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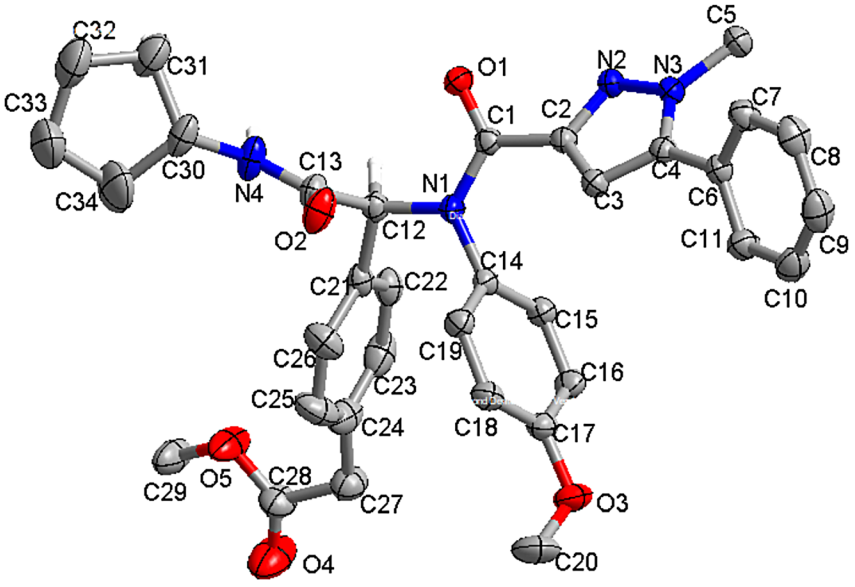
Crystal structure of methyl 2-(4-(2-(cyclopentyl-amino)-1-(*N*-(4-methoxyphenyl)-1-methyl-5-phenyl-1-*H*-pyrazole-3-carboxamido)-2-oxoethyl)phenyl)acetate, $C_{34}H_{36}N_4O_5$



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Abstract

$C_{34}H_{36}N_4O_5$, monoclinic, *C*2/*c* (no. 15), a = 29.9636(9) Å, b = 20.4000(3) Å, c = 14.2266(4) Å, β = 132.845(5)°, V = 6376.0(5) Å^3, Z = 8, $R_{gt}(F)$ = 0.0544, $wR_{ref}(F^2)$ = 0.1735, T = 293(2) K.

CCDC no.: 2122597

The asymmetric unit of the molecular structure is shown in the figure. Table 1 contains crystallographic data and

Table 1: Data collection and handling.

Crystal:	Colorless
Size:	0.17 × 0.13 × 0.10 mm
Wavelength:	CuKα radiation (1.54184 Å)
μ:	0.66 mm^{-1}
Diffractometer, scan mode:	Xcalibur, ω
θ_{max}, completeness:	67.1°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	41,586, 5692, 0.042
Criterion for I_{obs}, $N(hkl)_{gt}$:	I_{obs} > 2 σ(I_{obs}), 4502
$N(param)_{refined}$:	424
Programs:	CrysAlis^{PRO} [1], SHELX [2, 3], Olex2 [4]

Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

All starting materials are commercially available and were used without further purification. Add *p*-anisidine (123 mg,

