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ISSN 2414-3146

3-Nitrobenzaldehyde

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Received 5 January 2018
Accepted 15 January 2018

Edited by W. T. A. Harrison, University of Aberdeen, Scotland

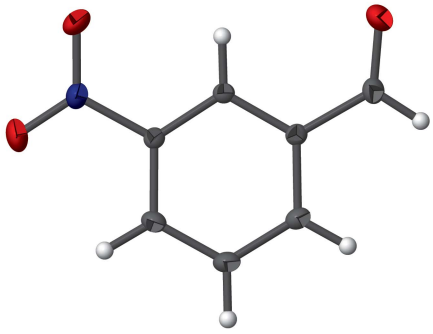
Keywords: crystal structure; π–π stacking interactions; nitrobenzaldehyde.

CCDC reference: 1816967

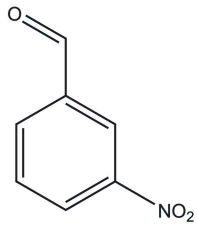
Structural data: full structural data are available from iucrdata.iucr.org

Polymorph I of the title compound, $C_7H_5NO_3$, is approximately planar: the dihedral angle between the benzene ring and the nitro group is 10.41 (4)° and the aldehyde O atom deviates from the ring plane by 0.165 (1) Å. In the crystal, aromatic π–π stacking interactions are observed [centroid–centroid separation = 3.7363 (5) Å].

3D view



Chemical scheme



Structure description

The existence of two polymorphic forms of the title compound, $C_7H_5NO_3$, has been known for almost eighty years (Lindpaintner, 1939); however, to date no crystal structure of the title compound has been reported. Here, we present the crystal structure of the stable polymorph (polymorph I). For the crystal structure of the closely related compound 2-nitrobenzaldehyde, see Coppens & Schmidt (1964) and Coppens (1964) and for the crystal structure of 4-nitrobenzalhyde, see Jackisch *et al.* (1989) and King & Bryant (1996).

The molecular structure of the title compound is shown in Fig. 1. The dihedral angle between the benzene ring and the nitro group is 10.41 (4)° and the aldehyde O atom deviates from the ring plane by 0.165 (1) Å. In the crystal, aromatic π–π stacking interactions are observed [centroid–centroid separation = 3.7363 (5) Å].

The melting point of the stable polymorph is 327 K, while the melting point of polymorph II is 322 K, as determined using the onset temperature of differential scanning calorimetry.

Synthesis and crystallization

A 100 mg mL^{-1} solution of 3-nitrobenzaldehyde (Merck, no indication of purity given) in acetone was filtered to obtain a clear yellow solution. Slow evaporation of a 1:1 mixture

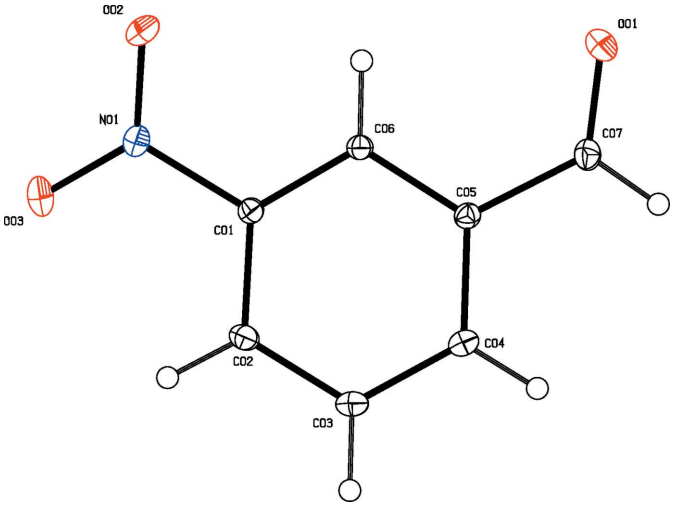


Figure 1
The molecular structure of the title compound, showing displacement ellipsoids drawn at the 30% probability level.

of this solution and heptane resulted in large colourless needle-shaped crystals of the title compound after two days.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1.

