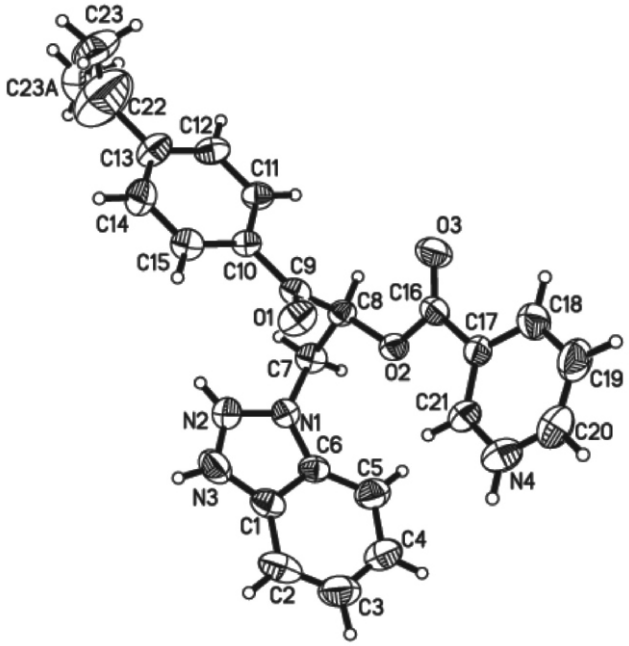
Crystal structure of 1-(4-ethylphenyl)-2-nicotinoyl-3-(1*H*-benzo[*d*]-1,2,3-triazol-1-yl)-1-oxopropan, $C_{23}H_{20}N_4O_3$

Wu-Lan Zeng*

Microscale Science Institute, Department of Chemistry and Chemical Engineering, Weifang University, Weifang 261061, Shandong, P. R. China

Received March 13, 2011, accepted and available on-line July 12, 2011; CCDC no. 1267/3447



Abstract

$C_{23}H_{20}N_4O_3$, orthorhombic, $Pca2_1$ (no. 29), a = 19.430(4) Å, b = 5.362(1) Å, c = 19.692(4) Å, V = 2051.6 Å^3, Z = 4, $R_{gt}(F)$ = 0.051, $wR_{ref}(F^2)$ = 0.133, T = 293 K.

Source of material

All the reagents and solvents from commercial sources were used without further purification. Bromine (3.2 g, 0.02 mol) was added dropwise to a solution of 1-(4-ethylphenyl)-3-(1*H*-benzo[*d*]-1,2,3-triazol-1-yl)-propan-1-one (5.58 g, 0.02 mol) and sodium acetate (1.6 g, 0.02 mol) in acetic acid (50 ml). The reaction proceeded for 7 h. After the reaction was completed, 50 ml of water and 20 ml of chloroform were added. The organic layer was washed successively with saturated sodium bicarbonate solution and brine. The solution was purified by flash column chromatography (silica gel, petroleum ether-ethylacetate (3:1 v/v) as eluent). It was further cooled with ice water, and then an acetone solution of nicotinic acid (10 ml, 2.46 g, 0.02 mol) and triethylamine (2.8 ml) were added. The mixture was stirred with ice water for about 5 h. The solution was filtered and concentrated to afford the target compound (m.p. 409-409.6 K). Single crystals were obtained by slow evaporation of petroleum ether-ethylacetate solution (3:1 v/v) at room temperature over a period of one week.

IR data are available in the CIF file.

* e-mail: wulanzeng@163.com

Discussion

Over the last two decades, many N-heterocyclic compounds were found in natural products and widely used as biologically active materials. The 1*H*-benzotriazole and its derivatives were studied because they exhibit a broad spectrum of pharmacological activities such as antifungal, antitumor and antineoplastic [1,2].

The bonds distances and angles in the title crystal structure agree with those in the related compounds 2-(1*H*-benzotriazol-1-yl)-1-(4-ethylbenzoyl)ethyl-2-chlorobenzoate [3] and 2-(1*H*-benzotriazol-1-yl)-1-(2-fluoro-benzoyl)ethyl-4-methylbenzoate [4] . The benzotriazole ring system is essentially planar with a dihedral angle of 0.69(3)° between the triazole ring (atoms N1–N3/C1/C6) and the benzene ring (C1–C6). The dihedral angles between the mean planes of the benzotriazole system and the pyridine ring (N4/C17–C21) and ethylphenyl ring (C10–C15) are 59.78° and 8.61°, respectively. The dihedral angle between pyridine ring (N4/C17–C21) and ethylphenyl ring (C10–C15) is 64.44°. C23/C23A describe the disordered position with the occupancies of 0.65 and 0.35, respectively.

