Crystal structure of 6-benzoyl-2-benzoxazolinone-3-propionitril, $C_{17}H_{12}N_2O_3$

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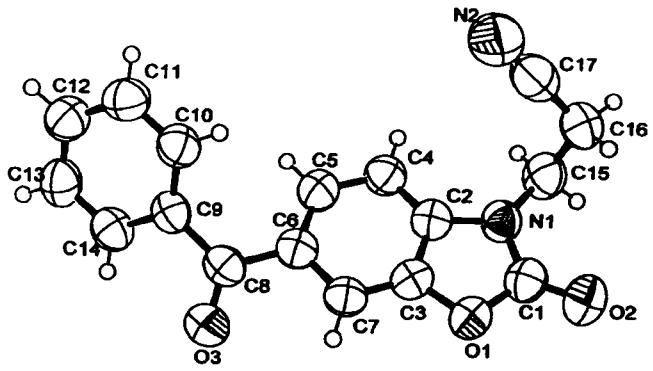
Received May 5, 2001, CCDC-No. 1267/655



Abstract

$C_{17}H_{12}N_2O_3$, monoclinic, $P12_1/n1$ (No. 14), $a = 10.661(1)$ Å, $b = 7.201(1)$ Å, $c = 18.651(1)$ Å, $\beta = 95.347(2)°$, $V = 1425.5$ Å^3, $Z = 4$, $R_{gt}(F) = 0.046$, $wR_{ref}(F^2) = 0.109$, $T = 293$ K.

Source of material

0.025 mole 6-benzoyl-2-benzoxazolinone and 0.03 mole triethylamine were stirred in 500 ml water. Then, 0.03 mole acrylonitrile was added to the reaction medium. The reaction mixture was heated at 323 K – 333 K for 6 hours and then at 298 K – 303 K for 18 hours. The solid was collected by filtration, washed with water until neutralized and air-dried at 298 K – 303 K. The title compound was recrystallized from ethanol (56% yield).

Discussion

The title compound, $C_{17}H_{12}N_2O_3$, is used as a starting material to synthesis analgesic compounds. This compound and its derivatives exhibite biological activity. It appeared expedient to continue our investigation and synthesize additional derivatives of nitriles. Thus, the object of this investigation was to prepare additional nitriles and to react these nitriles with alcoholic-hydrogen chloride solutions to afford the hydrogen chloride salts of imino-ester which upon neutralization with potassium carbonate would yield the title compound [1]. The bond lengths and angles are normal and the planar benzoxazolinone moiety forms a dihedral angle of 49(1)° with the benzene plane. This value is comparable with that in similar structure [2]. The bond lengths of C8—O3, C1—O2, C1—N1 and C2—N1 obtained in this study are 1.226(3) Å, 1.197(3) Å, 1.367(4) Å and 1.383(4) Å, respectively. In a similar structure given in the literature [2], those bond lengths have been reported to be 1.214(3) Å, 1.210(2) Å, 1.355(3) Å and 1.384(2) Å, respectively. In the present compound, the C1—O2 and C8—O3 bond lengths are found to be almost equal to those of a reported similar structure [3]. The obtained C1—O2 bond length is 1.197(3) Å and the C8—O3 bond length is 1.226(3) Å. The reported values are 1.199(10) Å and 1.219(10) Å, respectively. The torsion angles C15–C16–C17–N2, C4–C5–C6–C7 and N1–C15–C16–C17 are 37.0(2)°, –2.2(4)° and –66.1(4)°, respectively. The O3 atom lies 0.31(2) Å below the benzene plane.

Table 1. Data collection and handling.

Crystal:	cream, prismatic, size 0.15 × 0.20 × 0.25 mm
Wavelength:	Cu K_α radiation (1.54180 Å)
μ:	7.84 cm^{-1}
Diffractometer, scan mode:	Enraf-Nonius CAD4, ω/2θ
$2\theta_{max}$:	103.58°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	1578, 1578
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1490
$N(param)_{refined}$:	200
Programs:	SHELXS-97 [4], SHELXL-97 [5], MolEN [6], ORTEPII [7]

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

Atom	Site	x	y	z	U_{iso}
H(4)	4e	0.6400	0.3274	1.1585	0.087
H(5)	4e	0.4264	0.3929	1.1626	0.086
H(7)	4e	0.3540	0.2543	0.9533	0.078
H(10)	4e	0.2704	0.1750	1.1859	0.090
H(11)	4e	0.1692	0.1948	1.2893	0.100
H(12)	4e	0.0095	0.4083	1.2976	0.102
H(13)	4e	–0.0454	0.6056	1.2036	0.100
H(14)	4e	0.0548	0.5892	1.0997	0.090
H(15A)	4e	0.8543	0.2946	1.1116	0.100
H(15B)	4e	0.9184	0.2525	1.0411	0.100
H(16A)	4e	0.9036	–0.0687	1.0653	0.110
H(16B)	4e	0.9910	0.0360	1.1244	0.110

