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3,4′,5,5′-Tetramethoxy-2′-methylbiphenyl-4-ol

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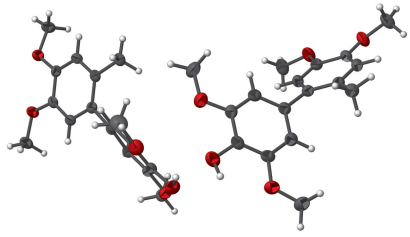
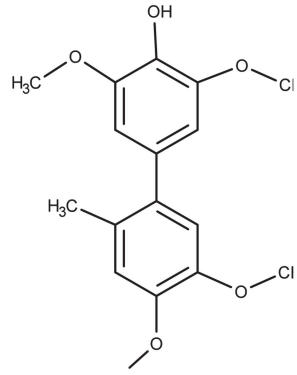
The asymmetric unit of the title compound, $C_{17}H_{20}O_5$, contains two independent molecules, *A* and *B*, with similar geometries [dihedral angles between the phenyl rings = 56.19 (8) and 54.98 (7)°, respectively]. Intramolecular O—H···O hydrogen bonds occur in both molecules. In the crystal, the *A* molecules form [1$\bar{1}$0] chains linked by O—H···O hydrogen bonds from the hydroxyl group to one of the methoxy O atoms. The *B* molecules form O—H···O hydrogen bonds to the hydroxyl O atoms of the *A* molecules and thus act as fixed spacers between the chains of molecule *A*. Some weak C—H···O contacts are also present.

3D view



Chemical scheme



Structure description

Non-symmetric biaryl systems are a frequently occurring structural motif and can be found in natural products (Bringmann *et al.*, 2005) and material science (Grimsdale *et al.*, 2009) and as a part of ligand building blocks for catalysis (Noyori, 2003). The cross-coupling of non-symmetric biaryl systems represents a significant, versatile and useful transformation in organic chemistry (Cepanec, 2004) while electrochemical synthesis offers access to new substances (Wiebe *et al.*, 2018) that could not previously be synthesized in classical chemistry (Waldvogel *et al.*, 2018; Lips *et al.*, 2018, 2019; Dörr *et al.*, 2019;Schulz *et al.*, 2017). Additionally, the electro-organic synthesis of biaryl systems is a sustainable (Wiebe *et al.*, 2017) and applicable metal- and reagent-free technology.

The triclinic unit cell of the title compound contains two independent molecules with nearly identical geometries (r.m.s. deviation = 0.143 Å) (Fig. 1). The dihedral angles between the least-squares planes of the phenyl rings are 56.19 (8) and 54.98 (7)° for molecules *A* and *B*, respectively. The molecular conformations are consolidated by intramolecular O—H···O hydrogen bonds (Table 1). All of the methoxy groups are located close to the planes of their connected phenyl rings. In the crystal, molecules *B* and

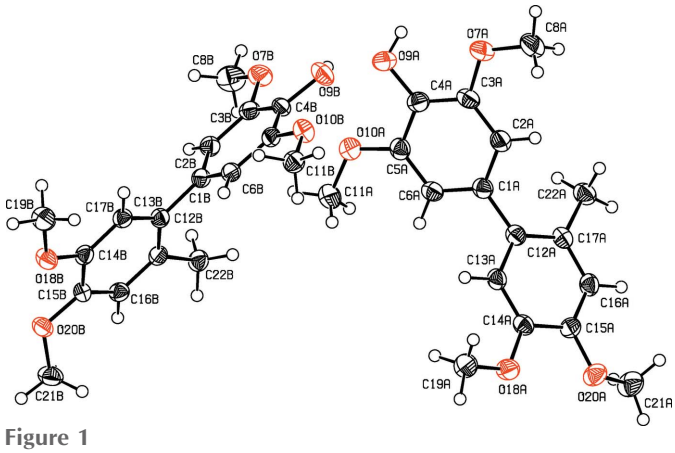


Figure 1
The molecular structure of the title compound with displacement ellipsoids drawn at the 50% probability level.

A are linked *via* an O9*B*—H9*B*···O9*A* hydrogen bond while molecule *A* shows an O9*A*—H9*A*···O20*A* bond, leading to a chain along [1$\bar{1}$0] (Fig. 2).

Synthesis and crystallization

A solution of 2,6-dimethoxyphenol (0.58 g, 3.8 mmol, 1.0 eq.), 1,2-dimethoxy-4-methylbenzene (1.73 g, 11.4 mmol, 3.0 eq.) and *N*-methyl-*N*,*N*,*N*-tributylammonium methylsulfate (0.77 g, 2.25 mmol) in 25 ml of 1,1,1,3,3,3-hexafluoro-propan-2-ol was transferred into a undivided beaker-type electrolysis cell equipped with boron-doped diamond electrodes. A constant current electrolysis with a current density of 5.2 mA cm^{-2} was performed at 323 K. After application of 731 C (2 F per 2,6-dimethoxyphenol) the electrolysis was stopped and the solvent mixture was recovered *in vacuo* (323 K, 200-70 mbar). The crude coupling product was purified by column chromatography [SiO_2, ethyl acetate/cyclohexane (1:4)] and the desired product was obtained as a slightly brown solid [yield: 68%, 0.79 g, 2.6 mmol); m.p. 367 K (cyclohexane/ethyl acetate (4:1)]; Suitable single crystals were obtained by slowly diluting a saturated solution of the title compound in ethyl acetate with *n*-heptane (diffusion method).

