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## (2E)-3-(5-Bromo-2-thienyl)-1-(4-hydroxyphenyl)prop-2-en-1-one

B. Narayana,<sup>a</sup> K. Lakshmana,<sup>a</sup> B. K. Sarojini,<sup>b</sup> H. S. Yathirajan<sup>c</sup> and Michael Bolte<sup>d\*</sup>

<sup>a</sup>Department of Studies in Chemistry, Mangalore University, Mangalagangotri 574 199, India, <sup>b</sup>Department of Chemistry, P. A. College of Engineering, Nadupadavu, Mangalore 574 153, India, <sup>c</sup>Department of Studies in Chemistry, University of Mysore, Manasagangotri, Mysore 570 006, India, and <sup>d</sup>Institut für Anorganische Chemie, J. W. Goethe-Universität Frankfurt, Max-von-Laue-Strasse 7, 60438 Frankfurt/Main, Germany

Correspondence e-mail: bolte@chemie.uni-frankfurt.de

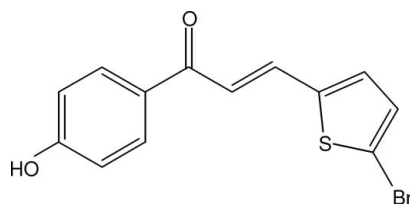
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Key indicators: single-crystal X-ray study;  $T = 173$  K; mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å;  $R$  factor = 0.031;  $wR$  factor = 0.066; data-to-parameter ratio = 15.5.

Geometric parameters of the title compound,  $\text{C}_{13}\text{H}_9\text{BrO}_2\text{S}$ , a chalcone derivative, are in the usual ranges. The  $\text{C}=\text{C}$  double bond is *trans* configured. The molecule is essentially planar (r.m.s. deviation for all non-H atoms = 0.069 Å). The crystal packing is stabilized by an  $\text{O}-\text{H}\cdots\text{O}$  hydrogen bond.

### Related literature

For related literature, see: Arty *et al.* (2000); Balaji *et al.* (2003); Batt *et al.* (1993); De Leon *et al.* (2003); Dimmock *et al.* (1999); Harrison *et al.* (2006); Modzelewska *et al.* (2006); Ng *et al.* (2006); Opletalova & Sedivy (1999); Opletalova *et al.* (2003); Patil *et al.* (2007); Sogawa *et al.* (1994); Won *et al.* (2005); Yathirajan *et al.* (2006); Zhao *et al.* (2000).



### Experimental

#### Crystal data

$\text{C}_{13}\text{H}_9\text{BrO}_2\text{S}$   
 $M_r = 309.17$   
 Orthorhombic, *Pbca*  
 $a = 13.2344$  (8) Å  
 $b = 11.0471$  (8) Å  
 $c = 16.1466$  (9) Å  
 $V = 2360.7$  (3) Å<sup>3</sup>  
 $Z = 8$   
 Mo  $K\alpha$  radiation  
 $\mu = 3.64$  mm<sup>-1</sup>  
 $T = 173$  (2) K  
 $0.31 \times 0.26 \times 0.21$  mm

#### Data collection

Stoe IPDSII two-circle diffractometer  
 Absorption correction: multi-scan (MULABS; Spek, 2003; Blessing,

1995)  
 $T_{\min} = 0.398$ ,  $T_{\max} = 0.515$   
 (expected range = 0.360–0.465)  
 18671 measured reflections  
 2461 independent reflections  
 2186 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.050$

#### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.031$   
 $wR(F^2) = 0.066$   
 $S = 1.07$   
 2461 reflections  
 159 parameters  
 H atoms treated by a mixture of independent and constrained refinement  
 $\Delta\rho_{\max} = 0.38$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.44$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{O2}-\text{H2O}\cdots\text{O1}^i$	0.92 (5)	1.79 (5)	2.701 (3)	173 (5)

Symmetry code: (i)  $x - \frac{1}{2}, -y + \frac{3}{2}, -z + 1$ .

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2456).

