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Crystal structure of piperidinium 4-nitrophenolate

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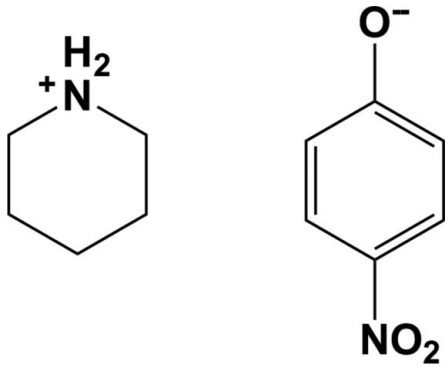
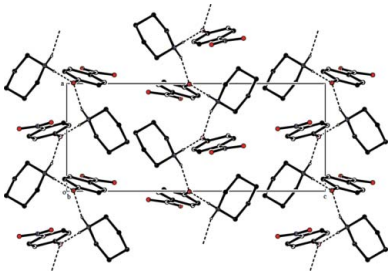
In the title molecular salt, $C_5H_{12}N^+\cdot C_6H_4NO_3^-$, the piperidine ring adopts a chair conformation and the cation is protonated at the N atom. In the anion, the nitro group is twisted at an angle of 10.30 (11)° with respect to the attached benzene ring. In the crystal, N—H···O hydrogen bonds link adjacent anions and cations into infinite chains propagating along [100]. The chains are linked by C—H···π interactions, forming sheets lying parallel to (001).

1. Chemical context

Piperidine derivatives exhibit a broad-spectrum of biological activities such as anti-bacterial and anti-cancer (Parthiban *et al.*, 2005). Nitro-aromatics are widely used either as materials or as intermediates in explosives, dyestuffs, pesticides and organic synthesis (Yan *et al.*, 2006). We report herein on the synthesis and crystal structure of the title molecular salt, prepared by mixing piperidine with 4-nitrophenol.





2. Structural commentary

The molecular structure of the title compound is illustrated in Fig. 1. The geometric parameters are close to those reported for similar structures *viz.* 1-acetyl-*c*-3,*t*-3-dimethyl-*r*-2,*c*-6-diphenylpiperidin-4-one (Aravindhan *et al.*, 2009), 4-nitrophenol-piperazine (2/1) (Nagapandiselvi *et al.*, 2013) and 2-carboxylatopyridinium-4-nitrophenol (1/1) (Sankar *et al.*, 2014). The piperidine ring (C8–C11/N2/C12) adopts a chair conformation with puckering parameters of Q = 0.5601 (17) Å, θ = 1.80 (17) and φ = 19 (10)°. The nitro group (N1/O2/O3) is twisted at an angle of 10.30 (11)° with respect to the attached benzene ring (C1–C6).

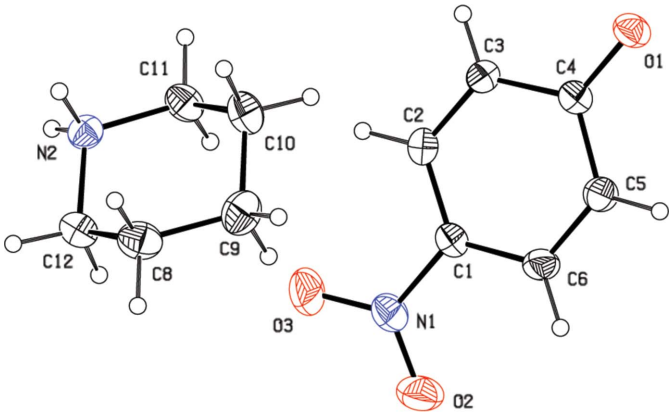


Figure 1
The molecular structure of the title salt, showing the atom labelling. Displacement ellipsoids are drawn at the 30% probability level.

3. Supramolecular features

In the crystal, adjacent cations and anions are linked by the N—H···O hydrogen bonds, which generate infinite chains along [100] (see Table 1 and Fig. 2). The chains are linked by C—H···π interactions, forming sheets lying parallel to the *ab* plane (Table 1).

4. Synthesis and crystallization

Piperidine (0.85 g) and 4-nitrophenol (1.39) in an equimolar (1:1) ratio were added to methanol as solvent and the mixture was stirred for 2 h, giving a clear solution. The solution was filtered into a beaker and sealed with parafilm and kept at room temperature for one week. Colourless crystals suitable for X-ray diffraction analysis were obtained after one week.

