

2-Amino-6-nitrobenzo-1,3-thiazole hydrate

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Key indicators

Single-crystal X-ray study

T = 150 K

Mean $\sigma(\text{C}-\text{C}) = 0.003 \text{ \AA}$

R factor = 0.039

wR factor = 0.105

Data-to-parameter ratio = 11.3

For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

The structure of the title compound, $\text{C}_7\text{H}_5\text{N}_3\text{O}_2\text{S}\cdot\text{H}_2\text{O}$, comprises molecules of the thiazole in an extensive hydrogen-bonding network with lattice water molecules. The essentially planar thiazole forms centrosymmetric, hydrogen-bonded dimers *via* a pair of $\text{N}-\text{H}\cdots\text{N}$ ($\text{H}\cdots\text{N}$ 2.11 Å) associations. The second amino H atom hydrogen bonds to the water O atom, while the water associates to one nitro O atom and two other water molecules. Complexity in the hydrogen-bonding network arises because the water molecule is close to an inversion centre, thus producing an $\text{Ow}\cdots\text{Ow}$ interaction.

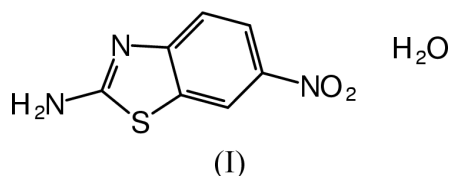
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Comment

A multitude of crystal structures of 2-aminobenzo-1,3-thiazole and its derivatives have been determined and provide an interesting subset of compounds in the 2-amino-1,3-thiazole range. Of the structures reported, most are pure organics that have fragments attached to the amino N atom. The structure of 2-aminobenzo-1,3-thiazole itself is unknown, but of the ring-substituted compounds, the structures of the 6-fluoro (Jainhuknan *et al.*, 1997), and both 4-nitro (Lokaj *et al.*, 1996) and 6-nitro (Glidewell *et al.*, 2001) analogues have been determined. In these three cases, the recorded hydrogen-bonding patterns are noteworthy, as well as the bond distances in the two nitro derivatives. In 2-amino-4-nitrobenzo-1,3-thiazole, the $\text{C}-\text{NH}_2$ and ring $\text{C}-\text{N}$ distances are equal to within 2σ [1.315 (2) and 1.312 (2) Å, respectively], a feature indicating complete π -electron delocalization. The corresponding bond distances in 2-amino-6-nitrobenzo-1,3-thiazole are 1.331 (3) and 1.321 (3) Å, and in 2-amino-6-fluorobenzo-1,3-thiazole are 1.358 (2) and 1.295 (2) Å, respectively [average values: 1.336 (17) and 1.313 (11) Å; Allen *et al.*, 1987]. The hydrogen-bonding patterns in all three structures include a dimer formation about the $\text{N}21-\text{H}\cdots\text{N}3$ association and, for both nitro analogues, at least one nitro O atom is also involved in the hydrogen-bonding array.



In this paper, the single-crystal structure of 2-amino-6-nitrobenzo-1,3-thiazole hydrate, (I), is reported as an addition to the non-hydrate structure. The hydrate structure was obtained from attempts to produce adducts of 2-amino-6-nitrobenzo-1,3-thiazole with aromatic carboxylic acids, as performed for 2-aminobenzo-1,3-thiazole (Lynch *et al.*, 1998, 1999).

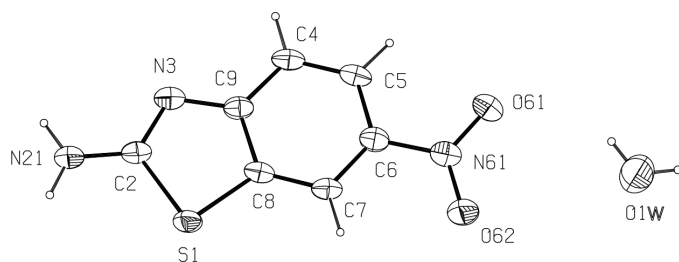


Figure 1
The molecular configuration and atom-numbering scheme for the title compound, showing 50% probability ellipsoids.

The structure of (I) (Fig. 1) comprises the thiazole molecule associated in an extensive hydrogen-bonded network with the water molecule. Hydrogen-bonding parameters are listed in Table 2. The bond lengths across the N21—C2—N3 site show a similar pattern to the 4-nitro analogue, with both bond lengths being equal [1.320 (3) Å]. The formation of dimers *via* the N21—H···N3 association is expected for this type of compound, but the location of the water molecule so near to a symmetry element provides an interesting hydrogen-bonding network (Fig. 2). The water network extends parallel to the *a* axis, while the thiazole molecules are inclined to the (100) plane. The water O atom is bound by the second amino H atom, while H1W associates to a nitro O atom. The suggested position of H2W allows for a three-centre association, linking two symmetry-generated water molecules.

