Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

N′-(2,4-Dinitrophenyl)acetohydrazide monohydrate

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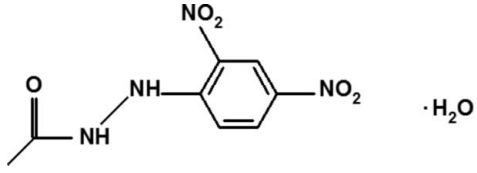
Received 18 June 2013; accepted 8 July 2013

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.071; wR factor = 0.195; data-to-parameter ratio = 29.9.

In the crystal structure of the title compound, $C_8H_8N_4O_5\cdot H_2O$, the organic and lattice water molecules are linked together *via* N—H···O and O—H···O hydrogen bonds. A C—H···O interaction is also observed between the organic molecules. These hydrogen bonds and interactions lead to the formation of a three-dimensional network. An intramolecular N—H···O hydrogen bond also occurs. The dihedral angle between the acetyl group and the almost planar hydrazide moiety [maximum deviation from the least-squares plane is 0.209 (2) Å for one of the nitro O atoms] is 88.5 (3)°.

Related literature

For background to the biological activity of hydrazines and hydrazones, see: Zahid & Sherazi (1997); Monfared *et al.* (2007). For a related crystal structure, see: Okabe *et al.* (1993). For hydrogen-bond graph-set motifs, see: Bernstein *et al.* (1995).



Experimental

Crystal data

$C_8H_8N_4O_5\cdot H_2O$
M_r = 258.20
Monoclinic, $P2_1/c$
a = 7.702 (2) Å
b = 7.057 (3) Å
c = 21.550 (4) Å
β = 109.044 (19)°
V = 1107.3 (6) Å^3
Z = 4
Ag $K\alpha$ radiation
λ = 0.56083 Å
μ = 0.08 mm^{-1}
T = 293 K
0.5 × 0.4 × 0.3 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
7469 measured reflections
5411 independent reflections
2771 reflections with $I > 2\sigma(I)$
R_{int} = 0.064
2 standard reflections every 120 min
intensity decay: 1%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.071
$wR(F^2)$ = 0.195
S = 0.98
5411 reflections
181 parameters
3 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
OW—H1W···O5	0.86 (1)	1.91 (1)	2.759 (3)	175 (3)
OW—H2W···O4^{i}	0.84 (1)	2.05 (1)	2.868 (3)	165 (3)
N1—H1N···OW^{ii}	0.88 (3)	2.04 (3)	2.883 (3)	160 (2)
N2—H2N···O1	0.90 (3)	1.96 (3)	2.607 (2)	128 (2)
N2—H2N···OW^{iii}	0.90 (3)	2.15 (3)	2.910 (3)	142 (2)
C6—H6···O5^{iv}	0.93	2.40	3.310 (3)	165

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

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS86* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012) and *DIAMOND* (Brandenburg & Putz, 2005); software used to prepare material for publication: *WinGX* (Farrugia, 2012).

This work was supported by the Tunisian Ministry of HEScR, and the Deanship of Scientific Research at King Saud University is also thanked for funding the paper through the Research Group Project No. RGP-VPP-089.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PV2637).

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supplementary materials

Acta Cryst. (2013). E**69**, o1249 [doi:10.1107/S1600536813018916]

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Comment

Hydrazine and its derivatives have been intensively investigated due to their attractive pharmacological applications (Zahid & Sherazi, 1997). In addition, hydrazones exhibit physiological activities in the treatment of several diseases such as tuberculosis (Monfared *et al.*, 2007). We report here the synthesis and the crystal structure of the title compound.

The bond distances and angles in the title compound (Fig. 1) agree very well with the corresponding bond distances and angles reported in a closely related compound (Okabe, *et al.*, 1993). There is a strong intramolecular N—H···O hydrogen bond that stabilizes the molecular structure of the title compound. In the crystal structure, pairs of symmetry-related molecules are connected into centrosymmetric clusters *via* medium O—H···O and N—H···O hydrogen bonds forming twenty-six-membered rings with an R_6^6 (26) motif (Bernstein, *et al.*, 1995). The water molecule is surrounded by three organic groups through hydrogen bonds type O—H···O and N—H···O. The crystal packing (Fig. 2) is stabilized by these intermolecular hydrogen bonds and C—H···O interactions (Table 1) resulting in a three dimensional network.

Experimental

An aqueous solution (10 ml) containing of 2,4-dinitro-phenylhydrazine (2 mmol) was added to pure acetic acid (10 ml). The obtained solution was stirred at 333 K for 30 min and then left to stand at room temperature. Yellow single crystals of the title compound were obtained after some days.