### References

- Arty, I. S., Timmermen, H., Samhoedi, M., Sastrohamidjojo, S. & Van Der Goto, H. (2000). *Eur. J. Med. Chem.* **35**, 449–457.  
 Balaji, R., Grande, D. & Nanjundan, S. (2003). *React. Funct. Polym.* **56**, 45–57.  
 Batt, D. G., Goodman, R., Jones, D. G., Kerr, J. R., Mantegna, J. R., McAllister, C., Newton, R. C., Nurnber, S., Welch, P. K. & Convington, M. B. (1993). *J. Med. Chem.* **36**, 1434–1442.  
 Blessing, R. H. (1995). *Acta Cryst.* **A51**, 33–38.  
 De Leon, E. J., Alcaraz, M. J., Dominguez, J. N., Charris, J. & Terencio, M. C. (2003). *Inflamm. Res.* **52**, 246–257.  
 Dimmock, J. R., Elias, D. W., Beazely, M. A. & Kandepu, N. M. (1999). *Curr. Med. Chem.* **6**, 1125–1149.  
 Harrison, W. T. A., Yathirajan, H. S., Ashalatha, B. V., Bindya, S. & Narayana, B. (2006). *Acta Cryst.* **E62**, o4164–o4165.  
 Modzelewska, A., Catherine Petit, C., Achanta, G., Davidson, N. E., Huang, P. & Khan, S. R. (2006). *Bioorg. Med. Chem.* pp. 3491–3495.  
 Ng, S.-L., Patil, P. S., Razak, I. A., Fun, H.-K. & Dharmaprakash, S. M. (2006). *Acta Cryst.* **E62**, o3200–o3202.  
 Opletalova, V., Jahodar, L., Jun, D. & Opletal, L. (2003). *Cesk. Farm.* **52**, 12–19.  
 Opletalova, V. & Sedivy, D. (1999). *Ceska Slov. Farm.* **48**, 252–255.  
 Patil, P. S., Ng, S.-L., Razak, I. A., Fun, H.-K. & Dharmaprakash, S. M. (2007). *Acta Cryst.* **E63**, o59–o60.  
 Sheldrick, G. M. (1990). *Acta Cryst.* **A46**, 467–473.  
 Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.  
 Sogawa, S., Nihro, Y., Ueda, H., Miki, T., Matsumoto, H. & Satoh, T. (1994). *Biol. Pharm. Bull.* **17**, 251–256.  
 Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.  
 Stoe & Cie (2001). *X-AREA*. Stoe & Cie, Darmstadt, Germany.  
 Won, S. J., Liu, C. T., Tsao, L. T., Weng, J. R., Ko, H. H., Wang, J. P. & Lin, C. N. (2005). *Eur. J. Med. Chem.* **40**, 103–112.  
 Yathirajan, H. S., Sarojini, B. K., Narayana, B., Ashalatha, B. V. & Bolte, M. (2006). *Acta Cryst.* **E62**, o3964–o3965.  
 Zhao, B., Lu, W.-Q., Zhou, Z.-H. & Wu, Y. (2000). *J. Mater. Chem.* **10**, 1513–1517.

**supplementary materials**

*Acta Cryst.* (2007). E63, o4552 [ doi:10.1107/S1600536807054359 ]

## (2E)-3-(5-Bromo-2-thienyl)-1-(4-hydroxyphenyl)prop-2-en-1-one

B. Narayana, K. Lakshmana, B. K. Sarojini, H. S. Yathirajan and M. Bolte

### Comment

Chalcone derivatives are of interest because of their pharmacological properties (Batt *et al.*, 1993; Sogawa *et al.*, 1994; Arty *et al.*, 2000). They have a wide variety of pharmaceutical activities including anticancer (Modzelewska *et al.*, 2006), anti-inflammatory (Won *et al.*, 2005) and antipyretic (De Leon *et al.*, 2003). Chalcones and their heterocyclic derivatives show numerous biological effects (Opletalova & Sedivy, 1999). The cytotoxic, anticancer, chemopreventative and mutagenic properties of a number of chalcones have been reviewed (Dimmock *et al.*, 1999). Chalcones and their analogues are used as potential therapeutic agents in diseases of the cardiovascular system (Opletalova *et al.*, 2003). Photo-cross-linkable polymers having the chalcone moiety act as negative photo resist materials used in a wide variety of applications (Balaji *et al.*, 2003). Chalcones are also used in designing effective second-order non-linear optical materials (Zhao *et al.*, 2000). The crystal structures of 3-(5-bromo-2-thienyl)-1-(4-methoxyphenyl)-prop-2-en-1-one (Patil *et al.*, 2007), 1-(3-bromo-2-thienyl)-3-(4-methoxyphenyl)prop-2-en-1-one (Harrison *et al.*, 2006), 1-(4-chlorophenyl)-3-(2-thienyl)prop-2-en-1-one (Ng *et al.*, 2006), 1-(3-bromo-2-thienyl)-3-(4,5-dimethoxy-2-nitrophenyl)prop-2-en-1-one (Yathirajan *et al.*, 2006) have been reported. The structure determination of the title compound, (I), was undertaken as a part of our study on chalcones.

