

(1*S*,2*S*)-1-(2-Bromo-4-hydroxy-3,5-dimethoxyphenyl)propane-1,2,3-triol

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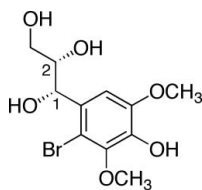
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 Key indicators: single-crystal X-ray study; $T = 200$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.022; wR factor = 0.032; data-to-parameter ratio = 13.8.

The title compound, $\text{C}_{11}\text{H}_{15}\text{BrO}_6$, is enantiomerically pure. The chirality at the stereogenic centres was installed *via* a Sharpless asymmetric dihydroxylation reaction and was determined to be *S* in each case. The crystal structure contains intermolecular hydrogen-bonding interactions.

Related literature

For related literature, see: Banwell *et al.* (2005); Sharpless *et al.* (1988).



Experimental

Crystal data

$\text{C}_{11}\text{H}_{15}\text{BrO}_6$	$V = 1269.51(4)$ Å ³
$M_r = 323.14$	$Z = 4$
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation
$a = 6.6489(1)$ Å	$\mu = 3.26$ mm ⁻¹
$b = 11.2217(2)$ Å	$T = 200$ K
$c = 17.0148(4)$ Å	$0.40 \times 0.40 \times 0.35$ mm

Data collection

Nonius KappaCCD diffractometer	23346 measured reflections
Absorption correction: multi-scan (SORTAV; Blessing 1995, 1997)	2906 independent reflections
$T_{\min} = 0.250$, $T_{\max} = 0.330$	2498 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.045$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.022$	Only H-atom coordinates refined
$wR(F^2) = 0.032$	$\Delta\rho_{\text{max}} = 0.52$ e Å ⁻³
$S = 1.02$	$\Delta\rho_{\text{min}} = -0.85$ e Å ⁻³
2906 reflections	Absolute structure: Flack (1983),
210 parameters	1219 Friedel pairs
44 restraints	Flack parameter: $-0.016(7)$

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O9—H9 ⁱ ··O11 ⁱ	0.74 (2)	2.05 (3)	2.732 (2)	154 (2)
O11—H11 ⁱ ··O14 ⁱⁱ	0.79 (2)	2.52 (3)	2.996 (2)	120 (2)
O11—H11 ⁱ ··O16 ⁱⁱ	0.79 (2)	2.06 (2)	2.817 (2)	161 (3)
O13—H13 ⁱ ··O9 ⁱ	0.79 (2)	1.97 (2)	2.727 (3)	159 (3)
O16—H16 ⁱ ··O17	0.78 (2)	2.30 (2)	2.759 (2)	118 (2)
O16—H16 ⁱ ··O13 ⁱⁱⁱ	0.78 (2)	2.01 (2)	2.696 (3)	146 (2)

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

Data collection: COLLECT (Nonius, 1997); cell refinement: DENZO/SCALEPACK (Otwinowski & Minor, 1997); data reduction: DENZO/SCALEPACK; program(s) used to solve structure: SIR92 (Altomare *et al.*, 1994); program(s) used to refine structure: CRYSTALS (Betteridge *et al.*, 2003); molecular graphics: ORTEPII (Johnson, 1976) in TEXSAN (Molecular Structure Corporation, 1992–1997); software used to prepare material for publication: CRYSTALS.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2601).

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supplementary materials

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(1*S*,2*S*)-1-(2-Bromo-4-hydroxy-3,5-dimethoxyphenyl)propane-1,2,3-triol

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Comment

In the stereoselective total synthesis of (+)-aiphanol, the enantiomer of the natural product (–)-aiphanol, the absolute stereochemistry on the 1,4-dioxane ring system was unequivocally determined (Banwell *et al.*, 2005). To achieve this the absolute stereochemistry at the stereogenic centres of the key intermediate employed in the synthesis of (+)-aiphanol, compound (II), was determined through single-crystal X-ray analysis of its brominated derivative, compound (I).

Compound (II) was brominated with pyridinium hydrobromide perbromide then the methoxymethyl (MOM) protecting group was removed under acidic conditions. The crude product was recrystallized from methanol-DCM to afford the title compound (I) as pure colourless crystals.

Compound (I) is enantiometrically pure. The absolute structure of the crystal has been determined by refinement of the Flack parameter. The chirality at the stereogenic centres was determined to be *S* in each case.