Refinement

H atoms were treated as riding, with C—H = 0.93 and 0.96 A° for phenyl and methyl H-atoms, respectively. The H-atoms bonded to O and N were located from difference maps and were allowed to refine freely. The U_{iso}(H) were allowed at 1.5U_{eq}(C methyl) or 1.2U_{eq}(C phenyl).

Computing details

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS86* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012) and *DIAMOND* (Brandenburg & Putz, 2005); software used to prepare material for publication: *WinGX* (Farrugia, 2012).

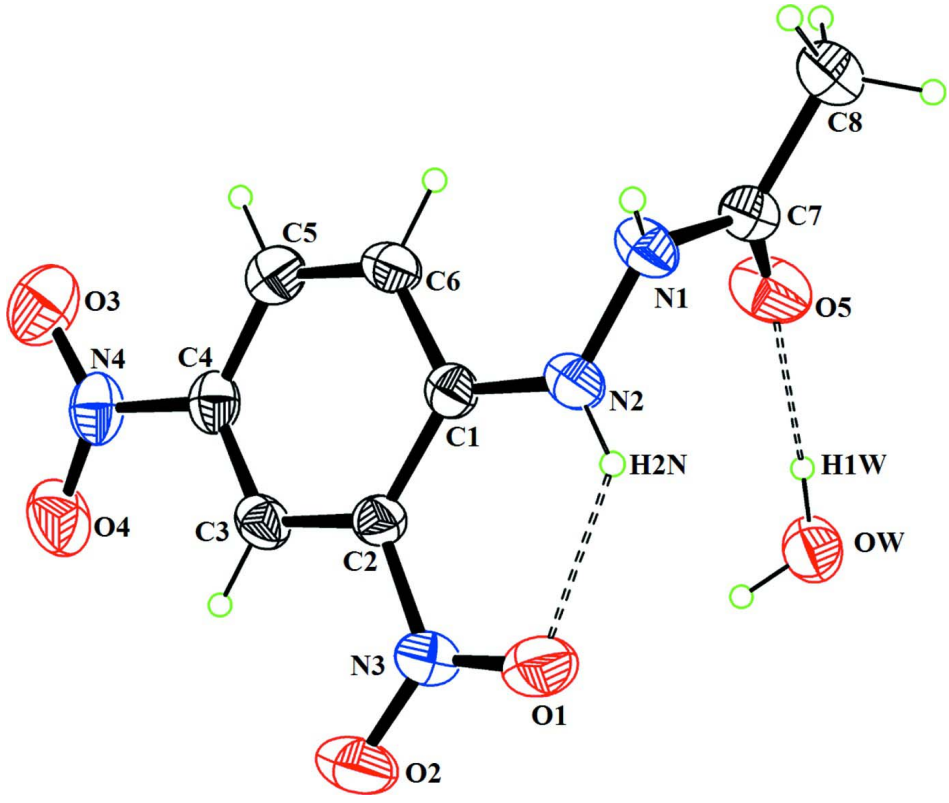

Figure 1

An *ORTEP* view of the title compound with the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms are represented as small spheres of arbitrary radii. Hydrogen bonds are represented as dashed lines.

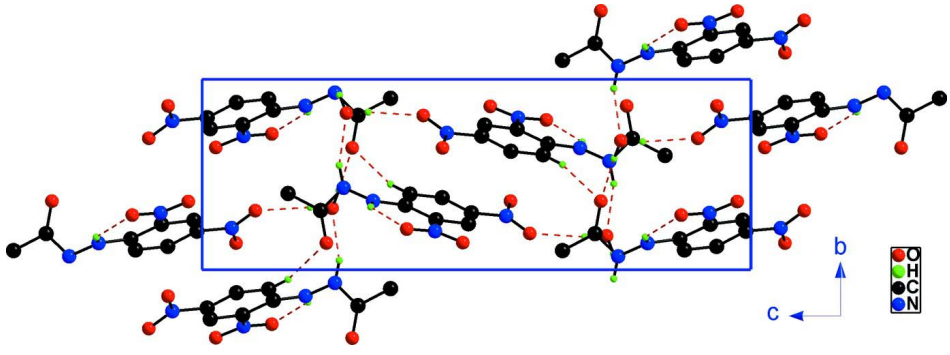

Figure 2

Unit cell packing of the title compound projected along the *a* axis. The H-atoms not involved in H-bonding are omitted.

