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(2*E*)-1-(4-Methylphenyl)-3-(2,3,5-trichlorophenyl)prop-2-en-1-one

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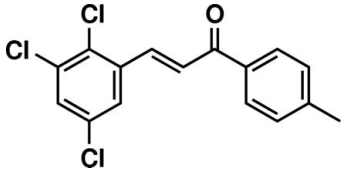
Key indicators: single-crystal X-ray study; T = 200 K; mean σ(C–C) = 0.004 Å; R factor = 0.064; wR factor = 0.168; data-to-parameter ratio = 27.8.

In the title molecule, $C_{16}H_{11}Cl_3O$, the dihedral angle between the two benzene rings is 33.2 (1)°. The crystal packing is stabilized by C—H···O hydrogen bonds.

Related literature

For the uses of chalcones, see: John *et al.* (2007). For related crystal structures, see: Thiruvalluvar *et al.* (2007*a*,*b*).



Experimental

Crystal data

$C_{16}H_{11}Cl_3O$
M_r = 325.60
Orthorhombic, *Pbca*
a = 7.6432 (4) Å
b = 10.5118 (4) Å
c = 36.2356 (12) Å
V = 2911.3 (2) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.62 mm^{-1}
T = 200 (2) K
0.34 × 0.27 × 0.22 mm

Data collection

Oxford Diffraction Gemini diffractometer
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2007)
T_{min} = 0.818, T_{max} = 0.877
38087 measured reflections
5035 independent reflections
2561 reflections with $I > 2\sigma(I)$
R_{int} = 0.069

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.064
$wR(F^2)$ = 0.168
S = 1.03
5035 reflections
181 parameters
H-atom parameters not refined
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.39 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C13—H13···O^{i}	0.95	2.43	3.372 (3)	169

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2007); cell refinement: *CrysAlis CCD*; data reduction: *CrysAlis RED* (Oxford Diffraction, 2007); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *DIAMOND* (Brandenburg, 1998); software used to prepare material for publication: *PLATON* (Spek, 2003).

RJB acknowledges the NSF–MRI program for funding to purchase the X-ray CCD diffractometer.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LX2037).

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supplementary materials

Acta Cryst. (2008). E**64**, o60 [**doi:10.1107/S1600536807062010**]

(2*E*)-1-(4-Methylphenyl)-3-(2,3,5-trichlorophenyl)prop-2-en-1-one

A. Thiruvalluvar, M. Subramanyam, R. J. Butcher, P. Karegoudar and B. S. Holla

Comment

Chalcones and their derivatives show some interesting physical properties like liquid crystalline nature and nonlinear optical effect(John *et al.*, 2007). Thiruvalluvar *et al.* (2007*a*,b) have reported related crystal structures of chalcones. As a continuation of our work on chalcones, we report here the *x*-ray crystal structure of the title molecule, $C_{16}H_{11}Cl_3O$, Fig.1.

The dihedral angle between the methylphenyl ring and the trichlorophenyl ring is 33.2 (1)°. The crystal packing is stabilized by C13—H13···O^{i} hydrogen bonds (Table 1 & Fig. 2; symmetry code as in Fig. 2)

Experimental

To the mixture of 2,3,5-trichlorobenzaldehyde (21 g, 0.1 mol) and 4-methylacetophenone (14.7 g, 0.11 mol) in methanol (100 ml), 20% of sodium hydroxide (8 g, 0.2 mol) was added at 288–293 K, after stirring for 8 h at 303 K. Solid obtained was filtered, washed with water and dried. The product was recrystallized using ethyl acetate. Yield was 27.6 g(85%).

Refinement

H atoms were positioned geometrically and allowed to ride on their parent atoms, with C—H = 0.93–0.96Å and U_{iso} = 1.2–1.5 times U_{eq}(C).

Figures

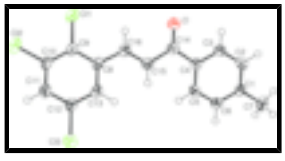

Fig. 1. The molecular structure of the title compound with the atomic numbering and 50% probability displacement ellipsoids. H atoms are shown as small spheres of arbitrary radius.

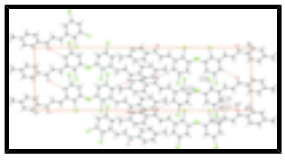

Fig. 2. The packing of the title compound, viewed down the *a* axis. Dashed lines indicate hydrogen bonds. [Symmetry code: (i) −*x* + 1/2, *y* − 1/2, *z*.]

