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3,5-Dinitropyridin-4(1*H*)-one monohydrate

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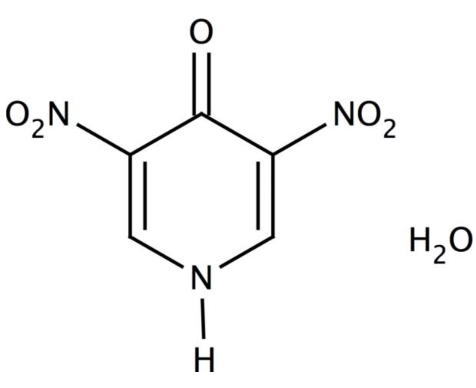
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.041; wR factor = 0.124; data-to-parameter ratio = 12.7.

The three independent organic molecules of 3,5-dinitropyridin-4(1*H*)-one monohydrate, $C_5H_3N_3O_5 \cdot H_2O$, each feature an N—H···O_{water} hydrogen bond. Each water molecule serves as hydrogen-bond donor to two carbonyl O atoms; these hydrogen bonds give rise to a layer motif. Two of the three formula units lie on special positions of site symmetry 2.

Related literature

The parent pyridin-4-one homolog crystallizes with five pyridone and six water molecules in the asymmetric unit; see: Jones (2001).



Experimental

Crystal data

$C_5H_3N_3O_5 \cdot H_2O$
M_r = 203.12
Orthorhombic, *Pbcn*
a = 21.728 (2) Å
b = 21.654 (2) Å
c = 6.5713 (5) Å
V = 3091.7 (4) Å^3
Z = 16
Mo $K\alpha$ radiation
μ = 0.16 mm^{-1}
T = 293 (2) K
0.45 × 0.45 × 0.20 mm

Data collection

Bruker APEXII diffractometer
Absorption correction: none
21800 measured reflections
3555 independent reflections
2852 reflections with $I > 2\sigma(I)$
R_{int} = 0.022

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.123
S = 1.08
3555 reflections
281 parameters
10 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3···O1w	0.85 (1)	1.86 (1)	2.703 (2)	172 (2)
N5—H5···O2w	0.85 (1)	1.84 (1)	2.692 (3)	180
N6—H6···O3w	0.85 (1)	1.87 (1)	2.723 (3)	180
O1w—H11···O8^{i}	0.85 (1)	2.04 (1)	2.878 (2)	168 (2)
O1w—H12···O11^{ii}	0.86 (1)	2.02 (1)	2.866 (2)	168 (2)
O2w—H21···O3^{iii}	0.84 (1)	2.05 (1)	2.888 (2)	173 (1)
O3w—H31···O3	0.84 (1)	2.05 (1)	2.890 (2)	172 (2)

Symmetry codes: (i) $-x+\frac{3}{2}, -y+\frac{3}{2}, z-\frac{1}{2}$; (ii) $x+\frac{1}{2}, y-\frac{1}{2}, -z+\frac{1}{2}$; (iii) $-x+1, -y+1, -z+1$.

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2008).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PK2110).

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supplementary materials

Acta Cryst. (2008). E**64**, o1701 [**doi:10.1107/S1600536808024604**]

3,5-Dinitropyridin-4(1*H*)-one monohydrate

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Comment

3,5-Dinitro-4-pyridinol, a specialty chemical, is assumed in chemical catalogs to exist in the enol form. The homolog, 4-pyridinol, is in fact 4-pyridinone·6/5hydrate. It has five independent pyridone and six water molecules that are hydrogen bonded to form layers (Jones, 2001). The presence of two electron-withdrawing groups in the title compound should enhance its propensity to form hydrogen bonds, and this is borne out in the present study. The three independent molecules of 3,5-dinitro-1*H*-pyridin-4-one hydrate (Fig. 1) each feature an N–H···O_{water} hydrogen bond; each water molecule serves as hydrogen-bond donor to two carbonyl oxygen atoms, and these hydrogen bonds give rise to a layered structure.

