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Benzene-1,3,5-tricarbonyl trichloride

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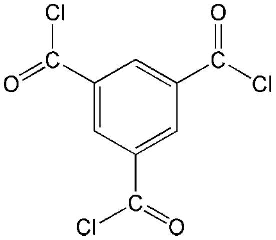
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.006 Å; R factor = 0.065; wR factor = 0.170; data-to-parameter ratio = 17.2.

In the title molecule, $C_9H_3Cl_3O_3$, there are three short interactions involving the benzene H atoms and the chloroformyl Cl atoms. In the crystal, molecules stack along the a axis with no significant non-bonded interactions.

Related literature

For the preparation of the title compound, see: Hamel *et al.* (1968). For applications of 1,3,5-tri(chloroformyl)benzene, see: Buch *et al.* (2008); Li *et al.* (2007). For related structures and hydrogen bonding, see: Leser & Rabinovich (1978*a*,*b*); Jeffrey *et al.* (1985).



Experimental

Crystal data

$C_9H_3Cl_3O_3$
M_r = 265.46
Orthorhombic, $P2_12_12_1$
a = 6.0230 (13) Å
b = 8.3306 (18) Å
c = 21.314 (5) Å
V = 1069.4 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.84 mm^{-1}
T = 298 K
0.30 × 0.20 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.787, T_{max} = 0.851
6552 measured reflections
2338 independent reflections
2267 reflections with $I > 2\sigma(I)$
R_{int} = 0.088

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.065
$wR(F^2)$ = 0.170
S = 1.22
2338 reflections
136 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}
Absolute structure: Flack (1983), 952 Friedel pairs
Flack parameter: 0.13 (15)

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

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2···Cl1	0.93	2.64	3.047 (5)	107
C4—H4···Cl2	0.93	2.62	3.036 (4)	108
C6—H6···Cl3	0.93	2.62	3.037 (4)	108

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus*; 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: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: VM2166).

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supplementary materials

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Benzene-1,3,5-tricarbonyl trichloride

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Comment

1,3,5-Tri(chloroformyl)benzene is a commonly used monomer, mainly used in the preparation of reverse osmosis membranes and polyamide nanofiltration membranes (Buch *et al.*, 2008; Li *et al.*, 2007), which has good application prospects in the rapid development of today's film industry. As part of our ongoing studies, the preparation and X-ray structure determination of the title compound, (I), was undertaken.

The molecular structure of the title compound is shown in Figure 1. Bond lengths and angles in (I) show normal values. The geometric parameters of the title molecule agree well with those reported for similar structures (Leser & Rabinovich, 1978*a*,*b*). The non-hydrogen atoms of the molecule lie in a nearly plane with an r.m.s deviation of 0.1118 Å. Three intramolecular hydrogen bonds are observed between the C—H of phenyl group and the Cl atom of the chloroformyl group (Jeffrey *et al.*, 1985) (Figure 1 and Table 1). The shortest contacts in the crystal packing are O1···C2^{i} [3.094 (6) Å], O2···Cl3^{ii} [3.191 (5) Å] and O3···C9^{iii} [3.216 (6) Å] [symmetry code (i): $-x,1/2+y,1/2-z$; (ii): $1+x,1+y,z$; (iii): $1/2+x,3/2-y,-z$].

Experimental

Compound (I) was synthesized according to the literature procedure of Hamel *et al.* (1968). Single crystals suitable for X-ray diffraction were grown by slow evaporation from 4:1 (V/V) mixed solution of petroleum ether and chloroform at room temperature.

Refinement

All H atoms were included in the riding-model approximation, with C—H distances of 0.93 Å, and the isotropic displacement parameters were set equal to $1.2U_{\rm eq}$ of the carrier atom.

Computing details

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus* (Bruker, 2001); 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: *SHELXTL* (Sheldrick, 2008).

