Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

1,2-Bis(2-bromobenzyl)diselane

Guoxiong Hua, Amy L. Fuller, Alexandra M. Z. Slawin* and J. Derek Woollins

Department of Chemistry, University of St Andrews, St Andrews KY16 9ST, Scotland
Correspondence e-mail: amzs@st-and.ac.uk

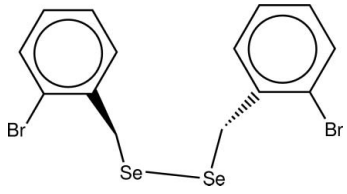
Received 12 September 2010; accepted 13 September 2010

Key indicators: single-crystal X-ray study; T = 125 K; mean σ(C–C) = 0.007 Å; R factor = 0.042; wR factor = 0.082; data-to-parameter ratio = 18.0.

In the title compound, $C_{14}H_{12}Br_2Se_2$, the Se—Se bond length [2.3034 (9) Å] is similar to those in diphenyl diselenide [2.3066 (7) and 2.3073 (10) Å] and shorter than that in 1,8-diselenonaphthalene [2.0879 (8)Å]. The molecule adopts a classical *gauche* conformation.

Related literature

Related structures are: diphenyl diselenide (Fuller *et al.*, 2010); di(2-bromomethyl)phenyldiselenide (Lari *et al.*, 2009) and 1,8 diseleno-naphthalene (Aucott *et al.*, 2004).



Experimental

Crystal data

$C_{14}H_{12}Br_2Se_2$
M_r = 497.98
Monoclinic, $P2_1/n$
a = 10.873 (3) Å
b = 9.002 (2) Å
c = 15.714 (4) Å
β = 106.102 (6)°
V = 1477.9 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 10.41 mm^{-1}
T = 125 K
0.15 × 0.09 × 0.09 mm

Data collection

Rigaku Saturn70 CCD diffractometer
Absorption correction: multi-scan *CrystalClear* (Rigaku Americas and Rigaku, 2009)
T_{min} = 0.153, T_{max} = 0.392
9089 measured reflections
3124 independent reflections
2671 reflections with $F^2 > 2\sigma(F^2)$
R_{int} = 0.058

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.082
S = 1.22
2939 reflections
163 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.76 e Å^{-3}
$\Delta\rho_{min}$ = −0.61 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Se1—C1	1.987 (5)	Se2—C8	1.986 (5)
Se1—Se2	2.3034 (8)		
C1—Se1—Se2—C8	88.8 (2)		

Data collection: *CrystalClear* (Rigaku Americas and Rigaku, 2009); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *CrystalStructure* (Rigaku Americas and Rigaku, 2010); software used to prepare material for publication: *CrystalStructure*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5357).

References

Aucott, S. M., Milton, H. L., Robertson, S. D., Slawin, A. M. Z. & Woollins, J. D. (2004). *Heteroat. Chem.* **15**, 531–542.
Fuller, A. L., Scott-Hayward, L. A. S., Li, Y., Bühl, M., Slawin, A. M. Z. & Woollins, J. D. (2010). *J. Am. Chem. Soc.* **132**, 5799–5802.
Lari, A., Rominger, F. & Gleiter, R. (2009). *Acta Cryst.* C**65**, o400–o403.
Rigaku Americas and Rigaku (2009). *CrystalClear*. Rigaku Americas, The Woodlands, Texas, USA, and Rigaku Corporation, Tokyo, Japan.
Rigaku Americas and Rigaku (2010). *CrystalStructure*. Rigaku Americas, The Woodlands, Texas, USA, and Rigaku Corporation, Tokyo, Japan.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

Acta Cryst. (2010). E**66**, o2579 [doi:10.1107/S1600536810036676]

1,2-Bis(2-bromobenzyl)diselane

Guoxiong Hua, Amy L. Fuller, Alexandra M. Z. Slawin and J. Derek Woollins

S1. Comment

We have recently reported (Fuller *et al.*, 2010) on the remarkable crystallization of PhSeSePh as one isomer. Here, the title compound (figure 1) is obtained as a mixture of both hands, the Se—Se bondlength, 2.3034 (9), is similar to that in diphenyldiselenide [2.3066 (7), 2.3073 (10) Å; Fuller *et al.*, 2010] and shorter than that in 1,8-diselenonaphthalene [2.0879 (8)Å; Aucott *et al.*, 2004).

