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Structure of a push–pull olefin prepared by ynamine hydroboration with a borandiol ester

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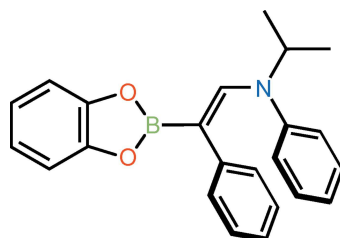
CCDC reference: 1997061

Supporting information: this article has supporting information at journals.iucr.org/e

N-[(*Z*)-2-(2*H*-1,3,2-Benzodioxaborol-2-yl)-2-phenylethenyl]-*N*-(propan-2-yl)-aniline, $C_{23}H_{22}BNO_2$, contains a $C=C$ bond that is conjugated with a donor and an acceptor group. An analysis that included similar push–pull olefins revealed that bond lengths in their $B-C=C-N$ core units correlate with the perceived acceptor and donor strength of the groups. The two phenyl groups in the molecule are rotated with respect to the plane that contains the BCCN atoms, and are close enough for significant π -stacking. Definite characterization of the title compound demonstrates, for the first time in a reliable way, that hydroboration of ynamines with borandiol esters is feasible. Compared to olefin hydroboration with borane, the ynamine substrate is activated enough to undergo reaction with the less active hydroboration reagent catecholborane.

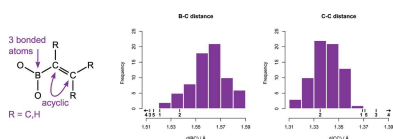
1. Chemical context

Boronic esters are frequently used to transfer organic groups to transition metals, for example in the transmetallation step of the Suzuki–Miyaura reaction. Hydroboration of ynamines with borandiol esters produces amino-functionalized boronic esters in one step and perfect atom economy.



For true ynamines, to the best of our knowledge, only two attempts of such reactions have been reported so far. These either failed (Witulski *et al.*, 2000) or were reported without reaction details and characterization data (Zhuo *et al.*, 2001). More recently it was found that the exceptionally active Pier's borane, $HB(C_6F_5)_2$, can readily hydroborate 1-propynyl-2,2,6,6-tetramethylpiperidine (Wang *et al.*, 2018). Borandiol esters are expected to be less reactive because the electron deficiency at the boron is reduced by π -donation from the oxygen atoms.

Given the limited precedent for ynamine hydroboration, the more comprehensive literature for enamine hydroboration was consulted (Goralski & Singaram, 2012; Dembitsky *et al.*, 2002), as their reactivity is expected to be controlled by similar effects. Compared to simple olefin substrates, conjugation of the $C=C$ bond with nitrogen dictates the regioselectivity and increases the reactivity of enamines. However, the presence of a nitrogen atom in the reactant and product enables the



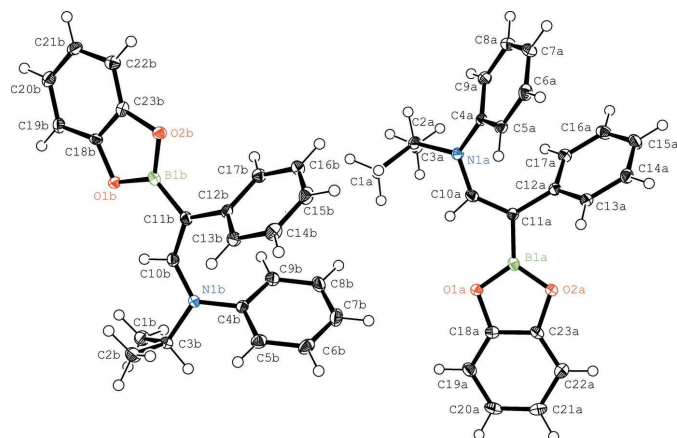


Figure 1
The molecular structures of the two independent molecules of the title compound **1** with displacement ellipsoids drawn at the 50% probability level.

formation of unreactive Lewis acid–base adducts with the hydroborating reagent. Building on the vast knowledge of the reactivity of different borane–amine adducts in hydroboration (Brown & Murray, 1984; Brown *et al.*, 1999), a bulky *iso*-propyl and an phenyl group were selected as substituents for the ynamine nitrogen. The former should weaken adducts for steric reasons, whereas the phenyl group is expected to reduce the nucleophilicity of the nitrogen by conjugation.

