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X-ray Structure Analysis Online

Crystal Structure of 4-Amino-3-(4′-chlorophenyl)-4-*H*-[1,2,4]-triazolo-5-thiol

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The crystal structure of 4-amino-3-(4′-chlorophenyl)-4*H*-[1,2,4]-triazolo-5-thiol (ACPTT) was determined by crystallographic methods. There are two crystallographically independent molecules in the asymmetric unit and are related by pseudo inversion symmetry with each other. A number of C–H···N and N–H···N types of intermolecular interactions stabilize the molecules in the unit cell in addition to van der Waals forces.

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Condensed 1,2,4-triazoles are biologically important compounds and are used as starting materials for the synthesis of many heterocycles. Apart from its extensive chemical significance, the 1,2,4-triazole nucleus is found to be associated with diverse medicinal properties and incorporated into a wide variety of therapeutically interesting drugs. The crystal structure determination of the title compound is carried out to establish the conformational aspects of the molecule (Fig. 1).

The crystal and experimental details are given in Table 1. The structure was solved by direct methods and refined by full-matrix least-squares to a final reliability value of 0.0995. All of the non-hydrogen atoms were refined anisotropically and the H atoms were allowed to ride on the parent atoms in the model. The atomic coordinates of the non-hydrogen atoms are presented in Table 2.

A ZORTEP plot of the molecule is shown in Fig. 2. There are two crystallographically independent molecules in the asymmetric unit. It is to be noted that the two molecules are related by pseudo inversion symmetry with each other. An in depth analysis of the coordinates in molecules A and B reveal some interesting features on the arrangement of the molecules in the unit cell. The *y*-coordinates of both molecules are approximately similar, whereas a translation of 1/2 and 1/2 in *x* and *z* coordinates lead to one and the same structure. This implies the possibility that the crystals can grow in monoclinic space group too, depending on the environment. In the present situation, the system can be described as a pseudo B-centered lattice upon translation of the coordinates by (1/2 0 1/2).

The bond lengths and bond angles are given in Table 3. The bond lengths and bond angles in both the molecules are comparable with the values reported in the literature.[1-3] The bonds N3–C2 = [1.316(6); 1.321(6)Å] and N4–C5 = [1.308(5); 1.309(5)Å] in molecules A and B, respectively show a double-bond character. The other N–C bonds in both of the molecules show an intermediate character, indicating a delocalization of the electron cloud around the C2–N3–N4–C5 atoms. The bond lengths C2–S13 = [1.673(4); 1.675(4)Å] in both molecules are comparable with the reported values.[4]

The triazole ring is planar, as can be seen from the torsion

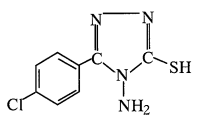

Fig. 1 Chemical diagram of ACPTT.

Table 1 Crystal and experimental data

CCDC No.	245938
Empirical formula	$C_8H_7N_4SCl$
Formula weight	226.69
Crystal system	Triclinic
Space group	$P\bar{1}$
Unit cell dimensions	a = 8.035(2)Å
	b = 11.496(2)Å
	c = 11.752(4)Å
	α = 66.58(1)°
	β = 73.07(1)°
	γ = 76.11(1)°
Z	4
V	943.3(4)Å^3
T	293(2)K
D_x	1.596 Mg/m^3
μ	5.354 mm^{-1}
Radiation (Cu K_α)	λ = 1.5418 Å
F(0 0 0)	464
θ range for data collection	4.20 to 72.43°
Reflections collected/unique	3811/3625 [R(int) = 0.0962]
Completeness to 2θ = 71.95	97.4%
Refinement method	Full-matrix least-squares on F^2
No. of parameters	256
Goodness-of-fit on F_2	1.415
Final R indices [1 > 2s(I)]	R1 = 0.0995, wR2 = 0.3010
R indices (all data)	R1 = 0.1085, wR2 = 0.3121
$(\Delta\rho)_{max}$	0.726 e.Å^{-3}
$(\Delta\rho)_{min}$	–0.807 e.Å^{-3}
Measurement: Siemens SMART CCD	
Programme system: SAINT	
Structure determination: SHELXS97	
Refinement: SHELXL97	
Structure drawing: ZORTEP	