(2E)-1-(4-Methylphenyl)-3-(2,3,5-trichlorophenyl)prop-2-en-1-one

Crystal data

$C_{16}H_{11}Cl_3O$	D_x = 1.486 Mg m^{-3}
M_r = 325.60	Melting point: 467(1) K
Orthorhombic, *Pbca*	Mo *K*α radiation

	λ = 0.71073 Å
Hall symbol: -P 2ac 2ab	Cell parameters from 8272 reflections
a = 7.6432 (4) Å	θ = 4.7–32.4º
b = 10.5118 (4) Å	μ = 0.62 mm^{-1}
c = 36.2356 (12) Å	T = 200 (2) K
V = 2911.3 (2) Å^3	Prism, colourless
Z = 8	0.34 × 0.27 × 0.22 mm
F_{000} = 1328	

Data collection

Oxford Diffraction Gemini diffractometer	5035 independent reflections
Radiation source: fine-focus sealed tube	2561 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.069
T = 200(2) K	θ_{max} = 32.5º
φ and ω scans	θ_{min} = 4.7º
Absorption correction: multi-scan (CrysAlis RED; Oxford Diffraction, 2007)	h = −11→10
T_{min} = 0.818, T_{max} = 0.877	k = −15→15
38087 measured reflections	l = −54→51

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.064	H-atom parameters not refined
$wR(F^2)$ = 0.168	$w = 1/[\sigma^2(F_o^2) + (0.0546P)^2 + 3.6539P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.03	$(\Delta/\sigma)_{max}$ = <0.001
5035 reflections	$\Delta\rho_{max}$ = 0.32 e Å^{-3}
181 parameters	$\Delta\rho_{min}$ = −0.39 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

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 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.04881 (12)	0.50228 (8)	0.19318 (2)	0.0482 (3)
Cl2	−0.10841 (14)	0.32270 (9)	0.25375 (2)	0.0598 (3)
Cl3	−0.06450 (13)	−0.07474 (8)	0.16142 (2)	0.0501 (3)
O	0.2908 (3)	0.52884 (19)	0.06638 (5)	0.0419 (7)
C1	0.1438 (4)	0.3651 (3)	−0.05988 (7)	0.0315 (8)
C2	0.2542 (4)	0.4610 (3)	−0.04685 (8)	0.0358 (9)
C3	0.2707 (4)	0.4818 (3)	−0.00924 (7)	0.0357 (9)
C4	0.1813 (3)	0.4086 (2)	0.01640 (7)	0.0265 (7)
C5	0.0720 (4)	0.3127 (3)	0.00328 (7)	0.0320 (8)
C6	0.0552 (4)	0.2926 (3)	−0.03434 (7)	0.0341 (8)
C7	0.1220 (4)	0.3410 (3)	−0.10050 (8)	0.0448 (10)
C8	0.0644 (3)	0.2917 (3)	0.14942 (7)	0.0294 (8)
C9	0.0205 (4)	0.3419 (3)	0.18411 (7)	0.0326 (8)
C10	−0.0503 (4)	0.2623 (3)	0.21107 (7)	0.0380 (9)
C11	−0.0769 (4)	0.1338 (3)	0.20478 (8)	0.0392 (9)
C12	−0.0301 (4)	0.0868 (3)	0.17038 (7)	0.0366 (9)
C13	0.0398 (4)	0.1617 (3)	0.14318 (7)	0.0328 (8)
C14	0.2066 (4)	0.4357 (3)	0.05641 (7)	0.0299 (8)
C15	0.1296 (4)	0.3471 (3)	0.08389 (7)	0.0350 (9)
C16	0.1330 (4)	0.3734 (3)	0.11973 (7)	0.0321 (8)
H2	0.31806	0.51195	−0.06379	0.0429*
H3	0.34525	0.54801	−0.00081	0.0428*
H5	0.00885	0.26093	0.02016	0.0385*
H6	−0.01996	0.22678	−0.04280	0.0409*
H7A	0.19620	0.26939	−0.10791	0.0671*
H7B	0.15616	0.41717	−0.11433	0.0671*
H7C	−0.00066	0.32072	−0.10576	0.0671*
H11	−0.12525	0.08011	0.22323	0.0470*
H13	0.07160	0.12530	0.12013	0.0393*
H15	0.07694	0.27026	0.07569	0.0420*
H16	0.18438	0.45194	0.12686	0.0385*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0641 (6)	0.0425 (4)	0.0379 (4)	0.0013 (4)	0.0016 (4)	−0.0104 (3)
Cl2	0.0763 (7)	0.0702 (6)	0.0328 (4)	−0.0083 (5)	0.0172 (4)	−0.0141 (4)
Cl3	0.0708 (6)	0.0372 (4)	0.0422 (4)	−0.0073 (4)	0.0041 (4)	0.0009 (3)
O	0.0503 (14)	0.0385 (12)	0.0370 (10)	−0.0092 (10)	−0.0045 (10)	−0.0030 (9)
C1	0.0329 (15)	0.0324 (14)	0.0292 (13)	0.0067 (12)	0.0021 (11)	−0.0019 (11)
C2	0.0360 (16)	0.0353 (15)	0.0361 (14)	−0.0053 (13)	0.0051 (13)	0.0031 (12)
C3	0.0342 (16)	0.0341 (15)	0.0387 (15)	−0.0083 (13)	−0.0009 (13)	0.0007 (12)
C4	0.0259 (13)	0.0239 (13)	0.0296 (12)	0.0032 (11)	−0.0010 (11)	0.0021 (10)
C5	0.0340 (15)	0.0293 (14)	0.0328 (13)	−0.0049 (12)	0.0030 (12)	0.0046 (11)

