

## 3-(2-Chlorophenyl)-1,5-bis(4-chlorophenyl)pentane-1,5-dione

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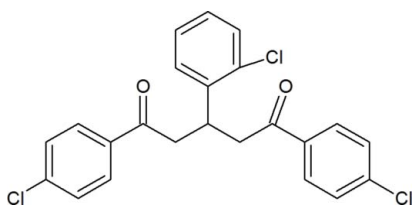
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Key indicators: single-crystal X-ray study;  $T = 296$  K; mean  $\sigma(\text{C}-\text{C}) = 0.002$  Å;  $R$  factor = 0.041;  $wR$  factor = 0.118; data-to-parameter ratio = 26.4.

In the title compound,  $\text{C}_{23}\text{H}_{17}\text{Cl}_3\text{O}_2$ , the dihedral angles between the 2-chlorophenyl group and the two 4-chlorophenyl groups are  $88.9$  (3) and  $12.9$  (2)°, while the angle between the mean planes of the two 4-chlorophenyl groups is  $77.9$  (5)°. The crystal packing is stabilized by intermolecular  $\text{C}-\text{H}\cdots\text{O}$  interactions between an H atom from each of the two 4-chlorophenyl groups and a ketone O atom in neighboring molecules, which link the molecules into chains diagonally along the  $ac$  plane of the unit cell. Additional intermolecular  $\pi-\pi$  stacking interactions occur between adjacent 2-chlorophenyl rings as well as between one of the 4-chlorophenyl rings and a 2-chlorophenyl ring, the distances between the centroids of interacting rings being  $3.931$  (6) and  $3.9915$  (4) Å, respectively.

### Related literature

For related structures, see: Insuasty *et al.* (2006); Teh *et al.* (2006); Huang *et al.* (2006); Qiu *et al.* (2006a,b); Butcher *et al.* (2007); Yathirajan *et al.* (2006, 2007). For related literature, see: Krohnke *et al.* (1976); Hirsch & Bailey, (1978).



### Experimental

#### Crystal data

$\text{C}_{23}\text{H}_{17}\text{Cl}_3\text{O}_2$   
 $M_r = 431.72$   
 Triclinic,  $P\bar{1}$   
 $a = 7.1717$  (8) Å  
 $b = 7.7000$  (15) Å  
 $c = 18.901$  (6) Å  
 $\alpha = 85.88$  (2)°  
 $\beta = 83.518$  (15)°  
 $\gamma = 77.656$  (12)°  
 $V = 1011.9$  (4) Å<sup>3</sup>  
 $Z = 2$   
 Mo  $K\alpha$  radiation  
 $\mu = 0.47$  mm<sup>-1</sup>  
 $T = 296$  K  
 $0.45 \times 0.39 \times 0.28$  mm

#### Data collection

Oxford Diffraction Gemini R CCD diffractometer  
 Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2007)  
 $T_{\min} = 0.712$ ,  $T_{\max} = 0.877$   
 14729 measured reflections  
 6690 independent reflections  
 3147 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.024$

#### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.041$   
 $wR(F^2) = 0.118$   
 $S = 0.93$   
 6690 reflections  
 253 parameters  
 H-atom parameters constrained  
 $\Delta\rho_{\max} = 0.24$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.33$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

| $D-\text{H}\cdots A$                          | $D-\text{H}$ | $\text{H}\cdots A$ | $D\cdots A$ | $D-\text{H}\cdots A$ |
|---|--------------|--------------------|-------------|----------------------|
| $\text{C7A}-\text{H7AA}\cdots\text{O1A}^i$    | 0.93         | 2.50               | 3.306 (2)   | 145                  |
| $\text{C7B}-\text{H7BA}\cdots\text{O1B}^{ii}$ | 0.93         | 2.51               | 3.2917 (17) | 142                  |

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

Data collection: *CrysAlisPro* (Oxford Diffraction, 2007); cell refinement: *CrysAlisPro*; data reduction: *CrysAlisPro*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2000); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2485).

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**supplementary materials**

*Acta Cryst.* (2007). E63, o4808-o4809 [ doi:10.1107/S1600536807059089 ]