All H atoms were observed in a difference electron-density map. They were then repositioned geometrically and their coordinates refined with restraints being applied to distances and bond angles. Intermolecular hydrogen-bonding interactions are observed between the O—H groups O9—H9 and O13—H13 with O11 and O9, respectively, of an adjacent molecule. Intermolecular hydrogen bonding interactions are also observed for O11—H11 with O14 and O16, and for O16—H16 with O13 and O17 of adjacent molecules.

Experimental

Compound (II) was prepared *via* a Sharpless asymmetric dihydroxylation reaction (Sharpless *et al.*, 1988) and was obtained in >95% e.e. as determined by chiral HPLC analysis (for experimental details see Banwell *et al.*, 2005). A magnetically stirred solution of compound (II) (20 mg, 0.07 mmol) in DCM (5 ml) maintained at 291 K was treated, in one portion, with pyridinium hydrobromide perbromide (22 mg, 0.07 mmol) then stirred for 10 minutes after which TLC analysis indicated no starting material remained. Consequently, the reaction mixture was quenched with sodium bisulphite (0.5 ml of a 1 *M* aqueous solution) then treated with NaHCO₃ (2 ml of a saturated solution). The DCM layer was separated, and the aqueous layer extracted with additional DCM (2 x 5 ml). The combined organic phases were washed with brine (1 x 10 ml) then dried (Na₂SO₄), filtered and concentrated under reduced pressure and the residue subjected to high vacuum for 4 h. The resulting material (21 mg, 0.06 mmol) in MeOH (5 ml) was treated with HCl (1 drop of conc. material) and the ensuing mixture stirred magnetically at 291 K for 18 h. The methanol was then removed under reduced pressure, water (10 ml) was added to the residue and the product extracted with ethyl acetate (3 x 10 ml). The combined organic phases were then dried (Na₂SO₄), filtered, and concentrated under reduced pressure. The residue thus obtained was subjected to column chromatography (silica, 19:1 *v/v* ethyl acetate - methanol elution) to afford, after concentration of the appropriate fractions (*R_f* = 0.4), a white solid. Recrystallization (from methanol-DCM) of this material afforded compound (I) (18 mg, 93%) as colourless crystals, m.p. 446–446 K. [α]_D +51.2° (c 0.3, MeOH); ¹H NMR (300 MHz, CD₃OD) δ: 7.06 (s, 1H, ArH), 5.04

supplementary materials

(d, J 3.8 Hz, 1H), 3.90 (s, 3H), 3.81 (s, 3H), 3.76 (m, 1H), 3.60 (m, 2H); ^{13}C NMR (75 MHz, CD_3OD) δ : 148.4 (C), 144.6 (C), 139.9 (C), 132.2 (C), 108.3 (C), 107.6 (C), 75.0 (CH), 72.2 (CH), 63.8 (CH_2), 59.7 (OCH_3), 55.6 (OCH_3).

Refinement

All hydrogen atoms were observed in difference electron density maps prior to their inclusion. The alcohol H atoms were included at these locations and the other H atoms were added at calculated positions. Positional coordinates were refined for all H atoms with restraints being applied to distances and angles involving them.

Figures

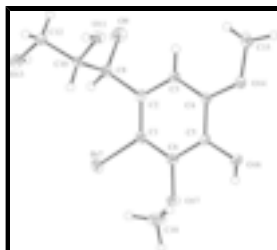


Fig. 1. The Molecular Structure of compound (I), with the atom labeling scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are drawn as spheres of small radii.

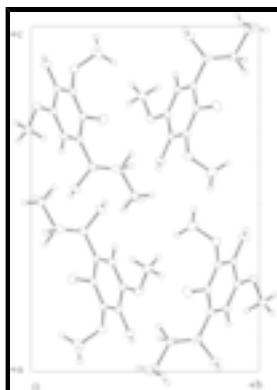


Fig. 2. Unit cell packing diagram of $\text{C}_{11}\text{H}_{15}\text{BrO}_6$. Hydrogen atoms are drawn as circles with small radii.

