Crystal structure of ethyl 4-(5-allyl-6-methyl-2-phenylpyrimidin-4-yl-amino)benzoate, $C_{23}H_{23}N_3O_2$

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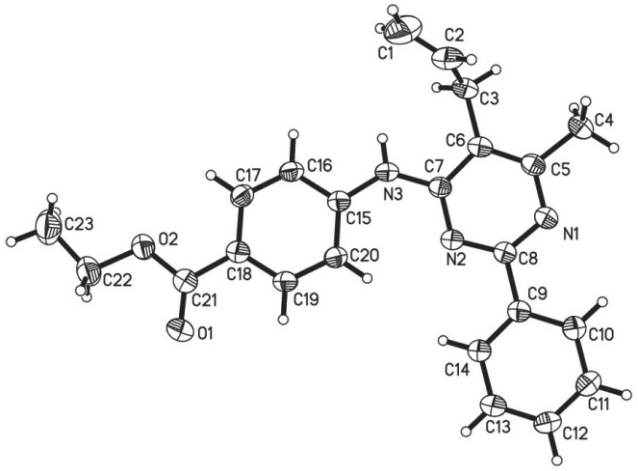

Abstract

$C_{23}H_{23}N_3O_2$, triclinic, $P\bar{1}$ (no. 2), a = 8.244(5) Å, b = 9.998(6) Å, c = 12.618(8) Å, α = 76.654(7)°, β = 84.834(7)°, γ = 81.708(7)°, V = 999.6 Å^3, Z = 2, $R_{gt}(F)$ = 0.0469, $wR_{ref}(F^2)$ = 0.1473, T = 296 K.

Table 1. Data collection and handling.

Crystal:	colourless prism, size 0.144×0.168×0.284 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.81 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ and ω
$2\theta_{max}$:	50°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	7084, 3453
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2843
$N(param)_{refined}$:	260
Programs:	SHELX, DIAMOND [5–6]

Source of material

Ethyl 4-(5-allyl-6-methyl-2-phenylpyrimidin-4-ylamino)benzoate was prepared according to the previousely discribed method [1, 2]. The colourless prism-shaped crystals suitable for X-ray diffraction were obtained by slow evaporation of a methanol solution.

Experimental details

The H atoms were calculated geometrically and refined as riding, with C–H = 0.93-0.97 Å, except for H3, and with U_{iso}(H) = 1.2U_{eq}(parent atom). Unfortunately, a data completeness of 98.3 % was achieved only.

Discussion

Pyrimidine derivatives have very interesting biological properties and many applications in the areas of pharmaceuticals, such as 4-(5-allyl-6-methyl-2-phenylpyrimidin-4-ylamino)benzoic acid which was identified owning potent inhibitory activity against phosphodiesterase 4 [2]. The title compound is one of its synthetical predecessor. In the crystal structure of the title compound, one phenyl ring (C9-C14)) directly linked at C8 is oriented at a dihedral angle of 3.92(9)° with respect to the pyrimidine ring, both are almost coplanar. Another phenyl ring (C15-C20) linked to C7 of pyrimidine ring is through bridge nitrogen atom (N3) with a dihedral angle of 9.22(9)°. The dihedral angle between the two phenyl rings is 12.06(10)°. The C–N distances of the pyrimidine ring are in the range 1.330(2)-1.347(2) Å with no significant bond fixation. In addition, pyrimidine ring distances are comparable with the corresponding bonds in 4,6-disubstituted 2-amino pyrimidines [3] and 4,5,6-tri-substituted 2-amino pyrimidines [4]. The bond lengths of C7–N3 and N3–C15 are 1.368(2) and 1.358(2)Å, respectively, indicating delocalization of nitrogen lone pair electron on to two adjacent aromatic rings. The torsion angles of allyl C1–C2–C3–C6 and ethyl ester C21–O2–C22–C23 moieties are –129.3(3)° and –179.6(2)°, indicating all atoms of the title compound, except for C1 and C2, almost in a plane.

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(3)	2*i*	0.216(2)	0.363(2)	0.422(2)	0.070(6)
H(1A)	2*i*	0.1972	0.1247	0.3629	0.182
H(1B)	2*i*	0.3537	0.0221	0.4139	0.182
H(2A)	2*i*	0.2744	0.0571	0.5747	0.107
H(3B)	2*i*	–0.0193	0.1369	0.5824	0.082
H(3C)	2*i*	0.0148	0.2441	0.4734	0.082
H(4A)	2*i*	–0.0665	0.1777	0.8527	0.078
H(4B)	2*i*	0.0355	0.0796	0.7834	0.078
H(4C)	2*i*	–0.1355	0.1584	0.7461	0.078
H(10A)	2*i*	0.1083	0.4766	0.9270	0.078
H(11A)	2*i*	0.1641	0.6297	1.0256	0.094
H(12A)	2*i*	0.2643	0.8327	0.9378	0.093
H(13A)	2*i*	0.3090	0.8808	0.7517	0.093
H(14A)	2*i*	0.2526	0.7296	0.6519	0.078
H(16A)	2*i*	0.3178	0.4371	0.2541	0.070
H(17A)	2*i*	0.4628	0.5869	0.1306	0.070
H(19A)	2*i*	0.4919	0.8018	0.3567	0.066
H(20A)	2*i*	0.3488	0.6506	0.4826	0.066
H(22A)	2*i*	0.8264	0.8606	0.0076	0.103
H(22B)	2*i*	0.6700	0.9624	–0.0347	0.103
H(23A)	2*i*	0.8125	0.8662	–0.1731	0.129
H(23B)	2*i*	0.6320	0.8296	–0.1530	0.129
H(23C)	2*i*	0.7780	0.7190	–0.1067	0.129