Experimental

4-Hydroxy-3-pyridine (19 g, 0.02 mol) was dissolved in fuming sulfuric acid (50% by SO_3 content) (60 ml), and to the solution was added an oleum-fuming nitric acid (1/3) mixture (50 ml). The temperature was kept at 0°C for an hour. The temperature was raised to 413 K over a period of one hour, and then held at 403 K for another 16 h. The mixture was poured into ice (200 g) to quench the reaction. Some 27 g of material was isolated. Crystals were obtained by recrystallization from water.

Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H 0.93 Å) and were included in the refinement using a riding model approximation, with *U*(H) 1.2U_{eq}(C). The amino and water H-atoms were refined with distance restraints of O–H = N–H 0.85 (1) and H···H 1.39 (1) Å; their temperature factors U_{iso} were freely refined.

Figures

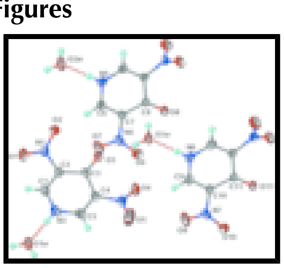


Fig. 1. Thermal ellipsoid plot (Barbour, 2001) plot of the three independent molecules of $C_5H_3N_3O_5·H_2O$ at the 50% probability level. Hydrogen atoms are drawn as spheres of arbitrary radius.

3,5-Dinitropyridin-4(1*H*)-one monohydrate

Crystal data

$C_5H_3N_3O_5·H_2O$	F_{000} = 1664

M_r = 203.12	D_x = 1.746 Mg m^{-3}
Orthorhombic, *Pbcn*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -P 2n 2ab	Cell parameters from 6494 reflections
a = 21.728 (2) Å	θ = 2.7–27.9º
b = 21.654 (2) Å	μ = 0.16 mm^{-1}
c = 6.5713 (5) Å	*T* = 293 (2) K
V = 3091.7 (4) Å^3	Block, colorless
Z = 16	0.45 × 0.45 × 0.20 mm

Data collection

Bruker APEXII diffractometer	3555 independent reflections
Radiation source: fine-focus sealed tube	2852 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.022
Detector resolution: 9 pixels mm^{-1}	θ_{max} = 27.5º
T = 293(2) K	θ_{min} = 2.7º
φ and ω scans	*h* = −28→27
Absorption correction: none	*k* = −28→27
21800 measured reflections	*l* = −8→8

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.040	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.123	$w = 1/[\sigma^2(F_o^2) + (0.0528P)^2 + 1.6904P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.08	$(\Delta/\sigma)_{max}$ < 0.001
3555 reflections	$\Delta\rho_{max}$ = 0.35 e Å^{-3}
281 parameters	$\Delta\rho_{min}$ = −0.31 e Å^{-3}
10 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.69961 (7)	0.46193 (6)	0.2892 (3)	0.0552 (4)
O2	0.62136 (6)	0.50982 (6)	0.4163 (3)	0.0464 (4)
O3	0.60319 (6)	0.62125 (6)	0.2292 (2)	0.0431 (4)
O4	0.61633 (6)	0.73545 (6)	0.0695 (3)	0.0486 (4)
O5	0.70310 (8)	0.77792 (8)	0.1081 (6)	0.1176 (12)
O6	0.60858 (6)	0.75858 (6)	0.5467 (3)	0.0496 (4)
O7	0.65774 (6)	0.67898 (7)	0.6579 (3)	0.0562 (4)