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Crystal data

$\text{C}_{11}\text{H}_{15}\text{BrO}_6$

$M_r = 323.14$

Orthorhombic, $P2_12_12_1$

$a = 6.6489$ (1) Å

$b = 11.2217$ (2) Å

$c = 17.0148$ (4) Å

$V = 1269.51$ (4) Å³

$Z = 4$

$F_{000} = 656$

$D_x = 1.691$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 17028 reflections

$\theta = 2.6$ – 27.5°

$\mu = 3.26$ mm⁻¹

$T = 200$ K

Block, colourless

$0.40 \times 0.40 \times 0.35$ mm

Data collection

Nonius KappaCCD diffractometer	2498 reflections with $I > 2.0\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.045$
$T = 200$ K	$\theta_{\text{max}} = 27.5^\circ$
φ and ω scans with CCD	$\theta_{\text{min}} = 3.0^\circ$
Absorption correction: multi-scan (SORTAV; Blessing 1995, 1997)	$h = -8 \rightarrow 8$
$T_{\text{min}} = 0.250$, $T_{\text{max}} = 0.330$	$k = -14 \rightarrow 14$
23346 measured reflections	$l = -22 \rightarrow 22$
2906 independent reflections	

Refinement

Refinement on F^2	Only H-atom coordinates refined
Least-squares matrix: full	Method, part 1, Chebychev polynomial, (Carruthers & Watkin, 1979, Prince, 1982) $[\text{weight}] = 1.0 / [A_0 * T_0(x) + A_1 * T_1(x) \dots + A_{n-1}] * T_{n-1}(x)$ where A_i are the Chebychev coefficients listed below and $x = F / F_{\text{max}}$ Method = Robust Weighting (Prince, 1982) $W = [\text{weight}] * [1 - (\Delta F / 6 * \text{sigma}(\Delta F)^2)]^2$ A_i are: 13.6 13.2 3.26
$R[F^2 > 2\sigma(F^2)] = 0.022$	$(\Delta/\sigma)_{\text{max}} = 0.016$
$wR(F^2) = 0.032$	$\Delta\rho_{\text{max}} = 0.52 \text{ e } \text{\AA}^{-3}$
$S = 1.02$	$\Delta\rho_{\text{min}} = -0.85 \text{ e } \text{\AA}^{-3}$
2906 reflections	Extinction correction: Larson (1970), Equation 22
210 parameters	Extinction coefficient: 89 (2)
44 restraints	Absolute structure: Flack (1983), 1219 Friedel pairs
Primary atom site location: structure-invariant direct methods	Flack parameter: -0.016 (7)
Hydrogen site location: inferred from neighbouring sites	

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Br7	0.28684 (4)	0.31423 (2)	0.730941 (16)	0.0364
O9	0.6736 (3)	0.1944 (2)	0.53031 (9)	0.0368
O11	0.9202 (2)	0.37486 (16)	0.58752 (10)	0.0278
O13	0.4452 (3)	0.48096 (16)	0.50205 (10)	0.0329
O14	1.0454 (2)	0.02665 (14)	0.76823 (11)	0.0306
O16	0.8140 (3)	0.06739 (16)	0.89022 (9)	0.0297
O17	0.4551 (2)	0.18876 (18)	0.87579 (9)	0.0319
C1	0.5309 (3)	0.22709 (17)	0.73816 (15)	0.0229
C2	0.6527 (3)	0.21166 (19)	0.67249 (13)	0.0222
C3	0.8289 (3)	0.14546 (19)	0.68246 (13)	0.0234
C4	0.8800 (3)	0.09653 (19)	0.75424 (13)	0.0229

