Bis(4-acetylphenyl) selenide
Hazem Bouraoui, Ali Boudjada, Sofiane Bouacida, Youcef Mechehoud, Jean Meinnel

To cite this version:
Hazem Bouraoui, Ali Boudjada, Sofiane Bouacida, Youcef Mechehoud, Jean Meinnel. Bis(4-acetylphenyl) selenide. Acta Crystallographica Section E: Structure Reports Online, International Union of Crystallography, 2011, 67 (4), pp.o941. <10.1107/S1600536811009962>. <hal-00615275>
Bis(4-acetylphenyl) selenide

Hazem Bouraoui,a Ali Boujdja,a Sofiane Bouacida,b‡ Youcef Mechehoudc and Jean Meinneld

*aLaboratoire de Cristallographie, Département de Physique, Université Mentouri-Constantine, 25000 Constantine, Algeria, bUnité de Recherche de Chimie de l’Environnement et Moléculaire Structurale, CHEMS, Université Mentouri-Constantine, 25000 Constantine, Algeria, and cUMR 6226 CNRS–Université Rennes 1 ‘Sciences Chimiques de Rennes’, Equipe ‘Matière Condensée et Systèmes Electroactifs’, 263 Avenue du Général Leclerc, F-35042 Rennes, France
Correspondence e-mail: bouacida_sofiane@yahoo.fr

Received 4 March 2011; accepted 16 March 2011

Key indicators: single-crystal X-ray study; T = 295 K; mean so(C—C) = 0.005 Å; R factor = 0.041; wR factor = 0.112; data-to-parameter ratio = 18.8.

In the title compound, C18H16O2Se, the dihedral angle between the benzene rings is 87.08 (11)°. In the crystal, molecules are linked into layers parallel to the bc plane by intermolecular C—H···O hydrogen bonds.

Related literature

For the synthesis of the title compound, see: Henry (1943). For biological properties and applications of organoselenide compounds, see: Clement et al. (1997); Anderson et al. (1996); Abdel-Hafez (2008); Woods et al. (1993); Hellberg et al. (1997). For a description of the Cambridge Structural Database, see: Allen (2002).

Experimental

Crystal data

C18H16O2Se
M r = 317.23
Monoclinic, P2 1/c
a = 14.9290 (7) Å
b = 7.7223 (3) Å
c = 13.8345 (6) Å
V = 1433.60 (11) Å 3
Z = 4

Mo Kα radiation
µ = 2.61 mm −1
T = 295 K
0.14 × 0.07 × 0.05 mm

Data collection

Nonius KappaCCD diffractometer
6274 measured reflections
3272 independent reflections

Refinement

R[F 2 > 2σ(F 2)] = 0.041
wR(F 2) = 0.112
S = 1.04
2954 reflections with I > 2σ(I)
R min = 0.027
174 parameters

Hydrogen-bond geometry (Å, °).

C2—H2···O2i 0.93 2.47 3.272 (5) 145
C12—H12···O1ii 0.93 2.53 3.317 (4) 143

Table 1

Symmetry codes: (i) x, −y + 1/2, z − 1/2; (ii) x, y − 1, z.

Data collection: SCALEPACK (Otwinowski & Minor, 1997); data reduction: SCALEPACK and DENZO (Otwinowski & Minor, 1997); program(s) used to solve structure: SIR2002 (Burla et al., 2003); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: ORTEP-3 (Farrugia, 1997) and DIAMOND (Brandenburg & Berndt, 2001); software used to prepare material for publication: WinGX (Farrugia, 1999).

This work was supported by the Laboratoire de Cristallographie, Département de Physique, Université Mentouri-Constantine, Algeria and UMR 6226 CNRS–Université Rennes 1 ‘Sciences Chimiques de Rennes’, France.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RZ2569).

