Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2-Amino-5-methylpyridinium 2-hydroxy-3,5-dinitrobenzoate

Madhukar Hemamalini‡ and Hoong-Kun Fun*§

X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia
Correspondence e-mail: hkfun@usm.my

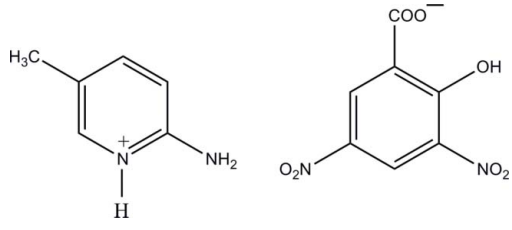
Received 12 April 2010; accepted 20 April 2010

Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.045; wR factor = 0.150; data-to-parameter ratio = 22.6.

In the title molecular salt, $C_6H_9N_2^+\cdot C_7H_3N_2O_7^-$, the 2-amino-5-methylpyridinium cation is essentially planar, with a maximum deviation of 0.023 (1) Å. There is an intramolecular O—H···O hydrogen bond in the 3,5-dinitrosalicylate anion, which generates an S(6) ring motif. In the crystal, the protonated N atom and the 2-amino group are hydrogen bonded to the carboxylate O atoms *via* a pair of N—H···O hydrogen bonds, forming an $R_2^2(8)$ ring motif. Weak intermolecular C—H···O interactions help to further stabilize the crystal structure.

Related literature

For background to the chemistry of substituted pyridines, see: Pozharski *et al.* (1997); Katritzky *et al.* (1996); Nahringbauer & Kvick (1977). For 3,5-dinitrosalicylic acid, see: Hindawey *et al.* (1980); Issa *et al.* (1981). For details of hydrogen bonding, see: Jeffrey & Saenger (1991); Jeffrey (1997); Scheiner (1997). For hydrogen-bond motifs, see: Bernstein *et al.* (1995). For bond-length data, see: Allen *et al.* (1987). For the stability of the temperature controller used in the data collection, see: Cosier & Glazer (1986).



Experimental

Crystal data

$C_6H_9N_2^+\cdot C_7H_3N_2O_7^-$
M_r = 336.27
Triclinic, $P\overline{1}$
a = 5.8673 (7) Å
b = 8.0991 (9) Å
c = 15.2437 (17) Å
α = 86.844 (3)°
β = 84.252 (3)°
γ = 81.209 (3)°
V = 711.69 (14) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.13 mm^{-1}
T = 100 K
0.29 × 0.14 × 0.08 mm

Data collection

Bruker APEX DUO CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.963, T_{max} = 0.990
12709 measured reflections
4947 independent reflections
3922 reflections with $I > 2\sigma(I)$
R_{int} = 0.023

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.150
S = 1.08
4947 reflections
219 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.51 e Å^{-3}
$\Delta\rho_{min}$ = −0.46 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1A···O7	0.82	1.66	2.4200 (13)	152
N1—H1···O6^{i}	0.86	1.82	2.6768 (14)	174
N2—H2A···O7^{i}	0.86	2.11	2.9668 (15)	176
N2—H2B···O1^{ii}	0.86	2.16	2.8468 (15)	137
N2—H2B···O2^{ii}	0.86	2.40	3.1723 (16)	149
C2—H2···O4^{iii}	0.93	2.49	3.4073 (16)	169
C4—H4···O3^{iv}	0.93	2.39	3.2371 (16)	151
C5—H5···O2^{ii}	0.93	2.44	3.2328 (17)	143

Symmetry codes: (i) $-x+1, -y+1, -z$; (ii) $x+1, y, z$; (iii) $x-1, y-1, z$; (iv) $-x+1, -y+1, -z+1$.

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

MH and HKF thank the Malaysian Government and Universiti Sains Malaysia for the Research University Golden Goose grant No. 1001/PFIZIK/811012. MH also thanks Universiti Sains Malaysia for a post-doctoral research fellowship.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5405).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
Bruker (2009). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Cosier, J. & Glazer, A. M. (1986). *J. Appl. Cryst.* **19**, 105–107.
Hindawey, A. M., Nasser, A. M. G., Issa, R. M. & Issa, Y. M. (1980). *Indian J. Chem. Sect. A*, **19**, 615–619.
Issa, Y. M., Hindawey, A. M., El-Kholy, A. E. & Issa, R. M. (1981). *Gazz. Chim. Ital.* **111**, 27–33.

