Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

3-Bromo-1-(2,4-dichlorophenyl)-2-methoxy-3-(4-methoxyphenyl)propan-1-one–2-bromo-3-(3-chloro-4-methoxyphenyl)-1-(4-chlorophenyl)-3-methoxypropan-1-one (0.91/0.09)

Ray J. Butcher,[a] Jerry P. Jasinski,[b]* B. Narayana,[c] Anil N. Mayekar[d] and H. S. Yathirajan[d]

[a]Department of Chemistry, Howard University, 525 College Street NW, Washington, DC 20059, USA, [b]Department of Chemistry, Keene State College, 229 Main Street, Keene, NH 03435-2001, USA, [c]Department of Studies in Chemistry, Mangalore University, Mangalagangotri 574 199, India, and [d]Department of Studies in Chemistry, University of Mysore, Manasagangotri, Mysore 570 006, India
Correspondence e-mail: jjasinski@keene.edu

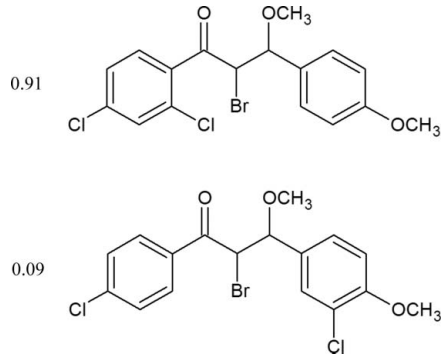
Received 5 September 2007; accepted 19 September 2007

Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.005 Å; disorder in main residue; R factor = 0.048; wR factor = 0.156; data-to-parameter ratio = 23.4.

The title co-crystal, $0.91C_{17}H_{15}BrCl_2O_3{\cdot}0.09C_{17}H_{15}BrCl_2O_3$, is a disordered mixture of two isomeric compounds. The ratio of the major and minor components in the co-crystal was obtained by refinement as 0.911 (3):0.089 (3). The dihedral angle between the two aromatic rings is 42.5 (2)°. Crystal packing is stabilized by intermolecular C—H···O hydrogen-bonding interactions which link the molecules into cyclic centrosymmetric $R_2^2(24)$ dimers.

Related literature

For related structures, see: Harrison *et al.* (2005); Butcher, Yathirajan, Anilkumar *et al.* (2006); Butcher, Yathirajan, Sarojini *et al.* (2006); Butcher, Yathirajan, Mithun *et al.* (2006); Sarojini *et al.* (2007); Yathirajan, Mayekar *et al.* (2007*a*); Yathirajan, Mayekar *et al.* (2007*b*); Yathirajan, Vijesh *et al.* (2007) . For related literature, see: Tam *et al.* (1989); Goto *et al.* (1991); Dinkova-Kostova *et al.* (1998); Nielson *et al.* (1998); Uchida *et al.* (1998); Liu *et al.* (2003); Indira *et al.* (2002); Rajas *et al.* (2002); Sarojini *et al.* (2006).



Experimental

Crystal data

$0.91C_{17}H_{15}BrCl_2O_3{\cdot}$-
$0.09C_{17}H_{15}BrCl_2O_3$
M_r = 418.09
Triclinic, $P\overline{1}$
a = 7.8013 (12) Å
b = 11.190 (2) Å
c = 11.438 (3) Å
α = 113.88 (2)°
β = 98.649 (16)°
γ = 95.991 (14)°
V = 887.4 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 2.63 mm^{-1}
T = 296 K
0.47 × 0.43 × 0.24 mm

Data collection

Oxford Diffraction Gemini R CCD diffractometer
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2007)
T_{min} = 0.574, T_{max} = 1.000 (expected range = 0.305–0.532)
10321 measured reflections
5156 independent reflections
2377 reflections with $I > 2\sigma(I)$
R_{int} = 0.028

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.156
S = 1.08
5156 reflections
220 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.73 e Å^{-3}
$\Delta\rho_{min}$ = −0.41 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C5—H5···O2^{i}	0.93	2.54	3.446 (4)	163

Symmetry code: (i) $-x+3, -y+2, -z+1$.

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: *WinGX* (Farrugia, 1999); software used to prepare material for publication: *WinGX*.

ANM thanks the University of Mysore for use of their research facilities. RJB acknowledges the NSF MRI program (grant No. CHE-0619278) for funds to purchase the X-ray diffractometer.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2459).

 doi:10.1107/S1600536807045977

References

Butcher, R. J., Yathirajan, H. S., Anilkumar, H. G., Sarojini, B. K. & Narayana, B. (2006). *Acta Cryst.* E**62**, o2525–o2527.

Butcher, R. J., Yathirajan, H. S., Mithun, A., Narayana, B. & Sarojini, B. K. (2006). *Acta Cryst.* E**62**, o2350–o2352.

Butcher, R. J., Yathirajan, H. S., Sarojini, B. K., Narayana, B. & Mithun, A. (2006). *Acta Cryst.* E**62**, o1629–o1630.

