Acta Cryst E CRYSTALLOGRAPHIC COMMUNICATIONS

ISSN 2056-9890

Crystal structures of 2-acetyl-4-ethynylphenol and 2-acetyl-4-(3-hydroxy-3-methylbut-1-yn-1-yl)-phenol

Jörg Hübscher, Robert Rosin, Wilhelm Seichter and Edwin Weber*

Institut für Organische Chemie, TU Bergakademie Freiberg, Leipziger Strasse 29, D-09596 Freiberg/Sachsen, Germany. *Correspondence e-mail: edwin.weber@chemie-tu.freiberg.de

Received 29 July 2016
Accepted 22 August 2016

Edited by H. Stoeckli-Evans, University of Neuchâtel, Switzerland

Keywords: crystal structure; 2-acetylphenol; ethynyl; dimethylhydroxymethyl; hydrogen bonding; C—H···π interactions.

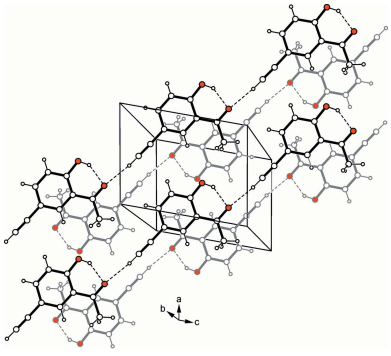
CCDC references: 1500395; 1500394

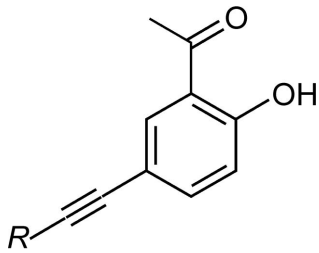
Supporting information: this article has supporting information at journals.iucr.org/e

In the title compounds, $C_{10}H_8O_2$, (I), and $C_{13}H_{14}O_3$, (II), the 2-acetyl-4-ethynylphenol unit displays a planar geometry, which is stabilized by an intramolecular O—H···O hydrogen bond. The crystal structure of (I) is constructed of infinite strands, along [101], of C—H···O=C hydrogen-bonded molecules, which in turn are linked by C—H···π interactions. In the crystal of (II), which crystallized with three independent molecules per asymmetric unit, the non-polar parts of the molecules form hydrophobic layered domains, parallel to $(10\bar{1})$, which are separated by the polar groups. While the 2-acetylphenol part of the molecules are involved in O—H···O=C hydrogen bonding, the ternary OH groups creates a cyclic pattern of O—H···O hydrogen bonds.

1. Chemical context

2-Acetylphenol and its derivatives are well known for their efficiency in the complexation of transition metal ions (Weber, 1977; Duckworth & Stephenson, 1969; Ali *et al.*, 2005). Such molecules, endowed with a 2-acetylphenol moiety, have been used as molecular linkers for the construction of coordination polymers and related porous framework structures (Hübscher *et al.*, 2013; Günthel *et al.*, 2015) that are the subject of great topical interest (MacGillivray, 2010; Furukawa *et al.*, 2013; Eddaoudi *et al.*, 2015). A corresponding linker design features a structure with terminal chelating 2-acetylphenol units attached to a linear central segment. In the course of the synthesis of respective linkers, the 2-acetylphenol derivatives (I) and (II), being substituted acetylenically in the 4-position, are important intermediates (Hübscher *et al.*, 2013). However, these compounds are not only of experimental preparative relevance but also show interesting structures in the crystalline state, as discussed in the present communication.





(I) R = H
(II) R = $C(CH_3)_2OH$

OPEN ACCESS

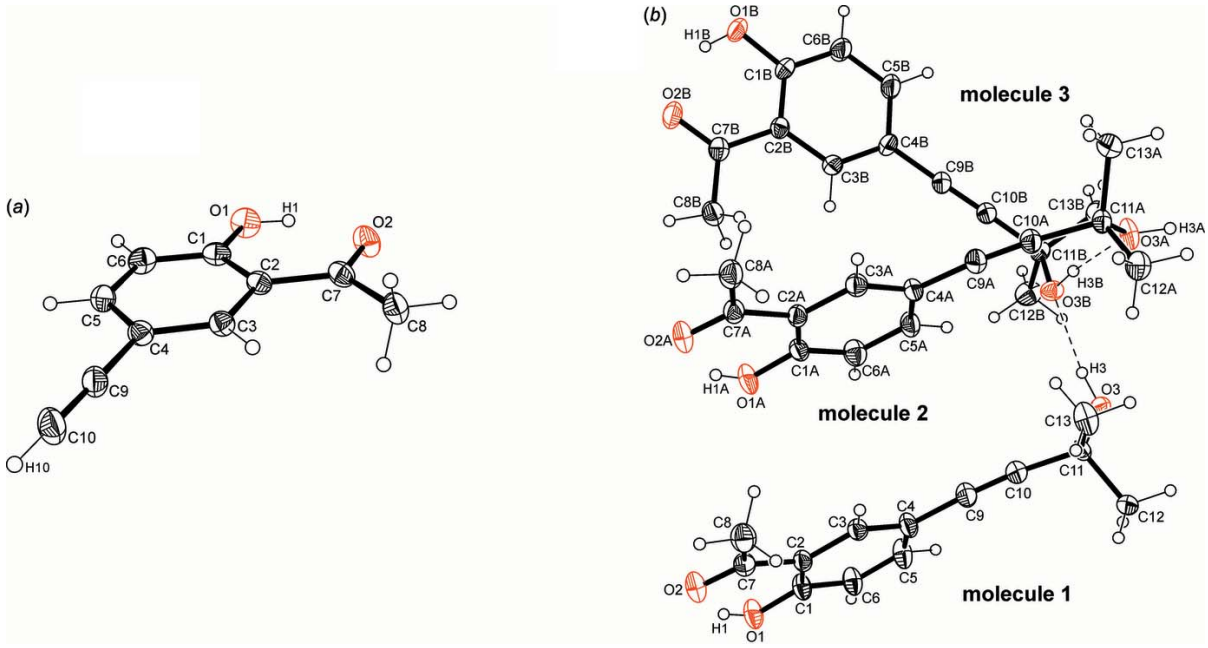


Figure 1
Perspective view of the molecular structure of the title compounds, (*a*) (I) and (*b*) (II), with the atom labelling. Displacement parameters are drawn at the 50% probability level.

2. Structural commentary

The crystal structures of the title compounds (I) and (II), crystallize in the space groups $P\overline{1}$ and $P2_1/c$, respectively. Perspective views of the molecules are depicted in Fig. 1. In (I) the asymmetric part of the unit cell contains one molecule (Fig. 1*a*). As a result of the presence of an intramolecular O–H···O hydrogen bond, the molecule has an almost planar geometry with largest atomic distances from the mean plane being −0.034 (1) Å for atom C5 and 0.069 (1) Å for atom O1. Because of substituent effects, the bond distances within the aromatic ring of the molecule deviate significantly from those observed in the polymorphous structures of ethynylbenzene (Dziubek *et al.* 2007; Thakur *et al.* 2010). Compound (II) crystallizes with three independent and conformationally non-equivalent molecules in the asymmetric unit. The molecules differ in their geometries around the dimethylhydroxymethyl structural element. These differences are expressed by the torsion angle along the atomic sequences $C_{ethynyl}-C-O-H$ which are 72.1 (2) and 83.9 (2)° (*gauche*) for molecules 1 and 3 and 173.0 (2)° (*anti*) for molecule 2 (Fig. 1*b*). The ethynyl segment of the molecules also deviates from linearity, possibly because of packing forces and intermolecular interactions.