Geometric parameters of the title compound are in the usual ranges. The C—C double bond is *trans* configured. The molecule is essentially planar (r.m.s. deviation for all non-H atoms 0.069 Å). The crystal packing is stabilized by a O—H···O hydrogen bond.

### Experimental

5-Bromo-2-thiophene carbaldehyde (1.91 g, 0.01 mol) and 4-hydroxyacetophenone (1.36 g, 0.01 mol) were stirred in ethanol (30 ml) at 298 K. 10 ml of a 10% aqueous NaOH solution was added slowly. The mixture was stirred for 2 h. The resulting precipitate was filtered off, washed with water and dried. The resulting crude product was recrystallized from 1,4-dioxane. Crystals suitable for single-crystal X-ray diffraction were grown by slow evaporation of the solution of the compound in 1,4-dioxane.

### Refinement

All H atoms were found in a difference map, but those bonded to C were geometrically positioned and refined with fixed individual displacement parameters [ $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$ ] using a riding model with C—H = 0.95 Å. The hydroxyl H atom was freely refined.

### Figures

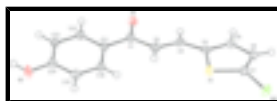


Fig. 1. Perspective view of the title compound with the atom numbering; displacement ellipsoids are at the 50% probability level.

## (2E)-3-(5-Bromo-2-thienyl)-1-(4-hydroxyphenyl)prop-2-en-1-one

### Crystal data

$C_{13}H_9BrO_2S$	$F_{000} = 1232$
$M_r = 309.17$	$D_x = 1.740 \text{ Mg m}^{-3}$
Orthorhombic, <i>Pbca</i>	Mo $K\alpha$ radiation
Hall symbol: -P 2ac 2ab	$\lambda = 0.71073 \text{ \AA}$
$a = 13.2344 (8) \text{ \AA}$	Cell parameters from 17692 reflections
$b = 11.0471 (8) \text{ \AA}$	$\theta = 3.5\text{--}26.7^\circ$
$c = 16.1466 (9) \text{ \AA}$	$\mu = 3.64 \text{ mm}^{-1}$
$V = 2360.7 (3) \text{ \AA}^3$	$T = 173 (2) \text{ K}$
$Z = 8$	Block, yellow
	$0.31 \times 0.26 \times 0.21 \text{ mm}$

### Data collection

Stoe IPDSII two-circle diffractometer	2461 independent reflections
Radiation source: fine-focus sealed tube	2186 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.050$
$T = 173(2) \text{ K}$	$\theta_{\text{max}} = 26.6^\circ$
$\omega$ scans	$\theta_{\text{min}} = 3.5^\circ$
Absorption correction: multi-scan (MULABS; Spek, 2003; Blessing, 1995)	$h = -16 \rightarrow 13$
$T_{\text{min}} = 0.398$ , $T_{\text{max}} = 0.515$	$k = -13 \rightarrow 13$
18671 measured reflections	$l = -20 \rightarrow 20$

### Refinement

Refinement on $F^2$	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.031$	$w = 1/[\sigma^2(F_o^2) + (0.0295P)^2 + 2.4425P]$
$wR(F^2) = 0.066$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.07$	$(\Delta/\sigma)_{\text{max}} = 0.001$
2461 reflections	$\Delta\rho_{\text{max}} = 0.38 \text{ e \AA}^{-3}$
159 parameters	$\Delta\rho_{\text{min}} = -0.44 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: SHELXL97, $F_c^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Secondary atom site location: difference Fourier map	Extinction coefficient: 0.0129 (5)

