

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

2',3,4,4'-Tetramethoxychalcone

Johannes H. van Tonder, Theunis J. Muller* and Barend C. B. Bezuidenhout

Department of Chemistry, University of the Free State, PO Box 339, Bloemfontein, 9300, South Africa

Correspondence e-mail: Muller.theunis@gmail.com

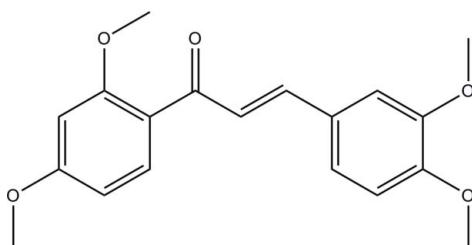
Received 17 May 2010; accepted 9 June 2010

Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.044; wR factor = 0.188; data-to-parameter ratio = 18.7.

In the title compound [systematic name: 1-(2,4-dimethoxyphenyl)-3-(3,4-dimethoxyphenyl)prop-2-en-1-one], $\text{C}_{19}\text{H}_{20}\text{O}_5$, the dihedral angle between the benzene rings is 26.88 (5°). One of the methoxy groups is twisted slightly away from the plane [$\text{C}-\text{O}-\text{C}$ torsion angle = -12.8 (3°)] while the others are almost co-planar [$\text{C}-\text{O}-\text{C}$ torsion angles = -3.2 (3), 2.6 (3) and -3.6 (3°)]. The crystal packing is stabilized by intermolecular $\text{C}-\text{H}\cdots\text{O}$ interactions. A weak intramolecular $\text{C}-\text{H}\cdots\text{O}$ interaction occurs.

Related literature

For properties and uses of chalcones, see: Marais *et al.* (2005); Fichou *et al.* (1988); Uchida *et al.* (1998). For the biological activity of flavenoids, see: Pietta *et al.* (2003). For related structures, see: Patil *et al.* (2006a,b,c); Teh *et al.* (2006a,b,c); Rosli *et al.* (2006). For the synthesis of the title compound, see: Kraus *et al.* (2008). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$\text{C}_{19}\text{H}_{20}\text{O}_5$
 $M_r = 328.35$
 Monoclinic, $P2_1/c$
 $a = 12.5839$ (7) Å
 $b = 11.7204$ (7) Å
 $c = 12.1339$ (6) Å
 $\beta = 109.489$ (2°)

$V = 1687.07$ (16) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.09$ mm⁻¹
 $T = 100$ K
 $0.49 \times 0.22 \times 0.07$ mm

Data collection

Bruker APEXII diffractometer
 Absorption correction: multi-scan
 (SADABS; Bruker, 2008)
 $T_{\min} = 0.976$, $T_{\max} = 0.994$

32681 measured reflections
 4205 independent reflections
 2539 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.039$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.044$
 $wR(F^2) = 0.188$
 $S = 1.12$
 4205 reflections
 225 parameters

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\max} = 0.21$ e Å⁻³
 $\Delta\rho_{\min} = -0.20$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{C}8-\text{H}8\cdots\text{O}1$	0.96 (2)	2.29 (2)	2.813 (3)	113.6 (15)
$\text{C}18-\text{H}18B\cdots\text{O}3^i$	0.96	2.46	3.253 (3)	140

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

Data collection: APEX2 (Bruker, 2008); cell refinement: SAINT-Plus (Bruker, 2008); data reduction: SAINT-Plus and XPREP (Bruker, 2008); program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: DIAMOND (Brandenburg & Putz, 2005); software used to prepare material for publication: WingGX (Farrugia, 1999).

The University of the Free State is gratefully acknowledged for financial support, Alice Brink for the data collection and Marietjie Schutte for her valuable input.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG2688).

