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2-Chloro-1-(3-methyl-3-phenylcyclobutyl)ethanone

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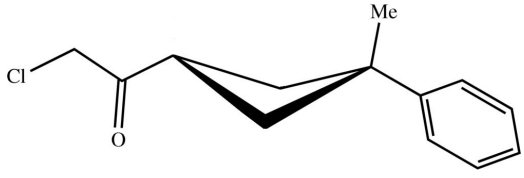
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.030; wR factor = 0.071; data-to-parameter ratio = 20.0.

The title compound, $C_{13}H_{15}ClO$, has a nonplanar conformation. The phenyl ring and chloroacetaldehyde group are in *cis* positions. Molecules are linked to one another by intermolecular C—H···O interactions, forming a $C(4)$ chain running parallel to the [001] direction. The cyclobutane ring is puckered, with a dihedral angle of 26.81 (13)°.

Related literature

For related literature, see: Akhmedov *et al.* (1991); Allen *et al.* (1987); Bernstein *et al.* (1995); Dehmlow & Schmidt (1990); Demir *et al.* (2006); Dinçer *et al.* (2004); Gompper & Christmann (1959); Roger *et al.* (1977); Özdemir *et al.* (2004); Çukurovalı *et al.* (2002).



Experimental

Crystal data

$C_{13}H_{15}ClO$
M_r = 222.70
Orthorhombic, $Pca2_1$
a = 9.4980 (9) Å
b = 15.6393 (11) Å
c = 7.8578 (7) Å
V = 1167.21 (17) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.30 mm^{-1}
T = 100 K
0.61 × 0.40 × 0.22 mm

Data collection

Stoe IPDS II diffractometer
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
T_{min} = 0.885, T_{max} = 0.944
6736 measured reflections
2743 independent reflections
2435 reflections with $I > 2\sigma(I)$
R_{int} = 0.035

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.071
S = 1.05
2743 reflections
137 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}
Absolute structure: Flack (1983), 1251 Friedel pairs
Flack parameter: −0.01 (5)

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

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1A···O1^{i}	0.97	2.46	3.244 (2)	137

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

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON* (Spek, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GW2017).

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supplementary materials

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2-Chloro-1-(3-methyl-3-phenylcyclobutyl)ethanone

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Comment

It has been shown that 3-substituted cyclobutane carboxylic acid deriatives have antidepressant activities and liquid crystal properties (Roger *et al.*, 1977; Dehmlow & Schmidt, 1990;). Substituted α-haloketones, like title compound, are used for different purposes, especially in the synthesis of heterocyclic compounds (Gompper & Christmann, 1959; Çukurovalı *et al.*, 2002). The extensive synthetic possibilies of this compound, due to the presence of active reaction sites, hold promise for the preparation of new heterocyclic chemicals. As a continuation of our investigations on the cyclobutane derivatives, a crystal structure determination of the title compound, (I), has been undertaken and the results are presented here.

In the crystal structure, the phenyl ring and chloroacetaldehyde group are in *cis* position with respect to the cyclobutane ring. Although close to being planar, the cyclobutane ring in (I) is more puckered than those in the literature [11.55 (3)°,Özdemir *et al.*, 2004; 19.8 (3)°, Dinçer *et al.*, 2004]. The C4/C3/C6 plane forms a dihedral angle of 26.81 (13)° with the C4/C5/C6 plane. However, the bond lengths and angles in the four-membered ring are normal (Allen *et al.*, 1987). The C—Cl and C═O bond distances are 1.7692 (17) and 1.211 (2) Å, repectively, and these values are significantly shorter than those in the literature [1.807 (12) and 1.187 (16) Å, respectively; Demir *et al.*, 2006].

In the crystal structure of (I), atom C1 in the molecule at (*x*, *y*, *z*) acts as hydrogen-bond donor to the O atom in the molecule at (−*x* + 1/2, *y*, *z* + 1/2), forming a C(4) (Bernstein *et al.*, 1995) chain running parallel to the [001] direction and generated by the c-glide plane at *x* = 1/4 (Fig. 2). There are no other significant interactions in the crystal structure of (I).

