-0.004(2)	0.003(2)	-0.004(2)
C(12A)	4e	0.1053(5)	-0.0545(2)	0.2418(4)	0.025(3)	0.026(2)	0.024(2)	0.002(2)	0.006(2)	0.000(2)
C(12B)	4e	-0.4081(5)	0.1620(2)	0.3656(4)	0.018(2)	0.042(3)	0.027(3)	0.002(2)	0.006(2)	0.001(2)
C(13A)	4e	0.0707(5)	-0.0406(2)	0.3421(4)	0.033(3)	0.037(3)	0.031(3)	0.000(2)	0.012(2)	-0.001(2)
C(13B)	4e	-0.3672(5)	0.1894(2)	0.4648(4)	0.021(3)	0.050(3)	0.032(3)	0.007(2)	0.002(2)	0.003(2)
C(14B)	4e	-0.3573(5)	0.1719(2)	0.5750(4)	0.025(3)	0.070(4)	0.025(3)	0.005(3)	-0.003(2)	-0.003(3)
C(14A)	4e	0.1379(6)	-0.0043(2)	0.4065(5)	0.044(3)	0.040(3)	0.038(3)	0.004(3)	0.006(3)	-0.014(2)
C(15B)	4e	-0.3838(5)	0.1278(2)	0.5882(5)	0.032(3)	0.077(4)	0.030(3)	0.011(3)	0.004(2)	0.019(3)
C(15A)	4e	0.2394(5)	0.0184(2)	0.3713(5)	0.040(3)	0.026(3)	0.050(3)	0.000(2)	0.001(3)	-0.013(2)

Table 3. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(16B)	4*e*	–0.4220(6)	0.0994(2)	0.4914(5)	0.035(3)	0.049(4)	0.063(4)	0.005(3)	0.016(3)	0.025(3)
C(16A)	4*e*	0.2721(6)	0.0060(2)	0.2696(5)	0.047(4)	0.026(3)	0.060(4)	–0.003(2)	0.019(3)	0.000(2)
C(17A)	4*e*	0.2062(5)	–0.0307(2)	0.2041(4)	0.046(3)	0.025(3)	0.035(3)	–0.001(2)	0.014(2)	–0.001(2)
C(17B)	4*e*	–0.4360(5)	0.1168(2)	0.3793(5)	0.028(3)	0.042(3)	0.040(3)	0.005(2)	0.006(2)	0.003(2)
C(22B)	4*e*	–0.5946(5)	0.0502(2)	–0.0987(4)	0.023(3)	0.035(3)	0.035(3)	0.001(2)	0.002(2)	–0.001(2)
C(22A)	4*e*	–0.0763(4)	–0.1226(1)	–0.3148(4)	0.025(2)	0.021(2)	0.022(2)	0.001(2)	0.005(2)	–0.000(2)
C(23A)	4*e*	–0.2025(5)	–0.1347(2)	–0.3898(4)	0.026(3)	0.033(3)	0.027(2)	–0.002(2)	0.009(2)	0.001(2)
C(23B)	4*e*	–0.5712(6)	0.0396(2)	–0.2044(5)	0.039(3)	0.051(4)	0.051(4)	–0.001(3)	0.006(3)	–0.014(3)
C(24B)	4*e*	–0.6546(8)	0.0079(2)	–0.2759(6)	0.072(5)	0.059(4)	0.064(5)	0.007(4)	–0.005(4)	–0.026(3)
C(24A)	4*e*	–0.2074(5)	–0.1437(2)	–0.5053(4)	0.031(3)	0.039(3)	0.025(2)	0.000(2)	0.001(2)	–0.003(2)
C(25A)	4*e*	–0.0896(5)	–0.1414(2)	–0.5437(4)	0.048(3)	0.038(3)	0.019(2)	0.006(2)	0.012(2)	0.001(2)
C(25B)	4*e*	–0.7560(8)	–0.0127(2)	–0.2383(9)	0.063(5)	0.033(4)	0.131(8)	0.005(3)	–0.039(5)	–0.015(4)
C(26B)	4*e*	–0.7784(7)	–0.0024(2)	–0.1323(8)	0.045(4)	0.037(4)	0.131(7)	–0.017(3)	–0.011(5)	0.031(4)
C(26A)	4*e*	0.0361(5)	–0.1302(2)	–0.4674(4)	0.033(3)	0.040(3)	0.033(3)	0.004(2)	0.015(2)	0.004(2)
C(27B)	4*e*	–0.6969(6)	0.0291(2)	–0.0610(5)	0.042(3)	0.045(3)	0.060(4)	–0.002(3)	0.011(3)	0.016(3)
C(27A)	4*e*	0.0437(5)	–0.1209(1)	–0.3518(4)	0.025(3)	0.027(2)	0.027(2)	–0.000(2)	0.007(2)	0.005(2)

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