Dinkova-Kostova, A. T., Abey-gunawardana, C. & Talalay, P. (1998). *J. Med. Chem.* **41**, 5287–5296.

Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.

Goto, Y., Hayashi, A., Kimura, Y. & Nakayama, M. (1991). *J. Cryst. Growth*, **108**, 688–698.

Harrison, W. T. A., Yathirajan, H. S., Sarojini, B. K., Narayana, B. & Anilkumar, H. G. (2005). *Acta Cryst.* C**61**, o728–o730.

Indira, J., Karat, P. P. & Sarojini, B. K. (2002). *J. Cryst. Growth*, **242**, 209–214.

Liu, M., Wilairat, P., Cropft, S. L., Tan, A. L. C. & Go, M. I. (2003). *Bioorg. Med. Chem.* **11**, 2729–2738.

Nielson, S. F., Christensen, S. B., Cruciani, G., Kharazmi, A. & Liljefors, T. (1998). *J. Med. Chem.* **41**, 4819–4832.

Oxford Diffraction (2007). *CrysAlisPro* (Version 171.31.8) and *CrysAlis RED* (Version 1.171.31.8). Oxford Diffraction Ltd, Abingdon, Oxfordshire, England.

Rajas, J., Paya, M., Domingues, J. N. & Ferrandiz, M. L. (2002). *Bioorg. Med. Chem. Lett.* **12**, 1951–1954.

Sarojini, B. K., Narayana, B., Ashalatha, B. V., Indira, J. & Lobo, K. J. (2006). *J. Cryst. Growth*, **295**, 54–59.

Sarojini, B. K., Narayana, B., Yathirajan, H. S., Mayekar, A. N. & Bolte, M. (2007). *Acta Cryst.* E**63**, o3755.

Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

Tam, W., Guerin, B., Calabrese, J. C. & Stevenson, S. H. (1989). *Chem. Phys. Lett.* **154**, 93–96.

Uchida, T., Kozawa, K., Sakai, T., Aoki, M., Yoguchi, H., Abduryim, A. & Watanabe, Y. (1998). *Mol. Cryst. Liq. Cryst.* **315**, 135–140.

Yathirajan, H. S., Mayekar, A. M., Narayana, B., Sarojini, B. K. & Bolte, M. (2007*b*). *Acta Cryst.* E**63**, o2345.

Yathirajan, H. S., Mayekar, A. N., Narayana, B., Sarojini, B. K. & Bolte, M. (2007*a*). *Acta Cryst.* E**63**, o2196–o2197.

Yathirajan, H. S., Vijesh, A. M., Narayana, B., Sarojini, B. K. & Bolte, M. (2007). *Acta Cryst.* E**63**, o2198–o2199.

supplementary materials

Acta Cryst. (2007). E**63**, o4123-o4124 [**doi:10.1107/S1600536807045977**]

3-Bromo-1-(2,4-dichlorophenyl)-2-methoxy-3-(4-methoxyphenyl)propan-1-one-2-bromo-3-(3-chloro-4-methoxyphenyl)-1-(4-chlorophenyl)-3-methoxypropan-1-one (0.91/0.09)

R. J. Butcher, J. P. Jasinski, B. Narayana, A. N. Mayekar and H. S. Yathirajan

Comment

For a structurally simple group of compounds, chalcones display an impressive array of biological activities, among which antimalarial, antiprotozoal, nitric oxide inhibition and anticancer activities have been reported in the literature (Liu *et al.*, 2003; Nielson *et al.*, 1998; Rajas *et al.*, 2002; Dinkova-Kostova *et al.*, 1998). Among several organic compounds reported for non-linear optical (NLO) properties, chalcone derivatives are notable materials for their excellent blue light transmittance and good crystallizability. They provide a necessary configuration to show NLO properties, with two planar rings connected through a conjugated double bond. The substitution of a bromo group on either of the phenyl rings greatly influences the non-centrosymmetric crystal packing. The bromo group can obviously improve the molecular first-order hyperpolarizabilities and can effectively reduce dipole-dipole interactions between the molecules (Goto *et al.*, 1991; Uchida *et al.*, 1998; Tam *et al.*, 1989; Indira *et al.*, 2002; Sarojini *et al.*, 2006). Chalcone derivatives usually have a lower melting temperature, which can be a drawback when these crystals are used in optical instruments. Chalcone dibromides usually have higher melting points and are thermally stable. The structures of chalcone dibromides *viz*., 2,3-dibromo-3-(4-bromo-6-methoxy-2-naphthyl)-1-(4-methoxyphenyl)propan-1-one (Sarojini *et al.*, 2007), 2,3-dibromo-1-(2,4-dichlorophenyl)-3-(4,5-dimethoxy-2-nitrophenyl)propan-1-one (Yathirajan, Mayekar *et al.*, 2007*a*), 2,3-dibromo-3-(5-bromo-6-methoxy-2-naphthyl)-1-(2,4-dichlorophenyl)propan-1-one (Yathirajan, Mayekar *et al.*, 2007*b*), 2,3-dibromo-1-(3-bromo-2-thienyl)-3-(4-fluorophenyl)propan-1-one (Yathirajan, Vijesh *et al.*, 2007), 2,3-dibromo-3-(4-methoxyphenyl)-1- phenylpropan-1-one (Butcher, Yathirajan, Anilkumar *et al.*, 2006), 2,3-Dibromo-1-(4-methoxyphenyl)-3-[4-(methylsulfanyl)phenyl]propan-1-one (Butcher, Yathirajan, Sarojini *et al.*, 2006), 2-bromo-3-hydroxy-1-(4-methylphenyl)-3-[4-(methylsulfanyl)phenyl] propan-1-one (Butcher, Yathirajan, Mithun *et al.*, 2006), 2,3-dibromo-1,3-diphenylpropan-1-one (Harrison *et al.*, 2005) have been reported. A new chalcone dibromide, was prepared by the bromination of the chalcone, (2E)-1-(2,4-dichlorophenyl)-3-(4-methoxyphenyl)prop-2-en-1-one. The title compound, a cocrystal, was obtained, during repeated crystallization in a mixture of ethyl acetate and methanol due to the reaction of 2,3-dibromo-1-(2,4-dichlorophenyl)-3-(4-methoxyphenyl)propan-1-one with methanol. We report here the crystal structure of the title compound.