References

Abdel-Hafez, Sh. H. (2008). Eur. J. Med. Chem. 43, 1971–1977.
Allen, F. H. (2002). Acta Cryst. B58, 380–388.
Anderson, C. M., Hallberg, A. & Haegberg, T. (1996). Adv. Drug Res. 28, 65–180.
Brandenburg, K. & Berndt, M. (2001). DIAMOND. Crystal Impact, Bonn, Germany.
Burla, M. C., Calandra, R., Camalli, M., Carozzini, B., Cascarano, G. L., De Caro, L., Giacovazzo, C., Polidori, G. & Spagna, R. (2003). J. Appl. Cryst. 36, 381-388.
Clement, I., Lisk, D. J., Ganther, H. & Thompson, H. J. (1997). Anticancer Res. 17, 3195–3199.
Farrugia, L. J. (1997). J. Appl. Cryst. 30, 565.
Farrugia, L. J. (1999). J. Appl. Cryst. 32, 837–838.
Hellberg, J., Remonem, T. & Johansson, M. (1997). Synth. Meth. 84, 251–255.
Henry, M. L. (1943). Org. Synth. Coll. 2, 238–240.
Nonius (1998). KappaCCD Reference Manual. Nonius BV, Delft, The Netherlands.
Otwinowski, Z. & Minor, W. (1997). Methods in Enzymology, Vol. 276, Macromolecular Crystallography, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
Sheldrick, G. M. (2008). Acta Cryst. A64, 112–122.
Woods, J. A., Hadfield, J. A., McGown, A. T. & Fox, B. W. (1993). Bioorg. Med. Chem. 1, 333–340.
supplementary materials
Bis(4-acetylphenyl) selenide

H. Bouraoui, A. Boudjada, S. Bouacida, Y. Mechehoud and J. Meinnel

Comment

Organoselenides and derivatives are of considerable interest in academia as anti-cancer (Clement et al., 1997), anti-oxydant (Anderson et al., 1996), anti-inflammatory and antiallergic agents (Abdel-Hafez, 2008), and in industry because of their wide involvement as key intermediates for the synthesis of pharmaceuticals (Woods et al., 1993), perfumes, fine chemicals and polymers (Hellberg et al., 1997). In the framework of our ongoing program related to the synthesis and pharmaceutical evaluation of new organoselenide derivatives, we report here the synthesis and crystal structure of the title compound.

In the molecule of the title compound (Fig. 1), the selenium atom is linked to two phenyl acetyl groups. All bond distances and angles are within the ranges of accepted values (CSD; Allen, 2002). The molecule is not planar, as can be seen from the dihedral angle of 87.08 (11)° between the planes of the two benzene rings. In the crystal structure, molecules are linked into chains running parallel to the c axis by intermolecular C2—H2···O2 hydrogen interactions (Fig. 2, Table 1). The chains are further connected by C12—H12···O1 hydrogen bonds to form layers parallel to the bc plane (Fig. 3).

Experimental

The title compound was prepared according to a literature method (Henry, 1943). Methyl acyl chloride (2.4 mmol) and anhydrous aluminium chloride (3.0 mmol) were dissolved in dry methylene chloride (4 ml). The reaction mixture was cooled at 0–5 °C, protected from atmospheric moisture, and stirred continuously from 15 min. A solution of diphenyl selenide (1 mmol) in methylene chloride (0.5 ml) was then added dropwise over a period of 5 min. The reaction mixture was allowed to reach room temperature gradually and stirred at this temperature overnight. The solution was then washed with ice water-HCl and extracted with methylene chloride. The organic layer was separated and dried over Na2SO4. Removal of the solvent afforded the crude title product which was recrystallized from CH3OH. Some crystals suitable for X-ray diffraction analysis were carefully isolated under polarizing microscope.

Refinement

All H atoms were localized in a Fourier difference map and introduced in calculated positions as riding on their parent C atoms, with Caryl—H = 0.93 Å, Cmethyl—H = 0.96 Å, and with Uiso(H) = 1.5Ueq(Cmethyl) or Uiso(H) = 1.2Ueq(Caryl).

Figures

Fig. 1. The molecular structure of the title compound with displacement ellipsoids drawn at the 50% probability level.
supplementary materials

Fig. 2. Packing diagram of the title compound viewed down the $b$ axis showing the chains parallel to the $c$ axis formed by hydrogen bonds (dashed line).