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

*Cg*1 is the centroid of the C1–C6 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O2	0.84	1.83	2.5696 (11)	146
C10—H10···O2^{i}	0.95	2.28	3.2214 (14)	171
C8—H8*C*···*Cg*1^{ii}	0.97	2.72	3.6024 (12)	150

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

3. Supramolecular features

Infinite strands of C—H···O hydrogen-bonded molecules [*d*(H···O) 2.28 Å] (Desiraju & Steiner, 1999) running along [101] represent the basic supramolecular aggregates of the crystal structure of (I). Within a given strand, the acetylenic hydrogen acts as a donor and the acyl oxygen as an acceptor site (Fig. 2 and Table 1). A view of the crystal packing reveals a layered arrangement of the molecular chains in the *ac* plane.

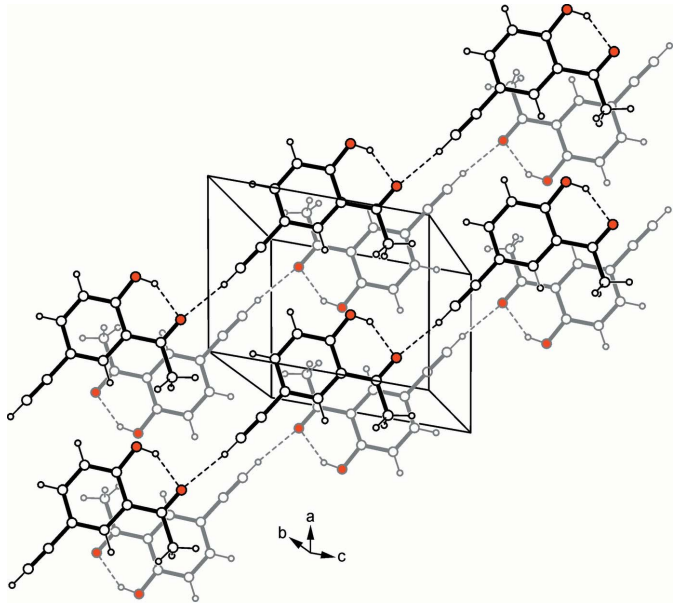


Figure 2
A partial view of the crystal packing of compound (I). Hydrogen bonds are shown as dashed lines (see Table 1), and O atoms as red circles.

Table 2
Hydrogen-bond geometry (Å, °) for (II).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O2	0.84	1.83	2.5639 (16)	145
O1—H1···O2A^{i}	0.84	2.60	3.1129 (15)	121
O3—H3···O3B^{ii}	0.84	1.90	2.7300 (12)	171
O1A—H1A···O2A	0.84	1.85	2.5832 (15)	145
O1A—H1A···O2B^{iii}	0.84	2.53	3.0303 (16)	119
O3A—H3A1···O3	0.84	1.99	2.8262 (13)	176
O1B—H1B···O2B	0.84	1.83	2.5611 (16)	145
O3B—H3B···O3A^{iv}	0.84	1.99	2.8203 (13)	172

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

As depicted in Fig. 2, the crystal of (I) lacks π–π arene stacking (Martinez & Iverson, 2012). Instead, the methyl hydrogen H8C forms a weak C—H···π contact [d(H···π) 2.72 Å; Table 1] (Nishio *et al.*, 2009), which connects the chains of consecutive layers.

Because of the presence of a dimethylhydroxymethyl residue as a terminal group, the crystal structure of (II) is composed of hexamers of O—H···O hydrogen-bonded molecules [d(H···O) 1.90, 1.99 Å], which create a cyclic hydrogen-bond motif of graph set $R_6^6(12)$ (Table 2 and Fig. 3). Furthermore, the hexamers are interconnected by weaker O—H···O hydrogen bonds involving the phenolic OH hydrogens H1 and H1A as donors and the acyl oxygen atoms O2A and O2B as acceptors [d(H···O) 2.60, 2.53 Å], forming layers parallel to (10$\bar{1}$). The molecules pack with the dimethylhydroxymethyl groups assembled in layered structure domains, separated by the non-polar parts of the molecules (Fig. 3).

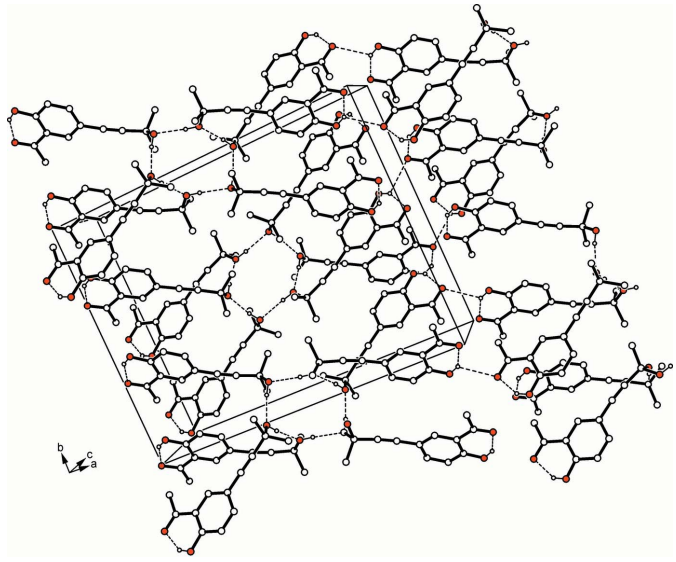


Figure 3
The crystal packing of compound (II), viewed along the c axis. Hydrogen bonds are shown as dashed lines (see Table 2) and C-bound H atoms have been omitted for clarity.

4. Database survey

A search in the Cambridge Structural Database (CSD, Version 5.37, update November 2015; Groom *et al.*, 2016) for *p*-substituted 2-acetylphenols excluding their co-crystals and complexes yielded 23 hits, only two of them containing the 4-ethynyl-2-acetylphenol element, namely 1,1′-[1,4-phenylenebis(ethyne-2,1-diyl(6-hydroxy-3,1-phenylene)]diethanone and 1,1′-[ethyne-1,2-diylbis(6-hydroxy-3,1-phenylene)]diethanone [CSD refcodes: TEVLAJ and TEVLEN; Hübscher *et al.*, 2013]. The presence of an acceptor instead of a donor substituent in *p*-position of the phenolic OH as in 4-cyano-2-acetophenol [LIWFUT; Filarowski *et al.*, 2007)], 4-nitro-2-acetophenol [GADBAP; Hibbs *et al.*, 2003)] and 4-chloro-2-acetophenol [DACGOE; Filarowski *et al.*, 2004)] markedly influences the pattern of non-covalent intermolecular bonding. In the first two cases, the crystal is constructed of the same kind of molecular strands in which the molecules are linked *via* C—H_{arene}···O=C bonding. Inter-strand association is accomplished by π–π stacking forces. In these structures, the *p*-substituents are excluded from intermolecular interactions. In the latter compound, the chlorine atom acts as a bifurcated acceptor for C—H···Cl bonding (Thallapally & Nangia, 2001), thus creating double strand-like supramolecular aggregates. Neither the OH nor the acetyl group are involved in intermolecular bonding.

