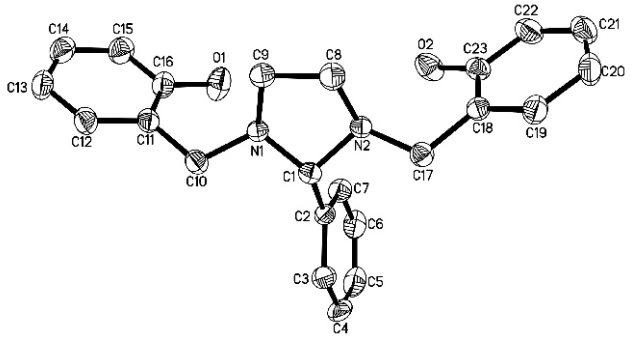
Crystal structure of 1,3-bis(*o*-hydroxybenzyl)-2-phenylimidazolidine, $C_{23}H_{24}N_2O_2$

Yan-Wei Li, Li-Qiao Zhang, Zi-Xing Ren, Su Zhang and Jian-Fang Ma*

Department of Chemistry, Northeast Normal University, Changchun 130024, P. R. China

Received March 24, 2011, accepted and available on-line August 15, 2011; CCDC no. 1267/3475



Abstract

$C_{23}H_{24}N_2O_2$, triclinic, $P\bar{1}$ (no. 2), a = 6.255(5) Å, b = 9.095(5) Å, c = 17.925(5) Å, α = 80.113(5)°, β = 87.715(5)°, γ = 74.388(5)°, V = 967.5 Å^3, Z = 2, $R_{gt}(F)$ = 0.043, $wR_{ref}(F^2)$ = 0.110, T = 293 K.

Source of material

All reagents and solvents were purchased from commercial sources and used as received. A solution of salicylaldehyde (8.54 g, 70 mmol) in methanol (80 mL) was added to a mixture of ethylenediamine (2.8 g, 46.7 mmol) in methanol (20 mL), and a yellow precipitate was obtained immediately. $NaBH_4$ (5.29 g, 140 mmol) was added slowly into the mixture with stirring over 1 h. After 200 mL of water were added, the precipitate of *N*,*N*′-bis(*o*-hydroxybenzyl)ethylenediamine was collected by filtration and dried in air. Recrystallization from methanol gave the white solid with a yield of 71 %. Benzaldehyde (6.36 g, 60 mmol) in methanol (20 mL) was slowly added to a mixture of the precipitate (5.44 g, 20 mmol) in methanol (250 mL). After the mixture was heated at reflux for 8 h and cooled to room temperature, white crystals of the title compound were collected and dried in air. Recrystallization from methanol gave the white target solid with a yield of 53%.

Experimental details

All H atoms on C atoms and O atoms were generated geometrically and refined as riding with d(C—H) = 0.93-0.97 Å, d(O—H) = 0.820-0.821 Å and U_{iso}(H) = 1.2 U_{eq}(C), U_{iso}(H) = 1.5 U_{eq}(O).

Discussion

The asymmetric unit of the title crystal structure contains one 1,3-bis(*o*-hydroxybenzyl)-2-phenylimidazolidine molecule. There are three benzene rings and one five-membered ring in the structure. The five-membered ring (C8, C9, N1, N2 and C1) displays an envelope conformation. Four atoms (C8, C9, N1 and N2) are almost coplanar, and the distances between these four atoms and the mean plane are all less than 0.05 Å. The dihedral angle between the plane of (C1, N1 and N2) and the mean plane of (C8, C9, N1 and N2) is 42.3 Å. ∠N1–C1–N2 = 100.9(1)°. The distance between C1 and the mean plane of (C8, C9, N1 and N2) is 0.63 Å. The benzene ring containing C2 to C7 atoms is nearly perpendicular to the five-membered ring, and the dihedral angle between these two rings is 87.16°. There are intramolecular hydrogen bonds between O1 and N1, as well as O2 and N2 (d(O1—N1) = 2.659 Å, d(O2—N2) = 2.640 Å, ∠O1–H1–N1 = 138.53 Å, ∠O2–H2–N2 = 142.91 Å), which stabilize the whole structure.