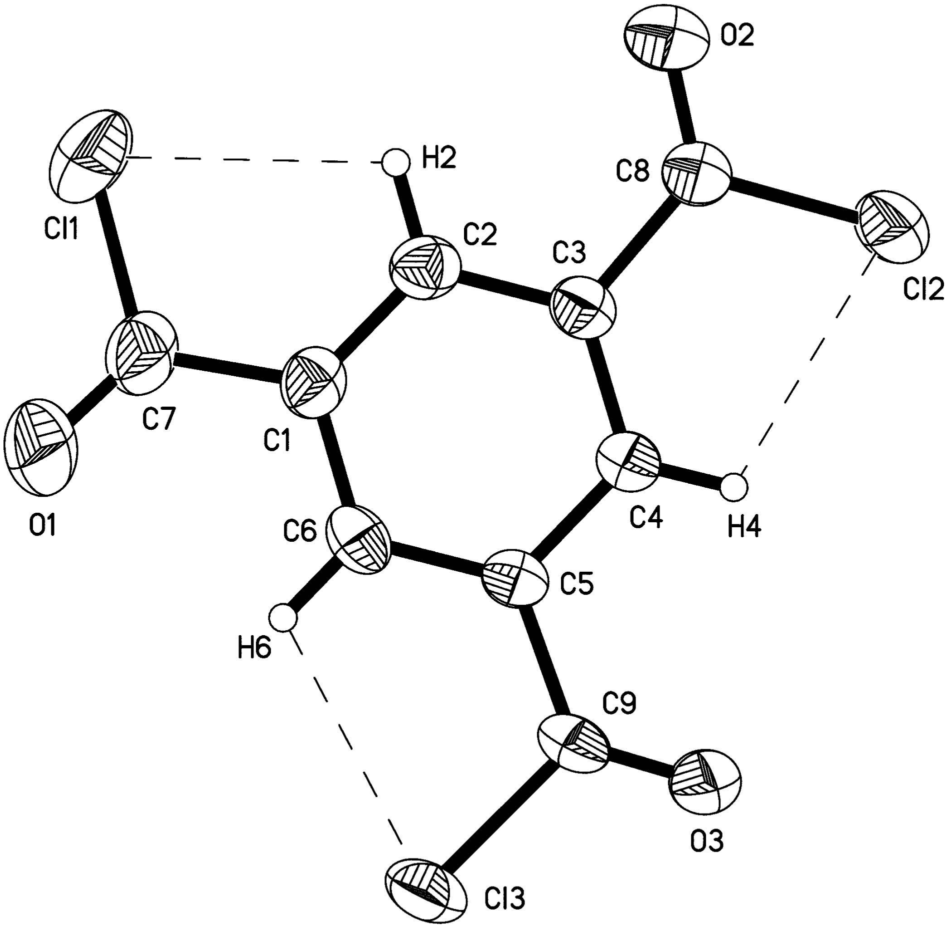

Figure 1

The molecular structure of (I), showing 30% probability displacement ellipsoids. Dashed lines indicate hydrogen bonds.

Benzene-1,3,5-tricarbonyl trichloride

Crystal data

$C_9H_3Cl_3O_3$
$M_r = 265.46$
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
$a = 6.0230$ (13) Å
$b = 8.3306$ (18) Å
$c = 21.314$ (5) Å
$V = 1069.4$ (4) Å^3
$Z = 4$

$F(000) = 528$
$D_x = 1.649$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3613 reflections
$\theta = 2.4$–28.2°
$\mu = 0.84$ mm^{-1}
$T = 298$ K
Block, colorless
0.30 × 0.20 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and ω scans

Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.787$, $T_{max} = 0.851$
6552 measured reflections
2338 independent reflections
2267 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.088$
$\theta_{\text{max}} = 27.0°$, $\theta_{\text{min}} = 2.6°$
$h = -7 \rightarrow 5$
$k = -9 \rightarrow 10$
$l = -27 \rightarrow 27$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.065$
$wR(F^2) = 0.170$
$S = 1.22$
2338 reflections
136 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
$w = 1/[\sigma^2(F_o^2) + (0.0522P)^2 + 0.5279P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\text{max}} < 0.001$
$\Delta\rho_{\text{max}} = 0.31$ e Å^{-3}
$\Delta\rho_{\text{min}} = -0.28$ e Å^{-3}
Absolute structure: Flack (1983), 952 Friedel pairs
Flack parameter: 0.13 (15)

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.4413 (7)	0.7665 (6)	0.83052 (18)	0.0582 (10)
C2	0.3612 (7)	0.9176 (6)	0.84269 (18)	0.0582 (9)
H2	0.2326	0.9534	0.8230	0.070*
C3	0.4728 (7)	1.0170 (5)	0.88445 (19)	0.0545 (9)
C4	0.6638 (6)	0.9669 (5)	0.91364 (18)	0.0497 (8)
H4	0.7392	1.0352	0.9408	0.060*
C5	0.7435 (6)	0.8124 (5)	0.90207 (17)	0.0482 (8)
C6	0.6328 (7)	0.7132 (5)	0.86085 (19)	0.0553 (9)
H6	0.6860	0.6102	0.8532	0.066*
C7	0.3370 (9)	0.6507 (8)	0.7862 (3)	0.0834 (16)
C8	0.3650 (9)	1.1749 (6)	0.8981 (3)	0.0764 (14)
C9	0.9471 (7)	0.7645 (5)	0.9372 (2)	0.0565 (9)
Cl1	0.1090 (3)	0.7279 (3)	0.74344 (9)	0.1188 (7)
Cl2	0.5232 (3)	1.30825 (15)	0.94387 (9)	0.0934 (5)
Cl3	1.0317 (2)	0.56293 (13)	0.92527 (7)	0.0775 (4)
O1	0.3901 (9)	0.5166 (6)	0.7783 (2)	0.1201 (18)
O2	0.1848 (8)	1.2126 (6)	0.8830 (3)	0.130 (2)
O3	1.0513 (6)	0.8462 (4)	0.97113 (18)	0.0759 (9)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.058 (2)	0.072 (3)	0.0452 (17)	0.0009 (19)	0.0046 (17)	−0.0110 (19)
C2	0.058 (2)	0.066 (2)	0.0502 (19)	0.008 (2)	−0.0014 (17)	0.0023 (19)
C3	0.058 (2)	0.0495 (19)	0.056 (2)	0.0058 (17)	0.0010 (18)	0.0014 (16)
C4	0.0481 (17)	0.0480 (18)	0.0530 (18)	0.0037 (15)	0.0025 (15)	0.0009 (16)
C5	0.0486 (18)	0.0464 (18)	0.0495 (18)	0.0055 (16)	0.0044 (15)	0.0063 (16)
C6	0.060 (2)	0.048 (2)	0.058 (2)	0.0056 (17)	0.0147 (18)	−0.0070 (17)
C7	0.076 (3)	0.103 (4)	0.070 (3)	0.003 (3)	−0.002 (2)	−0.034 (3)
C8	0.082 (3)	0.058 (2)	0.090 (3)	0.018 (2)	−0.029 (3)	−0.009 (2)
C9	0.0507 (19)	0.0460 (19)	0.073 (2)	0.0127 (16)	0.0077 (18)	0.0032 (19)
Cl1	0.1086 (11)	0.1431 (17)	0.1046 (11)	−0.0062 (12)	−0.0484 (10)	−0.0267 (12)
Cl2	0.0915 (9)	0.0520 (6)	0.1365 (13)	0.0142 (6)	−0.0241 (9)	−0.0208 (7)
Cl3	0.0730 (7)	0.0567 (6)	0.1028 (9)	0.0245 (5)	−0.0030 (6)	−0.0003 (6)
O1	0.123 (3)	0.112 (4)	0.125 (4)	0.020 (3)	−0.018 (3)	−0.076 (3)
O2	0.112 (3)	0.093 (3)	0.185 (5)	0.051 (3)	−0.071 (3)	−0.041 (3)
O3	0.0646 (18)	0.0658 (19)	0.097 (2)	0.0090 (15)	−0.0232 (18)	−0.0087 (19)

