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N-(2-Hydroxyethyl)-3,5-dinitrobenzamide

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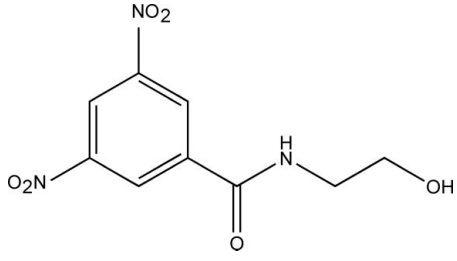
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.004 Å; R factor = 0.035; wR factor = 0.092; data-to-parameter ratio = 6.8.

The title compound, $C_9H_9N_3O_6$, was synthesized by the condensation of methyl 3,5-dinitrobenzoate and 2-aminoethanol. The non-centrosymmetric space group results in the formation of pseudo-chiral helices in the crystal structure, which exhibits a layer packing structure involving intramolecular N—H···O and O—H···O interactions.

Related literature

For related literature, see: Lin & Smith (1981); Morehouse & McGuire (1959); Percec (1981, 1982); Walde (1962).



Experimental

Crystal data

$C_9H_9N_3O_6$
M_r = 255.19
Orthorhombic, $P2_12_12_1$
a = 6.514 (4) Å
b = 9.097 (3) Å
c = 18.177 (3) Å
V = 1077.1 (8) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.14 mm^{-1}
T = 294 (2) K
0.46 × 0.45 × 0.33 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: none
1124 measured reflections
1118 independent reflections
945 reflections with $I > 2\sigma(I)$
R_{int} = 0.015
3 standard reflections
every 100 reflections
intensity decay: 3.4%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.092
S = 1.10
1118 reflections
164 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2*W*···O1^{i}	0.82	1.99	2.737 (3)	151
N1—H1*N*···O2^{i}	0.86	2.12	2.935 (3)	158

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

Data collection: *DIFRAC* (Gabe *et al.*, 1993); cell refinement: *DIFRAC*; data reduction: *NRCVAX* (Gabe *et al.*, 1989); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *DIAMOND* (Brandenburg, 1998); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LX2057).

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supporting information

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N-(2-Hydroxyethyl)-3,5-dinitrobenzamide

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S1. Comment

Non-ionic contrast agents, which are used in the field of intravascular and central nervous system visualization, are mostly complex molecules. However, the iodine in the molecule provides opacification to the *x*-rays and the remainder of the molecule provides the framework for transport of the iodine atoms. As a result, the structural arrangement of the molecule is very important in providing stability, solubility and biological safety in various organs (Lin & Smith, 1981). The title compound is an important intermediate in the synthesis of a variety of these molecules. It also can be used against coccidiosis and salmonella infection in poultry (Walde, 1962; Morehouse & McGuire, 1959).In addition, it plays an important role in the synthesis of copolymers (Percec, 1982; Percec, 1981). In this paper, we report the crystal structure of the title compound, N-(2-hydroxyethyl)-3,5-dinitrobenzamide (Fig. 1).

The title compound was crystallized in the non-centrosymmetric space group P212121 in spite of having no asymmetric carbon atom in the molecule. In the packing structure, an intermolecular O—H···O hydrogen bond leads to form pseudo-chiral helix about the 21 screw axis, propagating in the [100] direction. Non-centrosymmetric space group P212121 results in the formation of pseudo-chiral helix in the packing structure (Fig. 2). The crystal structure exhibits a layer packing structure with the intramolecular N—H···O and O—H···O hydrogen bonds (Fig. 2 and Table 1; symmetry code as in Fig. 2). On the other hand, adjacent molecules are linked into chains through van der Waals force to stabilize the crystal structure.

S2. Experimental

A mixture of methyl 3,5-dinitrobenzoate (5.65 g, 0.025 mol) and 50% aqueous 2-aminoethanol (30.5 g, 0.5 mol) was stirred for 10 h at room temperture. Then 30 ml water was added and the crystalline product was collected. Recrystallization of the crude product from ethanol gave N-(2-hydroxyethyl)-3,5-dinitrobenzamide (m.p. 416-417 K) (Lin & Smith, 1981). Single crystals of the title compound were obtained and used for X-ray diffraction studies at room temperature.

S3. Refinement

All H atoms were placed in idealized positions {C—H = 0.93 Å% (aromatic); C—H = 0.97 Å% (methylene); N—H = 0.86 Å%; O—H = 0.82 Å%} and refined as riding, with U_{iso}(H) = 1.2U_{eq}(C, N) or 1.5U_{eq}(O). Friedel pairs were merged at final refinement.

