

# Crystal structure and Hirshfeld surface analysis of (2E)-3-(3-bromo-4-fluorophenyl)-1-(3,4-dimethoxyphenyl)prop-2-en-1-one

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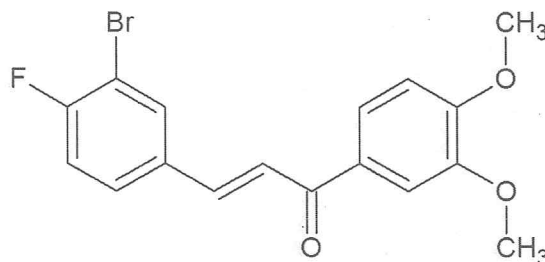
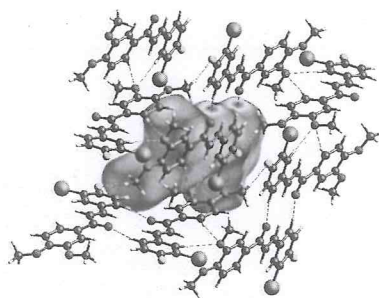
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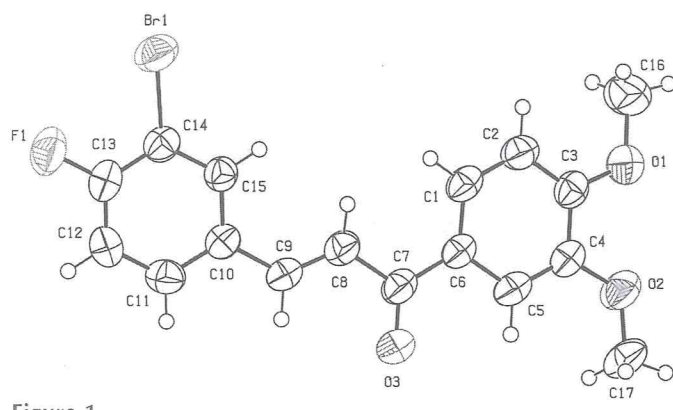
In the molecule of the title compound, C<sub>17</sub>H<sub>14</sub>BrFO<sub>3</sub>, the aromatic rings are tilted with respect to the enone bridge by 13.63 (14) and 4.27 (15)°, and form a dihedral angle 17.91 (17)°. In the crystal, centrosymmetrically related molecules are linked by pairs of C—H···O hydrogen bonds into dimeric units, forming rings of R<sub>2</sub><sup>2</sup>(14) graph-set motif. The dimers are further connected by weak C—H···O hydrogen interactions, forming layers parallel to (10 $\bar{1}$ ). Hirshfeld surface analysis shows that van der Waals interactions constitute the major contribution to the intermolecular interactions, with H···H contacts accounting for 29.7% of the surface.

## 1. Chemical context

Natural products are important sources to search for new agents for cancer therapies with minimal side effects. Chalcones, which are considered to be the precursors of flavonoids and isoflavonoids, are abundant in edible plants. They consist of open-chain flavonoids in which the two aromatic rings are joined by a three-carbon  $\alpha,\beta$ -unsaturated carbonyl system. These are coloured compounds because of the presence of the —CO—CH=CH— chromophore, the colour depending on the presence of other auxochromes. Accumulating evidence has shown that chalcones and their derivatives could inhibit tumor initiation and progression. In view of the above and in a continuation of our previous work on 3,4-dimethoxy chalcones (Sheshadri *et al.*, 2018), herewith we report the crystal and molecular structures of the title compound.



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**Figure 1**  
The molecular structure of the title compound with displacement ellipsoids drawn at the 50% probability level.

## 2. Structural commentary

The title compound (Fig. 1) is constructed by two aromatic rings (3-bromo-4-fluorophenyl and a 3,4-dimethoxyphenyl), which are linked by a  $C=C-C(=O)-C$  enone bridge. The molecule is twisted substantially about the enone bridge, as indicated by the dihedral angles of  $13.63$  ( $14$ ) and  $4.27$  ( $15$ ) $^\circ$  formed by the mean plane through  $C7-C8/O3$  [maximum deviation  $0.045$  ( $4$ )  $\text{\AA}$  for atom  $C7$ ] and the  $C1-C6$  and  $C10-C15$  aromatic rings. The dihedral angle between the mean planes of the 3,4-methoxyphenyl and 3-bromo-4-fluorophenyl rings is  $17.91$  ( $17$ ) $^\circ$ . The H atoms of the central propenone group are *trans* configured. The two methoxy groups attached to  $C16$  and  $C17$  are almost coplanar with the benzene ring, with the deviations of  $0.333$  ( $6$ )  $\text{\AA}$  for  $C16$  and  $-0.124$  ( $4$ )  $\text{\AA}$  for  $C17$ . The bond lengths and angles are comparable with those found in the related compounds (*2E*)-3-(3-chlorophenyl)-1-(3,4-dimethoxyphenyl)prop-2-en-1-one (Sheshadri *et al.*, 2018), (*E*)-3-(3,4-dimethoxyphenyl)-1-(1-hydroxynaphthalen-2-yl)prop-2-en-1-one (Ezhilarasi *et al.*, 2015), (*E*)-1-(3-bromophenyl)-3-(3,4-dimethoxyphenyl)prop-2-en-1-one (Escobar *et al.*, 2012) and (*E*)-3-(2-bromophenyl)-1-(3,4-dimethoxyphenyl)prop-2-en-1-one (Li *et al.*, 2012).

