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3,5-Dinitrobenzoyl chloride

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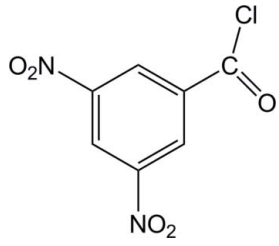
Key indicators: single-crystal X-ray study; T = 93 K; mean σ(C–C) = 0.003 Å; disorder in main residue; R factor = 0.038; wR factor = 0.097; data-to-parameter ratio = 12.3.

The carbonyl chloride group in the title compound, $C_7H_3ClN_2O_5$, is disordered over two orientations with occupancies of 0.505 (5) and 0.495 (5). The molecule is approximately planar, the dihedral angle between the carbonyl chloride plane and benzene ring being 9.6 (4)° in the major disorder component and 7.1 (4)° in the minor component. The nitro group at the 5-position is twisted, forming a dihedral angle of 6.7 (4)°. The crystal packing is stabilized by C—H···O hydrogen bonds.

Related literature

For general background to 3,5-dinitrobenzoyl chloride, see: Gennaro *et al.* (1993); Liu & Wang (2000); Saunders & Stacey (1942).



Experimental

Crystal data

$C_7H_3ClN_2O_5$ M_r = 230.56
Orthorhombic, $Pna2_1$
a = 18.295 (4) Å
b = 8.3924 (19) Å
c = 5.7362 (13) Å
V = 880.7 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.44 mm^{-1}
T = 93 K
0.37 × 0.33 × 0.27 mm

Data collection

Rigaku SPIDER diffractometer
Absorption correction: none
6904 measured reflections
2011 independent reflections
1835 reflections with $I > 2\sigma(I)$
R_{int} = 0.025

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.097
S = 1.03
2011 reflections
164 parameters
29 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.40 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}
Absolute structure: Flack (1983), 905 Friedel pairs
Flack parameter: 0.08 (9)

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

D—H···A	D—H	H···A	D···A	D—H···A
C6—H6···O4^{i}	0.95	2.44	3.386 (3)	173

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

Data collection: *RAPID-AUTO* (Rigaku 2004); cell refinement: *RAPID-AUTO*; data reduction: *RAPID-AUTO*; 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: CI2867).

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supporting information

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S1. Comment

3,5-Dinitrobenzoyl chloride is a useful disinfectant and preservative (Saunders *et al.*, 1942; Liu *et al.*, 2000). It was also used as a derivatization reagent for azide determination by capillary electrophoresis (Gennaro *et al.*, 1993). We report here the crystal structure of the title compound.

The carbonyl chloride group is disordered over two orientations (Fig. 1). Except for a long N1—O3 distance [1.339 (3) Å] all other bond lengths and angles are within expected ranges. The molecule is approximately planar. The plane of the carbonyl chloride group forms a dihedral angle of 9.6 (4)° with the benzene ring in the major component [7.1 (4)° in the minor component]. The N1/O2/O3 and N2/O4/O5 nitro groups form dihedral angles of 1.9 (3) and 6.7 (4)°, respectively, with the benzene ring.

The crystal packing is stabilized by C—H···O hydrogen bonds (Table 1).

S2. Experimental

A sample of commercial 3,5-dinitrobenzoylchloride (Aldrich) was crystallized by slow evaporation of a solution in carbon tetrachloride.

S3. Refinement

The carbonyl chloride group is disordered over two orientations with occupancies of 0.505 (5) and 0.495 (5). The CO distance involving disordered atoms was restrained to 1.22 (1) Å and in each disorder component and the carbonyl chloride group was restrained to be planar. The displacement parameters of atoms CL1′, O1′, O1 and O3 were restrained to an approximate isotropic behaviour. H atoms were positioned geometrically (C—H = 0.95 Å) and were allowed to ride on the C atoms to which they are bonded, with U_{iso}(H) = 1.2U_{eq}(C).