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.13 × 0.22 × 0.41 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.88 cm^{-1}
Diffractometer, scan mode:	CCD area detector, φ/ω
$2\theta_{max}$:	56.48°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	12373, 4602
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2118
$N(param)_{refined}$:	277
Programs:	SHELXS-97, SHELXL-97, SHELXTL [5]

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

Atom	Site	Occ.	x	y	z	U_{iso}
H(23A)	4*a*	0.65	0.5792	0.0416	–0.7940	0.203
H(23B)	4*a*	0.65	0.5741	0.0480	–0.7146	0.203
H(23C)	4*a*	0.65	0.5427	0.2656	–0.7577	0.203
C(23A)	4*a*	0.35	0.506(1)	–0.160(3)	–0.787(1)	0.134(7)
H(23D)	4*a*	0.35	0.5412	–0.1098	–0.8179	0.201
H(23E)	4*a*	0.35	0.4658	–0.2124	–0.8122	0.201
H(23F)	4*a*	0.35	0.5218	–0.2964	–0.7597	0.201
H(2A)	4*a*		0.1982	0.2112	–0.6467	0.100
H(3A)	4*a*		0.1195	0.5225	–0.6560	0.112
H(2B)	4*a*		0.0163	0.7206	–0.5748	0.116
H(3B)	4*a*		–0.0343	0.6494	–0.4718	0.134
H(4B)	4*a*		0.0012	0.3299	–0.4025	0.137
H(5A)	4*a*		0.0928	0.0788	–0.4310	0.109
H(7A)	4*a*		0.2246	–0.1454	–0.5750	0.077
H(7B)	4*a*		0.1854	–0.1715	–0.5058	0.077
H(8A)	4*a*		0.3023	–0.0776	–0.4853	0.066

Table 2. Continued.

Atom	Site	Occ.	x	y	z	U_{iso}
H(11A)	4*a*		0.3877	–0.1557	–0.5566	0.076
H(12A)	4*a*		0.4646	–0.2485	–0.6416	0.091
H(14A)	4*a*		0.4100	0.3576	–0.7423	0.103
H(15A)	4*a*		0.3352	0.4645	–0.6557	0.081

Table 2. Continued.

Atom	Site	Occ.	x	y	z	U_{iso}
H(18A)	4*a*		0.3451	0.4296	–0.2818	0.113
H(19A)	4*a*		0.3053	0.7507	–0.2115	0.124
H(20A)	4*a*		0.2057	0.9436	–0.2395	0.118
H(21A)	4*a*		0.1786	0.5205	–0.3954	0.095