## 3. Supramolecular features

In the crystal, centrosymmetrically related molecules are linked by pairs of  $C-H\cdots O$  hydrogen bonds into dimers forming rings with an  $R_2^2$  ( $14$ ) graph-set motif (Table 1, Fig. 2). The dimeric units are further connected by weak  $C-H\cdots O$  hydrogen bonds, forming layers parallel to  $(10\bar{1})$ .

In addition, weak  $C-Br\cdots\pi$  [ $C14-Br1 = 1.877$  ( $3$ )  $\text{\AA}$ ,  $Br1\cdots Cg1^i = 3.7959$  ( $16$ )  $\text{\AA}$ ,  $C14\cdots Cg1^i = 4.010$  ( $4$ )  $\text{\AA}$ ,  $C14-Br1\cdots Cg1^i = 82.54$  ( $11$ ) $^\circ$ ; symmetry code: (i)  $-1 + x, y, z$ ;  $Cg1$  is the centroid of the  $C1-C6$  ring] and  $C-F\cdots\pi$  [ $C13-F1 = 1.348$  ( $4$ )  $\text{\AA}$ ,  $F1\cdots Cg2^{ii} = 3.454$  ( $3$ )  $\text{\AA}$ ,  $C13\cdots Cg2^{ii} = 3.659$  ( $4$ )  $\text{\AA}$ ,  $C13-F1\cdots Cg2^{ii} = 87.78$  ( $19$ ) $^\circ$ ; symmetry code: (ii)  $-x, 1 - y, -z$ ;  $Cg2$  is the centroid of the  $C10-C15$  ring] interactions help to stabilize the crystal structure.

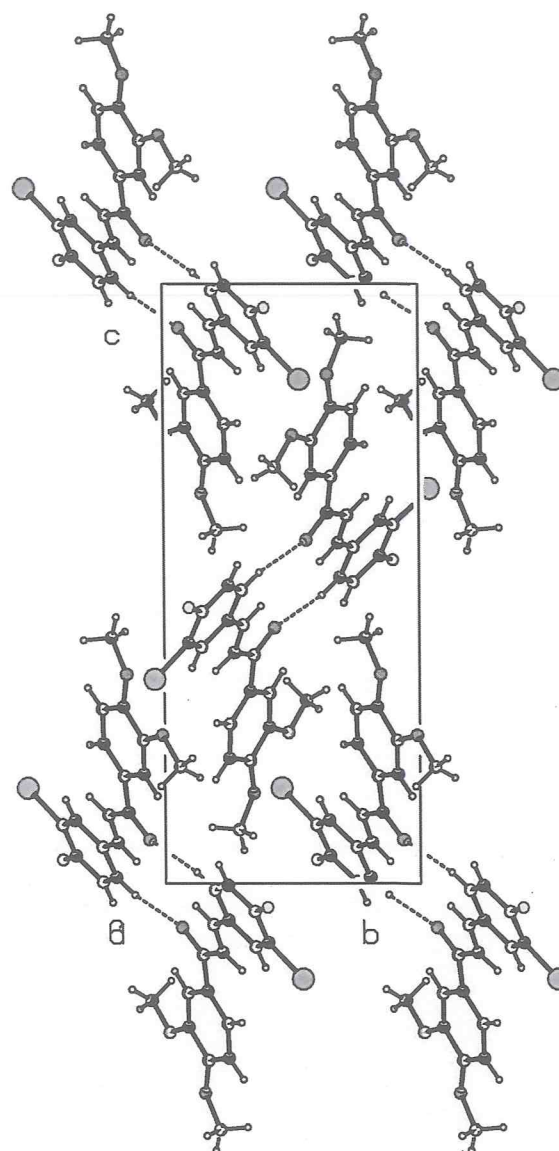
**Table 1**  
Hydrogen-bond geometry ( $\text{\AA}$ ,  $^\circ$ ).

| $D-H\cdots A$            | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|--------------------------|-------|-------------|-------------|---------------|
| $C15-H15A\cdots O2^i$    | 0.93  | 2.61        | 3.506 (5)   | 162           |
| $C11-H11A\cdots O3^{ii}$ | 0.93  | 2.46        | 3.358 (5)   | 162           |

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

## 4. Hirshfeld Surface Analysis

Molecular Hirshfeld surfaces (Hirshfeld, 1977; Spackman & Jayatilaka, 2009) are constructed based on the electron distribution calculated as the sum of spherical atom electron densities (Spackman & Byrom, 1997). Hirshfeld surface analysis is a tool for visualizing the intermolecular interactions; it can include comparisons to the van der Waals



**Figure 2**  
A view along the  $a$  axis of the crystal packing of the title compound. H atoms not involved in hydrogen bonding (dashed lines) are omitted for clarity.

