

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

2,2-Dichloro-1-(4-methylphenyl)-ethanone

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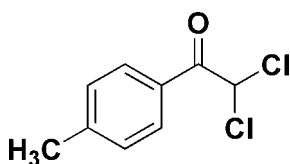
Received 23 December 2010; accepted 4 January 2011

Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å; R factor = 0.054; wR factor = 0.155; data-to-parameter ratio = 15.4.

The molecule of the title compound, $\text{C}_9\text{H}_8\text{Cl}_2\text{O}$, is almost planar: the dihedral angle between the benzene ring and the plane defined by the carbonyl O and ethane C atoms is $15.5(2)^\circ$. The crystal packing is stabilized by intermolecular $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds.

Related literature

For the preparation, see: Aston *et al.* (1943); Terent'ev *et al.* (2004). For synthetic use of the title compound and mandelic acid derivatives, see: Schiffers & Bolm (2008); Blay *et al.* (2006).



Experimental

Crystal data

$\text{C}_9\text{H}_8\text{Cl}_2\text{O}$
 $M_r = 203.05$
Monoclinic, $P2_1/c$
 $a = 6.650(5)$ Å
 $b = 9.959(7)$ Å
 $c = 14.475(11)$ Å
 $\beta = 92.921(9)^\circ$

$V = 957.4(12)$ Å³
 $Z = 4$
Mo $K\alpha$ radiation
 $\mu = 0.63$ mm⁻¹
 $T = 296$ K
 $0.32 \times 0.26 \times 0.14$ mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (SADABS; Bruker, 2005)
 $T_{\min} = 0.826$, $T_{\max} = 0.920$
4496 measured reflections
1694 independent reflections
874 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.061$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.054$
 $wR(F^2) = 0.155$
 $S = 1.03$
1694 reflections
110 parameters
H-atom parameters constrained
 $\Delta\rho_{\max} = 0.21$ e Å⁻³
 $\Delta\rho_{\min} = -0.27$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{C4}-\text{H4}\cdots\text{O1}^i$	0.93	2.58	3.42	150

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

Data collection: APEX2 (Bruker, 2008); cell refinement: SAINT (Bruker, 2008); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL (Sheldrick, 2008); software used to prepare material for publication: Mercury (Macrae *et al.*, 2006) and ORTEP-3 (Farrugia, 1997).

We thank the Natural Science Foundation of China (grant No. 20802092) for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5447).

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supplementary materials

Acta Cryst. (2011). E67, o337 [doi:10.1107/S1600536811000134]

2,2-Dichloro-1-(4-methylphenyl)ethanone

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Comment

The title compound, 2,2-Dichloro-1-(4-methylphenyl)ethanone, was obtained by chloration of 1-(4-methylphenyl)ethanone with concentrated hydrochloride and aqueous hydroperoxide in hot ethanol (Terent'ev *et al.*, 2004) and it was used for the preparation of substituted mandelic acid and derivatives.

In the title compound, C₉H₈Cl₂O, the lengths of two C—Cl bonds are different, the distance of C1—Cl1 is 1.757 Å, otherwise, the distance of C1—Cl2 is 1.762 Å. The molecule is nearly planar, the dihedral angle between the phenyl ring and the plane defined by O1, C2 and C1 is 15.5°. The packing of molecules in the crystal structure is stabilized by intermolecular C—H...O hydrogen bonds.

Experimental

To a stirred hot mixed aqueous concentrated hydrochloride and ethanol solution (300 cm³) of commercially available 1-*p*-tolylethanone (13.42 g, 0.10 mol) added dropwise aqueous hydroperoxide (35% wt in water, 0.272 mol), and the mixture was stirred for 30 min at 90–100°C. The solution was cooled to room temperature and diluted by addition of water (300 cm³), it was extracted by ether (2× 400 cm³), the combined organic layer was washed with 1 M NaOH (100 cm³), water (100 cm³), brine (2× 110 cm³), and dried over Na₂SO₄. Evaporation of the solvent afforded the title compound as a light yellow oli (19.8 g, 97%), which was solidified as a pale block after 24 h at room temperature. The melting point and the spectroscopic data of the title compound were consisted with the reported literature (Terent'ev *et al.*, 2004).