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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	0.63138 (8)	0.29531 (9)	0.77446 (17)	0.0490 (4)
C2	0.56345 (8)	0.29590 (9)	0.67687 (17)	0.0493 (4)
C3	0.52083 (8)	0.34335 (9)	0.58964 (18)	0.0527 (4)
H3	0.5286	0.3835	0.5721	0.063*
C4	0.46506 (8)	0.31855 (9)	0.53506 (17)	0.0505 (4)
C5	0.43220 (9)	0.20854 (10)	0.5533 (2)	0.0630 (5)
H5A	0.4175	0.2180	0.5944	0.095*
H5B	0.4516	0.1664	0.5807	0.095*
H5C	0.3987	0.2084	0.4620	0.095*
C6	0.40416 (9)	0.34888 (9)	0.44077 (19)	0.0545 (4)
C7	0.36642 (10)	0.35345 (11)	0.4646 (2)	0.0666 (5)
H7	0.3784	0.3353	0.5387	0.080*
C8	0.31063 (11)	0.38526 (13)	0.3770 (3)	0.0833 (7)
H8	0.2853	0.3884	0.3929	0.100*
C9	0.29276 (12)	0.41194 (13)	0.2675 (3)	0.0860 (8)
H9	0.2553	0.4330	0.2091	0.103*
C10	0.32993 (12)	0.40767 (13)	0.2439 (2)	0.0823 (7)
H10	0.3177	0.4258	0.1694	0.099*
C11	0.38557 (10)	0.37656 (11)	0.3302 (2)	0.0678 (5)
H11	0.4108	0.3742	0.3138	0.081*
C12	0.72917 (8)	0.32897 (9)	0.85233 (18)	0.0534 (4)
H12	0.7420	0.2872	0.8985	0.064*
C13	0.75146 (9)	0.38395 (11)	0.9488 (2)	0.0630 (5)
C14	0.63274 (8)	0.35871 (9)	0.62876 (17)	0.0498 (4)
C15	0.61058 (9)	0.32050 (10)	0.52443 (18)	0.0559 (5)
H15	0.6120	0.2750	0.5309	0.067*
C16	0.58639 (10)	0.34973 (11)	0.4109 (2)	0.0649 (5)
H16	0.5709	0.3239	0.3404	0.078*
C17	0.58501 (9)	0.41741 (11)	0.4013 (2)	0.0620 (5)
C18	0.60648 (10)	0.45566 (10)	0.5046 (2)	0.0635 (5)
H18	0.6051	0.5011	0.4982	0.076*
C19	0.63016 (9)	0.42595 (10)	0.6182 (2)	0.0582 (5)
H19	0.6444	0.4517	0.6879	0.070*
C20	0.57132 (17)	0.50852 (16)	0.2785 (3)	0.1092 (11)
H20A	0.5562	0.5179	0.1950	0.164*
H20B	0.6140	0.5183	0.3424	0.164*
H20C	0.5500	0.5349	0.2932	0.164*
C21	0.75690 (8)	0.33322 (9)	0.79462 (19)	0.0550 (5)
C22	0.76365 (9)	0.27708 (11)	0.7516 (2)	0.0676 (6)
H22	0.7533	0.2368	0.7628	0.081*
C23	0.78565 (11)	0.27971 (18)	0.6919 (3)	0.0902 (9)
H23	0.7905	0.2411	0.6650	0.108*
C24	0.80040 (12)	0.3386 (2)	0.6720 (3)	0.0945 (9)
C25	0.79447 (15)	0.39357 (19)	0.7162 (4)	0.1067 (11)
H25	0.8050	0.4338	0.7050	0.128*
C26	0.77327 (12)	0.39162 (12)	0.7774 (3)	0.0834 (7)
H26	0.7701	0.4303	0.8072	0.100*
C27[a]	0.8195 (4)	0.3250 (4)	0.5961 (8)	0.0900 (17)
H27A[a]	0.8048	0.2819	0.5574	0.108*
H27B[a]	0.7992	0.3566	0.5269	0.108*
C27A[b]	0.8244 (4)	0.3595 (5)	0.6082 (9)	0.0900 (17)
H27C[b]	0.7980	0.3418	0.5219	0.108*
H27D[b]	0.8237	0.4069	0.6023	0.108*
C28[a]	0.8856 (6)	0.3277 (10)	0.6709 (14)	0.0830 (19)
C28A[b]	0.8873 (7)	0.3355 (11)	0.6833 (17)	0.0830 (19)
C29[a]	0.9806 (7)	0.3061 (16)	0.856 (2)	0.113 (4)
H29A[a]	0.9812	0.2733	0.8085	0.169*
H29B[a]	1.0023	0.2902	0.9413	0.169*
H29C[a]	0.9995	0.3454	0.8606	0.169*
C29A[b]	0.9871 (8)	0.2975 (19)	0.881 (3)	0.113 (4)
H29D[b]	1.0057	0.3187	0.8546	0.169*
H29E[b]	1.0053	0.2552	0.9160	0.169*
H29F[b]	0.9931	0.3237	0.9447	0.169*
C30[a]	0.8384 (5)	0.4146 (6)	1.1788 (10)	0.082 (2)
H30[a]	0.8140	0.4536	1.1577	0.098*
C30A[b]	0.8402 (6)	0.4279 (8)	1.1504 (12)	0.082 (2)
H30A[b]	0.8108	0.4605	1.1305	0.098*
C31[a]	0.8628 (5)	0.3855 (6)	1.3047 (9)	0.117 (3)
H31A[a]	0.8304	0.3778	1.3033	0.141*
H31B[a]	0.8844	0.3448	1.3245	0.141*
C31A[b]	0.8762 (6)	0.4045 (7)	1.2881 (11)	0.117 (3)
H31C[b]	0.8499	0.4033	1.3051	0.141*
H31D[b]	0.8917	0.3606	1.2994	0.141*
C32[a]	0.9053 (5)	0.4391 (6)	1.3983 (9)	0.137 (3)
H32A[a]	0.9348	0.4228	1.4856	0.165*
H32B[a]	0.8833	0.4756	1.3937	0.165*
C32A[b]	0.9270 (7)	0.4499 (7)	1.3773 (11)	0.137 (3)
H32C[b]	0.9624	0.4269	1.4521	0.165*
H32D[b]	0.9156	0.4841	1.4049	0.165*
C33[a]	0.9368 (6)	0.4584 (7)	1.3463 (13)	0.164 (4)
H33A[a]	0.9415	0.5056	1.3496	0.197*
H33B[a]	0.9769	0.4387	1.4001	0.197*
C33A[b]	0.9396 (6)	0.4781 (8)	1.2990 (11)	0.164 (4)
H33C[b]	0.9761	0.4586	1.3247	0.197*
H33D[b]	0.9454	0.5251	1.3112	0.197*
C34[a]	0.8989 (5)	0.4353 (5)	1.2124 (10)	0.151 (3)
H34A[a]	0.9174	0.3983	1.2072	0.181*
H34B[a]	0.8914	0.4700	1.1566	0.181*
C34A[b]	0.8837 (6)	0.4619 (6)	1.1574 (11)	0.151 (3)
H34C[b]	0.8661	0.5020	1.1077	0.181*
H34D[b]	0.8960	0.4349	1.1221	0.181*
N1	0.66164 (7)	0.32829 (8)	0.74974 (14)	0.0517 (4)
N2	0.53554 (7)	0.24387 (8)	0.67494 (15)	0.0541 (4)
N3	0.47602 (7)	0.25854 (8)	0.58801 (15)	0.0523 (4)
N4	0.80523 (8)	0.37174 (10)	1.06539 (18)	0.0792 (6)
H4A[a]	0.8223	0.3352	1.0755	0.095*
H4B[b]	0.8189	0.3324	1.0899	0.095*
O1	0.65894 (6)	0.26604 (7)	0.87677 (13)	0.0611 (4)
O2	0.72369 (8)	0.43489 (9)	0.92033 (16)	0.0882 (6)
O3	0.56203 (9)	0.44125 (9)	0.28597 (16)	0.0833 (5)
O4[c]	0.90281 (15)	0.3293 (3)	0.6194 (3)	0.1492 (17)
O4A[d]	0.9149 (6)	0.3757 (9)	0.6784 (14)	0.1492 (17)
O5[c]	0.91995 (14)	0.3196 (3)	0.7947 (3)	0.1128 (13)
O5A[d]	0.9240 (6)	0.2900 (8)	0.7736 (13)	0.1128 (13)

