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Crystallographic report

Trimethylaminedibromopropoxycarbonylborane

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The conformation around the boron atom is nearly tetrahedral, with Br–B–Br bond angles of 109.14(19)° and slightly wider N–B–C bond angles up to 113.6(3)°. Copyright © 2005 John Wiley & Sons, Ltd.

KEYWORDS: crystal structure; amine dibromocarboxyborane ester; trimethylaminedibromopropoxycarbonylborane

COMMENT

A new one-pot method for the synthesis of trimethylaminedibromocarboxyborane esters from trimethylaminecarboxyborane in high purity and excellent yields was reported previously.[1] Since a boron-attached bromine atom is considered to be a good leaving group,[2] such compounds are considered as precursors for boron-substituted amine carboxyboranes, which are isoelectronic boron analogs of α-amino acids and their derivatives.[3] In this connection, the molecular structure has been determined, Fig. 1. The conformation around the boron atom is nearly ideally tetrahedral, with Br–B–Br bond angles of 109.14(19)° and slightly wider N–B–C bond angles up to 113.6(3)°. The observed data are comparable to those found in related compounds that contain an N–BBr_2–C fragment.[4,5]

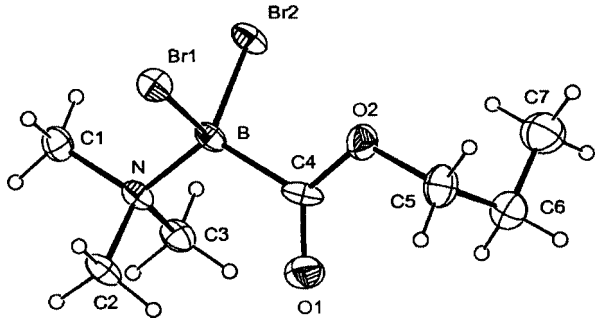

Figure 1. Molecular structure of $C_7H_{16}BBr_2NO_2$. Selected bond distances and angles are: B–Br1 2.026(4), B–Br2 2.030(4), B–N 1.603(5), B–C4 1.695(6), N–C1 1.491(5), O1–C4 1.199(5), O2–C4 1.296(5), O2–C5 1.469(5), C5–C6 1.500(7), C6–C7 1.516(7) Å; Br1–B–Br2 109.14(19), Br1–B–N 109.4(2), Br2–B–C4 108.9(2), O1–C4–B 121.2(3), O2–C4–B 110.8(3), N–B–C4 113.6(3), C1–N–C2 107.0(3), C1–N–B 112.7(3)°.

EXPERIMENTAL

The title compound was prepared from trimethylaminecarboxyborane in a one-pot method as described elsewhere.[1] Colorless single crystals were obtained from hot hexane. Crystal data for: $C_7H_{16}BBr_2\ NO_2$, $M = 316.84$, monoclinic, $P2_1/c$, $a = 15.0640(4)$, $b = 6.878\,00(10)$, $c = 11.7340(5)$ Å, $\beta = 109.0100(12)°$, $V = 1149.46(6)$ Å^3, $Z = 4$, Nonius KappaCCD difractometer, $T = 110(2)$ K, $R = 0.042$ (2364 data with $I \geq 2\sigma(I)$; $\theta_{max} = 28.2°$), $wR = 0.125$ (all 2734 data). Programs used: SHELXL-97, Denzo, SIR-92. CCDC number: CCDC 247984.

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