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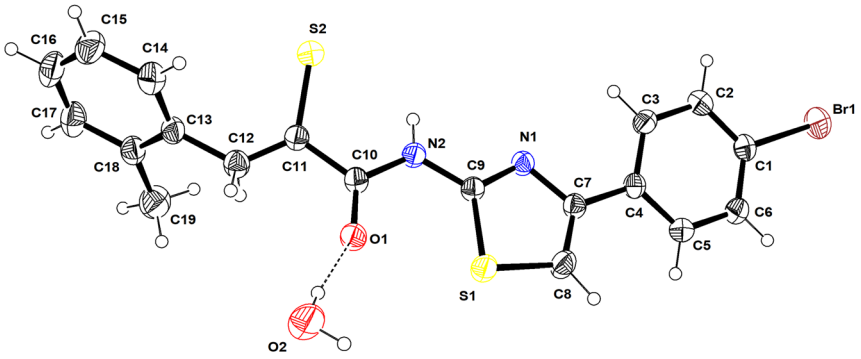
The crystal structure of *N*-[4-(4-bromophenyl)-1,3-thiazol-2-yl]-3-(2-methylphenyl)-2-sulfanylprop-2-enamide hydrate, $C_{19}H_{17}BrN_2O_2S_2$



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Abstract

$C_{19}H_{17}BrN_2O_2S_2$, triclinic, $P\bar{1}$ (no. 2), a = 7.8069(5) Å, b = 8.0787(5) Å, c = 15.7738(9) Å, α = 97.540(2)°, β = 90.179(2)°, γ = 104.507(2)°, V = 954.13(10) Å^3, Z = 2, $R_{gt}(F)$ = 0.0423, $wR_{ref}(F^2)$ = 0.1134, T = 296.15 K.

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The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

1 Source of materials

2-(4-Bromophenyl)-4-thiazolamine (0.25 g, 1 mmol) and 2-mercapto-3-(2-methylphenyl)-2-propenoic acid (0.19 g, 1 mmol) were mixed in dichloromethane (20 mL) and stirred for 10 h under the catalytic of N-(3-dimethylaminopropyl)-N′-ethylcarbodiimide hydrochloride (0.095 g, 0.5 mmol) and 1-hydroxybenzotriazole monohydrate (0.0068 g, 0.05 mmol). After filtration and drying, the title compound was obtained. Then, 5 mg of the title compound was dissolved in 5 mL of ethanol, and the solution was kept at room temperature for three days. Colourless block-shaped single crystals were obtained by slow evaporation.

2 Experimental details

Absorption corrections were applied by using multi-scan program [1]. Using Olex2 [2], the structure was solved with

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.42 × 0.23 × 0.16 mm
Wavelength:	Mo$K\alpha$ radiation (0.71073 Å)
μ:	2.39 mm^{-1}
Diffractometer, scan mode:	Bruker D8 venture
θ_{max}, completeness:	28.3°, >99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	10,730, 4580, 0.023
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 3316
$N(param)_{refined}$:	243
Programs:	Bruker [1], Olex2 [2], SHELX [3, 4]