N′-(2,4-Dinitrophenyl)acetohydrazide monohydrate

Crystal data

$C_8H_8N_4O_5·H_2O$
M_r = 258.20
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 7.702 (2) Å
b = 7.057 (3) Å
c = 21.550 (4) Å
β = 109.044 (19)°
V = 1107.3 (6) Å^3
Z = 4

$F(000) = 536$
$D_x = 1.549$ Mg m^{-3}
Ag $K\alpha$ radiation, $\lambda = 0.56083$ Å
Cell parameters from 25 reflections
$\theta = 9–11°$
$\mu = 0.08$ mm^{-1}
$T = 293$ K
Prism, yellow
0.5 × 0.4 × 0.3 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
non–profiled ω scans
7469 measured reflections
5411 independent reflections
2771 reflections with $I > 2\sigma(I)$
$R_{int} = 0.064$
$\theta_{max} = 28.0°$, $\theta_{min} = 2.2°$
$h = -12 \rightarrow 12$
$k = -11 \rightarrow 2$
$l = -36 \rightarrow 14$
2 standard reflections every 120 min
intensity decay: 1%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.071$
$wR(F^2) = 0.195$
$S = 0.98$
5411 reflections
181 parameters
3 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0627P)^2 + 0.2081P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.004$
$\Delta\rho_{max} = 0.25$ e Å^{-3}
$\Delta\rho_{min} = -0.23$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.027 (4)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'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 $F^2 > \sigma(F^2)$ 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}
OW	0.8341 (3)	0.6774 (3)	0.23696 (8)	0.0526 (5)
H1W	0.784 (4)	0.583 (3)	0.2497 (13)	0.088 (11)*
H2W	0.827 (4)	0.672 (5)	0.1972 (6)	0.091 (11)*
H2N	0.894 (4)	0.171 (4)	0.1927 (14)	0.073 (9)*
H1N	0.762 (3)	−0.050 (4)	0.2501 (11)	0.052 (7)*
C1	0.6447 (3)	0.1614 (3)	0.12985 (10)	0.0332 (4)
N2	0.7775 (3)	0.1390 (3)	0.18850 (8)	0.0416 (5)
C2	0.6778 (3)	0.2383 (3)	0.07398 (10)	0.0347 (5)
C4	0.3649 (3)	0.2020 (3)	0.00937 (10)	0.0387 (5)
N3	0.8583 (3)	0.2980 (3)	0.07563 (10)	0.0467 (5)

N4	0.2219 (3)	0.2185 (3)	−0.05403 (10)	0.0496 (5)
C3	0.5376 (3)	0.2593 (3)	0.01484 (10)	0.0383 (5)
H3	0.5615	0.3125	−0.0210	0.046*
N1	0.7353 (3)	0.0698 (3)	0.24205 (9)	0.0410 (5)
C6	0.4620 (3)	0.1034 (3)	0.12134 (10)	0.0385 (5)
H6	0.4349	0.0515	0.1568	0.046*
C5	0.3250 (3)	0.1218 (3)	0.06262 (11)	0.0422 (5)
H5	0.2065	0.0815	0.0580	0.051*
O5	0.6823 (3)	0.3600 (3)	0.27355 (9)	0.0607 (5)
C7	0.6972 (3)	0.1892 (3)	0.28405 (10)	0.0388 (5)
O4	0.2556 (3)	0.3098 (3)	−0.09698 (8)	0.0683 (6)
O2	0.8788 (3)	0.3578 (3)	0.02501 (9)	0.0723 (6)
O1	0.9859 (2)	0.2867 (3)	0.12714 (9)	0.0685 (6)
C8	0.6708 (3)	0.1000 (4)	0.34342 (10)	0.0492 (6)
H8A	0.7672	0.1403	0.3820	0.074*
H8B	0.6741	−0.0355	0.3397	0.074*
H8C	0.5542	0.1378	0.3465	0.074*
O3	0.0748 (3)	0.1398 (3)	−0.06187 (10)	0.0722 (6)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
OW	0.0634 (11)	0.0483 (11)	0.0435 (10)	−0.0030 (9)	0.0139 (9)	0.0001 (8)
C1	0.0400 (11)	0.0272 (10)	0.0339 (10)	0.0000 (9)	0.0142 (9)	−0.0027 (9)
N2	0.0431 (10)	0.0511 (12)	0.0333 (9)	−0.0026 (10)	0.0161 (8)	0.0035 (9)
C2	0.0404 (11)	0.0309 (11)	0.0367 (10)	−0.0045 (9)	0.0179 (9)	−0.0028 (9)
C4	0.0432 (12)	0.0331 (11)	0.0358 (10)	0.0044 (10)	0.0074 (9)	−0.0024 (9)
N3	0.0510 (12)	0.0503 (12)	0.0443 (10)	−0.0083 (10)	0.0234 (10)	0.0013 (10)
N4	0.0566 (13)	0.0416 (11)	0.0436 (11)	0.0072 (10)	0.0068 (10)	−0.0066 (10)
C3	0.0543 (14)	0.0305 (11)	0.0339 (10)	−0.0001 (10)	0.0194 (10)	0.0016 (9)
N1	0.0534 (11)	0.0387 (11)	0.0342 (9)	0.0053 (9)	0.0188 (8)	0.0067 (9)
C6	0.0458 (12)	0.0372 (12)	0.0390 (11)	−0.0009 (10)	0.0226 (10)	0.0023 (10)
C5	0.0395 (12)	0.0386 (13)	0.0498 (13)	0.0002 (10)	0.0166 (10)	−0.0039 (10)
O5	0.0877 (13)	0.0382 (10)	0.0690 (12)	0.0093 (9)	0.0431 (11)	0.0063 (9)
C7	0.0399 (11)	0.0404 (13)	0.0378 (11)	0.0004 (10)	0.0152 (9)	0.0037 (10)
O4	0.0883 (14)	0.0697 (13)	0.0383 (9)	0.0014 (11)	0.0090 (9)	0.0087 (10)
O2	0.0695 (12)	0.1000 (16)	0.0573 (11)	−0.0166 (11)	0.0342 (10)	0.0137 (11)
O1	0.0465 (10)	0.1019 (16)	0.0551 (11)	−0.0231 (10)	0.0138 (9)	0.0105 (11)
C8	0.0548 (14)	0.0557 (16)	0.0397 (12)	0.0029 (12)	0.0188 (11)	0.0065 (11)
O3	0.0496 (10)	0.0774 (14)	0.0732 (13)	−0.0013 (11)	−0.0025 (9)	−0.0012 (11)

