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(1*R*,2*R*)-1-(2-Bromo-4-hydroxy-3,5-dimethoxyphenyl)-2,3-dihydroxypropanol

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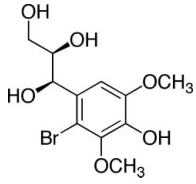
Key indicators: single-crystal X-ray study; T = 200 K; mean σ(C–C) = 0.004 Å; R factor = 0.022; wR factor = 0.058; data-to-parameter ratio = 13.8.

The title compound, $C_{11}H_{15}BrO_6$, is enantiomerically pure and the chirality at C-1 and C-2 was determined to be *R* in each case. The chirality at the stereogenic centres was installed *via* a Sharpless asymmetric dihydroxylation reaction. The crystal structure contains O—H···O hydrogen bonds.

Related literature

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



Experimental

Crystal data

$C_{11}H_{15}BrO_6$
M_r = 323.14
Orthorhombic, $P2_12_12_1$
a = 6.6461 (1) Å
b = 11.2214 (2) Å
c = 17.0108 (4) Å
V = 1268.64 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 3.26 mm^{-1}
T = 200 K
0.36 × 0.28 × 0.27 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995, 1997)
T_{min} = 0.380, T_{max} = 0.415
26121 measured reflections
2916 independent reflections
2296 reflections with $I > 2\sigma(I)$
R_{int} = 0.047

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.022
$wR(F^2)$ = 0.058
S = 0.91
2907 reflections
210 parameters
44 restraints
Only H-atom coordinates refined
$\Delta\rho_{max}$ = 0.70 e Å^{-3}
$\Delta\rho_{min}$ = −0.65 e Å^{-3}
Absolute structure: Flack (1983), 1214 Friedel pairs
Flack parameter: −0.007 (9)

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O9—H9···O11^{i}	0.77 (3)	1.98 (3)	2.730 (3)	164 (3)
O11—H11···O14^{ii}	0.80 (3)	2.35 (3)	2.989 (3)	137 (3)
O11—H11···O16^{ii}	0.80 (3)	2.09 (3)	2.812 (3)	150 (3)
O13—H13···O9^{i}	0.79 (3)	1.98 (3)	2.724 (3)	158 (3)
O16—H16···O17	0.80 (3)	2.33 (3)	2.752 (3)	114 (3)
O16—H16···O13^{iii}	0.80 (3)	1.97 (3)	2.690 (3)	149 (3)

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

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, 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: BT2560).

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supplementary materials

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(1*R*,2*R*)-1-(2-Bromo-4-hydroxy-3,5-dimethoxyphenyl)-2,3-dihydroxypropanol

S. Chand and A. C. Willis

Comment

In the stereoselective total synthesis 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 used in the synthesis, compound (II), was determined through single-crystal X-ray analysis of its brominated derivative (I).

Compound (I) was obtained through bromination of compound (II) with pyridinium hydrobromide perbromide followed by removal of the methoxymethyl (MOM) group under acidic conditions. The so-formed white solid was recrystallized from methanol-DCM to afford pure colourless crystals.

Compound (I) is enantiomerically pure and the absolute structure of the crystal has been determined by refinement of the Flack parameter. The outcome clearly indicates the absolute configuration at C-1 and C-2 as *R* 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) (25 mg, 0.09 mmol) in DCM (5 ml) maintained at 291 K was treated, in one portion, with pyridinium hydrobromide perbromide (32 mg, 0.1 mmol) and the ensuing mixture stirred for a further 10 minutes at which time TLC analysis indicated no starting material remained. Consequently, the reaction was quenched with sodium bisulfate (0.5 ml of a 1 *M* aqueous solution) then $NaHCO_3$ (2 ml of a saturated solution) was added. The DCM layer was separated, and the aqueous layer extracted with DCM (2 *x* 5 ml). The combined organic phases were washed with brine (1 *x* 10 ml) then dried (Na_2SO_4), filtered and the filtrate concentrated under reduced pressure. The resulting material (28 mg, 0.07 mmol) in MeOH (5 ml) was treated with conc. HCl (1 drop) and the ensuing mixture was 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 resulting mixture extracted with ethyl acetate (3 *x* 10 ml). The combined organic phases were then dried (Na_2SO_4), 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 of this material (from methanol-DCM) afforded the title compound (I) (20 mg, 91%) as colourless crystals, m.p. 445–446 K (Found: $M^{+\cdot}$, 324.0032 and 322.0053. $C_{11}H_{15}{}^{81}BrO_6$ and $C_{11}H_{15}{}^{79}BrO_6$ requires 324.0032 and 322.0052, respectively).