Table 1
Experimental details.

Crystal data	
Chemical formula	$C_7H_5NO_3$
M_r	151.12
Crystal system, space group	Monoclinic, $P2_1$
Temperature (K)	150
a, b, c (Å)	3.7363 (2), 7.0071 (3), 12.5877 (6)
β (°)	94.8144 (16)
V (Å^3)	328.39 (3)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.12
Crystal size (mm)	0.48 × 0.17 × 0.09
Data collection	
Diffractometer	Bruker D8 Quest *APEX3*
Absorption correction	Multi-scan (*SADABS*; Bruker, 2011)
T_{min}, T_{max}	0.713, 0.748
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	18662, 4005, 3735
R_{int}	0.021
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.909
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.030, 0.089, 1.05
No. of reflections	4005
No. of parameters	100
No. of restraints	1
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.38, −0.21
Absolute structure	Flack x determined using 1595 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013).
Absolute structure parameter	−0.02 (13)

Computer programs: *APEX3* (Bruker, 2012), *PEAKREF* (Schreurs, 2013), *SAINT* (Bruker, 2012), *SHELXT2014/4* (Sheldrick, 2015*a*), *SHELXL2016/6* (Sheldrick, 2015*b*), *PLATON* (Spek, 2009) and *ShelXLe* (Hübschle *et al.*, 2011).

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full crystallographic data

IUCrData (2018). **3**, x180092 [https://doi.org/10.1107/S2414314618000925]

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Crystal data

$C_7H_5NO_3$
M_r = 151.12
Monoclinic, $P2_1$
a = 3.7363 (2) Å
b = 7.0071 (3) Å
c = 12.5877 (6) Å
β = 94.8144 (16)°
V = 328.39 (3) Å^3
Z = 2
$F(000)$ = 156
D_x = 1.528 Mg m^{-3}
Melting point: 327 K
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9919 reflections
θ = 2.9–40.3°
μ = 0.12 mm^{-1}
T = 150 K
Needle, colourless
0.48 × 0.17 × 0.09 mm

Data collection

Bruker D8 Quest APEX3 diffractometer
Radiation source: sealed tube
Graphite monochromator
Detector resolution: 10.4 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2011)
T_{min} = 0.713, T_{max} = 0.748
18662 measured reflections
4005 independent reflections
3735 reflections with $I > 2\sigma(I)$
R_{int} = 0.021
θ_{max} = 40.3°, θ_{min} = 3.3°
h = −6→6
k = −12→12
l = −22→22

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.089
S = 1.05
4005 reflections
100 parameters
1 restraint
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
$w = 1/[\sigma^2(F_o^2) + (0.0643P)^2 + 0.0066P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.38 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}
Absolute structure: Flack x determined using 1595 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013).
Absolute structure parameter: −0.02 (13)