Table 2

Percentage contributions of interatomic contacts to the Hirshfeld surface for the title compound.

| Contact       | Percentage contribution |
|---------------|-------------------------|
| H...H         | 29.7                    |
| C...H/H...C   | 19.2                    |
| O...H/H...O   | 17.9                    |
| Br...H/H...Br | 11.2                    |
| F...H/H...F   | 6.8                     |
| Br...C/C...Br | 5.6                     |
| F...C/C...F   | 5.0                     |
| C...C         | 3.1                     |
| F...O/O...F   | 0.7                     |
| C...O/O...C   | 0.4                     |

envelope, which other molecules or atoms come into contact with when interactions are present. The Hirshfeld surface and two-dimensional fingerprint plots of the title compound were calculated using *CrystalExplorer17.5* (Turner *et al.*, 2017). In the Hirshfeld surface plotted over  $d_{\text{norm}}$  (Fig. 3), the white surfaces indicate contacts with distances equal to the sum of van der Waals radii, and the red and blue colours indicate distances shorter or longer than the van der Waals radii, respectively (Venkatesan *et al.*, 2016). The bright-red spots appearing near to O2, F1, Br1 and hydrogen atoms H15A, H16A, H17C indicate their role as donors and acceptors in the dominant C—H...O, C—H...F and C—H...Br contacts. The shape-index of the Hirshfeld surface is a tool to visualize the  $\pi$ - $\pi$  stacking interactions by the presence of adjacent red and blue triangles; if there are no adjacent red and/or blue triangles, then there are no  $\pi$ - $\pi$  interactions. The Hirshfeld surface of the title compound plotted over shape-index (Fig. 4) clearly suggest that this is the case here. The overall two-dimensional fingerprint plot and those delineated into H...H, C...H/H...C, O...H/H...O, Br...H/H...Br and F...H/H...F contacts (McKinnon *et al.*, 2007) are illustrated in Fig. 5a–f, respectively. Their relative contributions to the Hirshfeld surface are given in Table 2. The most important interaction is

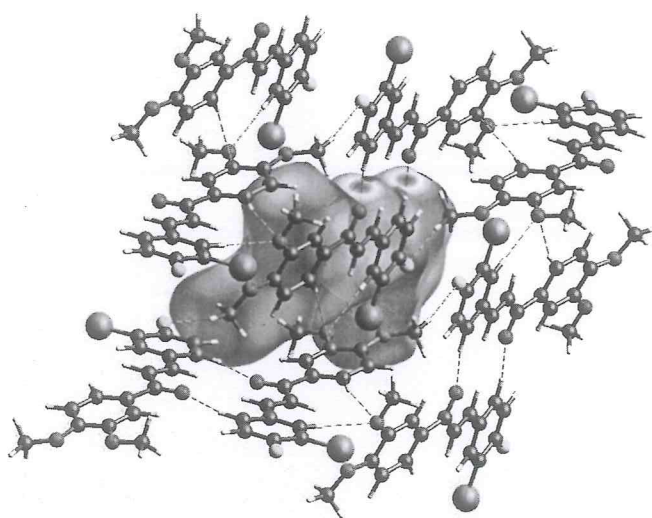


Figure 3

The Hirshfeld surface mapped over  $d_{\text{norm}}$  showing the C—H...O and C—H...F contacts.

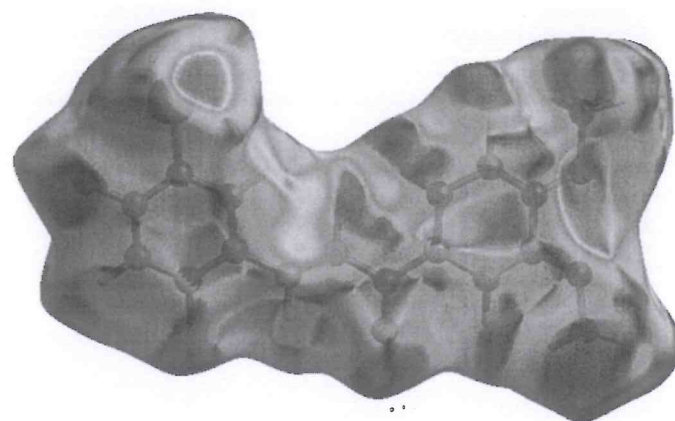


Figure 4

Hirshfeld surface of the title compound plotted over shape-index.