References

- Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2* pp. S1–19.
- Brandenburg, K. & Putz, H. (2005). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
- Bruker (2008). *APEX2*, *SAINt-Plus*, *XPREP* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
- Fichou, D., Watanabe, T., Takenda, T., Miyata, S., Goto, Y. & Nakayama, M. (1988). *Jpn. J. Appl. Phys.* **27**, L429–L430.
- Kraus, G. & Roy, S. (2008). *J. Nat. Prod.* **71**, 1961–1962.
- Marais, J. P. J., Ferreira, D. & Slade, D. (2005). *Phytochemistry*, **66**, 2145–2176.
- Patil, P. S., Ng, S.-L., Razak, I. A., Fun, H.-K. & Dharmaparakash, S. M. (2006c). *Acta Cryst.* **E62**, o4448–o4449.
- Patil, P. S., Teh, J. B.-J., Fun, H.-K., Razak, I. A. & Dharmaparakash, S. M. (2006a). *Acta Cryst.* **E62**, o896–o898.
- Patil, P. S., Teh, J. B.-J., Fun, H.-K., Razak, I. A. & Dharmaparakash, S. M. (2006b). *Acta Cryst.* **E62**, o1710–o1712.
- Pietta, P., Gardana, C. & Pietta, A. (2003). *Flavonoids in Health and Disease*. 2nd ed. edited by C. A. Rice-Evans & L. Packer. New York: Marcel Dekker, Inc.
- Rosli, M. M., Patil, P. S., Fun, H.-K., Razak, I. A. & Dharmaparakash, S. M. (2006). *Acta Cryst.* **E62**, o4228–o4230.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Teh, J. B.-J., Patil, P. S., Fun, H.-K., Razak, I. A. & Dharmaparakash, S. M. (2006a). *Acta Cryst.* **E62**, o890–o892.

Teh, J. B.-J., Patil, P. S., Fun, H.-K., Razak, I. A. & Dharmaprasad, S. M. (2006b). *Acta Cryst.* **E62**, o2399–o2400.
Teh, J. B.-J., Patil, P. S., Fun, H.-K., Razak, I. A. & Dharmaprasad, S. M. (2006c). *Acta Cryst.* **E62**, o2991–o2992.

Uchida, T., Kozawa, K., Sakai, T., Aoki, M., Yoguchi, H., Abdureyim, A. & Watanabe, Y. (1998). *Mol. Cryst. Liq. Cryst.* **62**, 135–140.

supplementary materials

Acta Cryst. (2010). E66, o1798-o1799 [doi:10.1107/S1600536810022142]

2',3,4,4'-Tetramethoxychalcone

J. H. van Tonder, T. J. Muller and B. C. B. Bezuidenhoudt

Comment

Flavonoids are a prominent class of secondary plant metabolites known for their wide range of biological active compounds that exhibit antibacterial, antifungal, antitumor and anti-inflammatory properties (Pietta *et al.*, 2003). Chalcones are an important subclass of these compounds and are often utilized as intermediates in the synthesis of a variety of cyclic flavonoids (Marais *et al.*, 2005). Furthermore, many chalcone derivatives are known to have excellent non-linear optical (NLO) properties (Fichou *et al.*, 1988; Uchida *et al.*, 1998; Patil *et al.*, 2006a,b). We report here a new chalcone which we have successfully synthesized (the title compound, (I)). Bond distances in (I) have normal values (Allen *et al.*, 1987) and bond angles and distances are comparable to those in related structures (Teh *et al.*, 2006a,b,c; Patil *et al.*, 2006a,b,c; Rosli *et al.* 2006). The least squares plane through the enone group (C7, C8, C9 and O3) exhibit dihedral angles of 29.2 (1)° and 4.5 (1)° with the C1—C6 and C10—C15 benzene rings, respectively. The dihedral angle between the two benzene rings is 26.88 (5)°. The methoxy group attached at C1 is slightly twisted away from the C1—C6 benzene ring plane, with a C16—O1—C1—C2 torsion angle of -12.8 (3)°. The methoxy groups at C3, C12 and C13 are almost co-planar with the C1—C6 and C10—C15 benzene rings with C17—O2—C3—C2, C18—O4—C12—C11 and C19—O5—C13—C14 torsion angles of -3.2 (3)°, 2.6 (3)° and -3.6 (3)°, respectively. An intramolecular C8—H8···O1 hydrogen bond is observed in the molecular structure of (I), while the molecules form chains through intermolecular C18—H18B···O3¹ hydrogen bonds (Table 1).