### 3-(2-Chlorophenyl)-1,5-bis(4-chlorophenyl)pentane-1,5-dione

J. P. Jasinski, R. J. Butcher, H. S. Yathirajan, B. Narayana and M. T. Swamy

#### Comment

1,5-Diketones are important synthetic intermediates and starting materials in the synthesis of many heterocyclic compounds (Hirsch & Bailey, 1978; Krohnke, 1976). The structures of related compounds *viz.*, 1,5-bis(4-chlorophenyl)-3-(2-chloroquinolin-3-yl)pentane-1,5-dione (Insuasty *et al.* 2006), 1,5-(4-dichlorophenyl)-3-(2,5-dimethoxyphenyl)pentane-1,5-dione (Teh *et al.* 2006), 3-(2-furyl)-1,5-bis(4-methylphenyl)pentane-1,5-dione (Huang *et al.* 2006), 1,5-bis(4-chlorophenyl)-3-(4-pyridyl)pentane-1,5-dione (Qiu *et al.* 2006a), 3-(3-chlorophenyl)-1,5-bis(4-nitrophenyl)pentane-1,5-dione (Qiu *et al.* 2006b), 1,5-bis(3-bromothiophen-2-yl)-3-(2,3,5-trichlorophenyl)pentane-1,5-dione, (Butcher *et al.* 2007), 1,5-bis(3-bromo-2-thienyl)-3-(3-nitrophenyl)pentane-1,5-dione (Yathirajan *et al.* 2006), 1,5-bis(4-bromophenyl)-3-(3-nitrophenyl)pentane-1,5-dione, (Yathirajan *et al.* 2007) have been reported. A new 1,5-dione, (I), C<sub>23</sub>H<sub>17</sub>Cl<sub>3</sub>O<sub>2</sub> was synthesized and the crystal structure is reported here.

In the title compound, C<sub>23</sub>H<sub>17</sub>Cl<sub>3</sub>O<sub>2</sub>, the dihedral angles between the 2-chlorophenyl group and the two 4-chlorophenyl groups are 88.9 (3) and 12.9 (2)°, while the angle between the mean planes of the two 4-chlorophenyl groups is 77.9 (5)° (Fig. 1). The crystal packing is stabilized by intermolecular C–H⋯O interactions between a hydrogen atom from each of the two 4-chlorophenyl groups and its nearby ketone oxygen in neighboring molecules which link the molecules into chains diagonally and oblique along the *ac* plane of the unit cell (Fig. 2). Additional intermolecular  $\pi$ - $\pi$  stacking interactions occur between adjacent 2-chlorophenyl rings [*Cg*3 = center of gravity of the 4-chlorophenyl ring (C3B–C8B); *Cg*3⋯*Cg*3 = 3.931 (6) Å; 1 - *x*, 1 - *y*, -*z*] as well as between one of the 4-chlorophenyl rings and a 2-chlorophenyl ring [*Cg*1 = center of gravity of the 2-chlorophenyl ring (C1–C6); *Cg*1⋯*Cg*3 = 3.931 (6) Å; *x*, -1 + *y*, *z*].

#### Experimental

4-Chloroacetophenone (1.54 g, 0.1 mol) in ethanol (30 ml) was mixed with 2-chlorobenzaldehyde (0.7 g, 0.05 mol) and the mixture was treated with an aqueous solution of sodium hydroxide (5 ml, 30%) (Fig. 3). This mixture was stirred well and left for 12 h. The resulting crude solid mass was collected by filtration, washed, dried and recrystallized from toluene (yield 85%, m.p.: 401 K). The initially formed 1-(4-methoxyphenyl)-3-(2-chlorophenyl)prop-2-en-1-one, underwent Michael addition, resulting in the formation of the novel title compound (I). Analysis found: C 63.90, H 3.94%; C<sub>23</sub>H<sub>17</sub>Cl<sub>3</sub>O<sub>2</sub> requires: C 63.98, H 3.97%.

#### Refinement

The H atoms were placed in their calculated positions and then refined using the riding model with C–H = 0.93 to 0.98 Å, and with *U*<sub>iso</sub>(H) = 1.19 or 1.21*U*<sub>eq</sub>(C).

Figures

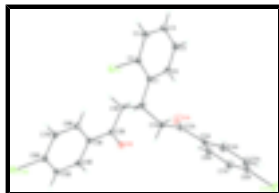


Fig. 1. Molecular structure of the title compound, showing atom labeling and 50% probability displacement ellipsoids.

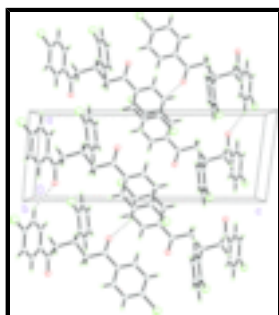


Fig. 2. Packing diagram of the title compound, viewed down the *b* axis. Dashed lines indicate intramolecular C—H...O hydrogen bonds.

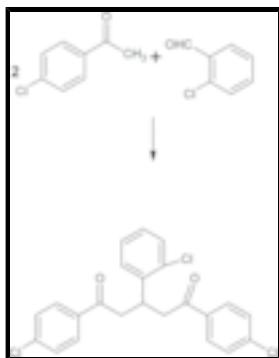


Fig. 3. Synthetic scheme for  $C_{23}H_{17}Cl_3O_2$ .