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.17 × 0.20 × 0.22 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.79 cm^{-1}
Diffractometer, scan mode:	Oxford Diffraction Gemini R Ultra, ω
$2\theta_{max}$:	58.34°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	7031, 4388
$N(param)_{refined}$:	246
Programs:	SHELXS-97, SHELXL-97 [3]

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

Atom	Site	x	y	z	U_{iso}
H(18)	2*i*	1.0476	0.2833	0.2118	0.048
H(3)	2*i*	0.9059	0.5252	0.2524	0.065
H(4)	2*i*	0.6379	0.6970	0.3130	0.081
H(5)	2*i*	0.3155	0.6362	0.3543	0.085
H(6)	2*i*	0.2532	0.4083	0.3353	0.075
H(7)	2*i*	0.5132	0.2397	0.2745	0.059
H(8A)	2*i*	1.0833	–0.1158	0.2369	0.069
H(8B)	2*i*	1.2533	–0.0137	0.2199	0.069
H(9A)	2*i*	1.1016	0.0717	0.1061	0.062
H(9B)	2*i*	0.9092	–0.0095	0.1282	0.062
H(10A)	2*i*	0.9676	0.3393	0.0728	0.058
H(10B)	2*i*	0.7364	0.4287	0.1012	0.058
H(12)	2*i*	0.8926	0.3803	–0.0607	0.070
H(13)	2*i*	0.7115	0.3392	–0.1618	0.082
H(14)	2*i*	0.4239	0.2250	–0.1406	0.084
H(15)	2*i*	0.3088	0.1592	–0.0194	0.078
H(17A)	2*i*	1.0169	0.1761	0.3642	0.052
H(17B)	2*i*	1.2405	0.0900	0.3297	0.052
H(19)	2*i*	1.3877	–0.0783	0.4452	0.065
H(20)	2*i*	1.3913	–0.2929	0.5360	0.079
H(21)	2*i*	1.0985	–0.4031	0.5431	0.081
H(22)	2*i*	0.8017	–0.2995	0.4623	0.074
H(1)	2*i*	0.5400	0.1966	0.1359	0.113
H(2)	2*i*	0.7604	–0.0034	0.3231	0.100

* Correspondence author (e-mail: majf247nenu@yahoo.com.cn)