Experimental

The title compound was synthesized according to the procedure by Kraus *et al.* (2008) Freshly ground KOH (1.55 g; 27.80 mmol; 5 eq.) was added to a cold (ice bath) stirring solution of 2',4'-dimethoxyacetophenone (1.00 g; 5.56 mmol) and 3,4-dimethoxybenzaldehyde (1.13 g; 7.12 mmol; 1.2 eq.) in EtOH (40 ml). The reaction mixture was allowed to heat to room temperature and stirred to completion (TLC). Ice was added to the reaction mixture prior to acidification with concentrated HCl (litmus). Extraction was performed with EtOAc (3 x 100 ml) and the organic fractions combined. The organic phase was neutralized with a saturated solution of NaHCO₃ (litmus), washed with water, dried (Na₂SO₄) and evaporated *in vacuo* at ca 40 °C. Crystallization from EtOH afforded the desired chalcone (1.55 g; 84.7%) as yellow needles. *R*_f0.16 (H:A; 8:2); Mp 88.3 °C; ¹H NMR (600 MHz, CDCl₃) δ 7.69 (1H, d, *J* = 8.61 Hz, H-6'), 7.58 (1H, d, *J* = 15.71 Hz, H-β), 7.33 (1H, d, *J* = 15.71 Hz, H-α), 7.14 (1H, dd, *J* = 1.92, 8.32 Hz, H-5), 7.08 (1H, d, *J* = 1.92 Hz, H-2), 6.84 (1H, d, *J* = 8.32 Hz, H-6), 6.52 (1H, dd, *J* = 2.25, 8.61 Hz, H-5'), 6.46 (1H, d, *J* = 2.25 Hz, H-3'), 3.88 (3H, s, -OCH₃), 3.87 (3H, s, -OCH₃), 3.85 (3H, s, -OCH₃), 3.82 (3H, s, -OCH₃); ¹³C NMR (151 MHz, CDCl₃) δ 190.58, 163.96, 160.21, 150.95, 149.11, 142.34 (C-β), 132.63 (C-6'), 128.39, 125.28 (C-α), 122.60 (C-5), 122.36, 111.13 (C-6), 110.24 (C-2), 105.14 (C-5'), 98.66 (C-3'), 55.94 (-OCH₃), 55.86 (-OCH₃), 55.71 (-OCH₃), 55.50 (-OCH₃).

Refinement

The aromatic H atoms were placed in geometrically idealized positions and constrained to ride on its parent atoms with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ and at a distance of 0.93 Å. The hydrogen atoms of the methine (H8 and H9) group were determined from a difference Fourier map and their positional parameters freely refined (C8—H8 = 0.96 (2) Å and C9—H9 = 1.01 (2) Å). The methyl H atoms were placed in geometrically idealized positions and constrained to ride on its parent atoms with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ and at a distance of 0.96 Å.

Figures

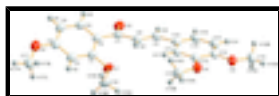


Fig. 1. Representation of the title compound, showing the numbering scheme and displacement ellipsoids (50% probability).