*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  $F^2 > 2\sigma(F^2)$  is used only for calculating  $R$ -factors(gt) *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 ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Br1	0.864761 (19)	0.01930 (2)	0.897108 (16)	0.02544 (11)
S1	0.79325 (4)	0.23561 (5)	0.78663 (4)	0.02105 (15)
O1	0.80436 (13)	0.61367 (15)	0.57092 (11)	0.0227 (4)
O2	0.32915 (13)	0.69338 (18)	0.52976 (12)	0.0272 (4)
H2O	0.318 (4)	0.761 (4)	0.499 (3)	0.077 (14)*
C1	0.73140 (17)	0.5591 (2)	0.60266 (14)	0.0170 (4)
C2	0.75165 (19)	0.4590 (2)	0.66109 (14)	0.0199 (5)
H2	0.6972	0.4235	0.6908	0.024*
C3	0.84604 (18)	0.41744 (19)	0.67284 (14)	0.0174 (5)
H3	0.8977	0.4551	0.6412	0.021*
C4	0.87774 (17)	0.3218 (2)	0.72835 (14)	0.0178 (5)
C5	0.97564 (18)	0.2833 (2)	0.74100 (14)	0.0191 (5)
H5	1.0323	0.3196	0.7147	0.023*
C6	0.98421 (19)	0.1849 (2)	0.79680 (14)	0.0202 (5)
H6	1.0463	0.1485	0.8126	0.024*
C7	0.89154 (19)	0.1490 (2)	0.82491 (14)	0.0195 (5)
C11	0.62570 (17)	0.5936 (2)	0.58404 (13)	0.0152 (4)
C12	0.60622 (18)	0.6967 (2)	0.53534 (14)	0.0178 (5)
H12	0.6612	0.7430	0.5146	0.021*
C13	0.50800 (17)	0.7317 (2)	0.51712 (13)	0.0169 (4)
H13	0.4962	0.8017	0.4844	0.020*
C14	0.42628 (17)	0.6641 (2)	0.54686 (13)	0.0176 (5)
C15	0.44386 (19)	0.5611 (2)	0.59508 (14)	0.0201 (5)
H15	0.3887	0.5147	0.6153	0.024*
C16	0.54232 (18)	0.5271 (2)	0.61317 (13)	0.0182 (5)
H16	0.5537	0.4571	0.6461	0.022*

*Atomic displacement parameters ( $\text{\AA}^2$ )*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Br1	0.02815 (16)	0.02015 (15)	0.02802 (15)	0.00418 (10)	0.00377 (10)	0.00766 (10)
S1	0.0174 (3)	0.0206 (3)	0.0251 (3)	0.0030 (2)	0.0005 (2)	0.0052 (2)

## supplementary materials

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O1	0.0151 (8)	0.0228 (9)	0.0303 (9)	-0.0014 (7)	0.0005 (7)	0.0082 (7)
O2	0.0135 (8)	0.0327 (10)	0.0355 (10)	0.0010 (7)	-0.0017 (7)	0.0147 (8)
C1	0.0174 (11)	0.0165 (10)	0.0171 (10)	0.0017 (8)	-0.0010 (9)	-0.0009 (9)
C2	0.0187 (11)	0.0192 (11)	0.0217 (11)	-0.0003 (9)	0.0005 (9)	0.0044 (9)
C3	0.0202 (12)	0.0135 (10)	0.0185 (11)	0.0005 (8)	-0.0018 (9)	-0.0012 (8)
C4	0.0171 (11)	0.0172 (11)	0.0191 (11)	0.0000 (8)	-0.0015 (9)	-0.0016 (8)
C5	0.0183 (11)	0.0178 (11)	0.0211 (11)	-0.0015 (9)	-0.0020 (9)	-0.0003 (9)
C6	0.0202 (11)	0.0204 (11)	0.0200 (11)	0.0044 (9)	-0.0034 (9)	-0.0002 (9)
C7	0.0243 (12)	0.0154 (10)	0.0189 (11)	0.0040 (9)	-0.0019 (9)	0.0005 (9)
C11	0.0156 (11)	0.0156 (10)	0.0144 (10)	0.0011 (8)	-0.0009 (8)	0.0003 (8)
C12	0.0167 (11)	0.0168 (10)	0.0200 (11)	-0.0018 (9)	0.0023 (9)	0.0021 (9)
C13	0.0168 (11)	0.0173 (10)	0.0167 (11)	0.0010 (8)	0.0003 (8)	0.0044 (8)
C14	0.0147 (11)	0.0209 (11)	0.0171 (10)	0.0009 (8)	-0.0003 (8)	0.0011 (9)
C15	0.0172 (11)	0.0212 (11)	0.0218 (11)	-0.0034 (9)	0.0019 (9)	0.0056 (9)
C16	0.0193 (11)	0.0169 (11)	0.0185 (11)	0.0003 (9)	-0.0007 (9)	0.0036 (9)

