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5-Chloro-1-phenylpentan-1-one

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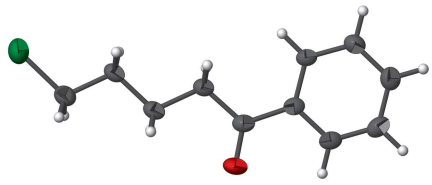
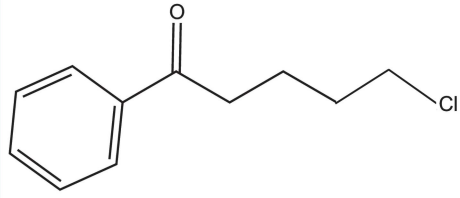
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{11}H_{13}ClO$, which is used as a starting material for the synthesis of some materials with possible medical applications, the molecular skeleton is slightly curved, with the dihedral angle of 4.7 (1)° between the mean planes of the chlorobutane and benzaldehyde fragments. In the crystal, weak C—H···O hydrogen bonds link the molecules into chains running along the [201] direction, and weak C—H···π interactions link these chains into layers parallel to the *ac* plane.

3D view



Chemical scheme



Structure description

The synthesis of the title compound, (I), was previously described in the preparation of building blocks for the synthesis of ω-phenylalkylpyrimidines and purines (Komissarov *et al.* 2010; Gromov *et al.* 1999). Another application is the use as coupling component for the synthesis of acyclic triaryl olefins, which were described as selective cyclooxygenase-2 inhibitors (Abdellatif *et al.*, 2010, 2011; Uddin *et al.* 2004*a*,*b*) and selective estrogen receptor modulators (Chen *et al.*, 2012; Shiina *et al.* 2007). In our study, (I) was obtained as product of the reaction of 5-chlorovaleryl chloride with benzene in the presence of aluminium chloride *via* Friedel–Crafts acylation.

The molecule of (I) (Fig. 1) is nearly flat, but not completely planar - the two mean planes formed by the chlorobutane (Cl1/C1–C4) fragment and by the rest of non-H atoms, respectively, are inclined to each other by 4.7 (1)°. In the crystal, weak C—H···O hydrogen bonds (Table 1) link the molecules into chains running along [201]. Weak C—H···π interactions (Table 1) link these chains into layers parallel to the *ac* plane. Van der Waals forces stabilize further the crystal packing (Fig. 2).

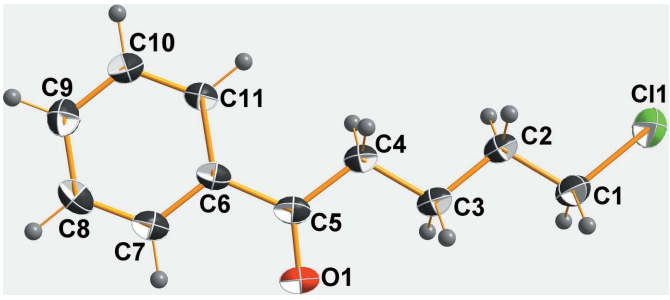


Figure 1
The molecular structure of (I), with atom labels and 50% probability displacement ellipsoids for non-H atoms.

Synthesis and crystallization

To an ice-cold suspension of aluminium chloride (1.76 g, 13.23 mmol) in 10 ml chloroform, 1.86 ml 5-chlorovaleryl chloride (14.40 mmol) and 0.99 ml benzene (11.54 mmol) were added under an argon atmosphere. After stirring for 1.5 h at room temperature, the reaction mixture was poured into a mixture of ice and water (15 ml). The organic layer was separated and washed three times with water (10 ml). The separated aqueous solution was extracted three times with $CHCl_3$ (15 ml), the combined organic layer was dried over Na_2SO_4 and the solvent was removed under vacuum. After purification with semi-preparative HPLC (ProStar; Varian; microsorb 60, C18; water/acetonitrile + 0.1% TFA; 35 min: 5/5, *v*/*v*; 7 ml min^{-1}). 5-Chloro-1-phenylpentan-1-one was obtained as a yellow solid in 91.6% yield. Colorless crystals were obtained by crystallization from acetonitrile/water + 0.1% TFA after chromatographic separation.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Acknowledgements

Help with the X-ray diffraction experiments by Dr A. Villinger and Dipl.-Chem. P. Thiele (University of Rostock) is gratefully acknowledged.