^{1}H NMR (300 MHz, CD_3OD) δ 7.05 (s, 1H, ArH), 5.04 (d, J 3.8 Hz, 1H), 3.89 (s, 3H), 3.80 (s, 3H), 3.75 (m, 1H), 3.60 (m, 2H); ^{13}C NMR (75 MHz, CD_3OD) δ 148.2 (C), 144.6 (C), 139.8 (C), 132.1 (C), 108.1 (C), 107.5 (C), 74.9 (CH), 72.0 (CH), 63.6 (CH_2), 59.5 (OCH_3), 55.4 (OCH_3); ν_{max} (NaCl)/cm^{-1} 3365 (broad), 2938, 1594, 1495, 1410, 1314, 1176, 1097, 856.

Refinement

Reflections with sinθ/λ<0.1 were rejected as unreliable as they are in the vicinity of the beam-stop shadow.

Figures

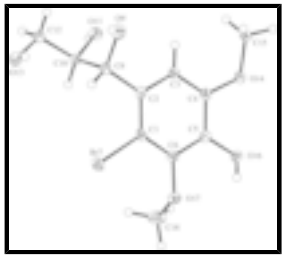


Fig. 1. The molecular structure of (I), with the atom labeling scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are drawn as spheres of small radius.

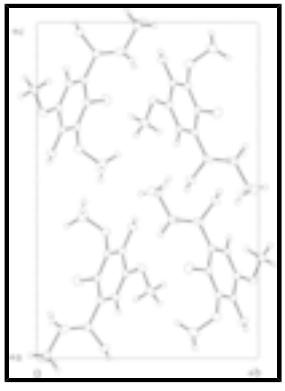


Fig. 2. Unit-cell packing diagram of $C_{11}H_{15}BrO_6$. Hydrogen atoms are drawn as circles with small radii.

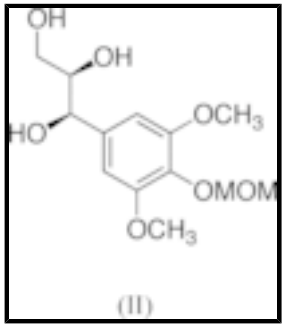


Fig. 3. The structure of compound (II)

(1*R*,2*R*)-1-(2-Bromo-4-hydroxy-3,5-dimethoxyphenyl)-2,3- dihydroxypropanol

Crystal data

$C_{11}H_{15}BrO_6$

M_r = 323.14

D_x = 1.692 Mg m^{-3}

Mo $K\alpha$ radiation

λ = 0.71073 Å

Orthorhombic, $P2_12_12_1$	Cell parameters from 14986 reflections
a = 6.6461 (1) Å	θ = 2.6–27.5º
b = 11.2214 (2) Å	μ = 3.26 mm^{-1}
c = 17.0108 (4) Å	T = 200 K
V = 1268.64 (4) Å^3	Block, colourless
Z = 4	0.36 × 0.28 × 0.27 mm
F_{000} = 656	