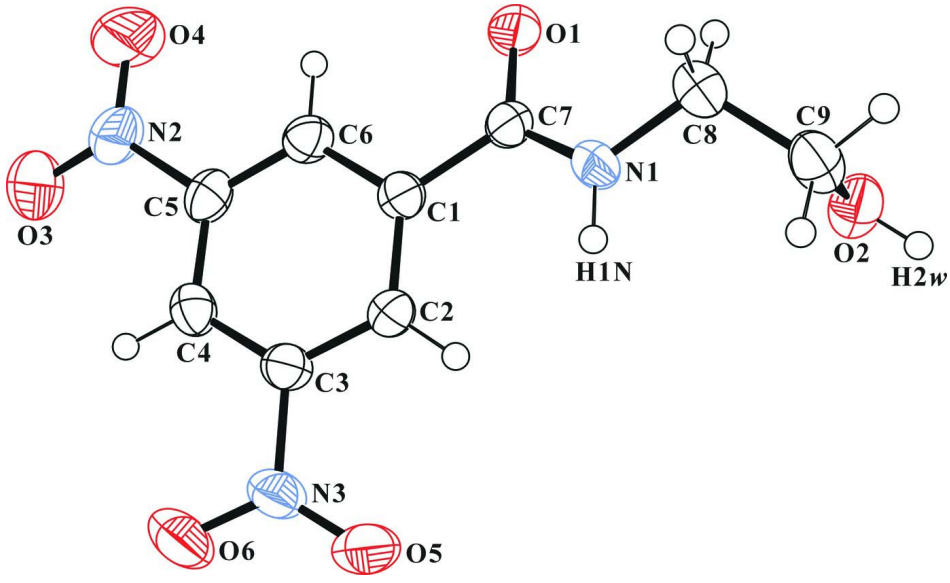

Figure 1

Molecular structure of title compound. Displacement ellipsoids are drawn at the 30% probability level.

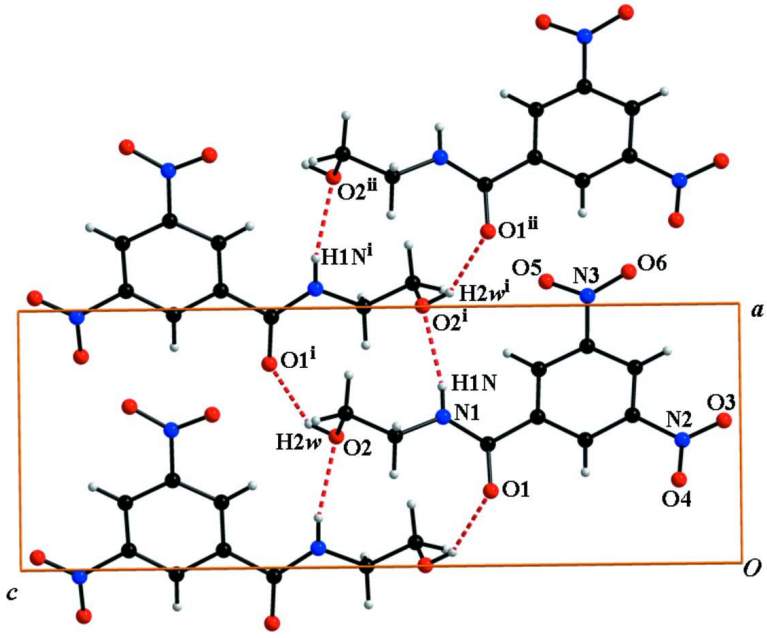

Figure 2

View of the structure projected on the ac plane. Hydrogen bonding shown as dashed lines. [Symmetry code: (i) x+1/2, -y+3/2, -z+1; (ii) x+1, y, z.]

N-(2-Hydroxyethyl)-3,5-dinitrobenzamide

Crystal data

$C_9H_9N_3O_6$
$M_r = 255.19$

Orthorhombic, $P2_12_12_1$
Hall symbol: p 2ac 2ab

a = 6.514 (4) Å
b = 9.097 (3) Å
c = 18.177 (3) Å
V = 1077.1 (8) Å^3
Z = 4
F(000) = 528
D_x = 1.574 Mg m^{-3}
Melting point = 416–417 K

Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 24 reflections
θ = 4.5–7.8°
μ = 0.14 mm^{-1}
T = 294 K
Block, colourless
0.46 × 0.45 × 0.33 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega/2\theta$ scans
1124 measured reflections
1118 independent reflections
945 reflections with $I > 2\sigma(I)$

R_{int} = 0.015
θ_{max} = 25.0°, θ_{min} = 2.2°
h = −3→7
k = −4→10
l = −10→21
3 standard reflections every 100 reflections
intensity decay: 3.4%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.092
S = 1.10
1118 reflections
164 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
$w = 1/[\sigma^2(F_o^2) + (0.0522P)^2 + 0.1396P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.219 (12)

