



Received 25 April 2019

Accepted 7 May 2019

Edited by W. T. A. Harrison, University of
Aberdeen, Scotland**Keywords:** crystal structure; bischalcone;
pentadienone bridge; C—H... π interactions;
Hirshfeld surface analysis.**CCDC reference:** 1914420**Supporting information:** this article has
supporting information at journals.iucr.org/e

Crystal structure and Hirshfeld surface analysis of a conformationally unsymmetrical bischalcone: (1*E*,4*E*)-1,5-bis(4-bromophenyl)penta-1,4-dien-3-one

Nabeel Arif Tawfeeq,^{a,b} Huey Chong Kwong,^a Mohamed Ibrahim Mohamed Tahir^{a*} and Thahira B. S. A. Ravooof^a^aDepartment of Chemistry, Faculty of Science, Universiti Putra Malaysia, 43400 UPM Serdang, Selangor Darul Ehsan, Malaysia, and ^bDepartment of Chemistry, College of Education for Women, University of Anbar, Iraq. *Correspondence e-mail: ibra@upm.edu.my

In the title bischalcone, C₁₇H₁₂Br₂O, the olefinic double bonds are almost coplanar with their attached 4-bromophenyl rings [torsion angles = −10.2 (4) and −6.2 (4)°], while the carbonyl double bond is in an *s-trans* conformation with respect to one of the C=C bonds and an *s-cis* conformation with respect to the other [C=C—C=O = 160.7 (3) and −15.2 (4)°, respectively]. The dihedral angle between the 4-bromophenyl rings is 51.56 (2)°. In the crystal, molecules are linked into a zigzag chain propagating along [001] by weak C—H... π interactions. The conformations of related bischalcones are surveyed and a Hirshfeld surface analysis is used to investigate and quantify the intermolecular contacts.

1. Chemical context

Dibenzalacetone, or bischalcone, [(1*E*,4*E*)-1,5-diphenylpenta-1,4-dien-3-one] was first prepared by the base-catalyzed Aldol condensation of benzaldehyde and acetone (Conard & Morris, 1932): it results in a highly conjugated system involving the α,β -unsaturated pentadienone (—C=C—(C=O)—C=C—) moiety. Bischalcones have a number of uses including anti-inflammatory (Mahapatra *et al.*, 2017) and anti-oxidant (Pandey & Syed, 2009) agents. Different bischalcones consist of two benzene rings substituted with different types of functional groups (electron donor or acceptor) bonded to the ends of the central α,β -unsaturated ketone which provides good configuration for the transfer of intramolecular charge (Fun *et al.*, 2011). In a continuation of our ongoing studies on the non-linear optical properties of various chalcone derivatives (Sim *et al.*, 2017; Kwong *et al.*, 2018), we report herein the synthesis, structure determination and Hirshfeld surface analysis of the title compound (I).

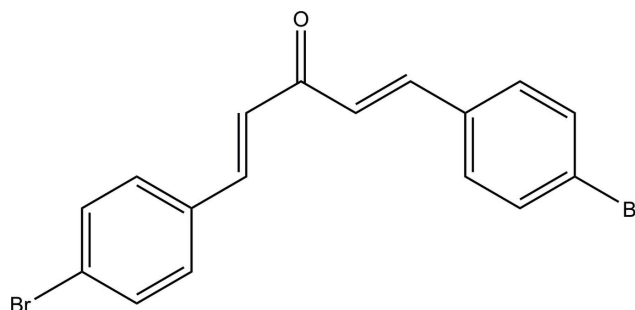
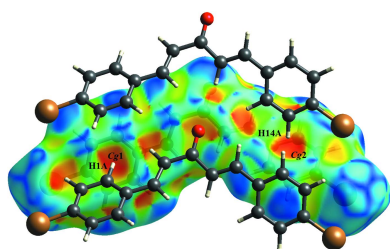


Table 1

Hydrogen-bond geometry (Å, °).

 $Cg1$ and $Cg2$ are the centroids of the C1–C16 and C12–C17 rings, respectively.

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|-------------------------------------|-------|-------------|-------------|---------------|
| C1–H1A \cdots Cg1 ⁱ | 0.93 | 2.86 | 3.539 (3) | 131 |
| C14–H14A \cdots Cg2 ⁱⁱ | 0.93 | 2.74 | 3.428 (3) | 131 |

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

2. Structural commentary

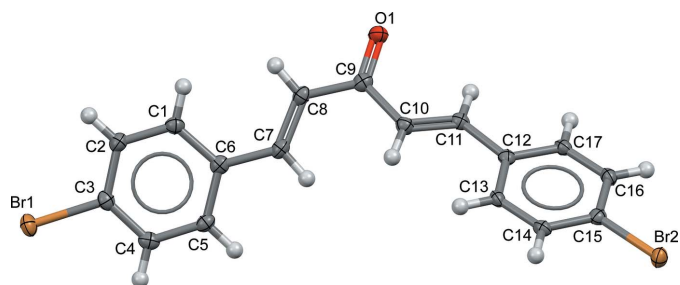
The asymmetric unit of (I) consists of a single molecule, consisting of two 4-bromophenyl rings connected by a penta-1,4-dien-3-one bridge (Fig. 1). The bond lengths and angles of the central chain are consistent with those in related structures (Butcher *et al.*, 2007a; Ruanwas *et al.*, 2011). The overall conformation of (I) can be described by the torsion angles between the olefinic double bonds and 4-bromophenyl rings [τ_1 (C1–C6–C7–C8); τ_4 (C13–C12–C11–C10)] and the carbonyl double bond [τ_2 (C7–C8–C9–O1); τ_3 (C11–C10–C9–O1)] (Fig. 2). The 4-bromophenyl rings in (I) are close to coplanar with their attached olefinic double bonds [$\tau_1 = -10.2$ (4)°; $\tau_4 = -6.2$ (4)°] but the conformations of the olefinic double bonds are very different: one is in *s-trans* [$\tau_2 = 160.7$ (3)°] conformation and in *s-cis* [$\tau_3 = -15.2$ (4)°] conformation with the central C=O double bond. These torsions result in an overall twisted shape for (I) with the dihedral angle between the 4-bromophenyl ring being 51.56 (2)°.