1-(2,4-dimethoxyphenyl)-3-(3,4-dimethoxyphenyl)prop-2-en-1-one

Crystal data

$\text{C}_{19}\text{H}_{20}\text{O}_5$

$M_r = 328.35$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 12.5839$ (7) Å

$b = 11.7204$ (7) Å

$c = 12.1339$ (6) Å

$\beta = 109.489$ (2)°

$V = 1687.07$ (16) Å³

$Z = 4$

$F(000) = 696$

$D_x = 1.293$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 7154 reflections

$\theta = 2.4$ – 23.6 °

$\mu = 0.09$ mm⁻¹

$T = 100$ K

Plate, colourless

$0.49 \times 0.22 \times 0.07$ mm

Data collection

Bruker APEXII
diffractometer

graphite

phi and ω scans

Absorption correction: multi-scan
(*SADABS*; Bruker, 2008)

$T_{\text{min}} = 0.976$, $T_{\text{max}} = 0.994$

32681 measured reflections

4205 independent reflections

2539 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.039$

$\theta_{\text{max}} = 28.4$ °, $\theta_{\text{min}} = 1.7$ °

$h = -16 \rightarrow 16$

$k = -15 \rightarrow 15$

$l = -11 \rightarrow 16$

Refinement

Refinement on F^2

Least-squares matrix: full

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

$$R[F^2 > 2\sigma(F^2)] = 0.044$$

$$wR(F^2) = 0.188$$

$$S = 1.12$$

4205 reflections

225 parameters

0 restraints

Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of independent and constrained refinement

$$w = 1/[\sigma^2(F_o^2) + (0.0998P)^2 + 0.0628P]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

$$(\Delta/\sigma)_{\max} = 0.001$$

$$\Delta\rho_{\max} = 0.21 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.20 \text{ e } \text{\AA}^{-3}$$

Special details

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

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.57536 (15)	0.12721 (14)	0.10767 (13)	0.0572 (4)
C2	0.66866 (16)	0.07753 (15)	0.09002 (15)	0.0635 (5)
H2	0.7138	0.027	0.1449	0.076*
C3	0.69406 (17)	0.10359 (16)	-0.00938 (16)	0.0665 (5)
C4	0.62632 (18)	0.17787 (18)	-0.09154 (16)	0.0724 (5)
H4	0.6422	0.1941	-0.1594	0.087*
C5	0.53595 (17)	0.22720 (17)	-0.07222 (14)	0.0654 (5)
H5	0.4918	0.2781	-0.1274	0.079*
C6	0.50707 (15)	0.20426 (14)	0.02731 (13)	0.0555 (4)
C7	0.40909 (15)	0.26733 (15)	0.04011 (13)	0.0582 (4)
C8	0.33980 (16)	0.21568 (16)	0.10368 (15)	0.0603 (5)
C9	0.25176 (15)	0.26831 (16)	0.11757 (14)	0.0596 (5)
C10	0.18227 (14)	0.22631 (15)	0.18413 (14)	0.0570 (4)
C11	0.20392 (14)	0.12066 (15)	0.24240 (14)	0.0571 (4)
H11	0.2627	0.0756	0.2367	0.068*
C12	0.14066 (14)	0.08267 (15)	0.30728 (14)	0.0574 (4)
C13	0.05259 (14)	0.15026 (17)	0.31711 (15)	0.0614 (5)
C14	0.02979 (16)	0.25345 (18)	0.26000 (16)	0.0692 (5)
H14	-0.0292	0.2982	0.2656	0.083*
C15	0.09404 (16)	0.29117 (17)	0.19425 (16)	0.0676 (5)
H15	0.0777	0.3612	0.1563	0.081*
C16	0.60374 (18)	0.01862 (18)	0.28377 (17)	0.0782 (6)
H16A	0.5762	0.0164	0.3486	0.117*

