[2,2'-Iminodiethanolato(2−)-κ3O,N,O']-[4-(methoxycarbonylmethyl)phenyl]boron

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Key indicators: single-crystal X-ray study; T = 153 K; mean σ(C−C) = 0.004 Å; R factor = 0.043; wR factor = 0.112; data-to-parameter ratio = 10.0.

The title compound, C13H18BNO4, was readily obtained from the reaction of methyl 4-boronobenzene acetate with ethanolamine. A combination of intermolecular N−H⋯O hydrogen bonds and C−H⋯π interactions leads to the pairwise association of molecules.

Related literature
For background to the biological importance of boron, see: Warrington (1923); Jabbour et al. (2004). For the use of boron-containing reagents in synthetic chemistry, see: Miyaura & Suzuki (1995); Corey et al. (1987); Liu et al. (2007); Jung & Lazarova (1999); Chan et al. (1998); Evans et al. (1998); Lam et al. (1998). For related structures, see: Rettig & Trotter (1975); Wang & Georghiou (2002).

Experimental
Crystal data
C13H18BNO4
M, = 263.10
Orthorhombic, P212121
a = 8.3776 (11) Å
b = 8.9269 (11) Å
c = 17.369 (2) Å

V = 1299.0 (3) Å³
Z = 4
Mo Kα radiation
μ = 0.10 mm⁻¹
T = 153 K
0.30 × 0.09 × 0.06 mm

Data collection
Rigaku Saturn diffractometer
Absorption correction: numerical
(NUMABS; Higashi, 1999)
T,min = 0.985, T,max = 0.997
16363 measured reflections
1725 independent reflections
1707 reflections with I > 2σ(I)
Rint = 0.037

Refinement
R[F² > 2σ(F²)] = 0.043
wR(F²) = 0.112
S = 1.17
1725 reflections
173 parameters
H-atom parameters constrained
Δρmax = 0.20 e Å⁻³
Δρmin = −0.22 e Å⁻³

Table 1
Hydrogen-bond geometry (Å, °).

Symmetry codes: (i) −x + 2, y + 1, −z + 1/2; (ii) −x + 2, y + 1, −z + 1/2.

Data collection: CrystalClear (Rigaku, 2002); cell refinement: CrystalClear; data reduction: CrystalClear; program(s) used to solve structure: SHELX97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: ORTEP-3 (Farrugia, 1997); software used to prepare material for publication: publCIF (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FJ2337).

References
supplementary materials
Boron is known to be an important trace element in higher plants (Warrington, 1923) and boron-containing compounds have been shown to have a range of diverse biological activities (Jabbour et al., 2004). The use of boron-containing reagents is also widespread in synthetic chemistry and this is due mainly to the pioneering work of H. C. Brown and coworkers and Suzuki and his coworkers (Miyaura & Suzuki, 1995). Corey, Bakshi and Shibata (Corey et al., 1987) discovered that a chiral oxazaborolidine ("CBS" reagent) which contains both boron-nitrogen and boron-oxygen bonds was capable of effecting enantioselective reduction of prochiral ketones, imines, and oximes to produce chiral alcohols, amines, and amino alcohols in excellent yields and ee's. Corey's group has also shown that chiral oxazaborolidine-aluminium bromide complexes (Liu et al. 2007) are also effective catalysts for enantioselective Diels-Alder reactions. In principle, oxazaborolidines are derived from reactions of a boronic acid and aminoalcohols and a less well known application of oxazaborolidines is to facilitate the conversion of a pinacolatoborane, by mild acid-catalysis (Jung & Lazarova, 1999), to the corresponding boronic acid, a key step for cupric acetate promoted coupling of an arylboronic acids with phenols (Chan et al., 1998; Evans et al., 1998; Lam et al. 1998).

There has been only one reported X-ray crystallographic study of the structure of a diethanolamine ester of a phenylboronic acid (1) (Rettig & Trotter, 1975). This compound which was named as B-phenyl-diptychboroxazolidine (alternative names: Tetrahydro-[1,3,2]oxa-borol[2,3-b][1,3,2]oxazaborole; [2,2'-(Imino-kN)bis[ethanolato-kO]](2-)]phenylboron) was measured on a diffractometer with Cu K\(_a\) radiation and it was revealed to be non-centrosymmetric and in the \(P2_1\) space group. The absolute configuration of the enantiomorphic crystal was determined in this study. In connection with our own work (Wang & Georghiou, 2002), crystals of (2), the corresponding diethanolamine ester of the 4-boronic acid derivative of methyl phenylacetate, were obtained and the structure of the molecule is reported here.