S2. Experimental

To a solution of 2-bromobenzyl bromide (15.0 g, 60 mmol) in 150 ml of dry ethanol was added potassium selenocyanate (9.5 g, 65.0 mmol) at ambient temperature. The mixture was stirred for 2 h. Then an eqeous solution of NaOH (4.8 g, 120 mmol in 200 ml of water) was added to the mixture and was continued stirring for another 2 h. After extracted by dichloromethane (300 ml) and washed three times with water (100 mLx3), the organic layer was dried over $MgSO_4$ overnight. The organic residue was further purified by silica gel column (dichloromethane as eluent) to give a bright yellow solid (14.5 g) in 97% yield. Selected IR (KBr, cm^{-1}): 2985(w), 1563(*m*), 1469(*m*), 1436(*m*), 1413(*m*), 1334(*m*), 1170(*m*), 1022(*s*), 755(*versus*), 718(*m*), 657(*m*), 589(*m*). ^{1}H NMR (CD_2Cl_2, d), 7.54 (dd, *J*(H,H) = 8.0 Hz, 2H, ArH), 7.29–7.09 (m, 6H, ArH), 4.00 (s, 4H, CH_2) p.p.m.. ^{13}C NMR (CD_2Cl_2, d), 138.6, 133.1, 131.0, 128.9, 127.5, 124.3, 33.4 p.p.m.. ^{77}Se NMR (CD_2Cl_2, d), 398.6 p.p.m.. MS (EI^+, m/*z*), 498 $[M]^+$. Accurate mass measurement [EI^+, m/*z*]: 489.7664 $[M]^+$, calculated mass for $C_{14}H_{12}Br_2{}^{76}Se_2$: 489.7685. Anal. Calcd. for $C_{14}H_{12}Br_2Se_2$: C, 33.78; H, 2.43. Found: C, 33.92; H, 2.62.

S3. Refinement

All H atoms were included in calculated positions and refined as riding atoms with U\ĩso\~(H) = 1.5 U\~eq\~. The highest peak in the difference map is 1.12 Å from atom Se2.

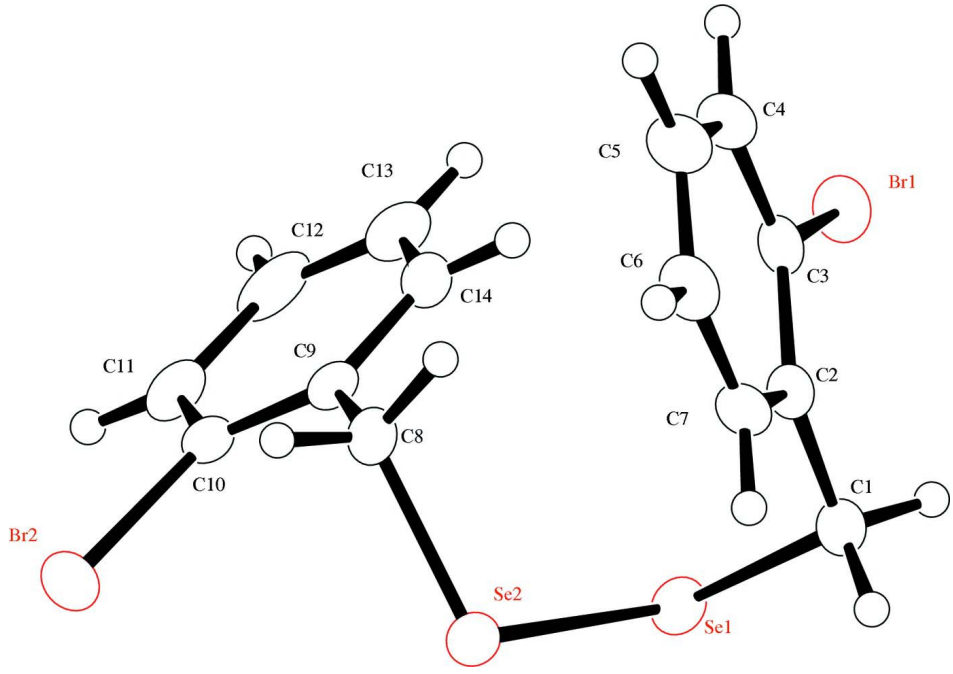


Figure 1

Anisotropic displacement ellipsoid plot of the title compound.