The mean planes of the two benzene rings are twisted with a dihedral angle of 42.5 (9)° (Fig.1). In the cocrystal the major occupied chlorine atom [Cl1; 0.911 (3)] and minor occupied hydrogen atom [H2; 0.089 (3)] are located on atom C2 of the *para*-chlorophenyl ring while the minor occupied chlorine atom [Cl3; 0.089 (3)] and major occupied hydrogen atom [H12; 0.911 (3)] are located on atom C12 of the 4-methoxyphenyl ring. The Br1–C8–C9–C10 and C17–O3–C9–C10 torsion angles are 61.6 (3)° and −72.7 (3)°, respectively.

Crystal packing is stabilized by intermolecular C—H···O hydrogen bonding interactions between the 4-methoxyphenyl oxygen atom (O2) of the molecule at (3 − *x*, 2 − *y*, 1 − *z*) and atom H5A of the molecule at (*x*, *y*, *z*), which form cyclic centrosymmetric $R^2_2(24)$ dimers (Fig. 2).

Experimental

(2E)-1-(2,4-Dichlorophenyl)-3-(2-methoxyphenyl)prop-2-en-1-one (3.07 g, 0.01 mole) was treated with bromine in acetic acid (30%) until the orange colour of the solution persisted (Fig.3). After stirring for half an hour, the contents were poured on to crushed ice. The resulting solid mass was collected by filtration. The compound was dried and recrystallized from ethanol. Crystals suitable for X-ray structure determination were obtained from a 1:1 mixture of ethyl acetate and methanol by slow evaporation (yield 70%; m.p. 365–369 K). Analysis found: C 44.74, H 3.58; $C_{34}H_{30}Br_2Cl_4O_6$ requires: C 48.83, H 3.62.

Refinement

H atoms were placed in calculated positions and were refined using a riding model, with C—H = 0.93–0.98 Å, and U_{iso}(H) = 1.19–1.56U_{eq}(C). The ratio of the major [Cl1 and H12] and minor [Cl3 and H2] components in the cocrystal was obtained by refinement as 0.911 (3):0.089 (3). Owing to the poor diffraction quality of the crystal, the ratio of observed to unique reflections is low (46%).

Figures

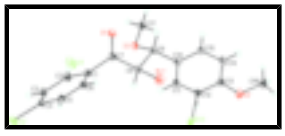


Fig. 1. Molecular structure of the title compound, showing atom labeling and 50% probability displacement ellipsoids. Both major [Cl1 and H12; 0.911 (3)] and minor [Cl3 and H2; 0.089 (3)] components of the cocrystal are displayed.

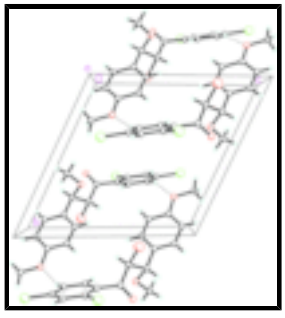


Fig. 2. Packing diagram of the title compound, viewed down the *a* axis. Dashed lines indicate intermolecular C—H···O hydrogen bonds. Only the major component (Cl1 and H12) of the cocrystal is displayed.

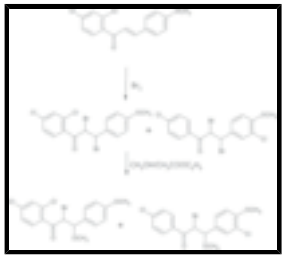


Fig. 3. Synthesis scheme for the title compound.

