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5-Nitro-2-(piperidin-1-yl)benzaldehyde

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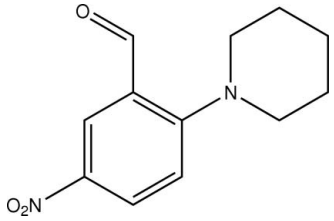
Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.002 Å; R factor = 0.045; wR factor = 0.111; data-to-parameter ratio = 13.4.

In the structure of the title compound, $C_{12}H_{14}N_2O_3$, the piperidine ring adopts a chair conformation and the aryl substitutent occupies an equatorial position.

Related literature

For the toxicity of nitroaromatics, see: Cronin *et al.* (1998); Shinoda *et al.* (1998). For piperidine ring conformations, see: Parkin *et al.* (2004). For ring conformational analysis, see: Cremer & Pople (1975). For reference bond lengths, see: Allen *et al.* (1987) and for bond angles, see; Codding & Kerr (1978).



Experimental

Crystal data

$C_{12}H_{14}N_2O_3$
M_r = 234.25
Triclinic, $P\overline{1}$
a = 5.686 (2) Å
b = 10.102 (5) Å
c = 10.221 (4) Å
α = 80.767 (2)°
β = 80.733 (3)°
γ = 86.034 (2)°
V = 571.4 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 295 K
0.30 × 0.25 × 0.25 mm

Data collection

Nonius KappaCCCD area-detector diffractometer
Absorption correction: none
11460 measured reflections
3280 independent reflections
2239 reflections with $I > 2.0\sigma(I)$
R_{int} = 0.03

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.111
S = 1.02
2058 reflections
154 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}

Data collection: *COLLECT* (Nonius, 1997); cell refinement: *DENZO/SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO/SCALEPACK*; program(s) used to solve structure: *SIR2004* (Burla *et al.*, 2005); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *PLATON* (Spek, 2009); software used to prepare material for publication: *CRYSTALS*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG2664).

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supplementary materials

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5-Nitro-2-(piperidin-1-yl)benzaldehyde

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Comment

Nitroaromatics are reactional intermediate compounds in chemical synthesis well known for their toxicity (Cronin *et al.*, 1998; Shinoda *et al.*,1998). Here we report the single-crystal X-ray determination of the title compound in order to have a best insight of its structure and then to undertake a study of its possible toxic activity. The molecular structure of this compound and its atomic labeling scheme are shown in Fig.1. In this one, the piperidine ring, (N2/C8/C9/C10/C11/C12), as previously reported (Parkin *et al.*, 2004), assumes a chair conformation, with the torsion angles mean value equal to 56.45 °, the puckering parameters (Cremer & Pople, 1975), being: Q = 0.5670 (17) Å, Phi = 365°, Theta = 1.59 (16)°. The C4 atom is in an equatorial position with respect to the piperidine ring. The system defined by (C1/C2/C3/C4/C5/C6), essentially planar, with a maximum deviation of 0.014 Å is an aromatic ring, according to the range of C—C bond lengths (1.3750 (19) Å to 1.4174 (17) Å) and C—C—C bond angles (117.61 (12) ° to 121.51 (12) °) (Peneloppe *et al.*, 1978). Values of selected bond lengths and angles are reported in table 1. The nitro group is confirmed throughout the N—O bond length characteristics, since distances d(O2—N1)=1.2195 (16)Å and d(O1—N1) =1.221 (16)Å are consistent with those encountered in the nitro group (Allen *et al.*, 1987). Besides d(C1—N1)=1.4553Å corresponds to a single bond length between an aromatic carbon and a nitrogen (Car—NO2). The bond length d(C7—O3)=1.2049 (18) Å, characterizes a normal double bond (CO) involved in an aldehyde function (Allen *et al.*, 1987).