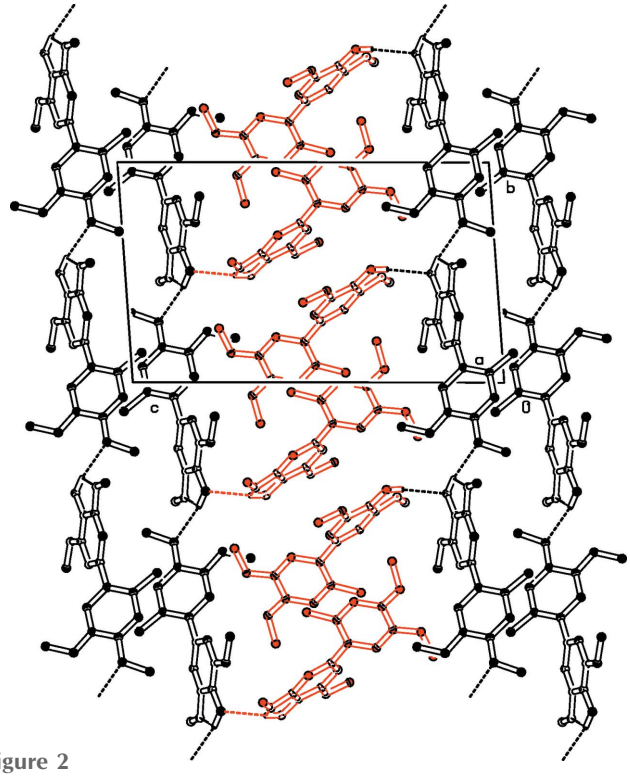


Figure 2
A partial packing diagram viewed along [100] with *A* molecules in black and *B* molecules in red. Hydrogen bonds are shown as dashed lines.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O9*A*—H9*A*···O7*A*	0.87 (3)	2.30 (3)	2.703 (2)	108 (2)
O9*B*—H9*B*···O7*B*	0.88 (3)	2.16 (3)	2.659 (2)	116 (2)
O9*A*—H9*A*···O20*A*^{i}	0.87 (3)	2.34 (3)	3.0000 (19)	133 (2)
O9*B*—H9*B*···O9*A*	0.88 (3)	2.23 (3)	2.992 (2)	146 (2)
C13*A*—H13*A*···O20*B*^{ii}	0.95	2.52	3.453 (2)	166
C13*B*—H13*B*···O9*B*^{iii}	0.95	2.55	3.498 (2)	172
C19*A*—H19*C*···O18*B*^{ii}	0.98	2.49	3.452 (3)	167
C19*B*—H19*D*···O18*A*^{iv}	0.98	2.54	3.475 (3)	159

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{17}H_{20}O_5$
M_r	304.33
Crystal system, space group	Triclinic, $P\bar{1}$
Temperature (K)	193
a, *b*, *c* (Å)	7.5318 (5), 11.0092 (6), 18.1896 (12)
α, β, γ (°)	85.600 (5), 87.945 (5), 78.702 (5)
V (Å^3)	1474.34 (16)
Z	4
Radiation type	Mo *K*α
μ (mm^{-1})	0.10
Crystal size (mm)	0.31 × 0.21 × 0.13
Data collection	
Diffractometer	Stoe *IPDS* 2T
Absorption correction	–
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	13532, 6992, 4168
R_{int}	0.032
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.658
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.046, 0.122, 0.96
No. of reflections	6992
No. of parameters	415
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.27, −0.23

Computer programs: *X-RED* and *X-AREA* (Stoe & Cie, 1996), *SIR2004* (Altomare *et al.*, 1999), *SHELXL2018* (Sheldrick, 2015) and *PLATON* (Spek, 2009).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

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full crystallographic data

IUCrData (2019). **4**, x191212 [https://doi.org/10.1107/S2414314619012124]

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Crystal data

$C_{17}H_{20}O_5$
M_r = 304.33
Triclinic, $P\overline{1}$
a = 7.5318 (5) Å
b = 11.0092 (6) Å
c = 18.1896 (12) Å
α = 85.600 (5)°
β = 87.945 (5)°
γ = 78.702 (5)°
V = 1474.34 (16) Å^3
Z = 4
$F(000)$ = 648
D_x = 1.371 Mg m^{-3}
Melting point: 367 K
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 5220 reflections
θ = 2.8–28.3°
μ = 0.10 mm^{-1}
T = 193 K
Block, colourless
0.31 × 0.21 × 0.13 mm