Experimental

The synthesis of the title compound was realised according to the literature method (Akhmedov *et al.*, 1991) with some modifications as given in the reaction sequence. The crystals which is suitable for X-ray analysis was obtained by the crystallization from n-hexane (yield 72%; m.p. 326 K). IR (ν, cm^{-1}): 1724 (C═O), 732 (–CH_2—Cl), 3049–3024 (Aromatics), 2980–2864 (Aliphatics); ^{1}H NMR ($CDCl_3$, p.p.m.): δ 1.54 (s, 3H, CH_3 on cyclobutane), 2.37 (m, 2H, –CH_2– in cyclobutane), 2.62 (m, 2H, –CH_2– in cyclobutane), 3.67 (q, j = 9.1 Hz, 1H, >CH–), 4.09 (s, 2H, –CH_2–), 7.13–7.34 (m, 5H, aromatics); ^{13}C NMR ($CDCl_6$, p.p.m.): δ 47.02, 203.66, 37.51, 37.05, 38.83, 30.56, 151.03, 124.78, 128.58, 125.94.

Refinement

H atoms were positioned geometrically and treated using a riding model, fixing the bond lengths at 0.93, 0.96, 0.97 and 0.98 Å for aromatic, methyl, methylene and methine H atoms, respectively. The displacement parameters of the H atoms were constrained to be U_{iso}(H)= 1.2U_{eq}(1.5U_{eq} for methyl) of the carrier atom. Refinement of the absolute structure parameter (Flack, 1983) yielded a value of −0.01 (5).

Figures

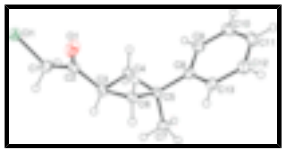


Fig. 1. An *ORTEP-3* (Farrugia, 1997) drawing of (I), showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 40% probability level.

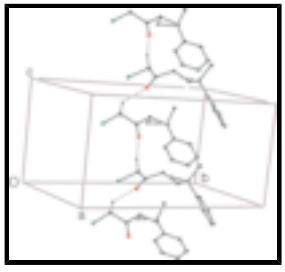


Fig. 2. Part of the crystal structure of (I), showing a C(4) chain along [001]. For the sake of clarity, H atoms bonded to C atoms not involved in the motif shown have been omitted.

Fig. 3. Reaction scheme for the preparation of (I).

2-Chloro-1-(3-methyl-3-phenylcyclobutyl)ethanone

Crystal data

$C_{13}H_{15}ClO$	F_{000} = 472
M_r = 222.70	D_x = 1.267 Mg m^{-3}
Orthorhombic, $Pca2_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2c -2ac	Cell parameters from 6312 reflections
a = 9.4980 (9) Å	θ = 2.5–28.0º
b = 15.6393 (11) Å	μ = 0.30 mm^{-1}
c = 7.8578 (7) Å	T = 100 K
V = 1167.21 (17) Å^3	Prism, colorless
Z = 4	0.61 × 0.40 × 0.22 mm

Data collection

Stoe IPDS II diffractometer	2743 independent reflections
Radiation source: sealed X-ray tube, 12 x 0.4 mm long-fine focus	2435 reflections with $I > 2\sigma(I)$
Monochromator: plane graphite	R_{int} = 0.035
Detector resolution: 6.67 pixels mm^{-1}	θ_{max} = 27.9º
T = 100 K	θ_{min} = 2.5º
ω scans	h = −12→10
Absorption correction: integration (X-RED32; Stoe & Cie, 2002)	k = −20→20
T_{min} = 0.885, T_{max} = 0.944	l = −10→10
6736 measured reflections	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.030$	$w = 1/[\sigma^2(F_o^2) + (0.038P)^2 + 0.0689P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.071$	$(\Delta/\sigma)_{max} = 0.001$
$S = 1.05$	$\Delta\rho_{max} = 0.20$ e Å^{-3}
2743 reflections	$\Delta\rho_{min} = -0.19$ e Å^{-3}
137 parameters	Extinction correction: SHELXL97, $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
1 restraint	Extinction coefficient: 0.0052 (14)
Primary atom site location: structure-invariant direct methods	Absolute structure: Flack (1983), 1251 Friedel pairs
Secondary atom site location: difference Fourier map	Flack parameter: −0.01 (5)

