Refinement of crystal structure of 2-biphenylboronic acid, $C_{12}H_{11}BO_2$

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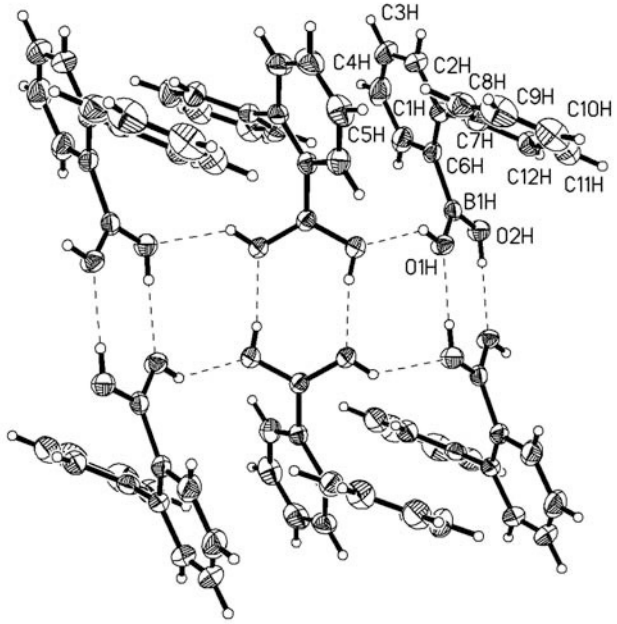

Abstract

$C_{12}H_{11}BO_2$, triclinic, $P\bar{1}$ (no. 2), a = 8.313(2) Å, b = 9.420(2) Å, c = 14.687(3) Å, α = 99.255(2)°, β = 104.533(2)°, γ = 100.311(2)°, V = 1069.5 Å^3, Z = 4, $R_{gt}(F)$ = 0.043, $wR_{ref}(F^2)$ = 0.113, T = 296 K.

Source of material

2-Biphenylboronic acid was purchased from Aldrich, and recrystallized from water/ethanol solution at room temperature to give the desired crystals suitable for single crystal X-ray diffraction investigation.

Discussion

Arylboronic acids $ArB(OH)_2$ are important starting materials in organic synthesis [1]. They are predominantly applied for the Suzuki cross-coupling reaction [2]. Recently arylboronic acids have also been employed as promising building blocks in crystal engineering and various types of novel supramoolecular assemblies have been generated [3,4]. Analogous to carboxylic acids, they are capable of forming dimeric units, this is the most basic and frequent structural motif found in the solid state [5].

The title crystal structure has two crystallographically independent molecules in the asymmetric unit. The benzene rings are not coplanar, with dihedral angles between the two benzene rings of 132.7° and 129.1° in the two molecules. The $B(OH)_2$ group is twisted by 125.5° and 127.2° from the palne of the benzene ring in the two independent molecules. In the crystal structure of the title compound, the $B(OH)_2$ moiety adopted the most preferred syn-anti conformation [6,7], there exist O–H···O hydrogen bonds between the adjacent molecules, which enforce the chain structure of the title compound.

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.16 × 0.18 × 0.38 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.81 cm^{-1}
Diffractometer, scan mode:	Bruker SMART APEXII CCD, φ/ω
$2\theta_{max}$:	51°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	8212, 3954
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2575
$N(param)_{refined}$:	275
Programs:	SHELXS-97 [8], SHELXL-97 [9], SHELXTL [10]

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

Atom	Site	x	y	z	U_{iso}
H(1)	2*i*	0.1217	0.7310	0.1263	0.074
H(2)	2*i*	0.1181	0.4250	0.0125	0.082
H(3)	2*i*	0.0060	–0.0772	0.0632	0.076
H(4)	2*i*	0.2198	0.2348	0.0831	0.072
H(2A)	2*i*	0.6379	0.8616	0.3866	0.093
H(3A)	2*i*	0.8507	0.8839	0.3135	0.113
H(4A)	2*i*	0.7983	0.7693	0.1549	0.104
H(5)	2*i*	0.5272	0.6364	0.0683	0.079
H(8)	2*i*	0.3585	0.9321	0.3905	0.090
H(9)	2*i*	0.1612	0.8978	0.4751	0.114
H(10)	2*i*	–0.0006	0.6664	0.4601	0.110
H(11)	2*i*	0.0338	0.4661	0.3608	0.087
H(12)	2*i*	0.2299	0.4979	0.2758	0.068
H(14)	2*i*	0.6138	0.3550	0.4016	0.074
H(15)	2*i*	0.4478	0.3490	0.5041	0.086
H(16)	2*i*	0.1660	0.2236	0.4489	0.083
H(17)	2*i*	0.0500	0.1054	0.2896	0.069
H(20)	2*i*	0.6462	0.4534	0.2499	0.077
H(21)	2*i*	0.8250	0.4523	0.1535	0.098
H(22)	2*i*	0.8385	0.2339	0.0636	0.097
H(23)	2*i*	0.6707	0.0146	0.0690	0.085
H(24)	2*i*	0.4899	0.0138	0.1650	0.068