‡ Additional correspondence author, e-mail: mhemamalini2k3@yahoo.co.in.
§ Thomson Reuters ResearcherID: A-3561-2009.

Jeffrey, G. A. (1997). *An Introduction to Hydrogen Bonding.* Oxford University Press.
Jeffrey, G. A. & Saenger, W. (1991). *Hydrogen Bonding in Biological Structures.* Berlin: Springer.
Katritzky, A. R., Rees, C. W. & Scriven, E. F. V. (1996). *Comprehensive Heterocyclic Chemistry II.* Oxford: Pergamon Press.
Nahringbauer, I. & Kvick, Å. (1977). *Acta Cryst.* B**33**, 2902–2905.
Pozharski, A. F., Soldatenkov, A. T. & Katritzky, A. R. (1997). *Heterocycles in Life and Society.* New York: Wiley.
Scheiner, S. (1997). *Hydrogen Bonding. A Theoretical Perspective.* Oxford University Press.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

supplementary materials

Acta Cryst. (2010). E**66**, o1194-o1195 [**doi:10.1107/S1600536810014480**]

2-Amino-5-methylpyridinium 2-hydroxy-3,5-dinitrobenzoate

M. Hemamalini and H.-K. Fun

Comment

Pyridine and its derivatives play an important role in heterocyclic chemistry (Pozharski *et al.*, 1997; Katritzky *et al.*, 1996). They are often involved in hydrogen-bond interactions (Jeffrey & Saenger, 1991; Jeffrey, 1997; Scheiner, 1997). The nitro-substituted aromatic acid 3,5-dinitrosalicylic acid (DNSA) has proven potential for formation of proton-transfer compounds, particularly because of its acid strength (pKa = 2.18), its interactive ortho-related phenolic substituent group together with the nitro substituents which have potential for both π···π interactions as well as hydrogen-bonding interactions. A large number of both neutral and proton-transfer compounds of Lewis bases with DNSA, together with their IR spectra have been reported (Hindawey *et al.*, 1980; Issa *et al.*, 1981) in the literature. Since our aim is to study some interesting hydrogen bonding interactions, the crystal structure of the title compound is presented here.

The asymmetric unit (Fig. 1) contains one 2-amino-5-methylpyridinium cation and one 3,5-dinitrosalicylate anion. The proton transfer from the carboxyl group to atom N1 of 2-amino-5-methylpyridine resulted in the widening of C1—N1—C2 angle of the pyridinium ring to 123.48 (10)°, compared to the corresponding angle of 117.4° in neutral 2-amino-5-methyl-pyridine (Nahringbauer & Kvick, 1977). The 2-amino-5-methylpyridinium cation is essentially planar, with a maximum deviation of 0.023 (1) Å for atom N1. The bond lengths and angles are normal (Allen *et al.*, 1987). The phenol oxygen atoms are bent slightly away from the mean plane of the benzene ring [torsion angle O1-C7-C8-C9 = 179.70 (12)°].

In the crystal packing (Fig. 2), the protonated N1 atom and the 2-amino group (N2) is hydrogen-bonded to the carboxylate oxygen atoms (O6 and O7) via a pair of intermolecular N1—H1···O6 and N2—H2A···O7 hydrogen bonds forming a ring motif $R_2^2(8)$ (Bernstein *et al.*, 1995). There is an intramolecular O—H···O hydrogen bond in the 3,5-dinitrosalicylate anion, which generates an *S*(6) ring motif. The crystal structure is further stabilized by weak C—H···O (Table 1) hydrogen bonds.

Experimental

A hot methanol solution (20 ml) of 2-amino-5-methylpyridine (27 mg, Aldrich) and 3,5-dinitrosalicylic acid (58 mg, Merck) were mixed and warmed over a heating magnetic stirrer for a few minutes. The resulting solution was allowed to cool slowly at room temperature and yellow blocks of (I) appeared after a few days.

Refinement

All hydrogen atoms were positioned geometrically [C–H = 0.93 or 0.96 Å, N–H = 0.86 Å and O–H = 0.82 Å] and were refined using a riding model, with U_{iso}(H) = 1.2 U_{eq}(C, N) or 1.5 U_{eq}(O). The methyl H atoms were positioned geometrically [C–H = 0.96 Å] and were refined using a riding model, with U_{iso}(H) = 1.5U_{eq}(C). A rotating group model was used for the methyl group.