Methyl \(p\)-[2,2'-iminobis[ethanolato]](2-)]\(N,O,O'\)phenylacetateboron (2; Figure 1) crystallized in the non-centrosymmetric space group \(P2_12_12_1\), however, data collection was performed using molybdenum radiation, and the absolute configuration could not be determined due to the lack of an atom with significant anomalous dispersion. Intermolecular hydrogen bonding between \(N1—H1···O2\) (\(N1···O2 = 2.921 (2) \AA\)) and \(C—H···\pi\) interactions between \(C10—H10B···Cg3\) (\(C10···Cg3= 3.618 (2)\); where \(Cg3\) is the centroid of \(C1—C6\)) leads to the pair-wise association of molecules (Figure 2). These molecular associates are related via the twofold screw axes in the crystal structure (viewed perpendicular to the \(b\) axis in Figure 3).

**Experimental**

To a solution of \(\text{PdCl}_2(\text{dppf})\) (160 mg, 0.18 mmol) in dioxane (24 ml) was added methyl 4-(trifluoroacetoxyacetate (1.58 g, 5.93 mmol), \(\text{Et}_3\text{N}\) (2.49 ml, 17.8 mmol) and 4,4,5,5-tetramethyl-1,3,2-dioxaborolan (1.30 ml, 8.9 mmol). After stirring for 20 h at 100 °C, the reaction mixture was extracted with benzene. The extract was purified by flash column chromatography
supplementary materials

(silica gel, 10%EtOAc in hexanes) to afford the arylboronate (1.47 g, 90%) $^1$HNMR (500 MHz, CDCl$_3$) 1.34 (s, 12H), 3.64 (s, 2H), 3.67(s, 3H), 7.27(d, $J=10$ Hz, 2H), 7.77(d, $J=10$ Hz, 2H). To a solution of the arylboronate (1.47 g, 5.3 mmol) in diethylether (53 ml) was added diethanolamine (0.6 ml, 5.8 mmol) in 2-propanol (10 ml). The resulting mixture was stirred at ambient temperature for 72 h, the reaction mixture was then filtered and the solid was washed with diethyl ether to give cyclic aminoarylboronate (1.08 g, 77%) as a colorless powder. Crystals suitable for X-ray diffraction analysis were obtained by crystallization from ethyl acetate solution. $^1$H NMR (500 MHz, CDCl$_3$) 2.49–2.51(m, 2H), 2.96–3.03(m, 2H), 3.58(s, 2H), 3.63(s, 3H), 3.70–3.72(m, 2H), 3.79–3.74(m, 2H), 5.83(s, 1H), 7.14(d, $J=7.5$ Hz, 2H), 7.43(d, $J=7.5$ Hz, 2H); $^{13}$C NMR 41.0, 51.1, 51.8, 63.2, 128.3, 132.7, 132.8, 172.6.

Refinement

H atoms were found in difference Fourier maps and subsequently placed in idealized positions with constrained distances and with $U_{iso}(H)$ values set to either 1.2Ueq or 1.5Ueq of the attached atom. They were refined on a riding model. All non-hydrogen atoms were refined anisotropically. This crystal was a weak anomalous scatterer collected with MoKa radiation, therefore, Friedel mates were merged (MERG 4) and absolute configuration was not determined.

Figures

Fig. 1. The molecular structure of (2), with atom labels and 50% probability displacement ellipsoids for non-H atoms.

Fig. 2. Intermolecular hydrogen bonds (long dashes) and C—H···π interactions (short dashes) between two associated molecules.

Fig. 3. Unit cell viewed perpendicular to the $b$ axis, showing the pair-wise ordering of molecules in the crystal lattice. (Cell axes: a = red, b = green, c = blue)
supplementary materials

[2,2'-Iminodiethanolato(2-)-κ³O,N,O'][4- (methoxycarbonylmethyl)phenyl]boron

Crystal data
C₁₃H₁₈BNO₄

$M_r$ = 263.10

Orthorhombic, $P2_12_12_1$

Hall symbol: P 2ac 2ab

$a$ = 8.3776 (11) Å

$b$ = 8.9269 (11) Å

$c$ = 17.369 (2) Å

$V$ = 1299.0 (3) Å³

$Z$ = 4

$F(000)$ = 560

$D_x$ = 1.345 Mg m⁻³

Melting point = 452–453 K

Mo Kα radiation, $\lambda$ = 0.71075 Å

Cell parameters from 5108 reflections

$\theta$ = 2.6–30.6°

$\mu$ = 0.10 mm⁻¹

$T$ = 153 K

Platelet, colorless

0.30 × 0.09 × 0.06 mm

Data collection

Rigaku Saturn diffractometer

Radiation source: fine-focus sealed tube

graphite - Rigaku SHINE

Detector resolution: 14.63 pixels mm⁻¹

$\omega$ scans

Absorption correction: numerical

(NUMABS; Higashi, 1999)