1,2-Bis(2-bromobenzyl)diselane

Crystal data

$C_{14}H_{12}Br_2Se_2$
$M_r = 497.98$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 10.873\ (3)$ Å
$b = 9.002\ (2)$ Å
$c = 15.714\ (4)$ Å
$\beta = 106.102\ (6)°$
$V = 1477.9\ (6)$ Å^3
$Z = 4$
$F(000) = 936.00$
$D_x = 2.238$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71075$ Å
Cell parameters from 4976 reflections
$\theta = 2.0–26.4°$
$\mu = 10.41$ mm^{-1}
$T = 125$ K
Prism, colorless
0.15 × 0.09 × 0.09 mm

Data collection

Rigaku Saturn70 CCD diffractometer
Detector resolution: 14.629 pixels mm^{-1}
ω scans
Absorption correction: multi-scan *CrystalClear* (Rigaku Americas and Rigaku, 2009)
$T_{min} = 0.153$, $T_{max} = 0.392$
9089 measured reflections
3124 independent reflections
2671 reflections with $F^2 > 2\sigma(F^2)$
$R_{int} = 0.058$
$\theta_{max} = 26.4°$
$h = -9\rightarrow13$
$k = -11\rightarrow9$
$l = -16\rightarrow19$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.082$
$S = 1.22$
2939 reflections
163 parameters
0 restraints
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map	$w = 1/[\sigma^2(F_o^2) + (0.0253P)^2 + 2.0566P]$ where $P = (F_o^2 + 2F_c^2)/3$
Hydrogen site location: inferred from neighbouring sites	$(\Delta/\sigma)_{max} = 0.001$
H-atom parameters constrained	$\Delta\rho_{max} = 0.76$ e Å^{-3}
	$\Delta\rho_{min} = -0.61$ e Å^{-3}

Special details

Geometry. ENTER SPECIAL DETAILS OF THE MOLECULAR GEOMETRY

Refinement. Refinement was performed using all reflections. The weighted *R*-factor (*wR*) and goodness of fit (*S*) are based on F^2. *R*-factor (gt) are based on *F*. The threshold expression of $F^2 > 2.0\ \sigma(F^2)$ is used only for calculating *R*-factor (gt).

