



Crystal structure of 5-(4-methylphenyl)-3-[(E)-2-(4-methylphenyl)ethenyl]cyclohex-2-en-1-one

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Received 9 April 2015; accepted 28 April 2015

Edited by M. Lopez-Rodriguez, Universidad de La Laguna, Tenerife

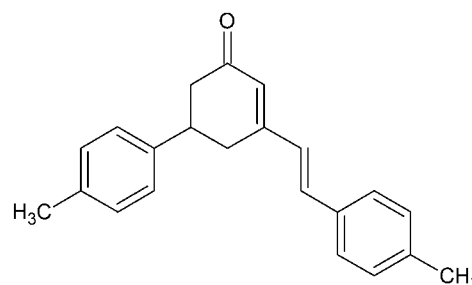
In the title compound, C₂₂H₂₂O, the dihedral angle between the planes of the benzene rings is 53.55 (7)°. Weak C—H···O interactions help to direct the packing, forming sheets lying parallel to (020).

Keywords: crystal structure; cyclohexenones; α,β -unsaturated ketones; C—H···O interactions.

CCDC reference: 1062089

1. Related literature

For the synthesis of cyclohexenones and their use as synthons, see: Mayekar *et al.* (2010); Suwito *et al.* (2014); Tabba *et al.* (1995); Bella *et al.* (2012); Xing *et al.* (2010); Martin & Prasad (2006). For various biological activities of cyclohexenone derivatives, see: Prasad *et al.* (2006); Kumar *et al.* (2003); Tatsuzaki *et al.* (2006); Yun *et al.* (2006); Kim *et al.* (2008); Yoon *et al.* (2007); Tanaka *et al.* (1997); Vyas *et al.* (2009). For the use of cyclohexenones as intermediates in synthesis, see: Mayekar *et al.* (2010); Bella *et al.* (2012); Xing *et al.* (2010); Martin & Prasad (2006). For the bioactivity of dehydrozingerone, chalcone and isoeugenol derivatives, see: Tatsuzaki *et al.* (2006).



2. Experimental

2.1. Crystal data

C₂₂H₂₂O
M_r = 302.39
 Monoclinic, *P*2₁/*c*
a = 4.9614 (1) Å
b = 30.7302 (6) Å
c = 11.0726 (2) Å
 β = 93.268 (1)°

V = 1685.44 (6) Å³
Z = 4
 Cu *K* α radiation
 μ = 0.55 mm⁻¹
T = 150 K
 0.31 × 0.11 × 0.08 mm

2.2. Data collection

Bruker D8 VENTURE PHOTON 12558 measured reflections
 100 CMOS diffractometer 3247 independent reflections
 Absorption correction: multi-scan 2529 reflections with *I* > 2 σ (*I*)
 (SADABS; Bruker, 2014) *R_{int}* = 0.042
T_{min} = 0.84, *T_{max}* = 0.96

2.3. Refinement

R[*F*² > 2 σ (*F*²)] = 0.050 210 parameters
wR(*F*²) = 0.131 H-atom parameters constrained
S = 1.05 $\Delta\rho_{\max}$ = 0.35 e Å⁻³
 3247 reflections $\Delta\rho_{\min}$ = -0.19 e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
C6—H6A···O1 ⁱ	0.99	2.60	3.515 (2)	154
C8—H8···O1 ⁱⁱ	0.95	2.47	3.353 (2)	155
C14—H14···O1 ⁱⁱ	0.95	2.55	3.410 (2)	151

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

Data collection: *APEX2* (Bruker, 2014); cell refinement: *SAINT* (Bruker, 2014); data reduction: *SAINT*; program(s) used to solve structure: *SHELXT* (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015b); molecular graphics: *DIAMOND* (Brandenburg & Putz, 2012); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

Acknowledgements

The support of NSF-MRI grant No. 1228232 for the purchase of the diffractometer and Tulane University for support of the Tulane Crystallography Laboratory are gratefully acknowledged.

Supporting information for this paper is available from the IUCr electronic archives (Reference: LR2135).