5. Synthesis and crystallization

Compounds (I) and (II) were synthesized following a literature procedure (Hübscher *et al.*, 2013). This involves the reaction of 2-acetyl-4-bromophenol with 2-methylbut-3-yn-2-ol (MEBYNOL) using a Sonogashira–Hagihara coupling process to give (II). A deblocking reaction of (II) under basic conditions yielded (I). Crystals of (I) and (II), suitable for X-ray diffraction analysis, were obtained from solutions of *n*-hexane/ethyl acetate (3:1, *v*/*v*) and cyclohexane, respectively, upon slow evaporation of the solvents at room temperature.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. All H atoms were placed geometrically in idealized positions and allowed to ride on their parent atoms: O—H = 0.84 and C—H = 0.95–98 Å with U_{iso}(H) = 1.5U_{eq}(C-methyl and O) and 1.2U_{eq}(C) for other H atoms.

Acknowledgements

We acknowledge the financial support within the Cluster of Excellence 'Structure Design of Novel High-Performance Materials *via* Atomic Design and Defect Engineering (ADDE)' provided to us by the European Union (European Regional Development Fund) and by the Ministry of Science and Art of Saxony (SMWK) as well as by the Deutsche Forschungsgemeinschaft (DFG Priority Program 1362 'Porous Metal-Organic Frameworks').

Table 3
Experimental details.

	(I)	(II)
Crystal data		
Chemical formula	$C_{10}H_8O_2$	$C_{13}H_{14}O_3$
M_r	160.16	218.24
Crystal system, space group	Triclinic, $P\overline{1}$	Monoclinic, $P2_1/c$
Temperature (K)	153	153
a, b, c (Å)	6.9725 (1), 7.3174 (1), 8.9189 (2)	22.5787 (6), 16.9306 (4), 9.2849 (2)
α, β, γ (°)	69.241 (1), 79.975 (1), 70.127 (1)	90, 101.815 (1), 90
V (Å^3)	399.42 (1)	3474.15 (14)
Z	2	12
Radiation type	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	0.09	0.09
Crystal size (mm)	0.55 × 0.41 × 0.15	0.36 × 0.18 × 0.09
Data collection		
Diffractometer	Bruker APEXII CCD area detector	Bruker APEXII CCD area detector
Absorption correction	Multi-scan (*SADABS*; Bruker, 2008)	Multi-scan (*SADABS*; Bruker, 2008)
T_{min}, T_{max}	0.956, 0.988	0.969, 0.992
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	8959, 2133, 1881	37857, 9244, 5584
R_{int}	0.018	0.043
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.684	0.684
Refinement		
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.038, 0.115, 1.06	0.047, 0.117, 0.89
No. of reflections	2133	9244
No. of parameters	111	448
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.39, −0.19	0.28, −0.22

Computer programs: *APEX2* and *SAINT* (Bruker, 2008), *SHELXS97* and *SHELXTL* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015) and *ORTEP-3 for Windows* (Farrugia, 2012).

References

Ali, H. M., Abdul Halim, S. N. & Ng, S. W. (2005). *Acta Cryst.* E**61**, m1429–m1430.

Bruker (2008). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.

Desiraju, G. R. & Steiner, T. (1999). In *The Weak Hydrogen Bond*. Oxford University Press.

Duckworth, V. F. & Stephenson, N. C. (1969). *Acta Cryst.* B**25**, 2245–2254.

Dziubek, K., Podsiadło, M. & Katrusiak, A. (2007). *J. Am. Chem. Soc.* **129**, 12620–12621.

Eddaoudi, M., Sava, D. F., Eubank, J. F., Adil, K. & Guillerm, V. (2015). *Chem. Soc. Rev.* **44**, 228–249.

Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.

Filarowski, A., Kochel, A., Hansen, P. E., Urbanowicz, A. & Szymborska, K. (2007). *J. Mol. Struct.* **844–845**, 77–82.

Filarowski, A., Koll, A., Kochel, A., Kalenik, J. & Hansen, P. E. (2004). *J. Mol. Struct.* **700**, 67–72.

Furukawa, H., Cordova, K. E., O'Keeffe, M. & Yaghi, O. M. (2013). *Science*, **341**, 1230444.

Groom, C. R., Bruno, I. J., Lightfoot, M. P. & Ward, S. C. (2016). *Acta Cryst.* B**72**, 171–179.

Günthel, M., Hübscher, J., Dittrich, R., Weber, E., Joseph, Y. & Mertens, F. (2015). *J. Polym. Sci. Part B Polym. Phys.* **53**, 335–344.

Hibbs, D. E., Overgaard, J. & Piltz, R. O. (2003). *Org. Biomol. Chem.* **1**, 1191–1198.

Hübscher, J., Günthel, M., Rosin, R., Seichter, W., Mertens, F. & Weber, E. (2013). *Z. Naturforsch. Teil B*, **68**, 214–222.

MacGillivray, L. R. (2010). *Metal-Organic Frameworks*. Hoboken: Wiley.

Martinez, C. R. & Iverson, B. L. (2012). *Chem. Sci.* **3**, 2191–2201.

Nishio, M., Umezawa, Y., Honda, K., Tsuboyama, S. & Suezawa, H. (2009). *CrystEngComm*, **11**, 1757–1788.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

Sheldrick, G. M. (2015). *Acta Cryst.* C**71**, 3–8.

Thakur, T. S., Sathishkumar, R., Dikundwar, A. G., Guru Row, T. N. & Desiraju, G. R. (2010). *Cryst. Growth Des.* **10**, 4246–4249.

Thallapally, P. K. & Nangia, A. (2001). *CrystEngComm*, **3**, 114–119.

Weber, J. H. (1977). *Synth. React. Inorg. Met.-Org. Chem.* **7**, 243–252.

supporting information

Acta Cryst. (2016). E**72**, 1370-1373 [https://doi.org/10.1107/S2056989016013451]

Crystal structures of 2-acetyl-4-ethynylphenol and 2-acetyl-4-(3-hydroxy-3-methylbut-1-yn-1-yl)phenol

Jörg Hübscher, Robert Rosin, Wilhelm Seichter and Edwin Weber

Computing details

For both compounds, data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT* (Bruker, 2008); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

(I) 2-Acetyl-4-ethynylphenol

Crystal data

$C_{10}H_8O_2$
$M_r = 160.16$
Triclinic, $P\overline{1}$
$a = 6.9725$ (1) Å
$b = 7.3174$ (1) Å
$c = 8.9189$ (2) Å
$\alpha = 69.241$ (1)°
$\beta = 79.975$ (1)°
$\gamma = 70.127$ (1)°
$V = 399.42$ (1) Å^3
$Z = 2$
$F(000) = 168$
$D_x = 1.332$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4876 reflections
$\theta = 2.5$–33.3°
$\mu = 0.09$ mm^{-1}
$T = 153$ K
Irregular, colourless
0.55 × 0.41 × 0.15 mm