3-Bromo-1-(2,4-dichlorophenyl)-2-methoxy-3-(4-methoxyphenyl)propan-1-one–\ 2-bromo-3-(3-chloro-4-methoxyphenyl)-1-(4-chlorophenyl)-3-methoxypropan-1-one (0.91/0.09)

Crystal data

$0.91C_{17}H_{15}BrCl_2O_3{\cdot}0.09C_{17}H_{15}BrCl_2O_3$	$Z = 2$
$M_r = 418.09$	$F_{000} = 420$

Triclinic, $P\bar{1}$	D_x = 1.565 Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 7.8013 (12) Å	Cell parameters from 3589 reflections
b = 11.190 (2) Å	θ = 4.7–32.4º
c = 11.438 (3) Å	μ = 2.63 mm^{-1}
α = 113.88 (2)º	T = 296 K
β = 98.649 (16)º	Block, pale yellow
γ = 95.991 (14)º	0.47 × 0.43 × 0.24 mm
V = 887.4 (4) Å^3	

Data collection

Oxford Diffraction Gemini R CCD diffractometer	5156 independent reflections
Radiation source: fine-focus sealed tube	2377 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.028
Detector resolution: 10.5081 pixels mm^{-1}	θ_{max} = 32.5º
T = 296 K	θ_{min} = 4.7º
φ and ω scans	h = −11→11
Absorption correction: multi-scan (CrysAlis RED; Oxford Diffraction, 2007)	k = −15→16
T_{min} = 0.574, T_{max} = 1.000	l = −16→17
10321 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
$R[F^2 > 2\sigma(F^2)]$ = 0.048	H-atom parameters constrained
$wR(F^2)$ = 0.156	$w = 1/[\sigma^2(F_o^2) + (0.0715P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.08	$(\Delta/\sigma)_{max}$ = 0.001
5156 reflections	$\Delta\rho_{max}$ = 0.73 e Å^{-3}
220 parameters	$\Delta\rho_{min}$ = −0.41 e Å^{-3}
Primary atom site location: structure-invariant direct methods	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 $F^2 > 2\text{sigma}(F^2)$ is used only for calculat-