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supporting information

Acta Cryst. (2015). E71, o436–o437 [doi:10.1107/S2056989015008324]

Crystal structure of 5-(4-methylphenyl)-3-[(*E*)-2-(4-methylphenyl)ethenyl]cyclohex-2-en-1-one

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S1. Structural commentary

From a chemical point of view, the most commonly used method for preparation of polyfunctionalized cyclohexenones is the Michael addition of carbanions to α,β -unsaturated ketones in presence of basic catalysts (Mayekar *et al.*, 2010; Suwito *et al.*, 2014; Tabba *et al.*, 1995). Cyclohexenones have been considered as efficient synthons in building spiranic compounds (Mayekar *et al.*, 2010) or intermediates in the synthesis of fused heterocycles such as benzoselenadiazoles and benzothiazoles (Bella *et al.*, 2012), benzopyrazoles (Xing *et al.*, 2010) or carbazole derivatives (Martin & Prasad, 2006). The existence of the α,β -unsaturated ketone moiety is a common feature of a large number of biologically active compounds which exhibit diverse pharmacological effects such as anti-microbial (Prasad *et al.*, 2006), anti-tumor (Kumar *et al.*, 2003), anti-cancer (Tatsuzaki *et al.*, 2006; Yun *et al.*, 2006) and radical scavenger activities (Kim *et al.*, 2008) as well as being inhibitors of topoisomerase I (Yoon *et al.*, 2007). Cyclohexenone derivatives, in particular, are well known lead molecules for the treatment of inflammation and autoimmune diseases (Tanaka *et al.*, 1997). Several reports have pointed out the importance of cyclohexenones for anti-microbial and anti-tubercular activity (Vyas *et al.*, 2009).

In the title compound (Fig. 1), the dihedral angle between the phenyl rings is 53.55 (7)°. Weak C6—H6A \cdots O1ⁱ (*i*: $x + 1, y, z$) interactions help to direct the packing (Fig. 2 and Table 1).

S2. Synthesis and crystallization

In 30 ml of methanol, a mixture of 1 mmol (262 mg) of (1*Z*,4*E*)-1,5-bis(4-methylphenyl)penta-1,4-dien-3-one and 1 mmol (100 mg) of acetylacetone was refluxed for 5 h in the presence of 10 mg of sodium methoxide. The resulting solid product was collected, filtered under vacuum, washed with cold ethanol and recrystallized from ethanol to afford colourless columns which were suitable for X-ray diffraction. Mp. 371 K.

S3. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. H-atoms were placed in calculated positions (C—H = 0.95 - 0.98 Å) and included as riding contributions with isotropic displacement parameters 1.2 - 1.5 times those of the attached carbon atoms. The 020 reflection was omitted from the final refinement as it was partially obscured by the beamstop.

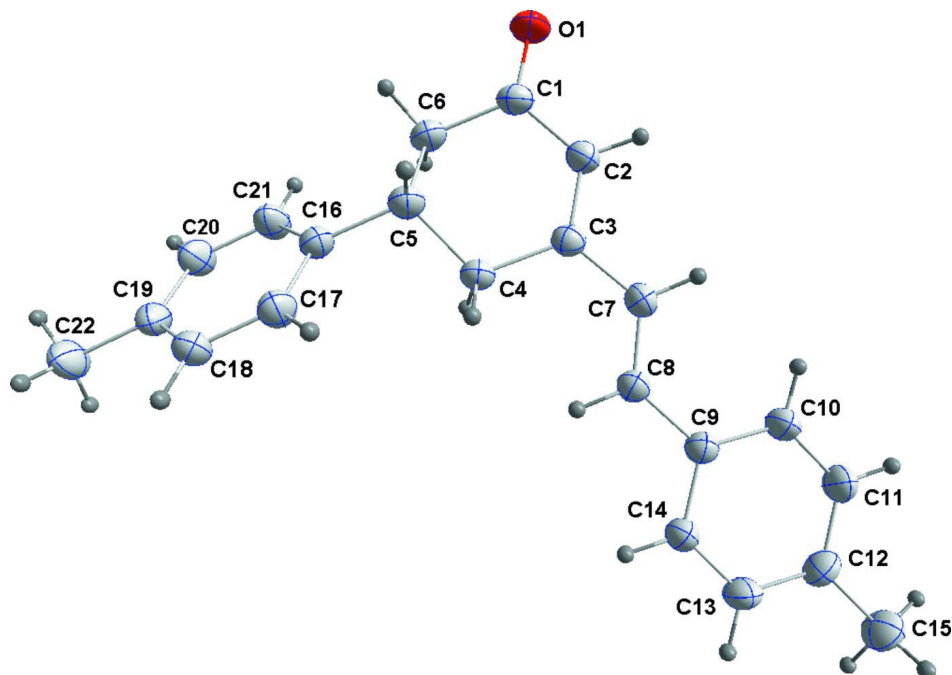


Figure 1

The title molecule with labeling scheme and 50% probability ellipsoids.

