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# (*E*)-4,4,4-Trichloro-1-phenylbut-2-en-1-one

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In the crystal structure of the title compound, $C_{10}H_7Cl_3O$, the carbonyl group is in an *s-cis* conformation and the configuration of the C═C double bond is *E*. With the exception of two Cl atoms, all the atoms lie on a crystallographic mirror plane. Intramolecular C—H···O and C—H···Cl hydrogen bonds are present.

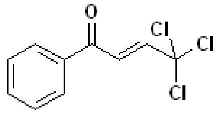
**Key indicators**

Single-crystal X-ray study
$T$ = 298 K
Mean $\sigma$(C–C) = 0.009 Å
$R$ factor = 0.079
$wR$ factor = 0.228
Data-to-parameter ratio = 13.2

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

## Comment

$\alpha,\beta$-Unsaturated ketones can be prepared by several methods and are versatile intermediates in organic syntheses (Nicolaou *et al.*, 2000). These compounds are often used in Michael additions (Ozeki *et al.*, 2004) and 1,4-addition reactions (Kisanga *et al.*, 2002). Some of them display biological activities (Tanaka *et al.*, 2003) and some interesting crystal structures of complexes containing $\alpha,\beta$-unsaturated ketones have been reported (Ortega-Jimenez *et al.*, 2000). The title compound, (I), is an intermediate in the preparation of a quinoline derivative.



(I)

In the molecule of (I) (Fig. 1), the carbonyl group is in an *s-cis* conformation and the configuration of the C═C double bond is *E*. All the atoms lie on a crystallographic mirror plane at $y = \frac{1}{4}$, with the exception of Cl1 and Cl1$^{i}$ [symmetry code: (i) $x, \frac{1}{2} - y, z$]. The C10—Cl1 bond is longer and C10—Cl2 shorter (Table 1) than the average value for C$sp^3$—Cl in trichloromethyl compounds (Allen *et al.*, 1987).

As in other $\alpha,\beta$-unsaturated ketones (Parthasarathi *et al.*, 2002), electron conjugation is observed between the central —CH═CH—C(═O)— group and the phenyl ring. Intramolecular C—H···O and C—H···Cl hydrogen bonds are present (Table 2).

## Experimental

The title compound was synthesized from triphenylbenzoxymethylenephosphorane (1.52 g, 4 mmol) and trichloroacetaldehyde (0.59 g, 4 mmol), refluxed for 2 h in dichloromethane (50 ml). After removal of solvent, the residue was purified by column chromatography on silica gel with 20:1 petroleum ether (60–90) and ethyl acetate. Single crystals suitable for X-ray diffraction (m.p. 394–397 K) were obtained by slow evaporation of an ethyl acetate/petroleum ether (1:2) solution. Spectroscopic analysis, IR (KBr, $\nu$ cm$^{-1}$): 2919, 1675, 1275, 955; $^1$H NMR (CDCl$_3$, p.p.m.): 7.98 (*d*, 2H), 7.64 (*m*, 1H), 7.52 (*m*, 2H), 7.42 (*d, J* = 14.6 Hz, 1H), 7.28(*d, J* = 14.6 Hz, 1H).

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### Crystal data

$C_{10}H_7Cl_3O$
$M_r = 249.51$
Orthorhombic, $Pnma$
$a = 10.4802$ (7) Å
$b = 7.1166$ (10) Å
$c = 14.69240$ (14) Å
$V = 1095.81$ (17) Å$^3$
$Z = 4$
$D_x = 1.512$ Mg m$^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 1773 reflections
$\theta = 2.4$–25.0°
$\mu = 0.80$ mm$^{-1}$
$T = 298$ (2) K
Block, colorless
0.29 × 0.25 × 0.20 mm

### Data collection

Bruker SMART APEX area-detector diffractometer
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
$T_{\min} = 0.802$, $T_{\max} = 0.857$
5398 measured reflections
1082 independent reflections
932 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.024$
$\theta_{\max} = 25.3°$
$h = -12 \rightarrow 12$
$k = -6 \rightarrow 8$
$l = -17 \rightarrow 17$

### Refinement

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)] = 0.079$
$wR(F^2) = 0.228$
$S = 1.08$
1082 reflections
82 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1112P)^2 + 2.0938P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 1.10$ e Å$^{-3}$
$\Delta\rho_{\min} = -0.58$ e Å$^{-3}$

**Table 1**
Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| Cl1—C10 | 1.791 (4) | C7—C8 | 1.482 (8) |
| Cl2—C10 | 1.730 (6) | C8—C9 | 1.294 (9) |
| O1—C7 | 1.216 (7) | C9—C10 | 1.479 (8) |
| C6—C7 | 1.473 (8) | | |
| C1—C6—C7 | 118.8 (5) | C6—C7—C8 | 119.4 (5) |
| C5—C6—C7 | 123.0 (5) | C9—C8—C7 | 122.0 (6) |
| O1—C7—C6 | 121.1 (5) | C8—C9—C10 | 128.3 (6) |
| O1—C7—C8 | 119.5 (5) | Cl2—C10—Cl1 | 110.3 (2) |

**Table 2**
Hydrogen-bonding geometry (Å, °).

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C9—H9···O1 | 0.93 | 2.42 | 2.767 (7) | 102 |
| C8—H8···Cl2 | 0.93 | 2.62 | 3.017 (6) | 107 |

All H atoms were initially located in a difference Fourier map and were then positioned geometrically and constrained to ride on their parent atoms, with C—H = 0.93 Å and $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C). The highest residual electron-density peak is 0.954 Å from atom Cl1.

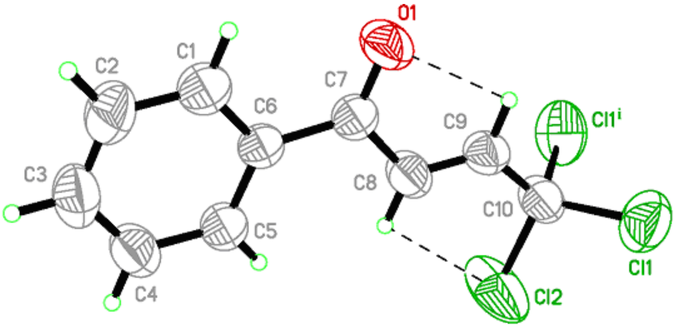

**Figure 1**
The structure of (I), showing the atomic numbering scheme and displacement ellipsoids at the 50% probability level [Symmetry code: (i) $x, \frac{1}{2} - y, z$]. The dashed lines indicate hydrogen bonds.

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL* (Bruker, 2002); software used to prepare material for publication: *SHELXTL*.

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