O8	0.5000	0.77618 (7)	0.7500	0.0389 (4)
O9	0.65904 (7)	0.92189 (7)	0.1991 (3)	0.0659 (5)
O10	0.61401 (6)	1.00203 (6)	0.0759 (3)	0.0482 (4)
O11	0.5000	1.02039 (7)	0.2500	0.0357 (4)
O1W	0.91543 (6)	0.62174 (6)	0.2293 (3)	0.0507 (4)
O2W	0.5000	0.46255 (9)	0.7500	0.0627 (7)
O3W	0.5000	0.70522 (8)	0.2500	0.0416 (5)
N1	0.67114 (7)	0.50953 (7)	0.3278 (3)	0.0360 (3)
N2	0.67066 (7)	0.73271 (7)	0.1089 (3)	0.0435 (4)
N3	0.79137 (7)	0.61956 (8)	0.1992 (3)	0.0434 (4)
N4	0.61008 (6)	0.70807 (7)	0.6311 (3)	0.0357 (3)
N5	0.5000	0.58686 (9)	0.7500	0.0376 (5)
N6	0.5000	0.83100 (9)	0.2500	0.0384 (5)
N7	0.61264 (7)	0.95139 (7)	0.1582 (3)	0.0377 (4)
C1	0.76100 (8)	0.56854 (9)	0.2549 (3)	0.0390 (4)
H1A	0.7829	0.5329	0.2857	0.047*
C2	0.69872 (8)	0.56805 (8)	0.2672 (3)	0.0315 (4)
C3	0.65995 (8)	0.62114 (7)	0.2208 (3)	0.0302 (4)
C4	0.69804 (8)	0.67347 (8)	0.1610 (3)	0.0337 (4)
C5	0.76066 (8)	0.67135 (9)	0.1539 (3)	0.0405 (4)
H5A	0.7825	0.7065	0.1169	0.049*
C6	0.55166 (8)	0.61756 (8)	0.7027 (3)	0.0345 (4)
H6A	0.5874	0.5957	0.6733	0.041*
C7	0.55244 (7)	0.68038 (7)	0.6972 (3)	0.0290 (4)
C8	0.5000	0.71922 (10)	0.7500	0.0269 (5)
C9	0.55223 (9)	0.86172 (8)	0.2128 (3)	0.0360 (4)
H9	0.5884	0.8398	0.1893	0.043*
C10	0.55324 (8)	0.92451 (7)	0.2090 (3)	0.0301 (4)
C11	0.5000	0.96327 (10)	0.2500	0.0278 (5)
H11	0.9376 (8)	0.6540 (6)	0.220 (4)	0.059 (7)*
H12	0.9385 (8)	0.5898 (6)	0.218 (4)	0.053 (7)*
H21	0.4680 (2)	0.4409 (7)	0.757 (4)	0.055 (7)*
H31	0.5318 (3)	0.6832 (7)	0.236 (4)	0.058 (7)*
H3	0.8306 (5)	0.6181 (10)	0.199 (4)	0.057 (7)*
H5	0.5000	0.5477 (5)	0.7500	0.051 (9)*
H6	0.5000	0.7915 (5)	0.2500	0.051 (9)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0543 (9)	0.0322 (7)	0.0790 (12)	0.0112 (6)	0.0045 (8)	0.0018 (7)
O2	0.0369 (7)	0.0417 (7)	0.0607 (9)	−0.0020 (6)	0.0068 (7)	0.0077 (7)
O3	0.0218 (6)	0.0344 (7)	0.0730 (10)	0.0014 (5)	0.0029 (6)	0.0061 (6)
O4	0.0381 (7)	0.0401 (7)	0.0676 (10)	0.0086 (6)	−0.0039 (7)	0.0016 (7)
O5	0.0504 (10)	0.0435 (10)	0.259 (4)	−0.0121 (8)	−0.0082 (15)	0.0441 (15)
O6	0.0415 (7)	0.0392 (7)	0.0681 (10)	−0.0090 (6)	0.0128 (7)	0.0059 (7)
O7	0.0273 (7)	0.0575 (9)	0.0837 (12)	0.0076 (6)	0.0053 (7)	−0.0044 (8)
O8	0.0326 (9)	0.0211 (8)	0.0631 (13)	0.000	0.0069 (8)	0.000