3. Supramolecular features

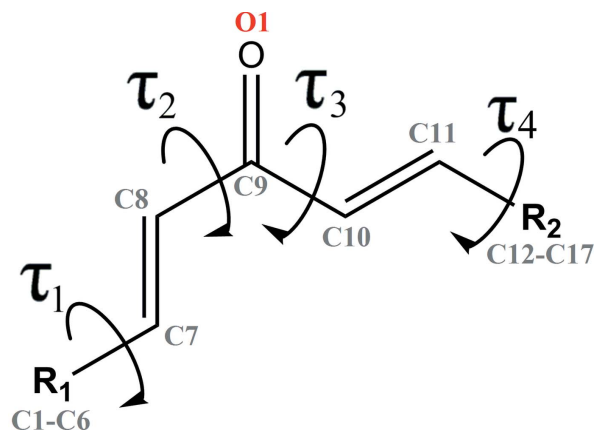
No classical hydrogen bonding is possible in (I) and in the crystal, molecules are linked by C–H \cdots π interactions (Table 1): the first of these results in a phenyl–phenyl T-shaped geometry *via* C1–H1A \cdots Cg1ⁱ (Fig. 3a). The C14–H14A \cdots Cg2ⁱⁱ (Fig. 3b) interactions lead to a zigzag chain along the *c*-axis direction.

4. Database survey

A survey of the Cambridge Structural Database (CSD, version 5.40, last update February 2019; (Groom *et al.*, 2016)) using

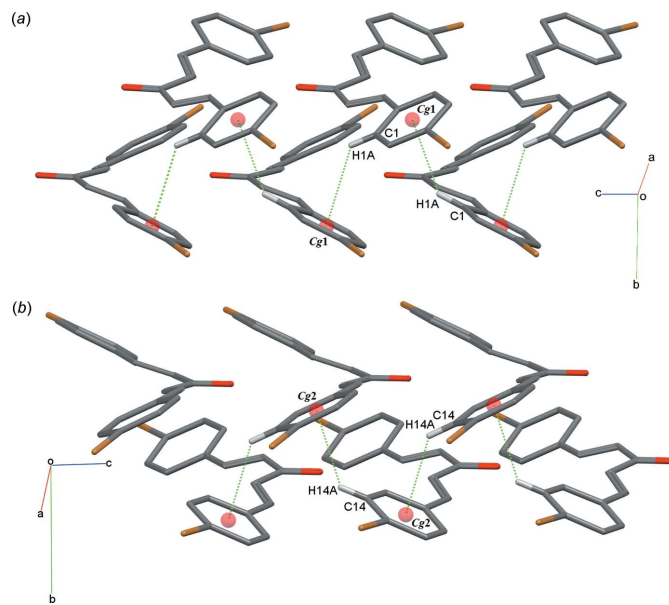

Figure 1

The molecular structure of the title compound showing 50% displacement ellipsoids.


Figure 2

General chemical diagram showing torsion angles, τ_1 , τ_2 , τ_3 and τ_4 in the title compound.

(1*E*,4*E*)-1,5-diphenylpenta-1,4-dien-3-one as the main skeleton revealed the presence of 27 structures containing a similar bischalcone moiety to the title compound but with different substituents on the terminal phenyl rings. The different substituents (R_1 and R_2) together with the torsion angles of the penta-4,4-dien-3-one connecting bridge are compiled in Table 2. For the conformationally symmetrical compounds (i.e. both C=C–C=O bonds are either *s-cis* or *s-trans*), the olefinic double bonds are close to coplanar with their attached phenyl rings as indicated by their τ_1 and τ_4 torsion angles, which fall in the range of 0.0–17.8°, except for the compounds AMEXUN and HUDLEY, which have somewhat larger τ_1 and τ_4 values of 22.5–27.4°. The olefinic double bonds for the symmetrical compounds are mostly in


Figure 3

A partial packing diagram of the title compound, with (a) C1–H1A \cdots π and (b) C14–H14A \cdots π interactions (dotted lines). Hydrogen atoms not involved in these interactions have been omitted for clarity.

Table 2
Torsion angles τ_1 , τ_2 , τ_3 and τ_4 ($^\circ$).