Data collection

Stoe IPDS 2T diffractometer
Radiation source: sealed X-ray tube, 12 x 0.4 mm long-fine focus
Detector resolution: 6.67 pixels mm^{-1}
rotation method scans
13532 measured reflections
6992 independent reflections
4168 reflections with $I > 2\sigma(I)$
R_{int} = 0.032
θ_{max} = 27.9°, θ_{min} = 2.8°
h = −9→9
k = −14→14
l = −22→23

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.122
S = 0.96
6992 reflections
415 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.065P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

Special details

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

Refinement. Hydrogen atoms attached to carbon atoms were placed at calculated positions and were refined in the riding-model approximation with isotropic displacement parameters. Hydroxyl hydrogen atoms were localized in difference maps and were refined without any constraints/restraints.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1A	0.9799 (2)	0.18167 (15)	0.12238 (9)	0.0296 (4)
C2A	1.0221 (3)	0.30034 (15)	0.11006 (10)	0.0340 (4)
H2A	1.137500	0.309233	0.090027	0.041*
C3A	0.8959 (3)	0.40467 (15)	0.12707 (9)	0.0326 (4)
C4A	0.7270 (3)	0.39390 (16)	0.15603 (9)	0.0321 (4)
C5A	0.6841 (2)	0.27607 (16)	0.16868 (9)	0.0312 (4)
C6A	0.8106 (2)	0.17168 (15)	0.15203 (9)	0.0303 (4)
H6A	0.780804	0.091848	0.161104	0.036*
O7A	0.92497 (19)	0.52477 (11)	0.11762 (7)	0.0415 (3)
C8A	1.1016 (3)	0.54224 (19)	0.09269 (13)	0.0485 (5)
H8A	1.122470	0.519130	0.041678	0.073*
H8B	1.193357	0.489944	0.124263	0.073*
H8C	1.109827	0.629598	0.095084	0.073*
O9A	0.5991 (2)	0.49493 (12)	0.17523 (8)	0.0413 (3)
H9A	0.626 (4)	0.564 (2)	0.1549 (16)	0.082 (9)*
O10A	0.51533 (18)	0.27390 (11)	0.19765 (8)	0.0428 (3)
C11A	0.4691 (3)	0.15525 (18)	0.21397 (13)	0.0477 (5)
H11A	0.474542	0.112132	0.168525	0.072*
H11B	0.346181	0.165727	0.235316	0.072*
H11C	0.554737	0.106225	0.249294	0.072*
C12A	1.1143 (2)	0.06707 (15)	0.10736 (9)	0.0287 (4)
C13A	1.1546 (2)	−0.02472 (15)	0.16586 (9)	0.0295 (4)
H13A	1.099942	−0.009578	0.213016	0.035*
C14A	1.2709 (2)	−0.13561 (15)	0.15652 (9)	0.0293 (4)
C15A	1.3497 (2)	−0.15842 (15)	0.08669 (10)	0.0297 (4)
C16A	1.3113 (2)	−0.06908 (16)	0.02949 (10)	0.0314 (4)
H16A	1.366084	−0.085124	−0.017537	0.038*
C17A	1.1942 (2)	0.04481 (15)	0.03803 (9)	0.0296 (4)
O18A	1.31709 (19)	−0.22968 (11)	0.21027 (7)	0.0375 (3)
C19A	1.2669 (3)	−0.20125 (18)	0.28398 (10)	0.0423 (5)
H19A	1.315309	−0.128816	0.295759	0.063*
H19B	1.316239	−0.272475	0.317834	0.063*
H19C	1.134578	−0.182842	0.289158	0.063*
O20A	1.46309 (18)	−0.27231 (11)	0.08059 (7)	0.0384 (3)
C21A	1.4887 (3)	−0.31556 (19)	0.00889 (11)	0.0458 (5)
H21A	1.370770	−0.316686	−0.011839	0.069*
H21B	1.562059	−0.399714	0.011582	0.069*
H21C	1.551004	−0.260204	−0.022681	0.069*
C22A	1.1508 (3)	0.13320 (16)	−0.02933 (10)	0.0364 (4)
H22A	1.191277	0.089587	−0.073735	0.055*
H22B	1.213269	0.202955	−0.027020	0.055*