**3-(2-Chlorophenyl)-1,5-bis(4-chlorophenyl)pentane-1,5-dione**

*Crystal data*

$C_{23}H_{17}Cl_3O_2$

$M_r = 431.72$

Triclinic, *P* $\bar{1}$

Hall symbol: -P 1

$a = 7.1717$  (8) Å

$b = 7.7000$  (15) Å

$c = 18.901$  (6) Å

$\alpha = 85.88$  (2)°

$\beta = 83.518$  (15)°

$\gamma = 77.656$  (12)°

$V = 1011.9$  (4) Å<sup>3</sup>

$Z = 2$

$F_{000} = 444$

$D_x = 1.417$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation

$\lambda = 0.71073$  Å

Cell parameters from 4806 reflections

$\theta = 4.5$ – $32.6$ °

$\mu = 0.47$  mm<sup>-1</sup>

$T = 296$  K

Prism, pale yellow

$0.45 \times 0.39 \times 0.28$  mm

*Data collection*

|  |  |
|--|--|
| Oxford Diffraction Gemini R CCD diffractometer                             | 6690 independent reflections           |
| Radiation source: fine-focus sealed tube                                   | 3147 reflections with $I > 2\sigma(I)$ |
| Monochromator: graphite  | $R_{\text{int}} = 0.024$               |
| Detector resolution: 10.5081 pixels $\text{mm}^{-1}$                       | $\theta_{\text{max}} = 32.7^\circ$     |
| $T = 296$ K  | $\theta_{\text{min}} = 4.6^\circ$      |
| $\varphi$ and $\omega$ scans   | $h = -10 \rightarrow 10$               |
| Absorption correction: multi-scan (CrysAlis RED; Oxford Diffraction, 2007) | $k = -11 \rightarrow 11$               |
| $T_{\text{min}} = 0.712$ , $T_{\text{max}} = 0.877$                        | $l = -28 \rightarrow 25$               |
| 14729 measured reflections   |  |

*Refinement*

|  |  |
|--|--|
| Refinement on $F^2$  | Secondary atom site location: difference Fourier map         |
| Least-squares matrix: full                                     | Hydrogen site location: inferred from neighbouring sites     |
| $R[F^2 > 2\sigma(F^2)] = 0.041$                                | H-atom parameters constrained                                |
| $wR(F^2) = 0.118$  | $w = 1/[\sigma^2(F_o^2) + (0.0598P)^2]$                      |
| $S = 0.93$   | where $P = (F_o^2 + 2F_c^2)/3$                               |
| 6690 reflections   | $(\Delta/\sigma)_{\text{max}} = 0.001$                       |
| 253 parameters   | $\Delta\rho_{\text{max}} = 0.24 \text{ e } \text{\AA}^{-3}$  |
| Primary atom site location: structure-invariant direct methods | $\Delta\rho_{\text{min}} = -0.33 \text{ e } \text{\AA}^{-3}$ |
|  | Extinction correction: none                                  |

*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  $F^2 > 2\sigma(F^2)$  is used only for calculating  $R$ -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement.  $R$ -factors based on  $F^2$  are statistically about twice as large as those based on  $F$ , and  $R$ -factors based on ALL data will be even larger.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )*

|      | $x$          | $y$          | $z$         | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|------|--------------|--------------|-------------|----------------------------------|
| Cl   | 0.99371 (5)  | 0.01789 (6)  | 0.20354 (3) | 0.07076 (15)                     |
| Cl1A | -0.20263 (8) | 0.29570 (8)  | 0.60074 (3) | 0.09081 (19)                     |
| Cl1B | 0.84843 (6)  | 0.85651 (6)  | 0.01648 (3) | 0.07311 (16)                     |
| O1A  | 0.59359 (15) | 0.16616 (18) | 0.37462 (6) | 0.0739 (4)                       |