H...H, contributing 29.7% to the overall crystal packing, which is reflected as widely scattered points of high density due to the large hydrogen content of the molecule. In the absence of C—H... $\pi$  interactions in the crystal, shown as a pair of characteristic wings the fingerprint plot, H...C/C...H contacts contribute 19.2% to the Hirshfeld surface (Fig. 5c). The O...H/H...O, Br...C/C...Br and F...C/C...F contacts in the structure with 17.9, 5.6 and 5.0% contributions, respectively, to the Hirshfeld surface have a symmetrical distribution of points (Fig. 5d–f). The other Br...C / C...Br, F...C / C...F, C...C, F...O / O...F and C...O / O...C contacts, having only small contributions to the Hirshfeld surface, have negligible directional impact on the molecular packing.

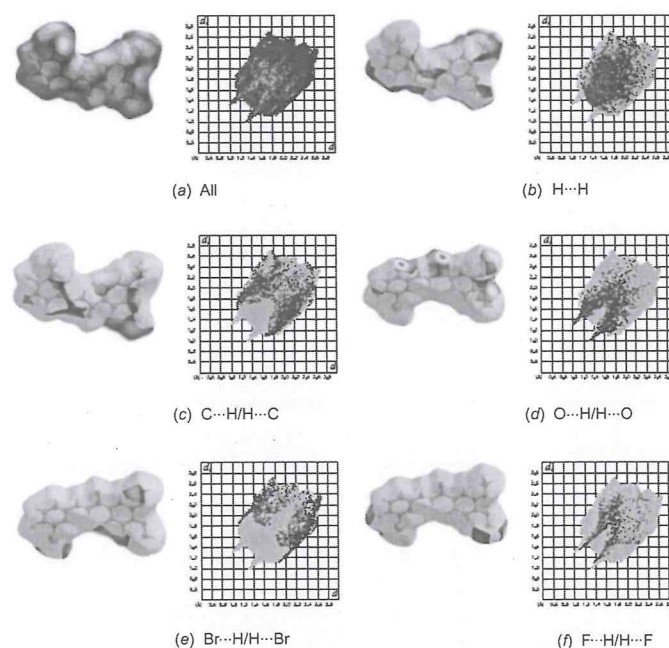


Figure 5

The two-dimensional fingerprint plots of the title compound, showing (a) all interactions, and delineated into (b) H...H, (c) C...H/H...C, (d) O...H/H...O, (e) Br...H/H...Br and (f) F...H/H...F interactions [ $d_e$  and  $d_i$  represent the distances from a point on the Hirshfeld surface to the nearest atoms outside (external) and inside (internal) the surface, respectively].

## 5. Synthesis and crystallization

The reagents and solvents for the synthesis were obtained from the Aldrich Chemical Co., and were used without additional purification. The title compound was synthesized as per the procedure reported earlier (Kumar *et al.*, 2013*a,b*; Chidan Kumar *et al.*, 2014). 1-(3,4-Dimethoxyphenyl) ethanone (0.01mol) and 3-bromo-4-fluorobenzaldehyde (0.01mol) were dissolved in 20 ml methanol. A catalytic amount of NaOH was added to the solution dropwise with vigorous stirring. The reaction mixture was stirred for about 6 h at room temperature. The progress of the reaction was monitored by TLC. The formed crude product was filtered, washed repeatedly with distilled water and recrystallized from ethanol to obtain the title chalcone. Crystals suitable for X-ray diffraction studies were obtained from an acetone solution by the slow evaporation technique at room temperature. The melting point (381–383 K) was determined by a Stuart Scientific (UK) apparatus. The purity of the compound was confirmed by thin layer chromatography using Merck silica gel 60 F254 coated aluminum plates.

## 6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. C-bound H atoms were positioned geometrically and refined using a riding model, with C–H = 0.93–0.6 Å, and with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$  or  $1.5U_{\text{eq}}(\text{C})$  for methyl H atoms.

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Table 3

Experimental details.

|   |   |
|---|---|
| Crystal data  |   |
| Chemical formula  | C <sub>17</sub> H <sub>14</sub> BrFO <sub>3</sub> |
| <i>M<sub>r</sub></i>  | 365.19  |
| Crystal system, space group   | Monoclinic, <i>P</i> <sub>2</sub> / <i>n</i>      |
| Temperature (K)   | 294   |
| <i>a</i> , <i>b</i> , <i>c</i> (Å)  | 8.9212 (12), 8.6601 (11), 20.538 (3)              |
| $\beta$ (°)   | 96.896 (3)  |
| <i>V</i> (Å <sup>3</sup> )  | 1575.2 (4)  |
| <i>Z</i>  | 4   |
| Radiation type  | Mo <i>K</i> $\alpha$                              |
| $\mu$ (mm <sup>-1</sup> )   | 2.63  |
| Crystal size (mm)   | 0.31 × 0.30 × 0.11                                |
| Data collection   |   |
| Diffractometer  | Bruker APEXII CCD                                 |
| Absorption correction   | Multi-scan ( <i>SADABS</i> ; Sheldrick, 2007)     |
| <i>T<sub>min</sub></i> , <i>T<sub>max</sub></i>   | 0.465, 0.755                                      |
| No. of measured, independent and observed [ <i>I</i> > 2 $\sigma$ ( <i>I</i> )] reflections                             | 11919, 3240, 2287                                 |
| <i>R<sub>int</sub></i>  | 0.031   |
| ( <i>sin</i> $\theta$ / $\lambda$ ) <sub>max</sub> (Å <sup>-1</sup> )   | 0.627   |
| Refinement  |   |
| <i>R</i> [ <i>F</i> <sup>2</sup> > 2 $\sigma$ ( <i>F</i> <sup>2</sup> )], <i>wR</i> ( <i>F</i> <sup>2</sup> ), <i>S</i> | 0.046, 0.149, 1.05                                |
| No. of reflections  | 3240  |
| No. of parameters   | 199   |
| H-atom treatment  | H-atom parameters constrained                     |
| $\Delta\rho_{\text{max}}$ , $\Delta\rho_{\text{min}}$ (e Å <sup>-3</sup> )  | 0.78, -0.66                                       |