H22C	1.019844	0.164474	−0.030977	0.055*
C1B	0.3944 (2)	0.27622 (14)	0.48112 (9)	0.0261 (4)
C2B	0.3044 (2)	0.33223 (15)	0.41793 (9)	0.0290 (4)
H2B	0.190413	0.314420	0.406677	0.035*
C3B	0.3824 (3)	0.41407 (15)	0.37162 (9)	0.0291 (4)
C4B	0.5515 (2)	0.43821 (14)	0.38575 (9)	0.0283 (4)
C5B	0.6380 (2)	0.38653 (14)	0.45020 (9)	0.0266 (4)
C6B	0.5587 (2)	0.30630 (14)	0.49753 (9)	0.0267 (4)
H6B	0.617609	0.271500	0.541746	0.032*
O7B	0.30278 (18)	0.47921 (11)	0.31022 (7)	0.0374 (3)
C8B	0.1337 (3)	0.4548 (2)	0.29006 (12)	0.0482 (5)
H8D	0.088563	0.509777	0.247053	0.072*
H8E	0.046793	0.469510	0.331326	0.072*
H8F	0.149065	0.368084	0.277882	0.072*
O9B	0.6339 (2)	0.51264 (11)	0.33720 (8)	0.0385 (3)
H9B	0.575 (3)	0.514 (2)	0.2964 (14)	0.067 (8)*
O10B	0.80286 (17)	0.41603 (11)	0.46072 (7)	0.0348 (3)
C11B	0.8942 (2)	0.36313 (16)	0.52674 (10)	0.0337 (4)
H11D	0.921109	0.272429	0.526178	0.051*
H11E	0.816650	0.387191	0.569692	0.051*
H11F	1.007510	0.393562	0.529543	0.051*
C12B	0.3161 (2)	0.18486 (15)	0.53019 (9)	0.0259 (4)
C13B	0.2925 (2)	0.20527 (15)	0.60577 (9)	0.0265 (4)
H13B	0.323504	0.277270	0.623403	0.032*
C14B	0.2250 (2)	0.12222 (15)	0.65462 (9)	0.0265 (4)
C15B	0.1776 (2)	0.01716 (15)	0.62843 (9)	0.0268 (4)
C16B	0.2012 (2)	−0.00287 (14)	0.55426 (9)	0.0276 (4)
H16B	0.169434	−0.074851	0.536936	0.033*
C17B	0.2700 (2)	0.07911 (15)	0.50431 (9)	0.0264 (4)
O18B	0.19864 (18)	0.13309 (10)	0.72899 (6)	0.0338 (3)
C19B	0.2269 (3)	0.24458 (17)	0.75752 (10)	0.0398 (5)
H19D	0.355463	0.249027	0.752602	0.060*
H19E	0.189646	0.245164	0.809695	0.060*
H19F	0.154858	0.316263	0.729890	0.060*
O20B	0.10809 (18)	−0.05989 (10)	0.67998 (6)	0.0336 (3)
C21B	0.1288 (3)	−0.18709 (15)	0.66423 (11)	0.0385 (5)
H21D	0.058455	−0.193066	0.620797	0.058*
H21E	0.085042	−0.234313	0.706616	0.058*
H21F	0.256938	−0.221171	0.654470	0.058*
C22B	0.3017 (2)	0.04457 (16)	0.42521 (9)	0.0308 (4)
H22D	0.205007	0.093332	0.394503	0.046*
H22E	0.301750	−0.044073	0.422886	0.046*
H22F	0.418809	0.062125	0.407079	0.046*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1A	0.0359 (10)	0.0294 (9)	0.0234 (9)	−0.0068 (8)	0.0002 (7)	−0.0016 (7)