## supplementary materials

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|      |              |               |              |            |
|------|--------------|---------------|--------------|------------|
| O1B  | 0.22035 (13) | 0.35855 (15)  | 0.14247 (6)  | 0.0630 (3) |
| C    | 0.55268 (17) | 0.10895 (17)  | 0.23407 (7)  | 0.0392 (3) |
| H0A  | 0.6438       | 0.1859        | 0.2371       | 0.047*     |
| C1   | 0.65483 (18) | -0.08305 (17) | 0.24669 (7)  | 0.0407 (3) |
| C2   | 0.85377 (19) | -0.13735 (19) | 0.23327 (7)  | 0.0464 (3) |
| C3   | 0.9461 (2)   | -0.3130 (2)   | 0.24106 (9)  | 0.0629 (4) |
| H3A  | 1.0786       | -0.3451       | 0.2314       | 0.075*     |
| C4   | 0.8422 (3)   | -0.4397 (2)   | 0.26302 (10) | 0.0718 (5) |
| H4A  | 0.9041       | -0.5583       | 0.2685       | 0.086*     |
| C5   | 0.6468 (3)   | -0.3926 (2)   | 0.27701 (10) | 0.0725 (5) |
| H5A  | 0.5758       | -0.4785       | 0.2925       | 0.087*     |
| C6   | 0.5560 (2)   | -0.2157 (2)   | 0.26790 (8)  | 0.0572 (4) |
| H6A  | 0.4231       | -0.1855       | 0.2765       | 0.069*     |
| C1A  | 0.37939 (18) | 0.16621 (19)  | 0.28840 (7)  | 0.0445 (3) |
| H1AA | 0.3097       | 0.2827        | 0.2731       | 0.053*     |
| H1AB | 0.2945       | 0.0835        | 0.2886       | 0.053*     |
| C2A  | 0.4284 (2)   | 0.17464 (19)  | 0.36322 (7)  | 0.0455 (3) |
| C3A  | 0.26998 (19) | 0.19997 (18)  | 0.42202 (7)  | 0.0438 (3) |
| C4A  | 0.3106 (2)   | 0.2179 (2)    | 0.49019 (8)  | 0.0632 (4) |
| H4AA | 0.4371       | 0.2101        | 0.4990       | 0.076*     |
| C5A  | 0.1676 (3)   | 0.2470 (3)    | 0.54553 (9)  | 0.0712 (5) |
| H5AA | 0.1968       | 0.2592        | 0.5914       | 0.085*     |
| C6A  | -0.0180 (2)  | 0.2578 (2)    | 0.53224 (8)  | 0.0585 (4) |
| C7A  | -0.0643 (2)  | 0.2376 (2)    | 0.46536 (9)  | 0.0646 (4) |
| H7AA | -0.1909      | 0.2435        | 0.4571       | 0.078*     |
| C8A  | 0.0818 (2)   | 0.2081 (2)    | 0.41048 (8)  | 0.0549 (4) |
| H8AA | 0.0525       | 0.1936        | 0.3649       | 0.066*     |
| C1B  | 0.49133 (18) | 0.12994 (18)  | 0.15762 (7)  | 0.0421 (3) |
| H1BA | 0.6040       | 0.0926        | 0.1245       | 0.051*     |
| H1BB | 0.4045       | 0.0509        | 0.1539       | 0.051*     |
| C2B  | 0.39471 (18) | 0.31623 (18)  | 0.13577 (7)  | 0.0423 (3) |
| C3B  | 0.51050 (18) | 0.44822 (18)  | 0.10689 (7)  | 0.0394 (3) |
| C4B  | 0.41837 (19) | 0.61665 (19)  | 0.08326 (7)  | 0.0446 (3) |
| H4BA | 0.2851       | 0.6452        | 0.0864       | 0.054*     |
| C5B  | 0.5207 (2)   | 0.74129 (19)  | 0.05538 (8)  | 0.0499 (4) |
| H5BA | 0.4577       | 0.8529        | 0.0391       | 0.060*     |
| C6B  | 0.7180 (2)   | 0.69894 (19)  | 0.05180 (7)  | 0.0462 (3) |
| C7B  | 0.81408 (19) | 0.5351 (2)    | 0.07542 (8)  | 0.0496 (4) |
| H7BA | 0.9473       | 0.5092        | 0.0734       | 0.060*     |
| C8B  | 0.71080 (19) | 0.40966 (19)  | 0.10217 (8)  | 0.0473 (3) |
| H8BA | 0.7751       | 0.2975        | 0.1174       | 0.057*     |

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

|      | $U^{11}$     | $U^{22}$   | $U^{33}$   | $U^{12}$      | $U^{13}$    | $U^{23}$    |
|------|--------------|------------|------------|---------------|-------------|-------------|
| Cl   | 0.03547 (19) | 0.0776 (3) | 0.0992 (4) | -0.01499 (18) | -0.0055 (2) | 0.0040 (2)  |
| Cl1A | 0.0917 (4)   | 0.1141 (4) | 0.0619 (3) | -0.0255 (3)   | 0.0284 (3)  | -0.0169 (3) |
| Cl1B | 0.0674 (3)   | 0.0560 (3) | 0.0960 (4) | -0.0229 (2)   | 0.0084 (2)  | 0.0000 (2)  |