Computer programs: *APEX2* and *SAINT* (Bruker, 2007), *SHELXS97* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *ORTEP-3 for Windows* (Farrugia, 2012) and *PLATON* (Spek, 2009).

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## supporting information

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## Crystal structure and Hirshfeld surface analysis of (2*E*)-3-(3-bromo-4-fluorophenyl)-1-(3,4-dimethoxyphenyl)prop-2-en-1-one

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### Computing details

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINTE* (Bruker, 2007); data reduction: *SAINTE* (Bruker, 2007); 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: *PLATON* (Spek, 2009).

### (2*E*)-3-(3-Bromo-4-fluorophenyl)-1-(3,4-dimethoxyphenyl)prop-2-en-1-one

#### Crystal data

|                                |   |
|--------------------------------|---|
| $C_{17}H_{14}BrFO_3$           | $F(000) = 736$  |
| $M_r = 365.19$                 | $D_x = 1.540 \text{ Mg m}^{-3}$                         |
| Monoclinic, $P2_1/n$           | Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$ |
| $a = 8.9212 (12) \text{ \AA}$  | Cell parameters from 3205 reflections                   |
| $b = 8.6601 (11) \text{ \AA}$  | $\theta = 2.4\text{--}22.9^\circ$                       |
| $c = 20.538 (3) \text{ \AA}$   | $\mu = 2.63 \text{ mm}^{-1}$                            |
| $\beta = 96.896 (3)^\circ$     | $T = 294 \text{ K}$                                     |
| $V = 1575.2 (4) \text{ \AA}^3$ | Block, colourless                                       |
| $Z = 4$                        | $0.31 \times 0.30 \times 0.11 \text{ mm}$               |

#### Data collection

|   |  |
|---|--|
| Bruker APEXII CCD diffractometer                            | 3240 independent reflections   |
| $\varphi$ and $\omega$ scans                                | 2287 reflections with $I > 2\sigma(I)$                                 |
| Absorption correction: multi-scan (SADABS; Sheldrick, 2007) | $R_{\text{int}} = 0.031$   |
| $T_{\text{min}} = 0.465$ , $T_{\text{max}} = 0.755$         | $\theta_{\text{max}} = 26.5^\circ$ , $\theta_{\text{min}} = 2.4^\circ$ |
| 11919 measured reflections                                  | $h = -11 \rightarrow 10$   |
|   | $k = -10 \rightarrow 10$   |
|   | $l = -25 \rightarrow 25$   |

#### Refinement

|                                 |  |
|---------------------------------|--|
| Refinement on $F^2$             | Hydrogen site location: inferred from neighbouring sites |
| Least-squares matrix: full      | H-atom parameters constrained                            |
| $R[F^2 > 2\sigma(F^2)] = 0.046$ | $w = 1/[\sigma^2(F_o^2) + (0.0824P)^2 + 0.5509P]$        |
| $wR(F^2) = 0.149$               | where $P = (F_o^2 + 2F_c^2)/3$                           |
| $S = 1.05$                      | $(\Delta/\sigma)_{\text{max}} < 0.001$                   |
| 3240 reflections                | $\Delta\rho_{\text{max}} = 0.78 \text{ e \AA}^{-3}$      |
| 199 parameters                  | $\Delta\rho_{\text{min}} = -0.65 \text{ e \AA}^{-3}$     |
| 0 restraints                    |  |