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Br(1)	0.94202 (5)	0.82306 (5)	0.39444 (3)	0.02997 (15)
Br(2)	0.24435 (5)	0.89337 (5)	0.36824 (4)	0.03177 (15)
Se(1)	0.63257 (5)	0.62609 (5)	0.28455 (3)	0.02562 (14)
Se(2)	0.48881 (5)	0.58891 (5)	0.36604 (3)	0.02668 (14)
C(1)	0.7876 (5)	0.5176 (5)	0.3532 (3)	0.0245 (10)
C(2)	0.8288 (5)	0.5639 (5)	0.4477 (3)	0.0213 (10)
C(3)	0.8944 (5)	0.6951 (5)	0.4770 (3)	0.0222 (10)
C(4)	0.9269 (5)	0.7397 (5)	0.5647 (3)	0.0275 (11)
C(5)	0.8920 (5)	0.6504 (6)	0.6263 (3)	0.0313 (11)
C(6)	0.8282 (5)	0.5183 (5)	0.5996 (3)	0.0279 (11)
C(7)	0.7967 (5)	0.4760 (5)	0.5117 (3)	0.0241 (10)
C(8)	0.5260 (5)	0.7583 (5)	0.4503 (3)	0.0247 (10)
C(9)	0.5175 (5)	0.9065 (5)	0.4057 (3)	0.0223 (10)
C(10)	0.4031 (5)	0.9784 (5)	0.3655 (3)	0.0217 (10)
C(11)	0.3992 (6)	1.1154 (5)	0.3247 (3)	0.0289 (12)
C(12)	0.5135 (6)	1.1812 (5)	0.3217 (3)	0.0359 (14)
C(13)	0.6279 (6)	1.1126 (5)	0.3605 (4)	0.0364 (14)
C(14)	0.6297 (5)	0.9771 (5)	0.4025 (3)	0.0298 (11)
H(1a)	0.7695	0.4097	0.3499	0.029*
H(1b)	0.8582	0.5359	0.3261	0.029*
H(4)	0.9722	0.8299	0.5825	0.033*
H(5)	0.9119	0.6802	0.6866	0.038*
H(6)	0.8059	0.4562	0.6420	0.033*
H(7)	0.7523	0.3852	0.4943	0.029*
H(8a)	0.4648	0.7561	0.4866	0.030*
H(8b)	0.6132	0.7460	0.4907	0.030*
H(11)	0.3198	1.1635	0.2991	0.035*
H(12)	0.5123	1.2740	0.2928	0.043*
H(13)	0.7059	1.1581	0.3585	0.044*
H(14)	0.7096	0.9312	0.4297	0.036*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br(1)	0.0302 (3)	0.0269 (3)	0.0381 (3)	−0.0038 (2)	0.0184 (3)	0.0042 (2)
Br(2)	0.0225 (3)	0.0318 (3)	0.0397 (3)	−0.0011 (2)	0.0065 (2)	−0.0027 (2)
Se(1)	0.0286 (3)	0.0285 (3)	0.0192 (2)	0.0043 (2)	0.0057 (2)	−0.00085 (18)
Se(2)	0.0256 (3)	0.0213 (2)	0.0342 (3)	−0.00192 (19)	0.0101 (2)	−0.00485 (19)
C(1)	0.022 (3)	0.022 (2)	0.030 (3)	0.003 (2)	0.007 (2)	0.0001 (19)
C(2)	0.019 (3)	0.020 (2)	0.026 (2)	0.0021 (19)	0.008 (2)	0.0020 (18)
C(3)	0.018 (3)	0.022 (2)	0.029 (3)	0.0021 (19)	0.010 (2)	0.0044 (19)
C(4)	0.023 (3)	0.030 (2)	0.029 (3)	−0.004 (2)	0.007 (2)	0.000 (2)
C(5)	0.028 (3)	0.041 (3)	0.021 (3)	0.000 (2)	0.000 (2)	0.001 (2)
C(6)	0.026 (3)	0.030 (2)	0.028 (3)	0.002 (2)	0.007 (2)	0.010 (2)
C(7)	0.020 (3)	0.020 (2)	0.031 (3)	−0.0019 (19)	0.004 (2)	0.0018 (19)
C(8)	0.023 (3)	0.028 (2)	0.022 (2)	0.006 (2)	0.005 (2)	−0.0009 (19)
C(9)	0.026 (3)	0.021 (2)	0.022 (2)	−0.0023 (19)	0.010 (2)	−0.0063 (18)
C(10)	0.025 (3)	0.022 (2)	0.020 (2)	−0.0031 (19)	0.008 (2)	−0.0052 (18)
C(11)	0.040 (3)	0.023 (2)	0.025 (3)	0.004 (2)	0.011 (2)	−0.0035 (19)
C(12)	0.065 (4)	0.020 (2)	0.032 (3)	−0.004 (3)	0.029 (3)	−0.005 (2)
C(13)	0.045 (4)	0.029 (3)	0.045 (3)	−0.015 (3)	0.028 (3)	−0.015 (2)
C(14)	0.025 (3)	0.032 (3)	0.035 (3)	−0.002 (2)	0.013 (2)	−0.011 (2)

Geometric parameters (Å, º)

