Acta Cryst E CRYSTALLOGRAPHIC COMMUNICATIONS

OPEN ACCESS

ISSN 2056-9890

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

**Joel T. Mague,[a] Shaaban K. Mohamed,[b,c] Mehmet Akkurt,[d] Antar A. Abdelhamid[e] and Mustafa R. Albayati[f]***

[a]Department of Chemistry, Tulane University, New Orleans, LA 70118, USA, [b]Chemistry and Environmental Division, Manchester Metropolitan University, Manchester M1 5GD, England, [c]Chemistry Department, Faculty of Science, Minia University, 61519 El-Minia, Egypt, [d]Department of Physics, Faculty of Sciences, Erciyes University, 38039 Kayseri, Turkey, [e]Department of Chemistry, Faculty of Science, Sohag University, 82524 Sohag, Egypt, and [f]Kirkuk University, College of Science, Department of Chemistry, Kirkuk, Iraq. *Correspondence e-mail: shaabankamel@yahoo.com

Received 9 April 2015; accepted 28 April 2015

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

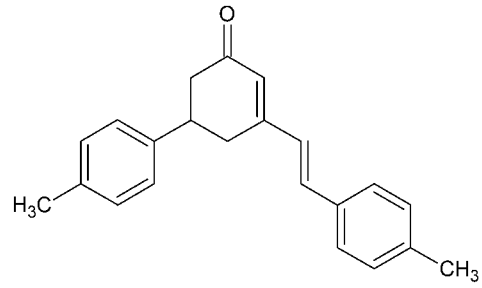
In the title compound, $C_{22}H_{22}O$, the dihedral angle between the planes of the benzene rings is 53.55 (7)°. Weak C—H···O interactions help to direct the packing, forming sheets lying parallel to (020).

**Keywords:** crystal structure; cyclohexenenones; $\alpha,\beta$-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

$C_{22}H_{22}O$
$M_r$ = 302.39
Monoclinic, $P2_1/c$
$a$ = 4.9614 (1) Å
$b$ = 30.7302 (6) Å
$c$ = 11.0726 (2) Å
$\beta$ = 93.268 (1)°
$V$ = 1685.44 (6) Å$^3$
$Z$ = 4
Cu $K\alpha$ radiation
$\mu$ = 0.55 mm$^{-1}$
$T$ = 150 K
0.31 × 0.11 × 0.08 mm

### 2.2. Data collection

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

### 2.3. Refinement

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

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C6—H6$A$···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, 2015*a*); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015*b*); 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).

## References


Bella, M., Schultz, M. & Milata, V. (2012). *Arkivoc*, **iv**, 242–251.
Brandenburg, K. & Putz, H. (2012). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
Bruker (2014). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Kim, B.-T. O. K.-J., Chun, J.-C. & Hwang, K.-J. (2008). *Bull. Korean Chem. Soc.* **29**, 1125–1130.
Kumar, S. K., Hager, E., Pettit, C., Gurulingappa, H., Davidson, N. E. & Khan, S. R. (2003). *J. Med. Chem.* **46**, 2813–2815.
Martin, A. E. & Prasad, K. J. (2006). *Acta Pharm.* **56**, 79–86.
Mayekar, A. N., Li, H., Yathirajan, H. S., Narayana, B. & Suchetha Kumari, N. (2010). *Int. J. Chem. (Can.)*, **2**, 114–123.
Prasad, Y. R., Kumar, P. R., Deepti, C. A. & Ramana, M. V. (2006). *Eur. J. Chem.* **3**, 236–241.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Sheldrick, G. M. (2015*a*). *Acta Cryst.* A**71**, 3–8.
Sheldrick, G. M. (2015*b*). *Acta Cryst.* C**71**, 3–8.
Suwito, H., Mustofa, J., Kristanti, A. N. & Puspaningsih, N. N. T. (2014). *J. Chem. Pharm. Res.* **6**, 1076–1088.
Tabba, H. D., Yousef, N. M. & Al-Arab, M. M. (1995). *Collect. Czech. Chem. Commun.* **60**, 594–604.
Tanaka, M., Nara, F., Suzuki, K., Hosoya, T. & Ogita, T. (1997). *J. Am. Chem. Soc.* **119**, 7871–7872.
Tatsuzaki, J., Bastow, K. F., Nakagawa-Goto, K., Nakamura, S., Itokawa, H. & Lee, K.-H. (2006). *J. Nat. Prod.* **69**, 1445–1449.
Vyas, D. H., Tala, S. D., Akbari, J. D., Dhaduk, M. F. & Joshi, H. S. (2009). *Indian J. Chem. Sect B*, **48**, 1405–1410.
Xing, R. G., Li, Y. N., Liu, Q., Meng, Q., Li, J., Shen, X., Liu, Z., Zhou, B., Yao, X. & Liu, Z. (2010). *Eur. J. Org. Chem.* pp. 6627–6632.
Yoon, G., Kang, B. Y. & Cheon, S. H. (2007). *Arch. Pharm. Res.* **30**, 313–316.
Yun, J.-M., Kweon, M.-H., Kwon, H., Hwang, J.-K. & Mukhtar, H. (2006). *Carcinogenesis*, **27**, 1454–1464.