Figures

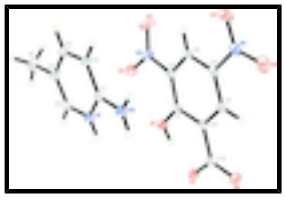


Fig. 1. The asymmetric unit of (I). Displacement ellipsoids are drawn at the 50% probability level.

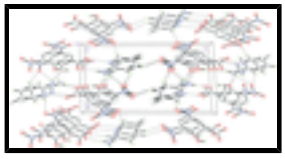


Fig. 2. The crystal packing of (I), showing hydrogen-bonded (dashed lines) networks. H atoms are not involing the hydrogen bond interactions are omitted for clarity.

2-Amino-5-methylpyridinium 2-hydroxy-3,5-dinitrobenzoate

Crystal data

$C_6H_9N_2^+ \cdot C_7H_3N_2O_7^-$	$Z = 2$
$M_r = 336.27$	$F(000) = 348$
Triclinic, $P\overline{1}$	$D_x = 1.569$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 5.8673$ (7) Å	Cell parameters from 5139 reflections
$b = 8.0991$ (9) Å	$\theta = 2.7$–$32.4°$
$c = 15.2437$ (17) Å	$\mu = 0.13$ mm^{-1}
$\alpha = 86.844$ (3)°	$T = 100$ K
$\beta = 84.252$ (3)°	Block, yellow
$\gamma = 81.209$ (3)°	0.29 × 0.14 × 0.08 mm
$V = 711.69$ (14) Å^3	

Data collection

Bruker APEX DUO CCD area-detector diffractometer	4947 independent reflections
Radiation source: fine-focus sealed tube	3922 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.023$
φ and ω scans	$\theta_{max} = 32.5°$, $\theta_{min} = 1.3°$
Absorption correction: multi-scan (SADABS; Bruker, 2009)	$h = -8 \rightarrow 8$
$T_{min} = 0.963$, $T_{max} = 0.990$	$k = -12 \rightarrow 11$
12709 measured reflections	$l = -22 \rightarrow 23$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map

$R[F^2 > 2\sigma(F^2)] = 0.045$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.150$	H-atom parameters constrained
$S = 1.08$	$w = 1/[\sigma^2(F_o^2) + (0.0741P)^2 + 0.3055P]$ where $P = (F_o^2 + 2F_c^2)/3$
4947 reflections	$(\Delta/\sigma)_{max} < 0.001$
219 parameters	$\Delta\rho_{max} = 0.51$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.46$ e Å^{-3}

Special details

Experimental. The crystal was placed in the cold stream of an Oxford Cryosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 100.0 (1) K.

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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 $F^2 > 2\sigma(F^2)$ 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}
N1	0.50349 (18)	0.28625 (13)	0.24313 (7)	0.0151 (2)
H1	0.4877	0.2768	0.1882	0.018*
N2	0.82364 (19)	0.41676 (14)	0.19955 (7)	0.0176 (2)
H2A	0.8060	0.4015	0.1454	0.021*
H2B	0.9352	0.4662	0.2121	0.021*
C1	0.6777 (2)	0.36374 (15)	0.26413 (8)	0.0146 (2)
C2	0.3509 (2)	0.22205 (16)	0.30452 (8)	0.0162 (2)
H2	0.2353	0.1692	0.2859	0.019*
C3	0.3654 (2)	0.23421 (16)	0.39271 (8)	0.0177 (2)
C4	0.5424 (2)	0.31915 (17)	0.41660 (8)	0.0196 (2)
H4	0.5553	0.3318	0.4761	0.023*
C5	0.6949 (2)	0.38321 (16)	0.35500 (8)	0.0180 (2)
H5	0.8088	0.4391	0.3726	0.022*
C6	0.2014 (2)	0.1628 (2)	0.46135 (9)	0.0252 (3)
H6A	0.0930	0.1115	0.4329	0.038*
H6B	0.1192	0.2508	0.4971	0.038*
H6C	0.2873	0.0806	0.4980	0.038*
O1	0.17251 (16)	0.61810 (13)	0.14298 (6)	0.01924 (19)
H1A	0.1638	0.6119	0.0899	0.029*
O2	0.10528 (18)	0.60967 (13)	0.31694 (7)	0.0243 (2)
O3	0.28454 (18)	0.76459 (15)	0.38862 (6)	0.0270 (2)