Data collection

Nonius KappaCCD diffractometer	2296 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.047
T = 200 K	θ_{max} = 27.5º
φ and ω scans with CCD	θ_{min} = 3.0º
Absorption correction: multi-scan (SORTAV; Blessing, 1995, 1997)	h = −8→8
T_{min} = 0.380, T_{max} = 0.415	k = −13→14
26121 measured reflections	l = −22→22
2916 independent reflections	

Refinement

Refinement on F^2	Only H-atom coordinates refined
Least-squares matrix: full	Method, part 1, Chebychev polynomial, (Carruthers & Watkin, 1979, *P*rince, 1982) [*w*eight] = 1.0/[A_0*T_0(x) + A_1*T_1(x) ··· + A_{n-1}]*T_{n-1}(x)] where A_i are the Chebychev coefficients listed below*w* and x = *F* /*F*max Method = Robust Weighting (*P*rince, 1982) W = [*w*eight] * [1-(delta*F*/6*sigma*F*)^2]^2 A_i are: 20.2 30.3 18.6 8.71 1.66
$R[F^2 > 2\sigma(F^2)]$ = 0.022	$(\Delta/\sigma)_{max}$ = 0.034
$wR(F^2)$ = 0.058	$\Delta\rho_{max}$ = 0.70 e Å^{-3}
S = 0.91	$\Delta\rho_{min}$ = −0.65 e Å^{-3}
2907 reflections	Extinction correction: Larson (1970), Equation 22
210 parameters	Extinction coefficient: 173 (9)
44 restraints	Absolute structure: Flack (1983), 1214 Friedel pairs
Primary atom site location: structure-invariant direct methods	Flack parameter: −0.007 (9)
Hydrogen site location: inferred from neighbouring sites	

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Br7	0.71340 (4)	0.68575 (2)	0.268949 (18)	0.0358
O9	0.3273 (3)	0.8058 (2)	0.46981 (11)	0.0372
O11	0.0799 (3)	0.62452 (19)	0.41226 (11)	0.0277