|     |             |             |             |              |              |             |
|-----|-------------|-------------|-------------|--------------|--------------|-------------|
| O1A | 0.0434 (6)  | 0.1231 (11) | 0.0596 (7)  | -0.0194 (6)  | -0.0097 (5)  | -0.0207 (7) |
| O1B | 0.0355 (5)  | 0.0703 (7)  | 0.0783 (8)  | -0.0073 (5)  | -0.0019 (5)  | 0.0118 (6)  |
| C   | 0.0321 (6)  | 0.0422 (7)  | 0.0430 (7)  | -0.0085 (5)  | -0.0022 (5)  | -0.0012 (6) |
| C1  | 0.0408 (7)  | 0.0434 (7)  | 0.0380 (7)  | -0.0088 (6)  | -0.0050 (6)  | -0.0014 (6) |
| C2  | 0.0412 (7)  | 0.0518 (8)  | 0.0449 (8)  | -0.0059 (6)  | -0.0061 (6)  | -0.0035 (6) |
| C3  | 0.0597 (9)  | 0.0604 (10) | 0.0619 (10) | 0.0079 (8)   | -0.0141 (8)  | -0.0075 (8) |
| C4  | 0.0958 (14) | 0.0450 (9)  | 0.0655 (11) | 0.0078 (9)   | -0.0128 (10) | -0.0028 (8) |
| C5  | 0.0992 (14) | 0.0477 (10) | 0.0722 (12) | -0.0243 (9)  | -0.0028 (10) | 0.0048 (8)  |
| C6  | 0.0560 (9)  | 0.0520 (9)  | 0.0626 (10) | -0.0152 (7)  | 0.0024 (8)   | 0.0025 (7)  |
| C1A | 0.0370 (6)  | 0.0525 (8)  | 0.0426 (8)  | -0.0044 (6)  | -0.0054 (6)  | -0.0062 (6) |
| C2A | 0.0436 (7)  | 0.0503 (8)  | 0.0434 (8)  | -0.0097 (6)  | -0.0063 (6)  | -0.0047 (6) |
| C3A | 0.0489 (8)  | 0.0460 (8)  | 0.0377 (8)  | -0.0106 (6)  | -0.0078 (6)  | -0.0028 (6) |
| C4A | 0.0553 (9)  | 0.0898 (12) | 0.0461 (9)  | -0.0146 (9)  | -0.0080 (8)  | -0.0116 (8) |
| C5A | 0.0819 (12) | 0.0957 (14) | 0.0375 (9)  | -0.0184 (10) | -0.0064 (9)  | -0.0131 (8) |
| C6A | 0.0639 (10) | 0.0621 (10) | 0.0472 (9)  | -0.0140 (8)  | 0.0086 (8)   | -0.0065 (7) |
| C7A | 0.0496 (9)  | 0.0892 (13) | 0.0559 (10) | -0.0167 (8)  | -0.0001 (8)  | -0.0095 (9) |
| C8A | 0.0502 (8)  | 0.0758 (11) | 0.0395 (8)  | -0.0145 (7)  | -0.0037 (7)  | -0.0059 (7) |
| C1B | 0.0411 (7)  | 0.0465 (8)  | 0.0381 (7)  | -0.0089 (6)  | 0.0003 (6)   | -0.0046 (6) |
| C2B | 0.0365 (7)  | 0.0524 (8)  | 0.0360 (7)  | -0.0047 (6)  | -0.0034 (6)  | -0.0022 (6) |
| C3B | 0.0367 (6)  | 0.0482 (8)  | 0.0317 (7)  | -0.0042 (6)  | -0.0047 (5)  | -0.0030 (6) |
| C4B | 0.0388 (7)  | 0.0464 (8)  | 0.0463 (8)  | -0.0010 (6)  | -0.0074 (6)  | -0.0056 (6) |
| C5B | 0.0518 (8)  | 0.0411 (8)  | 0.0531 (9)  | 0.0001 (6)   | -0.0081 (7)  | -0.0029 (7) |
| C6B | 0.0498 (8)  | 0.0462 (8)  | 0.0434 (8)  | -0.0130 (6)  | -0.0006 (6)  | -0.0042 (6) |
| C7B | 0.0361 (7)  | 0.0604 (9)  | 0.0500 (9)  | -0.0069 (6)  | -0.0039 (6)  | 0.0025 (7)  |
| C8B | 0.0377 (7)  | 0.0495 (8)  | 0.0496 (8)  | -0.0004 (6)  | -0.0049 (6)  | 0.0066 (6)  |

*Geometric parameters (Å, °)*

|          |             |          |             |
|----------|-------------|----------|-------------|
| Cl—C2    | 1.7439 (15) | C3A—C8A  | 1.379 (2)   |
| C11A—C6A | 1.7368 (16) | C4A—C5A  | 1.374 (2)   |
| C11B—C6B | 1.7380 (15) | C4A—H4AA | 0.9300      |
| O1A—C2A  | 1.2156 (16) | C5A—C6A  | 1.366 (2)   |
| O1B—C2B  | 1.2173 (15) | C5A—H5AA | 0.9300      |
| C—C1     | 1.5183 (18) | C6A—C7A  | 1.370 (2)   |
| C—C1A    | 1.5285 (18) | C7A—C8A  | 1.382 (2)   |
| C—C1B    | 1.5450 (19) | C7A—H7AA | 0.9300      |
| C—H0A    | 0.9800      | C8A—H8AA | 0.9300      |
| C1—C6    | 1.3759 (19) | C1B—C2B  | 1.5042 (19) |
| C1—C2    | 1.3976 (18) | C1B—H1BA | 0.9700      |
| C2—C3    | 1.379 (2)   | C1B—H1BB | 0.9700      |
| C3—C4    | 1.364 (3)   | C2B—C3B  | 1.4831 (19) |
| C3—H3A   | 0.9300      | C3B—C4B  | 1.3910 (19) |
| C4—C5    | 1.370 (3)   | C3B—C8B  | 1.3972 (18) |
| C4—H4A   | 0.9300      | C4B—C5B  | 1.371 (2)   |
| C5—C6    | 1.386 (2)   | C4B—H4BA | 0.9300      |
| C5—H5A   | 0.9300      | C5B—C6B  | 1.378 (2)   |
| C6—H6A   | 0.9300      | C5B—H5BA | 0.9300      |
| C1A—C2A  | 1.504 (2)   | C6B—C7B  | 1.372 (2)   |
| C1A—H1AA | 0.9700      | C7B—C8B  | 1.374 (2)   |