Refinement

All H atoms were placed in calculated positions and refined as riding, with C—H = 0.93–0.98 Å and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Figures

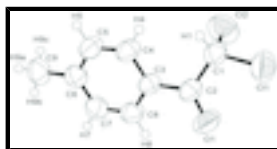


Fig. 1. The molecular structure of the title compound, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. Hydrogen atoms are drawn as spheres of arbitrary radius.

2,2-Dichloro-1-(4-methylphenyl)ethanone

Crystal data

$C_9H_8Cl_2O$	$F(000) = 416$
$M_r = 203.05$	$D_x = 1.409 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
$a = 6.650 (5) \text{ \AA}$	$\theta = 2.5\text{--}25.1^\circ$
$b = 9.959 (7) \text{ \AA}$	$\mu = 0.63 \text{ mm}^{-1}$
$c = 14.475 (11) \text{ \AA}$	$T = 296 \text{ K}$
$\beta = 92.921 (9)^\circ$	Block, colorless
$V = 957.4 (12) \text{ \AA}^3$	$0.32 \times 0.26 \times 0.14 \text{ mm}$
$Z = 4$	

Data collection

Bruker SMART APEX CCD area-detector diffractometer	1694 independent reflections
Radiation source: fine-focus sealed tube graphite	874 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{\text{int}} = 0.061$
Absorption correction: multi-scan (SADABS; Bruker, 2005)	$\theta_{\text{max}} = 25.1^\circ$, $\theta_{\text{min}} = 2.5^\circ$
$T_{\text{min}} = 0.826$, $T_{\text{max}} = 0.920$	$h = -7 \rightarrow 7$
4496 measured reflections	$k = -11 \rightarrow 9$
	$l = -17 \rightarrow 15$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.054$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.155$	H-atom parameters constrained
$S = 1.03$	$w = 1/[\sigma^2(F_o^2) + (0.0679P)^2]$
1694 reflections	where $P = (F_o^2 + 2F_c^2)/3$
110 parameters	$(\Delta/\sigma)_{\text{max}} < 0.001$
0 restraints	$\Delta\rho_{\text{max}} = 0.21 \text{ e \AA}^{-3}$
	$\Delta\rho_{\text{min}} = -0.27 \text{ e \AA}^{-3}$

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 $F^2 > \sigma(F^2)$ 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C11	1.21054 (15)	0.84106 (11)	0.13668 (8)	0.0912 (5)
C12	0.79412 (19)	0.87565 (15)	0.07241 (8)	0.1147 (6)
O1	0.9705 (4)	0.6849 (3)	0.2551 (2)	0.0966 (10)
C1	0.9678 (5)	0.8856 (4)	0.1682 (2)	0.0670 (10)
H1	0.9707	0.9780	0.1915	0.080*
C2	0.8918 (5)	0.7929 (4)	0.2428 (2)	0.0627 (9)
C3	0.7161 (5)	0.8368 (3)	0.2934 (2)	0.0562 (9)
C4	0.6500 (5)	0.9690 (4)	0.2946 (2)	0.0662 (10)
H4	0.7173	1.0350	0.2629	0.079*
C5	0.4837 (5)	1.0019 (4)	0.3433 (3)	0.0711 (10)
H5	0.4403	1.0907	0.3436	0.085*
C6	0.3800 (5)	0.9072 (4)	0.3915 (2)	0.0662 (10)
C7	0.4492 (6)	0.7764 (4)	0.3911 (3)	0.0747 (11)
H7	0.3823	0.7111	0.4238	0.090*
C8	0.6142 (6)	0.7406 (4)	0.3439 (3)	0.0691 (10)
H8	0.6588	0.6521	0.3453	0.083*
C9	0.1955 (5)	0.9460 (5)	0.4417 (3)	0.0876 (13)
H9A	0.2314	1.0114	0.4884	0.131*
H9B	0.1410	0.8679	0.4701	0.131*
H9C	0.0965	0.9834	0.3985	0.131*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.0822 (7)	0.0892 (9)	0.1049 (9)	0.0029 (6)	0.0303 (6)	-0.0065 (6)
C12	0.1093 (9)	0.1452 (13)	0.0881 (9)	0.0030 (8)	-0.0091 (7)	0.0200 (7)
O1	0.0959 (19)	0.0598 (18)	0.137 (3)	0.0230 (16)	0.0350 (18)	0.0209 (17)
C1	0.072 (2)	0.050 (2)	0.080 (3)	-0.0003 (18)	0.019 (2)	-0.0037 (18)
C2	0.065 (2)	0.049 (2)	0.075 (2)	0.002 (2)	0.0035 (19)	-0.0052 (19)
C3	0.0566 (19)	0.046 (2)	0.065 (2)	-0.0002 (17)	0.0016 (17)	0.0010 (17)
C4	0.062 (2)	0.049 (2)	0.088 (3)	-0.0012 (19)	0.0076 (19)	0.0019 (18)
C5	0.067 (2)	0.058 (3)	0.088 (3)	0.012 (2)	0.007 (2)	0.000 (2)
C6	0.063 (2)	0.073 (3)	0.063 (2)	0.000 (2)	0.0003 (18)	0.001 (2)
C7	0.081 (3)	0.068 (3)	0.076 (3)	-0.007 (2)	0.016 (2)	0.007 (2)
C8	0.080 (2)	0.051 (2)	0.076 (3)	0.000 (2)	0.003 (2)	0.0040 (19)
C9	0.070 (2)	0.113 (4)	0.081 (3)	0.009 (2)	0.014 (2)	0.002 (2)