*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 ( $\text{\AA}^2$ )*

|      | <i>x</i>     | <i>y</i>    | <i>z</i>     | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|------|--------------|-------------|--------------|----------------------------------|
| Br1  | -0.03000 (5) | 0.46149 (5) | 0.15921 (2)  | 0.0788 (2)                       |
| F1   | -0.2029 (2)  | 0.5975 (3)  | 0.03979 (14) | 0.0862 (7)                       |
| O1   | 1.0604 (3)   | 0.8509 (4)  | 0.34912 (13) | 0.0743 (8)                       |
| O2   | 1.1326 (3)   | 0.9899 (4)  | 0.24774 (15) | 0.0803 (8)                       |
| O3   | 0.6831 (3)   | 0.9371 (4)  | 0.07316 (15) | 0.0788 (8)                       |
| C1   | 0.7272 (4)   | 0.7591 (4)  | 0.23364 (19) | 0.0575 (9)                       |
| H1A  | 0.636474     | 0.705562    | 0.231962     | 0.069*                           |
| C2   | 0.8261 (4)   | 0.7559 (4)  | 0.29089 (18) | 0.0589 (9)                       |
| H2A  | 0.802350     | 0.699096    | 0.326693     | 0.071*                           |
| C3   | 0.9606 (4)   | 0.8373 (4)  | 0.29488 (18) | 0.0550 (8)                       |
| C4   | 0.9977 (4)   | 0.9146 (4)  | 0.23886 (18) | 0.0532 (8)                       |
| C5   | 0.8997 (4)   | 0.9162 (4)  | 0.18250 (18) | 0.0541 (8)                       |
| H5A  | 0.925561     | 0.968527    | 0.145921     | 0.065*                           |
| C6   | 0.7591 (3)   | 0.8392 (4)  | 0.17906 (17) | 0.0498 (7)                       |
| C7   | 0.6521 (4)   | 0.8551 (4)  | 0.11860 (18) | 0.0570 (8)                       |
| C8   | 0.5024 (4)   | 0.7777 (4)  | 0.11436 (19) | 0.0606 (9)                       |
| H8A  | 0.486595     | 0.702343    | 0.145064     | 0.073*                           |
| C9   | 0.3917 (4)   | 0.8126 (4)  | 0.06853 (17) | 0.0543 (8)                       |
| H9A  | 0.414505     | 0.883278    | 0.037089     | 0.065*                           |
| C10  | 0.2365 (4)   | 0.7534 (4)  | 0.06104 (17) | 0.0517 (8)                       |
| C11  | 0.1337 (4)   | 0.8057 (4)  | 0.01072 (18) | 0.0620 (9)                       |
| H11A | 0.164224     | 0.877235    | -0.018805    | 0.074*                           |
| C12  | -0.0149 (4)  | 0.7541 (5)  | 0.00294 (19) | 0.0669 (10)                      |
| H12A | -0.083256    | 0.790553    | -0.031380    | 0.080*                           |
| C13  | -0.0592 (4)  | 0.6488 (4)  | 0.0466 (2)   | 0.0610 (9)                       |
| C14  | 0.0408 (4)   | 0.5962 (4)  | 0.09817 (17) | 0.0542 (8)                       |
| C15  | 0.1889 (4)   | 0.6462 (4)  | 0.10559 (17) | 0.0514 (8)                       |
| H15A | 0.256713     | 0.609080    | 0.139942     | 0.062*                           |
| C16  | 1.0182 (6)   | 0.7928 (8)  | 0.4101 (2)   | 0.1109 (19)                      |
| H16A | 1.099315     | 0.809974    | 0.444494     | 0.166*                           |
| H16B | 0.929428     | 0.845664    | 0.420378     | 0.166*                           |
| H16C | 0.997848     | 0.684197    | 0.406049     | 0.166*                           |
| C17  | 1.1885 (5)   | 1.0598 (5)  | 0.1947 (2)   | 0.0817 (13)                      |
| H17A | 1.283889     | 1.107434    | 0.209172     | 0.123*                           |
| H17B | 1.201710     | 0.983432    | 0.162030     | 0.123*                           |
| H17C | 1.118491     | 1.136934    | 0.176371     | 0.123*                           |

Atomic displacement parameters ( $\text{\AA}^2$ )