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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}
C(1)	4*e*	0.7219(3)	0.1444(4)	0.9617(2)	0.073(2)	0.066(2)	0.078(2)	–0.005(1)	0.016(2)	0.002(1)
C(2)	4*e*	0.6203(3)	0.2486(4)	1.0542(2)	0.067(2)	0.060(2)	0.070(2)	–0.003(1)	0.008(1)	0.000(1)
C(3)	4*e*	0.5340(3)	0.2285(3)	0.9947(1)	0.073(2)	0.054(2)	0.064(2)	–0.007(1)	0.009(2)	0.000(1)
C(4)	4*e*	0.5829(3)	0.3112(4)	1.1182(2)	0.068(2)	0.081(2)	0.067(2)	–0.004(1)	0.000(1)	–0.004(1)
C(5)	4*e*	0.4546(3)	0.3496(4)	1.1200(2)	0.074(2)	0.071(2)	0.071(2)	0.000(1)	0.010(2)	–0.001(1)
C(6)	4*e*	0.3677(3)	0.3257(4)	1.0604(2)	0.070(2)	0.058(2)	0.069(2)	–0.002(1)	0.005(1)	0.003(1)
C(7)	4*e*	0.4092(3)	0.2666(3)	0.9947(2)	0.071(2)	0.058(2)	0.065(2)	–0.002(1)	0.003(1)	0.004(1)
C(8)	4*e*	0.2313(3)	0.3657(4)	1.0618(2)	0.071(2)	0.068(2)	0.075(2)	0.008(1)	0.002(2)	0.006(1)
C(9)	4*e*	0.1735(2)	0.3809(4)	1.1316(2)	0.061(2)	0.063(2)	0.077(2)	0.004(1)	0.004(1)	0.002(1)
C(10)	4*e*	0.2068(3)	0.2622(4)	1.1892(2)	0.077(2)	0.067(2)	0.080(2)	0.006(1)	0.008(2)	0.005(2)
C(11)	4*e*	0.1461(3)	0.2737(5)	1.2508(2)	0.089(2)	0.084(2)	0.078(2)	–0.004(2)	0.007(2)	0.009(2)
C(12)	4*e*	0.0510(3)	0.4018(5)	1.2560(2)	0.082(2)	0.098(2)	0.077(2)	–0.001(2)	0.012(2)	–0.003(2)
C(13)	4*e*	0.0182(3)	0.5186(5)	1.1999(2)	0.074(2)	0.086(2)	0.092(2)	0.008(2)	0.015(2)	–0.012(2)
C(14)	4*e*	0.0783(3)	0.5091(4)	1.1377(2)	0.071(2)	0.068(2)	0.085(2)	0.000(1)	0.003(2)	0.002(1)
C(15)	4*e*	0.8582(3)	0.2060(5)	1.0727(2)	0.069(2)	0.093(2)	0.089(2)	–0.012(2)	0.012(2)	–0.001(2)
C(16)	4*e*	0.9045(3)	0.0223(5)	1.1037(2)	0.065(2)	0.118(3)	0.093(2)	0.011(2)	0.005(2)	0.003(2)
C(17)	4*e*	0.8282(4)	–0.0453(6)	1.1588(2)	0.087(3)	0.109(3)	0.108(3)	0.016(2)	0.001(2)	0.019(2)
O(1)	4*e*	0.5964(2)	0.1653(2)	0.9371(1)	0.077(2)	0.070(1)	0.068(1)	–0.001(1)	0.014(1)	–0.0021(9)
O(2)	4*e*	0.7999(2)	0.0914(3)	0.9243(1)	0.081(2)	0.096(2)	0.093(2)	–0.003(1)	0.026(1)	–0.008(1)
O(3)	4*e*	0.1646(2)	0.3867(4)	1.0052(1)	0.084(2)	0.120(2)	0.079(2)	0.025(1)	0.002(1)	0.008(1)
N(1)	4*e*	0.7356(2)	0.1944(3)	1.0327(1)	0.060(2)	0.077(2)	0.074(2)	–0.004(1)	0.008(1)	–0.003(1)
N(2)	4*e*	0.7662(4)	–0.0971(7)	1.2013(2)	0.126(3)	0.163(4)	0.142(3)	0.026(3)	0.033(3)	0.058(3)

Acknowledgment. The authors wish to acknowledge the purchase of CAD4 diffractometer under grand DPT/TBAG1 of The Scientific and Technical Research Concil of Turkey.

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