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3,4-Dichlorohypnone

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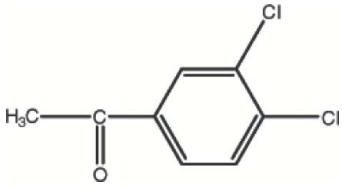
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.002 Å; R factor = 0.033; wR factor = 0.094; data-to-parameter ratio = 16.2.

The title compound [systematic name: 1-(3,4-dichlorophenyl)-ethanone], $C_8H_6Cl_2O$, was prepared by the reaction of aluminium trichloride with *o*-dichlorobenzene and acetyl chloride. There is π stacking in the structure.

Related literature

For related literature, see: Evans & Trotter (1988); Haisa *et al.* (1976); Hatanaka *et al.* (1989).



Experimental

Crystal data

$C_8H_6Cl_2O$
M_r = 189.03
Monoclinic, $P2_1/c$
a = 7.2878 (15) Å
b = 10.597 (2) Å
c = 10.610 (2) Å
β = 102.717 (3)°
V = 799.3 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.74 mm^{-1}
T = 294 (2) K
0.20 × 0.18 × 0.16 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.866, T_{max} = 0.890
4489 measured reflections
1637 independent reflections
1449 reflections with $I > 2\sigma(I)$
R_{int} = 0.018

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.094
S = 1.05
1637 reflections
101 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

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

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2284).

References

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Haisa, M., Kashino, S., Yuasa, T. & Akigawa, K. (1976). *Acta Cryst.* B**32**, 1326–1328.
Hatanaka, Y., Fukushima, S. & Hiyama, T. (1989). *Chem. Lett.* **18**, 1711–1714.
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supplementary materials

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3,4-Dichlorohypnone

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Comment

Hypnone derivate is an important kind of intermediate in industry of fine chemicals. They are extensively used as starting material in the synthetic of pesticide and medication (Hatanaka *et al.*, 1989). Because of the low melt point of them, the crysstal of hypnone derivate are rare. As part of our search for new hypnone derivate compounds we synthesized the title compound (I), and describe its structure here.

The distance of C7—O1 of 1.201 (2) Å is shorter than the C—O distance of 1.215 Å reported by Haisa *et al.* (1976). The C4—Cl2 and C3—Cl1 distances of 1.7063 (17) and 1.7072 (17) Å, respectively, are similar to the C—Cl distance of 1.747Å reported by Evans *et al.* (1988).

Experimental

A mixture of the *o*-dichloro benzene (0.1 mol), acetyl chloride (0.1 mol) and aluminium trichloride (0.15 mol), was stirred in refluxing trichloromethane (30 ml) for 5 h to afford the title compound (0.085 mol, yield 85%). Single crystals of the title compound (I) suitable for X-ray measurements were obtained by recrystallization from EtOH at room temperature.

Refinement

H atoms were fixed geometrically and allowed to ride on their attached atoms, with C—H = 0.93 - 0.96 Å , and U_{iso}=1.2-1.5U_{eq}.

Figures

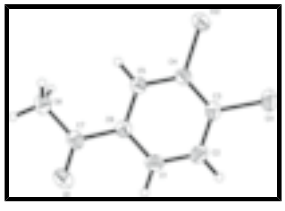

Fig. 1. The structure of the title compound (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme.

1-(3,4-dichlorophenyl)ethanone

Crystal data

$C_8H_6Cl_2O$	F_{000} = 384
M_r = 189.03	D_x = 1.571 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å

Hall symbol: -P 2ybc	Cell parameters from 3035 reflections
a = 7.2878 (15) Å	θ = 2.9–26.4º
b = 10.597 (2) Å	μ = 0.74 mm^{-1}
c = 10.610 (2) Å	T = 294 (2) K
β = 102.717 (3)º	Block, colourless
V = 799.3 (3) Å^3	0.20 × 0.18 × 0.16 mm
Z = 4	

Data collection

Bruker SMART CCD area-detector diffractometer	1637 independent reflections
Radiation source: fine-focus sealed tube	1449 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.018
T = 294(2) K	θ_{max} = 26.4º
φ and ω scans	θ_{min} = 2.8º
Absorption correction: multi-scan (SADABS; Bruker, 1997)	h = −9→8
T_{min} = 0.866, T_{max} = 0.890	k = −9→13
4489 measured reflections	l = −13→12

Refinement

Refinement on F^2	H-atom parameters constrained
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0506P)^2 + 0.2513P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.033	$(\Delta/\sigma)_{max}$ = 0.001
$wR(F^2)$ = 0.094	$\Delta\rho_{max}$ = 0.26 e Å^{-3}
S = 1.05	$\Delta\rho_{min}$ = −0.29 e Å^{-3}
1637 reflections	Extinction correction: none
101 parameters	
Primary atom site location: structure-invariant direct methods	
Secondary atom site location: difference Fourier map	
Hydrogen site location: inferred from neighbouring sites	