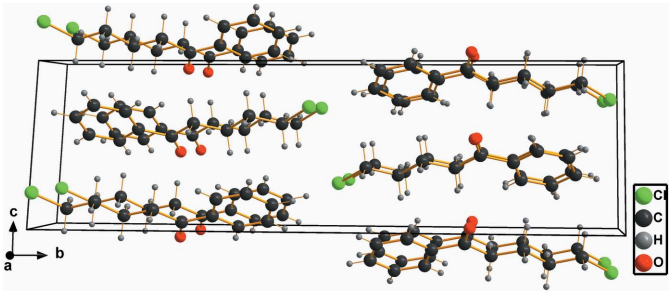


Figure 2
A portion of the crystal packing, viewed approximately along the *a* axis.

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

Cg is the centroid of the C6–C11 benzene ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C11—H11···O1^{i}	0.95	2.51	3.422 (2)	169
C2—H2*A*···*Cg*^{ii}	0.99	2.80	3.676 (2)	148

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{11}H_{13}ClO$
M_r	196.66
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	173
a, *b*, *c* (Å)	5.2434 (4), 25.902 (2), 7.7027 (6)
β (°)	104.756 (5)
V (Å^3)	1011.6 (1)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.33
Crystal size (mm)	0.32 × 0.27 × 0.26
Data collection	
Diffractometer	Bruker APEXII CCD diffractometer
Absorption correction	Multi-scan (*SADABS*; Bruker, 2007)
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	9116, 2343, 1411
R_{int}	0.042
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.652
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.043, 0.108, 1.01
No. of reflections	2343
No. of parameters	119
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.22, −0.31

Computer programs: *APEX2* (Bruker, 2007), *SAINT* (Bruker, 2007), *SHELXS97* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *SHELXTL* (Sheldrick, 2008).

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full crystallographic data

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Crystal data

$C_{11}H_{13}ClO$
M_r = 196.66
Monoclinic, $P2_1/c$
a = 5.2434 (4) Å
b = 25.902 (2) Å
c = 7.7027 (6) Å
β = 104.756 (5)°
V = 1011.6 (1) Å^3
Z = 4
$F(000)$ = 416
D_x = 1.291 Mg m^{-3}
Melting point = 320–321 K
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1422 reflections
θ = 2.9–22.7°
μ = 0.33 mm^{-1}
T = 173 K
Irregular block, colorless
0.32 × 0.27 × 0.26 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2007)
9116 measured reflections
2343 independent reflections
1411 reflections with $I > 2\sigma(I)$
R_{int} = 0.042
θ_{max} = 27.6°, θ_{min} = 1.6°
h = −6→5
k = −33→29
l = −10→10

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.108
S = 1.01
2343 reflections
119 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0362P)^2 + 0.2948P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}
Extinction correction: *SHELXL2014* (Sheldrick, 2015), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0021 (13)

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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.30681 (13)	0.47935 (2)	0.72353 (9)	0.0622 (2)
C1	0.0779 (4)	0.42960 (8)	0.6269 (3)	0.0403 (5)
H1A	−0.0813	0.4326	0.6731	0.048*
H1B	0.0231	0.4340	0.4949	0.048*
C2	0.1990 (4)	0.37676 (7)	0.6714 (2)	0.0330 (5)
H2A	0.2507	0.3722	0.8033	0.040*
H2B	0.3602	0.3741	0.6274	0.040*
C3	0.0082 (4)	0.33416 (8)	0.5870 (2)	0.0335 (5)
H3A	−0.0485	0.3395	0.4555	0.040*
H3B	−0.1503	0.3360	0.6342	0.040*
C4	0.1323 (4)	0.28088 (7)	0.6257 (2)	0.0311 (5)
H4A	0.2869	0.2788	0.5744	0.037*
H4B	0.1961	0.2763	0.7573	0.037*
C5	−0.0547 (4)	0.23775 (8)	0.5504 (2)	0.0322 (5)
O1	−0.2751 (3)	0.24672 (6)	0.4561 (2)	0.0515 (4)
C6	0.0313 (4)	0.18322 (8)	0.5912 (2)	0.0286 (4)
C7	−0.1480 (4)	0.14405 (8)	0.5232 (2)	0.0342 (5)
H7	−0.3185	0.1527	0.4514	0.041*
C8	−0.0816 (4)	0.09289 (8)	0.5587 (3)	0.0395 (5)
H8	−0.2066	0.0665	0.5128	0.047*
C9	0.1679 (4)	0.07998 (8)	0.6614 (3)	0.0386 (5)
H9	0.2143	0.0448	0.6855	0.046*
C10	0.3486 (4)	0.11831 (8)	0.7284 (3)	0.0352 (5)
H10	0.5196	0.1093	0.7983	0.042*
C11	0.2830 (4)	0.16993 (8)	0.6946 (2)	0.0307 (5)
H11	0.4085	0.1961	0.7416	0.037*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0628 (5)	0.0355 (4)	0.0776 (5)	0.0012 (3)	−0.0018 (3)	0.0025 (3)
C1	0.0395 (12)	0.0400 (13)	0.0373 (11)	0.0058 (10)	0.0023 (9)	0.0023 (9)
C2	0.0318 (11)	0.0358 (12)	0.0297 (10)	0.0081 (9)	0.0044 (8)	0.0026 (9)
C3	0.0306 (11)	0.0396 (12)	0.0283 (10)	0.0074 (9)	0.0040 (8)	0.0010 (9)
C4	0.0279 (10)	0.0366 (12)	0.0276 (9)	0.0028 (9)	0.0045 (8)	0.0003 (8)
C5	0.0270 (11)	0.0430 (13)	0.0257 (10)	0.0004 (9)	0.0049 (8)	−0.0009 (9)
O1	0.0338 (9)	0.0503 (10)	0.0572 (10)	0.0034 (8)	−0.0125 (7)	0.0044 (8)
C6	0.0267 (10)	0.0379 (12)	0.0214 (9)	−0.0013 (9)	0.0064 (8)	−0.0006 (8)
C7	0.0283 (11)	0.0438 (13)	0.0294 (10)	−0.0034 (10)	0.0051 (8)	−0.0007 (9)

