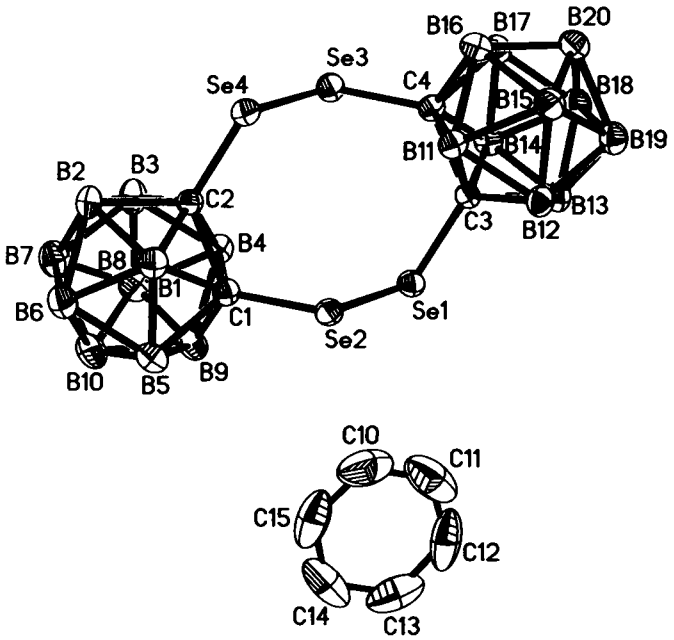
Z. Kristallogr. NCS 221 (2006) 419-420 / DOI 10.1524/ncrs.2006.0135

Crystal structure of 3,4,7,8-bis(1,2-dicarba-*closo*-dodecaborano[1,2])-1,2,5,6-tetraselenacyclooctane benzene solvate, $[(C_2B_{10}H_{10})Se_2]_2 \cdot C_6D_6$

Z. García-Hernández, B. Wrackmeyer*, M. Herberhold, T. Irrgang and R. Kempe*

Universität Bayreuth, Lehrstuhl für Anorganische Chemie II, 95440 Bayreuth, Germany

Received July 28, 2006, accepted and available on-line September 11, 2006; CCDC no. 1267/1859



Abstract

$C_{10}H_{26}B_{20}Se_4$, monoclinic, $P12_1/n1$ (no. 14), a = 10.3649(4) Å, b = 13.8582(7) Å, c = 18.3667(8) Å, β = 95.460(3)°, V = 2626.2 Å^3, Z = 4, $R_{gt}(F)$ = 0.040, $wR_{ref}(F^2)$ = 0.097, T = 193 K.

Source of material

In an NMR tube, tropylium bromide (0.15 g, 0.88 mmol) was added to a solution of 1,2-bis(trimethylsilylseleno)-1,2-dicarba-*closo*-dodecaborane(12) (0.1 g, 0.224 mmol) in 1.0 mL of deutered benzene, a yellow solution was observed and analyzed by NMR spectroscopy. After one month, the formation of yellow crystals was observed, the crystals were isolated and analyzed by X-ray diffraction (yield 0.012 g, 12 %).

Discussion

Recently, we described the molecular structures of cyclic compounds of tellurium [1] and selenium [2] containing two annellated [(dicarba-*closo*-dodecaborano)] units. The title compound is a new modification of the bis(*ortho*-carboran-1,2-diyl) selenium species containing two selena bridges. The molecule shows a nearly rectangular planar Se_4 arrangement (d(Se1—Se2) = 2.302 Å, d(Se3—Se4) = 2.300 Å, d(Se1···Se3) = 3.822 Å, d(Se2···Se4) = 3.703 Å; ∠Se2–Se1–Se3 = 88.44°, ∠Se1–Se2–Se4 = 91.51°, ∠Se2–Se4–Se3 = 91.44°, ∠Se4–Se3–Se1 = 88.59°). The mean deviation of this plane is 0.006 Å. The Se—C distances (av. 1.940 Å) and the C—C distances in the carborane units (av. 1.690 Å) are observed in the expected range. One uncoordinated benzene molecule was found.

Table 1. Data collection and handling.