C2A	0.0394 (11)	0.0321 (9)	0.0304 (10)	−0.0085 (8)	0.0043 (8)	−0.0008 (7)
C3A	0.0476 (12)	0.0266 (9)	0.0249 (9)	−0.0115 (8)	−0.0003 (8)	0.0002 (7)
C4A	0.0409 (11)	0.0289 (9)	0.0241 (9)	−0.0017 (8)	−0.0005 (8)	−0.0012 (7)
C5A	0.0336 (10)	0.0348 (9)	0.0247 (9)	−0.0064 (8)	0.0003 (8)	−0.0004 (7)
C6A	0.0383 (11)	0.0265 (8)	0.0265 (9)	−0.0078 (8)	−0.0014 (8)	0.0001 (7)
O7A	0.0514 (9)	0.0263 (6)	0.0471 (8)	−0.0108 (6)	0.0058 (7)	0.0002 (6)
C8A	0.0569 (14)	0.0379 (11)	0.0540 (14)	−0.0204 (10)	0.0114 (11)	−0.0008 (9)
O9A	0.0505 (9)	0.0273 (7)	0.0419 (8)	0.0004 (6)	0.0062 (7)	0.0008 (6)
O10A	0.0381 (8)	0.0344 (7)	0.0549 (9)	−0.0070 (6)	0.0100 (7)	−0.0007 (6)
C11A	0.0378 (12)	0.0423 (11)	0.0633 (15)	−0.0130 (9)	0.0102 (10)	0.0032 (10)
C12A	0.0320 (10)	0.0286 (8)	0.0269 (9)	−0.0091 (7)	0.0007 (7)	−0.0023 (7)
C13A	0.0344 (10)	0.0307 (9)	0.0239 (9)	−0.0078 (8)	0.0041 (7)	−0.0033 (7)
C14A	0.0345 (10)	0.0300 (9)	0.0242 (9)	−0.0087 (8)	−0.0008 (7)	0.0002 (7)
C15A	0.0323 (10)	0.0271 (8)	0.0299 (10)	−0.0049 (7)	0.0014 (8)	−0.0063 (7)
C16A	0.0350 (10)	0.0348 (9)	0.0253 (9)	−0.0088 (8)	0.0050 (8)	−0.0046 (7)
C17A	0.0331 (10)	0.0314 (9)	0.0261 (9)	−0.0109 (7)	0.0007 (7)	−0.0006 (7)
O18A	0.0509 (8)	0.0327 (6)	0.0248 (7)	0.0000 (6)	0.0014 (6)	0.0010 (5)
C19A	0.0521 (13)	0.0442 (11)	0.0246 (10)	0.0024 (9)	0.0019 (9)	0.0038 (8)
O20A	0.0469 (8)	0.0346 (7)	0.0296 (7)	0.0030 (6)	0.0017 (6)	−0.0063 (5)
C21A	0.0593 (14)	0.0429 (11)	0.0329 (11)	−0.0011 (10)	0.0014 (10)	−0.0114 (9)
C22A	0.0468 (12)	0.0338 (9)	0.0288 (10)	−0.0101 (8)	0.0019 (8)	0.0008 (8)
C1B	0.0297 (9)	0.0249 (8)	0.0229 (9)	−0.0033 (7)	0.0020 (7)	−0.0025 (6)
C2B	0.0282 (9)	0.0297 (9)	0.0280 (9)	−0.0034 (7)	−0.0003 (7)	−0.0005 (7)
C3B	0.0369 (10)	0.0248 (8)	0.0224 (9)	0.0012 (7)	0.0012 (7)	−0.0011 (7)
C4B	0.0383 (10)	0.0219 (8)	0.0249 (9)	−0.0078 (7)	0.0077 (7)	−0.0018 (6)
C5B	0.0276 (9)	0.0253 (8)	0.0275 (9)	−0.0055 (7)	0.0033 (7)	−0.0051 (7)
C6B	0.0295 (9)	0.0261 (8)	0.0229 (9)	−0.0019 (7)	0.0003 (7)	−0.0009 (7)
O7B	0.0448 (8)	0.0361 (7)	0.0278 (7)	−0.0022 (6)	−0.0053 (6)	0.0068 (5)
C8B	0.0519 (14)	0.0510 (12)	0.0389 (12)	−0.0035 (10)	−0.0156 (10)	0.0040 (9)
O9B	0.0515 (9)	0.0376 (7)	0.0282 (7)	−0.0166 (6)	0.0039 (7)	0.0052 (6)
O10B	0.0342 (7)	0.0383 (7)	0.0336 (7)	−0.0123 (6)	0.0008 (6)	0.0005 (5)
C11B	0.0305 (10)	0.0350 (9)	0.0350 (10)	−0.0053 (8)	−0.0012 (8)	−0.0012 (8)
C12B	0.0243 (9)	0.0273 (8)	0.0253 (9)	−0.0035 (7)	−0.0001 (7)	−0.0001 (7)
C13B	0.0268 (9)	0.0250 (8)	0.0280 (9)	−0.0048 (7)	0.0004 (7)	−0.0041 (7)
C14B	0.0273 (9)	0.0279 (8)	0.0225 (8)	−0.0012 (7)	0.0015 (7)	−0.0028 (7)
C15B	0.0275 (9)	0.0260 (8)	0.0262 (9)	−0.0053 (7)	0.0050 (7)	0.0004 (7)
C16B	0.0291 (9)	0.0238 (8)	0.0302 (9)	−0.0050 (7)	0.0000 (7)	−0.0048 (7)
C17B	0.0254 (9)	0.0272 (8)	0.0248 (9)	−0.0009 (7)	0.0004 (7)	−0.0022 (7)
O18B	0.0510 (8)	0.0298 (6)	0.0228 (6)	−0.0133 (6)	0.0061 (6)	−0.0051 (5)
C19B	0.0592 (13)	0.0350 (10)	0.0279 (10)	−0.0139 (9)	0.0060 (9)	−0.0101 (8)
O20B	0.0477 (8)	0.0250 (6)	0.0292 (7)	−0.0112 (5)	0.0090 (6)	−0.0025 (5)
C21B	0.0539 (13)	0.0248 (9)	0.0377 (11)	−0.0105 (9)	0.0009 (9)	−0.0017 (8)
C22B	0.0333 (10)	0.0324 (9)	0.0257 (9)	−0.0036 (8)	0.0016 (7)	−0.0039 (7)

Geometric parameters (Å, º)

C1A—C6A	1.388 (2)	C1B—C6B	1.390 (2)
C1A—C2A	1.404 (2)	C1B—C2B	1.394 (2)

