THE CRYSTAL STRUCTURE OF A DIMERIC COMPLEX FORMED BETWEEN BORIC ACID AND N-2-HYDROXYPHENYLSALICYLALDIMIN

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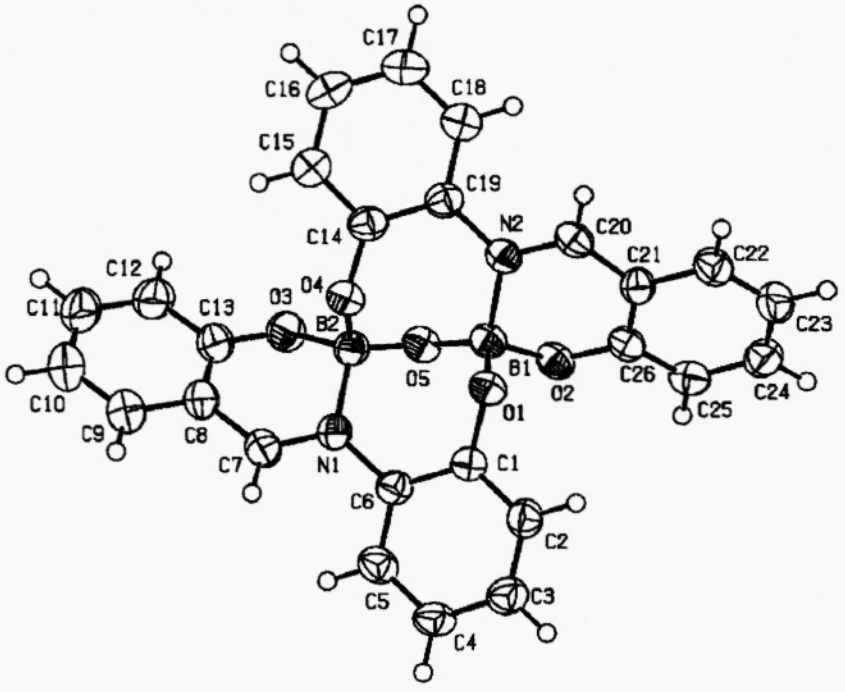

Figure 1. Molecular structure (50% probability ellipsoids) of $[C_{26}H_{18}B_2N_2O_5]$. Selected bond distances and angles; O1-B1 1.475(4), O2-B1 1.469(4), O3-B2 1.479(4), O4-B2 1.479(4), O5-B1 1.396(4), O5-B2 1.389(4), N1-B2 1.619(5), N2-B1 1.616(4) Å; B1-O5-B2 117.3(2), O1-B1-N2 101.8(2), O2-B1-O5 109.4(2), O2-B1-N2 107.1(2), O1-B1-O2 110.1(3), O1-B1-O5 115.4(3), O5-B1-N2 112.5(3), O4-B2-N1 101.7(3), O5-B2-N1 113.1(3), O3-B2-O4 109.3(3), O3-B2-O5 110.9(3), O3-B2-N1 106.2(3)°.

Comment

Boron is an essential trace element for plants, however it is not for animals including humans. Boron in the form of boric acid (H_3BO_3) is very stable in aqueous solution, has low reactivity and hence, it is generally difficult to find a useful complex-forming reagent for boric acid. Only a few organic reagents are available for the determination of boric acid by spectrophotometry. Azomethine-H, Berillon(III) and H-Resorcinol are some of known reagents for boric acid. These reagents contain ONO type donor atoms [1-4]. In this study, a dinuclear complex was prepared by a reaction between boric acid and a similar ONO type Schiff base, N-2-hydroxyphenylsalicylaldimin, in acetonitrile solution. As seen Fig 1, both boron atoms are in tetrahedral coordination sphere provided by one imin nitrogen and three oxygen atoms. The bond distances found in this study, 1.616(4)-1.619(5) Å for B-N bonds and 1.389(6)-1.479(4) Å for B-O bonds are in agreement with recently cited literatures [5-6].

Experimental

Preparation: 0.212 g (0.001 mole) N-2-hydroxyphenylsalicylaldimin was dissolved in about 40 mL of acetonitrile by heating. A solution of 0.062 g (0.001 mole) boric acid in about 20 mL hot acetonitrile was added into this solution. The mixture was set-aside for about 24 hours and light yellow crystalline complex was filtered and dried in air.

Crystallography:

Table 1. Crystal and experimental data

Formula= $C_{26}H_{18}B_2N_2O_5$	Formula weight = 460.04
Crystal system= monoclinic	Crystal size, mm = 0.20 x 0.20 x 0.15
Space group= *P*21/*c*	*a*, Å = 12.5070(10)
b, Å = 8.5110(10)	*c*, Å = 20.2380(5)
β ° = 94.64(2)	*V*, Å^3 = 2147.2(3)
Z = 4	Diffractometer: Enraf-Nonius CAD-4
Temperature, K = 295	μ (Cu Kα), mm^{-1} = 0.801
Trans. Factors (T_{min}, T_{max}) = 0.854, 0.887	D_x, g/cm^3 = 1.423
F(000) = 952	Θ_{max}, ° = 87.85
Reflns. Meas. = 5006	Reflns unique, R_{int} = 2438, 0.002
Reflns with I≥2σ(I) = 2039	Weighting scheme:
R, $R_w(F^2)$ = 0.042, 0.134	$w = 1/[\sigma^2(F_o^2) + (0.1713\ P)^2 + 2.6355\ P$
Deposition number: CCDC 155726	Where $P = (F_o^2 + 2F_c^2) / 3$
Abs correction = empirical via ψ scans [7]	$\Delta\rho_{min}$, $\Delta\rho_{max}$ (eA^{-3}) = -0.23, 0.29

Program used: Platon [8], WinGX [9], Shelxs-97 [10], Shelxl-97 [11].

Acknowledgements

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References

[1] F.J. Krug, J. Mortatti, L.C.R. Pessenda, E.A.G. Zagatto, H. Bergamin, *Anal. Chim. Acta*, 125, 29-35, 1981.
[2] W. Jin, L. Xiao, Y. Wu, *Anal. Chim. Acta*, 280, 69-74, 1993.
[3] L. Thunus, *Anal. Chim. Acta*, 318, 303-308, 1996.
[4] S. Motomizu, M. Oshima and Z. Jun, *Analyst*,115, 389-392.
[5] V. Barba, D. Cuahutle, M.E. Ochoa, R. Santillan, N. Fargan, *Inorg. Chim. Acta*, 303, 7-11, 2000.
[6] V. Barba, R. Luna, D. Castillo, R. Santillan, N. Fargan, *J.Organometallic Chem.*, 604, 273-282, 2000.
[7] C.K. Fair, MolEN. An interactive intelligent System for Crystal Structure Analysis, Enraf Nonius, Delft, The Netherlands, 1990
[8] A. L. Spek, PLATON, A Multipurpose Crystallographic Tool, Utrecht University, Utrecht, The Netherlands (2000).
[9] L. J. Farrugia, *J. Appl. Cryst.*, (1999), 32, 837-838.
[10] G. M. Sheldrick, SHELXS97, Program for the Solution of Crystal Structures, University of Göttingen, Germany (1997a).
[11] G. M. Sheldrick, SHELXL97, Program for the Refinement of Crystal Structures, University of Göttingen, Germany (1997b).

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