Ynamine hydroboration using a borandiol ester was reinvestigated and succeeded for a substrate that follows the developed design principles. The product of such a reaction contains a C=C double bond flanked by both an electron-donating group (EDG), the amine, and an electron-withdrawing group (EWG), the boronate. Therefore it belongs to the class of push–pull (captodative) olefins, which are known to have unusual properties such as weak π -bonds or biradical reactivity (Viehe *et al.*, 1985).

2. Structural commentary

The asymmetric unit contains two almost identical (*r.m.d.s* = 0.11 Å) independent molecules. As judged by the B1–C11–C10–N1 torsion angles of 171.6 (2) and 175.5 (2)°, the central C–C bond is only slightly twisted from planarity. Both phenyl groups in the molecule are rotated, by 43 (su?) and 49 (su?)°, with respect to that plane. The centroids of two phenyl groups in one molecule are on average 3.747 Å apart, which suggests intramolecular π -stacking. The mean distances are 1.521 Å for the B1–C11 bond, 1.365 Å for the C10–N1 bond and 1.369 Å for the central C10–C11 bond.

3. Supramolecular features

There is a short intermolecular contact between the boron atom and an aniline *ortho*-H of 2.856 Å ($\Sigma r_{VDW}[B,H] = 3.11$ Å). The shortest intermolecular distances between the catechol unit and boron are $B1A \cdots C21B^i = 3.540$ Å ($\Sigma r_{VDW}[B,C] = 3.68$ Å), $B1A \cdots C20B^i = 3.686$ Å,

Table 1

Comparison of bond lengths (in Å) in **1** with those in the similar compounds **2–5**.

Average distances and standard deviations are given whenever there is more than one molecule in the asymmetric unit. Typical bond lengths for vinylboranes and conjugated enamines were obtained from statistical analysis.

Compound	B–C	C–C	C–N	CCDC
1	1.521 (3)	1.369 (3)	1.365 (3)	title compound
2	1.537 (4)	1.335 (4)	1.394 (3)	861787 ^a
3	1.513 (4)	1.380 (3)	1.362 (3)	1529736
4	1.491 (7)	1.393 (6)	1.341 (6)	1843575
5	1.516 (2)	1.371 (2)	1.350 (2)	unpublished
Vinyl boronates	1.561 (15)	1.341 (12)	–	–
Enamines	–	~1.34	~1.36	–

Notes: (a) Hatayama & Okuno (2012); (b) Liu *et al.* (2017); (c) Wang *et al.* (2018).

$B1A \cdots H21B^i = 3.113$ Å and $B1A \cdots H20B^i = 3.381$ Å [symmetry code: (i) $\frac{1}{2} + x, 1 - y, z$]. In addition there is a short contact between one of the other catechol hydrogen atoms and the *meta*-carbon of the aniline, $H22A \cdots C6B(x, 1 + y, z)$ of 2.877 Å ($\Sigma r_{VDW}[C,H] = 2.97$ Å). All of these interactions involve atoms that are part of arenes and could be seen as intermolecular π -stacking.

Methyl hydrogen atoms of the isopropyl group are at van der Waals distances with one of the oxygen atoms (2.698 Å, $\Sigma r_{VDW}[O,H] = 2.70$ Å) and with one of the anilic *meta*-H atoms (2.388 Å, $\Sigma r_{VDW}[H,H] = 2.40$ Å). The nitrogen atom is sterically shielded by surrounding groups and does not have any close intermolecular neighbours.

4. Database survey

Contributions from the zwitterionic resonance structure $^-B=C-C=N^+$ are expected to increase with donor and acceptor group strength. This should be observable as a shortening of the B–C and C–N bonds and an elongation of the C–C bond. Following this idea, bond lengths of **1** were compared to those in the structurally related compounds **2–5** (Table 1, Fig. 2). C–N bond lengths are 1.341 Å (**4**), 1.350 Å (**5**), 1.362 Å (**3**), 1.365 Å (**1**), 1.394 Å (**2**). These numbers follow the expected N-donor strength, when the latter is

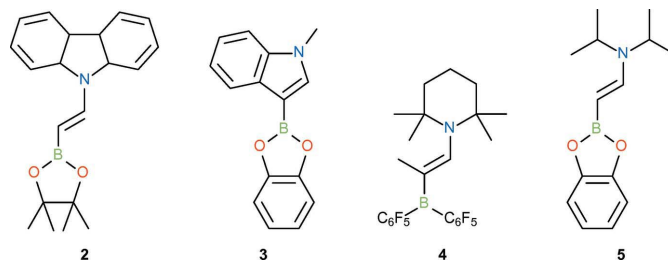


Figure 2

Chemical structure of reference compounds. 9-[(*E*)-2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)ethenyl]-9H-carbazole, **2** (Hatayama & Okuno, 2012), 3-(2H-1,3,2-benzodioxaborol-2-yl)-1-methyl-1H-indole, **3** (Liu *et al.*, 2017), 1-[2-bis(pentafluorophenyl)boranyl]prop-1-en-1-yl]-2,2,6,6-tetramethylpiperidine, **4** (Wang *et al.*, 2018) and N-[(*Z*)-2-(2H-1,3,2-benzodioxaborol-2-yl)-2-phenylethenyl]-N-(propan-2-yl)propan-2-amine, **5** (unpublished).