|     | $U^{11}$    | $U^{22}$    | $U^{33}$    | $U^{12}$     | $U^{13}$     | $U^{23}$     |
|-----|-------------|-------------|-------------|--------------|--------------|--------------|
| Br1 | 0.0718 (3)  | 0.0733 (3)  | 0.0950 (4)  | -0.0108 (2)  | 0.0252 (2)   | 0.0155 (2)   |
| F1  | 0.0526 (12) | 0.0955 (17) | 0.1080 (19) | -0.0115 (13) | -0.0013 (12) | -0.0013 (15) |
| O1  | 0.0636 (15) | 0.090 (2)   | 0.0675 (17) | -0.0085 (15) | 0.0020 (13)  | 0.0060 (14)  |
| O2  | 0.0642 (17) | 0.099 (2)   | 0.0778 (19) | -0.0323 (16) | 0.0096 (14)  | 0.0020 (16)  |
| O3  | 0.0607 (16) | 0.095 (2)   | 0.0808 (19) | -0.0177 (15) | 0.0098 (14)  | 0.0268 (16)  |
| C1  | 0.0516 (19) | 0.0472 (18) | 0.076 (2)   | -0.0054 (15) | 0.0188 (17)  | 0.0007 (16)  |
| C2  | 0.057 (2)   | 0.058 (2)   | 0.065 (2)   | -0.0037 (17) | 0.0155 (17)  | 0.0059 (16)  |
| C3  | 0.0477 (17) | 0.0525 (19) | 0.066 (2)   | 0.0040 (15)  | 0.0128 (16)  | -0.0029 (16) |
| C4  | 0.0463 (17) | 0.0494 (17) | 0.066 (2)   | +0.0043 (15) | 0.0138 (16)  | -0.0054 (16) |
| C5  | 0.0530 (19) | 0.0452 (17) | 0.068 (2)   | -0.0037 (15) | 0.0226 (17)  | -0.0006 (15) |
| C6  | 0.0457 (16) | 0.0420 (16) | 0.064 (2)   | -0.0016 (14) | 0.0159 (14)  | -0.0011 (14) |
| C7  | 0.0489 (18) | 0.0544 (19) | 0.070 (2)   | -0.0055 (16) | 0.0150 (16)  | 0.0036 (17)  |
| C8  | 0.052 (2)   | 0.059 (2)   | 0.071 (2)   | -0.0067 (17) | 0.0109 (17)  | 0.0097 (18)  |
| C9  | 0.0520 (19) | 0.054 (2)   | 0.059 (2)   | -0.0062 (16) | 0.0164 (16)  | 0.0026 (16)  |
| C10 | 0.0502 (18) | 0.0512 (19) | 0.0548 (19) | -0.0021 (15) | 0.0104 (15)  | -0.0036 (14) |
| C11 | 0.069 (2)   | 0.058 (2)   | 0.059 (2)   | -0.0038 (18) | 0.0096 (17)  | 0.0078 (17)  |
| C12 | 0.058 (2)   | 0.075 (2)   | 0.064 (2)   | 0.004 (2)    | -0.0050 (17) | 0.0049 (19)  |
| C13 | 0.0466 (19) | 0.061 (2)   | 0.075 (2)   | -0.0020 (17) | 0.0068 (17)  | -0.0090 (18) |
| C14 | 0.0529 (19) | 0.0468 (17) | 0.064 (2)   | -0.0014 (16) | 0.0130 (16)  | -0.0013 (15) |
| C15 | 0.0471 (17) | 0.0505 (18) | 0.057 (2)   | 0.0027 (15)  | 0.0079 (14)  | 0.0020 (15)  |
| C16 | 0.104 (4)   | 0.155 (5)   | 0.069 (3)   | -0.039 (4)   | -0.006 (3)   | 0.020 (3)    |
| C17 | 0.067 (3)   | 0.088 (3)   | 0.094 (3)   | -0.030 (2)   | 0.025 (2)    | 0.000 (2)    |

Geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

|         |           |          |           |
|---------|-----------|----------|-----------|
| Br1—C14 | 1.877 (3) | C8—H8A   | 0.9300    |
| F1—C13  | 1.348 (4) | C9—C10   | 1.466 (5) |
| O1—C3   | 1.345 (4) | C9—H9A   | 0.9300    |
| O1—C16  | 1.441 (5) | C10—C11  | 1.374 (5) |
| O2—C4   | 1.362 (4) | C10—C15  | 1.405 (5) |
| O2—C17  | 1.390 (5) | C11—C12  | 1.390 (5) |
| O3—C7   | 1.231 (4) | C11—H11A | 0.9300    |
| C1—C6   | 1.377 (5) | C12—C13  | 1.371 (5) |
| C1—C2   | 1.383 (5) | C12—H12A | 0.9300    |
| C1—H1A  | 0.9300    | C13—C14  | 1.377 (5) |
| C2—C3   | 1.385 (5) | C14—C15  | 1.381 (5) |
| C2—H2A  | 0.9300    | C15—H15A | 0.9300    |
| C3—C4   | 1.405 (5) | C16—H16A | 0.9600    |
| C4—C5   | 1.364 (5) | C16—H16B | 0.9600    |
| C5—C6   | 1.414 (5) | C16—H16C | 0.9600    |
| C5—H5A  | 0.9300    | C17—H17A | 0.9600    |
| C6—C7   | 1.479 (5) | C17—H17B | 0.9600    |
| C7—C8   | 1.487 (5) | C17—H17C | 0.9600    |
| C8—C9   | 1.315 (5) |          |           |

