Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

3,5-Dinitro-*N*-(1,3-thiazol-2-yl)-benzamide monohydrate

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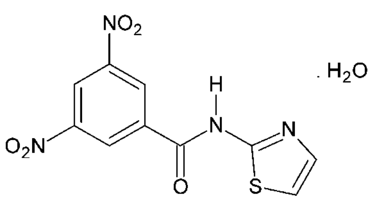
Received 2 February 2011; accepted 11 February 2011

Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.002 Å; R factor = 0.029; wR factor = 0.088; data-to-parameter ratio = 10.9.

In the title compound, $C_{10}H_6N_4O_5S\cdot H_2O$, the thiazole ring is twisted at a dihedral angle of 25.87 (7)° with respect to the benzene ring. The water molecule is linked with the benzamide molecules *via* N—H···O, O—H···N and O—H···O hydrogen bonds. In the crystal, π–π stacking is observed between nearly parallel [dihedral angle = 7.02 (7)°] thiazole and benzene rings of adjacent molecules, the centroid–centroid distances being 3.7107 (9) and 3.7158 (9) Å, respectively.

Related literature

For the effect of substituents on the structures of benzanilides, see: Gowda *et al.* (2008).



Experimental

Crystal data

$C_{10}H_6N_4O_5S\cdot H_2O$
M_r = 312.26
Monoclinic, $P2_1/c$
a = 13.7075 (12) Å
b = 6.9734 (6) Å
c = 13.8507 (13) Å
β = 108.512 (1)°
V = 1255.45 (19) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.30 mm^{-1}
T = 296 K
0.28 × 0.07 × 0.06 mm

Data collection

Bruker SMART 1000 CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)
T_{min} = 0.922, T_{max} = 0.983
6750 measured reflections
2214 independent reflections
1937 reflections with $I > 2\sigma(I)$
R_{int} = 0.015

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.088
S = 1.02
2214 reflections
203 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2N···O6	0.871 (19)	1.974 (19)	2.8313 (18)	167.9 (17)
O6—H6B···O1^{i}	0.75 (3)	2.38 (2)	3.0350 (19)	147 (2)
O6—H6C···N1^{ii}	0.84 (3)	2.14 (3)	2.964 (2)	168 (2)

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

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 2006); data reduction: *SAINT* and *CrystalStructure* (Rigaku/MSC, 2006); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

The authors are grateful to Allama Iqbal Open University, Islamabad, Pakistan, for the allocation of research and analytical laboratory facilities.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU5155).

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supporting information

Acta Cryst. (2011). E**67**, o660 [doi:10.1107/S1600536811005228]

3,5-Dinitro-*N*-(1,3-thiazol-2-yl)benzamide monohydrate

Sohail Saeed, Naghmana Rashid, Wing-Tak Wong and Rizwan Hussain

S1. Comment

In the present work, the structure of 3,5-dinitro-*N*-thiazol-2-yl-benzamide monohydrate has been determined to explore the effect of substituents on the structure of benzanilides (Gowda *et al.*, 2008).

The molecule is not planar. The thiazole ring is twisted to the benzene ring at a dihedral angle of 25.87 (7)°. The nitro groups are 12.30 (20)° and 15.68 (15)° from the phenyl ring plane of C5—C10. The thiazole ring is making a dihedral angle of 11.90 (2)° with the amido group which in turn makes a dihedral angle of 14.01 (4)° with the phenyl ring plane of C5—C10.

There are intermolecular N—H···O, O—H···N and O—H···O H-bond interactions, which link the molecules to form 2-D networks in the crystal lattice. There are also weak $\pi\cdots\pi$ interactions between neighbouring rings in the crystal lattice.

S2. Experimental

A solution of 3,5-dinitrobenzoyl chloride (0.01 mol) and 2-aminothiazole (0.01 mol) in anhydrous acetone was refluxed for 4 h. After completion of the reaction, the crude solid product was filtered, washed with water and purified by re-crystallization from ethyl acetate/water.