Fig. 3. Crystal packing of the title compound viewed down the $a$ axis showing a layer parallel to the $bc$ plane. H hydrogen bonds are shown as dashed lines.

$1\{-4-[(4\text{-acetylphenylidene})\text{selanyl}]\text{phenyl}\}\text{ethanone}$

Crystal data

$C_{16}H_{14}O_2Se$  
$M_r = 317.23$  
$D_x = 1.47 \text{ Mg m}^{-3}$

Monoclinic, $P2_1/c$  
Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ Å}$

$a = 14.9290 (7) \text{ Å}$  
$b = 7.7223 (3) \text{ Å}$  
$c = 13.8345 (6) \text{ Å}$

$\beta = 115.993 (2)^\circ$  
$V = 1433.60 (11) \text{ Å}^3$  
$Z = 4$

Needle, white  
$0.14 \times 0.07 \times 0.05 \text{ mm}$

Data collection

Nonius KappaCCD diffractometer  
$h = -19\rightarrow19$  
$k = -9\rightarrow10$  
$l = -17\rightarrow17$

$6274$ measured reflections  
$3272$ independent reflections  
$1904$ reflections with $I > 2\sigma(I)$

$R_{int} = 0.027$  
$\theta_{max} = 27.5^\circ$, $\theta_{min} = 3.0^\circ$

Refinement

Refinement on $F^2$  
Least-squares matrix: full  
$R[F^2 > 2\sigma(F^2)] = 0.041$

$wR(F^2) = 0.112$  
$S = 1.04$

Primary atom site location: structure-invariant direct methods  
Secondary atom site location: difference Fourier map  
Hydrogen site location: inferred from neighbouring sites  
$H$-atom parameters constrained

$w = 1/\left[\sigma^2(F_o^2) + (0.0407P)^2 + 0.5483P\right]$
where \( P = (F_o^2 + 2F_c^2)/3 \)

\((\Delta/\sigma)_\text{max} < 0.001\)

\(\Delta\rho_{\text{max}} = 0.48 \text{ e Å}^{-3}\)

\(\Delta\rho_{\text{min}} = -0.59 \text{ e Å}^{-3}\)

**Special details**

**Geometry.** All e.s.d.’s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.’s are taken into account individually in the estimation of e.s.d.’s in distances, angles and torsion angles; correlations between e.s.d.’s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.’s is used for estimating e.s.d.’s involving l.s. planes.

**Refinement.** Refinement of \( F^2 \) against ALL reflections. The weighted \( R \)-factor \( wR \) and goodness of fit \( S \) are based on \( F^2 \), conventional \( R \)-factors \( R \) are based on \( F \), with \( F \) set to zero for negative \( F^2 \). The threshold expression of \( F^2 > \sigma(F^2) \) is used only for calculating \( R \)-factors(ggt) etc. and is not relevant to the choice of reflections for refinement. \( R \)-factors based on \( F^2 \) are statistically about twice as large as those based on \( F \), and \( R \)-factors based on ALL data will be even larger.

**Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å\(^2\))**

| C1  | 0.1305 (3) | 0.6107 (4) | 0.4328 (3) | 0.0693 (8) |
| C2  | 0.0389 (3) | 0.6402 (5) | 0.3498 (3) | 0.0807 (10) |
| H2  | 0.0275     | 0.6113     | 0.2801     | 0.097*      |
| C3  | −0.0369 (3)| 0.7122 (5) | 0.3678 (3) | 0.0758 (9)  |
| H3  | −0.0985    | 0.7327     | 0.3101     | 0.091*      |
| C4  | −0.0222 (2)| 0.7546 (4) | 0.4719 (2) | 0.0631 (8)  |
| C5  | 0.0705 (3) | 0.7225 (4) | 0.5546 (3) | 0.0699 (8)  |
| C6  | 0.0820     | 0.7490     | 0.6247     | 0.084*      |
| H7  |           |            |            |             |
| C8  | 0.1463 (3) | 0.6524 (4) | 0.5365 (3) | 0.0727 (9)  |
| C9  | 0.2083     | 0.6330     | 0.5939     | 0.087*      |
| C10 | 0.2962 (2) | 0.7077 (4) | 0.3840 (2) | 0.0610 (7)  |
| C11 | 0.2529 (2) | 0.8705 (4) | 0.3634 (2) | 0.0643 (8)  |
| C12 | 0.1896     | 0.8864     | 0.3594     | 0.077*      |
| C13 | 0.3038 (2) | 1.0081 (4) | 0.3489 (2) | 0.0614 (7)  |
| C14 | 0.2743     | 1.1170     | 0.3352     | 0.074*      |
| C15 | 0.3987 (2) | 0.9885 (4) | 0.3543 (2) | 0.0550 (7)  |
| C16 | 0.4414 (2) | 0.8249 (4) | 0.3750 (2) | 0.0618 (8)  |
| C17 | 0.5049     | 0.8091     | 0.3793     | 0.074*      |
| C18 | 0.3904 (2) | 0.6849 (4) | 0.3891 (3) | 0.0683 (8)  |
| C19 | 0.4194     | 0.5755     | 0.4021     | 0.082*      |
| C20 | −0.1026 (3)| 0.8262 (4) | 0.4962 (3) | 0.0715 (9)  |
| C21 | 0.4524 (2) | 1.1412 (4) | 0.3393 (2) | 0.0613 (8)  |
| C22 | −0.2035 (3)| 0.8594 (6) | 0.4069 (3) | 0.0998 (13) |
| C23 | −0.2445    | 0.9155     | 0.4350     | 0.150*      |
| C24 | −0.2335    | 0.7515     | 0.3743     | 0.150*      |
| C25 | −0.1974    | 0.9326     | 0.3540     | 0.150*      |
| C26 | 0.5559 (2) | 1.1204 (5) | 0.3517 (3) | 0.0722 (9)  |
| C27 | 0.5821     | 1.2315     | 0.3462     | 0.108*      |
supplementary materials

H16B  0.5557  1.0456  0.2962  0.108*
H16C  0.5968  1.0707  0.4208  0.108*