supplementary materials

C5	0.7574 (3)	0.11533 (19)	0.81937 (12)	0.0229
C6	0.5814 (3)	0.1798 (2)	0.81127 (12)	0.0237
C8	0.6077 (4)	0.2688 (2)	0.59378 (13)	0.0232
C10	0.7057 (4)	0.39041 (19)	0.58618 (12)	0.0212
C12	0.6538 (4)	0.4546 (2)	0.50984 (14)	0.0276
C15	1.1565 (4)	-0.0135 (3)	0.70203 (15)	0.0348
C18	0.4909 (5)	0.2920 (3)	0.92280 (18)	0.0540
H9	0.581 (3)	0.176 (3)	0.5096 (15)	0.0440*
H11	0.971 (4)	0.4368 (17)	0.5962 (16)	0.0330*
H13	0.391 (4)	0.4210 (18)	0.4890 (17)	0.0400*
H16	0.708 (3)	0.058 (2)	0.9095 (14)	0.0360*
H31	0.917 (2)	0.1332 (11)	0.6391 (9)	0.0283*
H81	0.463 (2)	0.2807 (16)	0.5883 (10)	0.0280*
H101	0.666 (3)	0.4388 (15)	0.6317 (9)	0.0256*
H121	0.726 (3)	0.5283 (14)	0.5102 (10)	0.0332*
H122	0.695 (3)	0.4028 (15)	0.4670 (10)	0.0332*
H151	1.258 (3)	-0.0688 (15)	0.7219 (11)	0.0419*
H152	1.216 (3)	0.0546 (15)	0.6763 (11)	0.0419*
H153	1.067 (3)	-0.0565 (18)	0.6689 (11)	0.0419*
H181	0.410 (3)	0.286 (2)	0.9696 (11)	0.0649*
H182	0.631 (3)	0.292 (2)	0.9376 (13)	0.0649*
H183	0.453 (4)	0.3617 (17)	0.8953 (12)	0.0649*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br7	0.02617 (10)	0.03819 (12)	0.04482 (13)	0.01027 (12)	0.00424 (12)	0.00857 (14)
O9	0.0529 (12)	0.0345 (10)	0.0229 (8)	0.0013 (11)	-0.0115 (8)	-0.0090 (9)
O11	0.0190 (9)	0.0369 (10)	0.0275 (9)	-0.0057 (8)	-0.0009 (7)	-0.0011 (8)
O13	0.0293 (10)	0.0424 (11)	0.0268 (10)	0.0048 (8)	-0.0038 (8)	0.0048 (9)
O14	0.0298 (8)	0.0390 (9)	0.0231 (8)	0.0142 (7)	-0.0001 (9)	0.0054 (9)
O16	0.0275 (10)	0.0403 (10)	0.0212 (9)	0.0052 (8)	0.0036 (8)	0.0082 (7)
O17	0.0323 (8)	0.0352 (9)	0.0281 (8)	0.0025 (9)	0.0108 (7)	0.0022 (9)
C1	0.0169 (9)	0.0202 (10)	0.0316 (13)	0.0013 (8)	0.0005 (10)	0.0012 (10)
C2	0.0236 (11)	0.0214 (12)	0.0215 (11)	-0.0029 (8)	-0.0032 (9)	0.0020 (9)
C3	0.0231 (13)	0.0273 (12)	0.0197 (11)	0.0029 (9)	0.0005 (9)	0.0024 (9)
C4	0.0223 (10)	0.0223 (11)	0.0240 (14)	0.0030 (9)	-0.0015 (9)	0.0014 (9)
C5	0.0280 (15)	0.0213 (10)	0.0195 (11)	-0.0010 (9)	-0.0006 (9)	0.0031 (8)
C6	0.0247 (11)	0.0233 (11)	0.0229 (11)	-0.0024 (11)	0.0048 (9)	-0.0007 (11)
C8	0.0221 (11)	0.0247 (12)	0.0228 (12)	0.0025 (9)	-0.0041 (10)	0.0005 (10)
C10	0.0208 (10)	0.0238 (11)	0.0188 (11)	0.0015 (11)	-0.0013 (10)	-0.0012 (8)
C12	0.0284 (13)	0.0309 (13)	0.0234 (13)	-0.0001 (10)	0.0019 (10)	0.0035 (10)
C15	0.0343 (15)	0.0423 (16)	0.0279 (13)	0.0163 (12)	0.0023 (10)	0.0017 (11)
C18	0.075 (2)	0.048 (2)	0.0389 (17)	-0.0024 (18)	0.0217 (16)	-0.0141 (15)

Geometric parameters (\AA , $^\circ$)

Br7—C1	1.8988 (19)	C3—C4	1.382 (3)
O9—C8	1.434 (3)	C3—H31	0.950 (15)