ing *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 ($Å^2$)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Br1	0.82726 (6)	0.94242 (4)	0.28992 (5)	0.0906 (2)	
Cl1	0.51820 (12)	0.64546 (13)	0.37858 (10)	0.0805 (4)	0.911 (3)
Cl2	0.93309 (16)	0.61933 (11)	0.78055 (9)	0.0795 (3)	
Cl3	1.5542 (11)	1.1758 (9)	0.4443 (12)	0.070 (4)	0.089 (3)
O1	0.7642 (3)	0.6203 (3)	0.1876 (2)	0.0708 (7)	
O2	1.5216 (3)	1.2295 (3)	0.2055 (3)	0.0773 (7)	
O3	1.1774 (3)	0.6884 (2)	0.2033 (2)	0.0563 (6)	
C1	0.8768 (4)	0.6819 (3)	0.4134 (3)	0.0482 (7)	
C2	0.7306 (4)	0.6530 (4)	0.4590 (3)	0.0568 (8)	
H2	0.6188	0.6455	0.4120	0.068*	0.089 (3)
C3	0.7463 (5)	0.6348 (3)	0.5725 (3)	0.0605 (8)	
H3	0.6471	0.6162	0.6025	0.073*	
C4	0.9128 (5)	0.6449 (3)	0.6396 (3)	0.0533 (7)	
C5	1.0618 (5)	0.6736 (3)	0.5984 (3)	0.0596 (8)	
H5	1.1734	0.6803	0.6453	0.072*	
C6	1.0414 (4)	0.6920 (3)	0.4858 (3)	0.0555 (8)	
H6	1.1413	0.7120	0.4571	0.067*	
C7	0.8632 (4)	0.6976 (3)	0.2893 (3)	0.0528 (7)	
C8	0.9821 (4)	0.8176 (3)	0.2949 (3)	0.0521 (7)	
H8	1.0686	0.8582	0.3782	0.063*	
C9	1.0791 (4)	0.7814 (3)	0.1826 (3)	0.0474 (7)	
H9	0.9942	0.7374	0.0987	0.057*	
C10	1.1983 (4)	0.9004 (3)	0.1866 (3)	0.0508 (7)	
C11	1.3367 (5)	0.9743 (4)	0.2907 (3)	0.0606 (9)	
H11	1.3590	0.9505	0.3598	0.073*	
C12	1.4431 (5)	1.0832 (4)	0.2943 (4)	0.0651 (9)	
H12	1.5350	1.1326	0.3659	0.078*	0.911 (3)
C13	1.4126 (4)	1.1185 (3)	0.1917 (3)	0.0557 (8)	
C14	1.2783 (5)	1.0437 (4)	0.0851 (4)	0.0631 (9)	
H14	1.2590	1.0654	0.0144	0.076*	
C15	1.1711 (4)	0.9348 (3)	0.0838 (3)	0.0546 (8)	
H15	1.0797	0.8848	0.0120	0.065*	
C16	1.4996 (6)	1.2684 (5)	0.1019 (5)	0.0880 (13)	
H16A	1.5773	1.3512	0.1274	0.132*	
H16B	1.3798	1.2789	0.0818	0.132*	
H16C	1.5270	1.2015	0.0260	0.132*	
C17	1.2483 (6)	0.6162 (4)	0.0951 (3)	0.0718 (10)	
H17A	1.2922	0.5437	0.1065	0.108*	
H17B	1.3430	0.6735	0.0884	0.108*	
H17C	1.1581	0.5820	0.0168	0.108*	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.1155 (4)	0.0796 (3)	0.1043 (4)	0.0536 (3)	0.0558 (3)	0.0469 (3)
Cl1	0.0448 (5)	0.1334 (11)	0.0698 (7)	0.0109 (6)	0.0141 (4)	0.0504 (7)
Cl2	0.1141 (8)	0.0807 (7)	0.0548 (5)	0.0195 (6)	0.0207 (5)	0.0388 (5)
Cl3	0.038 (5)	0.041 (5)	0.107 (9)	−0.013 (4)	−0.009 (5)	0.022 (5)
O1	0.0804 (17)	0.0758 (17)	0.0433 (13)	−0.0065 (13)	0.0120 (12)	0.0181 (12)
O2	0.0675 (16)	0.0766 (18)	0.102 (2)	−0.0017 (13)	0.0239 (14)	0.0537 (16)
O3	0.0771 (15)	0.0559 (13)	0.0543 (13)	0.0260 (11)	0.0272 (11)	0.0340 (11)
C1	0.0504 (17)	0.0484 (17)	0.0520 (17)	0.0136 (13)	0.0217 (14)	0.0229 (14)
C2	0.0487 (17)	0.070 (2)	0.0563 (19)	0.0089 (15)	0.0155 (14)	0.0304 (17)
C3	0.066 (2)	0.065 (2)	0.062 (2)	0.0114 (17)	0.0291 (17)	0.0328 (17)
C4	0.066 (2)	0.0515 (18)	0.0498 (17)	0.0136 (15)	0.0212 (15)	0.0252 (14)
C5	0.061 (2)	0.060 (2)	0.068 (2)	0.0163 (16)	0.0141 (16)	0.0358 (18)
C6	0.0552 (19)	0.0548 (18)	0.065 (2)	0.0116 (15)	0.0243 (16)	0.0302 (16)
C7	0.0585 (19)	0.0526 (18)	0.0530 (18)	0.0177 (15)	0.0235 (15)	0.0223 (15)
C8	0.066 (2)	0.0511 (18)	0.0401 (15)	0.0113 (15)	0.0147 (14)	0.0185 (14)
C9	0.0609 (18)	0.0501 (17)	0.0349 (15)	0.0118 (15)	0.0129 (13)	0.0206 (13)
C10	0.0539 (18)	0.0575 (19)	0.0533 (18)	0.0174 (15)	0.0183 (15)	0.0318 (16)
C11	0.070 (2)	0.066 (2)	0.0474 (18)	0.0082 (18)	0.0003 (17)	0.0309 (17)
C12	0.066 (2)	0.065 (2)	0.065 (2)	0.0034 (18)	0.0036 (18)	0.0328 (18)
C13	0.0523 (18)	0.0565 (19)	0.065 (2)	0.0113 (16)	0.0214 (17)	0.0291 (17)
C14	0.070 (2)	0.076 (2)	0.069 (2)	0.021 (2)	0.0254 (19)	0.051 (2)
C15	0.064 (2)	0.058 (2)	0.0446 (17)	0.0087 (16)	0.0082 (14)	0.0258 (15)
C16	0.102 (3)	0.084 (3)	0.103 (3)	0.013 (2)	0.047 (3)	0.058 (3)
C17	0.100 (3)	0.068 (2)	0.059 (2)	0.035 (2)	0.036 (2)	0.0279 (18)

Geometric parameters (Å, °)

Br1—C8	1.952 (3)	C8—C9	1.523 (4)
Cl1—C2	1.746 (3)	C8—H8	0.98
Cl2—C4	1.736 (3)	C9—C10	1.523 (4)
Cl3—C12	1.641 (11)	C9—H9	0.98
O1—C7	1.213 (4)	C10—C15	1.372 (4)
O2—C13	1.368 (4)	C10—C11	1.377 (5)
O2—C16	1.412 (5)	C11—C12	1.383 (5)
O3—C17	1.407 (4)	C11—H11	0.93
O3—C9	1.427 (4)	C12—C13	1.379 (5)
C1—C2	1.383 (4)	C12—H12	0.93
C1—C6	1.386 (4)	C13—C14	1.375 (5)
C1—C7	1.490 (4)	C14—C15	1.398 (5)
C2—C3	1.383 (5)	C14—H14	0.93
C2—H2	0.93	C15—H15	0.93
C3—C4	1.374 (5)	C16—H16A	0.96
C3—H3	0.93	C16—H16B	0.96
C4—C5	1.374 (5)	C16—H16C	0.96
C5—C6	1.373 (5)	C17—H17A	0.96