Br1—C3	1.911 (4)	C6—H6	0.9500
Br2—C10	1.899 (5)	C7—H7	0.9500
Se1—C1	1.987 (5)	C8—C9	1.498 (6)
Se1—Se2	2.3034 (8)	C8—H8A	0.9900
Se2—C8	1.986 (5)	C8—H8B	0.9900
C1—C2	1.487 (6)	C9—C14	1.389 (7)
C1—H1A	0.9900	C9—C10	1.389 (7)
C1—H1B	0.9900	C10—C11	1.385 (6)
C2—C3	1.391 (6)	C11—C12	1.389 (8)
C2—C7	1.398 (6)	C11—H11	0.9500
C3—C4	1.384 (7)	C12—C13	1.371 (8)
C4—C5	1.389 (7)	C12—H12	0.9500
C4—H4	0.9500	C13—C14	1.385 (7)
C5—C6	1.383 (7)	C13—H13	0.9500
C5—H5	0.9500	C14—H14	0.9500
C6—C7	1.382 (7)		
C1—Se1—Se2	103.36 (13)	C2—C7—H7	119.3
C8—Se2—Se1	102.41 (14)	C9—C8—Se2	113.4 (3)
C2—C1—Se1	112.3 (3)	C9—C8—H8A	108.9
C2—C1—H1A	109.1	Se2—C8—H8A	108.9
Se1—C1—H1A	109.1	C9—C8—H8B	108.9
C2—C1—H1B	109.1	Se2—C8—H8B	108.9
Se1—C1—H1B	109.1	H8A—C8—H8B	107.7

H1A—C1—H1B	107.9	C14—C9—C10	117.1 (4)
C3—C2—C7	116.7 (4)	C14—C9—C8	118.9 (5)
C3—C2—C1	123.6 (4)	C10—C9—C8	124.0 (4)
C7—C2—C1	119.6 (4)	C11—C10—C9	122.2 (5)
C4—C3—C2	122.8 (4)	C11—C10—Br2	117.3 (4)
C4—C3—Br1	117.3 (3)	C9—C10—Br2	120.5 (3)
C2—C3—Br1	119.8 (3)	C10—C11—C12	118.9 (5)
C3—C4—C5	118.8 (5)	C10—C11—H11	120.6
C3—C4—H4	120.6	C12—C11—H11	120.6
C5—C4—H4	120.6	C13—C12—C11	120.2 (5)
C6—C5—C4	119.9 (5)	C13—C12—H12	119.9
C6—C5—H5	120.1	C11—C12—H12	119.9
C4—C5—H5	120.1	C12—C13—C14	119.9 (5)
C7—C6—C5	120.3 (4)	C12—C13—H13	120.0
C7—C6—H6	119.8	C14—C13—H13	120.0
C5—C6—H6	119.8	C13—C14—C9	121.6 (5)
C6—C7—C2	121.4 (4)	C13—C14—H14	119.2
C6—C7—H7	119.3	C9—C14—H14	119.2

C1—Se1—Se2—C8	88.8 (2)	Se1—Se2—C8—C9	55.2 (4)
Se2—Se1—C1—C2	−53.2 (3)	Se2—C8—C9—C14	−101.0 (4)
Se1—C1—C2—C3	−77.5 (5)	Se2—C8—C9—C10	78.0 (5)
Se1—C1—C2—C7	99.9 (4)	C14—C9—C10—C11	−0.8 (6)
C7—C2—C3—C4	−0.5 (7)	C8—C9—C10—C11	−179.8 (4)
C1—C2—C3—C4	176.9 (4)	C14—C9—C10—Br2	−178.7 (3)
C7—C2—C3—Br1	−179.8 (3)	C8—C9—C10—Br2	2.2 (6)
C1—C2—C3—Br1	−2.4 (6)	C9—C10—C11—C12	1.7 (7)
C2—C3—C4—C5	−0.3 (7)	Br2—C10—C11—C12	179.8 (3)
Br1—C3—C4—C5	179.0 (4)	C10—C11—C12—C13	−1.4 (7)
C3—C4—C5—C6	1.2 (8)	C11—C12—C13—C14	0.2 (7)
C4—C5—C6—C7	−1.3 (8)	C12—C13—C14—C9	0.8 (7)
C5—C6—C7—C2	0.5 (8)	C10—C9—C14—C13	−0.5 (7)
C3—C2—C7—C6	0.4 (7)	C8—C9—C14—C13	178.6 (4)
C1—C2—C7—C6	−177.1 (4)		