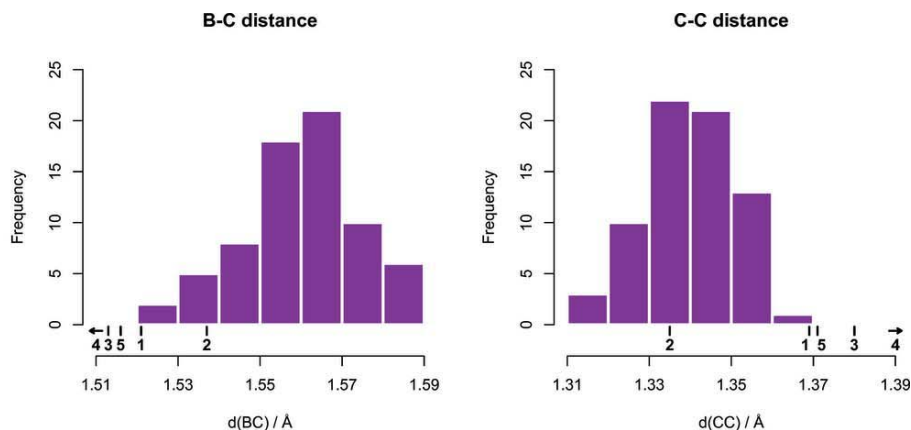
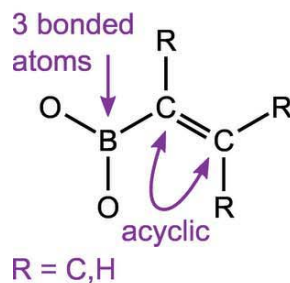


Figure 3

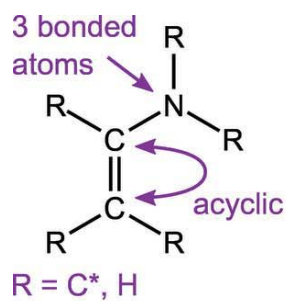
Statistical analysis of B–C and C–C bond lengths in vinyl boranes. The query substructure and restrictions are shown on the left. Seven problematic or irrelevant structures were removed. The bond distances of reference compounds are marked.

estimated by the number of conjugating substituents on the nitrogen: piperidine, diisopropyl > aniline > indole, carbazole. B–C lengths are 1.491 Å (**4**), 1.513 Å (**3**), 1.516 Å (**5**), 1.521 Å (**1**), 1.537 Å (**2**). Similarly, these numbers follow the B-acceptor strength: B(C₆F₅)₂ > catecholboron > pinacolboron (Adamczyk-Woźniak *et al.*, 2011). Following this, the zwitterionic resonance structure is most important in **4**, which has a strong donor and a strong acceptor. On the other end of the scale lies **2**, which has a weak donor and a weak acceptor. The other molecules, including **1**, lie between these two extremes.

In order to compare with olefins that either have a donor or an acceptor group, the Cambridge Structural Database (CSD, Version 5.41, update of November 2019; Groom *et al.*, 2016) was searched for vinyl boronates and enamines. Bond-length distributions and the exact query structures are shown in Fig. 3 and Fig. 4. The data set for vinyl boronates consists of about 90% of pinacol boronates and contains only a few catechol boronates. Compared with typical bond lengths in this data set, the B–C bond is shorter and the C=C bond is longer in **1–5**, which indicates stronger conjugation. The only exception

is **2**, whose C=C bond is shorter, most likely because it is part of the indole.