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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}
B(1)	2*i*	0.2338(3)	0.5854(2)	0.1071(1)	0.050(1)	0.035(1)	0.045(1)	0.0095(9)	0.014(1)	0.0137(9)
B(2)	2*i*	0.1669(2)	0.0817(2)	0.1394(2)	0.036(1)	0.033(1)	0.053(1)	0.0100(8)	0.0139(9)	0.0092(9)
O(1)	2*i*	0.0940(1)	0.6461(1)	0.09442(9)	0.0458(7)	0.0387(7)	0.0547(8)	0.0085(6)	0.0047(6)	–0.0003(6)
O(2)	2*i*	0.2126(2)	0.4486(1)	0.05228(9)	0.0553(8)	0.0385(7)	0.0620(9)	0.0134(6)	0.0039(6)	0.0042(6)
O(3)	2*i*	0.0681(2)	–0.0580(1)	0.11871(8)	0.0540(8)	0.0375(7)	0.0519(8)	0.0016(6)	0.0047(6)	0.0107(6)
O(4)	2*i*	0.1645(2)	0.1492(1)	0.06329(8)	0.0508(8)	0.0367(7)	0.0456(7)	–0.0013(6)	0.0044(6)	0.0073(5)
C(1)	2*i*	0.4492(2)	0.7368(2)	0.2718(1)	0.047(1)	0.042(1)	0.058(1)	0.0039(9)	–0.0005(9)	0.0155(9)
C(2)	2*i*	0.6140(3)	0.8162(2)	0.3221(2)	0.061(1)	0.068(2)	0.078(2)	–0.012(1)	–0.015(1)	0.026(1)
C(3)	2*i*	0.7418(3)	0.8289(3)	0.2786(2)	0.048(1)	0.088(2)	0.123(2)	–0.015(1)	–0.010(2)	0.051(2)
C(4)	2*i*	0.7108(3)	0.7615(3)	0.1840(2)	0.050(1)	0.083(2)	0.138(2)	0.012(1)	0.031(2)	0.055(2)
C(5)	2*i*	0.5482(3)	0.6819(2)	0.1325(2)	0.053(1)	0.058(1)	0.093(2)	0.012(1)	0.026(1)	0.028(1)
C(6)	2*i*	0.4147(2)	0.6679(2)	0.1742(1)	0.043(1)	0.0372(9)	0.063(1)	0.0084(8)	0.0115(9)	0.0167(9)
C(7)	2*i*	0.3171(2)	0.7179(2)	0.3232(1)	0.056(1)	0.050(1)	0.044(1)	0.0134(9)	–0.0035(9)	0.0085(9)
C(8)	2*i*	0.2937(3)	0.8377(2)	0.3841(2)	0.092(2)	0.060(1)	0.060(1)	0.024(1)	0.000(1)	0.003(1)
C(9)	2*i*	0.1755(4)	0.8173(4)	0.4347(2)	0.122(2)	0.101(2)	0.069(2)	0.059(2)	0.025(2)	0.002(2)
C(10)	2*i*	0.0790(4)	0.6792(4)	0.4258(2)	0.099(2)	0.121(2)	0.081(2)	0.056(2)	0.040(2)	0.032(2)
C(11)	2*i*	0.0993(3)	0.5600(3)	0.3667(2)	0.071(2)	0.083(2)	0.075(2)	0.027(1)	0.027(1)	0.030(1)
C(12)	2*i*	0.2171(2)	0.5795(2)	0.3159(1)	0.062(1)	0.055(1)	0.055(1)	0.016(1)	0.016(1)	0.013(1)
C(13)	2*i*	0.4343(2)	0.2329(2)	0.2815(1)	0.048(1)	0.0370(9)	0.043(1)	0.0116(8)	0.0050(8)	0.0086(8)
C(14)	2*i*	0.5000(3)	0.3040(2)	0.3783(1)	0.068(1)	0.057(1)	0.046(1)	0.012(1)	0.001(1)	0.005(1)
C(15)	2*i*	0.4010(3)	0.3007(2)	0.4398(2)	0.105(2)	0.065(1)	0.040(1)	0.025(1)	0.010(1)	0.005(1)
C(16)	2*i*	0.2332(3)	0.2263(2)	0.4070(2)	0.101(2)	0.069(1)	0.054(1)	0.033(1)	0.039(1)	0.017(1)
C(17)	2*i*	0.1642(3)	0.1556(2)	0.3114(1)	0.063(1)	0.055(1)	0.060(1)	0.013(1)	0.025(1)	0.015(1)
C(18)	2*i*	0.2615(2)	0.1573(2)	0.2466(1)	0.048(1)	0.0363(9)	0.044(1)	0.0111(8)	0.0143(8)	0.0098(8)
C(19)	2*i*	0.5478(2)	0.2333(2)	0.2183(1)	0.038(1)	0.048(1)	0.047(1)	0.0078(8)	0.0033(8)	0.0130(9)
C(20)	2*i*	0.6503(3)	0.3643(2)	0.2136(2)	0.056(1)	0.056(1)	0.076(1)	0.003(1)	0.014(1)	0.019(1)
C(21)	2*i*	0.7575(3)	0.3638(3)	0.1560(2)	0.061(2)	0.085(2)	0.105(2)	0.006(1)	0.030(1)	0.044(2)
C(22)	2*i*	0.7655(3)	0.2337(3)	0.1023(2)	0.061(1)	0.114(2)	0.092(2)	0.034(2)	0.040(1)	0.049(2)
C(23)	2*i*	0.6656(3)	0.1030(3)	0.1055(2)	0.069(1)	0.083(2)	0.077(2)	0.033(1)	0.033(1)	0.022(1)
C(24)	2*i*	0.5573(2)	0.1028(2)	0.1632(1)	0.054(1)	0.052(1)	0.066(1)	0.0145(9)	0.018(1)	0.014(1)

