Instrumental Achievements

Crystal Structure of 2(3*H*)-Benzoxazolone-3-propionitrile

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The aim of this X-ray diffraction study was to elucidate the crystal structure of benzoxazolone having propionitrile. To 500 ml of water were added 0.25 mol of 2-benzoxazolone, 0.3 mol of triethylamine, and 0.3 mol of acrylonitrile. The mixture was heated at 50 – 60˚C for 6 h and then stirred at room temperature for 18 h. At the end of this time, the solid material precipitated was filtered, washed with water to become neutral to turnusol paper, dried and crystallized from methanol (mp. 120˚C). The cell dimensions were determined from the angular settings of 25 reflections obtained from an Enraf-Nonius CAD-4 diffractometer. The space group $P2_1/n$ was determined from the systematic absences. Intensity data were collected in the range $2.2<\theta<74.3$ with variable-speed $\omega/2\theta$ scans using graphite-monochromated Cu K_α radiation. Three standard reflections were monitored at intervals of 120 min. Data were corrected for an intensity variation of 2%. The crystal and experimental data are listed in Table 1. The crystal structure was solved by a direct method using SHELXS-97,[1] and was refined by a full-matrix least-squares method using SHELXL-97[2] with anisotropic temperature factors for non-H atoms. The hydrogen atoms were located geometrically.

The final coordinates and equivalent thermal parameters for non-hydrogen atoms are given in Table 2; selected bond distances and angles are given in Table 3. The molecular structure of the title compound (Fig. 1) is shown in Fig. 2. The bond lengths and angles are in good agreement with the literature.[3,4] All atoms,

Table 1 Crystal and experimental data

Formula: $C_{10}H_8N_2O_2$	
Formula weight = 188.18	
Crystal system: monoclinic	
Space group: $P2_1/n$	$Z = 4$
a = 11.308(1) Å	
b = 5.8389(3) Å	$\beta = 104.502(8)^\circ$
c = 13.931(2) Å	
V = 890.5(2) Å^3	
D_x = 1.404 g/cm^3	
μ(Cu K_α) = 0.835 mm^{-1}	
T = 295 K	
Color: ivory	
F(0 0 0) = 392	
Radiation = 1.5418 Å (Cu K_α)	
$\theta_{max} = 74.3^\circ$	
R = 0.048	
wR = 0.1182	
h k l: h 0/14, k 0/7, l –17/16	
No. of reflections measured = 1809	
No. of reflections used = 1661 [F>2$\sigma(I)$]	
No. of parameters = 160	
Goodness-of-fit = 1.137	
$(\Delta/\sigma)_{max}$ = 0.021	
$(\Delta\rho)_{max}$ = 0.265 eÅ^{-3}	
$(\Delta\rho)_{min}$ = –0.206 eÅ^{-3}	
Measurements: Enraf-Nonius CAD-4 diffractometer	
Refinement: full matrix least-squares (SHELXL-97)	
Program system: CAD-4 EXPRESS Software	
Structure determination: SHELXS-97	
Treatment of hydrogen atoms: geometric calculation	

Table 2 Final coordinates and equivalent isotropic thermal parameters for non-hydrogen atoms

Atom	x	y	z	U_{eq}/Å^2
O1	0.9905(2)	0.1802(3)	0.5874(1)	0.0564(5)
O2	0.8191(2)	0.1787(3)	0.6435(2)	0.0683(6)
N1	0.9584(2)	0.4781(3)	0.6765(1)	0.0485(5)
N2	0.9289(3)	0.2260(5)	0.9116(2)	0.0831(8)
C1	1.0858(2)	0.3330(4)	0.5949(2)	0.0485(6)
C2	1.1838(2)	0.3136(4)	0.5533(2)	0.0567(6)
C3	1.2666(2)	0.4941(5)	0.5716(2)	0.0591(7)
C4	1.2510(2)	0.6801(5)	0.6296(2)	0.0587(6)
C5	1.1505(2)	0.6980(4)	0.6706(2)	0.0528(6)
C6	1.0685(2)	0.5195(3)	0.6511(1)	0.0443(5)
C7	0.9117(2)	0.2732(4)	0.6374(2)	0.0514(6)
C8	0.8947(2)	0.6324(4)	0.7281(2)	0.0530(6)
C9	0.9489(2)	0.6304(4)	0.8402(2)	0.0552(6)
C10	0.9377(2)	0.4054(5)	0.8820(2)	0.0588(6)

$U_{eq} = (1/3)\,\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* (\boldsymbol{a}_i \cdot \boldsymbol{a}_j)$.