Geometric parameters (Å, º)

OW—H1W	0.856 (10)	N4—O3	1.223 (3)
OW—H2W	0.841 (9)	N4—O4	1.224 (3)
C1—N2	1.351 (3)	C3—H3	0.9300
C1—C2	1.418 (3)	N1—C7	1.338 (3)
C1—C6	1.419 (3)	N1—H1N	0.88 (3)
N2—N1	1.386 (2)	C6—C5	1.364 (3)
N2—H2N	0.90 (3)	C6—H6	0.9300

C2—C3	1.384 (3)	C5—H5	0.9300
C2—N3	1.442 (3)	O5—C7	1.225 (3)
C4—C3	1.358 (3)	C7—C8	1.498 (3)
C4—C5	1.400 (3)	C8—H8A	0.9600
C4—N4	1.453 (3)	C8—H8B	0.9600
N3—O1	1.222 (2)	C8—H8C	0.9600
N3—O2	1.227 (2)		

H1W—OW—H2W	114 (2)	C4—C3—H3	120.1
N2—C1—C2	123.22 (18)	C2—C3—H3	120.1
N2—C1—C6	120.24 (18)	C7—N1—N2	120.3 (2)
C2—C1—C6	116.53 (18)	C7—N1—H1N	124.5 (16)
C1—N2—N1	120.55 (18)	N2—N1—H1N	113.5 (16)
C1—N2—H2N	119.4 (18)	C5—C6—C1	121.78 (19)
N1—N2—H2N	120.0 (18)	C5—C6—H6	119.1
C3—C2—C1	121.36 (18)	C1—C6—H6	119.1
C3—C2—N3	116.61 (18)	C6—C5—C4	119.25 (19)
C1—C2—N3	122.03 (18)	C6—C5—H5	120.4
C3—C4—C5	121.3 (2)	C4—C5—H5	120.4
C3—C4—N4	118.6 (2)	O5—C7—N1	121.5 (2)
C5—C4—N4	120.1 (2)	O5—C7—C8	122.7 (2)
O1—N3—O2	122.08 (19)	N1—C7—C8	115.7 (2)
O1—N3—C2	119.13 (17)	C7—C8—H8A	109.5
O2—N3—C2	118.79 (19)	C7—C8—H8B	109.5
O3—N4—O4	123.4 (2)	H8A—C8—H8B	109.5
O3—N4—C4	118.5 (2)	C7—C8—H8C	109.5
O4—N4—C4	118.1 (2)	H8A—C8—H8C	109.5
C4—C3—C2	119.74 (19)	H8B—C8—H8C	109.5

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
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C6—H6···O5^{iv}	0.93	2.40	3.310 (3)	165

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