Crystal structure of 1-[2-[6-(4-methoxyphenyl)-3(2*H*)-pyridazinone-2-yl]-acetyl]-4-(3-chlorophenyl)piperazine, $C_{23}H_{23}ClN_4O_3$

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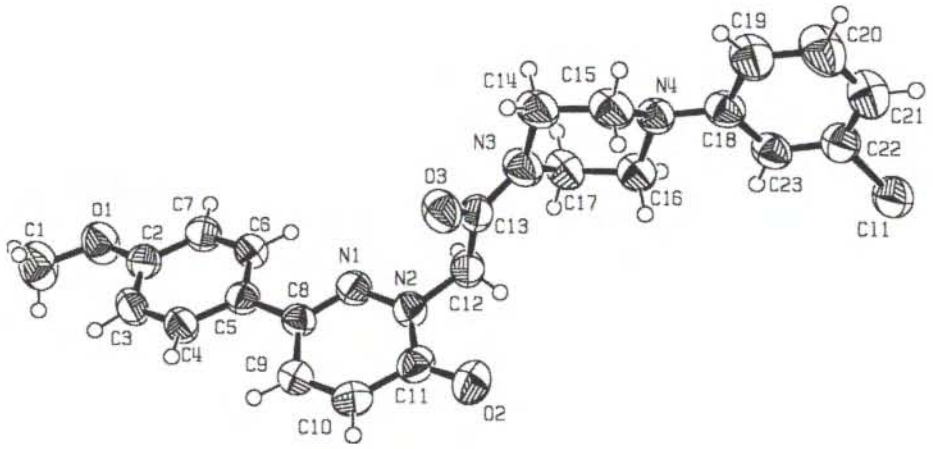

Abstract

$C_{23}H_{23}ClN_4O_3$, 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 Å^3, 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 K_α radiation (1.54184 Å)
μ:	18.23 cm^{-1}
Diffractometer, scan mode:	Enraf-Nonius CAD-4, $\omega/2\theta$
$2\theta_{max}$:	148.4°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	4441, 4441
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3274
$N(param)_{refined}$:	338
Programs:	SHELXS-97 [7], SHELXL-97 [8], ORTEPII [9]

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

Atom	Site	x	y	z	U_{iso}
H(1A)	2*i*	0.5148	1.6277	1.245	0.142
H(1B)	2*i*	0.6396	1.5475	1.313	0.142
H(1C)	2*i*	0.4839	1.5182	1.3777	0.142
H(3)	2*i*	0.300(4)	1.541(4)	1.282(4)	0.06(1)
H(4)	2*i*	0.134(5)	1.508(5)	1.210(4)	0.08(1)
H(6)	2*i*	0.4457	1.2508	1.0378	0.067
H(7)	2*i*	0.6144	1.2983	1.0981	0.071
H(9)	2*i*	–0.024(4)	1.439(4)	1.179(4)	0.049(9)
H(10)	2*i*	–0.184(4)	1.360(4)	1.130(4)	0.06(1)
H(12A)	2*i*	0.277(4)	1.079(4)	0.919(4)	0.05(1)
H(12B)	2*i*	0.127(4)	1.073(4)	0.917(4)	0.05(1)
H(14A)	2*i*	0.255(5)	1.296(5)	0.521(4)	0.07(1)
	2*i*	0.392(5)	1.203(4)	0.494(4)	0.06(1)
H(15A)	2*i*	0.143(6)	1.154(5)	0.505(5)	0.08(2)
H(15B)	2*i*	0.256(5)	1.181(5)	0.389(5)	0.07(1)
H(16A)	2*i*	0.321(4)	0.840(4)	0.657(4)	0.052(9)
H(16B)	2*i*	0.167(5)	0.939(4)	0.684(4)	0.06(1)
H(17A)	2*i*	0.3165	0.9589	0.7912	0.079
H(17B)	2*i*	0.4376	0.9876	0.6624	0.079
H(19)	2*i*	0.262(7)	1.076(6)	0.267(6)	0.11(2)
H(20)	2*i*	0.215	0.964	0.1537	0.112
H(21)	2*i*	0.1367	0.7643	0.2591	0.092
H(23)	2*i*	0.200(5)	0.771(4)	0.583(5)	0.07(1)

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}
Cl(1)	2*i*	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)	2*i*	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)	2*i*	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)	2*i*	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)	2*i*	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)	2*i*	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)	2*i*	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)	2*i*	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)	2*i*	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)	2*i*	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)	2*i*	–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)	2*i*	–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)	2*i*	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)	2*i*	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)	2*i*	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)	2*i*	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)	2*i*	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)	2*i*	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)	2*i*	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)	2*i*	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)	2*i*	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)	2*i*	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)	2*i*	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)	2*i*	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)	2*i*	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)	2*i*	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)	2*i*	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)	2*i*	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)	2*i*	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)	2*i*	–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)	2*i*	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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References

1. Gunesakaran, K.; Govindasamy, L.; Raj, S. S. S.; Velmurugan, D.; Karunakaran, S.; Kandaswamy, M.: 2,2′-Piperazin-1,4-diyl-dimethylene-bis[6-(benzyliminomethyl)-4-methylphenol]. Acta Crystallogr. **C52** (1996) 1027-1028.
2. Mairesse, P. G.; Boivin, J. C.; Thomas, D. J.; Bonte, J. ; Lesieur, D.: Structure de l'(Ethynyl-1 cyclohexyloxy)-1[(Méthoxy-2 phényl)-4 pipérazinyl-1]-3 propanol-2-(*RS*). Acta Crystallogr. **C43** (1987) 2128-2130.
3. Blaton, N. M.; Peters, O. M.; Ranter, C. J.: 2-Methylthio-10,11-dihydro-11-(4-methyl-piperazin-1-yl) dibenzo[*b,f*]thiepine Maleic Acid (Metitepine Maleate). Acta Crystallogr. **C51** (1995) 777-780.
4. Moreau, S.; Metin, J.; Coudert, P.; Couquelet, J.: 5(2-Chlorobenzyl)-6-methyl-3(2*H*)-pyridazinone. Acta Crystallogr. **C51** (1995) 1834-1836.
5. Fronczek, F. R.; Oliver, M. A.; Gandour, R. D.: (*E*)-and(*Z*)-2,3-Bis(2-methoxyphenyl)-2-butene, $C_{18}H_{20}O_2$. Acta Crystallogr. **C40** (1984) 504-506.
6. Fair, C. K. : MolEN. An Interactive Intelligent System for Crystal Structure Analysis. Enraf-Nonius, Delft, The Netherlands 1990.
7. Sheldrick, G. M.: SHELXS-97 Program for the Solution of Crystal Structures. University of Göttingen, Germany 1997.
8. Sheldrick, G. M.: SHELXL-97 Program for the Refinement of Crystal Structures. University of Göttingen, Germany 1997.
9. Johnson, C. K.: ORTEP-II. Report ORLN-5138. Oak Rigge National Laboratory, Tennessee, USA 1976.