O1    0.41088 (19) 1.2823 (3) 0.3175 (2) 0.0912 (8)
O2    -0.08802 (19) 0.8541 (4) 0.5879 (2) 0.0940 (8)
Se1   0.23415 (3)  0.50420 (5) 0.40667 (4) 0.08634 (18)

Atomic displacement parameters (Å²)

|     | U¹¹ | U²² | U³³ | U¹² | U¹³ | U²³ |
|-----|-----|-----|-----|-----|-----|-----|
| C1  | 0.078 (2) | 0.068 (2) | 0.074 (2) | -0.0169 (17) | 0.0436 (19) | -0.0041 (16) |
| C2  | 0.092 (3) | 0.094 (3) | 0.064 (2) | -0.023 (2) | 0.041 (2) | -0.0082 (18) |
| C3  | 0.070 (2) | 0.095 (3) | 0.0593 (19) | -0.0153 (19) | 0.0254 (17) | -0.0016 (17) |
| C4  | 0.069 (2) | 0.0613 (18) | 0.0599 (19) | -0.0142 (15) | 0.0291 (17) | 0.0000 (14) |
| C5  | 0.076 (2) | 0.077 (2) | 0.0569 (18) | -0.0084 (18) | 0.0297 (17) | -0.0080 (16) |
| C6  | 0.071 (2) | 0.078 (2) | 0.070 (2) | -0.0039 (17) | 0.0309 (18) | -0.0003 (17) |
| C7  | 0.075 (2) | 0.0568 (17) | 0.0604 (17) | -0.0054 (15) | 0.0377 (15) | -0.0072 (14) |
| C8  | 0.0614 (19) | 0.068 (2) | 0.0678 (19) | 0.0008 (15) | 0.0326 (16) | 0.0010 (15) |
| C9  | 0.0596 (18) | 0.0568 (17) | 0.0649 (18) | 0.0086 (15) | 0.0245 (15) | 0.0081 (15) |
| C10 | 0.0651 (18) | 0.0518 (16) | 0.0498 (15) | 0.0013 (14) | 0.0268 (13) | -0.0029 (13) |
| C11 | 0.070 (2) | 0.0547 (17) | 0.0726 (19) | 0.0023 (15) | 0.0428 (17) | -0.0032 (14) |
| C12 | 0.088 (2) | 0.0492 (16) | 0.084 (2) | 0.0072 (15) | 0.0521 (19) | -0.0029 (15) |
| C13 | 0.074 (2) | 0.066 (2) | 0.073 (2) | -0.0098 (16) | 0.0311 (18) | -0.0018 (17) |
| C14 | 0.071 (2) | 0.0548 (18) | 0.0572 (17) | -0.0027 (15) | 0.0275 (16) | 0.0018 (14) |
| C15 | 0.073 (3) | 0.119 (4) | 0.097 (3) | 0.007 (2) | 0.028 (2) | -0.001 (2) |
| C16 | 0.079 (2) | 0.075 (2) | 0.072 (2) | -0.0066 (18) | 0.0422 (18) | 0.0016 (17) |
| O1  | 0.0897 (17) | 0.0572 (14) | 0.126 (2) | 0.0055 (13) | 0.0468 (16) | 0.0179 (14) |
| O2  | 0.0921 (18) | 0.116 (2) | 0.0801 (17) | 0.0134 (15) | 0.0433 (14) | -0.0069 (15) |
| Se1 | 0.1093 (3) | 0.0598 (2) | 0.1188 (4) | -0.0126 (2) | 0.0767 (3) | -0.0080 (2) |