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)	2*i*	0.9148(2)	0.2459(2)	0.22280(8)	0.0392(8)	0.0355(7)	0.0436(8)	–0.0091(6)	–0.0002(6)	–0.0060(6)
C(2)	2*i*	0.7397(2)	0.3622(2)	0.25820(8)	0.0484(9)	0.0325(7)	0.0384(8)	–0.0035(6)	–0.0091(7)	–0.0072(6)
C(3)	2*i*	0.7752(3)	0.5005(2)	0.26914(9)	0.065(1)	0.0407(9)	0.055(1)	–0.0079(8)	–0.0188(8)	–0.0072(7)
C(4)	2*i*	0.6138(3)	0.6041(2)	0.3055(1)	0.100(1)	0.0374(8)	0.060(1)	–0.0002(9)	–0.028(1)	–0.0177(8)
C(5)	2*i*	0.4215(3)	0.5680(2)	0.3299(1)	0.081(1)	0.064(1)	0.049(1)	0.0205(9)	–0.0121(9)	–0.0188(9)
C(6)	2*i*	0.3844(3)	0.4324(2)	0.31863(9)	0.057(1)	0.065(1)	0.050(1)	0.0103(9)	–0.0009(8)	–0.0117(8)
C(7)	2*i*	0.5412(2)	0.3311(2)	0.28260(9)	0.0482(9)	0.0467(9)	0.0502(9)	–0.0038(7)	0.0000(7)	–0.0125(7)
C(8)	2*i*	1.0972(3)	–0.0114(2)	0.22079(9)	0.068(1)	0.0462(9)	0.0478(9)	0.0049(8)	0.0039(8)	–0.0107(7)
C(9)	2*i*	0.9904(3)	0.0572(2)	0.14388(9)	0.064(1)	0.0405(8)	0.0477(9)	–0.0045(7)	0.0031(8)	–0.0114(7)
C(10)	2*i*	0.8202(3)	0.3305(2)	0.08777(8)	0.057(1)	0.0405(8)	0.0442(9)	–0.0092(7)	0.0036(7)	–0.0032(7)
C(11)	2*i*	0.7079(3)	0.2977(2)	0.02214(8)	0.0515(9)	0.0365(8)	0.0403(8)	0.0023(7)	0.0017(7)	–0.0038(6)
C(12)	2*i*	0.7739(3)	0.3364(2)	–0.05179(9)	0.070(1)	0.0473(9)	0.047(1)	–0.0029(8)	0.0053(8)	–0.0018(7)
C(13)	2*i*	0.6665(4)	0.3109(2)	–0.1126(1)	0.095(2)	0.057(1)	0.0374(9)	0.006(1)	0.001(1)	–0.0047(8)
C(14)	2*i*	0.4944(4)	0.2441(2)	–0.0999(1)	0.089(1)	0.056(1)	0.051(1)	0.006(1)	–0.020(1)	–0.0125(8)
C(15)	2*i*	0.4255(3)	0.2052(2)	–0.0276(1)	0.068(1)	0.065(1)	0.059(1)	–0.0084(9)	–0.0133(9)	–0.0116(9)
C(16)	2*i*	0.5279(3)	0.2337(2)	0.03337(9)	0.054(1)	0.0557(9)	0.0433(9)	–0.0030(8)	–0.0009(8)	–0.0078(7)
C(17)	2*i*	1.0892(2)	0.0861(2)	0.34142(8)	0.0422(8)	0.0432(8)	0.0440(8)	–0.0104(7)	–0.0010(7)	–0.0064(7)
C(18)	2*i*	1.0933(2)	–0.0587(2)	0.39788(8)	0.0457(8)	0.0387(8)	0.0375(8)	–0.0036(7)	0.0048(7)	–0.0106(6)
C(19)	2*i*	1.2692(3)	–0.1225(2)	0.44810(9)	0.058(1)	0.057(1)	0.0439(9)	–0.0069(8)	–0.0030(8)	–0.0101(8)
C(20)	2*i*	1.2720(3)	–0.2511(2)	0.50266(9)	0.080(1)	0.064(1)	0.0392(9)	0.004(1)	–0.0085(9)	–0.0025(8)
C(21)	2*i*	1.0967(4)	–0.3163(2)	0.5069(1)	0.100(2)	0.047(1)	0.046(1)	–0.007(1)	0.014(1)	–0.0003(8)
C(22)	2*i*	0.9199(3)	–0.2548(2)	0.4586(1)	0.076(1)	0.0457(9)	0.062(1)	–0.0180(9)	0.012(1)	–0.0052(8)
C(23)	2*i*	0.9159(3)	–0.1261(2)	0.40409(9)	0.054(1)	0.0392(8)	0.0490(9)	–0.0091(7)	0.0078(8)	–0.0087(7)
N(1)	2*i*	0.8380(2)	0.2080(1)	0.15420(6)	0.0475(7)	0.0342(6)	0.0369(6)	–0.0064(5)	0.0015(5)	–0.0078(5)
N(2)	2*i*	0.9731(2)	0.0931(1)	0.27115(6)	0.0449(7)	0.0327(6)	0.0379(6)	–0.0031(5)	0.0009(5)	–0.0072(5)
O(1)	2*i*	0.4492(2)	0.1963(2)	0.10427(7)	0.0675(8)	0.118(1)	0.0506(7)	–0.0411(8)	0.0042(6)	–0.0134(7)
O(2)	2*i*	0.7356(2)	–0.0688(1)	0.35738(7)	0.0574(7)	0.0599(7)	0.0809(9)	–0.0239(6)	–0.0086(6)	0.0066(6)

Acknowledgments. We thank the Science Foundation for Young Teachers of Northeast Normal University (grant no. 20060304) and the Analysis and Testing Foundation of Northeast Normal University for support.

References

1. Kan, W.-Q.; Ma, J.-F.: Crystal structure of diaqua-(1,10-phenanthroline)-nickel(II) methanedisulfonate, $Ni(H_2O)_2(C_{12}H_8N_2)[CH_2(SO_3)_2]$. Z. Kristallogr. NCS **225** (2010) 329-330.
2. Xia, H.-T.; Liu, Y.-F.; Yanga, S.-P.; Wang, D.-Q.: 6,6′-Dimethoxy-2,2′-(2,2,4-trimethylimidazolidine-1,3-diyldimethylene)diphenol. Acta Crystallogr. **E63** (2007) o680-o682.
3. Sheldrick, G. M.: A short history of SHELX. Acta Crystallogr. **A64** (2008) 112-122.