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4-Acetamido-*N*-(3-amino-1,2,4-triazol-1-yl)benzenesulfonamide

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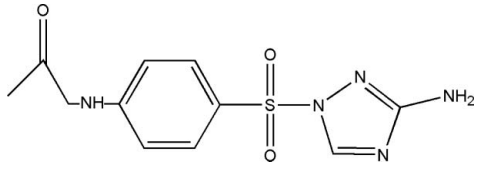
Key indicators: single-crystal X-ray study; T = 299 K; mean σ(C–C) = 0.002 Å; R factor = 0.040; wR factor = 0.112; data-to-parameter ratio = 15.1.

The title compound, $C_{10}H_{11}N_5O_3S$, was obtained unintentionally when our group attempted to synthesize a precursor of a copper corrosion inhibitor using *p*-acetylamidobenzenesulfonyl chloride and 3-amino-1-*H*-1,2,4-triazole. In the molecule the dihedral angle between the benzene ring and the triazole ring is 85.10 (6)°. In the crystal structure, molecules are linked by N—H···O and N—H···N hydrogen bonds to form a three-dimensional network.

Related literature

For related literature, see: Mai (2001); Sherif *et al.* (2007).



Experimental

Crystal data

$C_{10}H_{11}N_5O_3S$
M_r = 281.30
Monoclinic, $P2_1/c$
a = 7.6520 (5) Å
b = 11.2724 (8) Å
c = 14.0331 (10) Å
β = 94.047 (1)°
V = 1207.43 (14) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.28 mm^{-1}
T = 299 (2) K
0.30 × 0.30 × 0.25 mm

Data collection

Bruker APEX CCD area-detector diffractometer
Absorption correction: multi-scan *SADABS* (Sheldrick, 1996)
T_{min} = 0.920, T_{max} = 0.933
9872 measured reflections
2744 independent reflections
2513 reflections with $I > 2\sigma(I)$
R_{int} = 0.022

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.112
S = 1.04
2744 reflections
182 parameters
3 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···N3^{i}	0.857 (9)	2.198 (10)	3.0547 (18)	179 (2)
N5—H5A···O1^{ii}	0.852 (9)	2.230 (13)	2.9906 (19)	148.8 (18)
N5—H5B···N4^{iii}	0.867 (9)	2.189 (10)	3.0503 (18)	172.0 (19)
N5—H5A···O2	0.852 (9)	2.269 (17)	2.8848 (19)	129.3 (17)

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

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXTL* (Bruker, 2000).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2398).

References

Bruker (2000). *SMART*, *SAINT* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Mai, L. G. (2001). *Organism Synthesis Experiment*, 1st ed., pp. 171–172. Beijing: Higher Education Publishing Company.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Sherif, E. M., Erasmus, R. M. & Comins, J. D. (2007). *J. Colloid Interface Sci.* pp. 1–8.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

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supplementary materials

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4-Acetamido-*N*-(3-amino-1,2,4-triazol-1-yl)benzenesulfonamide

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Comment

The title molecule can easily form complexes with copper due to it containing many hetero atoms, so it can act as a type of copper corrosion inhibitor (Sherif *et al.*, 2007). Theoretically, the protons on atom N5 are more active than the proton on atom N2, therefore, when *p*-acetylamidobenzenesulfonyl chloride reacts with 3-amino-1,2,4-trizole, it should be the active proton which reacts first react (Mai, 2001), but in our experiment, the proton at atom N2 is involved in the reaction. This X-ray study denied the previously molecular structure referred to in the literature. Therefore, the activity of proton N5 under this condition is more active than the proton N2.

Experimental

The title compound was prepared by 3-amino-1-H-1,2,4-trizole (22 mmol) and *p*-acetamino benzene solfonyl chloride (25 mmol) in dry pyridine for 3 h. 3-amino-1-H −1,2,4-trizole was prepared by reacting aminoguanidine bicarbonate (250 mmol) and formic acid (17 mL) for 5 h. Then *p*-acetamino benzene solfonyl chloride was prepared from chlorosofonic acid (0.2 mol) and acetanilide (0.07 mol) in ice water bath. The solid product was collected by filtration. Single crystals suitable for crystallographic analysis were obtained by slow evaporation of a saturated THF-CH_3OH(1:1) solution of the title compound at room temperature.