C6	0.0349 (16)	0.0326 (14)	0.0347 (14)	−0.0054 (13)	−0.0023 (12)	−0.0026 (11)
C7	0.0472 (19)	0.057 (2)	0.0302 (14)	0.0024 (17)	0.0017 (14)	−0.0040 (14)
C8	0.0274 (14)	0.0352 (14)	0.0255 (12)	0.0041 (12)	−0.0020 (11)	−0.0007 (10)
C9	0.0338 (15)	0.0332 (14)	0.0309 (13)	0.0025 (12)	−0.0027 (12)	−0.0054 (11)
C10	0.0411 (17)	0.0489 (18)	0.0241 (12)	−0.0014 (15)	0.0030 (12)	−0.0066 (12)
C11	0.0407 (17)	0.0488 (18)	0.0280 (14)	−0.0054 (15)	0.0003 (12)	0.0007 (12)
C12	0.0435 (18)	0.0376 (16)	0.0286 (13)	−0.0007 (14)	−0.0025 (12)	−0.0012 (11)
C13	0.0360 (15)	0.0373 (15)	0.0250 (12)	0.0038 (13)	−0.0008 (12)	−0.0007 (11)
C14	0.0287 (14)	0.0282 (14)	0.0329 (13)	0.0030 (12)	−0.0029 (11)	−0.0006 (11)
C15	0.0437 (17)	0.0291 (14)	0.0323 (14)	−0.0019 (13)	−0.0021 (13)	−0.0014 (11)
C16	0.0333 (15)	0.0316 (14)	0.0314 (14)	0.0022 (12)	−0.0006 (12)	−0.0022 (11)

Geometric parameters (Å, °)

