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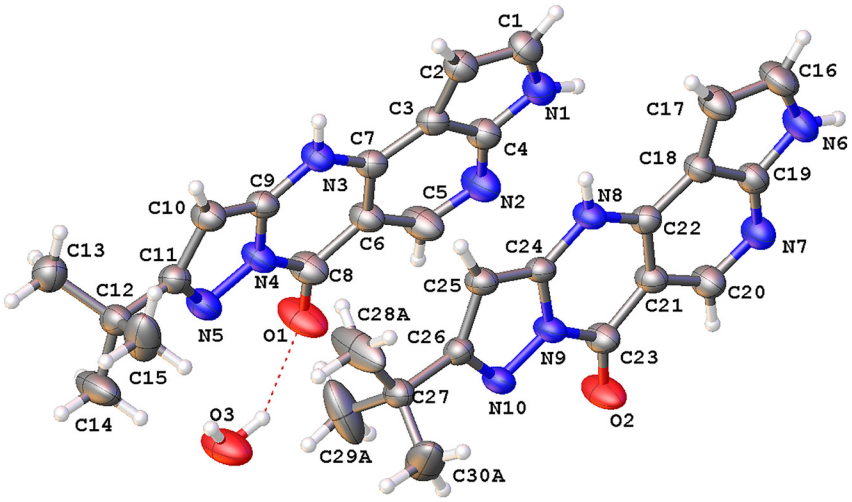
Crystal structure of 9-(*t*-butyl)-3,11-dihydro-6*H*-pyrazolo [1,5-*a*]pyrrolo[3′,2′:5,6]pyrido[4,3-*d*] pyrimidin-6-one hemihydrate, $C_{30}H_{32}N_{10}O_3$



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Abstract

$C_{30}H_{32}N_{10}O_3$, monoclinic, $P2_1/c$ (no. 14), a = 10.447(2) Å, b = 20.130(4) Å, c = 14.772(3) Å, β = 113.001(3)°, V = 2859.7(10) Å^3, Z = 4, $R_{gt}(F)$ = 0.0556, $wR_{ref}(F^2)$ = 0.1663, T = 296 K.

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The title crystal structure is shown in the figure. Tables 1 and 2 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.18 × 0.15 × 0.13 mm
Wavelength:	Mo*K*α radiation (0.71073 Å)
μ:	0.09 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	26.5°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	16503, 5904, 0.050
Criterion for I_{obs}, $N(hkl)_{gt}$:	I_{obs} > 2 $\sigma(I_{obs})$, 3552
$N(param)_{refined}$:	441
Programs:	Bruker [1], SHELX [2, 3], Olex2 [4]

Source of material

An oven-dried round bottom flask was charged with a stirring bar, 4-chloro-1*H*-pyrrolo[2,3-*b*]pyridine-5-carboxamide (100 mg, 0.5 mmol), 5-(*tert*-butyl)-1*H*-pyrazol-3-amine (70 mg, 0.5 mmol), cesium carbonate (500 mg, 1.5 mmol), and DMF (5 mL), and then the reaction mixture was stirred at 120 °C under nitrogen atmosphere for 8 h [5]. The product was obtained by column chromatography eluting with dichloromethane and methanol 15:1. The resulting solution was evaporated to get some colorless crystals.

Experimental details

All hydrogen atoms were placed in the calculated positions and all the non-hydrogen atoms were refined anisotropically.