C1A—C12A	1.491 (2)	C1B—C12B	1.487 (2)
C2A—C3A	1.387 (2)	C2B—C3B	1.386 (2)
C2A—H2A	0.9500	C2B—H2B	0.9500
C3A—O7A	1.379 (2)	C3B—O7B	1.370 (2)
C3A—C4A	1.383 (3)	C3B—C4B	1.388 (3)
C4A—O9A	1.379 (2)	C4B—O9B	1.373 (2)
C4A—C5A	1.397 (2)	C4B—C5B	1.389 (2)
C5A—O10A	1.362 (2)	C5B—O10B	1.367 (2)
C5A—C6A	1.387 (2)	C5B—C6B	1.390 (2)
C6A—H6A	0.9500	C6B—H6B	0.9500
O7A—C8A	1.434 (2)	O7B—C8B	1.418 (2)
C8A—H8A	0.9800	C8B—H8D	0.9800
C8A—H8B	0.9800	C8B—H8E	0.9800
C8A—H8C	0.9800	C8B—H8F	0.9800
O9A—H9A	0.87 (3)	O9B—H9B	0.88 (3)
O10A—C11A	1.424 (2)	O10B—C11B	1.432 (2)
C11A—H11A	0.9800	C11B—H11D	0.9800
C11A—H11B	0.9800	C11B—H11E	0.9800
C11A—H11C	0.9800	C11B—H11F	0.9800
C12A—C17A	1.396 (2)	C12B—C17B	1.397 (2)
C12A—C13A	1.408 (2)	C12B—C13B	1.409 (2)
C13A—C14A	1.374 (2)	C13B—C14B	1.382 (2)
C13A—H13A	0.9500	C13B—H13B	0.9500
C14A—O18A	1.370 (2)	C14B—O18B	1.371 (2)
C14A—C15A	1.402 (2)	C14B—C15B	1.397 (2)
C15A—C16A	1.374 (2)	C15B—O20B	1.375 (2)
C15A—O20A	1.3816 (19)	C15B—C16B	1.383 (2)
C16A—C17A	1.400 (2)	C16B—C17B	1.390 (2)
C16A—H16A	0.9500	C16B—H16B	0.9500
C17A—C22A	1.509 (2)	C17B—C22B	1.515 (2)
O18A—C19A	1.421 (2)	O18B—C19B	1.424 (2)
C19A—H19A	0.9800	C19B—H19D	0.9800
C19A—H19B	0.9800	C19B—H19E	0.9800
C19A—H19C	0.9800	C19B—H19F	0.9800
O20A—C21A	1.416 (2)	O20B—C21B	1.4284 (19)
C21A—H21A	0.9800	C21B—H21D	0.9800
C21A—H21B	0.9800	C21B—H21E	0.9800
C21A—H21C	0.9800	C21B—H21F	0.9800
C22A—H22A	0.9800	C22B—H22D	0.9800
C22A—H22B	0.9800	C22B—H22E	0.9800
C22A—H22C	0.9800	C22B—H22F	0.9800

C6A—C1A—C2A	118.54 (16)	C6B—C1B—C2B	119.18 (16)
C6A—C1A—C12A	119.64 (15)	C6B—C1B—C12B	120.36 (15)
C2A—C1A—C12A	121.77 (17)	C2B—C1B—C12B	120.46 (16)
C3A—C2A—C1A	120.20 (18)	C3B—C2B—C1B	119.59 (17)
C3A—C2A—H2A	119.9	C3B—C2B—H2B	120.2
C1A—C2A—H2A	119.9	C1B—C2B—H2B	120.2