C5—H5	0.93	C17—H17B	0.96
C6—H6	0.93	C17—H17C	0.96
C7—C8	1.523 (5)		
C13—O2—C16	118.3 (3)	C10—C9—H9	110.0
C17—O3—C9	112.8 (2)	C8—C9—H9	110.0
C2—C1—C6	117.6 (3)	C15—C10—C11	118.4 (3)
C2—C1—C7	122.8 (3)	C15—C10—C9	120.1 (3)
C6—C1—C7	119.6 (3)	C11—C10—C9	121.5 (3)
C1—C2—C3	121.8 (3)	C10—C11—C12	121.2 (3)
C1—C2—Cl1	120.9 (2)	C10—C11—H11	119.4
C3—C2—Cl1	117.2 (2)	C12—C11—H11	119.4
C1—C2—H2	119.1	C13—C12—C11	119.9 (3)
C3—C2—H2	119.1	C13—C12—Cl3	127.8 (5)
C4—C3—C2	118.1 (3)	C11—C12—Cl3	109.9 (5)
C4—C3—H3	121.0	C13—C12—H12	120.0
C2—C3—H3	121.0	C11—C12—H12	120.0
C3—C4—C5	122.2 (3)	O2—C13—C14	124.5 (3)
C3—C4—Cl2	118.3 (2)	O2—C13—C12	115.7 (3)
C5—C4—Cl2	119.5 (3)	C14—C13—C12	119.8 (3)
C6—C5—C4	118.1 (3)	C13—C14—C15	119.4 (3)
C6—C5—H5	120.9	C13—C14—H14	120.3
C4—C5—H5	120.9	C15—C14—H14	120.3
C5—C6—C1	122.2 (3)	C10—C15—C14	121.2 (3)
C5—C6—H6	118.9	C10—C15—H15	119.4
C1—C6—H6	118.9	C14—C15—H15	119.4
O1—C7—C1	123.1 (3)	O2—C16—H16A	109.5
O1—C7—C8	120.5 (3)	O2—C16—H16B	109.5
C1—C7—C8	116.4 (3)	H16A—C16—H16B	109.5
C7—C8—C9	112.5 (3)	O2—C16—H16C	109.5
C7—C8—Br1	105.6 (2)	H16A—C16—H16C	109.5
C9—C8—Br1	111.3 (2)	H16B—C16—H16C	109.5
C7—C8—H8	109.1	O3—C17—H17A	109.5
C9—C8—H8	109.1	O3—C17—H17B	109.5
Br1—C8—H8	109.1	H17A—C17—H17B	109.5
O3—C9—C10	112.0 (3)	O3—C17—H17C	109.5
O3—C9—C8	101.5 (2)	H17A—C17—H17C	109.5
C10—C9—C8	113.2 (2)	H17B—C17—H17C	109.5
O3—C9—H9	110.0		
C6—C1—C2—C3	0.1 (5)	C7—C8—C9—O3	−60.0 (3)
C7—C1—C2—C3	178.0 (3)	Br1—C8—C9—O3	−178.24 (19)
C6—C1—C2—Cl1	177.1 (3)	C7—C8—C9—C10	179.8 (2)
C7—C1—C2—Cl1	−5.0 (5)	Br1—C8—C9—C10	61.6 (3)
C1—C2—C3—C4	−0.6 (5)	O3—C9—C10—C15	125.6 (3)
Cl1—C2—C3—C4	−177.8 (3)	C8—C9—C10—C15	−120.4 (3)
C2—C3—C4—C5	0.7 (5)	O3—C9—C10—C11	−53.1 (4)
C2—C3—C4—Cl2	−178.9 (3)	C8—C9—C10—C11	60.8 (4)
C3—C4—C5—C6	−0.2 (5)	C15—C10—C11—C12	2.0 (5)
Cl2—C4—C5—C6	179.4 (3)	C9—C10—C11—C12	−179.2 (3)

C4—C5—C6—C1	−0.4 (5)	C10—C11—C12—C13	−0.8 (6)
C2—C1—C6—C5	0.4 (5)	C10—C11—C12—Cl3	162.9 (5)
C7—C1—C6—C5	−177.6 (3)	C16—O2—C13—C14	−2.2 (5)
C2—C1—C7—O1	−46.6 (5)	C16—O2—C13—C12	178.3 (4)
C6—C1—C7—O1	131.3 (3)	C11—C12—C13—O2	178.4 (3)
C2—C1—C7—C8	133.7 (3)	Cl3—C12—C13—O2	17.8 (7)
C6—C1—C7—C8	−48.4 (4)	C11—C12—C13—C14	−1.1 (5)
O1—C7—C8—C9	−47.3 (4)	Cl3—C12—C13—C14	−161.6 (6)
C1—C7—C8—C9	132.4 (3)	O2—C13—C14—C15	−177.7 (3)
O1—C7—C8—Br1	74.2 (3)	C12—C13—C14—C15	1.8 (5)
C1—C7—C8—Br1	−106.1 (2)	C11—C10—C15—C14	−1.3 (5)
C17—O3—C9—C10	−72.7 (3)	C9—C10—C15—C14	179.9 (3)
C17—O3—C9—C8	166.3 (3)	C13—C14—C15—C10	−0.6 (5)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C5—H5···O2^{i}	0.93	2.54	3.446 (4)	163

Symmetry codes: (i) −*x*+3, −*y*+2, −*z*+1.