Refinement

All the H-atoms were discernible in the difference Fourier map but H atoms bound to C atoms were included in calculated positions and allowed to ride during refinement, with C–H = 0.93—0.96 Å and U_{iso}(H) = 1.2U_{eq} (C of aromatic) or 1.5U_{eq}(C of methyl). H atoms bound to N atoms were located in a difference Fourier map and refined with restraints for N—H = 0.86 (1) Å, and U_{iso}(H) = 1.2U_{eq}(N).

Figures

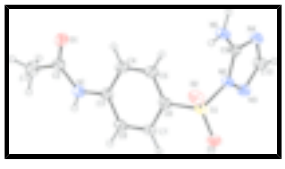

Fig. 1. The molecular structure of (I) with displacement ellipsoids drawn at the 50% probability level.

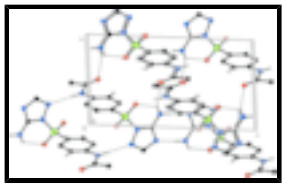

Fig. 2. The packing of the title molecule with hydrogen bonds shown as dashed lines.

4-Acetamido-*N*-(3-amino-1,2,4-triazol-1-yl)benzenesulfonamide

Crystal data

$C_{10}H_{11}N_5O_3S$	F_{000} = 584
M_r = 281.30	D_x = 1.547 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 6534 reflections
a = 7.6520 (5) Å	θ = 2.3–28.2º
b = 11.2724 (8) Å	μ = 0.28 mm^{-1}
c = 14.0331 (10) Å	T = 299 (2) K
β = 94.0470 (10)º	Block, colorless
V = 1207.43 (14) Å^3	0.30 × 0.30 × 0.25 mm
Z = 4	

Data collection

Bruker APEX CCD area-detector diffractometer	2744 independent reflections
Radiation source: fine-focus sealed tube	2513 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.022
T = 299(2) K	θ_{max} = 27.5º
φ and ω scans	θ_{min} = 2.3º
Absorption correction: multi-scan SADABS (Sheldrick, 1996)	h = −9→9
T_{min} = 0.920, T_{max} = 0.933	k = −8→14
9872 measured reflections	l = −18→18