Crystal:	light-yellow prism, size 0.40 × 0.45 × 0.80 mm
Wavelength:	Mo K_α radiation (0.71069 Å)
μ:	55.90 cm^{-1}
Diffractometer, scan mode:	Stoe IPDS II, ω
$2\theta_{max}$:	51.68°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	4965, 4965
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 3623
$N(param)_{refined}$:	307
Programs:	SIR97 [3], SHELXL-97 [4]

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

Atom	Site	x	y	z	U_{iso}
H(10)	4e	0.6767	0.1498	0.3749	0.096
H(11)	4e	0.8681	0.1593	0.3265	0.105
H(12)	4e	0.9515	0.3025	0.2963	0.091
H(13)	4e	0.8399	0.4449	0.3156	0.081
H(14)	4e	0.6404	0.4348	0.3693	0.084
H(15)	4e	0.5625	0.2834	0.3959	0.086
H(1)	4e	−0.3638	0.2920	0.5460	0.030
H(2)	4e	−0.4133	0.2468	0.6995	0.034
H(3)	4e	−0.1787	0.3035	0.7938	0.037
H(4)	4e	0.0111	0.3836	0.6982	0.031
H(5)	4e	−0.3408	0.5047	0.5350	0.034
H(6)	4e	−0.5278	0.4203	0.6277	0.037
H(7)	4e	−0.4100	0.4303	0.7821	0.042
H(8)	4e	−0.1464	0.5148	0.7824	0.039
H(9)	4e	−0.1066	0.5614	0.6293	0.035
H(10A)	4e	−0.3658	0.5881	0.6815	0.041
H(11A)	4e	−0.0179	0.1579	0.4940	0.026
H(12A)	4e	0.1743	0.2417	0.4024	0.032
H(13A)	4e	0.4038	0.3025	0.5015	0.032
H(14A)	4e	0.3482	0.2559	0.6532	0.029
H(15A)	4e	0.1508	0.0303	0.4133	0.033
H(16)	4e	0.1006	−0.0170	0.5652	0.032
H(17)	4e	0.3266	0.0416	0.6631	0.031
H(18)	4e	0.5195	0.1296	0.5743	0.034
H(19)	4e	0.4113	0.1187	0.4185	0.035
H(20)	4e	0.3643	−0.0404	0.5183	0.034

* Correspondence authors (e-mail: wrackmeyer@uni-bayreuth.de, kempe@uni-bayreuth.de)