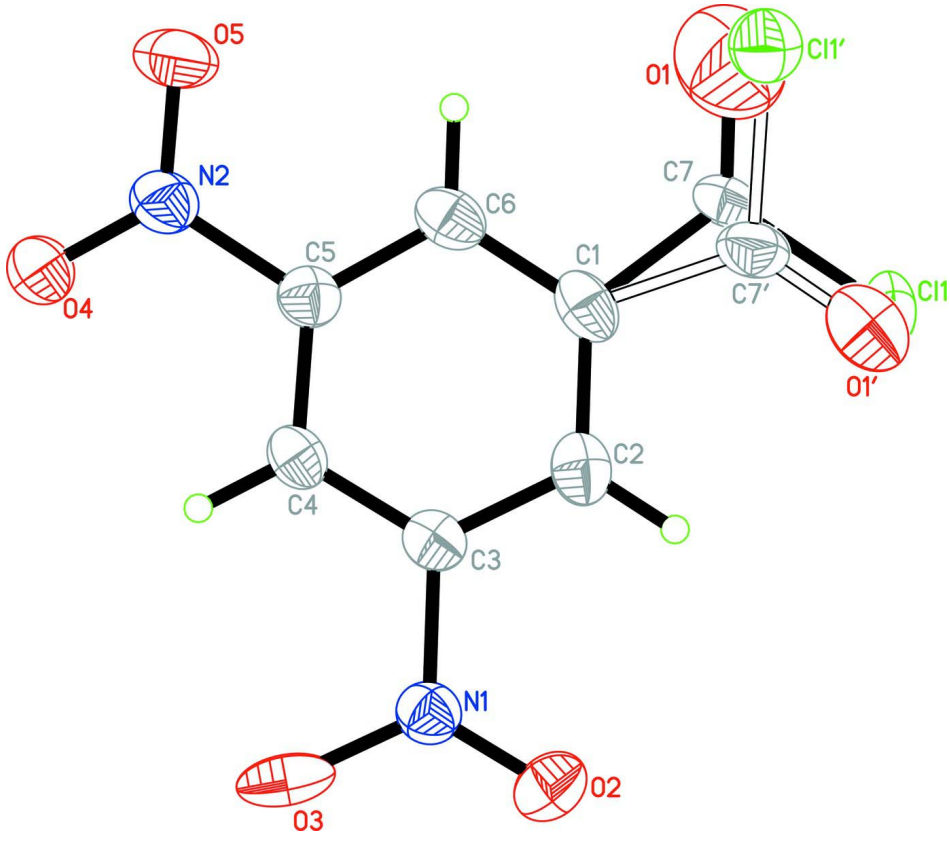

Figure 1

The molecular structure of the title compound, with the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. Both disorder components are shown.

3,5-Dinitrobenzoyl chloride

Crystal data

$C_7H_3ClN_2O_5$
$M_r = 230.56$
Orthorhombic, $Pna2_1$
Hall symbol: P 2c -2n
$a = 18.295$ (4) Å
$b = 8.3924$ (19) Å
$c = 5.7362$ (13) Å
$V = 880.7$ (3) Å^3
$Z = 4$

$F(000) = 464$
$D_x = 1.739$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2915 reflections
$\theta = 3.3$–$27.5°$
$\mu = 0.44$ mm^{-1}
$T = 93$ K
Block, colourless
0.37 × 0.33 × 0.27 mm

Data collection

Rigaku SPIDER diffractometer
Radiation source: Rotating Anode
Graphite monochromator
ω scans
6904 measured reflections
2011 independent reflections

1835 reflections with $I > 2\sigma(I)$
$R_{int} = 0.025$
$\theta_{max} = 27.5°$, $\theta_{min} = 3.3°$
$h = -23 \rightarrow 22$
$k = -10 \rightarrow 10$
$l = -7 \rightarrow 7$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.097$
$S = 1.03$
2011 reflections
164 parameters
29 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
$w = 1/[\sigma^2(F_o^2) + (0.0626P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.40$ e Å^{-3}
$\Delta\rho_{min} = -0.22$ e Å^{-3}
Absolute structure: Flack (1983), 905 Friedel pairs
Absolute structure parameter: 0.08 (9)