O7A—C3A—C4A	114.59 (15)	O7B—C3B—C2B	124.74 (17)
O7A—C3A—C2A	124.54 (17)	O7B—C3B—C4B	114.14 (15)
C4A—C3A—C2A	120.86 (16)	C2B—C3B—C4B	121.11 (16)
O9A—C4A—C3A	122.70 (16)	O9B—C4B—C3B	121.01 (16)
O9A—C4A—C5A	117.98 (17)	O9B—C4B—C5B	119.67 (17)
C3A—C4A—C5A	119.29 (16)	C3B—C4B—C5B	119.32 (16)
O10A—C5A—C6A	124.69 (16)	O10B—C5B—C4B	115.89 (15)
O10A—C5A—C4A	115.43 (15)	O10B—C5B—C6B	124.43 (16)
C6A—C5A—C4A	119.87 (17)	C4B—C5B—C6B	119.63 (17)
C5A—C6A—C1A	121.23 (16)	C5B—C6B—C1B	120.98 (16)
C5A—C6A—H6A	119.4	C5B—C6B—H6B	119.5
C1A—C6A—H6A	119.4	C1B—C6B—H6B	119.5
C3A—O7A—C8A	117.60 (14)	C3B—O7B—C8B	117.41 (15)
O7A—C8A—H8A	109.5	O7B—C8B—H8D	109.5
O7A—C8A—H8B	109.5	O7B—C8B—H8E	109.5
H8A—C8A—H8B	109.5	H8D—C8B—H8E	109.5
O7A—C8A—H8C	109.5	O7B—C8B—H8F	109.5
H8A—C8A—H8C	109.5	H8D—C8B—H8F	109.5
H8B—C8A—H8C	109.5	H8E—C8B—H8F	109.5
C4A—O9A—H9A	110.6 (18)	C4B—O9B—H9B	103.3 (17)
C5A—O10A—C11A	117.11 (14)	C5B—O10B—C11B	116.63 (14)
O10A—C11A—H11A	109.5	O10B—C11B—H11D	109.5
O10A—C11A—H11B	109.5	O10B—C11B—H11E	109.5
H11A—C11A—H11B	109.5	H11D—C11B—H11E	109.5
O10A—C11A—H11C	109.5	O10B—C11B—H11F	109.5
H11A—C11A—H11C	109.5	H11D—C11B—H11F	109.5
H11B—C11A—H11C	109.5	H11E—C11B—H11F	109.5
C17A—C12A—C13A	119.18 (15)	C17B—C12B—C13B	119.40 (15)
C17A—C12A—C1A	123.25 (15)	C17B—C12B—C1B	122.42 (14)
C13A—C12A—C1A	117.51 (15)	C13B—C12B—C1B	118.17 (15)
C14A—C13A—C12A	121.72 (16)	C14B—C13B—C12B	121.04 (15)
C14A—C13A—H13A	119.1	C14B—C13B—H13B	119.5
C12A—C13A—H13A	119.1	C12B—C13B—H13B	119.5
O18A—C14A—C13A	125.46 (15)	O18B—C14B—C13B	125.47 (15)
O18A—C14A—C15A	115.47 (15)	O18B—C14B—C15B	115.14 (15)
C13A—C14A—C15A	119.07 (16)	C13B—C14B—C15B	119.38 (15)
C16A—C15A—O20A	124.26 (15)	O20B—C15B—C16B	124.41 (15)
C16A—C15A—C14A	119.45 (15)	O20B—C15B—C14B	116.08 (14)
O20A—C15A—C14A	116.29 (15)	C16B—C15B—C14B	119.51 (15)
C15A—C16A—C17A	122.33 (16)	C15B—C16B—C17B	122.00 (16)
C15A—C16A—H16A	118.8	C15B—C16B—H16B	119.0
C17A—C16A—H16A	118.8	C17B—C16B—H16B	119.0
C12A—C17A—C16A	118.25 (15)	C16B—C17B—C12B	118.67 (15)
C12A—C17A—C22A	123.36 (16)	C16B—C17B—C22B	117.84 (15)
C16A—C17A—C22A	118.25 (15)	C12B—C17B—C22B	123.37 (16)
C14A—O18A—C19A	116.66 (13)	C14B—O18B—C19B	117.53 (13)
O18A—C19A—H19A	109.5	O18B—C19B—H19D	109.5
O18A—C19A—H19B	109.5	O18B—C19B—H19E	109.5

H19A—C19A—H19B	109.5	H19D—C19B—H19E	109.5
O18A—C19A—H19C	109.5	O18B—C19B—H19F	109.5
H19A—C19A—H19C	109.5	H19D—C19B—H19F	109.5
H19B—C19A—H19C	109.5	H19E—C19B—H19F	109.5
C15A—O20A—C21A	116.33 (14)	C15B—O20B—C21B	116.76 (12)
O20A—C21A—H21A	109.5	O20B—C21B—H21D	109.5
O20A—C21A—H21B	109.5	O20B—C21B—H21E	109.5
H21A—C21A—H21B	109.5	H21D—C21B—H21E	109.5
O20A—C21A—H21C	109.5	O20B—C21B—H21F	109.5
H21A—C21A—H21C	109.5	H21D—C21B—H21F	109.5
H21B—C21A—H21C	109.5	H21E—C21B—H21F	109.5
C17A—C22A—H22A	109.5	C17B—C22B—H22D	109.5
C17A—C22A—H22B	109.5	C17B—C22B—H22E	109.5
H22A—C22A—H22B	109.5	H22D—C22B—H22E	109.5
C17A—C22A—H22C	109.5	C17B—C22B—H22F	109.5
H22A—C22A—H22C	109.5	H22D—C22B—H22F	109.5
H22B—C22A—H22C	109.5	H22E—C22B—H22F	109.5