Experimental

[6.52 ml, (66 mmol)] of piperidine and [5.54 g, (66 mmol)] of sodium hydrogenocarbonate($NaHCO_3$) were added to [8 g, (43 mmol)] of distilled ethanol reflux during 24 h under shelter moister. After cooling to ambient temperature, the mixture was poured into 150 ml of dichloromethane then washed twice with 50 ml of water each time. After decantation, the organic layer was dried on magnesium sulfate, filtered and concentrated under reduced pressure. The residue was purifie by flash chromatography on silica gel using DCM/hexane(80/20)*v*/*v*. 8.8 g of the title compound were obtained with 85.51% yields. The melting point is 388k.

Refinement

All the H atoms were found by difference fourier. their position and displacement parameters U_{iso}(H) were refined to regularize their geometry(C—H in the range 0.94–0.99 Å) and U_{iso}(H) (1.2 times U_{eq} of the parent atom), after which their positions were refined isotropically with riding constraints.

Figures

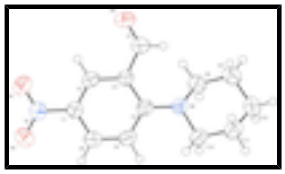

Fig. 1. The title compound structure and atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are shown as spheres of arbitary radius.

5-Nitro-2-(piperidin-1-yl)benzaldehyde

Crystal data

$C_{12}H_{14}N_2O_3$	$Z = 2$
$M_r = 234.25$	$F_{000} = 248$
Triclinic, $P\overline{1}$	$D_x = 1.362$ Mg m^{-3}
Hall symbol: -P 1	Melting point: 388 K
$a = 5.686$ (2) Å	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$b = 10.102$ (5) Å	Cell parameters from 3280 reflections
$c = 10.221$ (4) Å	$\theta = 2$–30º
$\alpha = 80.767$ (2)º	$\mu = 0.10$ mm^{-1}
$\beta = 80.733$ (3)º	$T = 295$ K
$\gamma = 86.034$ (2)º	Prism, orange
$V = 571.4$ (4) Å^3	0.30 × 0.25 × 0.25 mm

Data collection

Nonius KappaCCCD area detector diffractometer	2239 reflections with $I > 2.0\sigma(I)$
Monochromator: graphite	$R_{int} = 0.03$
$T = 295$ K	$\theta_{max} = 30.2$º
φ scans	$\theta_{min} = 2.0$º
Absorption correction: none	$h = 0 \rightarrow 8$
11460 measured reflections	$k = -14 \rightarrow 14$
3280 independent reflections	$l = -13 \rightarrow 14$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.045$	Method = Modified Sheldrick $w = 1/[\sigma^2(F^2) + (0.06P)^2 + 0.1P]$, where $P = (\max(F_o^2,0) + 2F_c^2)/3$
$wR(F^2) = 0.111$	$(\Delta/\sigma)_{max} = 0.0004$
$S = 1.02$	$\Delta\rho_{max} = 0.16$ e Å^{-3}
2058 reflections	$\Delta\rho_{min} = -0.15$ e Å^{-3}
154 parameters	Extinction correction: None

Primary atom site location: structure-invariant direct methods

Special details

Refinement. We had 3280 independent reflections but 2058 reflections were used in the refinement, instead of 3280 because the refinement was carried out under conditions I > 3σ(I).