supplementary materials

H16B	0.6836	0.0318	0.312	0.117*
H16C	0.5882	-0.0529	0.2429	0.117*
C17	0.86153 (19)	-0.0100 (2)	0.0519 (2)	0.0954 (7)
H17A	0.9212	-0.0335	0.0241	0.143*
H17B	0.8224	-0.0761	0.0653	0.143*
H17C	0.8927	0.0315	0.1237	0.143*
C18	0.24973 (17)	-0.08614 (17)	0.36379 (18)	0.0710 (5)
H18A	0.2528	-0.1544	0.4085	0.107*
H18B	0.3184	-0.0439	0.3967	0.107*
H18C	0.2405	-0.1061	0.2844	0.107*
C19	-0.09188 (18)	0.1716 (2)	0.4026 (2)	0.0927 (7)
H19A	-0.1252	0.1302	0.451	0.139*
H19B	-0.1484	0.1879	0.3286	0.139*
H19C	-0.0607	0.2418	0.4403	0.139*
O1	0.54938 (13)	0.10796 (12)	0.20672 (10)	0.0780 (4)
O2	0.78452 (13)	0.06147 (14)	-0.03354 (13)	0.0874 (5)
O3	0.38578 (12)	0.36122 (11)	-0.00560 (11)	0.0767 (4)
O4	0.15725 (11)	-0.01826 (11)	0.36644 (12)	0.0717 (4)
O5	-0.00515 (11)	0.10484 (13)	0.38441 (12)	0.0785 (4)
H8	0.3591 (17)	0.1401 (19)	0.1332 (17)	0.075 (6)*
H9	0.2294 (16)	0.3460 (17)	0.0808 (16)	0.071 (5)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0751 (11)	0.0504 (9)	0.0446 (8)	-0.0050 (8)	0.0182 (8)	-0.0034 (7)
C2	0.0785 (12)	0.0527 (10)	0.0560 (9)	-0.0032 (9)	0.0180 (9)	-0.0043 (8)
C3	0.0762 (12)	0.0620 (12)	0.0645 (10)	-0.0128 (9)	0.0279 (9)	-0.0147 (9)
C4	0.0920 (14)	0.0757 (13)	0.0567 (10)	-0.0123 (11)	0.0342 (10)	-0.0027 (9)
C5	0.0802 (13)	0.0633 (11)	0.0498 (9)	-0.0111 (9)	0.0176 (8)	0.0044 (8)
C6	0.0685 (10)	0.0508 (9)	0.0431 (8)	-0.0099 (8)	0.0132 (7)	-0.0021 (7)
C7	0.0725 (11)	0.0511 (10)	0.0446 (8)	-0.0058 (8)	0.0110 (7)	0.0012 (7)
C8	0.0714 (12)	0.0497 (10)	0.0566 (9)	-0.0034 (9)	0.0171 (8)	0.0030 (8)
C9	0.0670 (11)	0.0513 (10)	0.0526 (9)	-0.0035 (9)	0.0094 (8)	-0.0001 (8)
C10	0.0586 (10)	0.0538 (10)	0.0518 (8)	-0.0006 (8)	0.0092 (7)	-0.0036 (7)
C11	0.0563 (9)	0.0538 (10)	0.0592 (9)	0.0007 (8)	0.0167 (8)	-0.0018 (8)
C12	0.0565 (10)	0.0527 (10)	0.0593 (9)	-0.0041 (8)	0.0143 (8)	-0.0027 (8)
C13	0.0541 (9)	0.0673 (12)	0.0610 (9)	-0.0043 (8)	0.0168 (8)	-0.0096 (9)
C14	0.0586 (11)	0.0715 (13)	0.0739 (11)	0.0105 (9)	0.0172 (9)	-0.0051 (10)
C15	0.0668 (11)	0.0596 (11)	0.0677 (11)	0.0070 (9)	0.0110 (9)	0.0027 (9)
C16	0.0932 (14)	0.0743 (13)	0.0629 (10)	0.0076 (11)	0.0204 (10)	0.0213 (10)
C17	0.0753 (14)	0.1100 (19)	0.0954 (16)	0.0027 (13)	0.0213 (12)	-0.0291 (14)
C18	0.0812 (13)	0.0533 (11)	0.0818 (12)	0.0052 (9)	0.0316 (10)	0.0076 (9)
C19	0.0673 (12)	0.125 (2)	0.0908 (14)	0.0056 (13)	0.0336 (11)	-0.0140 (14)
O1	0.1039 (10)	0.0834 (10)	0.0517 (7)	0.0266 (8)	0.0326 (7)	0.0199 (6)
O2	0.0914 (10)	0.0909 (11)	0.0904 (10)	-0.0003 (8)	0.0444 (8)	-0.0111 (8)
O3	0.0972 (10)	0.0607 (8)	0.0724 (8)	0.0086 (7)	0.0284 (7)	0.0193 (7)
O4	0.0769 (9)	0.0598 (8)	0.0869 (9)	0.0028 (6)	0.0385 (7)	0.0107 (6)