Fig. 1

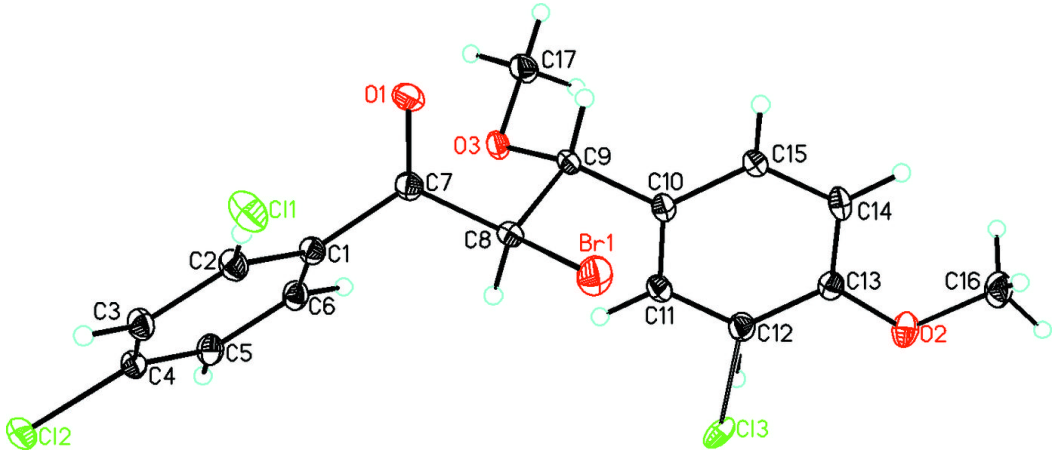

C17
O1
O3
C9
C15
C10
C7
Cl1
C14
C8
Br1
C2
C1
C13
C16
C3
C6
C11
C12
O2
C5
C4
Cl2
Cl3

Fig. 2

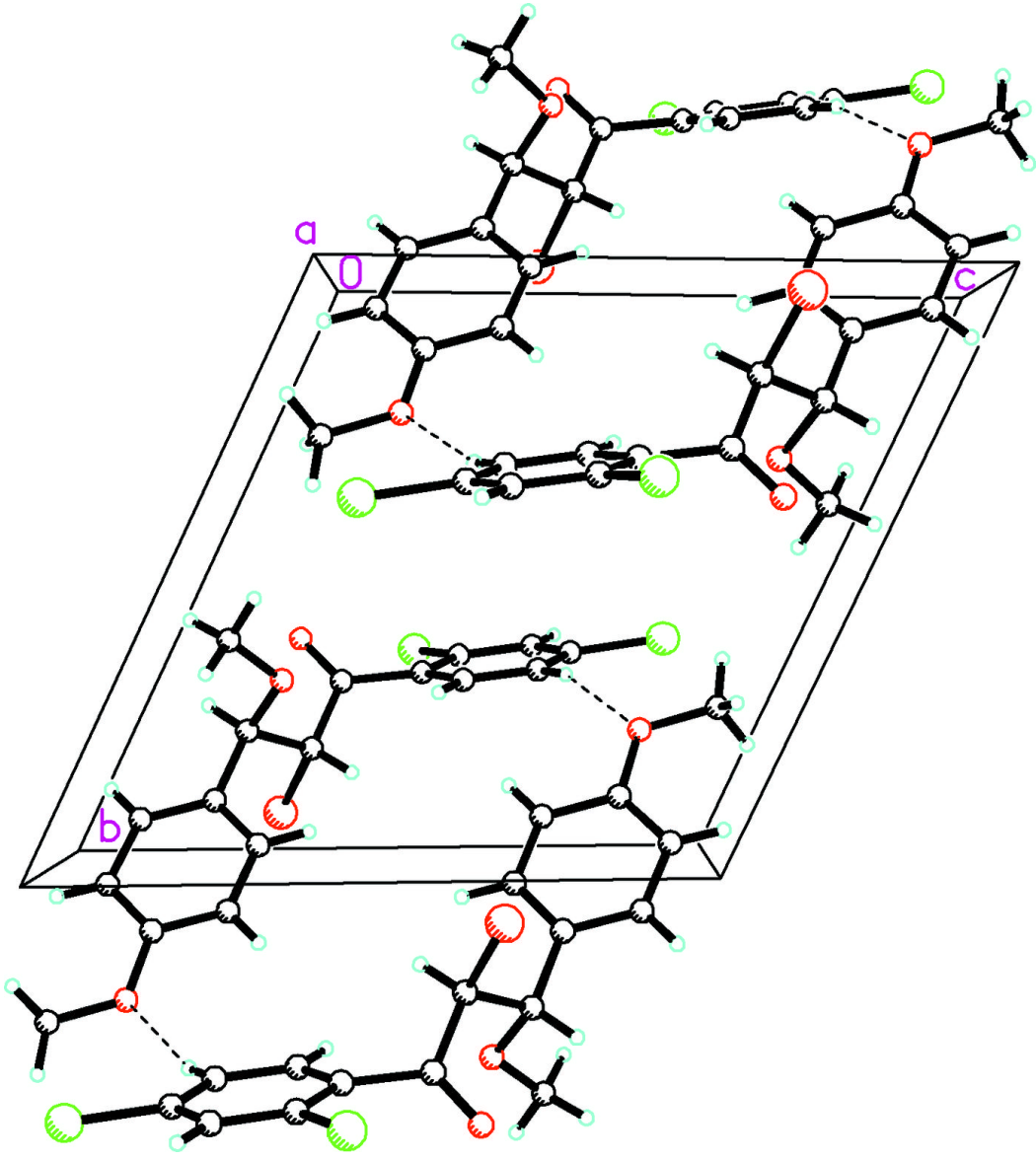