| Compound | R₁ | R₂ | τ_1 | τ_2 | τ_3 | τ_4 |
|---|-------------------------------|-------------------------------|------------------|-------------------|----------------|------------------|
| Symmetrical | | | | | | |
| AMEXUN (Mark <i>et al.</i> , 2016) | 4-(benzyloxy)-3-methoxyphenyl | 4-(benzyloxy)-3-methoxyphenyl | 27.4 | 19.3 | 20.4 | 22.5 |
| COGNOD01 (Rawal <i>et al.</i> , 2016) | 4-(diethylamino)phenyl | 4-(diethylamino)phenyl | 1.2, 4.0 | 11.0, 4.2 | 1.3, 2.9 | 0.7, 1.6 |
| DUMWIS (Fun <i>et al.</i> , 2010) | 2,4,5-trimethoxyphenyl | 2,4,5-trimethoxyphenyl | 11.6, 0.4, 11.8 | 0.7, 4.0, 5.5 | 3.0, 8.2, 6.0 | 9.8, 2.9, 3.7 |
| EDUSEE (Rawal <i>et al.</i> , 2016) | 4-(diethylamino)phenyl | 4-benzonitrile | 15.6, 5.9 | 0.1, 6.4 | 8.1, 7.7 | 3.7, 3.2 |
| GOLGOD (Shan <i>et al.</i> , 1999) | 4-methoxyphenyl | 4-methoxyphenyl | 3.2 | 153.4 | 152.9 | 2.7 |
| GOLGOD02 (Harrison <i>et al.</i> , 2006) | 4-methoxyphenyl | 4-methoxyphenyl | 2.4 | 152.2 | 152.2 | 2.4 |
| HIDMIQ (Zhou <i>et al.</i> , 1999) | 2-methoxyphenyl | 2-methoxyphenyl | 0.2 | 1.4 | 1.2 | 8.8 |
| HUDLEY (Feng <i>et al.</i> , 2009) | 2,4-dimethylphenyl | 2,4-dimethylphenyl | 26.1 | 3.1 | 1.1 | 24.6 |
| KOFCEO (Arshad <i>et al.</i> , 2008) | 4-methylphenyl | 4-methylphenyl | 16.6 | 4.0 | 13 | 180 |
| LEJNOE (Butcher <i>et al.</i> , 2006) | 4-chlorophenyl | 4-chlorophenyl | 17.8 | 9.8 | 9.8 | 17.8 |
| LESGAT (Park <i>et al.</i> , 2013) | 2-(trifluoromethyl)phenyl | 2-(trifluoromethyl)phenyl | 0.5 | 0.3 | 2.9 | 13.7 |
| SAFZOO (Samshuddin <i>et al.</i> , 2012) | 3-nitrophenyl | phenyl | 6.1 | 11.3 | 21.9 | 10.4 |
| SIMTUE (Nizam Mohideen <i>et al.</i> , 2007) | 2-chlorophenyl | 2-chlorophenyl | 8.8 | 3.4 | 0.9 | 0.5 |
| UPAWEO (Huang <i>et al.</i> , 2011) | 2,6-difluorophenyl | 2,6-difluorophenyl | 2.3 | 4.4 | 0.8 | 178 |
| UPAWEO01 (Schwarzer & Weber, 2014a) | 2,6-difluorophenyl | 2,6-difluorophenyl | 2.3 | 4.4 | 0.8 | 0.5 |
| WACXON (Hubig <i>et al.</i> , 1992) | <i>o</i> -tolyl | <i>o</i> -tolyl | 10.3 | 1.1 | 2.8 | 1.5 |
| XOHVOH (Schwarzer & Weber, 2014b) | pentafluorophenyl | pentafluorophenyl | 3.0, 7.9 | 1.0, 5.7 | 1.6, 3.4 | 5.7, 2.3 |
| XOHVUN (Schwarzer & Weber, 2014b) | pentafluorophenyl | phenyl | 5.6 | 2.4 | 3.3 | 7.8 |
| Unsymmetrical | | | | | | |
| (I) | 4-bromophenyl | 4-bromophenyl | 10.2 | 160.7 | 15.2 | 6.2 |
| IFAQAJ (Kapdi <i>et al.</i> , 2013) | 3,5-dimethoxyphenyl | 3,5-dimethoxyphenyl | 5.4 | 173.1 | 0.6 | 3.2 |
| LEJNOE01 (Maluleka & Mphahlele, 2017) | 4-chlorophenyl | 4-chlorophenyl | 11.2 | 160.2 | 13.6 | 6.6 |
| MESXEQ (Dravida <i>et al.</i> , 2018) | 2,6-dichlorophenyl | 2,6-dichlorophenyl | 46.8, 48.7, 51.8 | 175.3, 4.3, 178.7 | 7.5, 4.3, 15.5 | 32.7, 48.7, 51.8 |
| QAJNOG (Ruanwas <i>et al.</i> , 2011) | 2,4,6-trimethoxyphenyl | 2,4,6-trimethoxyphenyl | 6.6, 0.5 | 176.8, 169.4 | 1.2, 0.5 | 3.7, 11.8 |
| WIHBUL (Butcher <i>et al.</i> , 2007a) | 4-fluorophenyl | 4-fluorophenyl | 18.2, 18.7 | 169.0, 166.3 | 10.4, 8.8 | 21.8, 21.4 |
| XIFTOW (Butcher <i>et al.</i> , 2007b) | 3,4-dimethoxyphenyl | 3,4-dimethoxyphenyl | 1.6, 1.6 | 162.8, 170.8 | 23.3, 23.7 | 3.1, 20.3 |
| ZAPKIN (Chantrapomma <i>et al.</i> , 2016) | 4-ethoxyphenyl | 4-ethoxyphenyl | 17.2 | 168.4 | 17.1 | 13.8 |

Multiple sets of torsion angles are stated for compounds COGNOD01, DUMWIS, EDUSEE, XOHVOH, MESXEQ, QAJNOG, WIHBUL and XIFTOW because there is more than one independent molecule in their asymmetric units. A third molecule with full molecule disorder in compound WIHBUL was excluded from this table.

s-cis conformations with the carbonyl double bond (τ_2/τ_3 torsion angles of 0.1–21.9 $^\circ$). However, both the olefinic double bonds of compounds GOLGOD and GOLGOD02 are in *s-trans* conformations with the carbonyl double bond ($\tau_2/\tau_3 = 152.2$ – 153.4°). Furthermore, it may be noted that the symmetrical conformation at the penta-4,4-dien-3-one connection bridge is not affected by the different substituents at the **R₁** and **R₂** positions in EDUSEE, SAFZOO and XOHVUN. Most of the unsymmetrical compounds (one

C=C–C=O bond *s-cis* and one *s-trans*) have τ_1 and τ_4 values of 0.5– 17.2° , which indicates that the olefinic double bonds are close to coplanar to their attached phenyl ring. The outliers are MESXEQ and WIHBUL, which have τ_1 and τ_4 values of 18.2– 51.8° and 21.4– 51.8° , respectively. The torsion angles τ_2 and τ_3 for the unsymmetrical compounds, including (I), are in the ranges 160.2– 178.7° and 0.5– 23.7° , respectively, which indicate *s-trans* and *s-cis* conformations between the olefinic double bonds and the carbonyl double bond.

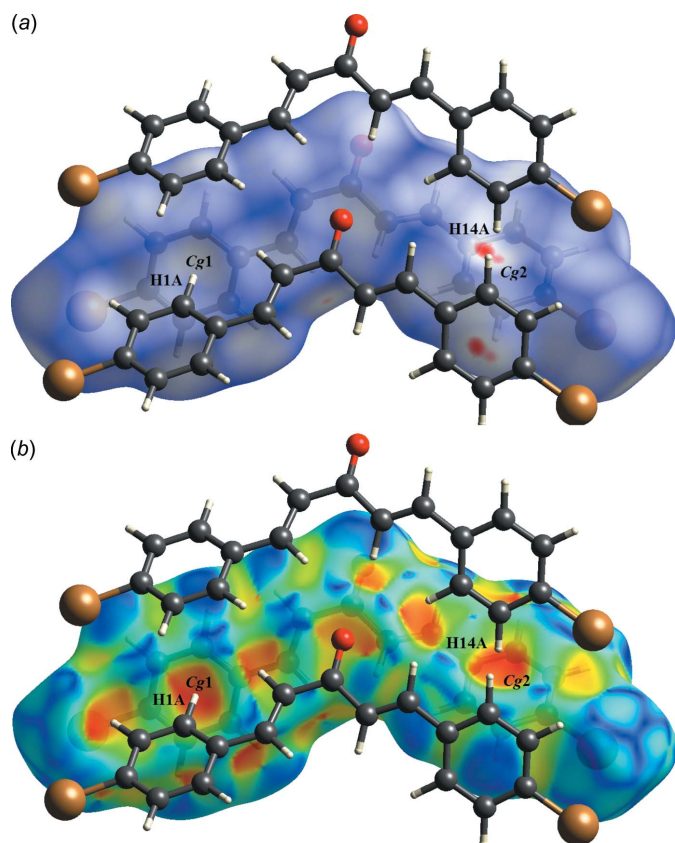


Figure 4
The Hirshfeld surface mapped with (a) d_{norm} and (b) shape-index for the title compound showing the C–H $\cdots\pi$ interactions.