For enamines the C–C bond length has an average of 1.341 Å, which is about 0.025 Å longer than the value of 1.316 (15) Å for regular internal olefins (Allen *et al.*, 2006). In **1**, **3**, **4** and **5**, the C–C bonds are on average 1.378 Å, and thereby longer than in enamines. C–N bond lengths for enamines are distributed more uniformly than C–C lengths. Inspection of the structures in which C–N distances are longer than 1.39 Å revealed that these structures typically either have a nitrogen whose lone pair is not coplanar with the C=C bond, or a nitrogen that is part of a carbazole or morpholine. As conjugation with the formal double bond between C10 and C11 is absent or short in these, only structures with C–N bond lengths below 1.39 Å were used for comparison. The average C–N bond length of about 1.36 Å for that subset is similar to the C–N bond lengths in **1**, **3**, **4** and **5**. Overall, comparison with enamines reveals that C–C bonds are longer in push–pull olefins, whereas C–N bond lengths are unaffected. This suggests that conjugation with the boron



C*: carbon atom that is not bound to any atom by a double or triple bond

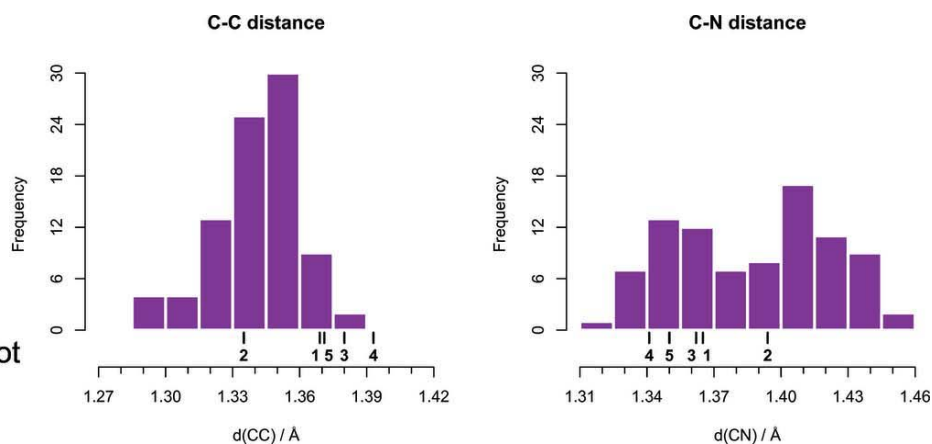


Figure 4

Statistical analysis of C–C and C–N bond lengths in enamines. The query substructure and restrictions are shown on the left. Problematic or irrelevant structures were removed. The bond distances of reference compounds are marked.

affects the C–C bond length more than the C–N bond length.

5. Synthesis and crystallization

The title compound was prepared by the multi-step sequence shown in Fig. 5.

***N*-isopropyl-*N*-(phenylethynyl)aniline:** In a 100 ml Schlenk flask, 5.8 ml of *N*-isopropyl amine (40 mmol, 1.0 eq.) were diluted with 40 ml of dry THF. 24.7 ml of an *n*-BuLi solution in hexanes (1.62 mol l⁻¹, 40 mmol, 1.0 eq.) were added over 5 min at 195 K. A colourless solid formed and after 15 min the suspension was warmed to room temperature over 30 min. Upon addition of 5.69 g of 2-chloroethynylbenzene (96%, 40 mmol, 1.0 eq., prepared according to Li *et al.*, 2014), the reaction mixture turned black. The sealed Schlenk flask was heated in an oil bath at 338 K (caution: the closed flask may burst if this temperature is exceeded). The reaction progress was monitored by GC–FID. After 6 h the reaction mixture was cooled to room temperature. 100 ml of *tert*-butyl methyl ether were added, the organic phase washed with ice-cold water (3 × 50 ml), dried with MgSO₄ and concentrated on the rotavap. 8.25 g of black viscous liquid were obtained and purified by Kugelrohr distillation (433 K, 0.2 mbar) to yield 6.34 g (purity 83 wt%, yield 56%) of the colourless liquid *N*-isopropyl-*N*-(phenylethynyl)aniline. ¹H NMR (400 MHz, CDCl₃): δ (ppm) = 1.40 (*d*, 6.5 Hz, 6H, 2 × CH₃), 4.10 (*hept*, 6.4 Hz, 1H, CH of *i*-Pr), 6.92 (*tt*, 7.4 Hz, 1.2 Hz, 1H, *para*-H of aniline), 7.16–7.33 (*m*, 7H, arene H), 7.38–7.41 (*m*, 2H, *ortho*-H of phenyl group). ¹³C{¹H} NMR (100 MHz, CDCl₃): δ (ppm) = 20.6 (*s*, 2 × CH₃), 49.6 (*s*, CH of *i*-Pr), 72.0 (*s*, alkynic carbon farther from N), 86.4 (*s*, alkynic carbon closer to N), 115.5 (*s*, *ortho*-C of aniline), 120.8 (*s*, *para*-C of aniline), 125.2 (*s*, *ipso*-C of phenyl group), 126.2 (*s*, *para*-C of phenyl group), 128.4 (*s*, *meta*-C of phenyl group), 129.3 (*s*, *meta*-C of aniline), 130.2 (*s*, *ortho*-C of phenyl group), 144.4 (*s*, *ipso*-C of aniline). EI–MS (70 eV) *m/z* = 236, 235 (*M*⁺), 220 (*M*⁺ – CH₃), 194, 193, 192 (*M*⁺ – C₃H₇), 165, 117, 115, 90, 89, 77, 63, 51, 43. ATR–IR ν (cm⁻¹)(%T) = 534 (70), 629 (61), 688 (22), 745 (18), 783 (82), 865 (87), 881 (87), 904 (87), 996 (80), 1025 (73), 1056 (66), 1129 (70), 1147 (52), 1170 (67), 1254 (41), 1312 (67), 1348 (77), 1367 (76), 1396 (51), 1490 (38), 1594 (46), 1932 (96), 2217 (38, C≡C-stretch), 2872 (94), 2932 (91), 2976 (80), 3034 (93), 3057 (93).