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
$wR(F^2)$ = 0.112	$w = 1/[\sigma^2(F_o^2) + (0.0659P)^2 + 0.3863P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.04	$(\Delta/\sigma)_{max}$ = 0.045
2744 reflections	$\Delta\rho_{max}$ = 0.28 e Å^{-3}
182 parameters	$\Delta\rho_{min}$ = −0.35 e Å^{-3}
3 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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 $F^2 > 2\text{sigma}(F^2)$ 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}
C1	0.4554 (3)	0.16048 (18)	−0.09977 (14)	0.0562 (5)
H1A	0.5714	0.1277	−0.0965	0.084*
H1B	0.4347	0.2040	−0.1583	0.084*
H1C	0.3713	0.0975	−0.0977	0.084*
C2	0.4378 (2)	0.24186 (16)	−0.01687 (11)	0.0408 (3)
C3	0.29405 (19)	0.43052 (14)	0.02422 (10)	0.0373 (3)
C4	0.3081 (3)	0.41761 (19)	0.12380 (12)	0.0566 (5)
H4	0.3579	0.3496	0.1516	0.068*
C5	0.2474 (3)	0.50655 (19)	0.18019 (11)	0.0571 (5)
H5	0.2568	0.4984	0.2463	0.069*
C6	0.1727 (2)	0.60772 (14)	0.13931 (10)	0.0373 (3)
C7	0.1588 (2)	0.62137 (14)	0.04080 (10)	0.0392 (3)
H7	0.1078	0.6892	0.0133	0.047*
C8	0.2210 (2)	0.53344 (15)	−0.01580 (10)	0.0402 (3)
H8	0.2142	0.5431	−0.0818	0.048*
C9	−0.00719 (19)	0.60099 (13)	0.37182 (9)	0.0336 (3)
C10	−0.2654 (2)	0.56760 (16)	0.31668 (12)	0.0456 (4)
H10	−0.3798	0.5393	0.3138	0.055*
N1	0.34335 (18)	0.34180 (13)	−0.03806 (9)	0.0421 (3)
H1	0.306 (3)	0.3514 (19)	−0.0966 (8)	0.051*
N2	−0.03538 (17)	0.65192 (12)	0.28261 (8)	0.0366 (3)
N3	−0.20576 (18)	0.62626 (13)	0.24615 (9)	0.0423 (3)
N4	−0.15328 (17)	0.54938 (13)	0.39494 (9)	0.0417 (3)
N5	0.14407 (18)	0.60225 (14)	0.42474 (9)	0.0437 (3)
H5A	0.229 (2)	0.6426 (17)	0.4060 (14)	0.052*
H5B	0.152 (3)	0.5649 (16)	0.4789 (9)	0.052*
O1	0.50430 (17)	0.21926 (12)	0.06242 (9)	0.0529 (3)
O2	0.23927 (18)	0.76165 (12)	0.27614 (9)	0.0534 (3)
O3	−0.00441 (18)	0.80197 (11)	0.15504 (9)	0.0508 (3)
S1	0.09973 (5)	0.72107 (3)	0.21183 (2)	0.03818 (14)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0618 (11)	0.0550 (11)	0.0515 (10)	0.0085 (9)	0.0010 (8)	−0.0103 (8)
C2	0.0335 (7)	0.0481 (9)	0.0405 (8)	−0.0008 (6)	0.0015 (6)	−0.0006 (7)
C3	0.0349 (7)	0.0459 (8)	0.0307 (7)	−0.0005 (6)	−0.0005 (5)	0.0010 (6)
C4	0.0801 (12)	0.0578 (11)	0.0315 (8)	0.0267 (10)	0.0008 (8)	0.0082 (7)
C5	0.0819 (13)	0.0631 (11)	0.0261 (7)	0.0244 (10)	0.0012 (7)	0.0068 (7)
C6	0.0406 (7)	0.0423 (8)	0.0291 (7)	−0.0013 (6)	0.0019 (5)	0.0021 (6)
C7	0.0479 (8)	0.0387 (8)	0.0306 (7)	−0.0020 (6)	−0.0005 (6)	0.0085 (6)
C8	0.0476 (8)	0.0466 (9)	0.0260 (6)	−0.0036 (7)	−0.0003 (6)	0.0056 (6)
C9	0.0435 (7)	0.0310 (7)	0.0263 (6)	0.0021 (6)	0.0022 (5)	−0.0011 (5)
C10	0.0410 (8)	0.0545 (10)	0.0407 (8)	−0.0060 (7)	−0.0015 (6)	0.0078 (7)
N1	0.0452 (7)	0.0503 (8)	0.0300 (6)	0.0050 (6)	−0.0035 (5)	−0.0010 (6)
N2	0.0399 (6)	0.0422 (7)	0.0273 (6)	−0.0029 (5)	0.0006 (5)	0.0037 (5)
N3	0.0406 (7)	0.0517 (8)	0.0337 (6)	−0.0033 (6)	−0.0035 (5)	0.0044 (6)
N4	0.0430 (7)	0.0478 (8)	0.0341 (6)	−0.0019 (6)	0.0019 (5)	0.0072 (5)
N5	0.0452 (7)	0.0522 (8)	0.0325 (6)	−0.0082 (6)	−0.0046 (5)	0.0081 (6)
O1	0.0503 (7)	0.0638 (8)	0.0435 (6)	0.0151 (6)	−0.0052 (5)	0.0021 (6)
O2	0.0607 (8)	0.0573 (8)	0.0416 (6)	−0.0213 (6)	−0.0002 (6)	−0.0023 (6)
O3	0.0710 (8)	0.0382 (6)	0.0436 (6)	0.0053 (6)	0.0060 (6)	0.0099 (5)
S1	0.0479 (2)	0.0357 (2)	0.0309 (2)	−0.00552 (15)	0.00257 (16)	0.00326 (13)