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Table 2 Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($Å^2 \times 10^3$) for the non-hydrogen atoms

Atom	*x*	*y*	*z*	$*U_{(eq)}$
Cl1A	-125(1)	8085(1)	3906(1)	56(1)
N1A	5140(4)	2190(3)	5113(3)	40(1)
C2A	5903(5)	1104(4)	5959(4)	42(1)
N3A	5257(5)	1266(3)	7060(3)	47(1)
N4A	4143(4)	2405(3)	6987(3)	45(1)
C5A	4099(5)	2959(4)	5783(4)	37(1)
C6A	3083(5)	4222(3)	5263(4)	38(1)
C7A	1766(5)	4670(4)	6114(4)	45(1)
C8A	787(5)	5872(4)	5697(4)	47(1)
C9A	1151(5)	6617(4)	4426(4)	43(1)
C10A	2494(5)	6210(3)	3573(4)	44(1)
C11A	3481(5)	4997(3)	3984(4)	41(1)
N12A	5361(5)	2413(4)	3831(4)	53(1)
S13A	7317(2)	-75(1)	5563(1)	52(1)
Cl1B	4752(1)	7984(1)	-980(1)	54(1)
N1B	10149(4)	2126(3)	207(3)	37(1)
C2B	11071(5)	1119(4)	1019(4)	40(1)
N3B	10614(5)	1376(3)	2077(3)	49(1)
N4B	9495(5)	2502(3)	1996(3)	48(1)
C5B	9243(5)	2960(4)	837(4)	38(1)
C6B	8170(5)	4199(4)	332(4)	38(1)
C7B	7529(5)	4938(4)	1099(4)	41(1)
C8B	6508(5)	6107(4)	696(4)	45(1)
C9B	6118(5)	6544(4)	-488(4)	41(1)
C10B	6766(5)	5848(4)	-1272(4)	46(1)
C11B	7807(5)	4668(4)	-871(4)	44(1)
N12B	10247(5)	2262(3)	-1033(3)	46(1)
S13B	12487(1)	-63(1)	624(1)	48(1)

$*U_{(eq)} = (1/3)\Sigma_i\Sigma_j a_i^* a_j^* \boldsymbol{a}_i \cdot \boldsymbol{a}_j$

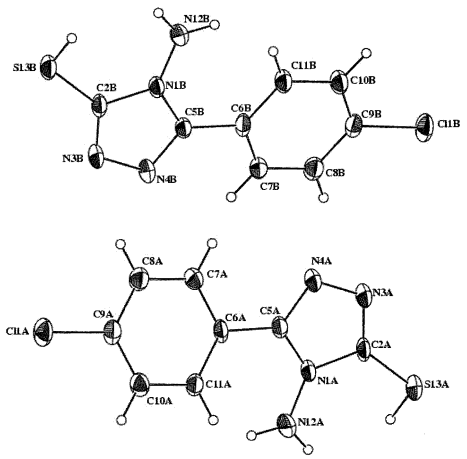

Fig. 2 ZORTEP plot of the molecules A and B of ACPTT with the thermal ellipsoids drawn at the 30% probability level.

angle values: [N1–C2–N3–N4 = –1.2(4)°, –1.7(4)°; C2–N3–N4–C5 = 0.5(5)°, 0.2(5)°; N3–N4–C5–N1 = –0.5(4)°, 1.4(4)°; N4–C5–N1–C2 = –1.3(4)°, –2.5(4)° for the molecules A and B respectively]. The dihedral angle between the phenyl ring and the triazole group in both molecules A and B are 0.2(2)° and 6.0(1)° respectively.

The packing of the molecules viewed down the *a*-axis in the unit cell is shown in Fig. 3. The molecules are stacked inversely one over the other along the *a*-direction ($\pi \cdots \pi$ interaction distance between the molecules is 3.673 Å). A number of C–H···N and N–H···N intermolecular interactions stabilize the molecules in the unit cell (Table 4).