[a]Occupancy: 0.537 (6), [b]Occupancy: 0.463 (6), [c]Occupancy: 0.793 (5), [d]Occupancy: 0.207 (5).

1 mmol, from Aladdin) and methanol (5 mL) into a 10 mL dried microwave bottle, stirred at room temperature for 10 min. Then methyl 2-(4-formylphenyl) acetate (178 mg

1 mmol, purchased from Macklin), molecular sieve 4A (0.2 g, from Aldrich), 1-methyl-5-phenyl-1*H*- pyrazole-3-carboxylic acid (1 mmol, purchased from Bidepharm) and cyclopentyl isocyanide (165 uL, 1.1 mmol) were added into the microwave bottle. The mixture was reacted in the microwave reactor at 80 °C for 1 h. After that, the reaction mixture was cooled down to room temperature, and the solvent was evaporated under reduced pressure to afford the yellow-brown crude product. The yellow-brown product was purified by column chromatography on a silica gel (200–300 μm) with petroleum ether:ethyl acetate (1:1) and ethyl acetate as eluent separately. Crystals of the target product were obtained by recrystallization from an acetonitrile solution at room temperature.

Experimental details

H atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms.

Comment

Pyrazole is a nitrogen-containing five-membered heterocyclic ring and corresponding derivatives show a wide range of anti-inflammatory [5, 6], anti-bacterial [7, 8], anti-malarial [9] and other activities. It is widely used in medicine [10] and fluorescent probes [11]. In recent years, multi-component reactions have gradually become a main method to create complex molecules [12, 13]. The title compound is a pyrazole heterocyclic compound synthesized by the Ugi reaction through ketone, amine, carboxylic acid and isonitrile [14].

The title crystal structure is shown in the figure, in which all values of the geometric parameters are normal [15]. There are two symmetry related N–H···O hydrogen bonds that connect neighboring molecules into dimers. In the crystal structure, the three phenyl rings and the pyrazolyl ring are almost planar. The cyclopentyl ring has a chair conformation. Possible π–π interactions are not observed. The torsion angle of C3–C4–C6–C9 is –126.2(2)° which shows the phenyl moiety bonded to C4 is not coplanar to the pyrazolyl moiety.

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

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