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.41536 (4)	0.37951 (2)	0.10882 (5)	0.03443 (11)
O1	0.27620 (14)	0.54032 (7)	0.00821 (18)	0.0385 (3)
C1	0.43719 (18)	0.47994 (10)	0.2090 (2)	0.0304 (3)
H1A	0.4059	0.4754	0.3262	0.036*
H1B	0.5366	0.4940	0.2106	0.036*
C2	0.35801 (15)	0.55188 (9)	0.1242 (2)	0.0243 (3)
C3	0.39456 (16)	0.63806 (9)	0.19372 (19)	0.0234 (3)
H3	0.4005	0.6363	0.3182	0.028*
C4	0.52987 (14)	0.67903 (9)	0.1165 (2)	0.0253 (3)
H4A	0.6116	0.6759	0.1903	0.030*
H4B	0.5521	0.6591	0.0027	0.030*
C5	0.45381 (14)	0.76651 (9)	0.1207 (2)	0.0228 (3)
C6	0.31190 (15)	0.71696 (9)	0.13509 (19)	0.0227 (3)
H6A	0.2485	0.7397	0.2207	0.027*
H6B	0.2639	0.7093	0.0272	0.027*

C7	0.48815 (19)	0.81500 (11)	0.2856 (2)	0.0278 (4)
H7A	0.4785	0.7771	0.3810	0.042*
H7B	0.4244	0.8622	0.2984	0.042*
H7C	0.5830	0.8360	0.2806	0.042*
C8	0.46899 (18)	0.82486 (10)	−0.03045 (19)	0.0231 (3)
C9	0.58479 (18)	0.82191 (11)	−0.1395 (2)	0.0285 (3)
H9	0.6562	0.7825	−0.1200	0.034*
C10	0.5941 (2)	0.87746 (12)	−0.2769 (2)	0.0346 (4)
H10	0.6713	0.8743	−0.3494	0.041*
C11	0.4903 (2)	0.93721 (11)	−0.3071 (2)	0.0356 (4)
H11	0.4973	0.9743	−0.3992	0.043*
C12	0.3753 (2)	0.94139 (11)	−0.1987 (2)	0.0330 (4)
H12	0.3051	0.9818	−0.2175	0.040*
C13	0.36494 (19)	0.88556 (10)	−0.06253 (19)	0.0263 (3)
H13	0.2870	0.8886	0.0089	0.032*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0380 (2)	0.02533 (17)	0.0400 (2)	0.00003 (15)	0.0113 (2)	−0.0010 (2)
O1	0.0412 (7)	0.0304 (6)	0.0439 (7)	−0.0022 (5)	−0.0176 (6)	−0.0011 (5)
C1	0.0332 (8)	0.0289 (8)	0.0290 (7)	0.0035 (7)	0.0012 (7)	−0.0001 (7)
C2	0.0226 (6)	0.0268 (7)	0.0234 (6)	−0.0009 (5)	0.0016 (6)	0.0011 (7)
C3	0.0235 (7)	0.0248 (8)	0.0219 (7)	−0.0003 (6)	−0.0007 (6)	0.0025 (6)
C4	0.0211 (6)	0.0280 (7)	0.0269 (6)	0.0002 (5)	0.0015 (8)	0.0038 (8)
C5	0.0214 (6)	0.0259 (7)	0.0210 (6)	−0.0012 (5)	0.0005 (7)	0.0010 (7)
C6	0.0215 (6)	0.0258 (7)	0.0208 (7)	0.0004 (5)	−0.0001 (6)	0.0006 (6)
C7	0.0314 (9)	0.0302 (9)	0.0218 (7)	−0.0007 (7)	−0.0012 (7)	−0.0009 (6)
C8	0.0240 (8)	0.0239 (7)	0.0213 (7)	−0.0054 (6)	−0.0012 (6)	−0.0032 (6)
C9	0.0270 (8)	0.0316 (8)	0.0269 (7)	−0.0060 (7)	0.0016 (6)	−0.0037 (7)
C10	0.0377 (10)	0.0407 (9)	0.0253 (7)	−0.0155 (8)	0.0036 (7)	−0.0003 (7)
C11	0.0491 (11)	0.0310 (9)	0.0268 (8)	−0.0157 (8)	−0.0050 (8)	0.0043 (7)
C12	0.0428 (10)	0.0271 (8)	0.0290 (8)	−0.0048 (7)	−0.0109 (7)	0.0009 (7)
C13	0.0298 (8)	0.0245 (8)	0.0246 (7)	−0.0029 (6)	−0.0014 (6)	−0.0003 (6)

