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The crystal structure of ethyl 5-methyl-7-(4-(phenylthio)phenyl)-4,7-dihydrotetrazolo[1,5-*a*]pyrimidine-6-carboxylate, $C_{20}H_{19}N_5O_2S$

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.38 × 0.18 × 0.16 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.19 mm ⁻¹
Diffractometer, scan mode:	Rigaku R-Axis, ω
$\theta_{\max}$, completeness:	27.5°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	19,858, 4459, 0.079
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2\sigma(I_{\text{obs}})$, 2433
$N(\text{param})_{\text{refined}}$:	253
Programs:	CRYSTALCLEAR [1], OLEX2 [2], SHELX [3, 4], Diamond [5]

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Abstract

$C_{20}H_{19}N_5O_2S$, monoclinic, $C2/c$ (no. 15), $a = 18.861(4)$ Å, $b = 12.000(2)$ Å, $c = 17.787(4)$ Å, $\beta = 105.15(3)^\circ$, $V = 3885.7(15)$ Å³, $Z = 8$, $R_{\text{gt}}(F) = 0.0654$, $wR_{\text{ref}}(F^2) = 0.1538$, $T = 293(2)$ K.

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

Source of material

In a 25 mL reaction vessel, a solution of 4-(phenylthio) benzaldehyde (10 mmol), ethyl 3-oxobutanoate (10 mmol), and 5-aminotetrazole (10 mmol) in 7.5 mL propane-1,2-diol and 2 mL water was sealed and kept at 120 °C for 12 h before it was cooled to room temperature. The reaction solution

was poured into 50 mL distilled water, stirred vigorously for 10 min, and then the solid of crude tetrazolopyrimidine was filtered out. Recrystallization was performed with methanol and DMF.

Experimental details

All the hydrogen atoms were calculated geometrically and with C–H distances ranging from 0.93 to 0.98 Å. $C_{\text{aryl}}\text{--H} = 0.93$ Å, with $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$. $C_{\text{methyl}}\text{--H} = 0.96$ Å, with $U_{\text{iso}}(\text{H}) = 1.5 U_{\text{eq}}(\text{C})$.

Comment

Tetrazolo[1,5-*a*]pyrimidines and their partially hydrogenated derivatives have received widespread attention because of their strong biological activity [6]. Medicines containing tetrazolo[1,5-*a*]pyrimidine rings mainly focus on anti-tumor [7] and virus aspects [8], including very important anti-hepatitis B virus varieties and anti-HIV varieties [9]. Pesticides containing pyrimidine rings have excellent performance in the three major fields of weeding, insecticide and sterilization and even plant growth regulators [10].

The three-component reaction of amine with the synthetic precursors of the unsaturated ketone, i.e., carbonyl compound and methylene-active compound are general and useful for the preparation of variety of

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
C1	0.10293 (14)	0.4344 (2)	0.02622 (17)	0.0401 (7)
C2	0.09083 (16)	0.4750 (3)	0.22658 (19)	0.0616 (9)
H2A	0.043457	0.507081	0.203778	0.092*
H2B	0.085031	0.407634	0.253459	0.092*
H2C	0.120195	0.526884	0.262732	0.092*
C3	0.12811 (15)	0.4491 (2)	0.16359 (17)	0.0443 (7)
C4	0.19514 (14)	0.4035 (2)	0.17203 (16)	0.0398 (7)
C5	0.23776 (16)	0.3650 (3)	0.24934 (19)	0.0488 (8)
C6	0.3524 (2)	0.2852 (4)	0.3191 (2)	0.0925 (14)
H6A	0.340461	0.208485	0.327998	0.111*
H6B	0.346435	0.329980	0.362429	0.111*
C7	0.4286 (2)	0.2929 (4)	0.3127 (3)	0.1227 (19)
H7A	0.461618	0.266496	0.360011	0.184*
H7B	0.433982	0.248027	0.269813	0.184*
H7C	0.439932	0.369041	0.304108	0.184*
C8	0.22978 (13)	0.3874 (2)	0.10410 (15)	0.0375 (6)
H8	0.253152	0.313848	0.108714	0.045*
C9	0.28637 (13)	0.4753 (2)	0.09983 (15)	0.0356 (6)
C10	0.26528 (14)	0.5842 (2)	0.07807 (16)	0.0440 (7)
H10	0.215682	0.602779	0.064703	0.053*
C11	0.31654 (15)	0.6650 (3)	0.07597 (16)	0.0460 (7)
H11	0.301420	0.737646	0.062207	0.055*
C12	0.39082 (15)	0.6384 (2)	0.09433 (16)	0.0425 (7)
C13	0.41231 (14)	0.5310 (3)	0.11661 (17)	0.0463 (7)
H13	0.461923	0.512493	0.129984	0.056*
C14	0.36053 (14)	0.4504 (2)	0.11919 (16)	0.0411 (7)
H14	0.375860	0.378246	0.134205	0.049*
C15	0.53947 (15)	0.6853 (3)	0.11311 (19)	0.0481 (8)
C16	0.57127 (19)	0.6546 (3)	0.05502 (19)	0.0668 (10)
H16	0.545154	0.662157	0.003135	0.080*
C17	0.6421 (2)	0.6124 (3)	0.0734 (2)	0.0773 (11)
H17	0.663541	0.592179	0.033917	0.093*
C18	0.68028 (19)	0.6004 (3)	0.1492 (2)	0.0659 (10)
H18	0.727972	0.572653	0.161500	0.079*
C19	0.64882 (19)	0.6289 (3)	0.2069 (2)	0.0635 (9)
H19	0.674703	0.619099	0.258614	0.076*
C20	0.57885 (18)	0.6723 (3)	0.18933 (19)	0.0580 (9)
H20	0.558115	0.692886	0.229300	0.070*
N1	0.08457 (12)	0.4748 (2)	0.08991 (14)	0.0477 (6)
H1	0.046263	0.516001	0.084625	0.057*
N2	0.16881 (11)	0.38971 (19)	0.03327 (13)	0.0395 (6)
N3	0.17196 (13)	0.3534 (2)	−0.03837 (14)	0.0515 (7)
N4	0.10870 (14)	0.3774 (2)	−0.08518 (15)	0.0574 (7)
N5	0.06328 (12)	0.4289 (2)	−0.04661 (14)	0.0513 (7)
O1	0.21873 (13)	0.3666 (2)	0.30874 (13)	0.0713 (7)
O2	0.30387 (11)	0.32593 (19)	0.24614 (12)	0.0623 (6)
S1	0.45180 (5)	0.74938 (8)	0.09012 (7)	0.0760 (3)

dihydro-tetrazolopyrimidines [11]. Further, to address green chemistry principles for the synthesis of dihydro-tetrazolopyrimidines one may concern carrying out these reactions under solvothermal conditions with propane-1,2-diol and water as mixture solvents [12].

In the title molecule all bond lengths are in normal ranges [13–15]. The plane of the tetrazole forms dihedral angles of 10.8(1)° and 87.43(9)° with the two phenyl rings. The dihedral angle between the phenyl rings is 81.8(1)°. The pyrimidine ring is in a flattened boat conformation. In the crystal, N–H⋯N hydrogen bonds connect adjacent molecules into dimers.

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