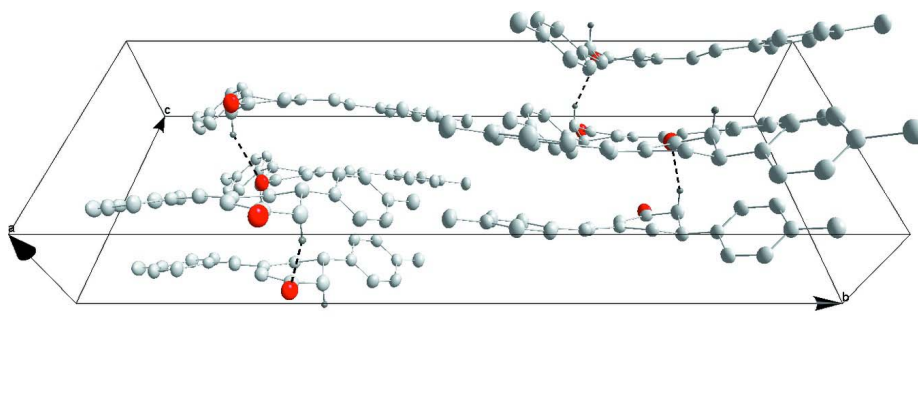


Figure 2

Packing viewed towards the $(10\bar{2})$ plane. Weak C—H...O interactions are shown as dotted lines.

5-(4-Methylphenyl)-3-[(*E*)-2-(4-methylphenyl)ethenyl]cyclohex-2-en-1-one

Crystal data

$C_{22}H_{22}O$

$M_r = 302.39$

Monoclinic, $P2_1/c$

$a = 4.9614 (1) \text{ \AA}$

$b = 30.7302 (6) \text{ \AA}$

$c = 11.0726 (2) \text{ \AA}$

$\beta = 93.268 (1)^\circ$

$V = 1685.44 (6) \text{ \AA}^3$

$Z = 4$

$F(000) = 648$

$D_x = 1.192 \text{ Mg m}^{-3}$

Cu $K\alpha$ radiation, $\lambda = 1.54178 \text{ \AA}$

Cell parameters from 7504 reflections

$\theta = 2.9\text{--}72.6^\circ$

$\mu = 0.55 \text{ mm}^{-1}$

$T = 150 \text{ K}$

Column, colourless

$0.31 \times 0.11 \times 0.08 \text{ mm}$

Data collection

Bruker D8 VENTURE PHOTON 100 CMOS
diffractometer
Radiation source: INCOATEC I μ S micro-focus
source
Mirror monochromator
Detector resolution: 10.4167 pixels mm⁻¹
 ω scans
Absorption correction: multi-scan
(SADABS; Bruker, 2014)

$T_{\min} = 0.84$, $T_{\max} = 0.96$
12558 measured reflections
3247 independent reflections
2529 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.042$
 $\theta_{\max} = 72.4^\circ$, $\theta_{\min} = 4.3^\circ$
 $h = -5 \rightarrow 6$
 $k = -38 \rightarrow 36$
 $l = -11 \rightarrow 13$

Refinement

Refinement on F^2
Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.050$
 $wR(F^2) = 0.131$
 $S = 1.05$
3247 reflections
210 parameters
0 restraints
Primary atom site location: structure-invariant
direct methods

Secondary atom site location: difference Fourier
map
Hydrogen site location: inferred from
neighbouring sites
H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0579P)^2 + 0.6292P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.35 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.19 \text{ e } \text{\AA}^{-3}$

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'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 $F^2 > \sigma(F^2)$ 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. H-atoms were placed in calculated positions (C—H = 0.95 - 0.98 Å) and included as riding contributions with isotropic displacement parameters 1.2 - 1.5 times those of the attached carbon atoms.