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}	Occ. (<1)
Cl1	0.43221 (12)	0.5812 (3)	0.3714 (5)	0.0418 (5)	0.505 (5)
C7	0.3864 (3)	0.4499 (11)	0.5519 (12)	0.0289 (16)	0.505 (5)
O1	0.3635 (5)	0.4892 (13)	0.7358 (18)	0.098 (5)	0.505 (5)
Cl1′	0.37524 (15)	0.5156 (3)	0.7640 (4)	0.0442 (5)	0.495 (5)
C7′	0.4064 (3)	0.4534 (13)	0.4960 (13)	0.035 (2)	0.495 (5)
O1′	0.4432 (5)	0.5374 (11)	0.377 (2)	0.083 (3)	0.495 (5)
C1	0.38140 (12)	0.2885 (3)	0.4413 (4)	0.0325 (5)	
C2	0.41455 (11)	0.2335 (3)	0.2394 (4)	0.0324 (5)	
H2	0.4468	0.2995	0.1526	0.039*	
C3	0.39917 (11)	0.0787 (2)	0.1677 (4)	0.0277 (4)	
C4	0.35290 (10)	−0.0199 (2)	0.2878 (4)	0.0276 (4)	
H4	0.3428	−0.1251	0.2355	0.033*	
C5	0.32173 (11)	0.0400 (2)	0.4872 (4)	0.0288 (4)	
C6	0.33439 (11)	0.1924 (2)	0.5677 (4)	0.0320 (5)	
H6	0.3115	0.2303	0.7057	0.038*	
N1	0.43482 (9)	0.0200 (2)	−0.0448 (3)	0.0308 (4)	
N2	0.27342 (10)	−0.0636 (2)	0.6268 (3)	0.0361 (4)	
O2	0.47388 (8)	0.10880 (19)	−0.1549 (3)	0.0384 (4)	
O3	0.42041 (8)	−0.1304 (2)	−0.1076 (3)	0.0426 (4)	
O4	0.25855 (9)	−0.19494 (18)	0.5508 (3)	0.0396 (4)	
O5	0.25255 (10)	−0.0136 (3)	0.8142 (4)	0.0579 (6)	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0443 (8)	0.0269 (9)	0.0541 (9)	−0.0110 (6)	0.0055 (7)	−0.0027 (8)
C7	0.033 (3)	0.026 (2)	0.028 (3)	0.006 (3)	0.003 (3)	−0.008 (3)
O1	0.108 (7)	0.085 (6)	0.100 (7)	0.003 (4)	−0.004 (5)	−0.006 (4)
Cl1′	0.0540 (9)	0.0372 (8)	0.0414 (9)	−0.0016 (7)	0.0034 (7)	0.0000 (8)
C7′	0.029 (3)	0.044 (3)	0.034 (4)	0.001 (3)	0.005 (3)	−0.014 (3)
O1′	0.104 (6)	0.056 (5)	0.088 (5)	−0.021 (4)	0.030 (4)	−0.030 (4)
C1	0.0386 (11)	0.0261 (10)	0.0328 (11)	0.0017 (8)	−0.0118 (9)	−0.0037 (9)
C2	0.0353 (11)	0.0284 (10)	0.0336 (11)	−0.0030 (8)	−0.0078 (9)	0.0027 (10)
C3	0.0295 (10)	0.0283 (10)	0.0253 (10)	0.0033 (7)	−0.0041 (8)	−0.0008 (8)
C4	0.0286 (9)	0.0258 (9)	0.0286 (11)	0.0003 (7)	−0.0061 (8)	−0.0030 (8)
C5	0.0280 (9)	0.0315 (10)	0.0268 (10)	−0.0007 (8)	−0.0032 (8)	−0.0012 (9)
C6	0.0352 (11)	0.0325 (10)	0.0283 (10)	0.0055 (9)	−0.0061 (9)	−0.0048 (9)
N1	0.0333 (9)	0.0310 (9)	0.0281 (9)	0.0011 (7)	−0.0009 (7)	−0.0007 (8)
N2	0.0370 (10)	0.0394 (10)	0.0320 (10)	−0.0078 (8)	0.0004 (9)	−0.0085 (9)
O2	0.0372 (8)	0.0431 (9)	0.0348 (9)	−0.0005 (6)	0.0045 (7)	0.0076 (7)
O3	0.0345 (8)	0.0728 (12)	0.0205 (7)	0.0114 (7)	0.0055 (6)	−0.0008 (9)
O4	0.0452 (9)	0.0383 (8)	0.0355 (8)	−0.0115 (7)	−0.0003 (7)	−0.0075 (7)
O5	0.0665 (12)	0.0637 (13)	0.0435 (12)	−0.0216 (10)	0.0226 (9)	−0.0250 (10)