S3. Refinement

All of the C-bound H atoms are observable from difference Fourier map but are all placed at geometrical positions with C—H = 0.93 Å for phenyl H-atoms. All C-bound H-atoms are refined using riding model with U_{iso}(H) = 1.2U_{eq}(C). Both the N– and O-bound H-atoms were located from a difference Fourier map and refined isotropically.

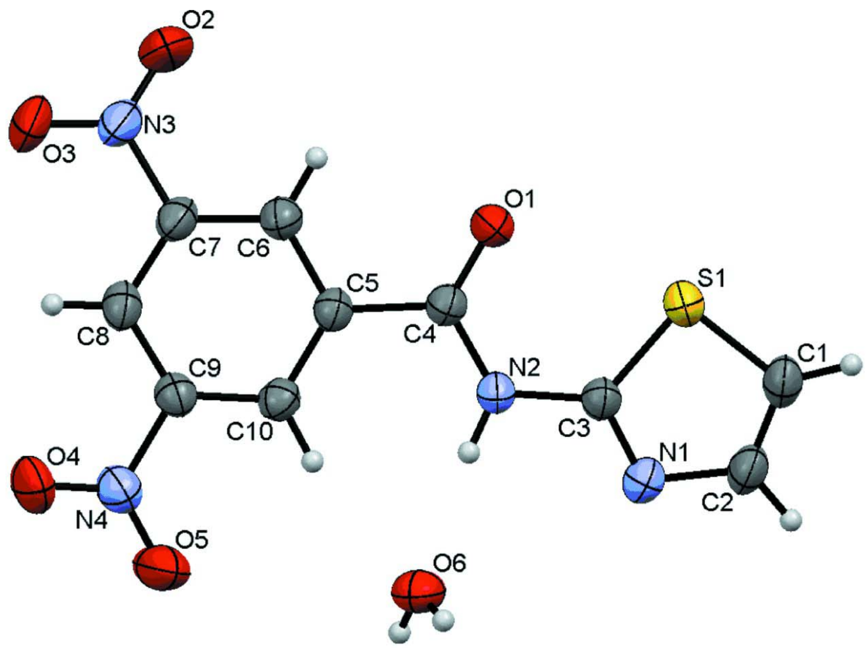

Figure 1

The *ORTEP* plot of the compound was shown at 50% probability thermal ellipsoids.

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Crystal data

$C_{10}H_6N_4O_5S \cdot H_2O$
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Hall symbol: -P 2ybc
$a = 13.7075$ (12) Å
$b = 6.9734$ (6) Å
$c = 13.8507$ (13) Å
$\beta = 108.512$ (1)°
$V = 1255.45$ (19) Å^3
$Z = 4$
$F(000) = 640$
$D_x = 1.652$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 9023 reflections
$\theta = 1.8$–25.0°
$\mu = 0.30$ mm^{-1}
$T = 296$ K
Needle, colourless
0.28 × 0.07 × 0.06 mm

Data collection

Bruker SMART 1000 CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)
$T_{min} = 0.922$, $T_{max} = 0.983$
6750 measured reflections
2214 independent reflections
1937 reflections with $I > 2\sigma(I)$
$R_{int} = 0.015$
$\theta_{max} = 25.0°$, $\theta_{min} = 3.0°$
$h = -16 \rightarrow 15$
$k = -8 \rightarrow 8$
$l = -16 \rightarrow 16$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.029$
$wR(F^2) = 0.088$
$S = 1.02$
2214 reflections
203 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 atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0569P)^2 + 0.2612P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.19$ e Å^{-3}
$\Delta\rho_{min} = -0.21$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0069 (14)