O4	0.93341 (17)	0.99542 (13)	0.26211 (7)	0.0237 (2)
O5	1.00019 (18)	1.02494 (14)	0.11971 (8)	0.0274 (2)
O6	0.55546 (18)	0.76372 (13)	−0.07527 (6)	0.0222 (2)
O7	0.26539 (17)	0.63243 (12)	−0.01529 (6)	0.01923 (19)
N3	0.25086 (18)	0.70325 (14)	0.31952 (7)	0.0166 (2)
N4	0.89119 (18)	0.97295 (13)	0.18590 (8)	0.0181 (2)
C7	0.3389 (2)	0.70035 (15)	0.15497 (8)	0.0136 (2)
C8	0.3891 (2)	0.74498 (15)	0.23904 (8)	0.0141 (2)
C9	0.5696 (2)	0.83248 (15)	0.24908 (8)	0.0152 (2)
H9	0.6004	0.8588	0.3049	0.018*
C10	0.7029 (2)	0.87992 (15)	0.17497 (8)	0.0148 (2)
C11	0.6613 (2)	0.84379 (15)	0.09005 (8)	0.0154 (2)
H11	0.7517	0.8788	0.0409	0.018*
C12	0.4831 (2)	0.75506 (14)	0.08054 (7)	0.0136 (2)
C13	0.4365 (2)	0.71642 (15)	−0.01015 (8)	0.0160 (2)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0161 (4)	0.0167 (5)	0.0128 (4)	−0.0032 (4)	−0.0022 (3)	−0.0010 (3)
N2	0.0184 (5)	0.0215 (5)	0.0143 (4)	−0.0086 (4)	−0.0004 (3)	−0.0001 (4)
C1	0.0161 (5)	0.0138 (5)	0.0138 (5)	−0.0018 (4)	−0.0014 (4)	−0.0003 (4)
C2	0.0144 (5)	0.0164 (5)	0.0180 (5)	−0.0032 (4)	−0.0014 (4)	−0.0009 (4)
C3	0.0183 (5)	0.0184 (5)	0.0156 (5)	−0.0022 (4)	0.0005 (4)	0.0009 (4)
C4	0.0238 (6)	0.0234 (6)	0.0123 (5)	−0.0048 (5)	−0.0033 (4)	−0.0007 (4)
C5	0.0200 (5)	0.0205 (6)	0.0149 (5)	−0.0058 (5)	−0.0039 (4)	−0.0005 (4)
C6	0.0225 (6)	0.0321 (7)	0.0206 (6)	−0.0078 (5)	0.0030 (5)	0.0045 (5)
O1	0.0192 (4)	0.0257 (5)	0.0154 (4)	−0.0105 (4)	−0.0028 (3)	−0.0014 (3)
O2	0.0271 (5)	0.0276 (5)	0.0202 (5)	−0.0141 (4)	0.0037 (4)	−0.0011 (4)
O3	0.0285 (5)	0.0422 (6)	0.0122 (4)	−0.0098 (5)	−0.0019 (4)	−0.0062 (4)
O4	0.0210 (4)	0.0230 (5)	0.0296 (5)	−0.0032 (4)	−0.0110 (4)	−0.0068 (4)
O5	0.0235 (5)	0.0269 (5)	0.0337 (6)	−0.0121 (4)	−0.0010 (4)	0.0021 (4)
O6	0.0286 (5)	0.0266 (5)	0.0127 (4)	−0.0099 (4)	0.0000 (3)	−0.0007 (3)
O7	0.0215 (4)	0.0242 (5)	0.0142 (4)	−0.0094 (4)	−0.0031 (3)	−0.0008 (3)
N3	0.0163 (4)	0.0197 (5)	0.0134 (4)	−0.0017 (4)	−0.0011 (3)	−0.0005 (4)
N4	0.0144 (4)	0.0149 (5)	0.0261 (5)	−0.0027 (4)	−0.0048 (4)	−0.0025 (4)
C7	0.0133 (5)	0.0139 (5)	0.0135 (5)	−0.0018 (4)	−0.0015 (4)	−0.0009 (4)
C8	0.0137 (5)	0.0158 (5)	0.0128 (5)	−0.0021 (4)	−0.0006 (4)	−0.0009 (4)
C9	0.0145 (5)	0.0151 (5)	0.0162 (5)	−0.0007 (4)	−0.0038 (4)	−0.0030 (4)
C10	0.0125 (5)	0.0132 (5)	0.0195 (5)	−0.0031 (4)	−0.0028 (4)	−0.0017 (4)
C11	0.0148 (5)	0.0140 (5)	0.0171 (5)	−0.0019 (4)	−0.0007 (4)	−0.0010 (4)
C12	0.0147 (5)	0.0144 (5)	0.0119 (5)	−0.0029 (4)	−0.0014 (4)	−0.0008 (4)
C13	0.0187 (5)	0.0162 (5)	0.0136 (5)	−0.0028 (4)	−0.0028 (4)	−0.0014 (4)

