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Key indicators

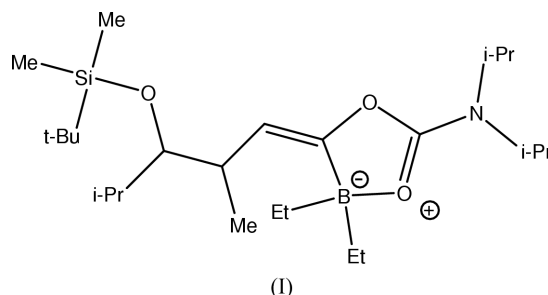
Single-crystal X-ray study
T = 193 K
Mean $\sigma(\text{C}-\text{C}) = 0.007 \text{ \AA}$
Disorder in main residue
R factor = 0.059
wR factor = 0.169
Data-to-parameter ratio = 10.6For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.A vinylic α -carbamoyloxyborane with a carbenoid-type bonding arrangement

In the crystal structure, the title compound, $\text{C}_{25}\text{H}_{52}\text{BNO}_3\text{Si}$, a vinylic α -carbamoyloxyborane, forms the five-membered ring of an alkylidene oxoniaboratadihydrofuran as a result of the bonding interaction of the carbonyl group with the boron atom. The ring is almost planar, with the boron atom in a tetrahedral configuration. The molecule exhibits a carbenoid-type bonding arrangement.

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Comment

Nucleophilic substitution at cyclopropane and, especially, at vinyl-type C atoms occurs only if the C atom in question bears both a metal and a leaving group (carbenoid) (Boche & Lohrenz, 2001; Kociński & Barber, 1990), or if a metallate rearrangement is involved. The latter requires a leaving group and an ate complex of Al, B, Cu, Ni, Zn or Zr at the same C atom (Nelson & Matthews, 1994; Harada *et al.*, 1993; Sidduri *et al.*, 1993). Here we describe the solid-state structure of the vinylic α -carbamoyloxyborane, (I), which was formed from the corresponding lithium borate and acetic acid (Birkinshaw & Kociński, 1991).



As a result of the bonding contact of the carbonyl group and to boron, the structure has to be described as an oxoniaboratadihydrofuran. A comparable situation can be found in the crystal structure of 2,2-diethyl-3,4-dimethoxy-5-[(1-phenylethyl)amino]1-oxonia-2-borata-1(5),3-cyclopentadiene (Barua *et al.*, 1987).

Most remarkably, the C1—O1 bond is lengthened to 1.460 (4) Å. In six non-metallated carbamoyloxy-substituted vinylic compounds, the mean $\text{C}_{\text{sp}^2}-\text{O}(\text{carbamoyloxy})$ value is 1.396 Å (Boche *et al.*, 1992). It thus turns out that a boron substituent together with a leaving group leads also to a carbenoid-type bonding situation, as is the case with lithium (Boche & Lohrenz, 2001; Boche *et al.*, 1992).

Experimental

The title compound was prepared from the corresponding lithium borate and acetic acid (Birkinshaw & Kociński, 1991).

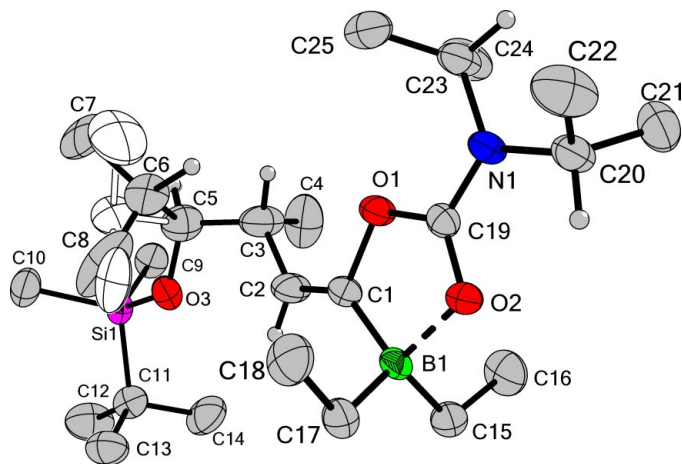


Figure 1
DIAMOND (Brandenburg, 2001) drawing of the title compound, showing the atomic numbering and disorder of the isopropyl group bonded to C5. Displacement ellipsoids are drawn at the 30% probability level. Methyl and methylene H atoms are not shown.

Crystal data

$C_{25}H_{52}BNO_3Si$
 $M_r = 453.58$
 Monoclinic, $P2_1/n$
 $a = 14.684$ (6) Å
 $b = 9.355$ (1) Å
 $c = 22.515$ (9) Å
 $\beta = 101.68$ (2)°
 $V = 3028.8$ (18) Å³
 $Z = 4$

$D_x = 0.995$ Mg m⁻³
 Cu $K\alpha$ radiation
 Cell parameters from 25 reflections
 $\theta = 35$ –50°
 $\mu = 0.84$ mm⁻¹
 $T = 193$ (2) K
 Needle, colorless
 $0.4 \times 0.2 \times 0.1$ mm

Data collection

Enraf–Nonius CAD-4 diffractometer
 ω scans
 Absorption correction: refined from ΔF (DIFABS; Walker & Stuart, 1983)
 $T_{\min} = 0.50$, $T_{\max} = 0.67$
 3959 measured reflections
 3786 independent reflections

2882 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.046$
 $\theta_{\text{max}} = 55.2^\circ$
 $h = 0 \rightarrow 15$
 $k = 0 \rightarrow 9$
 $l = -23 \rightarrow 23$
 2 standard reflections
 frequency: 60 min
 intensity decay: none

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.059$
 $wR(F^2) = 0.169$
 $S = 1.04$
 3786 reflections
 356 parameters
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0818P)^2 + 2.3718P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.55$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.18$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

B1–C17	1.594 (6)	O1–C19	1.322 (4)
B1–C15	1.599 (5)	O1–C1	1.460 (4)
B1–C1	1.609 (5)	O2–C19	1.259 (4)
B1–O2	1.614 (4)	C1–C2	1.308 (5)
C17–B1–C15	115.3 (3)	C19–O1–C1	108.1 (3)
C17–B1–C1	115.1 (3)	C19–O2–B1	110.0 (2)
C15–B1–C1	113.3 (3)	C2–C1–O1	114.7 (3)
C17–B1–O2	106.9 (3)	C2–C1–B1	138.1 (3)
C15–B1–O2	107.9 (3)	O1–C1–B1	107.1 (3)
C1–B1–O2	96.0 (2)		
C1–B1–O2–C19	2.9 (3)	O2–B1–C1–O1	–3.2 (3)
C19–O1–C1–C2	–176.1 (4)	O1–C1–C2–C3	3.5 (7)
C19–O1–C1–B1	2.8 (4)	B1–O2–C19–O1	–1.6 (4)
O2–B1–C1–C2	175.2 (5)	C1–O1–C19–O2	–0.8 (4)

The disorder of the isopropyl group bonded to C5 was refined using restraints for distances and temperature factors.

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg, 2001); software used to prepare material for publication: *SHELXTL* (Sheldrick, 1996).

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