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X-RAY STRUCTURE OF 1,2'-DIBORAZINE-2,6-DICHLORO-4,4',6'-TRI(DIISOPROPYLAMINO) TOLUENE SOLVATE

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Figure 1. Molecular structure of the diborazine 1,2':[(2,6-Cl4-4-(Pr3-N)B3N2H3)(4,4',6'-Pr3-N-B3N2H3)] 1 in the structure of the title compound. Selected bond distances (Å) and angles (°) for 1: B-Cl 1.782(6), N4-B(2) 1.501(8), N3-B(2) 1.42(1), N3-B(1) 1.45(1), N2-B(1) 1.44(1), N2-B(5) 1.46(1), N5-B(3) 1.388(7), N5-B(4) 1.450(6), N4-B(3) 1.429(6), N10-B(4) 1.42(1), N1-B(1) 1.42(1), N1-C(1) 1.459(9), N1-C(2) 1.49(1), N7-B(5) 1.427(9), N7-C(8) 1.46(1), N7-C(10) 1.46(1), N10-C(12) 1.471(3), B(2)-N(3)-B(1) 123.4(6), B(5)-N(6)-B(2) 123.9(4), B(3)-N(4)-B(2) 122.6(3), B(3a)-N(4)-B(3) 114.7(5), N(3)-B(2)-N(4) 120.9(6), N(6)-B(2)-N(4) 121.4(6), B(3)-N(5)-B(4) 125.4(4), B(1)-N(2)-B(5) 125.0(5), N(1)-B(1)-N(2) 123.4(7), N(2)-B(1)-N(3) 114.2(6), N(2)-B(5)-N(7) 122.6(7), N(1)-B(1)-N(3) 122.3(7), N(3)-B(2)-N(6) 118.6(5), N(6)-B(5)-N(7) 122.5(6), N(2)-B(5)-N(6) 114.9(5), N(10)-B(4)-N(5) 124.7(3), N(4)-B(3)-N(5) 122.1(4), N(5)-B(4)-N(5a) 110.7(6).

Comment
The unit cell contains four molecules of toluene and four molecules of the compound 1. The latter is a diborazine displaying a linked ring structure. The endo-rings B-N distances are between 1.388(7) and 1.46(1) Å falling within the range of the distances found in other borazines [1, 2]. It should be noted that this large range is also observed in the other previously described diborazine 1,2':(B3N2H3)2 [3]. As expected, the exo-rings B-N(ringo) bond lengths [av. 1.42(1) Å] are consistent with a B-N double-bond character [4]. The B – Cl bond lengths [1.782(6) Å] agree within experimental error with the values reported for 2,4,6-trichloroborazine, 1.76(2) Å [2] and for boron trichloride, ~1.73-1.76 Å [4]. The significant feature of this structure is the orthogonal arrangement of the borazine rings planes unlike in the diborazine 1,2':(B3N2H3)2 [3]. In the latter, the two rings are coplanar, as diphenyl in the solid state, and the inter-ring B-N bond is short [1.464(7) Å] mirroring its double-bond character. By contrast, in 1 the relative twist of the B3N planes...
[90.00(1)°] precludes any interaction between the lone pair of the N(4) nitrogen atom and the vacant p-orbital of the B(2) boron atom. Thus, this distance [1.502(8) Å] is longer than in 1,2'-B(N,NH)2 and resembles B-N single-bonds [1.58 Å] rather than B-N double-bond distances [1.41 Å]. This phenomenon is presumably related to the steric bulk of the borazine substituents in ortho and meta positions of both rings which precludes their coplanarity.

**Experimental**

**Preparation**

All experiments were performed under atmosphere of pure argon and anhydrous conditions using vacuum line and schlenk techniques with solvents purified by standard techniques. 2,4,6-trichloroborazine [5] and chloro(diisopropylamino)borane [6] were prepared as previously described and purified by vacuum sublimation and distillation, respectively. Triethylamine (17.8 mmol) was added to a solution of 2,4,6-trichloroborazine (5.94 mmol) and chloro(diisopropylamino)borane (17.8 mmol) in toluene (20 mL). The mixture was stirred for 24 h at room temperature and then heated under reflux conditions for 2 h. Under cooling, suitable crystals for X-ray analysis were obtained. 1H NMR (CDCl3): δ 1.12, 1.13 (CH3); 3.05 (NH): 3.32, 3.39 (CH). 13C NMR (CDCl3): 72.0.

**Table 1. Crystal data for 1,2'-[(2,6-C12-4-(Pr3N)B3N9H3)-(4',6'-Pr3N)B3N9H3] (C16H21)ClH3**

| Formula | C25H13B5Cl2N9 | Formula weight | 617.54 |
| Crystal system | orthorhombic | | |
| Space group | Cmc21 | | 0.30 x 0.22 x 0.15 |
| a, Å | 11.871(2) | | |
| b, Å | 18.919(4) | c, Å | 16.251(3) |
| g, Å3 | 3650(1) | Z | 4 |
| Diffractometer | Nonius Kappa CCD | | |
| μ(Mo-Kα), cm⁻¹ | 2.08 | | 0.940-0.970 |
| Dcalcd, g cm⁻³ | 1.124 | F(000) | 1328 |
| Density unique | 24.7 | Reflns meas. | 3028 |
| Reflns unique | 3028 | Reflns with I > 2σ(l) | 2119 |
| R(F²), Rw(F²) | 0.084, 0.232 | ρ, e Å⁻³ | 0.51 and -0.49 |

**References**


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