O9	0.0304 (7)	0.0501 (9)	0.1171 (16)	0.0104 (6)	0.0009 (8)	0.0031 (9)
O10	0.0399 (7)	0.0372 (7)	0.0673 (10)	−0.0056 (6)	0.0073 (7)	0.0088 (7)
O11	0.0305 (9)	0.0199 (8)	0.0567 (12)	0.000	0.0020 (8)	0.000
O1W	0.0263 (7)	0.0294 (7)	0.0962 (13)	0.0008 (5)	−0.0054 (7)	0.0013 (7)
O2W	0.0296 (10)	0.0253 (9)	0.133 (2)	0.000	−0.0014 (12)	0.000
O3W	0.0286 (9)	0.0244 (8)	0.0719 (14)	0.000	0.0004 (9)	0.000
N1	0.0345 (8)	0.0322 (8)	0.0414 (9)	0.0028 (6)	−0.0042 (7)	0.0014 (6)
N2	0.0356 (8)	0.0335 (8)	0.0614 (11)	−0.0008 (6)	0.0060 (8)	0.0067 (7)
N3	0.0206 (7)	0.0488 (10)	0.0608 (11)	0.0008 (6)	−0.0006 (7)	0.0012 (8)
N4	0.0275 (7)	0.0353 (8)	0.0442 (9)	−0.0025 (6)	0.0039 (6)	−0.0084 (7)
N5	0.0434 (12)	0.0192 (9)	0.0503 (14)	0.000	0.0058 (10)	0.000
N6	0.0460 (13)	0.0188 (9)	0.0504 (13)	0.000	0.0006 (10)	0.000
N7	0.0293 (7)	0.0333 (8)	0.0505 (10)	0.0011 (6)	0.0040 (7)	−0.0052 (7)
C1	0.0282 (9)	0.0412 (10)	0.0476 (11)	0.0067 (7)	−0.0031 (8)	−0.0008 (8)
C2	0.0270 (8)	0.0305 (8)	0.0371 (9)	0.0010 (6)	−0.0011 (7)	−0.0017 (7)
C3	0.0237 (8)	0.0296 (8)	0.0374 (9)	0.0006 (6)	0.0012 (7)	−0.0023 (7)
C4	0.0264 (8)	0.0324 (9)	0.0424 (10)	0.0001 (7)	0.0008 (7)	0.0007 (7)
C5	0.0288 (9)	0.0410 (10)	0.0517 (12)	−0.0062 (7)	0.0027 (8)	0.0011 (9)
C6	0.0339 (9)	0.0289 (8)	0.0406 (10)	0.0063 (7)	0.0034 (7)	−0.0019 (7)
C7	0.0261 (8)	0.0269 (8)	0.0339 (9)	−0.0002 (6)	0.0009 (7)	−0.0015 (6)
C8	0.0258 (11)	0.0233 (10)	0.0316 (12)	0.000	−0.0020 (9)	0.000
C9	0.0387 (10)	0.0270 (8)	0.0421 (10)	0.0055 (7)	0.0003 (8)	−0.0018 (7)
C10	0.0296 (8)	0.0239 (8)	0.0369 (9)	0.0001 (6)	−0.0006 (7)	−0.0009 (6)
C11	0.0281 (11)	0.0228 (10)	0.0323 (12)	0.000	−0.0021 (9)	0.000

Geometric parameters (Å, °)

O1—N1	1.2285 (19)	N5—C6	1.341 (2)
O2—N1	1.228 (2)	N5—C6^{i}	1.341 (2)
O3—C3	1.235 (2)	N5—H5	0.848 (10)
O4—N2	1.210 (2)	N6—C9	1.338 (2)
O5—N2	1.206 (2)	N6—C9^{ii}	1.338 (2)
O6—N4	1.227 (2)	N6—H6	0.854 (10)
O7—N4	1.225 (2)	N7—C10	1.455 (2)
O8—C8	1.233 (3)	C1—C2	1.356 (2)
O9—N7	1.223 (2)	C1—H1A	0.9300
O10—N7	1.223 (2)	C2—C3	1.457 (2)
O11—C11	1.237 (3)	C3—C4	1.457 (2)
O1W—H11	0.851 (9)	C4—C5	1.362 (2)
O1W—H12	0.858 (9)	C5—H5A	0.9300
O2W—H21	0.840 (8)	C6—C7	1.361 (2)
O3W—H31	0.844 (8)	C6—H6A	0.9300
N1—C2	1.457 (2)	C7—C8	1.458 (2)
N2—C4	1.455 (2)	C8—C7^{i}	1.458 (2)
N3—C1	1.338 (3)	C9—C10	1.360 (2)
N3—C5	1.338 (3)	C9—H9	0.9300
N3—H3	0.853 (10)	C10—C11	1.454 (2)
N4—C7	1.455 (2)	C11—C10^{ii}	1.454 (2)