Geometric parameters (Å, °)

C1—C2	1.495 (2)	C7—H7	0.9300
C1—H1A	0.9600	C8—H8	0.9300
C1—H1B	0.9600	C9—N4	1.321 (2)
C1—H1C	0.9600	C9—N5	1.3304 (19)
C2—O1	1.217 (2)	C9—N2	1.3803 (17)
C2—N1	1.360 (2)	C10—N3	1.299 (2)
C3—C8	1.389 (2)	C10—N4	1.361 (2)
C3—N1	1.397 (2)	C10—H10	0.9300
C3—C4	1.402 (2)	N1—H1	0.857 (9)
C4—C5	1.378 (3)	N2—N3	1.3968 (18)
C4—H4	0.9300	N2—S1	1.6757 (13)
C5—C6	1.382 (2)	N5—H5A	0.852 (9)
C5—H5	0.9300	N5—H5B	0.867 (9)
C6—C7	1.3876 (19)	O2—S1	1.4240 (13)
C6—S1	1.7494 (16)	O3—S1	1.4185 (12)
C7—C8	1.376 (2)		
C2—C1—H1A	109.5	C7—C8—H8	119.5
C2—C1—H1B	109.5	C3—C8—H8	119.5
H1A—C1—H1B	109.5	N4—C9—N5	125.85 (13)
C2—C1—H1C	109.5	N4—C9—N2	108.98 (13)
H1A—C1—H1C	109.5	N5—C9—N2	125.16 (14)
H1B—C1—H1C	109.5	N3—C10—N4	117.16 (15)

O1—C2—N1	123.68 (16)	N3—C10—H10	121.4
O1—C2—C1	121.88 (16)	N4—C10—H10	121.4
N1—C2—C1	114.44 (14)	C2—N1—C3	128.23 (13)
C8—C3—N1	117.61 (13)	C2—N1—H1	116.7 (14)
C8—C3—C4	119.35 (15)	C3—N1—H1	115.0 (14)
N1—C3—C4	122.97 (15)	C9—N2—N3	109.16 (12)
C5—C4—C3	119.46 (16)	C9—N2—S1	131.89 (11)
C5—C4—H4	120.3	N3—N2—S1	118.56 (9)
C3—C4—H4	120.3	C10—N3—N2	101.25 (12)
C4—C5—C6	120.55 (14)	C9—N4—C10	103.39 (13)
C4—C5—H5	119.7	C9—N5—H5A	119.2 (14)
C6—C5—H5	119.7	C9—N5—H5B	119.4 (13)
C5—C6—C7	120.37 (15)	H5A—N5—H5B	121.3 (19)
C5—C6—S1	120.06 (11)	O3—S1—O2	121.05 (8)
C7—C6—S1	119.55 (12)	O3—S1—N2	106.64 (7)
C8—C7—C6	119.32 (14)	O2—S1—N2	103.89 (7)
C8—C7—H7	120.3	O3—S1—C6	109.43 (7)
C6—C7—H7	120.3	O2—S1—C6	110.33 (8)
C7—C8—C3	120.93 (13)	N2—S1—C6	103.93 (7)
C8—C3—C4—C5	0.7 (3)	N4—C10—N3—N2	−1.2 (2)
N1—C3—C4—C5	−176.32 (19)	C9—N2—N3—C10	2.35 (17)
C3—C4—C5—C6	0.2 (3)	S1—N2—N3—C10	176.00 (12)
C4—C5—C6—C7	−0.4 (3)	N5—C9—N4—C10	−176.97 (16)
C4—C5—C6—S1	−178.89 (17)	N2—C9—N4—C10	1.91 (18)
C5—C6—C7—C8	−0.4 (2)	N3—C10—N4—C9	−0.4 (2)
S1—C6—C7—C8	178.12 (12)	C9—N2—S1—O3	−152.26 (14)
C6—C7—C8—C3	1.4 (2)	N3—N2—S1—O3	35.81 (13)
N1—C3—C8—C7	175.68 (14)	C9—N2—S1—O2	−23.32 (16)
C4—C3—C8—C7	−1.5 (3)	N3—N2—S1—O2	164.76 (12)
O1—C2—N1—C3	−4.9 (3)	C9—N2—S1—C6	92.15 (15)
C1—C2—N1—C3	175.40 (16)	N3—N2—S1—C6	−79.77 (13)
C8—C3—N1—C2	170.17 (15)	C5—C6—S1—O3	−167.40 (15)
C4—C3—N1—C2	−12.7 (3)	C7—C6—S1—O3	14.06 (15)
N4—C9—N2—N3	−2.79 (17)	C5—C6—S1—O2	57.04 (17)
N5—C9—N2—N3	176.09 (15)	C7—C6—S1—O2	−121.49 (13)
N4—C9—N2—S1	−175.29 (12)	C5—C6—S1—N2	−53.79 (16)
N5—C9—N2—S1	3.6 (2)	C7—C6—S1—N2	127.67 (13)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···N3^{i}	0.857 (9)	2.198 (10)	3.0547 (18)	179 (2)
N5—H5A···O1^{ii}	0.852 (9)	2.230 (13)	2.9906 (19)	148.8 (18)
N5—H5B···N4^{iii}	0.867 (9)	2.189 (10)	3.0503 (18)	172.0 (19)
N5—H5A···O2	0.852 (9)	2.269 (17)	2.8848 (19)	129.3 (17)