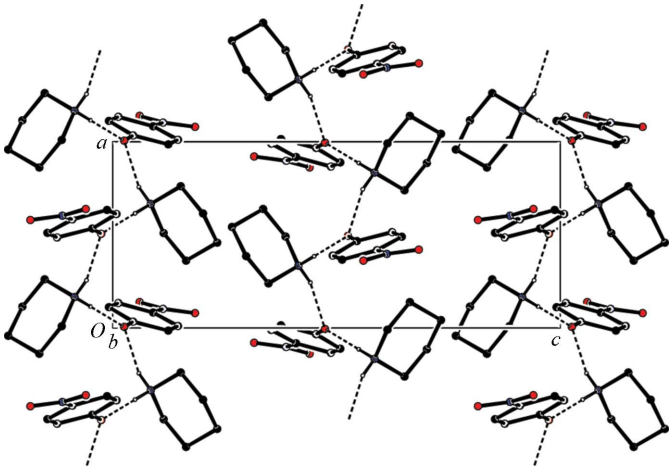


Figure 2
The crystal packing of the title salt, viewed along the *b* axis. Hydrogen bonds are shown as dashed lines (see Table 1 for details; H atoms not involved in hydrogen bonding have been omitted for clarity).

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

*Cg*1 is the centroid of the C1–C6 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*A*···O1^{i}	0.90	1.92	2.788 (2)	161
N2—H2*B*···O1^{ii}	0.90	1.80	2.6985 (15)	175
C6—H6···*Cg*1^{iii}	0.93	2.75	3.428 (3)	130

Symmetry codes: (i) $-x, y-\frac{1}{2}, -z+\frac{1}{2}$; (ii) $-x+\frac{1}{2}, -y+1, z+\frac{1}{2}$; (iii) $x+\frac{1}{2}, -y+\frac{1}{2}, -z$.

5. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The N-bound and C-bound H atoms were positioned geometrically and refined using a riding model: N—H = 0.90, C—H = 0.93 and 0.97 Å for CH and CH_2 H atoms, respectively, and with U_{iso}(H) = 1.2U_{eq}(N,C).

Acknowledgements

The authors wish to acknowledge the SAIF, IIT, Madras, for the data collection.

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Table 2
Experimental details.

Crystal data	
Chemical formula	$C_5H_{12}N^+·C_6H_4NO_3^-$
M_r	224.26
Crystal system, space group	Orthorhombic, $P2_12_12_1$
Temperature (K)	295
a, *b*, *c* (Å)	6.867 (5), 10.121 (4), 16.497 (6)
V (Å^3)	1146.6 (10)
Z	4
Radiation type	Mo *K*α
μ (mm^{-1})	0.10
Crystal size (mm)	0.26 × 0.22 × 0.20
Data collection	
Diffractometer	Bruker Kappa APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Sheldrick, 1996)
T_{min}, T_{max}	0.976, 0.981
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	6505, 2908, 2612
R_{int}	0.017
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.673
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.035, 0.091, 1.04
No. of reflections	2908
No. of parameters	146
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.13, −0.16

Computer programs: *APEX2* and *SAINT* (Bruker, 2004), *SHELXS97* and *SHELXL97* (Sheldrick, 2008) and *PLATON* (Spek, 2009).

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Crystal structure of piperidinium 4-nitrophenolate

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Computing details

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT* (Bruker, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008) and *PLATON* (Spek, 2009).

Piperidinium 4-nitrophenolate

Crystal data

$C_5H_{12}N^+·C_6H_4NO_3^-$
M_r = 224.26
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
a = 6.867 (5) Å
b = 10.121 (4) Å
c = 16.497 (6) Å
V = 1146.6 (10) Å^3
Z = 4
$F(000)$ = 480
D_x = 1.299 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 658 reflections
θ = 2.4–28.6°
μ = 0.10 mm^{-1}
T = 295 K
Block, colourless
0.26 × 0.22 × 0.20 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω and φ scan
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.976, T_{max} = 0.981
6505 measured reflections
2908 independent reflections
2612 reflections with $I > 2\sigma(I)$
R_{int} = 0.017
θ_{max} = 28.6°, θ_{min} = 2.4°
h = −6→9
k = −11→13
l = −22→10

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.091
S = 1.04
2908 reflections
146 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
$w = 1/[\sigma^2(F_o^2) + (0.0452P)^2 + 0.1138P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.13 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.119 (6)

Special details

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.