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}
S1	−0.17423 (3)	0.90512 (6)	0.12088 (3)	0.03884 (16)
O1	0.02312 (8)	0.95294 (19)	0.11971 (8)	0.0469 (3)
O2	0.35736 (11)	1.1250 (3)	0.08520 (11)	0.0793 (5)
O3	0.49514 (9)	1.0180 (3)	0.19517 (10)	0.0669 (4)
O4	0.48076 (9)	0.7824 (2)	0.51780 (10)	0.0590 (4)
O5	0.34262 (11)	0.8314 (3)	0.55577 (10)	0.0777 (5)
N1	−0.14748 (10)	0.9651 (2)	0.31080 (10)	0.0403 (3)
N2	0.00921 (9)	0.94048 (19)	0.27755 (10)	0.0351 (3)
H2N	0.0335 (15)	0.929 (3)	0.3435 (15)	0.046 (5)*
N3	0.40286 (11)	1.0453 (2)	0.16479 (11)	0.0505 (4)
N4	0.39071 (10)	0.8282 (2)	0.49550 (10)	0.0453 (4)
C1	−0.28028 (12)	0.9200 (2)	0.16129 (13)	0.0425 (4)
H1	−0.3480	0.9069	0.1193	0.051*
C2	−0.25176 (12)	0.9531 (2)	0.26157 (13)	0.0429 (4)
H2	−0.2996	0.9672	0.2960	0.052*
C3	−0.09807 (11)	0.9410 (2)	0.24567 (11)	0.0331 (3)
C4	0.06477 (11)	0.9452 (2)	0.21168 (11)	0.0338 (3)
C5	0.17976 (11)	0.9424 (2)	0.25696 (11)	0.0324 (3)
C6	0.23598 (11)	0.9885 (2)	0.19236 (12)	0.0368 (4)
H6	0.2027	1.0230	0.1251	0.044*
C7	0.34216 (11)	0.9820 (2)	0.23006 (12)	0.0379 (4)
C8	0.39520 (12)	0.9255 (2)	0.32777 (12)	0.0382 (4)
H8	0.4665	0.9150	0.3507	0.046*

C9	0.33678 (11)	0.8856 (2)	0.38975 (11)	0.0352 (3)
C10	0.23081 (11)	0.8948 (2)	0.35798 (11)	0.0340 (3)
H10	0.1944	0.8698	0.4029	0.041*
O6	0.07962 (11)	0.8457 (2)	0.48713 (10)	0.0503 (3)
H6B	0.0571 (18)	0.752 (4)	0.4970 (18)	0.076 (9)*
H6C	0.0932 (18)	0.912 (3)	0.5399 (19)	0.073 (8)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0299 (2)	0.0482 (3)	0.0354 (2)	−0.00058 (15)	0.00610 (16)	0.00073 (16)
O1	0.0341 (6)	0.0737 (9)	0.0322 (6)	0.0033 (5)	0.0092 (5)	0.0028 (5)
O2	0.0531 (8)	0.1362 (15)	0.0526 (8)	−0.0101 (8)	0.0222 (7)	0.0276 (9)
O3	0.0322 (7)	0.1079 (12)	0.0660 (9)	−0.0099 (7)	0.0233 (6)	−0.0043 (8)
O4	0.0356 (6)	0.0801 (10)	0.0533 (7)	0.0133 (6)	0.0030 (5)	0.0077 (7)
O5	0.0573 (9)	0.1376 (15)	0.0406 (7)	0.0245 (9)	0.0190 (6)	0.0190 (8)
N1	0.0314 (7)	0.0498 (8)	0.0414 (7)	0.0005 (6)	0.0139 (6)	0.0001 (6)
N2	0.0260 (7)	0.0474 (8)	0.0312 (7)	−0.0003 (5)	0.0078 (5)	−0.0010 (6)
N3	0.0379 (8)	0.0734 (11)	0.0447 (8)	−0.0118 (7)	0.0197 (7)	−0.0075 (7)
N4	0.0388 (8)	0.0546 (9)	0.0393 (7)	0.0046 (6)	0.0076 (6)	0.0011 (6)
C1	0.0275 (8)	0.0450 (9)	0.0529 (10)	−0.0009 (6)	0.0096 (7)	0.0055 (7)
C2	0.0289 (8)	0.0485 (10)	0.0540 (10)	0.0014 (6)	0.0170 (7)	0.0057 (8)
C3	0.0285 (7)	0.0355 (8)	0.0349 (8)	0.0006 (6)	0.0097 (6)	0.0016 (6)
C4	0.0299 (8)	0.0377 (8)	0.0340 (8)	0.0001 (6)	0.0107 (6)	−0.0008 (6)
C5	0.0280 (8)	0.0339 (8)	0.0355 (8)	−0.0005 (5)	0.0106 (6)	−0.0036 (6)
C6	0.0329 (8)	0.0437 (9)	0.0339 (8)	−0.0024 (6)	0.0107 (6)	−0.0032 (7)
C7	0.0329 (8)	0.0449 (9)	0.0398 (8)	−0.0054 (6)	0.0169 (7)	−0.0069 (7)
C8	0.0281 (7)	0.0435 (9)	0.0424 (9)	−0.0005 (6)	0.0105 (6)	−0.0091 (7)
C9	0.0322 (8)	0.0377 (8)	0.0341 (8)	0.0024 (6)	0.0082 (6)	−0.0037 (6)
C10	0.0330 (8)	0.0358 (8)	0.0352 (8)	0.0008 (6)	0.0137 (6)	−0.0021 (6)
O6	0.0545 (8)	0.0628 (9)	0.0371 (7)	−0.0040 (7)	0.0195 (6)	−0.0006 (6)