Table 3 Selected bond distances (Å), angles (˚) and torsion angles (˚)

O1	C7	1.373(3)		C5	C6	1.376(3)	
O1	C1	1.384(3)		C8	C9	1.528(3)	
O2	C7	1.206(3)		C9	C10	1.455(4)	
N1	C7	1.365(3)		N1	C6	1.399(3)	
N1	C8	1.451(3)		C1	C6	1.384(3)	
N2	C10	1.139(4)		C1	C2	1.377(3)	
C7	O1	C1	107.4(2)	C5	C4	C3	122.0(2)
C7	N1	C6	109.3(2)	C6	C5	C4	116.1(2)
C7	N1	C8	123.5(2)	C10	C9	C8	111.3(2)
N1	C7	O1	108.3(2)	N2	C10	C9	177.7(3)
C2	C1	O1	127.5(2)	C2	C3	C4	121.4(2)
C7	O1	C1	C2	-177.8(2)	C1	O1	C7	O2	179.7(2)
C4	C5	C6	N1	-178.4(2)	C1	O1	C7	N1	-1.0(2)
C2	C1	C6	C5	-1.1(3)	C7	N1	C8	C9	105.6(2)
O1	C1	C6	C5	179.8(2)	C6	N1	C8	C9	-80.4(2)
C7	N1	C6	C5	179.5(2)	C6	N1	C7	O2	179.6(2)
N1	C8	C9	C10	-62.6(3)	C8	C9	C10	N2	25(7)

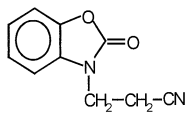

Fig. 1 Chemical structure of 2(3*H*)-benzoxazolone-3-propionitrile.

except for C9, C10 and N2, are coplanar (r.m.s. deviation 0.032 Å). The deviations of C9, C10 and N2 from the plane defined by C1, C2, C3, C4, C5, C6, N1, C7, O1, O2 and C8 are 1.269(3), 2.255(3) and 3.003(3)Å, respectively.

The cyanomethyl group is bonded to the benzoxazolone ring through the C8 atom. According to the values of the related angles (Table 3), the linear chain formed by atoms C9, C10 and N2 is linked with the planar 2-benzoxazolone moiety. The N1–C8–C9–C10 torsion angle is –62.6(3)˚.

The bond lengths of the triple bonds agree well with the reported values [N2≡C10=1.139(4)Å], which are found in (5-chloro-1,3-benzoxazol-2-ylthio)acetonitrile[5] and also in structure of some new D-secoestrone derivatives.[6]

There are three intermolecular C–H··O hydrogen bonds to O2 [C8··O2(–*x*+1/2+1,*y*+1/2,–*z*+1/2+1)=3.359(4), ∠C8–H8A··O2 =119˚; C8··O2 (*x*,*y*+1,*z*)=3.434(3), ∠C8–H8B··O2=157˚; C9··· O2 (–*x*+1/2+1,*y*+1/2,–*z*+1/2+1)=3.108(3), ∠C9–H9A··O2=120˚] and a C–H··N hydrogen bonds to N2 [C3··N2 (*x*+1/2,–*y*+1/2,*z*–1/2) =3.468(4), ∠C3–H3··N2=147˚].[7,8] The closest H··O distance is the intramolecular H8A··O2 of 2.58 Å interaction.[9]

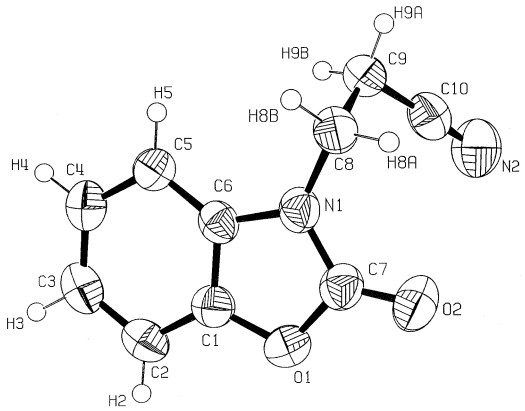

Fig. 2 Perspective view of the molecular structure of the title compound with the atom numbering scheme. The displacement ellipsoids are plotted at the 50% probability level.

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