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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
O1	0.2873 (3)	0.5989 (2)	0.34735 (10)	0.0471 (6)
O2	0.5044 (3)	0.7755 (2)	0.56083 (10)	0.0476 (6)
H2W	0.5535	0.8106	0.5985	0.071*
O3	0.5495 (4)	0.6710 (3)	0.02304 (11)	0.0717 (8)
O4	0.3233 (5)	0.5573 (4)	0.08631 (13)	0.1040 (12)
O5	1.0931 (4)	0.9372 (3)	0.26737 (12)	0.0799 (9)
O6	1.1272 (4)	0.8922 (3)	0.15286 (11)	0.0696 (8)

N1	0.5752 (4)	0.6420 (2)	0.41241 (11)	0.0370 (6)
H1N	0.6916	0.6865	0.4132	0.044*
N2	0.4757 (5)	0.6333 (3)	0.08103 (13)	0.0535 (7)
N3	1.0356 (4)	0.8822 (3)	0.21046 (13)	0.0464 (6)
C1	0.5713 (4)	0.6903 (3)	0.28124 (14)	0.0316 (6)
C2	0.7552 (4)	0.7679 (3)	0.27890 (13)	0.0332 (6)
H2A	0.8186	0.7982	0.3222	0.040*
C3	0.8421 (4)	0.7994 (3)	0.21153 (14)	0.0355 (6)
C4	0.7576 (4)	0.7570 (3)	0.14550 (13)	0.0366 (7)
H4	0.8202	0.7779	0.1007	0.044*
C5	0.5739 (5)	0.6816 (3)	0.14983 (14)	0.0386 (7)
C6	0.4801 (4)	0.6487 (3)	0.21553 (14)	0.0358 (6)
H6	0.3556	0.5987	0.2159	0.043*
C7	0.4681 (4)	0.6420 (3)	0.35076 (14)	0.0350 (7)
C8	0.5042 (5)	0.5698 (3)	0.47915 (13)	0.0437 (7)
H8A	0.5403	0.4664	0.4768	0.052*
H8B	0.3557	0.5765	0.4815	0.052*
C9	0.5928 (4)	0.6344 (3)	0.54759 (14)	0.0426 (7)
H9A	0.5652	0.5700	0.5890	0.051*
H9B	0.7404	0.6438	0.5424	0.051*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0326 (11)	0.0647 (13)	0.0441 (10)	−0.0133 (11)	0.0046 (9)	−0.0125 (10)
O2	0.0324 (10)	0.0700 (13)	0.0405 (10)	0.0039 (11)	−0.0015 (9)	−0.0121 (9)
O3	0.089 (2)	0.0951 (17)	0.0311 (11)	−0.0176 (17)	−0.0042 (13)	0.0000 (11)
O4	0.103 (2)	0.156 (3)	0.0536 (14)	−0.079 (2)	−0.0175 (15)	−0.0111 (17)
O5	0.0793 (19)	0.113 (2)	0.0478 (13)	−0.0607 (17)	−0.0059 (12)	0.0036 (13)
O6	0.0530 (14)	0.104 (2)	0.0523 (13)	−0.0241 (16)	0.0145 (12)	0.0089 (13)
N1	0.0305 (12)	0.0486 (13)	0.0319 (11)	−0.0083 (12)	0.0060 (10)	0.0000 (10)
N2	0.0617 (19)	0.0631 (16)	0.0356 (13)	−0.0109 (18)	−0.0113 (14)	−0.0048 (12)
N3	0.0415 (14)	0.0577 (14)	0.0400 (13)	−0.0152 (13)	0.0011 (13)	0.0105 (13)
C1	0.0287 (13)	0.0319 (12)	0.0341 (13)	0.0022 (12)	0.0013 (12)	−0.0024 (11)
C2	0.0334 (14)	0.0363 (13)	0.0300 (12)	−0.0038 (12)	−0.0027 (12)	−0.0013 (11)
C3	0.0320 (13)	0.0378 (13)	0.0367 (13)	−0.0040 (12)	0.0003 (13)	0.0023 (12)
C4	0.0415 (17)	0.0383 (14)	0.0300 (12)	0.0006 (14)	0.0033 (13)	0.0031 (11)
C5	0.0442 (17)	0.0414 (14)	0.0303 (13)	0.0005 (15)	−0.0081 (12)	−0.0026 (11)
C6	0.0281 (13)	0.0400 (13)	0.0391 (14)	−0.0011 (12)	−0.0031 (13)	−0.0039 (12)
C7	0.0310 (16)	0.0392 (15)	0.0346 (14)	−0.0051 (14)	0.0027 (12)	−0.0081 (11)
C8	0.0455 (16)	0.0502 (15)	0.0354 (13)	−0.0075 (15)	0.0068 (14)	0.0031 (12)
C9	0.0345 (15)	0.0576 (17)	0.0356 (13)	0.0019 (16)	0.0030 (12)	0.0081 (13)