|              |            |                 |            |
|--------------|------------|-----------------|------------|
| C3—O1—C16    | 118.2 (3)  | C11—C10—C9      | 120.0 (3)  |
| C4—O2—C17    | 119.9 (3)  | C15—C10—C9      | 121.2 (3)  |
| C6—C1—C2     | 122.0 (3)  | C10—C11—C12     | 121.5 (3)  |
| C6—C1—H1A    | 119.0      | C10—C11—H11A    | 119.3      |
| C2—C1—H1A    | 119.0      | C12—C11—H11A    | 119.3      |
| C1—C2—C3     | 120.0 (3)  | C13—C12—C11     | 119.0 (3)  |
| C1—C2—H2A    | 120.0      | C13—C12—H12A    | 120.5      |
| C3—C2—H2A    | 120.0      | C11—C12—H12A    | 120.5      |
| O1—C3—C2     | 125.2 (3)  | F1—C13—C12      | 119.7 (3)  |
| O1—C3—C4     | 116.0 (3)  | F1—C13—C14      | 119.4 (3)  |
| C2—C3—C4     | 118.8 (3)  | C12—C13—C14     | 120.8 (3)  |
| O2—C4—C5     | 125.1 (3)  | C13—C14—C15     | 120.3 (3)  |
| O2—C4—C3     | 114.2 (3)  | C13—C14—Br1     | 118.7 (3)  |
| C5—C4—C3     | 120.7 (3)  | C15—C14—Br1     | 121.0 (3)  |
| C4—C5—C6     | 120.8 (3)  | C14—C15—C10     | 119.7 (3)  |
| C4—C5—H5A    | 119.6      | C14—C15—H15A    | 120.1      |
| C6—C5—H5A    | 119.6      | C10—C15—H15A    | 120.1      |
| C1—C6—C5     | 117.7 (3)  | O1—C16—H16A     | 109.5      |
| C1—C6—C7     | 123.7 (3)  | O1—C16—H16B     | 109.5      |
| C5—C6—C7     | 118.5 (3)  | H16A—C16—H16B   | 109.5      |
| O3—C7—C6     | 120.6 (3)  | O1—C16—H16C     | 109.5      |
| O3—C7—C8     | 119.8 (3)  | H16A—C16—H16C   | 109.5      |
| C6—C7—C8     | 119.5 (3)  | H16B—C16—H16C   | 109.5      |
| C9—C8—C7     | 122.0 (3)  | O2—C17—H17A     | 109.5      |
| C9—C8—H8A    | 119.0      | O2—C17—H17B     | 109.5      |
| C7—C8—H8A    | 119.0      | H17A—C17—H17B   | 109.5      |
| C8—C9—C10    | 127.8 (3)  | O2—C17—H17C     | 109.5      |
| C8—C9—H9A    | 116.1      | H17A—C17—H17C   | 109.5      |
| C10—C9—H9A   | 116.1      | H17B—C17—H17C   | 109.5      |
| C11—C10—C15  | 118.7 (3)  |                 |            |
|              |            |                 |            |
| C6—C1—C2—C3  | 1.3 (5)    | C5—C6—C7—C8     | -178.8 (3) |
| C16—O1—C3—C2 | -7.8 (6)   | O3—C7—C8—C9     | -11.1 (6)  |
| C16—O1—C3—C4 | 171.3 (4)  | C6—C7—C8—C9     | 165.6 (3)  |
| C1—C2—C3—O1  | 175.4 (3)  | C7—C8—C9—C10    | -175.7 (3) |
| C1—C2—C3—C4  | -3.7 (5)   | C8—C9—C10—C11   | 178.4 (4)  |
| C17—O2—C4—C5 | -8.4 (6)   | C8—C9—C10—C15   | 0.1 (6)    |
| C17—O2—C4—C3 | 174.5 (4)  | C15—C10—C11—C12 | -0.4 (5)   |
| O1—C3—C4—O2  | 1.4 (5)    | C9—C10—C11—C12  | -178.8 (3) |
| C2—C3—C4—O2  | -179.4 (3) | C10—C11—C12—C13 | 0.0 (6)    |
| O1—C3—C4—C5  | -175.8 (3) | C11—C12—C13—F1  | -180.0 (3) |
| C2—C3—C4—C5  | 3.3 (5)    | C11—C12—C13—C14 | 1.0 (6)    |
| O2—C4—C5—C6  | -177.4 (3) | F1—C13—C14—C15  | 179.3 (3)  |
| C3—C4—C5—C6  | -0.5 (5)   | C12—C13—C14—C15 | -1.7 (6)   |
| C2—C1—C6—C5  | 1.6 (5)    | F1—C13—C14—Br1  | -2.8 (5)   |
| C2—C1—C6—C7  | -175.4 (3) | C12—C13—C14—Br1 | 176.2 (3)  |
| C4—C5—C6—C1  | -2.0 (5)   | C13—C14—C15—C10 | 1.2 (5)    |
| C4—C5—C6—C7  | 175.1 (3)  | Br1—C14—C15—C10 | -176.6 (2) |

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|             |           |                 |           |
|-------------|-----------|-----------------|-----------|
| C1—C6—C7—O3 | 174.9 (3) | C11—C10—C15—C14 | -0.2 (5)  |
| C5—C6—C7—O3 | -2.1 (5)  | C9—C10—C15—C14  | 178.2 (3) |
| C1—C6—C7—C8 | -1.9 (5)  |                 |           |

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*Hydrogen-bond geometry (Å, °)*

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| <i>D—H...A</i>              | <i>D—H</i> | <i>H...A</i> | <i>D...A</i> | <i>D—H...A</i> |
|-----------------------------|------------|--------------|--------------|----------------|
| C15—H15A...O2 <sup>i</sup>  | 0.93       | 2.61         | 3.506 (5)    | 162            |
| C11—H11A...O3 <sup>ii</sup> | 0.93       | 2.46         | 3.358 (5)    | 162            |

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

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