Crystal structure of 1-[2-[6-(4-methoxyphenyl)-3(2*H*)-pyridazinone-2-yl]-acetyl]-4-(3-chlorophenyl)piperazine, C₂₃H₂₃ClN₄O₃

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Abstract

C₂₃H₂₃ClN₄O₃, triclinic, *P* $\bar{1}$ (No. 2), *a* = 10.044(1) Å, *b* = 10.955(2) Å, *c* = 11.450(2) Å, α = 69.161(9)°, β = 70.761(5)°, γ = 72.90(1)°, *V* = 1089.1 Å³, *Z* = 2, *R*_{gt}(*F*) = 0.053, *wR*_{ref}(*F*²) = 0.157, *T* = 293 K.

Source of material

[6-(4-methoxyphenyl)-3(2*H*)-pyridazinone-2-yl]acetic acid (0.01 mol) in 40 ml dichloromethane at 273 K (ice-bath) was treated with triethylamine (3 ml) and 0.01 mol of ethyl chloroformate. After stirring the reaction mixture at 273 K for 15 min, 0.011 mol of 1-(3-chlorophenyl)piperazine was added to this solution. The final mixture was stirred at 273 K – 298 K for 24 h, evaporated to dryness, then treated with acetone. All solid materials thus obtained were washed with 1% solution of NaOH, then with water, dried and crystallized from methanol.

Discussion

The torsion angles C19–C18–N4–C15 [42.7 (6)°] and C23–C18–N4–C15 [–138.3 (4)°] define the position of the piperazine ring in the molecule. The piperazine ring may be described as a chair conformation with the N3 and N4 atoms deviating by –0.646(6) Å and 0.601(6) Å, respectively, from the plane of the C14, C15, C16 and C17 atoms. The bond lengths and angles are comparable to those observed in related piperazine derivatives [1–3]. The pyridazine and two phenyl rings (C2–C7 and C18–C23) are planar, the r.m.s. deviations from the mean planes

are 0.009 Å, 0.006 Å and 0.008 Å. The dihedral angle between the pyridazine ring and piperazine ring is 82.5(1)°. The two phenyl rings (C2–C7 and C18–C23) make dihedral angles of 88.2(1)° to each other and angles of 72.9(1)° and 21.9(2)° to the piperazine ring, respectively. In the two phenyl rings the bond and angles are in good agreement with the expected value for aromatic rings [2]. The methoxy group is almost coplanar with the aromatic rings, as shown by torsion angles 174.7(4)° for C7–C2–O1–C1. The CO [1.407(6) Å] and C=O [1.233(4) Å] distances are comparable with reported values [4, 5].

Table 1. Data collection and handling.

Crystal:	colourless, prismatic, size 0.12 × 0.18 × 0.40 mm
Wavelength:	Cu <i>K</i> _α radiation (1.54184 Å)
μ :	18.23 cm ^{–1}
Diffractometer, scan mode:	Enraf-Nonius CAD-4, $\omega/2\theta$
2 θ _{max} :	148.4°
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} :	4441, 4441
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ (<i>I</i> _{obs}), 3274
<i>N</i> (<i>param</i>) _{refined} :	338
Programs:	SHELXS-97 [7], SHELXL-97 [8], ORTEPII [9]

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Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U _{iso}
H(1A)	2i	0.5148	1.6277	1.245	0.142
H(1B)	2i	0.6396	1.5475	1.313	0.142
H(1C)	2i	0.4839	1.5182	1.3777	0.142
H(3)	2i	0.300(4)	1.541(4)	1.282(4)	0.06(1)
H(4)	2i	0.134(5)	1.508(5)	1.210(4)	0.08(1)
H(6)	2i	0.4457	1.2508	1.0378	0.067
H(7)	2i	0.6144	1.2983	1.0981	0.071
H(9)	2i	−0.024(4)	1.439(4)	1.179(4)	0.049(9)
H(10)	2i	−0.184(4)	1.360(4)	1.130(4)	0.06(1)
H(12A)	2i	0.277(4)	1.079(4)	0.919(4)	0.05(1)
H(12B)	2i	0.127(4)	1.073(4)	0.917(4)	0.05(1)
H(14A)	2i	0.255(5)	1.296(5)	0.521(4)	0.07(1)

Table 2. Continued.

Atom	Site	x	y	z	U _{iso}
	2i	0.392(5)	1.203(4)	0.494(4)	0.06(1)
H(15A)	2i	0.143(6)	1.154(5)	0.505(5)	0.08(2)
H(15B)	2i	0.256(5)	1.181(5)	0.389(5)	0.07(1)
H(16A)	2i	0.321(4)	0.840(4)	0.657(4)	0.052(9)
H(16B)	2i	0.167(5)	0.939(4)	0.684(4)	0.06(1)
H(17A)	2i	0.3165	0.9589	0.7912	0.079
H(17B)	2i	0.4376	0.9876	0.6624	0.079
H(19)	2i	0.262(7)	1.076(6)	0.267(6)	0.11(2)
H(20)	2i	0.215	0.964	0.1537	0.112
H(21)	2i	0.1367	0.7643	0.2591	0.092
H(23)	2i	0.200(5)	0.771(4)	0.583(5)	0.07(1)

Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
Cl(1)	2i	0.1139(1)	0.5969(1)	0.5139(1)	0.1119(9)	0.0694(7)	0.0755(7)	−0.0340(6)	−0.0032(6)	−0.0284(5)
C(1)	2i	0.5535(6)	1.5425(6)	1.2971(6)	0.081(3)	0.121(4)	0.117(4)	−0.019(3)	−0.036(3)	−0.066(4)
C(2)	2i	0.4788(4)	1.4268(4)	1.1962(3)	0.055(2)	0.061(2)	0.055(2)	−0.014(2)	−0.014(2)	−0.018(2)
C(3)	2i	0.3359(4)	1.4877(4)	1.2292(4)	0.064(2)	0.067(2)	0.063(2)	−0.010(2)	−0.012(2)	−0.035(2)
C(4)	2i	0.2333(4)	1.4596(4)	1.1909(4)	0.054(2)	0.063(2)	0.062(2)	−0.009(2)	−0.011(2)	−0.030(2)
C(5)	2i	0.2712(4)	1.3697(3)	1.1204(3)	0.055(2)	0.053(2)	0.044(2)	−0.014(1)	−0.012(1)	−0.010(2)
C(6)	2i	0.4172(4)	1.3106(4)	1.0864(3)	0.057(2)	0.055(2)	0.055(2)	−0.010(2)	−0.010(2)	−0.020(2)
C(7)	2i	0.5182(4)	1.3385(4)	1.1226(4)	0.052(2)	0.061(2)	0.064(2)	−0.008(2)	−0.013(2)	−0.021(2)
C(8)	2i	0.1622(4)	1.3317(3)	1.0863(3)	0.055(2)	0.050(2)	0.042(2)	−0.012(1)	−0.009(1)	−0.012(1)
C(9)	2i	0.0099(4)	1.3736(4)	1.1354(3)	0.055(2)	0.060(2)	0.053(2)	−0.014(2)	−0.008(2)	−0.020(2)
C(10)	2i	−0.0829(4)	1.3286(4)	1.1071(4)	0.049(2)	0.071(2)	0.066(2)	−0.016(2)	−0.004(2)	−0.023(2)
C(11)	2i	−0.0332(4)	1.2382(4)	1.0295(3)	0.062(2)	0.058(2)	0.052(2)	−0.022(2)	−0.016(2)	−0.008(2)
C(12)	2i	0.1809(5)	1.1240(4)	0.9005(4)	0.065(2)	0.053(2)	0.056(2)	−0.016(2)	−0.017(2)	−0.018(2)
C(13)	2i	0.1940(4)	1.2066(3)	0.7605(3)	0.066(2)	0.051(2)	0.053(2)	−0.018(2)	−0.015(2)	−0.015(1)
C(14)	2i	0.2932(7)	1.2067(5)	0.5341(4)	0.118(4)	0.054(2)	0.050(2)	−0.025(2)	0.000(2)	−0.012(2)
C(15)	2i	0.2340(7)	1.1419(4)	0.4722(4)	0.102(4)	0.050(2)	0.049(2)	−0.011(2)	−0.013(2)	−0.005(2)
C(16)	2i	0.2706(5)	0.9328(4)	0.6411(4)	0.075(2)	0.049(2)	0.051(2)	−0.008(2)	−0.017(2)	−0.009(2)
C(17)	2i	0.3343(5)	0.9997(4)	0.6986(4)	0.078(2)	0.060(2)	0.058(2)	−0.003(2)	−0.020(2)	−0.019(2)
C(18)	2i	0.2484(4)	0.9315(4)	0.4363(3)	0.068(2)	0.053(2)	0.049(2)	−0.008(2)	−0.012(2)	−0.011(2)
C(19)	2i	0.2522(6)	0.9874(5)	0.3049(4)	0.127(4)	0.060(2)	0.056(2)	−0.025(2)	−0.031(2)	−0.002(2)
C(20)	2i	0.2117(7)	0.9246(5)	0.2409(5)	0.151(5)	0.073(3)	0.058(2)	−0.019(3)	−0.046(3)	−0.006(2)
C(21)	2i	0.1663(6)	0.8050(4)	0.3023(5)	0.100(3)	0.062(2)	0.072(3)	−0.007(2)	−0.036(2)	−0.019(2)
C(22)	2i	0.1668(4)	0.7495(4)	0.4292(4)	0.071(2)	0.054(2)	0.063(2)	−0.010(2)	−0.014(2)	−0.017(2)
C(23)	2i	0.2059(4)	0.8096(4)	0.4970(4)	0.076(2)	0.056(2)	0.045(2)	−0.011(2)	−0.011(2)	−0.011(2)
N(1)	2i	0.2101(3)	1.2530(3)	1.0124(3)	0.056(2)	0.056(2)	0.049(2)	−0.013(1)	−0.013(1)	−0.013(1)
N(2)	2i	0.1142(3)	1.2088(3)	0.9858(3)	0.062(2)	0.055(2)	0.048(2)	−0.017(1)	−0.013(1)	−0.016(1)
N(3)	2i	0.2708(4)	1.1415(3)	0.6718(3)	0.093(2)	0.053(2)	0.052(2)	−0.016(2)	−0.009(2)	−0.015(1)
N(4)	2i	0.2883(3)	0.9964(3)	0.5018(3)	0.069(2)	0.052(2)	0.048(2)	−0.014(1)	−0.012(1)	−0.009(1)
O(1)	2i	0.5861(3)	1.4460(3)	1.2319(3)	0.062(2)	0.096(2)	0.092(2)	−0.011(1)	−0.024(1)	−0.048(2)
O(2)	2i	−0.1115(3)	1.1886(3)	1.0036(3)	0.071(2)	0.081(2)	0.087(2)	−0.027(1)	−0.021(1)	−0.030(2)
O(3)	2i	0.1379(3)	1.3245(3)	0.7322(3)	0.097(2)	0.052(2)	0.060(2)	−0.012(1)	−0.018(1)	−0.016(1)

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