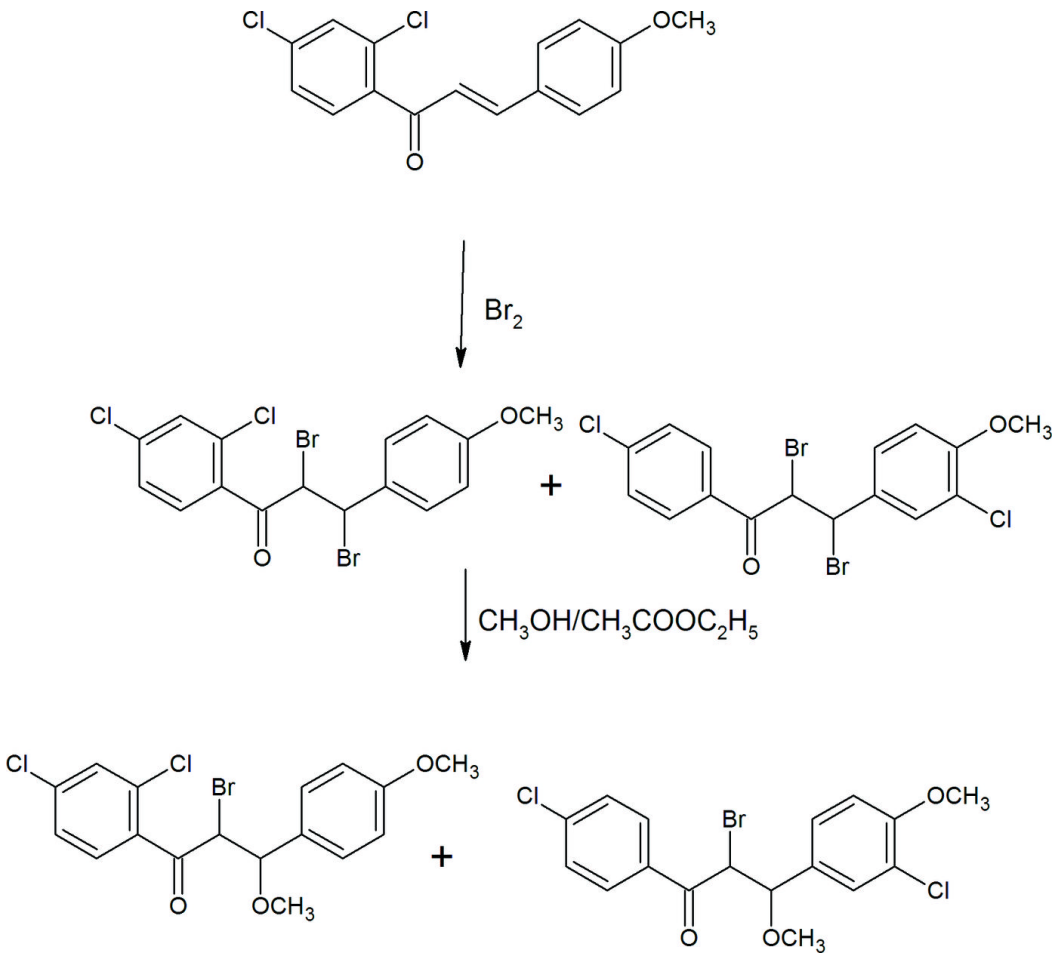


Fig. 3


Cl
Cl
OCH3
O
Br2
Cl
Cl
Br
OCH3
O
Br
+
Cl
Br
OCH3
Cl
O
Br
CH3OH/CH3COOC2H5
Cl
Cl
Br
OCH3
O
OCH3
+
Cl
Br
OCH3
Cl
O
OCH3