Geometric parameters (\AA , $^\circ$)

C11—C1	1.756 (4)	C5—C6	1.378 (5)
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supplementary materials

C12—C1	1.761 (4)	C5—H5	0.9300
O1—C2	1.206 (4)	C6—C7	1.382 (5)
C1—C2	1.527 (5)	C6—C9	1.508 (5)
C1—H1	0.9800	C7—C8	1.369 (5)
C2—C3	1.476 (5)	C7—H7	0.9300
C3—C4	1.388 (5)	C8—H8	0.9300
C3—C8	1.401 (5)	C9—H9A	0.9600
C4—C5	1.380 (5)	C9—H9B	0.9600
C4—H4	0.9300	C9—H9C	0.9600
C2—C1—C11	111.9 (3)	C4—C5—H5	119.0
C2—C1—C12	107.2 (2)	C5—C6—C7	117.9 (3)
C11—C1—C12	110.86 (19)	C5—C6—C9	120.6 (4)
C2—C1—H1	109.0	C7—C6—C9	121.5 (4)
C11—C1—H1	109.0	C8—C7—C6	121.5 (4)
C12—C1—H1	109.0	C8—C7—H7	119.2
O1—C2—C3	122.6 (3)	C6—C7—H7	119.2
O1—C2—C1	119.2 (3)	C7—C8—C3	120.2 (4)
C3—C2—C1	118.1 (3)	C7—C8—H8	119.9
C4—C3—C8	118.7 (3)	C3—C8—H8	119.9
C4—C3—C2	123.1 (3)	C6—C9—H9A	109.5
C8—C3—C2	118.2 (3)	C6—C9—H9B	109.5
C5—C4—C3	119.6 (3)	H9A—C9—H9B	109.5
C5—C4—H4	120.2	C6—C9—H9C	109.5
C3—C4—H4	120.2	H9A—C9—H9C	109.5
C6—C5—C4	122.0 (4)	H9B—C9—H9C	109.5
C6—C5—H5	119.0		
C11—C1—C2—O1	-19.0 (4)	C2—C3—C4—C5	179.7 (3)
C12—C1—C2—O1	102.8 (3)	C3—C4—C5—C6	0.1 (6)
C11—C1—C2—C3	164.5 (2)	C4—C5—C6—C7	1.0 (5)
C12—C1—C2—C3	-73.7 (3)	C4—C5—C6—C9	-178.3 (3)
O1—C2—C3—C4	166.2 (4)	C5—C6—C7—C8	-0.7 (5)
C1—C2—C3—C4	-17.4 (5)	C9—C6—C7—C8	178.5 (3)
O1—C2—C3—C8	-12.5 (5)	C6—C7—C8—C3	-0.7 (5)
C1—C2—C3—C8	163.8 (3)	C4—C3—C8—C7	1.9 (5)
C8—C3—C4—C5	-1.6 (5)	C2—C3—C8—C7	-179.4 (3)

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C4—H4 \cdots O1 ⁱ	0.93	2.58	3.42	150

Symmetry codes: (i) $-x+2, y+1/2, -z+1/2$.

Fig. 1