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	-0.2370 (3)	0.78680 (4)	0.50574 (12)	0.0422 (3)
C1	-0.0733 (4)	0.77848 (6)	0.42988 (16)	0.0324 (4)
C2	-0.0376 (4)	0.73445 (6)	0.38590 (16)	0.0320 (4)
H2	-0.1435	0.7119	0.4177	0.038*
C3	0.1391 (3)	0.72401 (5)	0.30189 (15)	0.0280 (4)
C4	0.2939 (4)	0.75888 (5)	0.23953 (15)	0.0298 (4)
H4A	0.2923	0.7521	0.1521	0.036*
H4B	0.4841	0.7583	0.2718	0.036*
C5	0.1810 (4)	0.80493 (6)	0.25518 (16)	0.0331 (4)
H5	0.0083	0.8068	0.2043	0.040*
C6	0.1127 (4)	0.81281 (6)	0.38459 (17)	0.0369 (4)
H6A	0.2814	0.8132	0.4368	0.044*

H6B	0.0257	0.8417	0.3905	0.044*
C7	0.1933 (4)	0.67865 (6)	0.27681 (16)	0.0313 (4)
H7	0.0800	0.6575	0.3107	0.038*
C8	0.3907 (4)	0.66425 (5)	0.20954 (15)	0.0295 (4)
H8	0.4884	0.6858	0.1686	0.035*
C9	0.4725 (3)	0.61914 (6)	0.19209 (15)	0.0296 (4)
C10	0.3664 (4)	0.58378 (6)	0.25307 (18)	0.0389 (4)
H10	0.2265	0.5885	0.3067	0.047*
C11	0.4619 (4)	0.54214 (6)	0.23644 (18)	0.0392 (4)
H11	0.3877	0.5188	0.2800	0.047*
C12	0.6632 (4)	0.53343 (6)	0.15784 (17)	0.0355 (4)
C13	0.7678 (4)	0.56848 (6)	0.09696 (17)	0.0394 (5)
H13	0.9057	0.5635	0.0424	0.047*
C14	0.6762 (4)	0.61052 (6)	0.11375 (16)	0.0341 (4)
H14	0.7534	0.6338	0.0712	0.041*
C15	0.7656 (5)	0.48793 (6)	0.1393 (2)	0.0466 (5)
H15A	0.7277	0.4700	0.2095	0.070*
H15B	0.9608	0.4888	0.1300	0.070*
H15C	0.6750	0.4754	0.0664	0.070*
C16	0.3727 (4)	0.83857 (5)	0.20647 (15)	0.0310 (4)
C17	0.4337 (4)	0.83682 (6)	0.08492 (16)	0.0348 (4)
H17	0.3555	0.8146	0.0345	0.042*
C18	0.6061 (4)	0.86683 (6)	0.03656 (16)	0.0357 (4)
H18	0.6431	0.8648	-0.0465	0.043*
C19	0.7260 (4)	0.89973 (6)	0.10667 (17)	0.0339 (4)
C20	0.6674 (4)	0.90135 (6)	0.22736 (18)	0.0394 (4)
H20	0.7471	0.9234	0.2777	0.047*
C21	0.4943 (4)	0.87140 (6)	0.27656 (17)	0.0378 (4)
H21	0.4585	0.8734	0.3597	0.045*
C22	0.9152 (4)	0.93214 (7)	0.0529 (2)	0.0450 (5)
H22A	1.1003	0.9209	0.0606	0.068*
H22B	0.9060	0.9599	0.0960	0.068*
H22C	0.8619	0.9366	-0.0328	0.068*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0419 (8)	0.0429 (7)	0.0440 (8)	0.0040 (6)	0.0210 (6)	-0.0051 (6)
C1	0.0284 (9)	0.0378 (10)	0.0315 (9)	0.0061 (7)	0.0061 (7)	0.0004 (7)
C2	0.0292 (9)	0.0332 (9)	0.0344 (9)	0.0010 (7)	0.0078 (7)	0.0028 (7)
C3	0.0261 (8)	0.0302 (9)	0.0277 (9)	0.0019 (7)	0.0017 (6)	0.0011 (6)
C4	0.0315 (9)	0.0296 (9)	0.0287 (9)	0.0011 (7)	0.0067 (7)	-0.0015 (6)
C5	0.0362 (9)	0.0314 (9)	0.0323 (9)	0.0015 (7)	0.0074 (7)	-0.0021 (7)
C6	0.0390 (10)	0.0312 (9)	0.0417 (10)	0.0040 (8)	0.0116 (8)	-0.0033 (7)
C7	0.0325 (9)	0.0292 (9)	0.0329 (9)	-0.0021 (7)	0.0076 (7)	0.0017 (7)
C8	0.0340 (9)	0.0285 (8)	0.0263 (8)	-0.0009 (7)	0.0051 (7)	0.0002 (6)
C9	0.0326 (9)	0.0289 (9)	0.0276 (9)	-0.0004 (7)	0.0038 (7)	-0.0006 (6)
C10	0.0439 (11)	0.0328 (9)	0.0417 (10)	-0.0010 (8)	0.0176 (8)	-0.0004 (8)