Geometric parameters (Å, º)

O1—C7	1.243 (4)	C1—C7	1.498 (4)
O2—C9	1.428 (3)	C2—C3	1.379 (3)
O2—H2W	0.8200	C2—H2A	0.9300

O3—N2	1.208 (3)	C3—C4	1.376 (4)
O4—N2	1.213 (4)	C4—C5	1.382 (4)
O5—N3	1.209 (3)	C4—H4	0.9300
O6—N3	1.209 (3)	C5—C6	1.374 (4)
N1—C7	1.320 (3)	C6—H6	0.9300
N1—C8	1.455 (3)	C8—C9	1.492 (4)
N1—H1N	0.8600	C8—H8A	0.9700
N2—C5	1.472 (4)	C8—H8B	0.9700
N3—C3	1.468 (3)	C9—H9A	0.9700
C1—C6	1.387 (3)	C9—H9B	0.9700
C1—C2	1.391 (4)		

C9—O2—H2W	109.5	C6—C5—C4	122.9 (2)
C7—N1—C8	122.7 (2)	C6—C5—N2	118.7 (3)
C7—N1—H1N	118.7	C4—C5—N2	118.4 (3)
C8—N1—H1N	118.7	C5—C6—C1	119.9 (2)
O3—N2—O4	123.8 (3)	C5—C6—H6	120.0
O3—N2—C5	119.0 (3)	C1—C6—H6	120.0
O4—N2—C5	117.3 (3)	O1—C7—N1	122.9 (3)
O6—N3—O5	123.9 (2)	O1—C7—C1	118.4 (2)
O6—N3—C3	118.3 (2)	N1—C7—C1	118.6 (2)
O5—N3—C3	117.8 (2)	N1—C8—C9	113.2 (2)
C6—C1—C2	118.8 (2)	N1—C8—H8A	108.9
C6—C1—C7	117.0 (2)	C9—C8—H8A	108.9
C2—C1—C7	124.2 (2)	N1—C8—H8B	108.9
C3—C2—C1	119.1 (2)	C9—C8—H8B	108.9
C3—C2—H2A	120.5	H8A—C8—H8B	107.7
C1—C2—H2A	120.5	O2—C9—C8	109.8 (2)
C4—C3—C2	123.5 (2)	O2—C9—H9A	109.7
C4—C3—N3	118.4 (2)	C8—C9—H9A	109.7
C2—C3—N3	118.1 (2)	O2—C9—H9B	109.7
C3—C4—C5	115.9 (2)	C8—C9—H9B	109.7
C3—C4—H4	122.1	H9A—C9—H9B	108.2
C5—C4—H4	122.1		

C6—C1—C2—C3	−0.8 (3)	O3—N2—C5—C4	−5.8 (4)
C7—C1—C2—C3	176.3 (2)	O4—N2—C5—C4	174.3 (3)
C1—C2—C3—C4	−0.6 (4)	C4—C5—C6—C1	−0.8 (4)
C1—C2—C3—N3	179.6 (2)	N2—C5—C6—C1	178.7 (2)
O6—N3—C3—C4	−10.1 (4)	C2—C1—C6—C5	1.4 (4)
O5—N3—C3—C4	169.5 (3)	C7—C1—C6—C5	−175.9 (3)
O6—N3—C3—C2	169.7 (3)	C8—N1—C7—O1	10.3 (4)
O5—N3—C3—C2	−10.7 (4)	C8—N1—C7—C1	−167.1 (2)
C2—C3—C4—C5	1.2 (4)	C6—C1—C7—O1	−16.4 (4)
N3—C3—C4—C5	−179.0 (2)	C2—C1—C7—O1	166.4 (2)
C3—C4—C5—C6	−0.6 (4)	C6—C1—C7—N1	161.2 (2)
C3—C4—C5—N2	180.0 (3)	C2—C1—C7—N1	−16.0 (4)
O3—N2—C5—C6	174.7 (3)	C7—N1—C8—C9	−154.7 (3)

O4—N2—C5—C6	−5.2 (4)	N1—C8—C9—O2	71.7 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2*W*···O1^{i}	0.82	1.99	2.737 (3)	151
N1—H1*N*···O2^{i}	0.86	2.12	2.935 (3)	158

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