Geometric parameters (Å, º)

C1—C2	1.373 (6)	C5—C6	1.378 (6)
C1—C6	1.394 (6)	C5—C9	1.491 (5)
C1—C7	1.489 (7)	C6—H6	0.9300
C2—C3	1.388 (6)	C7—O1	1.174 (7)
C2—H2	0.9300	C7—Cl1	1.769 (6)
C3—C4	1.373 (6)	C8—O2	1.175 (6)
C3—C8	1.496 (6)	C8—Cl2	1.758 (5)
C4—C5	1.396 (5)	C9—O3	1.174 (5)
C4—H4	0.9300	C9—Cl3	1.773 (4)
C2—C1—C6	119.6 (4)	C4—C5—C9	116.1 (4)
C2—C1—C7	124.4 (4)	C5—C6—C1	120.3 (4)
C6—C1—C7	115.9 (4)	C5—C6—H6	119.8
C1—C2—C3	119.9 (4)	C1—C6—H6	119.8
C1—C2—H2	120.1	O1—C7—C1	126.3 (6)
C3—C2—H2	120.1	O1—C7—Cl1	118.9 (5)
C4—C3—C2	121.0 (4)	C1—C7—Cl1	114.8 (4)
C4—C3—C8	122.8 (4)	O2—C8—C3	125.6 (5)
C2—C3—C8	116.1 (4)	O2—C8—Cl2	118.9 (4)
C3—C4—C5	119.2 (4)	C3—C8—Cl2	115.4 (3)
C3—C4—H4	120.4	O3—C9—C5	126.4 (4)
C5—C4—H4	120.4	O3—C9—Cl3	118.9 (3)
C6—C5—C4	119.9 (4)	C5—C9—Cl3	114.7 (3)
C6—C5—C9	123.9 (4)		
C6—C1—C2—C3	0.8 (6)	C2—C1—C7—O1	−173.6 (6)
C7—C1—C2—C3	−179.3 (4)	C6—C1—C7—O1	6.4 (9)
C1—C2—C3—C4	0.5 (6)	C2—C1—C7—Cl1	5.3 (7)
C1—C2—C3—C8	−175.9 (4)	C6—C1—C7—Cl1	−174.8 (3)

C2—C3—C4—C5	−1.5 (6)	C4—C3—C8—O2	−166.4 (7)
C8—C3—C4—C5	174.7 (4)	C2—C3—C8—O2	10.0 (9)
C3—C4—C5—C6	1.2 (6)	C4—C3—C8—Cl2	10.6 (6)
C3—C4—C5—C9	−177.8 (3)	C2—C3—C8—Cl2	−173.0 (4)
C4—C5—C6—C1	0.1 (6)	C6—C5—C9—O3	177.2 (4)
C9—C5—C6—C1	179.0 (4)	C4—C5—C9—O3	−3.8 (6)
C2—C1—C6—C5	−1.1 (6)	C6—C5—C9—Cl3	−3.1 (5)
C7—C1—C6—C5	179.0 (4)	C4—C5—C9—Cl3	175.9 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2···Cl1	0.93	2.64	3.047 (5)	107
C4—H4···Cl2	0.93	2.62	3.036 (4)	108
C6—H6···Cl3	0.93	2.62	3.037 (4)	108