$T_{\text{min}}$ = 0.985, $T_{\text{max}}$ = 0.997

1725 independent reflections

1707 reflections with $I > 2\sigma(I)$

$R_{\text{int}}$ = 0.037

$\theta_{\text{max}}$ = 27.5°, $\theta_{\text{min}}$ = 2.6°

$h$ = −10→10

$k$ = −11→11

$l$ = −22→22

16363 measured reflections

Refinement

Refinement on $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)]$ = 0.043

$wR(F^2) = 0.112$

$S$ = 1.17

1725 reflections

173 parameters

0 restraints

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$\Delta\rho_{\text{max}}$ = 0.20 e Å⁻³

$\Delta\rho_{\text{min}}$ = −0.22 e Å⁻³

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations

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between e.s.d.’s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.’s is used for estimating e.s.d.’s involving l.s. planes.

Refinement. Refinement of $F^2$ against ALL reflections. The weighted R-factor $wR$ and goodness of fit $S$ are based on $F^2$, conventional $R$-factors $R$ are based on $F$, with $F$ set to zero for negative $F^2$. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating $R$-factors($gt$) etc. and is not relevant to the choice of reflections for refinement. $R$-factors based on $F^2$ are statistically about twice as large as those based on $F$, and $R$-factors based on ALL data will be even larger.

The crystal was a weak anomalous scatterer collected with Mo Ka radiation. Friedel mates were merged (MERG 4) and the absolute configuration was not determined.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($\text{Å}^2$)

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### Geometric parameters (Å, °)

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supplementary materials

C7—O1—B1 109.67 (18) N1—C8—H8B 111.1
C9—O2—B1 110.55 (17) C7—C8—H8B 111.1
C12—O3—C13 114.9 (2) H8A—C8—H8B 109.1
C10—N1—C8 114.44 (19) O2—C9—C10 105.45 (19)
C10—N1—B1 105.44 (17) O2—C9—H9A 110.7
C8—N1—B1 103.17 (17) C10—C9—H9A 110.7
C10—N1—H1 111.1 O2—C9—H9B 110.7
C8—N1—H1 111.1 C10—C9—H9B 110.7
B1—N1—H1 111.1 H9A—C9—H9B 108.8
C2—C1—C6 116.5 (2) N1—C10—C9 104.23 (19)
C2—C1—B1 123.6 (2) N1—C10—H10A 110.9
C6—C1—B1 119.9 (2) C9—C10—H10A 110.9
C1—C2—C3 121.8 (2) N1—C10—H10B 110.9
C1—C2—H2 119.1 C9—C10—H10B 110.9
C3—C2—H2 119.1 H10A—C10—H10B 108.9
C4—C3—C2 120.7 (2) C12—C11—C4 110.81 (19)
C4—C3—H3 119.7 C12—C11—H11A 110.5
C2—C3—H3 119.7 C4—C11—H11A 109.5
C5—C4—C3 118.2 (2) C12—C11—H11B 109.5
C5—C4—C11 120.3 (2) C4—C11—H11B 109.5
C3—C4—C11 121.5 (2) H11A—C11—H11B 108.1
C4—C5—C6 121.0 (2) O4—C12—O3 123.2 (3)
C4—C5—H5 119.5 O4—C12—C11 124.8 (3)
C6—C5—H5 119.5 O3—C12—C11 112.0 (2)
C5—C6—C1 121.7 (2) O3—C13—H13A 109.5
C5—C6—H6 119.1 O3—C13—H13B 109.5
C1—C6—H6 119.1 H13A—C13—H13B 109.5
O1—C7—C8 104.28 (19) O3—C13—H13C 109.5
O1—C7—H7A 110.9 H13A—C13—H13C 109.5
C8—C7—H7A 110.9 H13B—C13—H13C 109.5
O1—C7—H7B 110.9 O1—B1—O2 112.28 (19)
C8—C7—H7B 110.9 O1—B1—C1 111.41 (18)
H7A—C7—H7B 108.9 O2—B1—C1 116.1 (2)
N1—C8—C7 103.34 (19) O1—B1—N1 101.97 (18)
N1—C8—H8A 111.1 O2—B1—N1 100.39 (17)
C7—C8—H8A 111.1 C1—B1—N1 113.35 (18)

Hydrogen-bond geometry (Å, °)

Cg3 is the centroid of the C1–C6 ring.

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Symmetry codes: (i) −x+2, y−1/2, −z+1/2; (ii) −x+2, y+1/2, −z+1/2.
Fig. 1
Fig. 3