# supporting information

*Acta Cryst.* (2015). E**71**, o436–o437 [doi:10.1107/S2056989015008324]

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

**Joel T. Mague, Shaaban K. Mohamed, Mehmet Akkurt, Antar A. Abdelhamid and Mustafa R. Albayati**

## 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···O1$^{i}$ (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*,4E)-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.

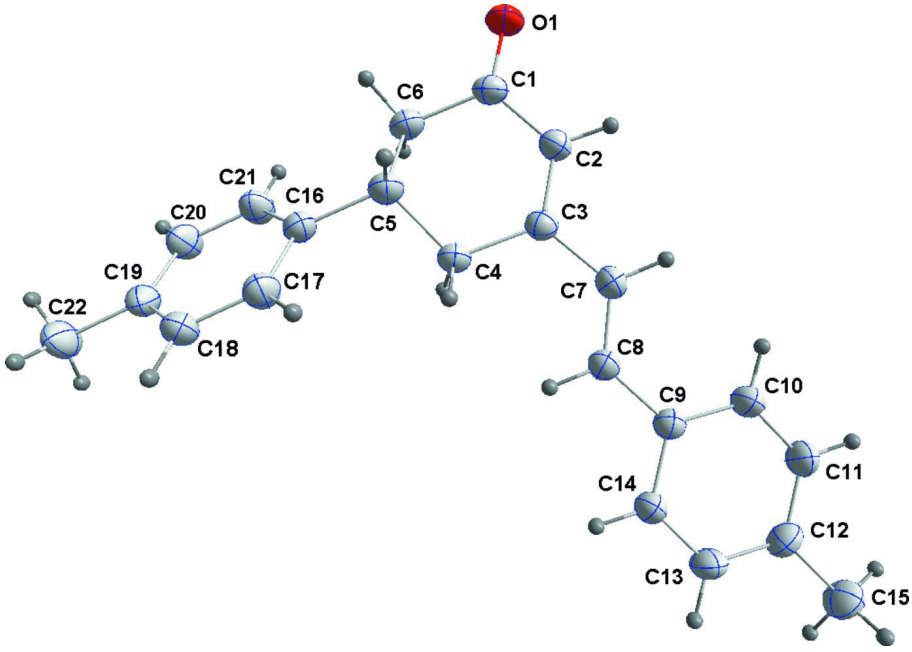



**Figure 1**

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

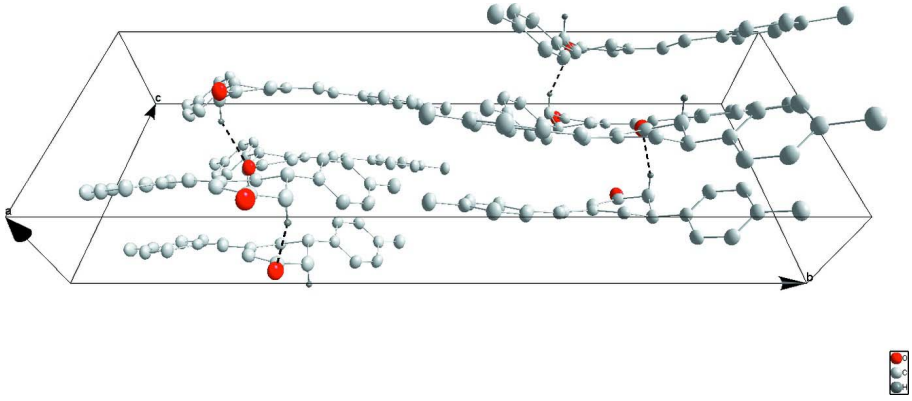


**Figure 2**

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) Å
$b = 30.7302$ (6) Å
$c = 11.0726$ (2) Å
$\beta = 93.268$ (1)°
$V = 1685.44$ (6) Å$^3$
$Z = 4$

$F(000) = 648$
$D_x = 1.192$ Mg m$^{-3}$
Cu $K\alpha$ radiation, $\lambda = 1.54178$ Å
Cell parameters from 7504 reflections
$\theta = 2.9$–$72.6°$
$\mu = 0.55$ mm$^{-1}$
$T = 150$ K
Column, colourless
0.31 × 0.11 × 0.08 mm

*Data collection*

Bruker D8 VENTURE PHOTON 100 CMOS diffractometer
Radiation source: INCOATEC I$\mu$S micro-focus source
Mirror monochromator
Detector resolution: 10.4167 pixels mm$^{-1}$
$\omega$ 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 $I > 2\sigma(I)$
$R_{int}$ = 0.042
$\theta_{max}$ = 72.4°, $\theta_{min}$ = 4.3°
$h$ = −5→6
$k$ = −38→36
$l$ = −11→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 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.19 e Å$^{-3}$

*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 (Å$^2$)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| 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 (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| 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, º)*

| | | | |
|---|---|---|---|
| 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, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C6—H6*A*···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*+3/2, *z*−1/2.