The torsion angles most clearly illustrate the absence of an intramolecular mirror plane.

The observed value of the distance between the atoms C(1) and C(8) is 3.396 Å. The EFF calculated value is 3.525 Å. When the C(1)...C(8) distance and the valency angle at C(9) are constrained to the observed values during the EFF calculation, the energy rises 1.4 kJ mol⁻¹ and m shows up again. An alternate empirical force field (EAS, Engler, Andose & von R. Schleyer, 1973) gives 3.602 Å for the non-bonded distance C(1)...C(8), 117.4° for the valency angle at C(9) and the energy increases by 3.3 kJ mol⁻¹ when the observed values are used. Therefore, the prediction of geometric details of the strained structure of (I) using MM2 is better than with EAS. However, the former set of force-field parameters is also capable of improvement.

The crystals were provided by Dr D. Tavernier, State University at Gent, Belgium.

References


Dimeric tert-Butylaminoboron Difluoride, [(CH₃)₃CNHBF₂]₂

BY PETER G. JONES

Institut für Anorganische Chemie der Universität Göttingen, Tannmannstrasse 4, D-3400 Göttingen, Federal Republic of Germany

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Abstract. Mₑ = 241.88, orthorhombic, space group Pbca, a = 7.615 (4), b = 17.007 (8), c = 9.706 (5) Å, V = 1257 (1) Å³, Z = 4 (dimers), Dₓ = 1.278 (1) g cm⁻³, F(000) = 512, room temperature, λ(Mo Kα) = 0.71069 Å, μ(Mo Kα) = 1.1 cm⁻¹, R = 0.037 for 776 unique observed reflections. The dimers possess crystallographic inversion symmetry, with B–N 1.595 (4) Å, and B–F 1.355 (4) and 1.366 (4) Å. The B₂N₂ rings are exactly planar.

Introduction. The dehydrohalogenation of the title compound (I) (Elter, Glemser & Herzog, 1971; Greenwood, Hooton & Walker, 1966; Greenwood & Robinson, 1968) leads to (amongst other, non-cyclic, products) the tetrameric azaborane [(CH₃)₃CNBF₄]₄ (Elter, Noltemeyer & Sheldrick, 1984). We were therefore interested to discover whether (I) already contained the eight-membered BN ring in the solid state, or if, similarly to compounds RENBX₂ [R = Me, X = F, Hazell (1966); R = Et, X = F, Edwards & Stadler (1970); R = Me, X = Cl, Hess (1963)], it was dimeric. The molecular weight in solution suggests a dimeric formulation, although a tetramer gradually forms on standing (Greenwood et al., 1966).

Experimental. Dₓ not determined. Colourless rhombic prisms, sealed in glass capillaries to prevent slow hydrolysis by atmospheric moisture. Stoe two-circle diffractometer, monochromated Mo Kα radiation. Crystal 1, mounted about c, 0.45 x 0.4 x 0.2 mm, layers 0–7, 1029 reflections. Crystal 2, mounted about a, 0.55 x 0.45 x 0.3 mm, layers 0–7, 1495 reflections. Interlayer scale factors derived from least-squares analysis of common reflections after Lp corrections. No absorption correction. 870 unique reflections (R_{int} 0.023, 2θ_{max} 55°), of which 776 with F > 4σ(F) used for all calculations. Cell constants refined from θ₀ values of 231 strong reflections from various positive and negative layers (Clegg & Sheldrick, 1984). Structure solution by routine direct methods. Refinement on F to R 0.037, R_w 0.036 [non-H atoms anisotropic, H(1) refining freely isotropic, other H using riding model...
with C–H 0.96 Å, H–C–H 109.5°, U(H) = 1.2 Ueq(C); weighting scheme $w^{-1} = \sigma^2(F) + 0.0001F^2$; 86 parameters, $S = 2.0$, slope of normal probability plot 1.6. Max. $\Delta/\sigma$ 0.002; max. and min. heights in final $\Delta$F map +0.16, −0.14 e Å$^{-3}$. No correction for secondary extinction. Calculations performed with program system SHELXTL (Sheldrick, 1978).

Discussion. Final atomic coordinates and derived parameters are given in Tables 1 and 2.* The structure is shown in Figs. 1 and 2. Compound (1) is dimeric; since the dimers possess a crystallographic centre of symmetry, the BEN2 ring is constrained to be exactly planar. The B–N and B–F bond lengths are similar to those of the other $R_2NBX_2$ dimers, although the low accuracy of the earlier studies precludes useful comparisons. The shortest intermolecular contacts between non-H atoms are F(2)...C(2) 3.18 (2), F(1)...N(1) 3.29 (2) Å (second atoms at $-l+x,y,z$; $-0.5+x,y,1.5-z$ respectively).

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39422 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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References