O5 0.0707 (8) 0.0858 (10) 0.0877 (9) -0.0012 (7) 0.0378 (7) -0.0051 (7)

Geometric parameters (Å, °)

C1—O1	1.3654 (19)	C12—C13	1.399 (2)
C1—C2	1.390 (3)	C13—O5	1.369 (2)
C1—C6	1.395 (2)	C13—C14	1.376 (3)
C2—C3	1.381 (3)	C14—C15	1.384 (3)
C2—H2	0.93	C14—H14	0.93
C3—O2	1.359 (2)	C15—H15	0.93
C3—C4	1.382 (3)	C16—O1	1.418 (2)
C4—C5	1.364 (3)	C16—H16A	0.96
C4—H4	0.93	C16—H16B	0.96
C5—C6	1.398 (2)	C16—H16C	0.96
C5—H5	0.93	C17—O2	1.430 (3)
C6—C7	1.489 (3)	C17—H17A	0.96
C7—O3	1.223 (2)	C17—H17B	0.96
C7—C8	1.473 (3)	C17—H17C	0.96
C8—C9	1.327 (3)	C18—O4	1.419 (2)
C8—H8	0.96 (2)	C18—H18A	0.96
C9—C10	1.460 (3)	C18—H18B	0.96
C9—H9	1.01 (2)	C18—H18C	0.96
C10—C15	1.384 (3)	C19—O5	1.418 (3)
C10—C11	1.407 (2)	C19—H19A	0.96
C11—C12	1.367 (2)	C19—H19B	0.96
C11—H11	0.93	C19—H19C	0.96
C12—O4	1.363 (2)		
O1—C1—C2	121.95 (15)	O5—C13—C12	115.08 (17)
O1—C1—C6	116.73 (16)	C14—C13—C12	119.51 (17)
C2—C1—C6	121.24 (15)	C13—C14—C15	120.49 (17)
C3—C2—C1	119.70 (17)	C13—C14—H14	119.8
C3—C2—H2	120.1	C15—C14—H14	119.8
C1—C2—H2	120.1	C14—C15—C10	121.04 (18)
O2—C3—C2	124.22 (19)	C14—C15—H15	119.5
O2—C3—C4	115.62 (17)	C10—C15—H15	119.5
C2—C3—C4	120.16 (18)	O1—C16—H16A	109.5
C5—C4—C3	119.46 (17)	O1—C16—H16B	109.5
C5—C4—H4	120.3	H16A—C16—H16B	109.5
C3—C4—H4	120.3	O1—C16—H16C	109.5
C4—C5—C6	122.60 (18)	H16A—C16—H16C	109.5
C4—C5—H5	118.7	H16B—C16—H16C	109.5
C6—C5—H5	118.7	O2—C17—H17A	109.5
C1—C6—C5	116.82 (17)	O2—C17—H17B	109.5
C1—C6—C7	125.93 (15)	H17A—C17—H17B	109.5
C5—C6—C7	117.21 (15)	O2—C17—H17C	109.5
O3—C7—C8	120.85 (17)	H17A—C17—H17C	109.5
O3—C7—C6	118.74 (16)	H17B—C17—H17C	109.5
C8—C7—C6	120.39 (15)	O4—C18—H18A	109.5
C9—C8—C7	122.73 (18)	O4—C18—H18B	109.5