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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}
C1	0.9093 (2)	0.93311 (11)	0.76724 (16)	0.0447 (6)
H1A	0.995356	0.946982	0.812694	0.054*
C2	0.8244 (2)	0.88836 (10)	0.78561 (16)	0.0405 (5)
H2	0.841525	0.866556	0.844711	0.049*
C3	0.7055 (2)	0.88157 (10)	0.69735 (15)	0.0357 (5)
C4	0.7251 (2)	0.92348 (10)	0.62811 (15)	0.0406 (5)
C5	0.5273 (3)	0.89385 (12)	0.50471 (17)	0.0565 (7)
H5	0.465090	0.897077	0.439506	0.068*
C6	0.4929 (2)	0.84959 (10)	0.56547 (15)	0.0405 (6)
C7	0.5830 (2)	0.84369 (10)	0.66482 (14)	0.0338 (5)
C8	0.3703 (3)	0.80853 (11)	0.52296 (16)	0.0468 (6)
C9	0.4391 (2)	0.76006 (10)	0.68822 (14)	0.0348 (5)
C10	0.3928 (2)	0.70872 (10)	0.72622 (16)	0.0422 (6)
H10	0.428050	0.694095	0.791093	0.051*
C11	0.2796 (2)	0.68216 (10)	0.64636 (15)	0.0390 (5)
C12	0.2007 (2)	0.61909 (11)	0.64614 (16)	0.0448 (6)
C13	0.1291 (3)	0.62573 (12)	0.71787 (18)	0.0533 (7)
H13A	0.074538	0.586705	0.714379	0.080*
H13B	0.069629	0.664042	0.700969	0.080*
H13C	0.197930	0.630640	0.783479	0.080*
C14	0.0935 (3)	0.60425 (16)	0.5439 (2)	0.0836 (11)
H14A	0.045125	0.563985	0.545449	0.125*
H14B	0.139195	0.599134	0.499210	0.125*
H14C	0.028223	0.640261	0.522415	0.125*
C15	0.3070 (3)	0.56253 (12)	0.6819 (2)	0.0743 (9)
H15A	0.374862	0.572998	0.746091	0.111*
H15B	0.352344	0.557101	0.637020	0.111*
H15C	0.260189	0.522052	0.684914	0.111*
C16	1.3056 (3)	0.96265 (13)	0.68488 (18)	0.0594 (7)
H16	1.381316	0.981722	0.735126	0.071*
C17	1.2237 (3)	0.91382 (13)	0.69746 (19)	0.0529 (7)
H17	1.231 (2)	0.8944 (11)	0.7545 (18)	0.053 (7)*
C18	1.1228 (2)	0.89875 (10)	0.60212 (15)	0.0375 (5)
C19	1.1494 (2)	0.94113 (10)	0.53594 (16)	0.0400 (5)
C20	0.9793 (3)	0.90075 (12)	0.40211 (17)	0.0459 (6)
H20	0.930 (2)	0.9003 (11)	0.3294 (18)	0.062 (7)*
C21	0.9410 (2)	0.85458 (10)	0.45924 (15)	0.0385 (5)
C22	1.0123 (2)	0.85388 (10)	0.56187 (15)	0.0346 (5)
C23	0.8327 (2)	0.80734 (11)	0.40892 (15)	0.0431 (6)
C24	0.8681 (2)	0.76590 (10)	0.57415 (14)	0.0324 (5)
C25	0.8088 (2)	0.71718 (10)	0.60892 (16)	0.0375 (5)
H25	0.830 (2)	0.7079 (10)	0.6788 (16)	0.046 (6)*
C26	0.7142 (2)	0.68506 (10)	0.52384 (15)	0.0353 (5)
C27	0.6283 (2)	0.62407 (11)	0.51898 (15)	0.0425 (6)
C28A[a]	0.6427 (11)	0.6027 (5)	0.6188 (5)	0.106 (4)
H28A[a]	0.736789	0.589035	0.655868	0.159*
H28B[a]	0.580903	0.566256	0.613326	0.159*
H28C[a]	0.619710	0.639123	0.651696	0.159*
C28B[b]	0.5671 (12)	0.6251 (7)	0.5902 (9)	0.103 (4)
H28D[b]	0.637203	0.635369	0.653724	0.154*
H28E[b]	0.527536	0.582439	0.592389	0.154*
H28F[b]	0.495559	0.658365	0.572422	0.154*
C29A[a]	0.4703 (6)	0.6440 (4)	0.4625 (7)	0.099 (3)
H29A[a]	0.413349	0.604934	0.449854	0.148*
H29B[a]	0.458266	0.664983	0.401348	0.148*
H29C[a]	0.443401	0.674335	0.502149	0.148*
C29B[b]	0.5347 (11)	0.6070 (5)	0.4179 (6)	0.104 (4)
H29D[b]	0.496844	0.563419	0.417048	0.156*
H29E[b]	0.460594	0.638889	0.395778	0.156*
H29F[b]	0.584224	0.607859	0.375188	0.156*
C30A[a]	0.6579 (9)	0.5726 (3)	0.4614 (7)	0.090 (2)
H30A[a]	0.631377	0.587693	0.394892	0.135*
H30B[a]	0.606333	0.533314	0.462167	0.135*
H30C[a]	0.755562	0.562831	0.489030	0.135*
C30B[b]	0.7386 (8)	0.5631 (3)	0.5540 (9)	0.096 (3)
H30D[b]	0.792391	0.562046	0.514128	0.144*
H30E[b]	0.689240	0.521905	0.546947	0.144*
H30F[b]	0.799484	0.569198	0.621662	0.144*
N1	0.8491 (2)	0.95452 (9)	0.67206 (13)	0.0452 (5)
H1	0.884081	0.982923	0.644678	0.054*
N2	0.6398 (2)	0.93116 (10)	0.53200 (14)	0.0545 (6)
N3	0.55128 (18)	0.80141 (8)	0.72508 (12)	0.0371 (4)
H3	0.602441	0.800761	0.786961	0.044*
N4	0.35224 (19)	0.76509 (8)	0.59052 (12)	0.0403 (5)
N5	0.2520 (2)	0.71599 (9)	0.56403 (13)	0.0450 (5)
N6	1.2604 (2)	0.97955 (10)	0.58755 (14)	0.0526 (6)
H6	1.296418	1.009522	0.563289	0.063*
N7	1.0797 (2)	0.94471 (9)	0.43773 (13)	0.0464 (5)
N8	0.97254 (19)	0.81096 (8)	0.61764 (13)	0.0377 (5)
H8	1.017 (2)	0.8088 (10)	0.6828 (16)	0.046 (7)*
N9	0.80704 (19)	0.76369 (8)	0.47332 (12)	0.0372 (5)
N10	0.71097 (19)	0.71314 (8)	0.44169 (12)	0.0395 (5)
O1	0.2909 (2)	0.80857 (9)	0.43710 (12)	0.0740 (6)
O2	0.7678 (2)	0.80251 (9)	0.32083 (11)	0.0683 (6)
O3	0.1075 (2)	0.70990 (10)	0.32388 (12)	0.0819 (7)
H3A	0.134034	0.687827	0.380827	0.123*
H3B	0.165584	0.744077	0.334967	0.123*

[a]Occupancy: 0.552 (6), [b]Occupancy: 0.448 (6).

Comment

Derivatives similar to the title compound have been reported to have antitumor activity, anti pulmonary fibrosis activity and janus kinase protein inhibitory activity [6–10]. Exactly the same skeleton as the title compound has not been reported in the literature, which may arouse the interest of researchers.

The asymmetric unit of the title structure contains two molecules 9-(*tert*-butyl)-3,11-dihydro-6*H*-pyrazolo [1,5-*a*] pyrrolo[3′,2′:5,6]pyrido[4,3-*d*]pyrimidin-6-one molecules and a water molecule. There is a disorder of the *t*-butyl group and the ratio is 0.552:0.448. All other values of the geometric parameters are normal. The bond lengths and angles are in the expected ranges.

Author contributions: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.

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Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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