Cl1—C9	1.731 (3)	C10—C11	1.385 (4)
Cl2—C10	1.730 (3)	C11—C12	1.388 (4)
Cl3—C12	1.749 (3)	C12—C13	1.370 (4)
O—C14	1.226 (4)	C14—C15	1.485 (4)
C1—C2	1.397 (4)	C15—C16	1.328 (4)
C1—C6	1.377 (4)	C2—H2	0.9500
C1—C7	1.503 (4)	C3—H3	0.9500
C2—C3	1.386 (4)	C5—H5	0.9500
C3—C4	1.386 (4)	C6—H6	0.9500
C4—C5	1.393 (4)	C7—H7A	0.9800
C4—C14	1.490 (4)	C7—H7B	0.9800
C5—C6	1.385 (4)	C7—H7C	0.9800
C8—C9	1.404 (4)	C11—H11	0.9500
C8—C13	1.398 (4)	C13—H13	0.9500
C8—C16	1.473 (4)	C15—H15	0.9500
C9—C10	1.396 (4)	C16—H16	0.9500
Cl1···Cl2	3.1344 (12)	C6···H3^{x}	2.9400
Cl2···C9^{i}	3.627 (3)	C13···H7A^{xi}	3.0100
Cl2···Cl1	3.1344 (12)	C13···H15	2.7100
Cl2···Cl3^{ii}	3.5154 (11)	C14···H6^{viii}	2.7400
Cl3···Cl2^{iii}	3.5154 (11)	C15···H5	2.6500
Cl3···C7^{iv}	3.592 (3)	C15···H13	2.7100
Cl1···H16	2.6700	H2···H7B	2.4200
Cl3···H7A^{iv}	2.9900	H3···O	2.4800
O···C13^{v}	3.372 (3)	H3···C3^{vii}	2.9700
O···H3	2.4800	H3···H3^{vii}	2.5700
O···H16	2.4700	H3···C5^{v}	2.8600
O···H13^{v}	2.4300	H3···C6^{v}	2.9400
O···H15^{v}	2.7500	H5···C15	2.6500
C1···C14^{vi}	3.402 (4)	H5···H15	2.0800
C3···C3^{vii}	3.589 (4)	H6···H7C	2.4900
C3···C5^{vi}	3.402 (4)	H6···C4^{xi}	2.8600

C4···C5^{vi}	3.583 (4)	H6···C14^{xi}	2.7400
C4···C4^{vi}	3.576 (3)	H7A···C13^{viii}	3.0100
C5···C4^{vi}	3.583 (4)	H7A···Cl3^{iv}	2.9900
C5···C3^{vi}	3.402 (4)	H7B···H2	2.4200
C6···C14^{vi}	3.578 (4)	H7C···H6	2.4900
C7···Cl3^{iv}	3.592 (3)	H13···C15	2.7100
C7···C13^{viii}	3.548 (4)	H13···H15	2.2200
C9···Cl2^{ix}	3.627 (3)	H13···O^{x}	2.4300
C13···O^{x}	3.372 (3)	H15···C5	2.6600
C13···C7^{xi}	3.548 (4)	H15···C13	2.7100
C14···C6^{vi}	3.578 (4)	H15···H5	2.0800
C14···C1^{vi}	3.402 (4)	H15···H13	2.2200
C3···H3^{vii}	2.9700	H15···O^{x}	2.7500
C4···H6^{viii}	2.8600	H16···Cl1	2.6700
C5···H15	2.6600	H16···O	2.4700
C5···H3^{x}	2.8600		
C2—C1—C6	118.0 (2)	C4—C14—C15	118.8 (2)
C2—C1—C7	121.3 (3)	C14—C15—C16	121.2 (3)
C6—C1—C7	120.7 (3)	C8—C16—C15	125.9 (3)
C1—C2—C3	120.1 (3)	C1—C2—H2	120.00
C2—C3—C4	121.8 (3)	C3—C2—H2	120.00
C3—C4—C5	117.9 (2)	C2—C3—H3	119.00
C3—C4—C14	118.8 (2)	C4—C3—H3	119.00
C5—C4—C14	123.3 (2)	C4—C5—H5	120.00
C4—C5—C6	120.1 (3)	C6—C5—H5	120.00
C1—C6—C5	122.1 (3)	C1—C6—H6	119.00
C9—C8—C13	118.7 (3)	C5—C6—H6	119.00
C9—C8—C16	121.3 (3)	C1—C7—H7A	109.00
C13—C8—C16	120.0 (2)	C1—C7—H7B	109.00
Cl1—C9—C8	120.4 (2)	C1—C7—H7C	109.00
Cl1—C9—C10	120.0 (2)	H7A—C7—H7B	109.00
C8—C9—C10	119.6 (3)	H7A—C7—H7C	109.00
Cl2—C10—C9	120.4 (2)	H7B—C7—H7C	109.00
Cl2—C10—C11	117.9 (2)	C10—C11—H11	121.00
C9—C10—C11	121.8 (3)	C12—C11—H11	121.00
C10—C11—C12	117.2 (3)	C8—C13—H13	120.00
Cl3—C12—C11	118.3 (2)	C12—C13—H13	120.00
Cl3—C12—C13	118.9 (2)	C14—C15—H15	119.00
C11—C12—C13	122.8 (3)	C16—C15—H15	119.00
C8—C13—C12	119.9 (3)	C8—C16—H16	117.00
O—C14—C4	120.5 (2)	C15—C16—H16	117.00
O—C14—C15	120.8 (2)		
C6—C1—C2—C3	−0.7 (5)	C9—C8—C13—C12	1.6 (4)
C7—C1—C2—C3	179.5 (3)	C16—C8—C13—C12	−177.6 (3)
C2—C1—C6—C5	0.4 (5)	C9—C8—C16—C15	−158.6 (3)