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Br1	−0.02511 (4)	0.96692 (5)	0.79994 (2)	0.06904 (15)
C1	0.1478 (4)	0.9205 (3)	0.72251 (17)	0.0417 (6)
C2	0.1010 (4)	0.7847 (4)	0.65776 (18)	0.0450 (6)
H2A	−0.013885	0.714451	0.652199	0.054*
C3	0.2281 (4)	0.7539 (3)	0.60076 (17)	0.0413 (6)
H3	0.196943	0.663010	0.556299	0.050*
C4	0.4007 (3)	0.8561 (3)	0.60869 (15)	0.0352 (5)
C5	0.4430 (4)	0.9936 (3)	0.67508 (17)	0.0417 (6)
H5	0.557471	1.064629	0.680932	0.050*
C6	0.3177 (4)	1.0260 (4)	0.73230 (17)	0.0441 (6)
H6	0.347368	1.117241	0.776650	0.053*
C7	0.5354 (3)	0.8191 (3)	0.54920 (15)	0.0374 (5)
C8	0.7131 (4)	0.8799 (4)	0.55991 (18)	0.0512 (7)
H8	0.769974	0.954474	0.607300	0.061*
C9	0.6162 (3)	0.6900 (3)	0.42989 (15)	0.0351 (5)
C10	0.7262 (3)	0.5710 (3)	0.29823 (16)	0.0363 (5)
C11	0.6742 (3)	0.4646 (3)	0.21356 (16)	0.0372 (5)
C12	0.7777 (4)	0.4894 (4)	0.14743 (17)	0.0443 (6)
H12A	0.891071	0.475775	0.166437	0.053*
H12B	0.796375	0.610717	0.141849	0.053*
C13	0.7479 (4)	0.3989 (4)	0.05943 (17)	0.0444 (6)
C14	0.7112 (4)	0.2203 (4)	0.0438 (2)	0.0585 (8)
H14	0.701224	0.157185	0.089614	0.070*
C15	0.6892 (5)	0.1347 (5)	−0.0388 (2)	0.0687 (10)
H15	0.665674	0.014990	−0.048562	0.082*
C16	0.7024 (4)	0.2274 (5)	−0.1062 (2)	0.0657 (9)
H16	0.687110	0.170511	−0.161945	0.079*
C17	0.7378 (4)	0.4026 (5)	−0.09179 (19)	0.0602 (8)
H17	0.746490	0.463760	−0.138262	0.072*
C18	0.7614 (4)	0.4927 (4)	−0.00933 (18)	0.0476 (7)
C19	0.7960 (6)	0.6869 (4)	0.0052 (2)	0.0693 (9)
H19A	0.703065	0.719013	0.037934	0.104*
H19B	0.799404	0.729418	−0.048987	0.104*
H19C	0.907392	0.735692	0.035845	0.104*
N1	0.4802 (3)	0.7088 (3)	0.47415 (13)	0.0367 (5)
N2	0.5933 (3)	0.5875 (3)	0.35109 (14)	0.0388 (5)
O1	0.8801 (2)	0.6447 (3)	0.31849 (12)	0.0490 (5)
O2	1.2286 (3)	0.6042 (4)	0.33132 (18)	0.0836 (8)
H2B	1.255186	0.648689	0.383035	0.125*
H2C	1.121844	0.608014	0.323791	0.125*
S1	0.82041 (9)	0.80169 (11)	0.47489 (5)	0.0516 (2)
S2	0.46879 (10)	0.31741 (10)	0.20506 (5)	0.04803 (19)
H2	0.490 (4)	0.544 (4)	0.3335 (19)	0.045 (8)*

the ShelXT [3] structure solution program using intrinsic phasing and refined with the ShelXT [4] refinement package using least squares minimisation.

3 Comment

Researchers have long shown a keen interest in heterocyclic compounds that incorporate sulphur and nitrogen atoms [5, 6]. In the crystal structure of the title compound, the asymmetric unit consists of one formula unit of the title molecule and one water molecule. This compound's structure is novel and has been reported for the first time. The dihedral angle between the bromine-substituted benzene ring and the thiazole ring is 15.42°. Similarly, the dihedral angle between the methyl-substituted benzene ring and the thiazole ring is 79.58°. Furthermore, the dihedral angle between these two benzene rings is 69.05°. There is intramolecular hydrogen bond N2–H2···S2 in the molecule. In the crystal phase, molecules are bound by intermolecular hydrogen bonds N2–H2···O2, O2–H2C···O1 and O2–H2b···N1. The single-crystal structure analysis confirms that all bond lengths and bond angles of the compound fall within a reasonable range.

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References

1. Bruker. *Apex3 v. 2016.9-0 and SAINT v. 8.37A*; Bruker Axs Inc.: Madison, Wisconsin, USA, 2016.
2. Dolomanov O. V., Bourhis L. J., Gildea R. J., Howard J. A. K., Puschmann H. OLEX2: a complete structure solution, refinement and analysis program. *J. Appl. Crystallogr.* 2009, *42*, 339–341.
3. Sheldrick G. M. *SHELXTL* – integrated space-group and crystal-structure determination. *Acta Crystallogr.* 2015, *A71*, 3–8.
4. Sheldrick G. M. Crystal structure refinement with SHELXL. *Acta Crystallogr.* 2015, *C71*, 3–8.
5. Cheng K., Xue J. Y., Zhu H. L. Design, synthesis and antibacterial activity studies of thiazole derivatives as potent ecKAS III inhibitors. *Bioorg. Med. Chem. Lett.* 2013, *23*, 4235–4238.
6. Sharma D., Kumar S., Narasimhan B., Ramasamy K., Lim S. M., Shah S. A. A., Mani V. 4-(4–Bromophenyl)-thiazol-2-amine derivatives: synthesis biological activity and molecular docking study with ADME profile. *BMC Chem.* 2019, *13*, 60.