Experimental

Crystals of the title compound were separated from a partially evaporated ethanol solution containing an equimolar amount of 3-aminobenzoic acid.

Crystal data

$C_7H_5N_3O_2S \cdot H_2O$	$D_x = 1.621 \text{ Mg m}^{-3}$
$M_r = 213.22$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 5386 reflections
$a = 3.8930$ (2) Å	$\theta = 2.9\text{--}32.0^\circ$
$b = 10.8743$ (5) Å	$\mu = 0.35 \text{ mm}^{-1}$
$c = 20.6378$ (8) Å	$T = 150$ (2) K
$\beta = 91.335$ (4) $^\circ$	Block, yellow
$V = 873.44$ (7) Å ³	$0.20 \times 0.15 \times 0.10 \text{ mm}$
$Z = 4$	

Data collection

Bruker–Nonius KappaCCD area-detector diffractometer	1504 independent reflections
φ and ω scans	1247 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SORTAV; Blessing, 1995)	$R_{\text{int}} = 0.037$
$T_{\text{min}} = 0.933$, $T_{\text{max}} = 0.970$	$\theta_{\text{max}} = 25.0^\circ$
5105 measured reflections	$h = -4 \rightarrow 4$
	$k = -12 \rightarrow 12$
	$l = -20 \rightarrow 24$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0469P)^2 + 0.3321P]$
$R[F^2 > 2\sigma(F^2)] = 0.039$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.105$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.14$	$\Delta\rho_{\text{max}} = 0.32 \text{ e \AA}^{-3}$
1504 reflections	$\Delta\rho_{\text{min}} = -0.25 \text{ e \AA}^{-3}$
133 parameters	
H-atom parameters constrained	

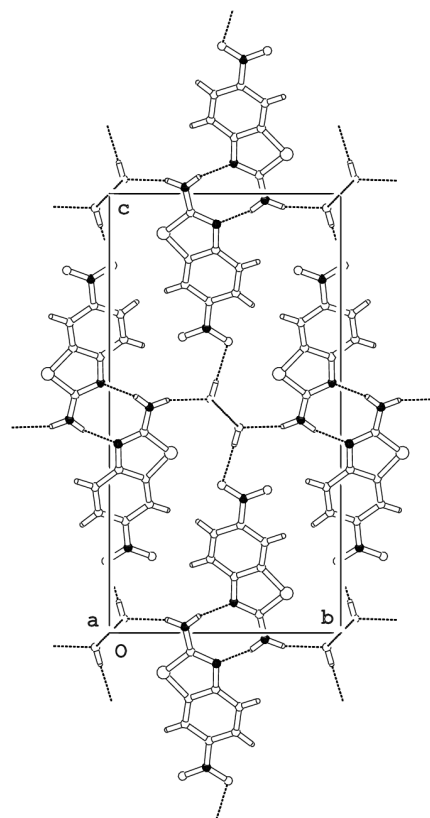


Figure 2
Packing diagram for the title compound. Hydrogen-bonding associations are shown as dotted lines.

Table 1

Selected interatomic distances (Å).

S1—C8	1.740 (2)	C2—N3	1.320 (3)
S1—C2	1.766 (2)	C2—N21	1.320 (3)

Table 2

Hydrogen-bonding geometry (Å, $^\circ$).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
N21—H21···N3 ⁱ	0.88	2.11	2.970 (3)	165
N21—H22···O1W ⁱⁱⁱ	0.88	2.11	2.946 (3)	159
O1W—H1W···O61 ⁱⁱⁱ	0.84	2.06	2.894 (3)	175
O1W—H2W···O1W ^{iv}	0.81	2.17	2.772 (6)	132
O1W—H2W···O1W ^v	0.81	2.30	2.651 (8)	107

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

All thiazole H atoms were included in the refinement, at calculated positions, as riding models, with C—H set to 0.95 Å and N—H set to 0.88 Å. H1W was initially located in a difference synthesis, while H2W was generated at the most probable position based on the position of H1W and the positions of adjacent symmetry-generated water molecules. Both H1W and H2W were restrained to an O—H distance of 0.83 Å and an H···H distance of 1.35 Å, while displacement parameters were refined as riding models.

Data collection: DENZO (Otwinowski & Minor, 1997) and COLLECT (Hooft, 1998); cell refinement: DENZO and COLLECT; data reduction: DENZO and COLLECT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine

structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON97* (Spek, 1997); software used to prepare material for publication: *SHELXL97*.

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