O13	0.5555 (3)	0.51920 (18)	0.49792 (12)	0.0326
O14	−0.0461 (3)	0.97329 (16)	0.23181 (13)	0.0298
O16	0.1867 (3)	0.93279 (17)	0.10984 (10)	0.0286
O17	0.5445 (3)	0.81131 (19)	0.12419 (11)	0.0314
C1	0.4695 (3)	0.7733 (2)	0.26163 (17)	0.0234
C2	0.3472 (4)	0.7884 (2)	0.32756 (15)	0.0228
C3	0.1705 (4)	0.8547 (2)	0.31769 (15)	0.0238
C4	0.1200 (4)	0.9032 (2)	0.24574 (14)	0.0237
C5	0.2422 (4)	0.8849 (2)	0.18035 (14)	0.0237
C6	0.4186 (3)	0.8203 (2)	0.18853 (14)	0.0241
C8	0.3924 (4)	0.7314 (2)	0.40585 (15)	0.0232
C10	0.2937 (4)	0.6088 (2)	0.41383 (13)	0.0215
C12	0.3465 (4)	0.5455 (2)	0.49012 (16)	0.0274
C15	−0.1565 (4)	1.0135 (3)	0.29855 (17)	0.0345
C18	0.5083 (7)	0.7077 (3)	0.0766 (2)	0.0545
H9	0.415 (4)	0.824 (3)	0.4971 (18)	0.0440*
H11	0.024 (5)	0.571 (2)	0.3902 (18)	0.0340*
H13	0.609 (5)	0.577 (2)	0.514 (2)	0.0400*
H16	0.291 (4)	0.943 (3)	0.0876 (18)	0.0350*
H31	0.085 (2)	0.8677 (11)	0.3605 (10)	0.0285*
H81	0.537 (3)	0.7206 (18)	0.4101 (12)	0.0279*
H101	0.335 (3)	0.5596 (17)	0.3687 (11)	0.0258*
H121	0.307 (3)	0.5945 (18)	0.5340 (11)	0.0328*
H122	0.272 (3)	0.4704 (16)	0.4909 (13)	0.0328*
H151	−0.256 (3)	1.071 (2)	0.2799 (13)	0.0414*
H152	−0.069 (3)	1.053 (2)	0.3358 (13)	0.0414*
H153	−0.222 (4)	0.9479 (18)	0.3252 (13)	0.0414*
H181	0.594 (4)	0.712 (2)	0.0322 (14)	0.0654*
H182	0.370 (3)	0.707 (2)	0.0596 (17)	0.0654*
H183	0.537 (5)	0.6359 (19)	0.1056 (14)	0.0654*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br7	0.02523 (12)	0.03501 (13)	0.04712 (15)	0.00987 (11)	0.00413 (13)	0.00840 (14)
O9	0.0540 (14)	0.0312 (9)	0.0263 (9)	0.0018 (10)	−0.0115 (8)	−0.0080 (9)
O11	0.0191 (9)	0.0343 (10)	0.0296 (10)	−0.0032 (7)	−0.0012 (7)	−0.0022 (8)
O13	0.0291 (10)	0.0390 (11)	0.0296 (10)	0.0046 (8)	−0.0038 (8)	0.0037 (9)
O14	0.0279 (9)	0.0356 (9)	0.0260 (8)	0.0122 (7)	0.0002 (9)	0.0057 (9)
O16	0.0269 (10)	0.0357 (9)	0.0233 (9)	0.0055 (8)	0.0019 (8)	0.0072 (7)
O17	0.0309 (9)	0.0312 (9)	0.0321 (9)	0.0015 (9)	0.0095 (7)	0.0030 (9)
C1	0.0194 (10)	0.0188 (9)	0.0319 (13)	0.0000 (8)	0.0008 (10)	0.0009 (10)
C2	0.0237 (11)	0.0210 (12)	0.0238 (12)	−0.0015 (8)	−0.0043 (9)	0.0008 (9)
C3	0.0236 (14)	0.0280 (11)	0.0197 (11)	0.0028 (9)	−0.0006 (9)	0.0010 (9)
C4	0.0221 (11)	0.0226 (10)	0.0263 (14)	0.0031 (9)	−0.0018 (9)	0.0019 (9)
C5	0.0256 (15)	0.0233 (10)	0.0221 (11)	−0.0020 (9)	−0.0010 (9)	0.0012 (9)
C6	0.0242 (11)	0.0222 (11)	0.0260 (12)	−0.0014 (11)	0.0049 (9)	0.0010 (11)
C8	0.0210 (12)	0.0238 (11)	0.0249 (13)	0.0010 (9)	−0.0033 (9)	0.0005 (10)

C10	0.0201 (10)	0.0226 (10)	0.0219 (11)	0.0003 (11)	0.0002 (11)	−0.0009 (8)
C12	0.0273 (13)	0.0301 (13)	0.0247 (13)	0.0000 (9)	0.0013 (10)	0.0022 (10)
C15	0.0348 (15)	0.0391 (15)	0.0296 (13)	0.0172 (12)	0.0033 (11)	0.0022 (12)
C18	0.073 (2)	0.048 (2)	0.0422 (18)	−0.0036 (17)	0.0220 (17)	−0.0157 (15)

Geometric parameters (Å, °)