***N*-[(*Z*)-2-(2*H*-1,3,2-benzodioxaborol-2-yl)-2-phenylethenyl]-*N*-(propan-2-yl)aniline:** Under a counterflow of argon, 3.08 g of *N*-isopropyl-*N*-(phenylethynyl)aniline (13.1 mmol, 1.0 eq.) were placed in an oven-dried 20 ml Schlenk flask with a Young valve. The flask and its contents were purged three times by applying high vacuum followed by flushing with argon. 6.5 ml dry *tert*-butyl methyl ether were added and the mixture stirred vigorously to ensure mixing of the two liquids.

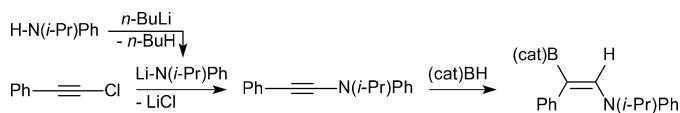


Figure 5

Reaction sequence used for the synthesis of the title compound.

Table 2

Experimental details.

Crystal data			
Chemical formula	C ₂₃ H ₂₂ BNO ₂		
<i>M</i> _r	355.22		
Crystal system, space group	Orthorhombic, <i>Pca</i> ₂₁		
Temperature (K)	100		
<i>a</i> , <i>b</i> , <i>c</i> (Å)	17.8540 (11), 11.5361 (6), 18.5366 (12)		
<i>V</i> (Å ³)	3817.9 (4)		
<i>Z</i>	8		
Radiation type	Mo <i>K</i> α		
μ (mm ⁻¹)	0.08		
Crystal size (mm)	0.38 × 0.2 × 0.07		
Data collection			
Diffractometer	Bruker–Nonius Kappa APEXII		
Absorption correction	Multi-scan (<i>SADABS</i> ; Bruker, 2015)		
<i>T</i> _{min} , <i>T</i> _{max}	0.703, 0.733		
No. of measured, independent and observed [<i>I</i> > 2σ(<i>I</i>)] reflections	60829, 8781, 7379		
<i>R</i> _{int}	0.047		
(sin θ/λ) _{max} (Å ⁻¹)	0.652		
Refinement			
<i>R</i> [[<i>F</i> ² > 2σ(<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.039, 0.086, 1.03		
No. of reflections	8781		
No. of parameters	491		
No. of restraints	1		
H-atom treatment	H-atom parameters constrained		
$\Delta\rho_{\max}$, $\Delta\rho_{\min}$ (e Å ⁻³)	0.25, -0.24		

Computer programs: *APEX2* and *SAINT* (Bruker, 2015), *SHELXT* (Sheldrick, 2015a), *SHELXL* (Sheldrick, 2015b) and *OLEX2* (Dolomanov *et al.*, 2009).

2.1 ml of catecholborane (19.5 mmol, 1.5 eq.) were added, the flask closed, and the reaction mixture heated to 323 K for 16 h. Cooling to room temperature led to the precipitation of the product. The supernatant was removed and the precipitate recrystallized from 40 ml of *tert*-butyl ether. X-ray quality crystals were obtained in a yield of 1.88g (40%). Notes: (*a*) Schlenk techniques are necessary because ynamines and catecholborane are moisture-sensitive; (*b*) the reaction also works well in diethyl ether, 1,4-dioxane or THF.