C7—C1—C6—C5	−179.8 (3)	C13—C8—C16—C15	20.6 (4)
C1—C2—C3—C4	0.7 (5)	Cl1—C9—C10—Cl2	−0.6 (4)
C2—C3—C4—C5	−0.4 (4)	Cl1—C9—C10—C11	179.3 (2)
C2—C3—C4—C14	179.5 (3)	C8—C9—C10—Cl2	−179.0 (2)
C3—C4—C5—C6	0.1 (4)	C8—C9—C10—C11	0.8 (5)
C14—C4—C5—C6	−179.8 (3)	Cl2—C10—C11—C12	179.8 (2)
C3—C4—C14—O	7.2 (4)	C9—C10—C11—C12	0.0 (5)
C3—C4—C14—C15	−172.1 (3)	C10—C11—C12—Cl3	−179.3 (2)
C5—C4—C14—O	−172.9 (3)	C10—C11—C12—C13	0.0 (5)
C5—C4—C14—C15	7.8 (4)	Cl3—C12—C13—C8	178.4 (2)
C4—C5—C6—C1	−0.1 (5)	C11—C12—C13—C8	−0.9 (5)
C13—C8—C9—Cl1	179.9 (2)	O—C14—C15—C16	7.5 (5)
C13—C8—C9—C10	−1.6 (4)	C4—C14—C15—C16	−173.2 (3)
C16—C8—C9—Cl1	−0.9 (4)	C14—C15—C16—C8	−178.6 (3)
C16—C8—C9—C10	177.6 (3)		

Symmetry codes: (i) *x*−1/2, *y*, −*z*+1/2; (ii) −*x*, *y*+1/2, −*z*+1/2; (iii) −*x*, *y*−1/2, −*z*+1/2; (iv) −*x*, −*y*, −*z*; (v) −*x*+1/2, *y*+1/2, *z*; (vi) −*x*, −*y*+1, −*z*; (vii) −*x*+1, −*y*+1, −*z*; (viii) *x*+1/2, −*y*+1/2, −*z*; (ix) *x*+1/2, *y*, −*z*+1/2; (x) −*x*+1/2, *y*−1/2, *z*; (xi) *x*−1/2, −*y*+1/2, −*z*.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C13—H13···O^{x}	0.95	2.43	3.372 (3)	169

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

Fig. 1

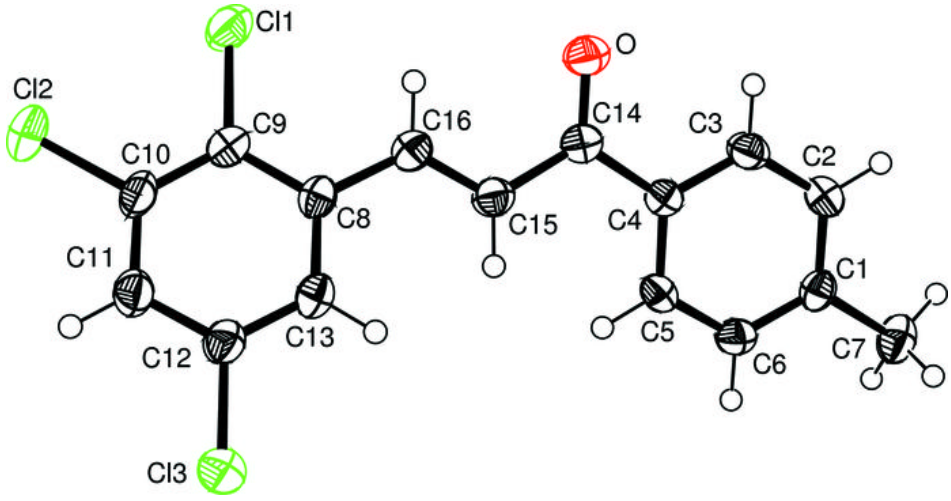

Fig. 2