Data collection

Bruker APEXII CCD area detector diffractometer
phi and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2008)
$T_{min} = 0.956$, $T_{max} = 0.988$
8959 measured reflections
2133 independent reflections
1881 reflections with $I > 2\sigma(I)$
$R_{int} = 0.018$
$\theta_{max} = 29.1°$, $\theta_{min} = 2.5°$
$h = -9 \rightarrow 9$
$k = -10 \rightarrow 9$
$l = -12 \rightarrow 12$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.115$
$S = 1.06$
2133 reflections
111 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0597P)^2 + 0.0965P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.39$ e Å^{-3}
$\Delta\rho_{min} = -0.19$ 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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	1.41916 (10)	0.74175 (12)	0.41955 (9)	0.0302 (2)
H1	1.3947	0.7603	0.3254	0.045*
O2	1.20310 (12)	0.78960 (13)	0.19487 (9)	0.0330 (2)
C1	1.24964 (13)	0.73237 (14)	0.51663 (11)	0.0216 (2)
C2	1.06267 (13)	0.75622 (13)	0.45971 (10)	0.01892 (19)
C3	0.89112 (13)	0.75319 (13)	0.56854 (10)	0.01892 (19)
H3	0.7646	0.7693	0.5317	0.023*
C4	0.90292 (13)	0.72710 (13)	0.72934 (11)	0.01990 (19)
C5	1.09230 (14)	0.69865 (14)	0.78338 (11)	0.0229 (2)
H5	1.1027	0.6783	0.8933	0.027*
C6	1.26244 (14)	0.70005 (15)	0.67882 (12)	0.0245 (2)
H6	1.3895	0.6788	0.7174	0.029*
C7	1.04995 (14)	0.78735 (14)	0.28831 (11)	0.0219 (2)
C8	0.85185 (15)	0.81574 (16)	0.22710 (11)	0.0260 (2)
H8A	0.8665	0.8497	0.1099	0.039*
H8B	0.7450	0.9274	0.2564	0.039*
H8C	0.8142	0.6887	0.2748	0.039*
C9	0.72595 (14)	0.73051 (15)	0.83939 (11)	0.0233 (2)
C10	0.58198 (16)	0.73495 (18)	0.93333 (12)	0.0303 (2)
H10	0.4670	0.7385	1.0084	0.036*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0169 (3)	0.0405 (4)	0.0325 (4)	−0.0114 (3)	0.0054 (3)	−0.0114 (3)
O2	0.0275 (4)	0.0478 (5)	0.0258 (4)	−0.0153 (3)	0.0108 (3)	−0.0163 (3)
C1	0.0160 (4)	0.0208 (4)	0.0276 (5)	−0.0062 (3)	0.0026 (3)	−0.0084 (3)
C2	0.0178 (4)	0.0195 (4)	0.0200 (4)	−0.0062 (3)	0.0015 (3)	−0.0075 (3)
C3	0.0165 (4)	0.0209 (4)	0.0198 (4)	−0.0060 (3)	0.0003 (3)	−0.0073 (3)
C4	0.0188 (4)	0.0204 (4)	0.0197 (4)	−0.0055 (3)	0.0005 (3)	−0.0066 (3)
C5	0.0234 (4)	0.0245 (4)	0.0216 (4)	−0.0065 (3)	−0.0038 (3)	−0.0080 (3)
C6	0.0186 (4)	0.0268 (5)	0.0299 (5)	−0.0069 (3)	−0.0049 (3)	−0.0093 (4)
C7	0.0227 (4)	0.0230 (4)	0.0208 (4)	−0.0084 (3)	0.0043 (3)	−0.0091 (3)
C8	0.0269 (5)	0.0333 (5)	0.0207 (4)	−0.0104 (4)	0.0005 (3)	−0.0117 (4)
C9	0.0229 (4)	0.0286 (5)	0.0181 (4)	−0.0072 (3)	−0.0019 (3)	−0.0073 (3)
C10	0.0241 (5)	0.0454 (6)	0.0199 (4)	−0.0100 (4)	0.0018 (3)	−0.0107 (4)

Geometric parameters (Å, º)

O1—C1	1.3447 (10)	C4—C9	1.4357 (12)
O1—H1	0.8400	C5—C6	1.3760 (13)
O2—C7	1.2359 (11)	C5—H5	0.9500
C1—C6	1.3945 (13)	C6—H6	0.9500
C1—C2	1.4144 (12)	C7—C8	1.4954 (13)
C2—C3	1.4043 (11)	C8—H8A	0.9800
C2—C7	1.4776 (12)	C8—H8B	0.9800
C3—C4	1.3915 (12)	C8—H8C	0.9800
C3—H3	0.9500	C9—C10	1.1896 (14)
C4—C5	1.4081 (13)	C10—H10	0.9500
C1—O1—H1	109.5	C4—C5—H5	119.6
O1—C1—C6	117.45 (8)	C5—C6—C1	120.47 (8)
O1—C1—C2	122.56 (8)	C5—C6—H6	119.8
C6—C1—C2	119.99 (8)	C1—C6—H6	119.8
C3—C2—C1	118.61 (8)	O2—C7—C2	120.17 (8)
C3—C2—C7	121.40 (8)	O2—C7—C8	119.66 (8)
C1—C2—C7	119.98 (8)	C2—C7—C8	120.17 (8)
C4—C3—C2	121.23 (8)	C7—C8—H8A	109.5
C4—C3—H3	119.4	C7—C8—H8B	109.5
C2—C3—H3	119.4	H8A—C8—H8B	109.5
C3—C4—C5	118.87 (8)	C7—C8—H8C	109.5
C3—C4—C9	121.18 (8)	H8A—C8—H8C	109.5
C5—C4—C9	119.94 (8)	H8B—C8—H8C	109.5
C6—C5—C4	120.77 (8)	C10—C9—C4	178.07 (10)
C6—C5—H5	119.6	C9—C10—H10	180.0
O1—C1—C2—C3	−177.21 (8)	C9—C4—C5—C6	−178.37 (8)
C6—C1—C2—C3	2.03 (13)	C4—C5—C6—C1	0.82 (14)
O1—C1—C2—C7	1.68 (14)	O1—C1—C6—C5	176.86 (8)
C6—C1—C2—C7	−179.08 (8)	C2—C1—C6—C5	−2.41 (14)
C1—C2—C3—C4	−0.09 (13)	C3—C2—C7—O2	179.92 (8)
C7—C2—C3—C4	−178.96 (8)	C1—C2—C7—O2	1.07 (14)
C2—C3—C4—C5	−1.46 (13)	C3—C2—C7—C8	−0.24 (13)
C2—C3—C4—C9	178.01 (8)	C1—C2—C7—C8	−179.09 (8)
C3—C4—C5—C6	1.11 (14)		

Hydrogen-bond geometry (Å, º)

Cg1 is the centroid of the C1–C6 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O2	0.84	1.83	2.5696 (11)	146
C10—H10···O2^{i}	0.95	2.28	3.2214 (14)	171
C8—H8*C*···*Cg*1^{ii}	0.97	2.72	3.6024 (12)	150

Symmetry codes: (i) *x*−1, *y*, *z*+1; (ii) −*x*+2, −*y*+3, −*z*+1.