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 $F^2 > 2\text{sigma}(F^2)$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.08265 (18)	0.22326 (12)	0.09031 (7)	0.0380 (3)
C2	0.0102 (2)	0.31257 (12)	0.14583 (7)	0.0404 (3)
H2	−0.0220	0.2848	0.1979	0.049*
C3	−0.0144 (2)	0.44223 (12)	0.12419 (7)	0.0403 (3)
H3	−0.0638	0.5014	0.1621	0.048*
C4	0.03355 (18)	0.48874 (11)	0.04556 (7)	0.0373 (3)
C5	0.1115 (2)	0.39408 (13)	−0.00839 (8)	0.0474 (3)
H5	0.1491	0.4211	−0.0600	0.057*
C6	0.1336 (2)	0.26352 (13)	0.01258 (8)	0.0465 (3)
H6	0.1820	0.2029	−0.0247	0.056*
C8	0.6387 (2)	0.21154 (17)	0.33603 (10)	0.0570 (4)
H8A	0.7057	0.2749	0.3702	0.068*
H8B	0.7348	0.1503	0.3149	0.068*
C9	0.5422 (3)	0.28329 (19)	0.26652 (10)	0.0626 (4)
H9A	0.6386	0.3342	0.2371	0.075*
H9B	0.4854	0.2197	0.2294	0.075*
C10	0.3854 (3)	0.37423 (16)	0.29817 (10)	0.0545 (4)
H10A	0.4442	0.4428	0.3312	0.065*
H10B	0.3199	0.4164	0.2530	0.065*
C11	0.2391 (2)	0.29893 (14)	0.34801 (9)	0.0480 (3)
H11A	0.1444	0.3601	0.3703	0.058*
H11B	0.1703	0.2370	0.3135	0.058*
C12	0.4907 (2)	0.13639 (13)	0.38617 (9)	0.0488 (3)
H12A	0.4341	0.0663	0.3537	0.059*
H12B	0.5549	0.0963	0.4324	0.059*
N1	0.10251 (18)	0.08685 (11)	0.11247 (8)	0.0487 (3)
N2	0.33415 (17)	0.22610 (10)	0.41493 (6)	0.0405 (3)
H2A	0.2438	0.1784	0.4415	0.049*
H2B	0.3851	0.2845	0.4502	0.049*
O1	0.00554 (15)	0.61052 (8)	0.02564 (6)	0.0458 (2)
O2	0.1387 (2)	0.00445 (11)	0.06015 (8)	0.0730 (4)
O3	0.0795 (2)	0.05548 (12)	0.18390 (7)	0.0771 (4)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0355 (6)	0.0359 (5)	0.0427 (6)	0.0007 (5)	−0.0004 (5)	0.0057 (5)
C2	0.0423 (6)	0.0453 (6)	0.0337 (5)	−0.0013 (6)	−0.0019 (5)	0.0049 (5)
C3	0.0434 (7)	0.0414 (6)	0.0361 (6)	0.0028 (5)	0.0005 (5)	−0.0033 (5)
C4	0.0324 (6)	0.0364 (6)	0.0431 (6)	−0.0006 (5)	0.0018 (5)	0.0030 (5)
C5	0.0545 (8)	0.0445 (7)	0.0432 (7)	0.0065 (6)	0.0179 (6)	0.0089 (5)
C6	0.0517 (8)	0.0412 (6)	0.0466 (7)	0.0096 (6)	0.0155 (6)	0.0019 (5)
C8	0.0425 (7)	0.0574 (8)	0.0710 (10)	0.0055 (7)	0.0071 (7)	−0.0019 (7)
C9	0.0635 (10)	0.0723 (10)	0.0520 (8)	−0.0044 (9)	0.0162 (8)	0.0073 (8)
C10	0.0568 (9)	0.0489 (8)	0.0577 (8)	−0.0017 (7)	−0.0023 (7)	0.0146 (7)
C11	0.0388 (7)	0.0459 (7)	0.0594 (8)	0.0031 (6)	−0.0029 (6)	0.0053 (6)
C12	0.0495 (8)	0.0408 (6)	0.0559 (7)	0.0053 (6)	−0.0015 (7)	0.0016 (6)
N1	0.0516 (7)	0.0396 (6)	0.0547 (7)	−0.0015 (5)	0.0013 (6)	0.0090 (5)
N2	0.0433 (6)	0.0377 (5)	0.0406 (5)	−0.0053 (4)	0.0014 (4)	−0.0029 (4)
O1	0.0494 (5)	0.0346 (4)	0.0535 (5)	0.0023 (4)	0.0079 (5)	0.0061 (4)
O2	0.1004 (10)	0.0403 (5)	0.0782 (8)	0.0118 (6)	0.0251 (7)	0.0021 (5)
O3	0.1256 (13)	0.0504 (6)	0.0554 (6)	−0.0028 (7)	−0.0012 (7)	0.0188 (5)

Geometric parameters (Å, º)