C11	0.0471 (11)	0.0299 (9)	0.0415 (11)	-0.0027 (8)	0.0109 (8)	0.0032 (7)
C12	0.0420 (10)	0.0287 (9)	0.0354 (10)	0.0038 (8)	-0.0006 (8)	-0.0027 (7)
C13	0.0438 (11)	0.0358 (10)	0.0401 (10)	0.0053 (8)	0.0150 (8)	-0.0029 (8)
C14	0.0402 (10)	0.0306 (9)	0.0326 (9)	-0.0001 (7)	0.0115 (8)	0.0010 (7)
C15	0.0545 (13)	0.0334 (10)	0.0522 (13)	0.0081 (9)	0.0064 (10)	-0.0006 (9)
C16	0.0358 (9)	0.0267 (8)	0.0309 (9)	0.0027 (7)	0.0069 (7)	-0.0011 (7)
C17	0.0418 (10)	0.0309 (9)	0.0321 (9)	-0.0019 (8)	0.0058 (7)	-0.0044 (7)
C18	0.0418 (10)	0.0350 (10)	0.0312 (9)	0.0033 (8)	0.0102 (8)	0.0012 (7)
C19	0.0322 (9)	0.0305 (9)	0.0396 (10)	0.0032 (7)	0.0077 (7)	0.0023 (7)
C20	0.0435 (11)	0.0352 (10)	0.0399 (11)	-0.0067 (8)	0.0064 (8)	-0.0068 (8)
C21	0.0468 (11)	0.0361 (10)	0.0315 (10)	-0.0041 (8)	0.0099 (8)	-0.0056 (7)
C22	0.0444 (12)	0.0414 (11)	0.0506 (12)	-0.0057 (9)	0.0143 (9)	0.0013 (9)

Geometric parameters (Å, °)

O1—C1	1.228 (2)	C11—H11	0.9500
C1—C2	1.452 (2)	C12—C13	1.387 (3)
C1—C6	1.506 (3)	C12—C15	1.506 (2)
C2—C3	1.352 (2)	C13—C14	1.386 (2)
C2—H2	0.9500	C13—H13	0.9500
C3—C7	1.450 (2)	C14—H14	0.9500
C3—C4	1.508 (2)	C15—H15A	0.9800
C4—C5	1.535 (2)	C15—H15B	0.9800
C4—H4A	0.9900	C15—H15C	0.9800
C4—H4B	0.9900	C16—C21	1.390 (3)
C5—C6	1.511 (2)	C16—C17	1.397 (2)
C5—C16	1.524 (2)	C17—C18	1.386 (3)
C5—H5	1.0000	C17—H17	0.9500
C6—H6A	0.9900	C18—C19	1.388 (3)
C6—H6B	0.9900	C18—H18	0.9500
C7—C8	1.339 (2)	C19—C20	1.385 (3)
C7—H7	0.9500	C19—C22	1.514 (3)
C8—C9	1.460 (2)	C20—C21	1.391 (3)
C8—H8	0.9500	C20—H20	0.9500
C9—C14	1.394 (2)	C21—H21	0.9500
C9—C10	1.398 (2)	C22—H22A	0.9800
C10—C11	1.381 (3)	C22—H22B	0.9800
C10—H10	0.9500	C22—H22C	0.9800
C11—C12	1.388 (3)		
O1—C1—C2	121.44 (16)	C12—C11—H11	119.1
O1—C1—C6	121.55 (16)	C13—C12—C11	117.25 (16)
C2—C1—C6	116.89 (15)	C13—C12—C15	121.09 (17)
C3—C2—C1	123.22 (16)	C11—C12—C15	121.66 (17)
C3—C2—H2	118.4	C14—C13—C12	121.60 (17)
C1—C2—H2	118.4	C14—C13—H13	119.2
C2—C3—C7	119.64 (15)	C12—C13—H13	119.2
C2—C3—C4	120.88 (15)	C13—C14—C9	121.04 (16)