(II) 2-acetyl-4-(3-hydroxy-3-methylbut-1-yn-1-yl)phenol

Crystal data

$C_{13}H_{14}O_3$
$M_r = 218.24$
Monoclinic, $P2_1/c$
$a = 22.5787$ (6) Å
$b = 16.9306$ (4) Å
$c = 9.2849$ (2) Å
$\beta = 101.815$ (1)°
$V = 3474.15$ (14) Å^3
$Z = 12$
$F(000) = 1392$
$D_x = 1.252$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 5422 reflections
$\theta = 2.5$–30.0°
$\mu = 0.09$ mm^{-1}
$T = 153$ K
Irregular, colourless
0.36 × 0.18 × 0.09 mm

Data collection

Bruker APEXII CCD area detector diffractometer
phi and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2008)
$T_{min} = 0.969$, $T_{max} = 0.992$
37857 measured reflections
9244 independent reflections
5584 reflections with $I > 2\sigma(I)$
$R_{int} = 0.043$
$\theta_{max} = 29.1°$, $\theta_{min} = 1.5°$
$h = -30\rightarrow30$
$k = -22\rightarrow15$
$l = -12\rightarrow12$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.047$
$wR(F^2) = 0.117$
$S = 0.89$
9244 reflections
448 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0549P)^2 + 0.7504P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.28$ e Å^{-3}
$\Delta\rho_{min} = -0.22$ 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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.89700 (5)	0.94178 (6)	0.79757 (13)	0.0366 (3)
H1	0.9285	0.9250	0.7731	0.055*
O2	0.96006 (5)	0.84850 (7)	0.67152 (12)	0.0346 (3)
O3	0.56111 (4)	0.64727 (5)	0.60432 (10)	0.0205 (2)
H3	0.5789	0.6073	0.6458	0.031*
C1	0.85079 (7)	0.89274 (9)	0.74568 (17)	0.0269 (3)
C2	0.85610 (6)	0.82660 (8)	0.65630 (15)	0.0211 (3)
C3	0.80510 (6)	0.77861 (8)	0.60989 (15)	0.0215 (3)
H3A	0.8082	0.7340	0.5498	0.026*
C4	0.75036 (7)	0.79445 (9)	0.64918 (17)	0.0263 (3)
C5	0.74677 (7)	0.86060 (11)	0.7373 (2)	0.0421 (5)

H5	0.7095	0.8724	0.7652	0.051*
C6	0.79568 (7)	0.90872 (10)	0.7843 (2)	0.0431 (5)
H6	0.7919	0.9534	0.8438	0.052*
C7	0.91531 (7)	0.80842 (9)	0.61934 (16)	0.0240 (3)
C8	0.92118 (7)	0.74117 (9)	0.51956 (17)	0.0307 (4)
H8A	0.9608	0.7434	0.4922	0.046*
H8B	0.8892	0.7447	0.4307	0.046*
H8C	0.9173	0.6912	0.5702	0.046*
C9	0.69681 (7)	0.74734 (9)	0.59880 (17)	0.0268 (3)
C10	0.65027 (7)	0.71275 (8)	0.55868 (16)	0.0249 (3)
C11	0.59180 (6)	0.67451 (8)	0.49244 (15)	0.0203 (3)
C12	0.54963 (7)	0.73505 (9)	0.40490 (18)	0.0314 (4)
H12A	0.5115	0.7095	0.3593	0.047*
H12B	0.5686	0.7576	0.3281	0.047*
H12C	0.5417	0.7772	0.4707	0.047*
C13	0.60249 (7)	0.60527 (9)	0.39664 (17)	0.0323 (4)
H13A	0.6330	0.5700	0.4534	0.048*
H13B	0.6168	0.6250	0.3106	0.048*
H13C	0.5646	0.5762	0.3641	0.048*
O1A	0.09288 (5)	0.39654 (6)	0.19886 (12)	0.0314 (3)
H1A	0.0606	0.4125	0.2208	0.047*
O2A	0.02652 (5)	0.48991 (6)	0.31900 (12)	0.0314 (3)
O3A	0.44017 (4)	0.60416 (6)	0.48406 (11)	0.0266 (2)
H3A1	0.4762	0.6151	0.5226	0.040*
C1A	0.13877 (6)	0.44376 (8)	0.26152 (15)	0.0223 (3)
C2A	0.13134 (6)	0.50939 (8)	0.35040 (15)	0.0201 (3)
C3A	0.18251 (6)	0.55480 (8)	0.40964 (15)	0.0218 (3)
H3A2	0.1782	0.5995	0.4685	0.026*
C4A	0.23926 (6)	0.53601 (8)	0.38434 (16)	0.0225 (3)
C5A	0.24516 (7)	0.47049 (8)	0.29560 (16)	0.0255 (3)
H5A	0.2838	0.4572	0.2770	0.031*
C6A	0.19582 (7)	0.42545 (9)	0.23548 (17)	0.0278 (3)
H6A	0.2006	0.3814	0.1755	0.033*
C7A	0.07086 (6)	0.52920 (8)	0.37736 (16)	0.0229 (3)
C8A	0.06356 (7)	0.59680 (9)	0.47539 (17)	0.0281 (3)
H8A1	0.0217	0.5987	0.4889	0.042*
H8A2	0.0733	0.6462	0.4305	0.042*
H8A3	0.0909	0.5898	0.5711	0.042*
C9A	0.29267 (7)	0.57970 (8)	0.45030 (16)	0.0238 (3)
C10A	0.33979 (6)	0.60994 (8)	0.50234 (16)	0.0222 (3)
C11A	0.40037 (6)	0.64057 (8)	0.56760 (15)	0.0197 (3)
C12A	0.41846 (7)	0.61565 (9)	0.72763 (16)	0.0274 (3)
H12D	0.4181	0.5579	0.7341	0.041*
H12E	0.3898	0.6377	0.7830	0.041*
H12F	0.4592	0.6352	0.7691	0.041*
C13A	0.40330 (7)	0.73018 (8)	0.55269 (19)	0.0344 (4)
H13D	0.4440	0.7487	0.5977	0.052*
H13E	0.3737	0.7548	0.6025	0.052*