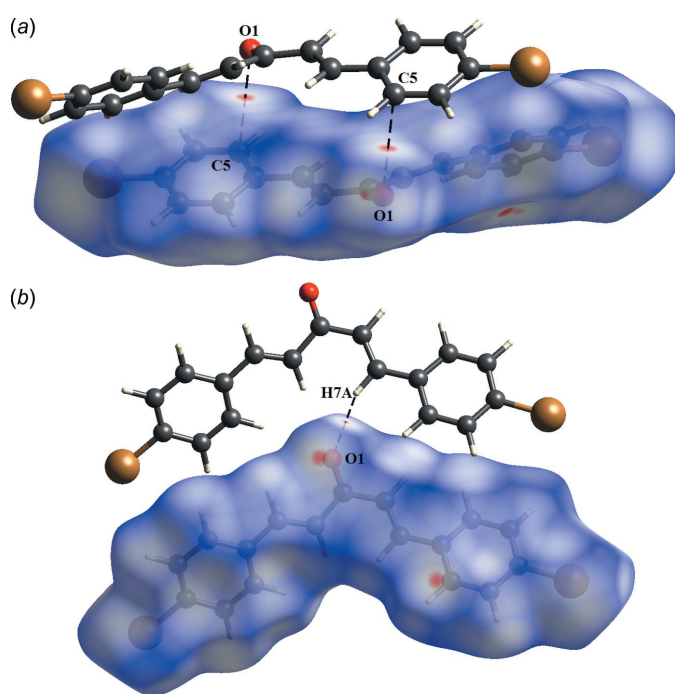


Figure 5
The Hirshfeld surface mapped with d_{norm} showing (a) the C5 \cdots O1 short contact and (b) the weak C7–H7A \cdots O1 interaction.

5. Hirshfeld surface analysis

The Hirshfeld surfaces mapped with normalized contact distance d_{norm} and the two-dimensional fingerprint plots for (I) were generated using *CrystalExplorer17.5* (Turner *et al.*, 2017). The darkest red spots on the Hirshfeld surface mapped with d_{norm} (Fig. 4a) correspond to the C14–H14A \cdots Cg2ⁱⁱ interaction. Even through the C1–H1A \cdots Cg1ⁱ interaction is not visible in the d_{norm} surface mapping, this interaction can be seen as a unique pattern of a red ‘circle’ on the shape-index surface mapping (Fig. 4b). Besides the C–H $\cdots\pi$ interactions, the d_{norm} surface mapping indicated a short contact between atom O1 and C5 with a distance of 0.06 Å shorter than the sum of the van der Waals radii of O and C atoms (3.22 Å; Fig. 5a). Together with this short contact, another weak C7–H7A \cdots O1 interaction was also revealed as light spots on the d_{norm} surface (Fig. 5b).

As illustrated in Fig. 6, the corresponding fingerprint plots for (I) are shown with characteristic pseudo-symmetric wings in the d_e and d_i diagonal axes. The H \cdots C/C \cdots H contacts are

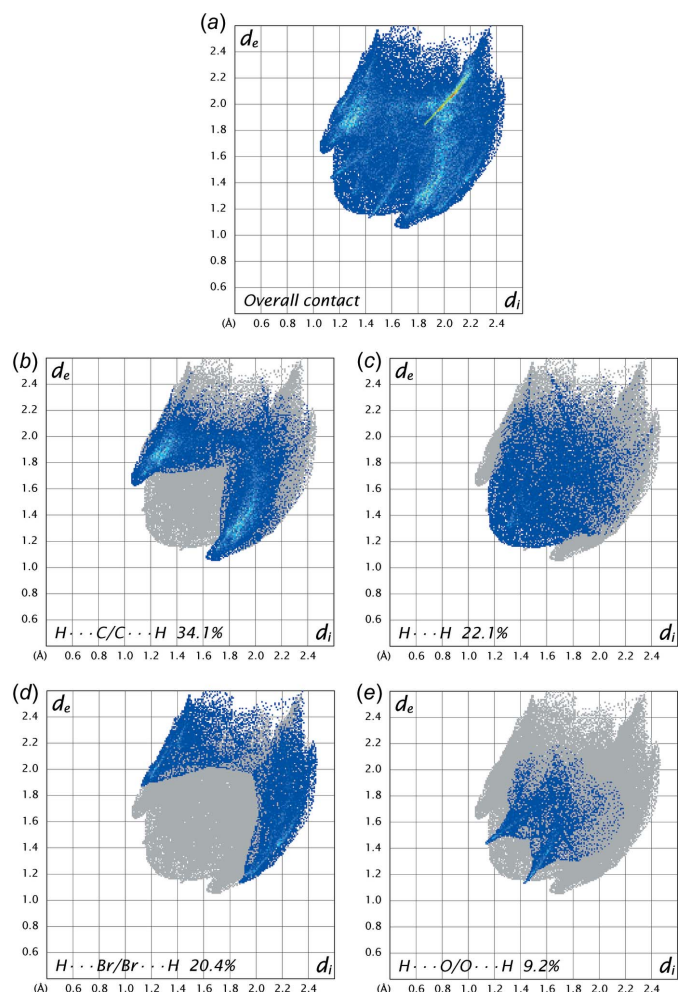


Figure 6
The two-dimensional fingerprint plots of the title compound for different intermolecular contacts and their percentage contributions to the Hirshfeld surface. d_e and d_i are the distances from the Hirshfeld surface to the nearest atom interior and exterior, respectively, to the surface.