Geometric parameters (Å, º)

S1—C1	1.7187 (16)	C1—H1	0.9300
S1—C3	1.7304 (15)	C2—H2	0.9300
O1—C4	1.2205 (18)	C4—C5	1.500 (2)
O2—N3	1.215 (2)	C5—C10	1.391 (2)
O3—N3	1.2147 (18)	C5—C6	1.391 (2)
O4—N4	1.2162 (17)	C6—C7	1.382 (2)
O5—N4	1.2171 (18)	C6—H6	0.9300
N1—C3	1.299 (2)	C7—C8	1.375 (2)
N1—C2	1.377 (2)	C8—C9	1.376 (2)
N2—C4	1.3618 (19)	C8—H8	0.9300
N2—C3	1.3947 (19)	C9—C10	1.379 (2)
N2—H2N	0.871 (19)	C10—H10	0.9300
N3—C7	1.477 (2)	O6—H6B	0.75 (3)
N4—C9	1.471 (2)	O6—H6C	0.84 (3)

C1—C2	1.338 (2)		

C1—S1—C3	88.30 (8)	O1—C4—C5	121.21 (13)
C3—N1—C2	109.65 (14)	N2—C4—C5	117.15 (13)
C4—N2—C3	123.06 (13)	C10—C5—C6	119.83 (13)
C4—N2—H2N	126.6 (12)	C10—C5—C4	123.35 (13)
C3—N2—H2N	110.2 (12)	C6—C5—C4	116.81 (13)
O3—N3—O2	124.34 (15)	C7—C6—C5	118.77 (14)
O3—N3—C7	117.90 (15)	C7—C6—H6	120.6
O2—N3—C7	117.74 (14)	C5—C6—H6	120.6
O4—N4—O5	123.90 (14)	C8—C7—C6	123.06 (14)
O4—N4—C9	118.23 (13)	C8—C7—N3	117.61 (14)
O5—N4—C9	117.87 (13)	C6—C7—N3	119.28 (14)
C2—C1—S1	110.49 (12)	C7—C8—C9	116.21 (14)
C2—C1—H1	124.8	C7—C8—H8	121.9
S1—C1—H1	124.8	C9—C8—H8	121.9
C1—C2—N1	116.08 (14)	C8—C9—C10	123.66 (14)
C1—C2—H2	122.0	C8—C9—N4	117.92 (14)
N1—C2—H2	122.0	C10—C9—N4	118.41 (13)
N1—C3—N2	120.67 (14)	C9—C10—C5	118.33 (13)
N1—C3—S1	115.47 (11)	C9—C10—H10	120.8
N2—C3—S1	123.84 (11)	C5—C10—H10	120.8
O1—C4—N2	121.63 (13)	H6B—O6—H6C	108 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*N*···O6	0.871 (19)	1.974 (19)	2.8313 (18)	167.9 (17)
O6—H6*B*···O1^{i}	0.75 (3)	2.38 (2)	3.0350 (19)	147 (2)
O6—H6*C*···N1^{ii}	0.84 (3)	2.14 (3)	2.964 (2)	168 (2)

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