## supplementary materials

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|               |              |                  |              |
|---------------|--------------|------------------|--------------|
| C1A—H1AB      | 0.9700       | C7B—H7BA         | 0.9300       |
| C2A—C3A       | 1.488 (2)    | C8B—H8BA         | 0.9300       |
| C3A—C4A       | 1.375 (2)    |                  |              |
| C1—C—C1A      | 112.96 (11)  | C6A—C5A—C4A      | 119.09 (15)  |
| C1—C—C1B      | 107.79 (11)  | C6A—C5A—H5AA     | 120.5        |
| C1A—C—C1B     | 110.64 (10)  | C4A—C5A—H5AA     | 120.5        |
| C1—C—H0A      | 108.4        | C5A—C6A—C7A      | 121.53 (14)  |
| C1A—C—H0A     | 108.4        | C5A—C6A—C11A     | 120.38 (13)  |
| C1B—C—H0A     | 108.4        | C7A—C6A—C11A     | 118.08 (13)  |
| C6—C1—C2      | 115.99 (13)  | C6A—C7A—C8A      | 118.44 (15)  |
| C6—C1—C       | 121.92 (12)  | C6A—C7A—H7AA     | 120.8        |
| C2—C1—C       | 121.97 (12)  | C8A—C7A—H7AA     | 120.8        |
| C3—C2—C1      | 122.17 (14)  | C3A—C8A—C7A      | 121.32 (14)  |
| C3—C2—C1      | 117.63 (12)  | C3A—C8A—H8AA     | 119.3        |
| C1—C2—C1      | 120.19 (11)  | C7A—C8A—H8AA     | 119.3        |
| C4—C3—C2      | 119.73 (16)  | C2B—C1B—C        | 114.25 (11)  |
| C4—C3—H3A     | 120.1        | C2B—C1B—H1BA     | 108.7        |
| C2—C3—H3A     | 120.1        | C—C1B—H1BA       | 108.7        |
| C3—C4—C5      | 120.13 (16)  | C2B—C1B—H1BB     | 108.7        |
| C3—C4—H4A     | 119.9        | C—C1B—H1BB       | 108.7        |
| C5—C4—H4A     | 119.9        | H1BA—C1B—H1BB    | 107.6        |
| C4—C5—C6      | 119.39 (16)  | O1B—C2B—C3B      | 120.07 (12)  |
| C4—C5—H5A     | 120.3        | O1B—C2B—C1B      | 119.50 (13)  |
| C6—C5—H5A     | 120.3        | C3B—C2B—C1B      | 120.43 (11)  |
| C1—C6—C5      | 122.57 (15)  | C4B—C3B—C8B      | 118.21 (13)  |
| C1—C6—H6A     | 118.7        | C4B—C3B—C2B      | 119.49 (11)  |
| C5—C6—H6A     | 118.7        | C8B—C3B—C2B      | 122.30 (12)  |
| C2A—C1A—C     | 114.47 (11)  | C5B—C4B—C3B      | 121.13 (12)  |
| C2A—C1A—H1AA  | 108.6        | C5B—C4B—H4BA     | 119.4        |
| C—C1A—H1AA    | 108.6        | C3B—C4B—H4BA     | 119.4        |
| C2A—C1A—H1AB  | 108.6        | C4B—C5B—C6B      | 119.10 (13)  |
| C—C1A—H1AB    | 108.6        | C4B—C5B—H5BA     | 120.5        |
| H1AA—C1A—H1AB | 107.6        | C6B—C5B—H5BA     | 120.5        |
| O1A—C2A—C3A   | 121.13 (13)  | C7B—C6B—C5B      | 121.52 (13)  |
| O1A—C2A—C1A   | 120.22 (13)  | C7B—C6B—C11B     | 119.20 (11)  |
| C3A—C2A—C1A   | 118.61 (12)  | C5B—C6B—C11B     | 119.28 (11)  |
| C4A—C3A—C8A   | 118.37 (13)  | C6B—C7B—C8B      | 119.09 (13)  |
| C4A—C3A—C2A   | 119.48 (13)  | C6B—C7B—H7BA     | 120.5        |
| C8A—C3A—C2A   | 122.15 (12)  | C8B—C7B—H7BA     | 120.5        |
| C5A—C4A—C3A   | 121.23 (15)  | C7B—C8B—C3B      | 120.94 (13)  |
| C5A—C4A—H4AA  | 119.4        | C7B—C8B—H8BA     | 119.5        |
| C3A—C4A—H4AA  | 119.4        | C3B—C8B—H8BA     | 119.5        |
| C1A—C—C1—C6   | 38.87 (18)   | C4A—C5A—C6A—C7A  | 1.0 (3)      |
| C1B—C—C1—C6   | -83.68 (15)  | C4A—C5A—C6A—C11A | -179.63 (14) |
| C1A—C—C1—C2   | -145.14 (12) | C5A—C6A—C7A—C8A  | -0.9 (3)     |
| C1B—C—C1—C2   | 92.31 (14)   | C11A—C6A—C7A—C8A | 179.71 (13)  |
| C6—C1—C2—C3   | -0.3 (2)     | C4A—C3A—C8A—C7A  | 1.4 (2)      |
| C—C1—C2—C3    | -176.50 (13) | C2A—C3A—C8A—C7A  | -177.85 (15) |