Br7—C1	1.899 (2)	C3—C4	1.381 (3)
O9—C8	1.438 (3)	C3—H31	0.933 (17)
O9—H9	0.773 (18)	C4—C5	1.392 (4)
O11—C10	1.432 (3)	C5—C6	1.385 (3)
O11—H11	0.799 (17)	C8—C10	1.530 (3)
O13—C12	1.426 (3)	C8—H81	0.972 (17)
O13—H13	0.789 (18)	C10—C12	1.521 (4)
O14—C4	1.376 (3)	C10—H101	0.985 (17)
O14—C15	1.425 (3)	C12—H121	0.964 (17)
O16—C5	1.365 (3)	C12—H122	0.977 (17)
O16—H16	0.797 (17)	C15—H151	0.973 (17)
O17—C6	1.381 (3)	C15—H152	0.965 (18)
O17—C18	1.437 (4)	C15—H153	0.968 (17)
C1—C2	1.395 (4)	C18—H181	0.948 (18)
C1—C6	1.392 (4)	C18—H182	0.963 (18)
C2—C3	1.401 (3)	C18—H183	0.964 (18)
C2—C8	1.508 (3)		
Br7···O14^{i}	3.252 (2)	O11···O14^{v}	2.989 (3)
Br7···C4^{i}	3.368 (2)	O13···O16^{i}	2.690 (3)
Br7···C5^{i}	3.497 (2)	O13···O17^{i}	3.194 (3)
Br7···O11^{ii}	3.514 (2)	O13···C3^{iv}	3.525 (3)
Br7···O16^{i}	3.571 (2)	O14···C10^{vi}	3.340 (3)
O9···O13^{iii}	2.724 (3)	O17···C12^{vii}	3.348 (3)
O9···O11^{iv}	2.730 (3)	O17···C10^{vii}	3.566 (3)
O11···O16^{v}	2.812 (3)		
C8—O9—H9	112 (3)	C2—C8—H81	108.4 (12)
C10—O11—H11	112 (2)	O9—C8—H81	108.3 (12)
C12—O13—H13	108 (3)	C10—C8—H81	107.8 (12)
C4—O14—C15	117.2 (2)	C8—C10—O11	108.2 (2)
C5—O16—H16	104 (3)	C8—C10—C12	113.4 (2)
C6—O17—C18	113.8 (2)	O11—C10—C12	107.6 (2)
Br7—C1—C2	120.47 (19)	C8—C10—H101	108.3 (12)
Br7—C1—C6	117.50 (18)	O11—C10—H101	109.5 (12)
C2—C1—C6	122.0 (2)	C12—C10—H101	109.8 (12)
C1—C2—C3	117.2 (2)	C10—C12—O13	113.6 (2)
C1—C2—C8	122.8 (2)	C10—C12—H121	109.3 (12)
C3—C2—C8	119.9 (2)	O13—C12—H121	108.2 (13)
C2—C3—C4	121.3 (2)	C10—C12—H122	107.3 (13)
C2—C3—H31	119.8 (11)	O13—C12—H122	108.2 (13)
C4—C3—H31	118.9 (11)	H121—C12—H122	110.1 (15)