Table 3
Experimental details.

| | |
|--|--|
| Crystal data | |
| Chemical formula | C ₁₇ H ₁₂ Br ₂ O |
| <i>M_r</i> | 392.09 |
| Crystal system, space group | Monoclinic, <i>P</i> 2 ₁ / <i>c</i> |
| Temperature (K) | 100 |
| <i>a</i> , <i>b</i> , <i>c</i> (Å) | 17.5920 (2), 14.0777 (3), 5.7956 (1) |
| β (°) | 98.742 (1) |
| <i>V</i> (Å ³) | 1418.63 (4) |
| <i>Z</i> | 4 |
| Radiation type | Cu <i>K</i> α |
| μ (mm ⁻¹) | 7.17 |
| Crystal size (mm) | 0.12 × 0.06 × 0.03 |
| Data collection | |
| Diffractometer | Agilent SuperNova Dual diffractometer with an Atlas detector |
| Absorption correction | Multi-scan (<i>CrysAlis PRO</i> ; Agilent, 2014) |
| <i>T_{min}</i> , <i>T_{max}</i> | 0.64, 0.79 |
| No. of measured, independent and observed [<i>I</i> > 2σ(<i>I</i>)] reflections | 17456, 2524, 2372 |
| <i>R_{int}</i> | 0.031 |
| (sin θ/λ) _{max} (Å ⁻¹) | 0.597 |
| Refinement | |
| <i>R</i> [<i>F</i> ² > 2σ(<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i> | 0.022, 0.056, 1.15 |
| No. of reflections | 2524 |
| No. of parameters | 181 |
| H-atom treatment | H-atom parameters constrained |
| $\Delta\rho_{\max}$, $\Delta\rho_{\min}$ (e Å ⁻³) | 0.42, -0.40 |

Computer programs: *CrysAlis PRO* (Agilent, 2014), *SHELXS97* (Sheldrick, 2008), *SHELXL2013* (Sheldrick, 2015), *Mercury* (Macrae *et al.*, 2006) and *PLATON* (Spek, 2009).

the most populated contacts and contribute 34.1% to the total intermolecular contacts, followed by H···H (22.1%), H···Br/Br···H (20.4%) and H···O/O···H (9.2%) contacts (Fig. 6). As the C—H···π bonds are the main interaction in the crystal, the most populated H···C/C···H contacts appear as two symmetrical narrow wings at diagonal axes *d_e* + *d_i* ≈ 2.7 Å (Fig. 6*b*). The H···H contacts appear in the central region of the fingerprint plots with *d_e* = *d_i* = 2.4 Å (Fig. 6*c*). With the presence of relatively larger bromine atoms in the structure, the H···Br/Br···H contacts appear as symmetrical broad wing at diagonal axes of *d_e* + *d_i* ≈ 3.0 Å (Fig. 6*d*). Two symmetric spikes in the fingerprint plots with a short spike at *d_e* + *d_i* ≈ 2.7 Å represent the H···O/O···H contacts (Fig. 6*e*), indicating the presence of the weak C7—H7A···O1 interaction. The percentage contributions for other contacts are less than 15% in the Hirshfeld surface mapping.

6. Synthesis and crystallization

A mixture of 4-bromobenzaldehyde (4.9 g, 12.5 mmol) and acetone (0.363 g, 6.25 mmol) dissolved in absolute ethanol (30 ml) was slowly added to an aqueous solution of potassium hydroxide (4.0 g in 20 ml water). The mixture was vigorously stirred at room temperature for two h and then 20 ml chilled water was added. The resulting yellow precipitate was recovered by vacuum filtration and washed with cold water

(100 ml). The crude product was recrystallized from absolute ethanol solution as yellow blocks.

(1*E*,4*E*)-1,5-Bis(4-bromophenyl)penta-1,4-dien-3-one; pure yellow solid (4.6 g, 88.6%), m.p. 484 K; **IR** ν_{\max} 594, 687, 813, 979, 1066, 1181, 1320, 1398, 1480, 1581, 1643 cm⁻¹, **UV-Vis** λ_{\max} 227 and 317 nm, **¹H NMR**: δ_{H} (500MHz, CDCl₃) 7.02 (2H, H-1), 7.45 (4H, H-2), 7.54 (4H, H-3), 7.67 (2H, H-4); **¹³C NMR**: δ_{C} (125MHz, CDCl₃) 124.62, 125.69, 129.25, 131.74, 133.29, 141.83, 188.22; **HRMS** (ES): *MH*⁺, found: 392 C₁₇H₁₂Br₂O⁺ requires: 391.92.

7. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. C-bound H atoms were positioned geometrically (C—H = 0.93 Å) and refined using a riding model with *U*_{iso}(H) = 1.5*U*_{eq}(C).

Acknowledgements

We thank the Universiti Putra Malaysia for the use of their facilities and the University of Anbar, Ministry of Higher Education, in Iraq for a scholarship (NAT).

Funding information

This research was funded by the UPM under the Research University Grant Scheme (RUGS No. 05-01-11-1234RU).

References

- Agilent (2014). *CrysAlis PRO*. Agilent Technologies Ltd, Yarnton, England.
- Arshad, M. N., Tahir, M. N., Asghar, M. N., Khan, I. U. & Ashfaq, M. (2008). *Acta Cryst.* **E64**, o1413.
- Butcher, R. J., Jasinski, J. P., Sarojini, B. K., Yathirajan, H. S., Bindya, S. & Narayana, B. (2007*a*). *Acta Cryst.* **E63**, o3213–o3214.
- Butcher, R. J., Jasinski, J. P., Yathirajan, H. S., Bindya, S., Narayana, B. & Sarojini, B. K. (2007*b*). *Acta Cryst.* **E63**, o3115.
- Butcher, R. J., Yathirajan, H. S., Sarojini, B. K., Narayana, B. & Vijaya Raj, K. K. (2006). *Acta Cryst.* **E62**, o1973–o1975.
- Chantrapromma, S., Ruanwas, P., Boonnak, N., Chantrapromma, K. & Fun, H.-K. (2016). *Crystallogr. Rep.* **61**, 1081–1085.
- Conard, C. R. & Morris, A. D. (1932). *Org. Synth.* **12**, 22.
- Dravida Thendral, E., Mohamooda Sumaya, U., Gomathi, S., Biruntha, K. & Usha, G. (2018). *IUCr Data* **3**, x171822.
- Feng, Z., Li, J. & Lin, Y. (2009). *Acta Cryst.* **E65**, o2275.
- Fun, H.-K., Loh, W.-S., Sarojini, B. K., Khaleel, V. M. & Narayana, B. (2011). *Acta Cryst.* **E67**, o2651–o2652.
- Fun, H.-K., Ruanwas, P. & Chantrapromma, S. (2010). *Acta Cryst.* **E66**, o307–o308.
- Groom, C. R., Bruno, I. J., Lightfoot, M. P. & Ward, S. C. (2016). *Acta Cryst.* **B72**, 171–179.
- Harrison, W. T. A., Sarojini, B. K., Vijaya Raj, K. K., Yathirajan, H. S. & Narayana, B. (2006). *Acta Cryst.* **E62**, o1522–o1523.
- Huang, J.-D., Tang, Q.-Q., Chen, X.-Y., Ye, Y. & Wang, Y. (2011). *Acta Cryst.* **E67**, o758.
- Hubig, S. M., Drouin, M., Michel, A. & Harvey, P. D. (1992). *Inorg. Chem.* **31**, 5375–5380.
- Kapdi, A. R., Whitwood, A. C., Williamson, D. C., Lynam, J. M., Burns, M. J., Williams, T. J., Reay, A. J., Holmes, J. & Fairlamb, I. J. S. (2013). *J. Am. Chem. Soc.* **135**, 8388–8399.