H11—O1W—H12	109.1 (14)	O3—C3—C4	125.31 (15)
O2—N1—O1	123.08 (16)	O3—C3—C2	124.72 (15)
O2—N1—C2	119.15 (14)	C4—C3—C2	109.97 (14)
O1—N1—C2	117.76 (15)	C5—C4—N2	115.48 (16)
O5—N2—O4	121.96 (17)	C5—C4—C3	123.38 (16)
O5—N2—C4	118.53 (17)	N2—C4—C3	121.12 (15)
O4—N2—C4	119.51 (15)	N3—C5—C4	121.24 (17)
C1—N3—C5	120.47 (16)	N3—C5—H5A	119.4
C1—N3—H3	117.6 (16)	C4—C5—H5A	119.4
C5—N3—H3	121.8 (16)	N5—C6—C7	120.80 (16)
O7—N4—O6	123.11 (15)	N5—C6—H6A	119.6
O7—N4—C7	118.20 (15)	C7—C6—H6A	119.6
O6—N4—C7	118.66 (14)	C6—C7—N4	115.51 (15)
C6—N5—C6^{i}	120.6 (2)	C6—C7—C8	124.09 (16)
C6—N5—H5	119.71 (10)	N4—C7—C8	120.39 (14)
C6^{i}—N5—H5	119.71 (10)	O8—C8—C7^{i}	125.23 (9)
C9—N6—C9^{ii}	120.4 (2)	O8—C8—C7	125.23 (9)
C9—N6—H6	119.82 (11)	C7^{i}—C8—C7	109.54 (19)
C9^{ii}—N6—H6	119.82 (10)	N6—C9—C10	120.94 (17)
O10—N7—O9	123.05 (16)	N6—C9—H9	119.5
O10—N7—C10	118.79 (14)	C10—C9—H9	119.5
O9—N7—C10	118.16 (16)	C9—C10—C11	124.11 (16)
N3—C1—C2	120.99 (17)	C9—C10—N7	114.74 (15)
N3—C1—H1A	119.5	C11—C10—N7	121.15 (14)
C2—C1—H1A	119.5	O11—C11—C10^{ii}	125.25 (9)
C1—C2—N1	115.69 (15)	O11—C11—C10	125.25 (9)
C1—C2—C3	123.94 (16)	C10^{ii}—C11—C10	109.50 (19)
N1—C2—C3	120.36 (14)		
C5—N3—C1—C2	−0.1 (3)	C6^{i}—N5—C6—C7	−1.47 (13)
N3—C1—C2—N1	179.48 (18)	N5—C6—C7—N4	−176.15 (14)
N3—C1—C2—C3	0.5 (3)	N5—C6—C7—C8	3.0 (3)
O2—N1—C2—C1	152.19 (18)	O7—N4—C7—C6	−26.7 (2)
O1—N1—C2—C1	−27.0 (3)	O6—N4—C7—C6	151.55 (18)
O2—N1—C2—C3	−28.8 (3)	O7—N4—C7—C8	154.12 (15)
O1—N1—C2—C3	151.95 (18)	O6—N4—C7—C8	−27.7 (2)
C1—C2—C3—O3	179.23 (19)	C6—C7—C8—O8	178.50 (13)
N1—C2—C3—O3	0.3 (3)	N4—C7—C8—O8	−2.34 (18)
C1—C2—C3—C4	−0.1 (3)	C6—C7—C8—C7^{i}	−1.50 (13)
N1—C2—C3—C4	−179.00 (16)	N4—C7—C8—C7^{i}	177.66 (18)
O5—N2—C4—C5	−16.1 (3)	C9^{ii}—N6—C9—C10	−0.98 (13)
O4—N2—C4—C5	164.46 (19)	N6—C9—C10—C11	2.0 (3)
O5—N2—C4—C3	162.5 (3)	N6—C9—C10—N7	−177.52 (15)
O4—N2—C4—C3	−16.9 (3)	O10—N7—C10—C9	152.08 (18)
O3—C3—C4—C5	179.9 (2)	O9—N7—C10—C9	−27.5 (3)
C2—C3—C4—C5	−0.7 (3)	O10—N7—C10—C11	−27.5 (2)
O3—C3—C4—N2	1.4 (3)	O9—N7—C10—C11	152.96 (17)