¹H NMR (600 MHz, CDCl₃): δ (ppm) = 1.34 (*d*, 6.8 Hz, 6H, CH₃ of *i*-Pr), 3.89 (*hept*, 6.8 Hz, 1H, CH of *i*-Pr), 6.77–6.80 (*m*, 2H, H5 & H9), 6.81–6.85 (*m*, 2H, H7 & H15), 6.85–6.92 (*m*, 6H, H6 & H8 & H13 & H14 & H16 & H17), 6.95–6.98 (*m*, 2H, 2 × catechol-H), 7.11–7.14 (*m*, 2H, 2 × catechol-H), 7.53 (*s*, 1H, H10).

¹³C{¹H} NMR (151 MHz, CDCl₃): δ (ppm) = 22.2 (*s*, C1 & C2), 57.4 (*s*, C3), 98.1 (*br s*, C11), 111.7 (*s*, 2 × catechol-C), 121.8 (*s*, 2 × catechol-C), 124.3 (*s*, C15), 124.6 (*s*, C7), 126.3 (*s*, C5 & C9), 126.9 (*s*, C14 & C16), 128.0 (*s*, C6 & C8), 129.4 (*s*, C13 & C17), 138.8 (*s*, C12), 143.3 (*s*, C4), 144.9 (*s*, C10), 148.9 (*s*, C18 & C23). Inverse gated [¹³C{¹H}] NMR with *D*₁ = 60 s measured to get integrable ¹³C NMR.

¹¹B NMR (160 MHz, CDCl₃): δ (ppm) = 33.2 (*s*).

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. H atoms were positioned geometrically and refined as riding: C–H = 0.95–0.98 Å and *U*_{iso}(H)

= $1.2U_{\text{eq}}(\text{C})$ or $1.5U_{\text{eq}}(\text{C-methyl})$. The absolute structure was not determined because of unreliable Flack and Hooft parameters.

Acknowledgements

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1 supporting information

2 Structure of a push–pull olefin prepared by ynamine hydroboration with a
3 borandiol ester

4 Joël Gubler and Peter Chen*

5 Computing details

6 Data collection: *APEX2* (Bruker, 2015); cell refinement: *S SAINT* (Bruker, 2015); data reduction: *S SAINT* (Bruker, 2015);
7 program(s) used to solve structure: *SHELXT* (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL* (Sheldrick,
8 2015b); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *OLEX2*
9 (Dolomanov *et al.*, 2009).

10 *N*-[(*Z*)-2-(2*H*-1,3,2-Benzodioxaborol-2-yl)-2-phenylethenyl]-*N*-(propan-2-yl)aniline11 *Crystal data*12 $C_{23}H_{22}BNO_2$ 13 $M_r = 355.22$ 14 Orthorhombic, *Pca*2₁15 $a = 17.8540$ (11) Å16 $b = 11.5361$ (6) Å17 $c = 18.5366$ (12) Å18 $V = 3817.9$ (4) Å³19 $Z = 8$ 20 $F(000) = 1504$ $D_x = 1.236$ Mg m⁻³Mo *K*α radiation, $\lambda = 0.71073$ Å

Cell parameters from 9981 reflections

 $\theta = 2.5$ – 26.8° $\mu = 0.08$ mm⁻¹ $T = 100$ K

Plate, clear light yellow

0.38 × 0.2 × 0.07 mm

21 *Data collection*22 Bruker–Nonius Kappa APEXII
diffractometer

23 Radiation source: sealed tube

24 Graphite monochromator

25 Detector resolution: 8.33 pixels mm⁻¹26 ω and φ scans27 Absorption correction: multi-scan
(*SADABS*; Bruker, 2015)28 $T_{\min} = 0.703$, $T_{\max} = 0.733$

60829 measured reflections

8781 independent reflections

7379 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.047$ $\theta_{\max} = 27.6^\circ$, $\theta_{\min} = 2.8^\circ$ $h = -23 \rightarrow 23$ $k = -14 \rightarrow 14$ $l = -24 \rightarrow 24$ 29 *Refinement*30 Refinement on F^2

31 Least-squares matrix: full

32 $R[F^2 > 2\sigma(F^2)] = 0.039$ 33 $wR(F^2) = 0.086$ 34 $S = 1.03$

35 8781 reflections

36 491 parameters

37 1 restraint

38 Primary atom site location: structure-invariant
direct methodsHydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2) + (0.0401P)^2 + 0.7053P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} < 0.001$ $\Delta\rho_{\max} = 0.25$ e Å⁻³ $\Delta\rho_{\min} = -0.24$ e Å⁻³

Absolute structure: Flack x determined using
3185 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons,
Flack and Wagner, Acta Cryst. B69 (2013)
249-259).