H13F	0.3941	0.7446	0.4483	0.052*
O1B	0.09750 (5)	0.76866 (6)	0.69610 (12)	0.0308 (3)
H1B	0.0648	0.7519	0.7142	0.046*
O2B	0.02975 (5)	0.67941 (6)	0.81596 (12)	0.0312 (3)
O3B	0.39230 (5)	0.49085 (5)	1.26894 (10)	0.0223 (2)
H3B	0.4058	0.5279	1.3266	0.033*
C1B	0.14361 (6)	0.72470 (8)	0.76915 (16)	0.0231 (3)
C2B	0.13506 (6)	0.66126 (8)	0.86175 (15)	0.0197 (3)
C3B	0.18585 (6)	0.61886 (8)	0.93290 (15)	0.0207 (3)
H3B1	0.1805	0.5758	0.9947	0.025*
C4B	0.24368 (6)	0.63788 (8)	0.91583 (16)	0.0225 (3)
C5B	0.25058 (7)	0.70103 (9)	0.82282 (18)	0.0318 (4)
H5B	0.2899	0.7147	0.8095	0.038*
C6B	0.20166 (7)	0.74333 (9)	0.75085 (18)	0.0326 (4)
H6B	0.2074	0.7857	0.6880	0.039*
C7B	0.07363 (7)	0.64181 (8)	0.88234 (15)	0.0218 (3)
C8B	0.06475 (7)	0.57612 (8)	0.98361 (16)	0.0260 (3)
H8B1	0.0226	0.5756	0.9951	0.039*
H8B2	0.0915	0.5841	1.0798	0.039*
H8B3	0.0744	0.5256	0.9422	0.039*
C9B	0.29578 (6)	0.59485 (8)	0.99253 (16)	0.0234 (3)
C10B	0.33953 (7)	0.56037 (8)	1.05638 (16)	0.0220 (3)
C11B	0.39376 (6)	0.51369 (8)	1.12051 (15)	0.0192 (3)
C12B	0.39370 (8)	0.43695 (8)	1.03571 (17)	0.0321 (4)
H12G	0.4307	0.4073	1.0753	0.048*
H12H	0.3919	0.4487	0.9316	0.048*
H12I	0.3584	0.4053	1.0455	0.048*
C13B	0.45099 (7)	0.56111 (9)	1.11999 (16)	0.0263 (3)
H13G	0.4512	0.6084	1.1809	0.039*
H13H	0.4521	0.5767	1.0189	0.039*
H13I	0.4865	0.5287	1.1598	0.039*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0213 (6)	0.0381 (6)	0.0493 (7)	−0.0113 (5)	0.0050 (6)	−0.0164 (6)
O2	0.0213 (6)	0.0484 (7)	0.0351 (7)	−0.0103 (5)	0.0080 (5)	−0.0079 (5)
O3	0.0189 (5)	0.0210 (5)	0.0220 (5)	0.0017 (4)	0.0049 (4)	0.0048 (4)
C1	0.0189 (8)	0.0300 (8)	0.0299 (8)	−0.0060 (6)	0.0006 (7)	−0.0063 (6)
C2	0.0171 (7)	0.0260 (7)	0.0191 (7)	−0.0034 (6)	0.0009 (6)	0.0023 (6)
C3	0.0198 (7)	0.0232 (7)	0.0204 (7)	−0.0028 (6)	0.0017 (6)	0.0008 (6)
C4	0.0184 (8)	0.0301 (7)	0.0283 (8)	−0.0069 (6)	0.0002 (7)	−0.0013 (6)
C5	0.0186 (8)	0.0521 (11)	0.0565 (12)	−0.0055 (8)	0.0097 (8)	−0.0235 (9)
C6	0.0238 (9)	0.0481 (10)	0.0576 (12)	−0.0044 (8)	0.0091 (9)	−0.0309 (9)
C7	0.0216 (8)	0.0315 (7)	0.0189 (7)	−0.0017 (6)	0.0038 (6)	0.0036 (6)
C8	0.0274 (9)	0.0356 (8)	0.0305 (9)	0.0002 (7)	0.0090 (7)	−0.0021 (7)
C9	0.0208 (8)	0.0298 (8)	0.0292 (8)	−0.0033 (6)	0.0037 (7)	0.0004 (6)
C10	0.0221 (8)	0.0256 (7)	0.0266 (8)	−0.0013 (6)	0.0041 (7)	0.0019 (6)

C11	0.0178 (7)	0.0233 (7)	0.0198 (7)	−0.0054 (6)	0.0038 (6)	0.0018 (6)
C12	0.0248 (8)	0.0315 (8)	0.0348 (9)	−0.0078 (7)	−0.0015 (7)	0.0154 (7)
C13	0.0264 (9)	0.0423 (9)	0.0297 (9)	−0.0057 (7)	0.0091 (7)	−0.0122 (7)
O1A	0.0187 (6)	0.0355 (6)	0.0388 (7)	−0.0094 (5)	0.0029 (5)	−0.0100 (5)
O2A	0.0163 (5)	0.0419 (6)	0.0355 (6)	−0.0057 (5)	0.0044 (5)	−0.0029 (5)
O3A	0.0160 (5)	0.0374 (6)	0.0272 (6)	−0.0055 (5)	0.0062 (5)	−0.0113 (5)
C1A	0.0178 (7)	0.0254 (7)	0.0223 (8)	−0.0052 (6)	0.0007 (6)	0.0027 (6)
C2A	0.0159 (7)	0.0254 (7)	0.0182 (7)	−0.0015 (6)	0.0018 (6)	0.0043 (6)
C3A	0.0205 (8)	0.0234 (7)	0.0211 (7)	−0.0007 (6)	0.0035 (6)	0.0008 (6)
C4A	0.0170 (7)	0.0243 (7)	0.0248 (8)	−0.0040 (6)	0.0009 (6)	0.0027 (6)
C5A	0.0162 (7)	0.0291 (7)	0.0315 (8)	−0.0008 (6)	0.0058 (7)	−0.0006 (6)
C6A	0.0248 (8)	0.0277 (7)	0.0309 (9)	−0.0018 (6)	0.0059 (7)	−0.0069 (6)
C7A	0.0180 (7)	0.0295 (7)	0.0205 (7)	−0.0009 (6)	0.0023 (6)	0.0055 (6)
C8A	0.0202 (8)	0.0329 (8)	0.0319 (9)	0.0007 (7)	0.0074 (7)	0.0004 (7)
C9A	0.0191 (8)	0.0251 (7)	0.0270 (8)	−0.0004 (6)	0.0046 (6)	0.0000 (6)
C10A	0.0188 (7)	0.0234 (7)	0.0244 (8)	−0.0003 (6)	0.0044 (6)	−0.0019 (6)
C11A	0.0154 (7)	0.0217 (6)	0.0215 (7)	−0.0001 (6)	0.0025 (6)	−0.0025 (6)
C12A	0.0267 (8)	0.0315 (8)	0.0236 (8)	−0.0001 (7)	0.0041 (7)	−0.0034 (6)
C13A	0.0286 (9)	0.0226 (7)	0.0484 (11)	−0.0029 (7)	−0.0008 (8)	0.0019 (7)
O1B	0.0201 (6)	0.0336 (6)	0.0370 (6)	0.0063 (5)	0.0018 (5)	0.0147 (5)
O2B	0.0178 (5)	0.0398 (6)	0.0348 (6)	0.0047 (5)	0.0025 (5)	0.0082 (5)
O3B	0.0278 (6)	0.0200 (5)	0.0186 (5)	0.0002 (4)	0.0036 (5)	0.0013 (4)
C1B	0.0200 (8)	0.0244 (7)	0.0228 (8)	0.0039 (6)	−0.0008 (6)	0.0043 (6)
C2B	0.0178 (7)	0.0219 (6)	0.0184 (7)	0.0001 (6)	0.0011 (6)	−0.0016 (5)
C3B	0.0207 (8)	0.0203 (6)	0.0202 (7)	0.0002 (6)	0.0024 (6)	0.0016 (5)
C4B	0.0187 (7)	0.0247 (7)	0.0224 (8)	0.0038 (6)	0.0005 (6)	0.0027 (6)
C5B	0.0181 (8)	0.0376 (9)	0.0389 (10)	0.0003 (7)	0.0041 (7)	0.0123 (7)
C6B	0.0227 (8)	0.0365 (8)	0.0385 (10)	0.0022 (7)	0.0061 (7)	0.0188 (7)
C7B	0.0202 (7)	0.0256 (7)	0.0190 (7)	−0.0004 (6)	0.0028 (6)	−0.0038 (6)
C8B	0.0215 (8)	0.0296 (7)	0.0271 (8)	−0.0029 (6)	0.0055 (7)	0.0011 (6)
C9B	0.0182 (8)	0.0251 (7)	0.0259 (8)	−0.0010 (6)	0.0022 (6)	0.0026 (6)
C10B	0.0194 (7)	0.0230 (7)	0.0231 (8)	−0.0013 (6)	0.0033 (6)	0.0015 (6)
C11B	0.0199 (7)	0.0202 (6)	0.0168 (7)	0.0024 (6)	0.0018 (6)	0.0001 (5)
C12B	0.0419 (10)	0.0257 (7)	0.0267 (9)	0.0038 (7)	0.0021 (7)	−0.0059 (6)
C13B	0.0199 (8)	0.0331 (8)	0.0251 (8)	0.0013 (6)	0.0029 (6)	0.0058 (6)