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References

1. Hall, D. G.: Boronic Acids: Preparation and Applications in Organic Synthesis and Medicine. Wiley-VCH, Weinheim 2005.
2. Miyaura, M.; Suzuki, A.: Palladium-Catalyzed Cross-Coupling Reactions of Organoboron Compounds. Chem. Rev. **95** (1995) 2457-2483.
3. Fournier, J. H.; Maris, T.; Wuest, J. D.; Guo, W. Z.; Galoppini, E.: Molecular Tectonics. Use of the Hydrogen Bonding of Boronic Acids To Direct Supramolecular Construction. J. Am. Chem. Soc. **125** (2003)1002-1006.
4. Pedireddi, V. R.; SeethaLekshmi, N.: Boronic acids in the design and synthesis of supramolecular assemblies. Tetrahedron Lett. **45** (2004) 1903-1906.
5. Rodríguez-Cuamatzi, P.; Arillo-Flores, O. I.; Bernal-Uruchurtu, M. I.; Höpfl, H.: Theoretical and Experimental Evaluation of Homo- and Heterodimeric Hydrogen-Bonded Motif containing Boronic Acids, Carboxylic Acids, and carboxylate Anions: Application for the Generation of Highly Stable Hydrogen bonded Supramolecular Systems. Cryst. Growth Des. **5** (2005)167-175.
6. SeethaLekshmi, N.; Pedireddi, V. R.: Supramolecular Architecture in Some 4-Halophenylboronic Acids. Cryst. Growth Des. **7** (2007)1985-1963.
7. SeethaLekshmi, N.; Pedireddi, V. R.: Solid-State Structure of 4-Carboxyphenylboronic Acid and Its Hydrates. Cryst. Growth Des. **7** (2007)944-949.
8. Sheldrick, G. M.: SHELXS-97. Program for the Solution of Crystal Structures. University of Göttingen, Germany 1997.
9. Sheldrick, G. M.: SHELXL-97. Program for the Refinement of Crystal Structures. University of Göttingen, Germany 1997.
10. Sheldrick, G. M.: SHELXTL. Structure Determination Software Suite. Version 6.14. Bruker AXS, Madison, Wisconsin, USA 2000.