Special details

Experimental. Polymorph I

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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O01	0.3805 (2)	0.36964 (11)	0.47803 (5)	0.02774 (14)
O02	0.5659 (2)	0.81726 (10)	0.79109 (6)	0.02858 (15)
O03	0.3141 (2)	0.75360 (14)	0.93565 (6)	0.03273 (17)
N01	0.39008 (18)	0.71338 (9)	0.84537 (5)	0.01838 (11)
C01	0.26607 (18)	0.52935 (9)	0.79964 (5)	0.01429 (10)
C02	0.1223 (2)	0.39635 (11)	0.86644 (6)	0.01736 (12)
H02	0.101303	0.424953	0.939430	0.021*
C03	0.0102 (2)	0.22081 (11)	0.82413 (6)	0.01891 (13)
H03	−0.087439	0.127606	0.868273	0.023*
C04	0.0419 (2)	0.18223 (10)	0.71667 (6)	0.01706 (12)
H04	−0.036577	0.062838	0.687503	0.020*
C05	0.18828 (17)	0.31822 (9)	0.65163 (5)	0.01416 (10)
C06	0.30344 (19)	0.49536 (10)	0.69286 (5)	0.01405 (10)
H06	0.403384	0.588642	0.649244	0.017*
C07	0.2287 (2)	0.26924 (11)	0.53892 (6)	0.01863 (12)
H07	0.129789	0.151915	0.512489	0.022*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O01	0.0390 (3)	0.0266 (3)	0.0188 (2)	−0.0037 (3)	0.0094 (2)	−0.0013 (2)
O02	0.0373 (3)	0.0184 (2)	0.0311 (3)	−0.0106 (3)	0.0088 (3)	−0.0032 (2)
O03	0.0461 (4)	0.0320 (3)	0.0209 (3)	−0.0065 (3)	0.0074 (3)	−0.0112 (3)
N01	0.0201 (3)	0.0162 (2)	0.0187 (2)	−0.00059 (19)	0.00057 (19)	−0.00306 (18)
C01	0.0145 (2)	0.0137 (2)	0.0148 (2)	−0.00082 (17)	0.00209 (18)	−0.00065 (17)
C02	0.0185 (3)	0.0190 (3)	0.0148 (2)	−0.0012 (2)	0.0030 (2)	0.00250 (19)
C03	0.0203 (3)	0.0171 (3)	0.0196 (3)	−0.0025 (2)	0.0034 (2)	0.0042 (2)
C04	0.0173 (3)	0.0139 (2)	0.0200 (3)	−0.00153 (19)	0.0015 (2)	0.0017 (2)
C05	0.0143 (2)	0.0129 (2)	0.0153 (2)	0.00052 (19)	0.00169 (18)	0.00008 (19)
C06	0.0148 (2)	0.0132 (2)	0.0145 (2)	−0.00045 (17)	0.00272 (17)	0.00045 (17)
C07	0.0216 (3)	0.0172 (2)	0.0174 (3)	0.0010 (2)	0.0028 (2)	−0.0031 (2)

Geometric parameters (Å, º)

O01—C07	1.2152 (10)	C03—C04	1.3941 (10)
O02—N01	1.2262 (9)	C03—H03	0.9500
O03—N01	1.2271 (9)	C04—C05	1.3974 (9)
N01—C01	1.4711 (9)	C04—H04	0.9500

C01—C06	1.3836 (9)	C05—C06	1.3992 (10)
C01—C02	1.3928 (9)	C05—C07	1.4797 (9)
C02—C03	1.3909 (11)	C06—H06	0.9500
C02—H02	0.9500	C07—H07	0.9500
O03—N01—O02	123.79 (8)	C03—C04—C05	120.36 (6)
O03—N01—C01	118.29 (7)	C03—C04—H04	119.8
O02—N01—C01	117.92 (6)	C05—C04—H04	119.8
C06—C01—C02	123.15 (6)	C04—C05—C06	120.73 (6)
C06—C01—N01	118.48 (6)	C04—C05—C07	118.69 (6)
C02—C01—N01	118.36 (6)	C06—C05—C07	120.56 (6)
C03—C02—C01	118.65 (6)	C01—C06—C05	117.39 (6)
C03—C02—H02	120.7	C01—C06—H06	121.3
C01—C02—H02	120.7	C05—C06—H06	121.3
C02—C03—C04	119.72 (6)	O01—C07—C05	124.13 (7)
C02—C03—H03	120.1	O01—C07—H07	117.9
C04—C03—H03	120.1	C05—C07—H07	117.9
O03—N01—C01—C06	170.48 (8)	C03—C04—C05—C06	−0.38 (10)
O02—N01—C01—C06	−10.01 (10)	C03—C04—C05—C07	177.92 (7)
O03—N01—C01—C02	−10.63 (11)	C02—C01—C06—C05	0.25 (10)
O02—N01—C01—C02	168.88 (7)	N01—C01—C06—C05	179.09 (6)
C06—C01—C02—C03	−0.08 (11)	C04—C05—C06—C01	−0.02 (9)
N01—C01—C02—C03	−178.92 (7)	C07—C05—C06—C01	−178.30 (6)
C01—C02—C03—C04	−0.34 (11)	C04—C05—C07—O01	−173.33 (8)
C02—C03—C04—C05	0.56 (11)	C06—C05—C07—O01	4.98 (12)