Absolute structure parameter: -0.7 (4)

39 *Special details*

40 **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 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.

41 **Refinement.** 8 reflections were omitted (some are equivalents). These were checked visually and are all results of high background around the beamstop (beginning ice formation or crystalline powder covering the sample). 0 1 0 is clearly shadowed by the beamstop. Absolute structure is not claimed due to unreliable Flack and Hooft parameters.

42 *Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)*

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
44 O1B	0.18188 (9)	0.59842 (13)	0.60028 (9)	0.0155 (4)
45 O2B	0.30086 (9)	0.67252 (13)	0.61169 (9)	0.0158 (4)
46 N1B	0.23745 (11)	0.25154 (16)	0.65127 (11)	0.0159 (4)
47 C1B	0.10503 (15)	0.2002 (2)	0.62440 (14)	0.0210 (6)
48 H1BA	0.0874	0.2806	0.6251	0.032*
49 H1BB	0.0642	0.1485	0.6390	0.032*
50 H1BC	0.1215	0.1800	0.5756	0.032*
51 C2B	0.14877 (15)	0.2209 (2)	0.75303 (14)	0.0205 (6)
52 H2BA	0.1928	0.2152	0.7844	0.031*
53 H2BB	0.1096	0.1686	0.7707	0.031*
54 H2BC	0.1301	0.3008	0.7533	0.031*
55 C3B	0.17032 (14)	0.1869 (2)	0.67659 (13)	0.0160 (5)
56 H3B	0.1840	0.1028	0.6776	0.019*
57 C4B	0.30570 (13)	0.18933 (19)	0.64199 (14)	0.0147 (5)
58 C5B	0.32986 (14)	0.1121 (2)	0.69452 (14)	0.0189 (5)
59 H5B	0.3009	0.1007	0.7369	0.023*
60 C6B	0.39606 (14)	0.0518 (2)	0.68521 (16)	0.0230 (6)
61 H6B	0.4126	-0.0006	0.7214	0.028*
62 C7B	0.43822 (15)	0.0673 (2)	0.62365 (16)	0.0275 (6)
63 H7B	0.4841	0.0267	0.6177	0.033*
64 C8B	0.41341 (15)	0.1423 (2)	0.57066 (15)	0.0238 (6)
65 H8B	0.4420	0.1522	0.5279	0.029*
66 C9B	0.34704 (14)	0.2033 (2)	0.57938 (14)	0.0191 (5)
67 H9B	0.3301	0.2543	0.5426	0.023*
68 C10B	0.23179 (13)	0.36795 (19)	0.63924 (13)	0.0151 (5)
69 H10B	0.1820	0.3949	0.6321	0.018*
70 C11B	0.28538 (13)	0.4526 (2)	0.63563 (13)	0.0156 (5)
71 C12B	0.36555 (13)	0.4370 (2)	0.65557 (13)	0.0142 (5)
72 C13B	0.38562 (14)	0.3878 (2)	0.72178 (13)	0.0172 (5)
73 H13B	0.3476	0.3630	0.7541	0.021*
74 C14B	0.46041 (15)	0.3748 (2)	0.74087 (14)	0.0209 (5)