Geometric parameters (Å, º)

O1—C1	1.3432 (17)	C6A—H6A	0.9500
O1—H1	0.8400	C7A—C8A	1.492 (2)
O2—C7	1.2311 (17)	C8A—H8A1	0.9800
O3—C11	1.4376 (16)	C8A—H8A2	0.9800
O3—H3	0.8400	C8A—H8A3	0.9800
C1—C6	1.390 (2)	C9A—C10A	1.1903 (19)
C1—C2	1.413 (2)	C10A—C11A	1.472 (2)
C2—C3	1.4031 (19)	C11A—C12A	1.518 (2)
C2—C7	1.479 (2)	C11A—C13A	1.5261 (19)
C3—C4	1.385 (2)	C12A—H12D	0.9800

C3—H3A	0.9500	C12A—H12E	0.9800
C4—C5	1.399 (2)	C12A—H12F	0.9800
C4—C9	1.444 (2)	C13A—H13D	0.9800
C5—C6	1.370 (2)	C13A—H13E	0.9800
C5—H5	0.9500	C13A—H13F	0.9800
C6—H6	0.9500	O1B—C1B	1.3448 (16)
C7—C8	1.491 (2)	O1B—H1B	0.8400
C8—H8A	0.9800	O2B—C7B	1.2314 (17)
C8—H8B	0.9800	O3B—C11B	1.4382 (16)
C8—H8C	0.9800	O3B—H3B	0.8400
C9—C10	1.194 (2)	C1B—C6B	1.392 (2)
C10—C11	1.486 (2)	C1B—C2B	1.4135 (19)
C11—C12	1.517 (2)	C2B—C3B	1.3988 (19)
C11—C13	1.520 (2)	C2B—C7B	1.476 (2)
C12—H12A	0.9800	C3B—C4B	1.3849 (19)
C12—H12B	0.9800	C3B—H3B1	0.9500
C12—H12C	0.9800	C4B—C5B	1.403 (2)
C13—H13A	0.9800	C4B—C9B	1.442 (2)
C13—H13B	0.9800	C5B—C6B	1.370 (2)
C13—H13C	0.9800	C5B—H5B	0.9500
O1A—C1A	1.3434 (16)	C6B—H6B	0.9500
O1A—H1A	0.8400	C7B—C8B	1.4962 (19)
O2A—C7A	1.2313 (17)	C8B—H8B1	0.9800
O3A—C11A	1.4403 (16)	C8B—H8B2	0.9800
O3A—H3A1	0.8400	C8B—H8B3	0.9800
C1A—C6A	1.393 (2)	C9B—C10B	1.1951 (19)
C1A—C2A	1.4144 (19)	C10B—C11B	1.4765 (19)
C2A—C3A	1.4030 (19)	C11B—C12B	1.5189 (19)
C2A—C7A	1.477 (2)	C11B—C13B	1.5221 (19)
C3A—C4A	1.3862 (19)	C12B—H12G	0.9800
C3A—H3A2	0.9500	C12B—H12H	0.9800
C4A—C5A	1.404 (2)	C12B—H12I	0.9800
C4A—C9A	1.440 (2)	C13B—H13G	0.9800
C5A—C6A	1.371 (2)	C13B—H13H	0.9800
C5A—H5A	0.9500	C13B—H13I	0.9800

C1—O1—H1	109.5	H8A1—C8A—H8A2	109.5
C11—O3—H3	109.5	C7A—C8A—H8A3	109.5
O1—C1—C6	117.24 (13)	H8A1—C8A—H8A3	109.5
O1—C1—C2	123.17 (13)	H8A2—C8A—H8A3	109.5
C6—C1—C2	119.58 (13)	C10A—C9A—C4A	174.00 (16)
C3—C2—C1	118.43 (13)	C9A—C10A—C11A	175.11 (15)
C3—C2—C7	122.16 (13)	O3A—C11A—C10A	104.90 (11)
C1—C2—C7	119.37 (13)	O3A—C11A—C12A	109.62 (11)
C4—C3—C2	121.85 (13)	C10A—C11A—C12A	110.24 (12)
C4—C3—H3A	119.1	O3A—C11A—C13A	109.47 (12)
C2—C3—H3A	119.1	C10A—C11A—C13A	111.50 (12)
C3—C4—C5	118.10 (13)	C12A—C11A—C13A	110.93 (12)