Geometric parameters (Å, °)

N1—C1	1.3509 (15)	O1—H1A	0.8200
N1—C2	1.3668 (15)	O2—N3	1.2302 (14)
N1—H1	0.8600	O3—N3	1.2348 (14)

N2—C1	1.3355 (15)	O4—N4	1.2404 (15)
N2—H2A	0.8600	O5—N4	1.2291 (15)
N2—H2B	0.8600	O6—C13	1.2331 (15)
C1—C5	1.4181 (16)	O7—C13	1.3067 (15)
C2—C3	1.3652 (17)	N3—C8	1.4546 (15)
C2—H2	0.9300	N4—C10	1.4563 (15)
C3—C4	1.4164 (18)	C7—C8	1.4208 (16)
C3—C6	1.5019 (18)	C7—C12	1.4372 (16)
C4—C5	1.3681 (18)	C8—C9	1.3861 (16)
C4—H4	0.9300	C9—C10	1.3794 (17)
C5—H5	0.9300	C9—H9	0.9300
C6—H6A	0.9600	C10—C11	1.3956 (17)
C6—H6B	0.9600	C11—C12	1.3793 (16)
C6—H6C	0.9600	C11—H11	0.9300
O1—C7	1.2957 (14)	C12—C13	1.4943 (16)
C1—N1—C2	123.48 (10)	O2—N3—O3	122.24 (11)
C1—N1—H1	118.2	O2—N3—C8	119.70 (10)
C2—N1—H1	118.3	O3—N3—C8	118.06 (11)
C1—N2—H2A	120.0	O5—N4—O4	123.31 (11)
C1—N2—H2B	120.0	O5—N4—C10	118.76 (11)
H2A—N2—H2B	120.0	O4—N4—C10	117.93 (11)
N2—C1—N1	119.16 (11)	O1—C7—C8	123.94 (11)
N2—C1—C5	123.65 (11)	O1—C7—C12	120.07 (10)
N1—C1—C5	117.18 (11)	C8—C7—C12	115.98 (10)
C3—C2—N1	121.14 (11)	C9—C8—C7	122.15 (11)
C3—C2—H2	119.4	C9—C8—N3	116.22 (10)
N1—C2—H2	119.4	C7—C8—N3	121.63 (10)
C2—C3—C4	116.59 (11)	C10—C9—C8	118.97 (11)
C2—C3—C6	122.06 (12)	C10—C9—H9	120.5
C4—C3—C6	121.34 (12)	C8—C9—H9	120.5
C5—C4—C3	122.13 (11)	C9—C10—C11	122.23 (11)
C5—C4—H4	118.9	C9—C10—N4	118.70 (11)
C3—C4—H4	118.9	C11—C10—N4	119.06 (11)
C4—C5—C1	119.43 (11)	C12—C11—C10	118.54 (11)
C4—C5—H5	120.3	C12—C11—H11	120.7
C1—C5—H5	120.3	C10—C11—H11	120.7
C3—C6—H6A	109.5	C11—C12—C7	122.12 (10)
C3—C6—H6B	109.5	C11—C12—C13	118.93 (10)
H6A—C6—H6B	109.5	C7—C12—C13	118.95 (10)
C3—C6—H6C	109.5	O6—C13—O7	123.31 (11)
H6A—C6—H6C	109.5	O6—C13—C12	120.35 (11)
H6B—C6—H6C	109.5	O7—C13—C12	116.34 (10)
C7—O1—H1A	109.5		
C2—N1—C1—N2	177.44 (11)	N3—C8—C9—C10	−178.15 (10)
C2—N1—C1—C5	−2.27 (17)	C8—C9—C10—C11	0.44 (18)
C1—N1—C2—C3	0.56 (19)	C8—C9—C10—N4	179.53 (11)
N1—C2—C3—C4	1.24 (18)	O5—N4—C10—C9	−175.21 (11)
N1—C2—C3—C6	−179.26 (12)	O4—N4—C10—C9	4.88 (17)