- Kwong, H. C., Rakesh, M. S., Chidan Kumar, C. S., Maidur Shivaraj, R., Patil Parutagouda, S., Quah Ching, K., Win, Y.-F., Parlak, C. & Chandrāju, S. (2018). *Z. Kristallogr. Cryst. Mater.* **233**, 349–360.
- Macrae, C. F., Edgington, P. R., McCabe, P., Pidcock, E., Shields, G. P., Taylor, R., Towler, M. & van de Streek, J. (2006). *J. Appl. Cryst.* **39**, 453–457.
- Mahapatra, D. K., Bharti, S. K. & Asati, V. (2017). *Curr. Top. Med. Chem.* **17**, 3146–3169.
- Maluleka, M. & Mphahlele, M. J. (2017). *Z. Kristallogr. New Cryst. Struct.* **232**, 1049.
- Mark, E. L., Huma, A. B. & Jeremy, K. (2016). Private communication (Refcode CCDC 921980). CCDC, Cambridge, England.
- Nizam Mohideen, M., Thenmozhi, S., Subbiah Pandi, A., Murugan, R. & Narayanan, S. S. (2007). *Acta Cryst.* **E63**, o4379.
- Pandey, K. B. & Rizvi, S. I. (2009). *Oxid. Med. Cell. Longev.* **2**, 270–278.
- Park, D. H., Ramkumar, V. & Parthiban, P. (2013). *Acta Cryst.* **E69**, o177.
- Rawal, M., Garrett, K. E., Johnson, L. E., Kaminsky, W., Jucov, E., Shelton, D. P., Timofeeva, T., Eichinger, B. E., Tillack, A. F., Robinson, B. H., Elder, D. L. & Dalton, L. R. (2016). *J. Opt. Soc. Am. B*, **33**, E160–E170.
- Ruanwas, P., Chantrapromma, S. & Fun, H.-K. (2011). *Acta Cryst.* **E67**, o33–o34.
- Samshuddin, S., Butcher, R. J., Akkurt, M., Narayana, B., Sarojini, B. K. & Yathirajan, H. S. (2012). *Acta Cryst.* **E68**, o74–o75.
- Schwarzer, A. & Weber, E. (2014a). *Acta Cryst.* **C70**, 202–206.
- Schwarzer, A. & Weber, E. (2014b). *Cryst. Growth Des.* **14**, 2335–2342.
- Shan, Y., Zhou, H. & Huang, S. D. (1999). *Z. Kristallogr. New Cryst. Struct.* **214**, 381.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Sheldrick, G. M. (2015). *Acta Cryst.* **C71**, 3–8.
- Sim, A., Chidan Kumar, C. S., Kwong, H. C., Then, L. Y., Win, Y.-F., Quah, C. K., Naveen, S., Chandrāju, S., Lokanath, N. K. & Warad, I. (2017). *Acta Cryst.* **E73**, 896–900.
- Spek, A. L. (2009). *Acta Cryst.* **D65**, 148–155.
- Turner, M., McKinnon, J., Wolff, S., Grimwood, D. J., Spackman, P. R., Jayatilaka, D. & Spackman, M. (2017). *Crystal Explorer17*. University of Western Australia.
- Zhou, H., Lai, C. & Montes, I. (1999). *Z. Kristallogr. New Cryst. Struct.* **214**, 53.

supporting information

Acta Cryst. (2019). E75, 774-779 [https://doi.org/10.1107/S2056989019006480]

Crystal structure and Hirshfeld surface analysis of a conformationally unsymmetrical bischalcone: (1*E*,4*E*)-1,5-bis(4-bromophenyl)penta-1,4-dien-3-one

Nabeel Arif Tawfeeq, Huey Chong Kwong, Mohamed Ibrahim Mohamed Tahir and Thahira B. S. A. Ravoof

Computing details

Data collection: *CrysAlis PRO* (Agilent, 2014); cell refinement: *CrysAlis PRO* (Agilent, 2014); data reduction: *CrysAlis PRO* (Agilent, 2014); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2013* (Sheldrick, 2015); molecular graphics: *SHELXL2013* (Sheldrick, 2015) and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *SHELXL2013* (Sheldrick, 2015) and *PLATON* (Spek, 2009).

(1*E*,4*E*)-1,5-Bis(4-bromophenyl)penta-1,4-dien-3-one

Crystal data

C₁₇H₁₂Br₂O

M_r = 392.09

Monoclinic, *P*2₁/*c*

a = 17.5920 (2) Å

b = 14.0777 (3) Å

c = 5.7956 (1) Å

β = 98.742 (1)°

V = 1418.63 (4) Å³

Z = 4

F(000) = 768

D_x = 1.836 Mg m⁻³

Cu *Kα* radiation, λ = 1.54178 Å

Cell parameters from 11112 reflections

θ = 4–76°

μ = 7.17 mm⁻¹

T = 100 K

Block, yellow

0.12 × 0.06 × 0.03 mm

Data collection

Agilent SuperNova Dual
diffractometer with an Atlas detector

Graphite monochromator

ω scans

Absorption correction: multi-scan
(*CrysAlis PRO*; Agilent, 2014)

T_{min} = 0.64, *T_{max}* = 0.79

17456 measured reflections

2524 independent reflections

2372 reflections with *I* > 2σ(*I*)

R_{int} = 0.031

θ_{max} = 67.1°, θ_{min} = 4.0°

h = -21→18

k = -16→16

l = -6→6

Refinement

Refinement on *F*²

Least-squares matrix: full

R[*F*² > 2σ(*F*²)] = 0.022

wR(*F*²) = 0.056

S = 1.15

2524 reflections

181 parameters

0 restraints

Hydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0179P)^2 + 1.9865P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.003$

$$\Delta\rho_{\max} = 0.42 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.40 \text{ e } \text{\AA}^{-3}$$