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 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	0.09028 (8)	0.22661 (5)	0.39055 (6)	0.06076 (19)
Cl2	0.12712 (9)	0.44153 (5)	0.19719 (5)	0.0671 (2)
O1	0.4462 (2)	0.73876 (15)	0.70133 (14)	0.0665 (4)
C1	0.2990 (2)	0.50645 (18)	0.61880 (17)	0.0466 (4)
H1	0.3372	0.5199	0.7073	0.056*
C2	0.2314 (3)	0.39160 (18)	0.57462 (18)	0.0495 (4)
H2	0.2227	0.3271	0.6325	0.059*
C3	0.1763 (2)	0.37135 (16)	0.44448 (18)	0.0421 (4)
C4	0.1904 (2)	0.46613 (16)	0.35993 (16)	0.0409 (4)
C5	0.2580 (2)	0.58170 (16)	0.40441 (16)	0.0409 (4)
H5	0.2675	0.6458	0.3464	0.049*
C6	0.3120 (2)	0.60317 (16)	0.53495 (16)	0.0383 (4)
C7	0.3855 (2)	0.72665 (17)	0.58718 (18)	0.0462 (4)
C8	0.3836 (4)	0.83410 (19)	0.4994 (2)	0.0637 (5)
H8A	0.4336	0.9073	0.5485	0.096*
H8B	0.2567	0.8507	0.4540	0.096*
H8C	0.4592	0.8146	0.4385	0.096*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0682 (3)	0.0402 (3)	0.0742 (4)	−0.0130 (2)	0.0164 (3)	−0.0048 (2)
Cl2	0.1019 (4)	0.0493 (3)	0.0420 (3)	−0.0029 (2)	−0.0015 (2)	−0.00618 (19)
O1	0.0833 (10)	0.0658 (10)	0.0479 (8)	−0.0126 (8)	0.0089 (7)	−0.0161 (7)
C1	0.0508 (9)	0.0526 (10)	0.0374 (8)	−0.0012 (8)	0.0117 (7)	0.0003 (7)
C2	0.0566 (10)	0.0464 (10)	0.0486 (10)	−0.0027 (8)	0.0182 (8)	0.0091 (8)
C3	0.0387 (8)	0.0353 (8)	0.0540 (10)	−0.0018 (6)	0.0138 (7)	−0.0021 (7)
C4	0.0432 (8)	0.0399 (9)	0.0385 (8)	0.0028 (7)	0.0065 (7)	−0.0021 (7)
C5	0.0460 (9)	0.0363 (8)	0.0403 (9)	0.0011 (7)	0.0095 (7)	0.0014 (7)
C6	0.0362 (8)	0.0406 (9)	0.0398 (8)	0.0017 (6)	0.0122 (6)	−0.0022 (7)
C7	0.0469 (9)	0.0463 (10)	0.0471 (10)	−0.0023 (7)	0.0139 (8)	−0.0109 (8)
C8	0.0870 (15)	0.0401 (10)	0.0653 (13)	−0.0095 (10)	0.0195 (11)	−0.0077 (9)

Geometric parameters (Å, °)

Cl1—C3	1.7072 (17)	C4—C5	1.365 (2)
Cl2—C4	1.7063 (17)	C5—C6	1.373 (2)
O1—C7	1.201 (2)	C5—H5	0.9300
C1—C2	1.357 (3)	C6—C7	1.475 (2)
C1—C6	1.374 (3)	C7—C8	1.469 (3)
C1—H1	0.9300	C8—H8A	0.9600
C2—C3	1.367 (3)	C8—H8B	0.9600
C2—H2	0.9300	C8—H8C	0.9600
C3—C4	1.366 (2)		

C2—C1—C6	121.12 (17)	C6—C5—H5	120.0
C2—C1—H1	119.4	C5—C6—C1	118.97 (16)
C6—C1—H1	119.4	C5—C6—C7	121.72 (16)
C1—C2—C3	119.55 (17)	C1—C6—C7	119.31 (15)
C1—C2—H2	120.2	O1—C7—C8	120.41 (18)
C3—C2—H2	120.2	O1—C7—C6	119.75 (17)
C4—C3—C2	120.00 (16)	C8—C7—C6	119.84 (16)
C4—C3—Cl1	121.05 (14)	C7—C8—H8A	109.5
C2—C3—Cl1	118.95 (14)	C7—C8—H8B	109.5
C5—C4—C3	120.43 (16)	H8A—C8—H8B	109.5
C5—C4—Cl2	118.92 (14)	C7—C8—H8C	109.5
C3—C4—Cl2	120.65 (14)	H8A—C8—H8C	109.5
C4—C5—C6	119.93 (16)	H8B—C8—H8C	109.5
C4—C5—H5	120.0		

C6—C1—C2—C3	0.4 (3)	C4—C5—C6—C1	0.9 (2)
C1—C2—C3—C4	0.4 (3)	C4—C5—C6—C7	−179.87 (15)
C1—C2—C3—Cl1	−179.62 (14)	C2—C1—C6—C5	−1.0 (3)
C2—C3—C4—C5	−0.5 (3)	C2—C1—C6—C7	179.71 (16)
Cl1—C3—C4—C5	179.49 (13)	C5—C6—C7—O1	−173.66 (17)
C2—C3—C4—Cl2	178.55 (14)	C1—C6—C7—O1	5.6 (3)
Cl1—C3—C4—Cl2	−1.4 (2)	C5—C6—C7—C8	6.5 (3)
C3—C4—C5—C6	−0.1 (3)	C1—C6—C7—C8	−174.27 (18)
Cl2—C4—C5—C6	−179.19 (12)		

Fig. 1