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	−0.2540 (2)	−0.19297 (12)	0.65442 (13)	0.0538
N2	0.1530 (2)	0.26552 (11)	0.75678 (11)	0.0494
C1	−0.1479 (2)	−0.07454 (13)	0.68028 (13)	0.0446
C2	0.0235 (2)	−0.08957 (12)	0.76296 (12)	0.0437
C3	0.1255 (2)	0.02260 (12)	0.78871 (12)	0.0421
C4	0.0534 (2)	0.15293 (12)	0.73040 (12)	0.0428
C5	−0.1174 (3)	0.16295 (13)	0.64329 (14)	0.0514
C6	−0.2181 (3)	0.05102 (14)	0.61906 (14)	0.0516
C7	0.3268 (3)	−0.00219 (15)	0.86587 (14)	0.0530
C8	0.1313 (3)	0.28678 (15)	0.89744 (13)	0.0539
C9	0.3194 (3)	0.37797 (17)	0.91473 (16)	0.0665
C10	0.3053 (4)	0.51042 (17)	0.82255 (17)	0.0676
C11	0.3178 (3)	0.48655 (15)	0.67935 (16)	0.0630
C12	0.1293 (3)	0.39255 (14)	0.66607 (15)	0.0573
O1	−0.1742 (2)	−0.30357 (10)	0.69841 (14)	0.0772
O2	−0.4183 (2)	−0.17831 (12)	0.58925 (14)	0.0796
O3	0.3735 (2)	−0.10961 (12)	0.92869 (13)	0.0761
H2	0.0775	−0.1770	0.8003	0.0524*
H5	−0.1691	0.2482	0.6013	0.0617*
H6	−0.3366	0.0604	0.5608	0.0619*
H7	0.4264	0.0706	0.8620	0.0636*
H81	0.1423	0.1992	0.9549	0.0647*
H82	−0.0258	0.3295	0.9232	0.0647*
H91	0.3019	0.3901	1.0089	0.0798*
H92	0.4712	0.3336	0.8926	0.0798*
H101	0.4289	0.5645	0.8298	0.0812*
H102	0.1507	0.5572	0.8515	0.0812*
H111	0.2952	0.5712	0.6206	0.0756*
H112	0.4750	0.4465	0.6503	0.0756*
H121	−0.0316	0.4328	0.6880	0.0688*
H122	0.1462	0.3734	0.5738	0.0688*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0571 (7)	0.0479 (7)	0.0585 (7)	−0.0037 (5)	−0.0156 (6)	−0.0069 (5)
N2	0.0716 (8)	0.0387 (5)	0.0387 (5)	−0.0033 (5)	−0.0132 (5)	−0.0032 (4)
C1	0.0483 (7)	0.0414 (7)	0.0447 (7)	−0.0017 (5)	−0.0089 (5)	−0.0067 (5)

C2	0.0490 (7)	0.0396 (6)	0.0400 (6)	0.0018 (5)	−0.0067 (5)	−0.0001 (5)
C3	0.0477 (7)	0.0415 (6)	0.0365 (6)	−0.0001 (5)	−0.0085 (5)	−0.0023 (5)
C4	0.0519 (7)	0.0396 (6)	0.0361 (6)	0.0001 (5)	−0.0066 (5)	−0.0043 (5)
C5	0.0636 (9)	0.0401 (7)	0.0508 (7)	0.0056 (6)	−0.0187 (6)	−0.0011 (5)
C6	0.0570 (8)	0.0492 (7)	0.0510 (7)	0.0025 (6)	−0.0204 (6)	−0.0044 (6)
C7	0.0565 (8)	0.0502 (8)	0.0527 (8)	−0.0027 (6)	−0.0168 (6)	−0.0006 (6)
C8	0.0693 (9)	0.0528 (8)	0.0409 (7)	−0.0078 (7)	−0.0078 (6)	−0.0093 (6)
C9	0.0804 (11)	0.0674 (10)	0.0576 (9)	−0.0135 (8)	−0.0267 (8)	−0.0066 (8)
C10	0.0807 (11)	0.0571 (9)	0.0718 (10)	−0.0192 (8)	−0.0251 (9)	−0.0094 (8)
C11	0.0770 (11)	0.0475 (8)	0.0622 (9)	−0.0086 (7)	−0.0112 (8)	0.0018 (7)
C12	0.0848 (11)	0.0394 (7)	0.0501 (8)	−0.0030 (7)	−0.0215 (7)	−0.0026 (6)
O1	0.0870 (8)	0.0420 (6)	0.1096 (10)	−0.0025 (5)	−0.0414 (7)	−0.0056 (6)
O2	0.0848 (8)	0.0650 (7)	0.1011 (10)	−0.0102 (6)	−0.0519 (8)	−0.0082 (7)
O3	0.0848 (8)	0.0595 (7)	0.0874 (9)	−0.0029 (6)	−0.0478 (7)	0.0128 (6)