C3—C4—O14	124.9 (2)	O14—C15—H151	107.3 (13)
C3—C4—C5	120.6 (2)	O14—C15—H152	111.0 (14)
O14—C4—C5	114.5 (2)	H151—C15—H152	108.7 (15)
C4—C5—O16	119.1 (2)	O14—C15—H153	111.3 (13)
C4—C5—C6	119.4 (2)	H151—C15—H153	110.5 (15)
O16—C5—C6	121.5 (2)	H152—C15—H153	108.0 (15)
C1—C6—C5	119.6 (2)	O17—C18—H181	107.8 (15)
C1—C6—O17	122.2 (2)	O17—C18—H182	109.7 (15)
C5—C6—O17	118.1 (2)	H181—C18—H182	109.5 (16)
C2—C8—O9	111.2 (2)	O17—C18—H183	110.8 (15)
C2—C8—C10	112.0 (2)	H181—C18—H183	109.4 (16)
O9—C8—C10	109.0 (2)	H182—C18—H183	109.5 (16)
Br7—C1—C2—C3	179.6 (2)	O17—C6—C1—C2	176.3 (2)
Br7—C1—C2—C8	−3.8 (3)	O17—C6—C5—C4	−175.1 (2)
Br7—C1—C6—O17	−4.1 (3)	C1—C2—C3—C4	−0.1 (3)
Br7—C1—C6—C5	−179.9 (2)	C1—C2—C8—C10	−90.6 (3)
O9—C8—C2—C1	147.1 (2)	C1—C6—O17—C18	91.6 (3)
O9—C8—C2—C3	−36.4 (3)	C1—C6—C5—C4	0.9 (3)
O9—C8—C10—O11	59.3 (2)	C2—C1—C6—C5	0.4 (3)
O9—C8—C10—C12	−60.0 (3)	C2—C3—C4—C5	1.5 (4)
O11—C10—C8—C2	−64.2 (3)	C2—C8—C10—C12	176.5 (2)
O11—C10—C12—O13	177.4 (2)	C3—C2—C1—C6	−0.8 (3)
O13—C12—C10—C8	−63.0 (3)	C3—C2—C8—C10	85.9 (3)
O14—C4—C3—C2	−177.2 (2)	C3—C4—O14—C15	10.0 (4)
O14—C4—C5—O16	−2.0 (3)	C3—C4—C5—C6	−1.9 (4)
O14—C4—C5—C6	177.0 (2)	C4—C3—C2—C8	−176.9 (2)
O16—C5—C4—C3	179.2 (2)	C5—C4—O14—C15	−168.8 (2)
O16—C5—C6—O17	3.8 (3)	C5—C6—O17—C18	−92.5 (3)
O16—C5—C6—C1	179.8 (2)	C6—C1—C2—C8	175.8 (2)

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O9—H9···O11^{iv}	0.77 (3)	1.98 (3)	2.730 (3)	164 (3)
O11—H11···O14^{v}	0.80 (3)	2.35 (3)	2.989 (3)	137 (3)
O11—H11···O16^{v}	0.80 (3)	2.09 (3)	2.812 (3)	150 (3)
O13—H13···O9^{iv}	0.79 (3)	1.98 (3)	2.724 (3)	158 (3)
O16—H16···O17	0.80 (3)	2.33 (3)	2.752 (3)	114 (3)
O16—H16···O13^{vii}	0.80 (3)	1.97 (3)	2.690 (3)	149 (3)