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 (\AA^2)

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|------|--------------|--------------|--------------|----------------------------------|
| Br1 | 0.14690 (2) | 0.38377 (2) | -0.19437 (5) | 0.02381 (9) |
| Br2 | 1.00765 (2) | 0.35118 (2) | 0.21721 (5) | 0.02140 (9) |
| O1 | 0.58973 (11) | 0.35870 (14) | 0.9112 (3) | 0.0230 (4) |
| C1 | 0.33281 (15) | 0.33927 (19) | 0.3232 (4) | 0.0190 (5) |
| H1A | 0.341393 | 0.310601 | 0.469408 | 0.023* |
| C2 | 0.25910 (14) | 0.34239 (19) | 0.2021 (4) | 0.0193 (5) |
| H2A | 0.218193 | 0.316997 | 0.266165 | 0.023* |
| C3 | 0.24703 (14) | 0.38441 (19) | -0.0186 (4) | 0.0192 (5) |
| C4 | 0.30666 (15) | 0.42466 (18) | -0.1141 (4) | 0.0193 (5) |
| H4A | 0.297492 | 0.453469 | -0.260084 | 0.023* |
| C5 | 0.38031 (14) | 0.42161 (18) | 0.0103 (4) | 0.0187 (5) |
| H5A | 0.420665 | 0.448675 | -0.053317 | 0.022* |
| C6 | 0.39517 (14) | 0.37826 (18) | 0.2313 (4) | 0.0176 (5) |
| C7 | 0.47449 (15) | 0.37678 (18) | 0.3537 (4) | 0.0187 (5) |
| H7A | 0.512770 | 0.395031 | 0.268379 | 0.022* |
| C8 | 0.49705 (15) | 0.35175 (18) | 0.5758 (4) | 0.0188 (5) |
| H8A | 0.460021 | 0.328563 | 0.659851 | 0.023* |
| C9 | 0.57702 (15) | 0.35856 (18) | 0.6962 (4) | 0.0194 (5) |
| C10 | 0.64033 (14) | 0.36427 (18) | 0.5544 (4) | 0.0186 (5) |
| H10A | 0.630488 | 0.349029 | 0.396510 | 0.022* |
| C11 | 0.71104 (14) | 0.39078 (18) | 0.6484 (4) | 0.0169 (5) |
| H11A | 0.717134 | 0.413278 | 0.800928 | 0.020* |
| C12 | 0.78029 (14) | 0.38804 (18) | 0.5353 (4) | 0.0165 (5) |
| C13 | 0.77978 (14) | 0.34717 (18) | 0.3144 (4) | 0.0166 (5) |
| H13A | 0.733445 | 0.327076 | 0.229449 | 0.020* |
| C14 | 0.84699 (14) | 0.33627 (18) | 0.2209 (4) | 0.0174 (5) |
| H14A | 0.846014 | 0.308769 | 0.074547 | 0.021* |
| C15 | 0.91600 (14) | 0.36684 (18) | 0.3479 (4) | 0.0180 (5) |
| C16 | 0.91859 (14) | 0.40921 (18) | 0.5654 (4) | 0.0180 (5) |
| H16A | 0.964994 | 0.430278 | 0.647991 | 0.022* |
| C17 | 0.85050 (14) | 0.41952 (19) | 0.6572 (4) | 0.0171 (5) |
| H17A | 0.851669 | 0.447926 | 0.802583 | 0.021* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|--------------|--------------|--------------|---------------|---------------|---------------|
| Br1 | 0.01735 (14) | 0.02515 (16) | 0.02724 (15) | 0.00374 (11) | -0.00210 (10) | 0.00155 (11) |
| Br2 | 0.01568 (14) | 0.02560 (16) | 0.02334 (15) | -0.00101 (10) | 0.00433 (10) | -0.00280 (11) |
| O1 | 0.0235 (9) | 0.0274 (11) | 0.0181 (9) | -0.0039 (8) | 0.0034 (7) | 0.0004 (7) |
| C1 | 0.0212 (13) | 0.0189 (13) | 0.0173 (12) | 0.0008 (10) | 0.0041 (10) | -0.0004 (10) |
| C2 | 0.0175 (12) | 0.0185 (13) | 0.0225 (13) | -0.0010 (10) | 0.0054 (10) | 0.0002 (10) |
| C3 | 0.0182 (12) | 0.0179 (13) | 0.0209 (12) | 0.0038 (10) | 0.0009 (10) | -0.0009 (10) |
| C4 | 0.0252 (13) | 0.0153 (13) | 0.0174 (12) | 0.0039 (11) | 0.0040 (10) | -0.0002 (10) |
| C5 | 0.0203 (12) | 0.0163 (13) | 0.0208 (12) | -0.0001 (10) | 0.0074 (10) | -0.0009 (10) |
| C6 | 0.0200 (13) | 0.0141 (12) | 0.0192 (12) | 0.0006 (10) | 0.0048 (10) | -0.0035 (10) |
| C7 | 0.0183 (12) | 0.0165 (13) | 0.0225 (12) | -0.0013 (10) | 0.0067 (10) | -0.0013 (10) |
| C8 | 0.0183 (12) | 0.0169 (13) | 0.0220 (13) | -0.0023 (10) | 0.0054 (10) | -0.0026 (10) |
| C9 | 0.0218 (13) | 0.0157 (13) | 0.0213 (13) | 0.0002 (10) | 0.0050 (10) | -0.0005 (10) |
| C10 | 0.0194 (13) | 0.0193 (14) | 0.0168 (12) | 0.0009 (10) | 0.0022 (10) | -0.0008 (10) |
| C11 | 0.0190 (12) | 0.0144 (13) | 0.0171 (11) | 0.0020 (10) | 0.0022 (9) | 0.0001 (9) |
| C12 | 0.0170 (12) | 0.0147 (12) | 0.0175 (12) | 0.0007 (10) | 0.0014 (9) | 0.0031 (10) |
| C13 | 0.0151 (12) | 0.0175 (13) | 0.0157 (11) | -0.0007 (10) | -0.0022 (9) | 0.0009 (9) |
| C14 | 0.0201 (12) | 0.0166 (13) | 0.0147 (11) | 0.0000 (10) | 0.0005 (10) | -0.0002 (10) |
| C15 | 0.0177 (12) | 0.0179 (13) | 0.0187 (12) | 0.0021 (10) | 0.0034 (10) | 0.0034 (10) |
| C16 | 0.0166 (12) | 0.0188 (13) | 0.0172 (12) | -0.0013 (10) | -0.0019 (9) | 0.0005 (10) |
| C17 | 0.0195 (12) | 0.0173 (13) | 0.0137 (11) | -0.0015 (10) | -0.0007 (9) | 0.0003 (10) |