C6A—C1A—C2A—C3A	−0.3 (3)	C6B—C1B—C2B—C3B	1.8 (2)
C12A—C1A—C2A—C3A	−177.76 (16)	C12B—C1B—C2B—C3B	−177.90 (14)
C1A—C2A—C3A—O7A	179.41 (17)	C1B—C2B—C3B—O7B	−176.54 (15)
C1A—C2A—C3A—C4A	−0.2 (3)	C1B—C2B—C3B—C4B	2.1 (2)
O7A—C3A—C4A—O9A	−1.2 (3)	O7B—C3B—C4B—O9B	−5.7 (2)
C2A—C3A—C4A—O9A	178.50 (16)	C2B—C3B—C4B—O9B	175.51 (14)
O7A—C3A—C4A—C5A	−179.23 (15)	O7B—C3B—C4B—C5B	174.06 (14)
C2A—C3A—C4A—C5A	0.5 (3)	C2B—C3B—C4B—C5B	−4.8 (2)
O9A—C4A—C5A—O10A	1.5 (2)	O9B—C4B—C5B—O10B	0.7 (2)
C3A—C4A—C5A—O10A	179.66 (16)	C3B—C4B—C5B—O10B	−179.08 (14)
O9A—C4A—C5A—C6A	−178.27 (16)	O9B—C4B—C5B—C6B	−176.83 (14)
C3A—C4A—C5A—C6A	−0.1 (3)	C3B—C4B—C5B—C6B	3.4 (2)
O10A—C5A—C6A—C1A	179.81 (17)	O10B—C5B—C6B—C1B	−176.82 (14)
C4A—C5A—C6A—C1A	−0.4 (3)	C4B—C5B—C6B—C1B	0.4 (2)
C2A—C1A—C6A—C5A	0.6 (3)	C2B—C1B—C6B—C5B	−3.0 (2)
C12A—C1A—C6A—C5A	178.15 (16)	C12B—C1B—C6B—C5B	176.61 (14)
C4A—C3A—O7A—C8A	175.72 (17)	C2B—C3B—O7B—C8B	−4.4 (2)
C2A—C3A—O7A—C8A	−3.9 (3)	C4B—C3B—O7B—C8B	176.86 (15)
C6A—C5A—O10A—C11A	2.1 (3)	C4B—C5B—O10B—C11B	−179.75 (14)
C4A—C5A—O10A—C11A	−177.66 (17)	C6B—C5B—O10B—C11B	−2.4 (2)
C6A—C1A—C12A—C17A	123.53 (19)	C6B—C1B—C12B—C17B	−124.34 (18)
C2A—C1A—C12A—C17A	−59.0 (2)	C2B—C1B—C12B—C17B	55.3 (2)
C6A—C1A—C12A—C13A	−53.5 (2)	C6B—C1B—C12B—C13B	54.1 (2)
C2A—C1A—C12A—C13A	123.95 (19)	C2B—C1B—C12B—C13B	−126.26 (17)
C17A—C12A—C13A—C14A	0.1 (3)	C17B—C12B—C13B—C14B	0.2 (2)
C1A—C12A—C13A—C14A	177.27 (17)	C1B—C12B—C13B—C14B	−178.29 (15)
C12A—C13A—C14A—O18A	−179.88 (17)	C12B—C13B—C14B—O18B	179.38 (16)
C12A—C13A—C14A—C15A	−0.8 (3)	C12B—C13B—C14B—C15B	−0.7 (2)
O18A—C14A—C15A—C16A	−179.86 (16)	O18B—C14B—C15B—O20B	1.1 (2)
C13A—C14A—C15A—C16A	0.9 (3)	C13B—C14B—C15B—O20B	−178.75 (15)

O18A—C14A—C15A—O20A	0.2 (2)	O18B—C14B—C15B—C16B	−179.25 (15)
C13A—C14A—C15A—O20A	−179.01 (16)	C13B—C14B—C15B—C16B	0.9 (2)
O20A—C15A—C16A—C17A	179.45 (16)	O20B—C15B—C16B—C17B	179.13 (16)
C14A—C15A—C16A—C17A	−0.5 (3)	C14B—C15B—C16B—C17B	−0.4 (3)
C13A—C12A—C17A—C16A	0.3 (3)	C15B—C16B—C17B—C12B	−0.1 (3)
C1A—C12A—C17A—C16A	−176.64 (17)	C15B—C16B—C17B—C22B	176.21 (16)
C13A—C12A—C17A—C22A	175.88 (17)	C13B—C12B—C17B—C16B	0.2 (2)
C1A—C12A—C17A—C22A	−1.1 (3)	C1B—C12B—C17B—C16B	178.64 (16)
C15A—C16A—C17A—C12A	−0.1 (3)	C13B—C12B—C17B—C22B	−175.87 (15)
C15A—C16A—C17A—C22A	−175.92 (17)	C1B—C12B—C17B—C22B	2.5 (3)
C13A—C14A—O18A—C19A	−12.0 (3)	C13B—C14B—O18B—C19B	6.2 (3)
C15A—C14A—O18A—C19A	168.81 (17)	C15B—C14B—O18B—C19B	−173.66 (15)
C16A—C15A—O20A—C21A	−22.8 (3)	C16B—C15B—O20B—C21B	25.8 (2)
C14A—C15A—O20A—C21A	157.19 (18)	C14B—C15B—O20B—C21B	−154.65 (15)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O9*A*—H9*A*···O7*A*	0.87 (3)	2.30 (3)	2.703 (2)	108 (2)
O9*B*—H9*B*···O7*B*	0.88 (3)	2.16 (3)	2.659 (2)	116 (2)
O9*A*—H9*A*···O20*A*^{i}	0.87 (3)	2.34 (3)	3.0000 (19)	133 (2)
O9*B*—H9*B*···O9*A*	0.88 (3)	2.23 (3)	2.992 (2)	146 (2)
C13*A*—H13*A*···O20*B*^{ii}	0.95	2.52	3.453 (2)	166
C13*B*—H13*B*···O9*B*^{iii}	0.95	2.55	3.498 (2)	172
C19*A*—H19*C*···O18*B*^{ii}	0.98	2.49	3.452 (3)	167
C19*B*—H19*D*···O18*A*^{iv}	0.98	2.54	3.475 (3)	159

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