O9—H9	0.740 (16)	C4—C5	1.392 (3)
O11—C10	1.436 (3)	C5—C6	1.383 (3)
O11—H11	0.786 (16)	C8—C10	1.518 (3)
O13—C12	1.424 (3)	C8—H81	0.975 (15)
O13—H13	0.796 (16)	C10—C12	1.525 (3)
O14—C4	1.372 (2)	C10—H101	0.982 (15)
O14—C15	1.420 (3)	C12—H121	0.958 (16)
O16—C5	1.373 (2)	C12—H122	0.972 (15)
O16—H16	0.787 (16)	C15—H151	0.976 (15)
O17—C6	1.385 (2)	C15—H152	0.966 (16)
O17—C18	1.428 (3)	C15—H153	0.949 (16)
C1—C2	1.391 (3)	C18—H181	0.964 (16)
C1—C6	1.393 (3)	C18—H182	0.967 (16)
C2—C3	1.397 (3)	C18—H183	0.946 (17)
C2—C8	1.515 (3)		
Br7...O14 ⁱ	3.250 (2)	O11...O16 ^v	2.817 (2)
Br7...C4 ⁱ	3.366 (2)	O11...O14 ^v	2.996 (2)
Br7...C5 ⁱ	3.498 (2)	O13...O16 ⁱ	2.696 (3)
Br7...O11 ⁱⁱ	3.516 (2)	O13...O17 ⁱ	3.193 (2)
Br7...O16 ⁱ	3.573 (2)	O13...C3 ^{iv}	3.531 (3)
O9...O13 ⁱⁱⁱ	2.727 (3)	O14...C10 ^{vi}	3.349 (3)
O9...O11 ^{iv}	2.732 (2)	O17...C12 ^{vii}	3.349 (3)
O9...C8 ⁱⁱⁱ	3.600 (3)	O17...C10 ^{vii}	3.574 (3)
C8—O9—H9	105 (2)	C2—C8—H81	109.7 (10)
C10—O11—H11	109 (2)	O9—C8—H81	108.0 (11)
C12—O13—H13	107 (2)	C10—C8—H81	107.0 (11)
C4—O14—C15	117.42 (19)	C8—C10—O11	108.41 (19)
C5—O16—H16	100.0 (20)	C8—C10—C12	113.56 (19)
C6—O17—C18	113.7 (2)	O11—C10—C12	107.2 (2)
Br7—C1—C2	120.65 (18)	C8—C10—H101	108.3 (10)
Br7—C1—C6	117.36 (16)	O11—C10—H101	108.7 (10)
C2—C1—C6	121.99 (19)	C12—C10—H101	110.5 (10)
C1—C2—C3	117.2 (2)	C10—C12—O13	113.5 (2)
C1—C2—C8	122.8 (2)	C10—C12—H121	106.7 (11)
C3—C2—C8	119.9 (2)	O13—C12—H121	108.1 (11)
C2—C3—C4	121.7 (2)	C10—C12—H122	107.0 (10)
C2—C3—H31	119.8 (10)	O13—C12—H122	109.3 (11)
C4—C3—H31	118.6 (10)	H121—C12—H122	112.3 (13)
C3—C4—O14	125.3 (2)	O14—C15—H151	106.6 (11)
C3—C4—C5	120.0 (2)	O14—C15—H152	108.8 (11)
O14—C4—C5	114.72 (19)	H151—C15—H152	112.1 (14)
C4—C5—O16	118.64 (19)	O14—C15—H153	107.9 (12)
C4—C5—C6	119.7 (2)	H151—C15—H153	108.1 (14)
O16—C5—C6	121.6 (2)	H152—C15—H153	112.9 (14)
C1—C6—O17	122.27 (19)	O17—C18—H181	108.3 (13)
C1—C6—C5	119.46 (19)	O17—C18—H182	107.9 (13)
O17—C6—C5	118.2 (2)	H181—C18—H182	109.0 (14)

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C2—C8—O9	111.03 (19)	O17—C18—H183	110.4 (13)
C2—C8—C10	111.77 (19)	H181—C18—H183	108.3 (15)
O9—C8—C10	109.15 (19)	H182—C18—H183	112.9 (15)
Br7—C1—C2—C3	-179.4 (2)	O17—C6—C1—C2	-176.1 (2)
Br7—C1—C2—C8	4.1 (3)	O17—C6—C5—C4	175.0 (2)
Br7—C1—C6—O17	3.8 (3)	C1—C2—C3—C4	0.4 (3)
Br7—C1—C6—C5	179.8 (2)	C1—C2—C8—C10	90.4 (3)
O9—C8—C2—C1	-147.4 (2)	C1—C6—O17—C18	-91.3 (3)
O9—C8—C2—C3	36.2 (3)	C1—C6—C5—C4	-1.2 (3)
O9—C8—C10—O11	-58.7 (2)	C2—C1—C6—C5	-0.1 (3)
O9—C8—C10—C12	60.3 (3)	C2—C3—C4—C5	-1.7 (3)
O11—C10—C8—C2	64.6 (2)	C2—C8—C10—C12	-176.4 (2)
O11—C10—C12—O13	-177.4 (2)	C3—C2—C1—C6	0.5 (3)
O13—C12—C10—C8	62.9 (3)	C3—C2—C8—C10	-86.0 (3)
O14—C4—C3—C2	177.2 (2)	C3—C4—O14—C15	-10.0 (3)
O14—C4—C5—O16	2.1 (3)	C3—C4—C5—C6	2.1 (3)
O14—C4—C5—C6	-177.0 (2)	C4—C3—C2—C8	177.0 (2)
O16—C5—C4—C3	-178.8 (2)	C5—C4—O14—C15	169.0 (2)
O16—C5—C6—O17	-4.1 (3)	C5—C6—O17—C18	92.7 (3)
O16—C5—C6—C1	179.7 (2)	C6—C1—C2—C8	-176.0 (2)