C2—C3—C4—C5	−1.29 (19)	O5—N4—C10—C11	3.91 (17)
C6—C3—C4—C5	179.21 (13)	O4—N4—C10—C11	−176.00 (11)
C3—C4—C5—C1	−0.4 (2)	C9—C10—C11—C12	−1.15 (18)
N2—C1—C5—C4	−177.56 (12)	N4—C10—C11—C12	179.76 (11)
N1—C1—C5—C4	2.14 (18)	C10—C11—C12—C7	0.51 (18)
O1—C7—C8—C9	179.70 (12)	C10—C11—C12—C13	179.74 (11)
C12—C7—C8—C9	−1.52 (17)	O1—C7—C12—C11	179.60 (11)
O1—C7—C8—N3	−1.24 (19)	C8—C7—C12—C11	0.77 (17)
C12—C7—C8—N3	177.54 (10)	O1—C7—C12—C13	0.37 (17)
O2—N3—C8—C9	−171.71 (11)	C8—C7—C12—C13	−178.46 (10)
O3—N3—C8—C9	8.51 (17)	C11—C12—C13—O6	−0.21 (18)
O2—N3—C8—C7	9.17 (18)	C7—C12—C13—O6	179.05 (11)
O3—N3—C8—C7	−170.60 (11)	C11—C12—C13—O7	−179.85 (11)
C7—C8—C9—C10	0.96 (18)	C7—C12—C13—O7	−0.59 (16)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1A···O7	0.82	1.66	2.4200 (13)	152
N1—H1···O6^{i}	0.86	1.82	2.6768 (14)	174
N2—H2A···O7^{i}	0.86	2.11	2.9668 (15)	176
N2—H2B···O1^{ii}	0.86	2.16	2.8468 (15)	137
N2—H2B···O2^{ii}	0.86	2.40	3.1723 (16)	149
C2—H2···O4^{iii}	0.93	2.49	3.4073 (16)	169
C4—H4···O3^{iv}	0.93	2.39	3.2371 (16)	151
C5—H5···O2^{ii}	0.93	2.44	3.2328 (17)	143

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

Fig. 1

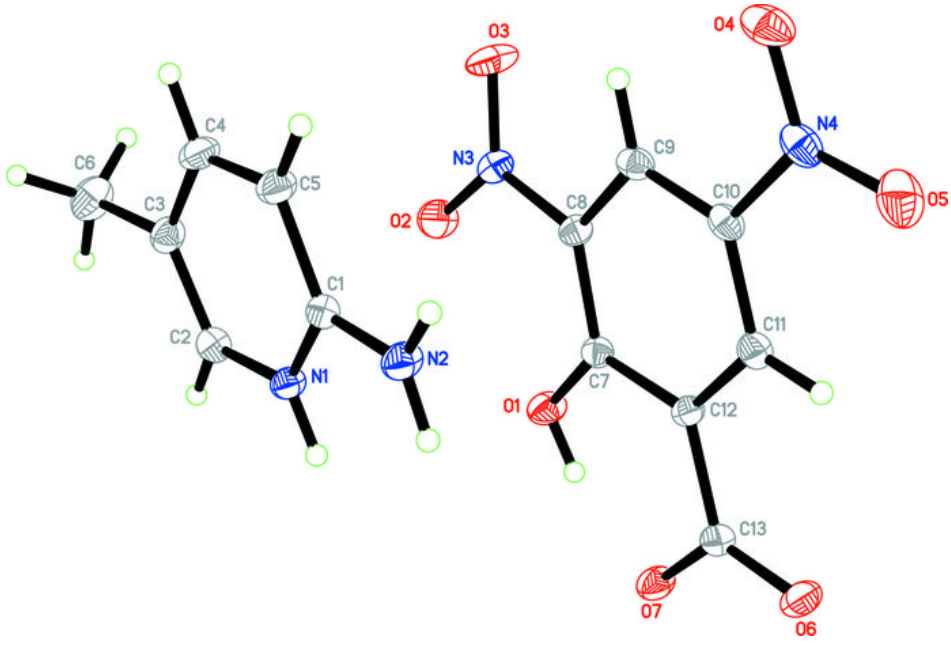

O3
O4
C4
N3
C9
N4
C6
C5
C8
C10
O5
O2
C3
C1
C11
C2
N2
C7
N1
O1
C12
C13
O7
O6

Fig. 2