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

Fig. 1

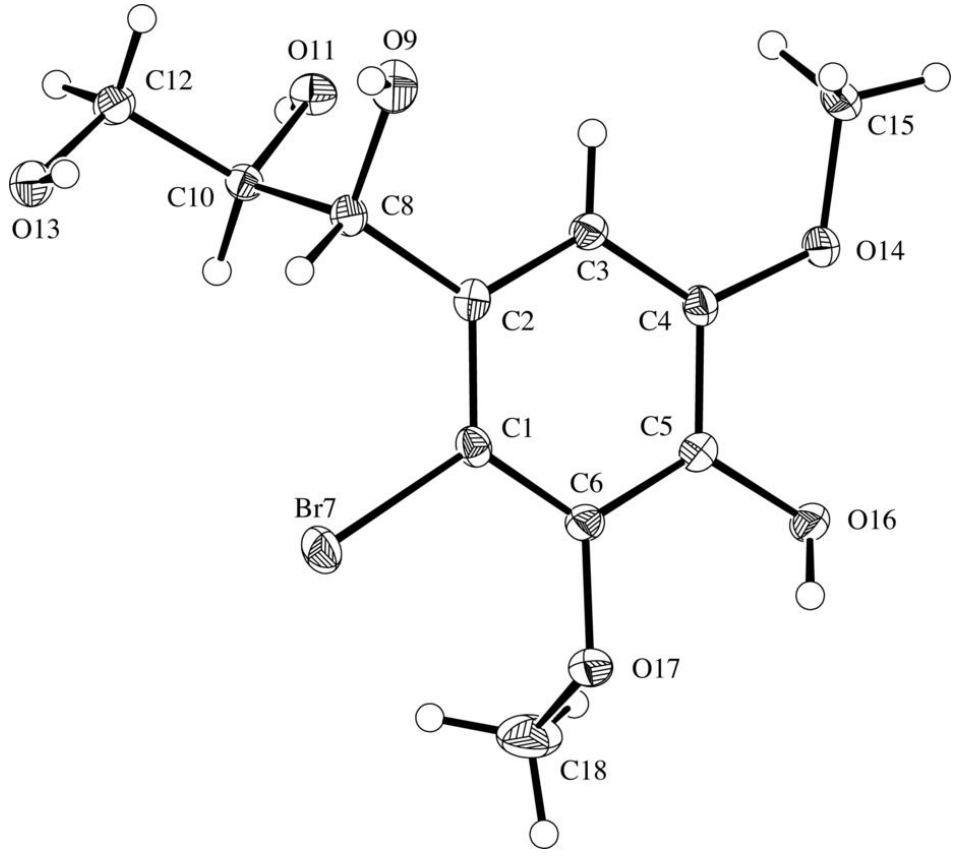