Geometric parameters (Å, °)

|     |     |     |     |     |     |
|-----|-----|-----|-----|-----|-----|
| C13—O2 | 1.209 (4) | C2—H2 | 0.9300 |
| C13—C4 | 1.486 (4) | C3—C4 | 1.397 (4) |
| C13—C15 | 1.494 (5) | C3—H3 | 0.9300 |
| C14—O1 | 1.224 (4) | C4—C5 | 1.378 (4) |
| C14—C16 | 1.488 (4) | C5—C6 | 1.373 (4) |
| C14—C10 | 1.490 (4) | C5—H5 | 0.9300 |
| C15—H15A | 0.9600 | C6—H6 | 0.9300 |
| C15—H15B | 0.9600 | C7—C8 | 1.385 (4) |
| C15—H15C | 0.9600 | C7—C12 | 1.389 (4) |
| C16—H16A | 0.9600 | C8—C9 | 1.371 (4) |
| C16—H16B | 0.9600 | C8—H8 | 0.9300 |
| C16—H16C | 0.9600 | C9—C10 | 1.394 (4) |
| Se1—C7 | 1.918 (3) | C9—H9 | 0.9300 |
| Se1—C1 | 1.921 (3) | C10—C11 | 1.387 (4) |
| C1—C2 | 1.366 (5) | C11—C12 | 1.385 (4) |
| C1—C6 | 1.386 (4) | C11—H11 | 0.9300 |
| C2—C3 | 1.378 (5) | C12—H12 | 0.9300 |
| Bond | Angle (°) | Bond | Angle (°) |
|------|----------|------|----------|
| O2—C13—C4 | 120.8 (3) | C5—C4—C3 | 117.4 (3) |
| O2—C13—C15 | 119.3 (3) | C5—C4—C13 | 119.7 (3) |
| C4—C13—C15 | 119.9 (3) | C3—C4—C13 | 122.9 (3) |
| O1—C14—C16 | 120.8 (3) | C6—C5—C4 | 121.9 (3) |
| O1—C14—C10 | 119.6 (3) | C6—C5—H5 | 119.1 |
| C16—C14—C10 | 119.6 (3) | C4—C5—H5 | 119.1 |
| C13—C15—H15A | 109.5 | C5—C6—C1 | 120.0 (3) |
| C13—C15—H15B | 109.5 | C5—C6—H6 | 120.0 |
| H15A—C15—H15B | 109.5 | C1—C6—H6 | 120.0 |
| C13—C15—H15C | 109.5 | C8—C7—C12 | 119.7 (3) |
| H15A—C15—H15C | 109.5 | C8—C7—Se1 | 124.2 (2) |
| H15B—C15—H15C | 109.5 | C12—C7—Se1 | 116.0 (2) |
| C14—C16—H16A | 109.5 | C9—C8—C7 | 119.7 (3) |
| C14—C16—H16B | 109.5 | C9—C8—H8 | 120.1 |
| H16A—C16—H16B | 109.5 | C7—C8—H8 | 120.1 |
| C14—C16—H16C | 109.5 | C8—C9—C10 | 121.5 (3) |
| H16A—C16—H16C | 109.5 | C8—C9—H9 | 119.2 |
| C7—Se1—C1 | 99.58 (13) | C11—C10—C9 | 118.3 (3) |
| C2—C1—C6 | 119.0 (3) | C11—C10—C14 | 121.5 (3) |
| C2—C1—Se1 | 120.3 (3) | C11—C10—C14 | 121.5 (3) |
| C6—C1—Se1 | 120.6 (3) | C12—C11—C10 | 120.6 (3) |
| C1—C2—C3 | 120.9 (3) | C12—C11—C10 | 119.7 |
| C1—C2—H2 | 119.5 | C10—C11—C10 | 119.7 |
| C3—C2—H2 | 119.5 | C11—C12—C7 | 120.1 (3) |
| C2—C3—C4 | 120.7 (3) | C11—C12—H12 | 120.0 |
| C2—C3—H3 | 119.6 | C7—C12—H12 | 120.0 |
| C4—C3—H3 | 119.6 | C4—C3—H3 | 119.6 |
| C7—Se1—C1—C2 | 91.3 (3) | C1—Se1—C7—C8 | −15.7 (3) |
| C7—Se1—C1—C6 | −91.3 (3) | C1—Se1—C7—C12 | 164.3 (2) |
| C6—C1—C2—C3 | 0.7 (5) | C12—C7—C8—C9 | −0.4 (5) |
| Se1—C1—C2—C3 | 178.2 (3) | Se1—C7—C8—C9 | 179.6 (2) |
| C1—C2—C3—C4 | −0.9 (5) | C7—C8—C9—C10 | 0.0 (5) |
| C2—C3—C4—C5 | 0.3 (5) | C8—C9—C10—C11 | 0.0 (4) |
| C2—C3—C4—C13 | −177.6 (3) | C8—C9—C10—C14 | −179.4 (3) |
| O2—C13—C4—C5 | −1.2 (5) | O1—C14—C10—C11 | 177.4 (3) |
| C15—C13—C4—C5 | −179.3 (3) | C16—C14—C10—C11 | −2.9 (4) |
| O2—C13—C4—C3 | 176.7 (3) | O1—C14—C10—C9 | −3.3 (4) |
| C15—C13—C4—C3 | −1.5 (5) | C16—C14—C10—C9 | 176.4 (3) |
| C3—C4—C5—C6 | 0.3 (5) | C9—C10—C11—C12 | 0.4 (4) |
| C13—C4—C5—C6 | 178.3 (3) | C14—C10—C11—C12 | 179.7 (3) |
| C4—C5—C6—C1 | −0.5 (5) | C10—C11—C12—C7 | −0.7 (5) |
| C2—C1—C6—C5 | −0.1 (5) | C8—C7—C12—C11 | 0.7 (5) |
| Se1—C1—C6—C5 | −177.5 (3) | Se1—C7—C12—C11 | −179.3 (2) |

Hydrogen-bond geometry (Å, °)

| D—H···A | D—H | H···A | D···A | D—H···A |
|---------|------|------|------|----------|
| C2—H2···O2i | 0.93 | 2.47 | 3.272 (5) | 145 |
supplementary materials

C12—H12···O1\textsuperscript{ii}  

|        |        |        |        |
|--------|--------|--------|--------|
| 0.93   | 2.53   | 3.317 (4) | 143    |

Symmetry codes: (i) \(x, -y+3/2, z-1/2\); (ii) \(x, y-1, z\).

Fig. 1
Fig. 2