### *Geometric parameters (Å, °)*

Br1—C7	1.881 (2)	C5—H5	0.9500
S1—C7	1.729 (2)	C6—C7	1.366 (4)
S1—C4	1.744 (2)	C6—H6	0.9500
O1—C1	1.249 (3)	C11—C16	1.407 (3)
O2—C14	1.354 (3)	C11—C12	1.408 (3)
O2—H2O	0.92 (5)	C12—C13	1.388 (3)
C1—C2	1.478 (3)	C12—H12	0.9500
C1—C11	1.481 (3)	C13—C14	1.399 (3)
C2—C3	1.344 (3)	C13—H13	0.9500
C2—H2	0.9500	C14—C15	1.398 (3)
C3—C4	1.448 (3)	C15—C16	1.387 (3)
C3—H3	0.9500	C15—H15	0.9500
C4—C5	1.379 (3)	C16—H16	0.9500
C5—C6	1.417 (3)		
C7—S1—C4	90.71 (11)	C6—C7—Br1	126.56 (18)
C14—O2—H2O	118 (3)	S1—C7—Br1	120.08 (14)
O1—C1—C2	118.9 (2)	C16—C11—C12	117.8 (2)
O1—C1—C11	121.5 (2)	C16—C11—C1	122.6 (2)
C2—C1—C11	119.6 (2)	C12—C11—C1	119.6 (2)
C3—C2—C1	120.9 (2)	C13—C12—C11	121.0 (2)
C3—C2—H2	119.5	C13—C12—H12	119.5
C1—C2—H2	119.5	C11—C12—H12	119.5
C2—C3—C4	127.3 (2)	C12—C13—C14	120.2 (2)
C2—C3—H3	116.4	C12—C13—H13	119.9
C4—C3—H3	116.4	C14—C13—H13	119.9
C5—C4—C3	126.1 (2)	O2—C14—C15	117.8 (2)
C5—C4—S1	110.76 (17)	O2—C14—C13	122.4 (2)
C3—C4—S1	123.13 (18)	C15—C14—C13	119.8 (2)
C4—C5—C6	113.9 (2)	C16—C15—C14	119.6 (2)
C4—C5—H5	123.0	C16—C15—H15	120.2
C6—C5—H5	123.0	C14—C15—H15	120.2

C7—C6—C5	111.2 (2)	C15—C16—C11	121.7 (2)
C7—C6—H6	124.4	C15—C16—H16	119.2
C5—C6—H6	124.4	C11—C16—H16	119.2
C6—C7—S1	113.35 (17)		
O1—C1—C2—C3	-7.7 (3)	O1—C1—C11—C16	174.3 (2)
C11—C1—C2—C3	173.2 (2)	C2—C1—C11—C16	-6.6 (3)
C1—C2—C3—C4	179.1 (2)	O1—C1—C11—C12	-5.7 (3)
C2—C3—C4—C5	-177.9 (2)	C2—C1—C11—C12	173.4 (2)
C2—C3—C4—S1	4.2 (3)	C16—C11—C12—C13	0.3 (3)
C7—S1—C4—C5	-0.92 (18)	C1—C11—C12—C13	-179.7 (2)
C7—S1—C4—C3	177.3 (2)	C11—C12—C13—C14	-0.3 (3)
C3—C4—C5—C6	-177.9 (2)	C12—C13—C14—O2	-179.0 (2)
S1—C4—C5—C6	0.3 (3)	C12—C13—C14—C15	0.0 (3)
C4—C5—C6—C7	0.8 (3)	O2—C14—C15—C16	179.2 (2)
C5—C6—C7—S1	-1.5 (3)	C13—C14—C15—C16	0.1 (3)
C5—C6—C7—Br1	177.93 (17)	C14—C15—C16—C11	-0.1 (4)
C4—S1—C7—C6	1.41 (19)	C12—C11—C16—C15	-0.1 (3)
C4—S1—C7—Br1	-178.06 (14)	C1—C11—C16—C15	179.9 (2)

*Hydrogen-bond geometry* ( $\text{\AA}$ ,  $^\circ$ )

<i>D</i> —H $\cdots$ <i>A</i>	<i>D</i> —H	H $\cdots$ <i>A</i>	<i>D</i> $\cdots$ <i>A</i>	<i>D</i> —H $\cdots$ <i>A</i>
O2—H2O $\cdots$ O1 <sup>i</sup>	0.92 (5)	1.79 (5)	2.701 (3)	173 (5)

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

Fig. 1