Geometric parameters (Å, º)

Cl1—C7	1.728 (8)	C3—N1	1.468 (3)
C7—O1	1.182 (9)	C4—C5	1.374 (3)
C7—C1	1.499 (9)	C4—H4	0.95
Cl1′—C7′	1.720 (9)	C5—C6	1.380 (3)
C7′—O1′	1.190 (8)	C5—N2	1.476 (3)
C7′—C1	1.491 (11)	C6—H6	0.95
C1—C6	1.384 (3)	N1—O2	1.210 (2)
C1—C2	1.387 (3)	N1—O3	1.339 (3)
C2—C3	1.391 (3)	N2—O5	1.216 (3)
C2—H2	0.95	N2—O4	1.216 (2)
C3—C4	1.370 (3)		
O1—C7—C1	127.5 (9)	C2—C3—N1	117.96 (19)
O1—C7—Cl1	121.9 (9)	C3—C4—C5	117.00 (19)
C1—C7—Cl1	110.6 (4)	C3—C4—H4	121.5
O1′—C7′—C1	127.1 (9)	C5—C4—H4	121.5
O1′—C7′—Cl1′	121.3 (10)	C4—C5—C6	123.3 (2)
C1—C7′—Cl1′	111.6 (5)	C4—C5—N2	118.99 (18)
C6—C1—C2	121.02 (19)	C6—C5—N2	117.72 (19)
C6—C1—C7′	128.4 (3)	C5—C6—C1	118.0 (2)
C2—C1—C7′	110.5 (3)	C5—C6—H6	121.0
C6—C1—C7	110.1 (3)	C1—C6—H6	121.0
C2—C1—C7	128.9 (3)	O2—N1—O3	123.80 (18)
C1—C2—C3	118.0 (2)	O2—N1—C3	119.29 (18)

C1—C2—H2	121.0	O3—N1—C3	116.89 (16)
C3—C2—H2	121.0	O5—N2—O4	124.1 (2)
C4—C3—C2	122.7 (2)	O5—N2—C5	117.64 (18)
C4—C3—N1	119.31 (18)	O4—N2—C5	118.26 (18)
O1′—C7′—C1—C6	−174.9 (4)	C2—C3—C4—C5	−0.4 (3)
Cl1′—C7′—C1—C6	5.2 (4)	N1—C3—C4—C5	179.13 (17)
O1′—C7′—C1—C2	7.5 (3)	C3—C4—C5—C6	0.6 (3)
Cl1′—C7′—C1—C2	−172.4 (2)	C3—C4—C5—N2	−177.98 (18)
O1′—C7′—C1—C7	−161.7 (9)	C4—C5—C6—C1	−0.6 (3)
Cl1′—C7′—C1—C7	18.3 (8)	N2—C5—C6—C1	178.06 (18)
O1—C7—C1—C6	10.2 (3)	C2—C1—C6—C5	0.3 (3)
Cl1—C7—C1—C6	−169.9 (2)	C7′—C1—C6—C5	−177.1 (3)
O1—C7—C1—C2	−171.9 (4)	C7—C1—C6—C5	178.4 (2)
Cl1—C7—C1—C2	8.1 (4)	C4—C3—N1—O2	177.70 (19)
O1—C7—C1—C7′	−158.9 (9)	C2—C3—N1—O2	−2.7 (3)
Cl1—C7—C1—C7′	21.1 (8)	C4—C3—N1—O3	−1.0 (3)
C6—C1—C2—C3	0.0 (3)	C2—C3—N1—O3	178.60 (18)
C7′—C1—C2—C3	177.8 (2)	C4—C5—N2—O5	172.6 (2)
C7—C1—C2—C3	−177.8 (3)	C6—C5—N2—O5	−6.1 (3)
C1—C2—C3—C4	0.1 (3)	C4—C5—N2—O4	−5.4 (3)
C1—C2—C3—N1	−179.43 (17)	C6—C5—N2—O4	175.9 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C6—H6···O4^{i}	0.95	2.44	3.386 (3)	173

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