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Table 3 Bond lengths (Å), and bond angles (°)

Atoms	Mol. A	Mol. B	Atoms	Mol. A	Mol. B
Bond length (Å)			**Bond angles (°)**		
Cl1-C9	1.729(4)	1.737(4)	C2-N1-N12	124.6(3)	123.9(3)
N1-C2	1.387(4)	1.386(4)	C2-N1-C5	108.1(3)	108.0(3)
N1-N12	1.389(5)	1.383(5)	N12-N1-C5	127.3(3)	128.0(3)
N1-C5	1.384(5)	1.383(5)	N3-C2-N1	103.6(3)	103.7(3)
C2-N3	1.316(6)	1.321(6)	N3-C2-S13	131.5(3)	131.3(3)
C2-S13	1.673(4)	1.675(4)	N1-C2-S13	124.9(3)	125.0(3)
N3-N4	1.384(5)	1.374(5)	C2-N3-N4	113.9(3)	113.7(3)
N4-C5	1.308(5)	1.309(5)	C5-N4-N3	104.4(3)	104.8(3)
C5-C6	1.471(5)	1.470(5)	N4-C5-N1	109.9(3)	109.8(3)
C6-C7	1.384(6)	1.388(6)	N4-C5-C6	123.1(4)	122.6(4)
C6-C11	1.396(5)	1.391(5)	N1-C5-C6	126.9(3)	127.6(4)
C7-C8	1.390(5)	1.376(5)	C7-C6-C11	120.0(3)	119.2(3)
C8-C9	1.378(6)	1.381(6)	C7-C6-C5	117.3(3)	117.4(4)
C9-C10	1.374(6)	1.367(6)	C11-C6-C5	122.5(3)	123.4(4)
C10-C11	1.401(5)	1.392(5)	C6-C7-C8	120.5(4)	120.9(4)
			C9-C8-C7	119.2(4)	119.2(4)
			C10-C9-C8	121.3(4)	121.0(4)
			C10-C9-C11	119.9(3)	119.3(3)
			C8-C9-C11	118.8(3)	119.7(3)
			C9-C10-C11	119.9(4)	120.0(4)
			C6-C11-C10	119.1(4)	119.7(4)

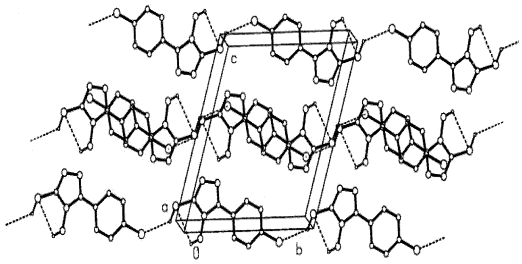

Fig. 3 Packing of the molecules viewed down the *a*-axis of ACPTT.

Table 4 Hydrogen bondings [Å & (°)]

D-H	D···A	H···A	D-H···A	*Sym
C7A -H7A 0.930(3)	C7A ...N12B 3.501(4)	H7A ...N12B 2.636(3)	C7A -H7A ...N12B 154.96(3)	(1)
C8B -H8B 0.930(5)	C8B ...N4A 3.613(7)	H8B ...N4A 2.723(4)	C8B -H8B ...N4A 160.53(3)	(2)
C10B -H10B 0.930(4)	C10B ...N12A 3.540(6)	H10B ...N12A 2.674(4)	C10B -H10B ...N12A 155.25(3)	(3)
N12B -H12C 0.860(4)	N12B ...N4A 3.308(5)	H12C ...N4A 2.835(3)	N12B -H12C ...N4A 116.34(3)	(4)

*Symmetry equivalent positions:
(1) $x - 1, +y, +z + 1$ (3) $-x + 1, -y + 1, -z$
(2) $-x + 1, -y + 1, -z + 1$ (4) $x + 1, +y, +z - 1$

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