C8	0.0358 (12)	0.0437 (14)	0.0397 (11)	−0.0112 (10)	0.0106 (10)	−0.0052 (10)
C9	0.0423 (13)	0.0343 (13)	0.0418 (12)	0.0003 (10)	0.0153 (10)	0.0009 (10)
C10	0.0306 (11)	0.0391 (13)	0.0354 (11)	0.0046 (10)	0.0076 (9)	0.0020 (9)
C11	0.0248 (10)	0.0371 (12)	0.0287 (10)	−0.0013 (9)	0.0042 (8)	−0.0021 (8)

Geometric parameters (Å, º)

Cl1—C1	1.789 (2)	C5—O1	1.221 (2)
C1—C2	1.511 (3)	C5—C6	1.491 (3)
C1—H1A	0.9900	C6—C7	1.392 (3)
C1—H1B	0.9900	C6—C11	1.400 (2)
C2—C3	1.520 (3)	C7—C8	1.380 (3)
C2—H2A	0.9900	C7—H7	0.9500
C2—H2B	0.9900	C8—C9	1.386 (3)
C3—C4	1.522 (3)	C8—H8	0.9500
C3—H3A	0.9900	C9—C10	1.379 (3)
C3—H3B	0.9900	C9—H9	0.9500
C4—C5	1.502 (3)	C10—C11	1.389 (3)
C4—H4A	0.9900	C10—H10	0.9500
C4—H4B	0.9900	C11—H11	0.9500
C2—C1—Cl1	111.07 (14)	H4A—C4—H4B	107.7
C2—C1—H1A	109.4	O1—C5—C6	119.69 (18)
Cl1—C1—H1A	109.4	O1—C5—C4	120.95 (19)
C2—C1—H1B	109.4	C6—C5—C4	119.36 (16)
Cl1—C1—H1B	109.4	C7—C6—C11	118.92 (18)
H1A—C1—H1B	108.0	C7—C6—C5	118.17 (17)
C1—C2—C3	111.59 (16)	C11—C6—C5	122.92 (17)
C1—C2—H2A	109.3	C8—C7—C6	120.89 (18)
C3—C2—H2A	109.3	C8—C7—H7	119.6
C1—C2—H2B	109.3	C6—C7—H7	119.6
C3—C2—H2B	109.3	C7—C8—C9	119.90 (19)
H2A—C2—H2B	108.0	C7—C8—H8	120.0
C2—C3—C4	111.86 (15)	C9—C8—H8	120.0
C2—C3—H3A	109.2	C10—C9—C8	119.9 (2)
C4—C3—H3A	109.2	C10—C9—H9	120.0
C2—C3—H3B	109.2	C8—C9—H9	120.0
C4—C3—H3B	109.2	C9—C10—C11	120.62 (19)
H3A—C3—H3B	107.9	C9—C10—H10	119.7
C5—C4—C3	113.21 (15)	C11—C10—H10	119.7
C5—C4—H4A	108.9	C10—C11—C6	119.74 (18)
C3—C4—H4A	108.9	C10—C11—H11	120.1
C5—C4—H4B	108.9	C6—C11—H11	120.1
C3—C4—H4B	108.9		

Hydrogen-bond geometry (Å, º)

Cg is the centroid of the C6–C11 benzene ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C11—H11···O1^{i}	0.95	2.51	3.422 (2)	169
C2—H2*A*···*Cg*^{ii}	0.99	2.80	3.676 (2)	148

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