C7—C3—C4	119.37 (14)	C13—C14—H14	119.5
C3—C4—C5	113.86 (14)	C9—C14—H14	119.5
C3—C4—H4A	108.8	C12—C15—H15A	109.5
C5—C4—H4A	108.8	C12—C15—H15B	109.5
C3—C4—H4B	108.8	H15A—C15—H15B	109.5
C5—C4—H4B	108.8	C12—C15—H15C	109.5
H4A—C4—H4B	107.7	H15A—C15—H15C	109.5
C6—C5—C16	113.91 (15)	H15B—C15—H15C	109.5
C6—C5—C4	110.97 (14)	C21—C16—C17	117.07 (16)
C16—C5—C4	110.25 (14)	C21—C16—C5	123.69 (16)
C6—C5—H5	107.1	C17—C16—C5	119.24 (16)
C16—C5—H5	107.1	C18—C17—C16	121.24 (17)
C4—C5—H5	107.1	C18—C17—H17	119.4
C1—C6—C5	112.23 (15)	C16—C17—H17	119.4
C1—C6—H6A	109.2	C17—C18—C19	121.53 (17)
C5—C6—H6A	109.2	C17—C18—H18	119.2
C1—C6—H6B	109.2	C19—C18—H18	119.2
C5—C6—H6B	109.2	C20—C19—C18	117.33 (17)
H6A—C6—H6B	107.9	C20—C19—C22	121.69 (17)
C8—C7—C3	125.02 (16)	C18—C19—C22	120.98 (17)
C8—C7—H7	117.5	C19—C20—C21	121.51 (17)
C3—C7—H7	117.5	C19—C20—H20	119.2
C7—C8—C9	127.22 (16)	C21—C20—H20	119.2
C7—C8—H8	116.4	C16—C21—C20	121.32 (17)
C9—C8—H8	116.4	C16—C21—H21	119.3
C14—C9—C10	117.34 (16)	C20—C21—H21	119.3
C14—C9—C8	118.65 (15)	C19—C22—H22A	109.5
C10—C9—C8	123.97 (16)	C19—C22—H22B	109.5
C11—C10—C9	120.96 (17)	H22A—C22—H22B	109.5
C11—C10—H10	119.5	C19—C22—H22C	109.5
C9—C10—H10	119.5	H22A—C22—H22C	109.5
C10—C11—C12	121.80 (17)	H22B—C22—H22C	109.5
C10—C11—H11	119.1		
O1—C1—C2—C3	179.54 (18)	C10—C11—C12—C15	-179.6 (2)
C6—C1—C2—C3	-4.5 (3)	C11—C12—C13—C14	0.1 (3)
C1—C2—C3—C7	170.34 (16)	C15—C12—C13—C14	-179.66 (19)
C1—C2—C3—C4	-5.8 (3)	C12—C13—C14—C9	-0.6 (3)
C2—C3—C4—C5	-14.9 (2)	C10—C9—C14—C13	0.3 (3)
C7—C3—C4—C5	169.05 (15)	C8—C9—C14—C13	177.83 (17)
C3—C4—C5—C6	44.1 (2)	C6—C5—C16—C21	5.5 (3)
C3—C4—C5—C16	171.31 (15)	C4—C5—C16—C21	-120.04 (19)
O1—C1—C6—C5	-149.12 (18)	C6—C5—C16—C17	-174.85 (17)
C2—C1—C6—C5	34.9 (2)	C4—C5—C16—C17	59.6 (2)
C16—C5—C6—C1	-179.03 (15)	C21—C16—C17—C18	-0.6 (3)
C4—C5—C6—C1	-53.9 (2)	C5—C16—C17—C18	179.74 (17)
C2—C3—C7—C8	-169.72 (18)	C16—C17—C18—C19	0.2 (3)
C4—C3—C7—C8	6.4 (3)	C17—C18—C19—C20	0.2 (3)

C3—C7—C8—C9	172.86 (17)	C17—C18—C19—C22	179.64 (18)
C7—C8—C9—C14	177.15 (18)	C18—C19—C20—C21	-0.3 (3)
C7—C8—C9—C10	-5.5 (3)	C22—C19—C20—C21	-179.72 (19)
C14—C9—C10—C11	0.4 (3)	C17—C16—C21—C20	0.5 (3)
C8—C9—C10—C11	-176.95 (18)	C5—C16—C21—C20	-179.83 (18)
C9—C10—C11—C12	-0.9 (3)	C19—C20—C21—C16	0.0 (3)
C10—C11—C12—C13	0.6 (3)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
C6—H6A \cdots O1 ⁱ	0.99	2.60	3.515 (2)	154
C8—H8 \cdots O1 ⁱⁱ	0.95	2.47	3.353 (2)	155
C14—H14 \cdots O1 ⁱⁱ	0.95	2.55	3.410 (2)	151

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