C3—C4—C9	122.75 (14)	C11A—C12A—H12D	109.5
C5—C4—C9	119.12 (14)	C11A—C12A—H12E	109.5
C6—C5—C4	121.52 (15)	H12D—C12A—H12E	109.5
C6—C5—H5	119.2	C11A—C12A—H12F	109.5
C4—C5—H5	119.2	H12D—C12A—H12F	109.5
C5—C6—C1	120.52 (15)	H12E—C12A—H12F	109.5
C5—C6—H6	119.7	C11A—C13A—H13D	109.5
C1—C6—H6	119.7	C11A—C13A—H13E	109.5
O2—C7—C2	120.13 (13)	H13D—C13A—H13E	109.5
O2—C7—C8	119.69 (14)	C11A—C13A—H13F	109.5
C2—C7—C8	120.18 (13)	H13D—C13A—H13F	109.5
C7—C8—H8A	109.5	H13E—C13A—H13F	109.5
C7—C8—H8B	109.5	C1B—O1B—H1B	109.5
H8A—C8—H8B	109.5	C11B—O3B—H3B	109.5
C7—C8—H8C	109.5	O1B—C1B—C6B	117.64 (13)
H8A—C8—H8C	109.5	O1B—C1B—C2B	122.65 (13)
H8B—C8—H8C	109.5	C6B—C1B—C2B	119.71 (13)
C10—C9—C4	175.55 (16)	C3B—C2B—C1B	118.45 (13)
C9—C10—C11	173.44 (16)	C3B—C2B—C7B	121.72 (12)
O3—C11—C10	111.07 (11)	C1B—C2B—C7B	119.82 (13)
O3—C11—C12	105.15 (11)	C4B—C3B—C2B	121.84 (13)
C10—C11—C12	109.53 (11)	C4B—C3B—H3B1	119.1
O3—C11—C13	109.47 (11)	C2B—C3B—H3B1	119.1
C10—C11—C13	110.13 (12)	C3B—C4B—C5B	118.28 (13)
C12—C11—C13	111.41 (13)	C3B—C4B—C9B	121.27 (13)
C11—C12—H12A	109.5	C5B—C4B—C9B	120.44 (13)
C11—C12—H12B	109.5	C6B—C5B—C4B	121.22 (14)
H12A—C12—H12B	109.5	C6B—C5B—H5B	119.4
C11—C12—H12C	109.5	C4B—C5B—H5B	119.4
H12A—C12—H12C	109.5	C5B—C6B—C1B	120.48 (14)
H12B—C12—H12C	109.5	C5B—C6B—H6B	119.8
C11—C13—H13A	109.5	C1B—C6B—H6B	119.8
C11—C13—H13B	109.5	O2B—C7B—C2B	120.07 (13)
H13A—C13—H13B	109.5	O2B—C7B—C8B	120.09 (13)
C11—C13—H13C	109.5	C2B—C7B—C8B	119.84 (13)
H13A—C13—H13C	109.5	C7B—C8B—H8B1	109.5
H13B—C13—H13C	109.5	C7B—C8B—H8B2	109.5
C1A—O1A—H1A	109.5	H8B1—C8B—H8B2	109.5
C11A—O3A—H3A1	109.5	C7B—C8B—H8B3	109.5
O1A—C1A—C6A	116.82 (13)	H8B1—C8B—H8B3	109.5
O1A—C1A—C2A	123.16 (13)	H8B2—C8B—H8B3	109.5
C6A—C1A—C2A	120.02 (13)	C10B—C9B—C4B	178.88 (16)
C3A—C2A—C1A	118.21 (13)	C9B—C10B—C11B	174.01 (15)
C3A—C2A—C7A	121.67 (13)	O3B—C11B—C10B	110.51 (11)
C1A—C2A—C7A	120.12 (12)	O3B—C11B—C12B	105.61 (11)
C4A—C3A—C2A	121.56 (13)	C10B—C11B—C12B	109.64 (12)
C4A—C3A—H3A2	119.2	O3B—C11B—C13B	109.33 (11)
C2A—C3A—H3A2	119.2	C10B—C11B—C13B	110.54 (11)

C3A—C4A—C5A	118.87 (13)	C12B—C11B—C13B	111.12 (12)
C3A—C4A—C9A	122.18 (13)	C11B—C12B—H12G	109.5
C5A—C4A—C9A	118.91 (13)	C11B—C12B—H12H	109.5
C6A—C5A—C4A	120.76 (14)	H12G—C12B—H12H	109.5
C6A—C5A—H5A	119.6	C11B—C12B—H12I	109.5
C4A—C5A—H5A	119.6	H12G—C12B—H12I	109.5
C5A—C6A—C1A	120.58 (14)	H12H—C12B—H12I	109.5
C5A—C6A—H6A	119.7	C11B—C13B—H13G	109.5
C1A—C6A—H6A	119.7	C11B—C13B—H13H	109.5
O2A—C7A—C2A	119.97 (13)	H13G—C13B—H13H	109.5
O2A—C7A—C8A	120.10 (13)	C11B—C13B—H13I	109.5
C2A—C7A—C8A	119.94 (13)	H13G—C13B—H13I	109.5
C7A—C8A—H8A1	109.5	H13H—C13B—H13I	109.5
C7A—C8A—H8A2	109.5		

O1—C1—C2—C3	179.09 (14)	C9A—C4A—C5A—C6A	177.42 (14)
C6—C1—C2—C3	−0.3 (2)	C4A—C5A—C6A—C1A	−0.1 (2)
O1—C1—C2—C7	1.3 (2)	O1A—C1A—C6A—C5A	−179.82 (13)
C6—C1—C2—C7	−178.10 (15)	C2A—C1A—C6A—C5A	0.1 (2)
C1—C2—C3—C4	0.0 (2)	C3A—C2A—C7A—O2A	177.17 (13)
C7—C2—C3—C4	177.71 (13)	C1A—C2A—C7A—O2A	−2.1 (2)
C2—C3—C4—C5	0.2 (2)	C3A—C2A—C7A—C8A	−2.9 (2)
C2—C3—C4—C9	177.97 (13)	C1A—C2A—C7A—C8A	177.88 (13)
C3—C4—C5—C6	−0.1 (3)	O1B—C1B—C2B—C3B	179.88 (13)
C9—C4—C5—C6	−177.96 (17)	C6B—C1B—C2B—C3B	0.1 (2)
C4—C5—C6—C1	−0.2 (3)	O1B—C1B—C2B—C7B	−0.7 (2)
O1—C1—C6—C5	−179.02 (17)	C6B—C1B—C2B—C7B	179.48 (14)
C2—C1—C6—C5	0.4 (3)	C1B—C2B—C3B—C4B	0.6 (2)
C3—C2—C7—O2	−174.40 (13)	C7B—C2B—C3B—C4B	−178.83 (13)
C1—C2—C7—O2	3.3 (2)	C2B—C3B—C4B—C5B	−0.7 (2)
C3—C2—C7—C8	5.2 (2)	C2B—C3B—C4B—C9B	178.53 (13)
C1—C2—C7—C8	−177.15 (13)	C3B—C4B—C5B—C6B	0.3 (2)
O1A—C1A—C2A—C3A	−179.71 (13)	C9B—C4B—C5B—C6B	−178.99 (15)
C6A—C1A—C2A—C3A	0.3 (2)	C4B—C5B—C6B—C1B	0.3 (3)
O1A—C1A—C2A—C7A	−0.4 (2)	O1B—C1B—C6B—C5B	179.67 (15)
C6A—C1A—C2A—C7A	179.61 (13)	C2B—C1B—C6B—C5B	−0.5 (2)
C1A—C2A—C3A—C4A	−0.8 (2)	C3B—C2B—C7B—O2B	−178.68 (13)
C7A—C2A—C3A—C4A	179.90 (13)	C1B—C2B—C7B—O2B	1.9 (2)
C2A—C3A—C4A—C5A	0.9 (2)	C3B—C2B—C7B—C8B	1.1 (2)
C2A—C3A—C4A—C9A	−176.86 (13)	C1B—C2B—C7B—C8B	−178.30 (13)
C3A—C4A—C5A—C6A	−0.4 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O2	0.84	1.83	2.5639 (16)	145
O1—H1···O2*A*^{i}	0.84	2.60	3.1129 (15)	121
O3—H3···O3*B*^{ii}	0.84	1.90	2.7300 (12)	171

O1*A*—H1*A*···O2*A*	0.84	1.85	2.5832 (15)	145
O1*A*—H1*A*···O2*B*^{iii}	0.84	2.53	3.0303 (16)	119
O3*A*—H3*A*1···O3	0.84	1.99	2.8262 (13)	176
O1*B*—H1*B*···O2*B*	0.84	1.83	2.5611 (16)	145
O3*B*—H3*B*···O3*A*^{iv}	0.84	1.99	2.8203 (13)	172

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