Geometric parameters (Å, °)

Cl1—C1	1.7692 (17)	C6—H6B	0.9700
O1—C2	1.211 (2)	C7—H7A	0.9600
C1—C2	1.509 (2)	C7—H7B	0.9600
C1—H1A	0.9700	C7—H7C	0.9600
C1—H1B	0.9700	C8—C13	1.393 (2)
C2—C3	1.495 (2)	C8—C9	1.395 (2)
C3—C6	1.5334 (19)	C9—C10	1.388 (2)
C3—C4	1.559 (2)	C9—H9	0.9300
C3—H3	0.9800	C10—C11	1.379 (3)
C4—C5	1.5475 (19)	C10—H10	0.9300
C4—H4A	0.9700	C11—C12	1.386 (3)
C4—H4B	0.9700	C11—H11	0.9300

C5—C8	1.505 (2)	C12—C13	1.385 (2)
C5—C7	1.536 (2)	C12—H12	0.9300
C5—C6	1.5589 (19)	C13—H13	0.9300
C6—H6A	0.9700		
C2—C1—Cl1	114.01 (12)	C5—C6—H6A	113.8
C2—C1—H1A	108.7	C3—C6—H6B	113.8
Cl1—C1—H1A	108.7	C5—C6—H6B	113.8
C2—C1—H1B	108.7	H6A—C6—H6B	111.1
Cl1—C1—H1B	108.7	C5—C7—H7A	109.5
H1A—C1—H1B	107.6	C5—C7—H7B	109.5
O1—C2—C3	123.96 (13)	H7A—C7—H7B	109.5
O1—C2—C1	122.75 (14)	C5—C7—H7C	109.5
C3—C2—C1	113.26 (14)	H7A—C7—H7C	109.5
C2—C3—C6	119.78 (13)	H7B—C7—H7C	109.5
C2—C3—C4	114.81 (13)	C13—C8—C9	118.07 (15)
C6—C3—C4	88.53 (11)	C13—C8—C5	119.21 (14)
C2—C3—H3	110.6	C9—C8—C5	122.71 (15)
C6—C3—H3	110.6	C10—C9—C8	120.48 (17)
C4—C3—H3	110.6	C10—C9—H9	119.8
C5—C4—C3	88.29 (10)	C8—C9—H9	119.8
C5—C4—H4A	113.9	C11—C10—C9	120.80 (17)
C3—C4—H4A	113.9	C11—C10—H10	119.6
C5—C4—H4B	113.9	C9—C10—H10	119.6
C3—C4—H4B	113.9	C10—C11—C12	119.31 (16)
H4A—C4—H4B	111.1	C10—C11—H11	120.3
C8—C5—C7	110.25 (11)	C12—C11—H11	120.3
C8—C5—C4	118.32 (15)	C13—C12—C11	120.08 (18)
C7—C5—C4	110.82 (14)	C13—C12—H12	120.0
C8—C5—C6	116.20 (13)	C11—C12—H12	120.0
C7—C5—C6	111.58 (13)	C12—C13—C8	121.26 (16)
C4—C5—C6	88.04 (10)	C12—C13—H13	119.4
C3—C6—C5	88.80 (10)	C8—C13—H13	119.4
C3—C6—H6A	113.8		
Cl1—C1—C2—O1	6.8 (2)	C4—C5—C6—C3	19.01 (13)
Cl1—C1—C2—C3	−171.24 (11)	C7—C5—C8—C13	−75.72 (17)
O1—C2—C3—C6	8.6 (2)	C4—C5—C8—C13	155.30 (13)
C1—C2—C3—C6	−173.33 (13)	C6—C5—C8—C13	52.5 (2)
O1—C2—C3—C4	−94.80 (19)	C7—C5—C8—C9	103.18 (17)
C1—C2—C3—C4	83.25 (17)	C4—C5—C8—C9	−25.8 (2)
C2—C3—C4—C5	141.38 (13)	C6—C5—C8—C9	−128.62 (16)
C6—C3—C4—C5	19.00 (12)	C13—C8—C9—C10	−0.8 (2)
C3—C4—C5—C8	−137.59 (13)	C5—C8—C9—C10	−179.69 (15)
C3—C4—C5—C7	93.69 (14)	C8—C9—C10—C11	0.8 (3)
C3—C4—C5—C6	−18.69 (12)	C9—C10—C11—C12	−0.1 (3)
C2—C3—C6—C5	−136.82 (14)	C10—C11—C12—C13	−0.5 (3)
C4—C3—C6—C5	−18.86 (12)	C11—C12—C13—C8	0.5 (2)
C8—C5—C6—C3	139.81 (13)	C9—C8—C13—C12	0.2 (2)
C7—C5—C6—C3	−92.64 (13)	C5—C8—C13—C12	179.10 (14)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1A···O1^{i}	0.97	2.46	3.244 (2)	137