|                 |              |                  |              |
|-----------------|--------------|------------------|--------------|
| C6—C1—C2—C1     | 178.12 (11)  | C6A—C7A—C8A—C3A  | -0.3 (3)     |
| C—C1—C2—C1      | 1.90 (18)    | C1—C—C1B—C2B     | -177.57 (10) |
| C1—C2—C3—C4     | -0.5 (2)     | C1A—C—C1B—C2B    | 58.46 (15)   |
| C1—C2—C3—C4     | -178.91 (13) | C—C1B—C2B—O1B    | -95.04 (15)  |
| C2—C3—C4—C5     | 0.3 (3)      | C—C1B—C2B—C3B    | 84.40 (14)   |
| C3—C4—C5—C6     | 0.7 (3)      | O1B—C2B—C3B—C4B  | -4.2 (2)     |
| C2—C1—C6—C5     | 1.3 (2)      | C1B—C2B—C3B—C4B  | 176.39 (11)  |
| C—C1—C6—C5      | 177.49 (14)  | O1B—C2B—C3B—C8B  | 176.04 (13)  |
| C4—C5—C6—C1     | -1.5 (3)     | C1B—C2B—C3B—C8B  | -3.40 (19)   |
| C1—C—C1A—C2A    | 67.11 (15)   | C8B—C3B—C4B—C5B  | 0.7 (2)      |
| C1B—C—C1A—C2A   | -171.94 (11) | C2B—C3B—C4B—C5B  | -179.14 (13) |
| C—C1A—C2A—O1A   | 12.0 (2)     | C3B—C4B—C5B—C6B  | -0.9 (2)     |
| C—C1A—C2A—C3A   | -170.18 (12) | C4B—C5B—C6B—C7B  | 0.1 (2)      |
| O1A—C2A—C3A—C4A | 1.4 (2)      | C4B—C5B—C6B—C11B | 179.99 (11)  |
| C1A—C2A—C3A—C4A | -176.42 (14) | C5B—C6B—C7B—C8B  | 1.1 (2)      |
| O1A—C2A—C3A—C8A | -179.32 (16) | C11B—C6B—C7B—C8B | -178.87 (11) |
| C1A—C2A—C3A—C8A | 2.9 (2)      | C6B—C7B—C8B—C3B  | -1.3 (2)     |
| C8A—C3A—C4A—C5A | -1.4 (3)     | C4B—C3B—C8B—C7B  | 0.5 (2)      |
| C2A—C3A—C4A—C5A | 177.96 (16)  | C2B—C3B—C8B—C7B  | -179.73 (13) |
| C3A—C4A—C5A—C6A | 0.2 (3)      |                  |              |

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

| $D-H\cdots A$                       | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|-------------------------------------|-------|-------------|-------------|---------------|
| C7A—H7AA $\cdots$ O1A <sup>i</sup>  | 0.93  | 2.50        | 3.306 (2)   | 145           |
| C7B—H7BA $\cdots$ O1B <sup>ii</sup> | 0.93  | 2.51        | 3.2917 (17) | 142           |