Symmetry codes: (i) $-x+1, y+1/2, -z+3/2$; (ii) $x-1, y, z$; (iii) $x+1/2, -y+1/2, -z+1$; (iv) $x-1/2, -y+1/2, -z+1$; (v) $-x+2, y+1/2, -z+3/2$; (vi) $-x+2, y-1/2, -z+3/2$; (vii) $-x+1, y-1/2, -z+3/2$.

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O9—H9 \cdots O11 ^{iv}	0.74 (2)	2.05 (3)	2.732 (2)	154 (2)
O11—H11 \cdots O14 ^v	0.79 (2)	2.52 (3)	2.996 (2)	120 (2)
O11—H11 \cdots O16 ^v	0.79 (2)	2.06 (2)	2.817 (2)	161 (3)
O13—H13 \cdots O9 ^{iv}	0.79 (2)	1.97 (2)	2.727 (3)	159 (3)
O16—H16 \cdots O17	0.78 (2)	2.30 (2)	2.759 (2)	118 (2)
O16—H16 \cdots O13 ^{vii}	0.78 (2)	2.01 (2)	2.696 (3)	146 (2)

Symmetry codes: (iv) $x-1/2, -y+1/2, -z+1$; (v) $-x+2, y+1/2, -z+3/2$; (vii) $-x+1, y-1/2, -z+3/2$.

Fig. 1

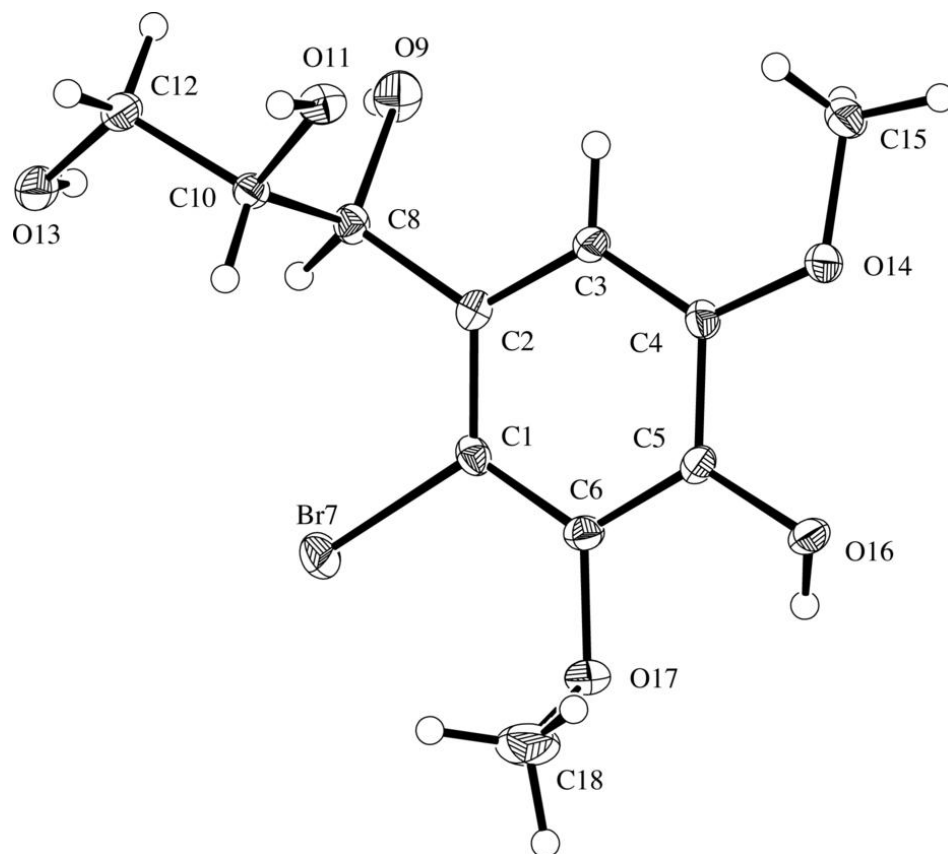


Fig. 2