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

Fig. 1

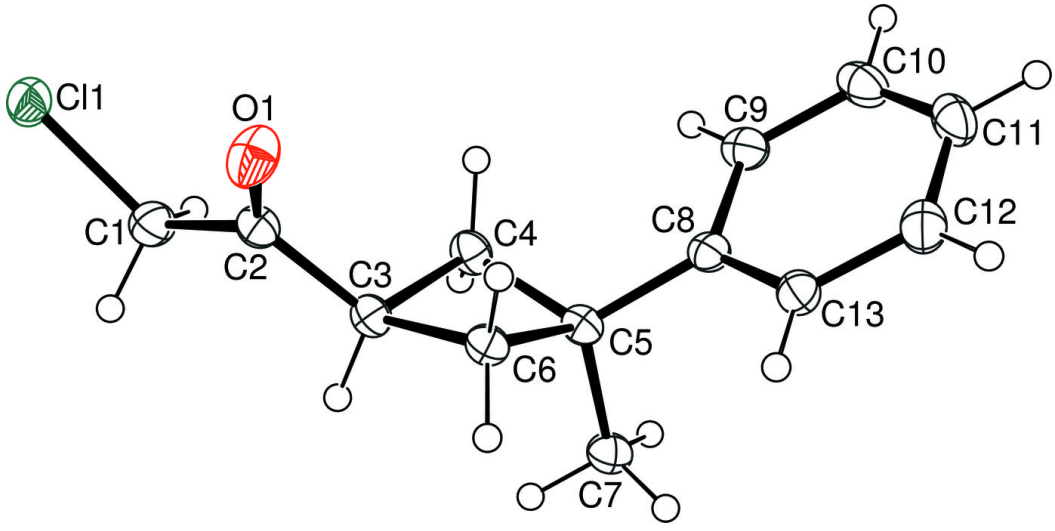


Fig. 2

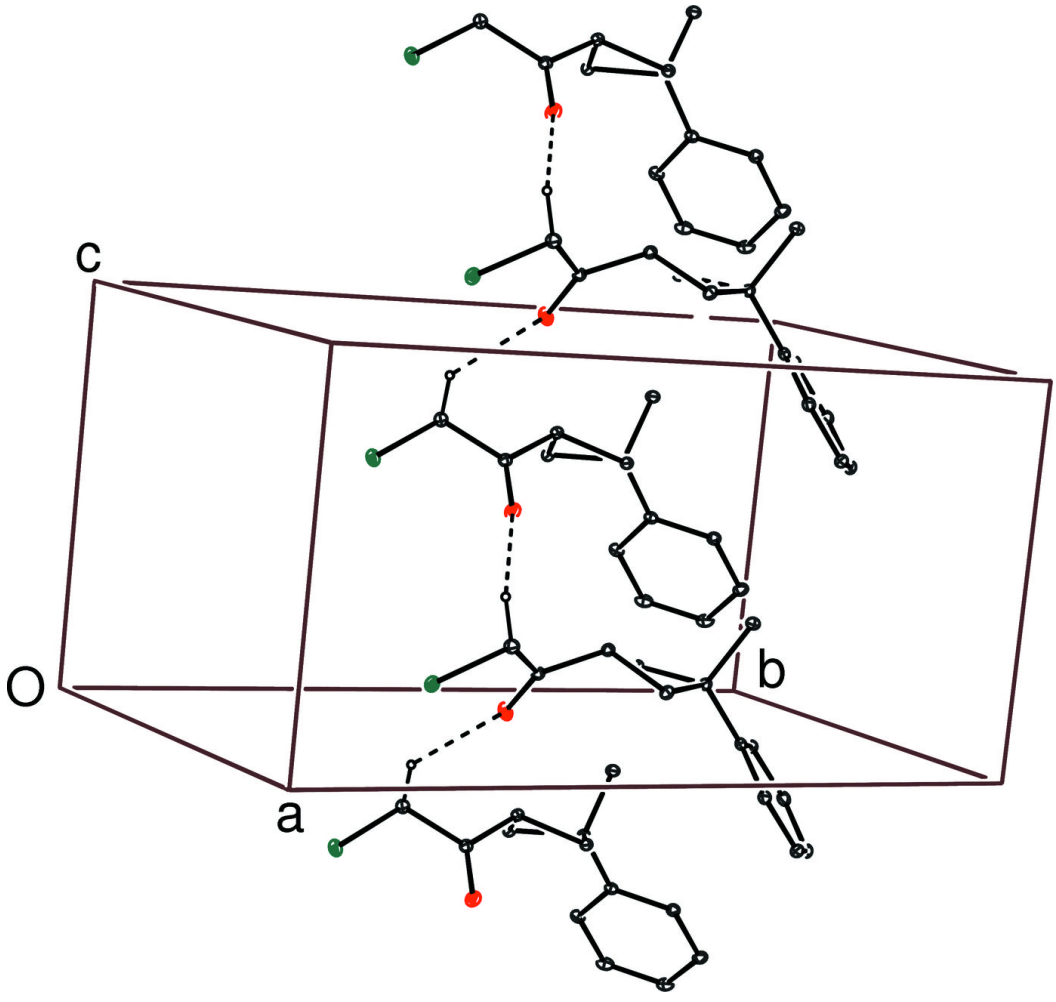
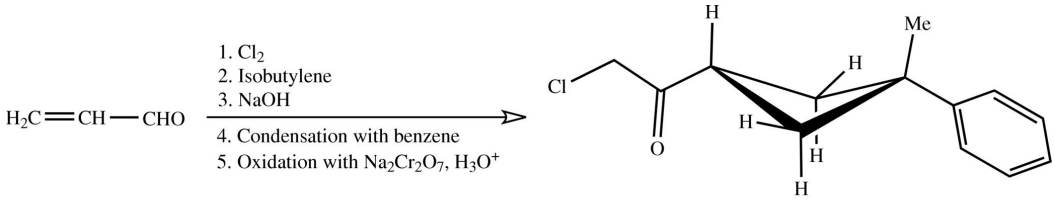


Fig. 3


H2C═CH—CHO
1. Cl2
2. Isobutylene
3. NaOH
4. Condensation with benzene
5. Oxidation with Na2Cr2O7, H3O+
Cl
O
H
H
H
H
Me

(I)