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

Fig. 1

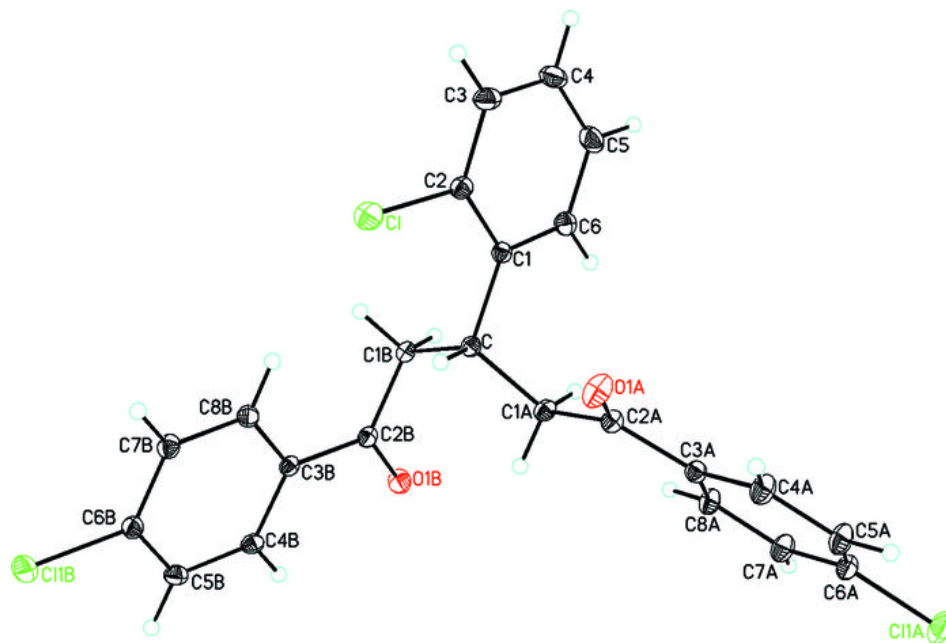


Fig. 2

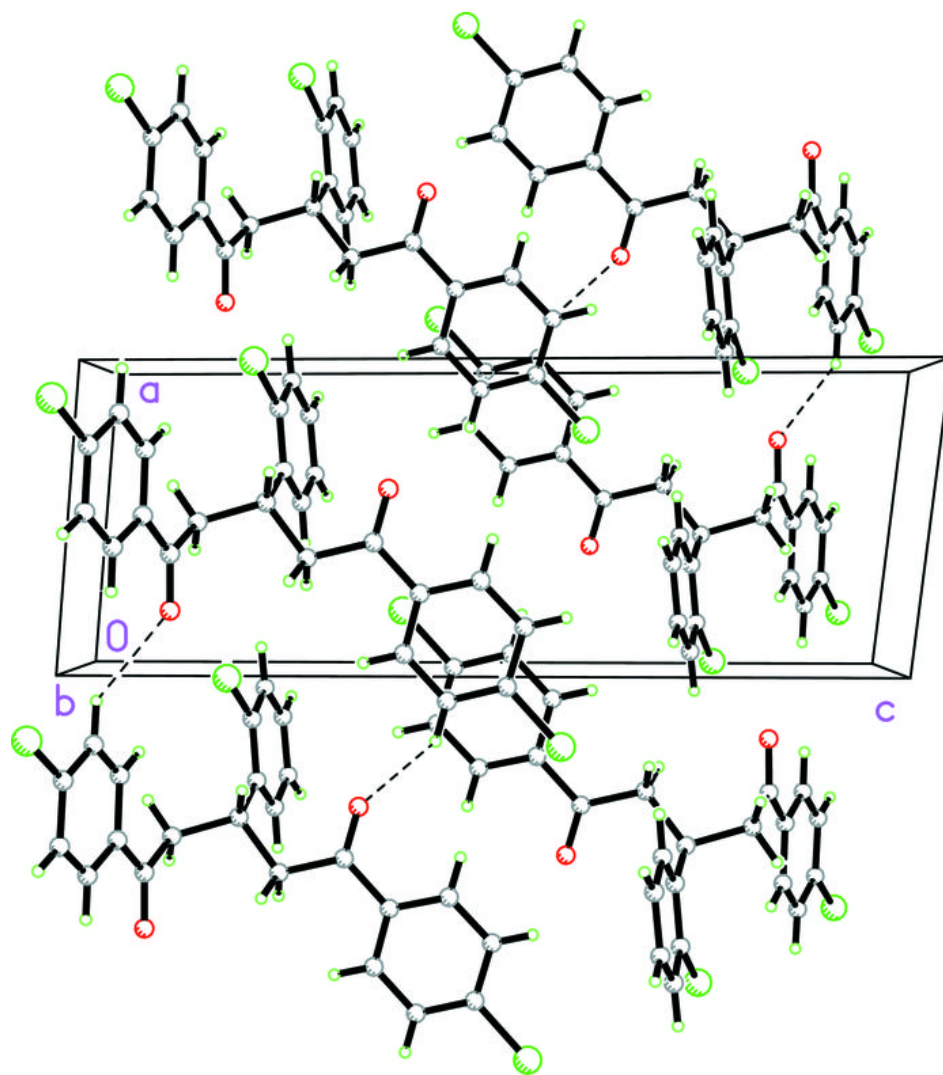


Fig. 3