C1—C2	1.3798 (18)	C9—C10	1.510 (2)
C1—C6	1.3902 (18)	C9—H9A	0.9700
C1—N1	1.4347 (17)	C9—H9B	0.9700
C2—C3	1.3703 (18)	C10—C11	1.506 (2)
C2—H2	0.9300	C10—H10A	0.9700
C3—C4	1.4187 (17)	C10—H10B	0.9700
C3—H3	0.9300	C11—N2	1.4793 (18)
C4—O1	1.2900 (15)	C11—H11A	0.9700
C4—C5	1.4129 (18)	C11—H11B	0.9700
C5—C6	1.374 (2)	C12—N2	1.4848 (19)
C5—H5	0.9300	C12—H12A	0.9700
C6—H6	0.9300	C12—H12B	0.9700
C8—C9	1.511 (2)	N1—O2	1.2257 (17)
C8—C12	1.516 (2)	N1—O3	1.2306 (16)
C8—H8A	0.9700	N2—H2A	0.9000
C8—H8B	0.9700	N2—H2B	0.9000
C2—C1—C6	120.73 (12)	H9A—C9—H9B	108.2
C2—C1—N1	119.70 (11)	C11—C10—C9	110.87 (13)
C6—C1—N1	119.55 (12)	C11—C10—H10A	109.5
C3—C2—C1	119.92 (11)	C9—C10—H10A	109.5
C3—C2—H2	120.0	C11—C10—H10B	109.5
C1—C2—H2	120.0	C9—C10—H10B	109.5
C2—C3—C4	121.83 (12)	H10A—C10—H10B	108.1
C2—C3—H3	119.1	N2—C11—C10	111.42 (13)
C4—C3—H3	119.1	N2—C11—H11A	109.3

O1—C4—C5	122.95 (11)	C10—C11—H11A	109.3
O1—C4—C3	121.02 (11)	N2—C11—H11B	109.3
C5—C4—C3	116.03 (11)	C10—C11—H11B	109.3
C6—C5—C4	122.36 (12)	H11A—C11—H11B	108.0
C6—C5—H5	118.8	N2—C12—C8	110.68 (12)
C4—C5—H5	118.8	N2—C12—H12A	109.5
C5—C6—C1	119.09 (12)	C8—C12—H12A	109.5
C5—C6—H6	120.5	N2—C12—H12B	109.5
C1—C6—H6	120.5	C8—C12—H12B	109.5
C9—C8—C12	111.16 (14)	H12A—C12—H12B	108.1
C9—C8—H8A	109.4	O2—N1—O3	121.66 (13)
C12—C8—H8A	109.4	O2—N1—C1	119.64 (12)
C9—C8—H8B	109.4	O3—N1—C1	118.70 (12)
C12—C8—H8B	109.4	C11—N2—C12	112.69 (11)
H8A—C8—H8B	108.0	C11—N2—H2A	109.1
C10—C9—C8	110.09 (13)	C12—N2—H2A	109.1
C10—C9—H9A	109.6	C11—N2—H2B	109.1
C8—C9—H9A	109.6	C12—N2—H2B	109.1
C10—C9—H9B	109.6	H2A—N2—H2B	107.8
C8—C9—H9B	109.6		

C6—C1—C2—C3	0.8 (2)	C12—C8—C9—C10	56.35 (18)
N1—C1—C2—C3	−178.12 (12)	C8—C9—C10—C11	−56.27 (19)
C1—C2—C3—C4	−0.2 (2)	C9—C10—C11—N2	55.67 (18)
C2—C3—C4—O1	178.45 (13)	C9—C8—C12—N2	−55.31 (17)
C2—C3—C4—C5	−1.21 (19)	C2—C1—N1—O2	169.09 (14)
O1—C4—C5—C6	−177.44 (14)	C6—C1—N1—O2	−9.8 (2)
C3—C4—C5—C6	2.2 (2)	C2—C1—N1—O3	−9.8 (2)
C4—C5—C6—C1	−1.8 (2)	C6—C1—N1—O3	171.33 (14)
C2—C1—C6—C5	0.2 (2)	C10—C11—N2—C12	−55.30 (16)
N1—C1—C6—C5	179.09 (14)	C8—C12—N2—C11	54.81 (16)

Hydrogen-bond geometry (Å, º)

Cg1 is the centroid of the C1–C6 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*A*···O1^{i}	0.90	1.92	2.788 (2)	161
N2—H2*B*···O1^{ii}	0.90	1.80	2.6985 (15)	175
C6—H6···*Cg*1^{iii}	0.93	2.75	3.428 (3)	130

Symmetry codes: (i) −*x*, *y*−1/2, −*z*+1/2; (ii) −*x*+1/2, −*y*+1, *z*+1/2; (iii) *x*+1/2, −*y*+1/2, −*z*.