Fig. 2

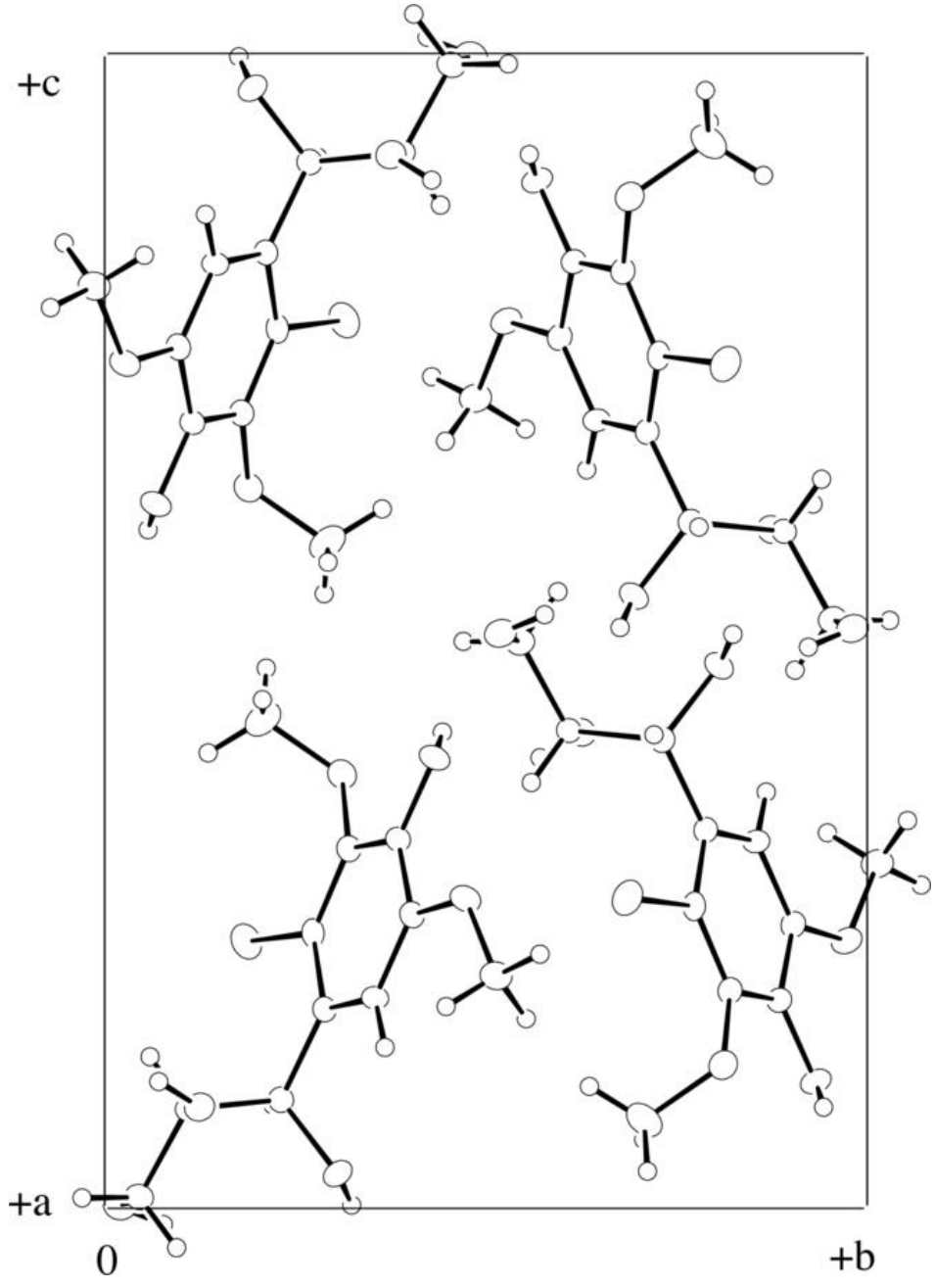
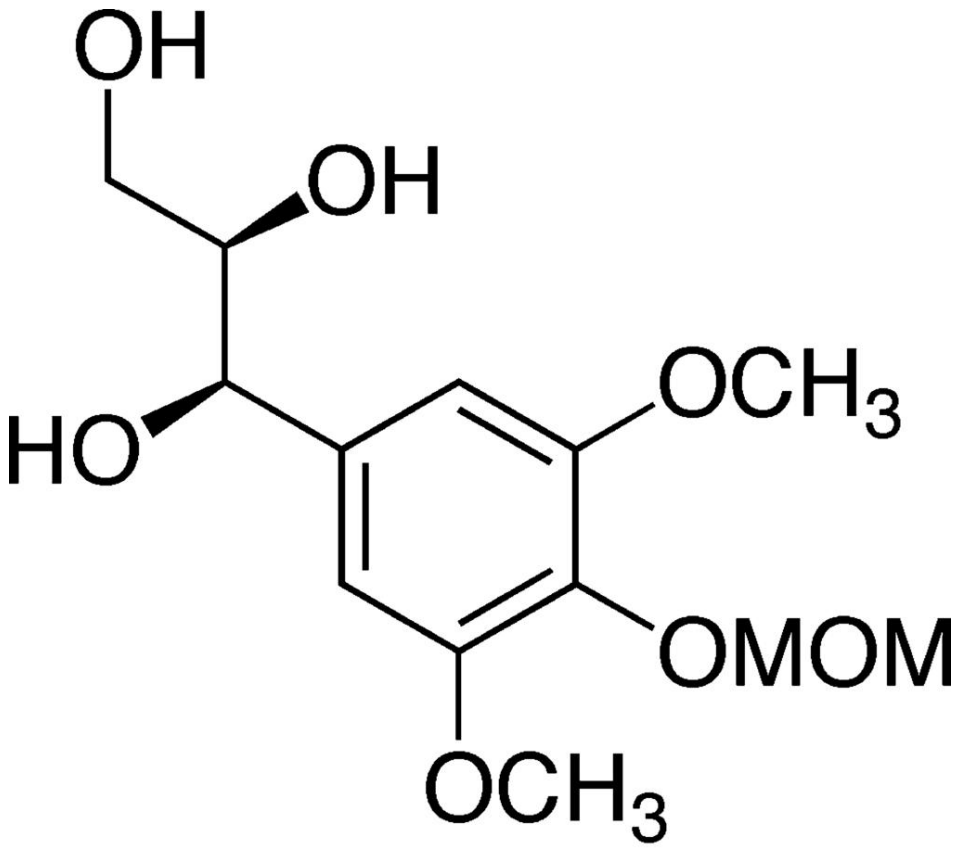


Fig. 3



(II)