Geometric parameters (\AA , $^\circ$)

| | | | |
|-----------|-----------|--------------|-----------|
| Br1—C3 | 1.896 (3) | C8—H8A | 0.9300 |
| Br2—C15 | 1.895 (2) | C9—C10 | 1.484 (4) |
| O1—C9 | 1.232 (3) | C10—C11 | 1.333 (4) |
| C1—C2 | 1.378 (4) | C10—H10A | 0.9300 |
| C1—C6 | 1.402 (4) | C11—C12 | 1.469 (3) |
| C1—H1A | 0.9300 | C11—H11A | 0.9300 |
| C2—C3 | 1.396 (4) | C12—C17 | 1.399 (3) |
| C2—H2A | 0.9300 | C12—C13 | 1.402 (3) |
| C3—C4 | 1.380 (4) | C13—C14 | 1.382 (4) |
| C4—C5 | 1.384 (4) | C13—H13A | 0.9300 |
| C4—H4A | 0.9300 | C14—C15 | 1.389 (4) |
| C5—C6 | 1.407 (4) | C14—H14A | 0.9300 |
| C5—H5A | 0.9300 | C15—C16 | 1.389 (4) |
| C6—C7 | 1.466 (4) | C16—C17 | 1.390 (4) |
| C7—C8 | 1.336 (4) | C16—H16A | 0.9300 |
| C7—H7A | 0.9300 | C17—H17A | 0.9300 |
| C8—C9 | 1.475 (4) | | |
| C2—C1—C6 | 121.6 (2) | C8—C9—C10 | 118.9 (2) |
| C2—C1—H1A | 119.2 | C11—C10—C9 | 121.4 (2) |
| C6—C1—H1A | 119.2 | C11—C10—H10A | 119.3 |
| C1—C2—C3 | 118.7 (2) | C9—C10—H10A | 119.3 |

| | | | |
|--------------|--------------|-----------------|--------------|
| C1—C2—H2A | 120.6 | C10—C11—C12 | 126.6 (2) |
| C3—C2—H2A | 120.6 | C10—C11—H11A | 116.7 |
| C4—C3—C2 | 121.5 (2) | C12—C11—H11A | 116.7 |
| C4—C3—Br1 | 119.17 (19) | C17—C12—C13 | 118.3 (2) |
| C2—C3—Br1 | 119.33 (19) | C17—C12—C11 | 119.7 (2) |
| C3—C4—C5 | 119.1 (2) | C13—C12—C11 | 121.8 (2) |
| C3—C4—H4A | 120.4 | C14—C13—C12 | 121.1 (2) |
| C5—C4—H4A | 120.4 | C14—C13—H13A | 119.5 |
| C4—C5—C6 | 121.2 (2) | C12—C13—H13A | 119.5 |
| C4—C5—H5A | 119.4 | C13—C14—C15 | 119.3 (2) |
| C6—C5—H5A | 119.4 | C13—C14—H14A | 120.4 |
| C1—C6—C5 | 117.9 (2) | C15—C14—H14A | 120.4 |
| C1—C6—C7 | 123.6 (2) | C16—C15—C14 | 121.3 (2) |
| C5—C6—C7 | 118.5 (2) | C16—C15—Br2 | 119.99 (19) |
| C8—C7—C6 | 126.3 (2) | C14—C15—Br2 | 118.71 (19) |
| C8—C7—H7A | 116.9 | C15—C16—C17 | 118.7 (2) |
| C6—C7—H7A | 116.9 | C15—C16—H16A | 120.6 |
| C7—C8—C9 | 124.1 (2) | C17—C16—H16A | 120.6 |
| C7—C8—H8A | 117.9 | C16—C17—C12 | 121.3 (2) |
| C9—C8—H8A | 117.9 | C16—C17—H17A | 119.3 |
| O1—C9—C8 | 119.5 (2) | C12—C17—H17A | 119.3 |
| O1—C9—C10 | 121.6 (2) | | |
| | | | |
| C6—C1—C2—C3 | 0.9 (4) | O1—C9—C10—C11 | -15.2 (4) |
| C1—C2—C3—C4 | -1.7 (4) | C8—C9—C10—C11 | 165.4 (2) |
| C1—C2—C3—Br1 | 176.9 (2) | C9—C10—C11—C12 | 171.9 (2) |
| C2—C3—C4—C5 | 1.2 (4) | C10—C11—C12—C17 | 179.3 (3) |
| Br1—C3—C4—C5 | -177.34 (19) | C10—C11—C12—C13 | -6.2 (4) |
| C3—C4—C5—C6 | 0.0 (4) | C17—C12—C13—C14 | 1.4 (4) |
| C2—C1—C6—C5 | 0.3 (4) | C11—C12—C13—C14 | -173.2 (2) |
| C2—C1—C6—C7 | 179.9 (2) | C12—C13—C14—C15 | -0.3 (4) |
| C4—C5—C6—C1 | -0.8 (4) | C13—C14—C15—C16 | -0.8 (4) |
| C4—C5—C6—C7 | 179.6 (2) | C13—C14—C15—Br2 | 179.91 (19) |
| C1—C6—C7—C8 | -10.2 (4) | C14—C15—C16—C17 | 0.9 (4) |
| C5—C6—C7—C8 | 169.4 (3) | Br2—C15—C16—C17 | -179.82 (19) |
| C6—C7—C8—C9 | -175.0 (2) | C15—C16—C17—C12 | 0.1 (4) |
| C7—C8—C9—O1 | 160.7 (3) | C13—C12—C17—C16 | -1.3 (4) |
| C7—C8—C9—C10 | -19.8 (4) | C11—C12—C17—C16 | 173.4 (2) |

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

Cg1 and Cg2 are the centroids of the C1—C16 and C12—C17 rings, respectively.

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|-------------------------------------|-------|-------------|-------------|---------------|
| C1—H1A \cdots Cg1 ⁱ | 0.93 | 2.86 | 3.539 (3) | 131 |
| C14—H14A \cdots Cg2 ⁱⁱ | 0.93 | 2.74 | 3.428 (3) | 131 |

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