C2—C3—C4—N2	−179.23 (17)	C9—C10—C11—O11	179.01 (13)
C1—N3—C5—C4	−0.7 (3)	N7—C10—C11—O11	−1.48 (19)
N2—C4—C5—N3	179.75 (19)	C9—C10—C11—C10^{ii}	−0.99 (13)
C3—C4—C5—N3	1.2 (3)	N7—C10—C11—C10^{ii}	178.52 (19)

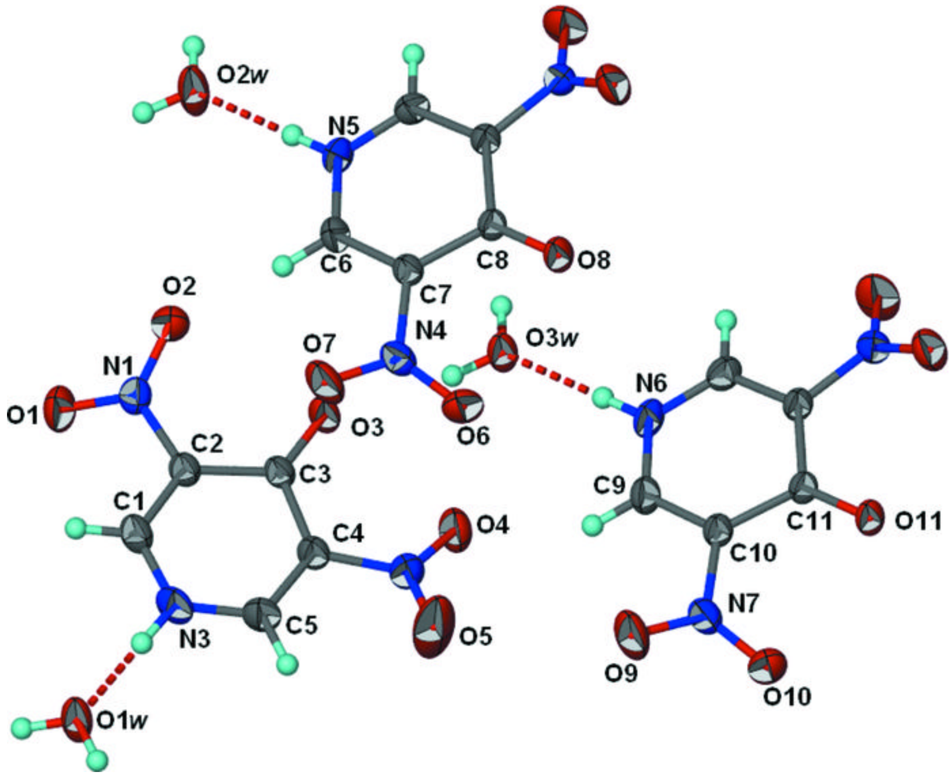
Symmetry codes: (i) −*x*+1, *y*, −*z*+3/2; (ii) −*x*+1, *y*, −*z*+1/2.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3···O1w	0.85 (1)	1.86 (1)	2.703 (2)	172 (2)
N5—H5···O2w	0.85 (1)	1.84 (1)	2.692 (3)	180
N6—H6···O3w	0.85 (1)	1.87 (1)	2.723 (3)	180
O1w—H11···O8^{iii}	0.85 (1)	2.04 (1)	2.878 (2)	168 (2)
O1w—H12···O11^{iv}	0.86 (1)	2.02 (1)	2.866 (2)	168 (2)
O2w—H21···O3^{v}	0.84 (1)	2.05 (1)	2.888 (2)	173 (1)
O3w—H31···O3	0.84 (1)	2.05 (1)	2.890 (2)	172 (2)

Symmetry codes: (iii) −*x*+3/2, −*y*+3/2, *z*−1/2; (iv) *x*+1/2, *y*−1/2, −*z*+1/2; (v) −*x*+1, −*y*+1, −*z*+1.

Fig. 1


O2w
N5
C6
C8
O8
O2
C7
N4
O3w
O7
N1
N6
O1
O3
O6
C2
C3
C9
C11
O11
C1
C4
O4
C10
N7
C5
N3
O5
O9
O10
O1w