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

Atom	Site	Occ.	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(23)	4*a*	0.65	0.5510(4)	0.089(2)	–0.7561(5)	0.127(6)	0.149(9)	0.131(7)	0.006(5)	0.024(6)	–0.031(6)
O(1)	4*a*		0.2956(1)	0.4678(4)	–0.5320(1)	0.096(2)	0.042(1)	0.104(2)	0.005(1)	0.021(2)	–0.010(1)
O(2)	4*a*		0.2439(1)	0.1781(4)	–0.4390(1)	0.058(1)	0.067(1)	0.063(1)	0.007(1)	0.001(1)	–0.007(1)
O(3)	4*a*		0.3507(1)	0.1914(6)	–0.3979(2)	0.076(2)	0.127(3)	0.096(2)	0.036(2)	–0.021(2)	–0.033(2)
N(1)	4*a*		0.1597(1)	0.1341(5)	–0.5572(2)	0.066(2)	0.059(2)	0.061(2)	–0.003(2)	–0.002(2)	–0.001(2)
N(2)	4*a*		0.1680(2)	0.2491(7)	–0.6165(2)	0.079(2)	0.104(3)	0.067(2)	0.009(2)	–0.001(2)	–0.003(2)
N(3)	4*a*		0.1239(2)	0.4247(7)	–0.6217(2)	0.089(2)	0.102(3)	0.089(3)	0.010(2)	–0.018(2)	0.028(2)
N(4)	4*a*		0.1803(2)	0.7507(7)	–0.3189(2)	0.100(3)	0.101(3)	0.101(3)	0.021(2)	0.016(2)	–0.022(2)
C(1)	4*a*		0.0853(2)	0.4325(7)	–0.5650(2)	0.062(2)	0.072(3)	0.085(3)	0.002(2)	–0.016(2)	–0.006(2)
C(2)	4*a*		0.0311(2)	0.5921(8)	–0.5466(3)	0.078(3)	0.083(3)	0.128(5)	0.020(2)	–0.031(3)	–0.012(3)
C(3)	4*a*		0.0018(2)	0.548(1)	–0.4860(3)	0.081(3)	0.124(4)	0.131(5)	0.025(3)	–0.008(3)	–0.040(4)
C(4)	4*a*		0.0239(2)	0.355(1)	–0.4435(3)	0.080(3)	0.156(4)	0.107(4)	0.022(3)	0.013(3)	–0.015(4)
C(5)	4*a*		0.0774(2)	0.2036(8)	–0.4600(2)	0.068(2)	0.105(3)	0.100(4)	0.008(2)	0.011(2)	0.008(2)
C(6)	4*a*		0.1075(2)	0.2456(7)	–0.5224(2)	0.051(2)	0.061(2)	0.073(2)	–0.002(2)	–0.007(2)	–0.007(2)
C(7)	4*a*		0.2086(2)	–0.0541(5)	–0.5355(2)	0.070(2)	0.043(2)	0.080(2)	0.001(2)	–0.003(2)	–0.008(2)
C(8)	4*a*		0.2704(2)	0.0554(6)	–0.4984(2)	0.058(2)	0.045(2)	0.063(2)	0.009(2)	0.004(2)	–0.005(2)
C(9)	4*a*		0.3070(2)	0.2470(6)	–0.5420(2)	0.058(2)	0.041(2)	0.072(2)	0.004(2)	–0.010(2)	–0.006(2)
C(10)	4*a*		0.3533(1)	0.1663(6)	–0.5970(2)	0.051(2)	0.043(2)	0.064(2)	–0.002(2)	0.002(2)	–0.003(2)
C(11)	4*a*		0.3923(2)	–0.0488(6)	–0.5935(2)	0.057(2)	0.051(2)	0.081(2)	0.004(2)	0.000(2)	0.002(2)
C(12)	4*a*		0.4381(2)	–0.1046(7)	–0.6451(2)	0.062(2)	0.068(2)	0.097(3)	0.011(2)	0.009(2)	–0.009(2)
C(13)	4*a*		0.4458(2)	0.0431(9)	–0.7006(2)	0.091(3)	0.088(3)	0.094(3)	0.006(3)	0.023(2)	–0.006(3)
C(14)	4*a*		0.4063(2)	0.2547(8)	–0.7044(2)	0.102(3)	0.082(3)	0.073(3)	–0.006(3)	0.012(2)	0.008(2)
C(15)	4*a*		0.3608(2)	0.3182(6)	–0.6526(2)	0.071(2)	0.055(2)	0.077(3)	0.000(2)	–0.001(2)	–0.002(2)
C(16)	4*a*		0.2923(2)	0.2591(7)	–0.3950(2)	0.064(2)	0.075(2)	0.062(2)	0.014(2)	–0.008(2)	0.001(2)
C(17)	4*a*		0.2654(2)	0.4463(6)	–0.3473(2)	0.070(2)	0.067(2)	0.054(2)	0.009(2)	0.004(2)	–0.000(2)
C(18)	4*a*		0.3040(2)	0.5116(9)	–0.2914(2)	0.095(3)	0.114(4)	0.074(3)	0.020(3)	–0.012(2)	–0.018(2)
C(19)	4*a*		0.2807(3)	0.7013(9)	–0.2496(2)	0.118(4)	0.119(4)	0.073(3)	–0.002(3)	0.004(3)	–0.031(3)
C(20)	4*a*		0.2204(3)	0.8123(8)	–0.2668(3)	0.124(4)	0.080(3)	0.090(4)	–0.005(3)	0.031(3)	–0.014(3)
C(21)	4*a*		0.2048(2)	0.5682(7)	–0.3580(2)	0.080(3)	0.086(3)	0.071(3)	0.014(2)	0.006(2)	–0.015(2)
C(22)	4*a*		0.4939(4)	–0.027(2)	–0.7590(4)	0.144(6)	0.160(7)	0.188(7)	–0.004(5)	0.088(5)	–0.030(5)

Acknowledgment. This work was supported by Natural Science Foundation of Shandong Province (grant nos. Y2008B29 and ZR2010BM033).

References

1. Zhang, S. S.; Zhang, H. Q.; Li, D.; Sun, L. H.; Ma, C. P.; Wang, W.; Wan, J.; Qu, B.: A novel benzotriazole derivative inhibits proliferation of human hepatocarcinoma cells by increasing oxidative stress concomitant mitochondrial damage. Eur. J. Pharmacol. **584** (2008) 144-152.
2. Gu, H. B.; Long, L.; Li, P. P.; Wang, L.; Chen, W. Y.: Synthesis, crystal ctructure and antimicrobial activity of a new ternary copper(II) complex with *p*-chlorophenoxyacetic acid and 2-amino benzothiazole. Chin. J. Struct. Chem. **29** (2010) 676-681.
3. Zeng, W. L.; Jian, F.: 2-(1*H*-Benzotriazol-1-yl)-1-(4-ethylbenzoyl)ethyl 2-chlorobenzoate. Acta Crystallogr. **E65** (2009) o2165.
4. Zeng, W. L.; Jian, F.: 2-(1*H*-Benzotriazol-1-yl)-1-(2-fluorobenzoyl)ethyl 4-methylbenzoate. Acta Crystallogr. **E65** (2009) o2036.
5. Sheldrick, G. M.: A short history of SHELX. Acta Crystallogr. **A64** (2008) 112-122.