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

Fig. 1

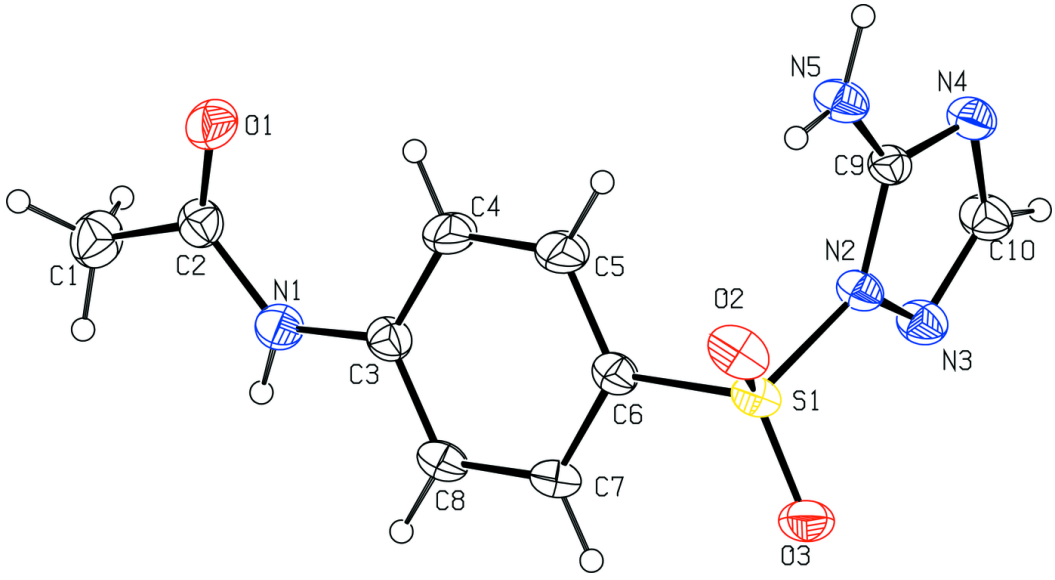

Fig. 2