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Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
N(1)	2*i*	0.0707(2)	0.3767(1)	0.7713(1)	0.0522(7)	0.0497(7)	0.0605(8)	–0.0108(6)	–0.0029(6)	–0.0111(6)
N(2)	2*i*	0.1944(2)	0.5077(1)	0.6113(1)	0.0507(7)	0.0446(7)	0.0552(8)	–0.0075(5)	–0.0044(6)	–0.0145(6)
N(3)	2*i*	0.2287(2)	0.4288(2)	0.4501(1)	0.0674(8)	0.0496(8)	0.0589(9)	–0.0178(6)	–0.0012(6)	–0.0214(7)
O(1)	2*i*	0.6316(2)	0.9109(1)	0.1765(1)	0.0893(9)	0.0589(8)	0.0842(9)	–0.0260(7)	0.0031(7)	–0.0191(7)
O(2)	2*i*	0.6164(2)	0.7811(1)	0.0568(1)	0.0772(8)	0.0741(8)	0.0581(7)	–0.0238(6)	0.0019(6)	–0.0119(6)
C(1)	2*i*	0.2580(5)	0.0812(4)	0.4228(3)	0.197(4)	0.113(2)	0.160(3)	–0.026(2)	0.050(3)	–0.080(2)
C(2)	2*i*	0.2103(3)	0.1025(2)	0.5169(2)	0.119(2)	0.057(1)	0.100(2)	–0.011(1)	–0.003(1)	–0.033(1)
C(3)	2*i*	0.0611(2)	0.1937(2)	0.5415(2)	0.079(1)	0.057(1)	0.076(1)	–0.0254(9)	–0.0022(9)	–0.0206(9)
C(4)	2*i*	–0.0378(2)	0.1640(2)	0.7802(2)	0.064(1)	0.057(1)	0.074(1)	–0.0193(8)	–0.0016(8)	–0.0101(8)
C(5)	2*i*	0.0447(2)	0.2832(2)	0.7150(1)	0.0454(8)	0.0462(8)	0.066(1)	–0.0067(6)	–0.0059(7)	–0.0105(7)
C(6)	2*i*	0.0922(2)	0.2963(2)	0.6059(1)	0.0483(8)	0.0461(8)	0.067(1)	–0.0066(6)	–0.0068(7)	–0.0169(7)
C(7)	2*i*	0.1722(2)	0.4131(2)	0.5570(1)	0.0458(7)	0.0448(8)	0.0555(9)	–0.0046(6)	–0.0052(6)	–0.0125(7)
C(8)	2*i*	0.1434(2)	0.4845(2)	0.7163(1)	0.0444(7)	0.0457(8)	0.0546(9)	–0.0046(6)	–0.0037(6)	–0.0115(7)
C(9)	2*i*	0.1742(2)	0.5862(2)	0.7782(1)	0.0489(8)	0.0498(9)	0.0563(9)	–0.0066(6)	–0.0035(7)	–0.0144(7)
C(10)	2*i*	0.1486(2)	0.5583(2)	0.8908(2)	0.076(1)	0.065(1)	0.059(1)	–0.0204(8)	0.0052(8)	–0.0169(8)
C(11)	2*i*	0.1821(3)	0.6499(2)	0.9501(2)	0.098(1)	0.088(1)	0.059(1)	–0.028(1)	0.002(1)	–0.027(1)
C(12)	2*i*	0.2418(3)	0.7707(2)	0.8980(2)	0.096(1)	0.077(1)	0.072(1)	–0.030(1)	–0.003(1)	–0.033(1)
C(13)	2*i*	0.2680(3)	0.7992(2)	0.7872(2)	0.105(2)	0.063(1)	0.073(1)	–0.034(1)	–0.003(1)	–0.0196(9)
C(14)	2*i*	0.2344(2)	0.7084(2)	0.7274(2)	0.085(1)	0.058(1)	0.056(1)	–0.0218(9)	–0.0028(8)	–0.0143(8)
C(15)	2*i*	0.3168(2)	0.5276(2)	0.3814(1)	0.0467(8)	0.0482(8)	0.0555(9)	–0.0056(6)	–0.0048(6)	–0.0159(7)
C(16)	2*i*	0.3530(2)	0.5109(2)	0.2751(1)	0.0639(9)	0.058(1)	0.062(1)	–0.0162(8)	–0.0024(8)	–0.0254(8)
C(17)	2*i*	0.4392(2)	0.6005(2)	0.2011(1)	0.0604(9)	0.065(1)	0.0538(9)	–0.0107(8)	–0.0019(7)	–0.0207(8)
C(18)	2*i*	0.4919(2)	0.7120(2)	0.2305(1)	0.0446(7)	0.0501(9)	0.0595(9)	–0.0041(6)	–0.0063(7)	–0.0136(7)
C(19)	2*i*	0.4565(2)	0.7279(2)	0.3360(1)	0.0555(8)	0.0502(9)	0.064(1)	–0.0100(7)	–0.0051(7)	–0.0212(7)
C(20)	2*i*	0.3706(2)	0.6378(2)	0.4118(1)	0.0585(9)	0.0548(9)	0.0559(9)	–0.0119(7)	–0.0008(7)	–0.0199(7)
C(21)	2*i*	0.5858(2)	0.8126(2)	0.1542(1)	0.0528(9)	0.055(1)	0.062(1)	–0.0044(7)	–0.0065(7)	–0.0109(8)
C(22)	2*i*	0.7195(3)	0.8664(3)	–0.0209(2)	0.094(2)	0.096(2)	0.068(1)	–0.036(1)	0.007(1)	–0.008(1)
C(23)	2*i*	0.7370(3)	0.8159(3)	–0.1222(2)	0.108(2)	0.141(2)	0.073(2)	–0.037(2)	0.010(1)	–0.018(2)

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