supplementary materials

C9—C8—H8	120.1 (12)	H18A—C18—H18B	109.5
C7—C8—H8	117.1 (12)	O4—C18—H18C	109.5
C8—C9—C10	126.39 (18)	H18A—C18—H18C	109.5
C8—C9—H9	118.9 (11)	H18B—C18—H18C	109.5
C10—C9—H9	114.7 (11)	O5—C19—H19A	109.5
C15—C10—C11	117.73 (17)	O5—C19—H19B	109.5
C15—C10—C9	120.67 (17)	H19A—C19—H19B	109.5
C11—C10—C9	121.57 (16)	O5—C19—H19C	109.5
C12—C11—C10	121.57 (16)	H19A—C19—H19C	109.5
C12—C11—H11	119.2	H19B—C19—H19C	109.5
C10—C11—H11	119.2	C1—O1—C16	119.85 (15)
O4—C12—C11	124.71 (16)	C3—O2—C17	118.05 (17)
O4—C12—C13	115.65 (15)	C12—O4—C18	117.21 (14)
C11—C12—C13	119.64 (17)	C13—O5—C19	117.86 (18)
O5—C13—C14	125.41 (17)		
O1—C1—C2—C3	-177.22 (16)	C15—C10—C11—C12	-0.2 (2)
C6—C1—C2—C3	-0.7 (3)	C9—C10—C11—C12	178.26 (15)
C1—C2—C3—O2	178.59 (16)	C10—C11—C12—O4	-179.63 (15)
C1—C2—C3—C4	-0.7 (3)	C10—C11—C12—C13	-0.4 (2)
O2—C3—C4—C5	-177.74 (17)	O4—C12—C13—O5	-0.4 (2)
C2—C3—C4—C5	1.6 (3)	C11—C12—C13—O5	-179.68 (14)
C3—C4—C5—C6	-1.2 (3)	O4—C12—C13—C14	-179.86 (15)
O1—C1—C6—C5	177.78 (15)	C11—C12—C13—C14	0.9 (3)
C2—C1—C6—C5	1.1 (2)	O5—C13—C14—C15	179.90 (17)
O1—C1—C6—C7	0.2 (2)	C12—C13—C14—C15	-0.7 (3)
C2—C1—C6—C7	-176.47 (15)	C13—C14—C15—C10	0.1 (3)
C4—C5—C6—C1	-0.2 (3)	C11—C10—C15—C14	0.3 (3)
C4—C5—C6—C7	177.62 (16)	C9—C10—C15—C14	-178.11 (16)
C1—C6—C7—O3	150.16 (17)	C2—C1—O1—C16	-12.8 (3)
C5—C6—C7—O3	-27.4 (2)	C6—C1—O1—C16	170.51 (17)
C1—C6—C7—C8	-31.7 (2)	C2—C3—O2—C17	-3.2 (3)
C5—C6—C7—C8	150.79 (16)	C4—C3—O2—C17	176.09 (18)
O3—C7—C8—C9	-1.9 (3)	C11—C12—O4—C18	2.6 (2)
C6—C7—C8—C9	179.96 (15)	C13—C12—O4—C18	-176.60 (15)
C7—C8—C9—C10	-176.58 (15)	C14—C13—O5—C19	-3.6 (3)
C8—C9—C10—C15	178.36 (17)	C12—C13—O5—C19	176.98 (16)
C8—C9—C10—C11	0.0 (3)		

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C8—H8 \cdots O1	0.96 (2)	2.29 (2)	2.813 (3)	113.6 (15)
C18—H18B \cdots O3 ⁱ	0.96	2.46	3.253 (3)	140

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

Fig. 1