Geometric parameters (Å, °)

N1—O1	1.2210 (16)	C1—C6	1.3839 (19)
N1—C1	1.4553 (18)	C6—H61	0.959
N1—O2	1.2195 (16)	C8—C9	1.507 (2)
C3—C4	1.4174 (17)	C8—H81	0.984
C3—C2	1.3878 (19)	C8—H82	0.980
C3—C7	1.4773 (19)	C12—C11	1.516 (2)
C4—N2	1.3868 (17)	C12—H122	0.981
C4—C5	1.4081 (19)	C12—H121	0.982
N2—C8	1.4720 (18)	C9—C10	1.513 (2)
N2—C12	1.4694 (17)	C9—H92	0.956
C5—C6	1.376 (2)	C9—H91	0.978
C5—H51	0.949	C11—C10	1.511 (2)
C2—C1	1.3750 (19)	C11—H112	0.977
C2—H21	0.955	C11—H111	0.977
C7—O3	1.2049 (18)	C10—H101	0.939
C7—H71	0.951	C10—H102	0.993
O1—N1—C1	118.62 (12)	C9—C8—H81	111.4
O1—N1—O2	122.42 (13)	N2—C8—H82	108.6
C1—N1—O2	118.96 (12)	C9—C8—H82	108.4
C4—C3—C2	120.29 (12)	H81—C8—H82	108.4
C4—C3—C7	122.63 (12)	N2—C12—C11	109.92 (13)
C2—C3—C7	116.75 (12)	N2—C12—H122	108.8
C3—C4—N2	120.58 (12)	C11—C12—H122	110.7
C3—C4—C5	117.61 (12)	N2—C12—H121	109.0
N2—C4—C5	121.79 (12)	C11—C12—H121	110.9
C4—N2—C8	118.01 (11)	H122—C12—H121	107.5
C4—N2—C12	118.40 (11)	C8—C9—C10	110.77 (14)
C8—N2—C12	111.78 (11)	C8—C9—H92	107.4
C4—C5—C6	121.51 (12)	C10—C9—H92	109.5
C4—C5—H51	120.3	C8—C9—H91	109.3
C6—C5—H51	118.1	C10—C9—H91	112.0
C3—C2—C1	119.98 (12)	H92—C9—H91	107.7

C3—C2—H21	119.5	C12—C11—C10	111.64 (13)
C1—C2—H21	120.4	C12—C11—H112	108.8
C3—C7—O3	123.42 (14)	C10—C11—H112	108.6
C3—C7—H71	117.1	C12—C11—H111	108.5
O3—C7—H71	119.4	C10—C11—H111	110.5
N1—C1—C2	119.43 (12)	H112—C11—H111	108.8
N1—C1—C6	119.35 (12)	C9—C10—C11	110.19 (14)
C2—C1—C6	121.20 (12)	C9—C10—H101	110.3
C1—C6—C5	119.35 (13)	C11—C10—H101	110.4
C1—C6—H61	120.6	C9—C10—H102	107.9
C5—C6—H61	120.1	C11—C10—H102	109.6
N2—C8—C9	110.88 (12)	H101—C10—H102	108.3
N2—C8—H81	109.1		
C12—N2—C8—C9	−58.84 (17)	C8—C9—C10—C11	−54.06 (18)
C8—N2—C12—C11	58.21 (16)	C9—C10—C11—C12	54.45 (18)
N2—C8—C9—C10	56.24 (17)	C10—C11—C12—N2	−56.22 (17)

Fig. 1