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}
C(1)	4*e*	–0.1751(4)	0.3922(3)	0.5985(2)	0.022(2)	0.025(2)	0.020(2)	0.001(2)	0.002(2)	–0.000(2)
C(2)	4*e*	–0.2007(4)	0.3008(3)	0.6564(2)	0.024(2)	0.020(2)	0.019(2)	0.001(2)	0.003(2)	–0.000(2)
C(3)	4*e*	0.1898(4)	0.2446(3)	0.5400(2)	0.019(2)	0.020(2)	0.020(2)	0.002(2)	0.002(2)	–0.001(2)
C(4)	4*e*	0.1634(4)	0.1527(3)	0.5971(2)	0.024(2)	0.025(2)	0.014(2)	0.001(2)	–0.000(2)	0.000(2)
C(10)	4*e*	0.710(1)	0.2112(7)	0.3631(4)	0.117(8)	0.064(5)	0.056(5)	–0.005(5)	–0.004(5)	0.021(4)
C(11)	4*e*	0.822(1)	0.2169(7)	0.3345(5)	0.116(8)	0.077(6)	0.063(5)	0.051(6)	–0.027(5)	–0.018(5)
C(12)	4*e*	0.8710(7)	0.3004(9)	0.3169(4)	0.044(4)	0.16(1)	0.028(3)	0.005(5)	0.004(3)	–0.018(5)
C(13)	4*e*	0.8059(9)	0.3842(6)	0.3284(4)	0.096(6)	0.065(5)	0.035(3)	–0.038(5)	–0.029(4)	0.012(3)
C(14)	4*e*	0.6878(8)	0.3783(6)	0.3596(4)	0.076(5)	0.078(5)	0.048(4)	0.044(5)	–0.035(4)	–0.035(4)
C(15)	4*e*	0.6430(7)	0.2893(8)	0.3755(4)	0.051(4)	0.132(8)	0.032(3)	–0.015(5)	0.008(3)	–0.005(4)
Se(1)	4*e*	0.12977(4)	0.37497(3)	0.55356(3)	0.0246(2)	0.0206(2)	0.0300(2)	–0.0009(2)	0.0047(2)	–0.0010(2)
Se(2)	4*e*	–0.08653(4)	0.37006(3)	0.51136(2)	0.0260(2)	0.0258(2)	0.0178(2)	0.0033(2)	0.0023(2)	0.0022(2)
Se(3)	4*e*	0.06850(5)	0.16730(4)	0.68281(2)	0.0303(3)	0.0336(3)	0.0172(2)	0.0064(2)	0.0029(2)	0.0038(2)
Se(4)	4*e*	–0.14614(4)	0.16999(3)	0.63752(3)	0.0272(3)	0.0221(2)	0.0289(2)	–0.0005(2)	0.0066(2)	0.0013(2)
B(1)	4*e*	–0.3240(5)	0.3374(4)	0.5934(3)	0.024(3)	0.030(3)	0.022(2)	0.001(2)	0.001(2)	0.000(2)
B(2)	4*e*	–0.3523(5)	0.3096(4)	0.6857(3)	0.025(3)	0.033(3)	0.027(3)	0.001(2)	0.009(2)	–0.001(2)
B(3)	4*e*	–0.2109(6)	0.3440(4)	0.7425(3)	0.031(3)	0.039(3)	0.023(3)	0.000(2)	0.007(2)	–0.009(2)
B(4)	4*e*	–0.0961(6)	0.3927(4)	0.6856(3)	0.030(3)	0.029(3)	0.020(2)	–0.002(2)	0.002(2)	–0.005(2)
B(5)	4*e*	–0.3086(5)	0.4649(4)	0.5866(3)	0.029(3)	0.026(3)	0.030(3)	0.005(2)	0.001(2)	–0.002(2)
B(6)	4*e*	–0.4208(6)	0.4141(4)	0.6425(3)	0.028(3)	0.034(3)	0.030(3)	0.004(2)	0.005(2)	–0.002(2)
B(7)	4*e*	–0.3498(6)	0.4196(5)	0.7352(3)	0.035(3)	0.042(3)	0.027(3)	0.004(3)	0.007(2)	–0.006(3)
B(8)	4*e*	–0.1915(6)	0.4708(4)	0.7355(3)	0.038(3)	0.032(3)	0.026(3)	0.002(2)	0.002(2)	–0.008(2)
B(9)	4*e*	–0.1677(5)	0.4989(4)	0.6436(3)	0.028(3)	0.028(3)	0.030(3)	0.003(2)	0.002(2)	–0.006(2)
B(10)	4*e*	–0.3232(6)	0.5148(4)	0.6745(3)	0.033(3)	0.031(3)	0.038(3)	0.006(2)	0.005(3)	–0.011(2)
B(11)	4*e*	0.0891(5)	0.1508(4)	0.5086(3)	0.026(3)	0.024(2)	0.016(2)	–0.002(2)	0.000(2)	–0.003(2)
B(12)	4*e*	0.2054(5)	0.2017(4)	0.4541(3)	0.029(3)	0.030(3)	0.021(2)	0.002(2)	0.005(2)	–0.002(2)
B(13)	4*e*	0.3438(5)	0.2384(4)	0.5139(3)	0.020(3)	0.034(3)	0.027(3)	0.002(2)	0.005(2)	0.000(2)
B(14)	4*e*	0.3117(5)	0.2102(4)	0.6053(3)	0.021(3)	0.030(3)	0.021(2)	0.001(2)	–0.004(2)	–0.001(2)
B(15)	4*e*	0.1911(5)	0.0749(4)	0.4610(3)	0.030(3)	0.027(3)	0.026(3)	0.000(2)	0.002(2)	–0.004(2)
B(16)	4*e*	0.1614(5)	0.0463(4)	0.5520(3)	0.029(3)	0.023(3)	0.026(3)	0.002(2)	–0.001(2)	0.000(2)
B(17)	4*e*	0.2972(5)	0.0816(4)	0.6111(3)	0.027(3)	0.027(3)	0.024(3)	0.005(2)	0.001(2)	0.001(2)
B(18)	4*e*	0.4129(5)	0.1344(4)	0.5577(3)	0.022(3)	0.036(3)	0.026(3)	0.005(2)	0.000(2)	–0.002(2)
B(19)	4*e*	0.3477(6)	0.1282(4)	0.4640(3)	0.028(3)	0.034(3)	0.024(3)	0.003(2)	0.004(2)	–0.002(2)
B(20)	4*e*	0.3194(5)	0.0323(4)	0.5242(3)	0.031(3)	0.026(3)	0.027(3)	0.005(2)	0.002(2)	–0.002(2)

Acknowledgment. We thank the Fonds der Chemischen Industrie for financial support.

References

1. Herberhold, M.; Milius, W.; Jin, G.-X.; Kremnitz, W.; Wrackmeyer, B.: Molecular Structures of some Tellurium Derivatives of 1,2-Dicarba-*closo*-dodecaborane(12). Z. Anorg. Allg. Chem. (2006) in press.
2. Wrackmeyer, B.; García-Hernández, Z.; Kempe, R.; Herberhold, M.: Novel 1,2-dicarba-*closo*-dodecaborane(12) Derivatives of Selenium. Eur. J. Inorg. Chem. (2006) in press.
3. Altomare, A.; Burla, M. C.; Camalli, M.; Cascarano, G. L.; Giacovazzo, C.; Guagliardi, A.; Moliterni, A. G. G.; Polidori, G.; Spagna, R.: SIR97: a new tool for crystal structure determination and refinement. J. Appl. Crystallogr. **32** (1999) 115-119.
4. Sheldrick, G. M.: SHELXL-97. Program for the Refinement of Crystal Structures. University of Göttingen, Germany 1997.