75	H14B	0.4731	0.3410	0.7860	0.025*
76	C15B	0.51658 (14)	0.4110 (2)	0.69433 (14)	0.0210 (6)
77	H15B	0.5677	0.4012	0.7071	0.025*
78	C16B	0.49740 (14)	0.4614 (2)	0.62906 (14)	0.0188 (5)
79	H16B	0.5356	0.4869	0.5972	0.023*
80	C17B	0.42288 (14)	0.4749 (2)	0.60995 (14)	0.0169 (5)
81	H17B	0.4106	0.5103	0.5652	0.020*
82	C18B	0.18063 (14)	0.71629 (19)	0.58605 (13)	0.0140 (5)
83	C19B	0.12056 (14)	0.7829 (2)	0.56544 (13)	0.0178 (5)
84	H19B	0.0715	0.7518	0.5614	0.021*
85	C20B	0.13606 (15)	0.8997 (2)	0.55079 (14)	0.0198 (5)
86	H20B	0.0966	0.9492	0.5356	0.024*
87	C21B	0.20801 (15)	0.9445 (2)	0.55794 (13)	0.0192 (5)
88	H21B	0.2165	1.0242	0.5478	0.023*
89	C22B	0.26793 (14)	0.8754 (2)	0.57958 (13)	0.0179 (5)
90	H22B	0.3171	0.9059	0.5849	0.022*
91	C23B	0.25211 (13)	0.76087 (19)	0.59287 (13)	0.0151 (5)
92	B1B	0.25681 (15)	0.5724 (2)	0.61526 (14)	0.0149 (6)
93	O1A	0.59813 (9)	-0.09865 (13)	0.40292 (10)	0.0176 (4)
94	O2A	0.71747 (9)	-0.17253 (13)	0.39845 (9)	0.0174 (4)
95	N1A	0.65302 (11)	0.24387 (17)	0.34366 (11)	0.0161 (4)
96	C1A	0.52189 (14)	0.2942 (2)	0.37802 (15)	0.0228 (6)
97	H1AA	0.5416	0.3104	0.4263	0.034*
98	H1AB	0.5027	0.2146	0.3764	0.034*
99	H1AC	0.4813	0.3485	0.3671	0.034*
100	C2A	0.58421 (14)	0.3081 (2)	0.32256 (14)	0.0177 (5)
101	H2A	0.5976	0.3923	0.3213	0.021*
102	C3A	0.55869 (14)	0.2754 (2)	0.24731 (15)	0.0227 (6)
103	H3AA	0.5407	0.1952	0.2473	0.034*
104	H3AB	0.6008	0.2829	0.2137	0.034*
105	H3AC	0.5180	0.3272	0.2323	0.034*
106	C4A	0.72025 (13)	0.3083 (2)	0.35237 (13)	0.0139 (5)
107	C5A	0.76423 (13)	0.2910 (2)	0.41332 (13)	0.0150 (5)
108	H5A	0.7496	0.2358	0.4487	0.018*
109	C6A	0.82952 (14)	0.3543 (2)	0.42245 (14)	0.0199 (5)
110	H6A	0.8596	0.3423	0.4640	0.024*
111	C7A	0.85112 (15)	0.4353 (2)	0.37098 (15)	0.0233 (6)
112	H7A	0.8964	0.4776	0.3768	0.028*
113	C8A	0.80630 (14)	0.4541 (2)	0.31124 (16)	0.0227 (6)
114	H8A	0.8204	0.5105	0.2765	0.027*
115	C9A	0.74113 (14)	0.3912 (2)	0.30178 (14)	0.0182 (5)
116	H9A	0.7106	0.4047	0.2607	0.022*
117	C10A	0.64818 (14)	0.1276 (2)	0.35658 (13)	0.0157 (5)
118	H10A	0.5984	0.1001	0.3624	0.019*
119	C11A	0.70180 (13)	0.0433 (2)	0.36266 (12)	0.0136 (5)
120	C12A	0.78275 (13)	0.0582 (2)	0.34518 (13)	0.0143 (5)
121	C13A	0.83799 (13)	0.0207 (2)	0.39292 (13)	0.0159 (5)
122	H13A	0.8239	-0.0154	0.4369	0.019*

123	C14A	0.91359 (14)	0.0353 (2)	0.37700 (15)	0.0184 (5)
124	H14A	0.9505	0.0104	0.4105	0.022*
125	C15A	0.93517 (15)	0.0860 (2)	0.31277 (14)	0.0209 (6)
126	H15A	0.9868	0.0967	0.3021	0.025*
127	C16A	0.88076 (14)	0.1212 (2)	0.26390 (14)	0.0209 (6)
128	H16A	0.8952	0.1549	0.2193	0.025*
129	C17A	0.80555 (14)	0.1074 (2)	0.27994 (13)	0.0166 (5)
130	H17A	0.7689	0.1317	0.2460	0.020*
131	C18A	0.59638 (14)	-0.2147 (2)	0.42114 (13)	0.0155 (5)
132	C19A	0.53579 (14)	-0.2820 (2)	0.43916 (14)	0.0196 (5)
133	H19A	0.4864	-0.2514	0.4402	0.024*
134	C20A	0.55064 (15)	-0.3974 (2)	0.45574 (14)	0.0212 (6)
135	H20A	0.5102	-0.4469	0.4685	0.025*
136	C21A	0.62240 (15)	-0.4420 (2)	0.45425 (14)	0.0221 (6)
137	H21A	0.6302	-0.5212	0.4661	0.027*
138	C22A	0.68384 (15)	-0.3727 (2)	0.43561 (14)	0.0215 (6)
139	H22A	0.7335	-0.4024	0.4346	0.026*
140	C23A	0.66814 (14)	-0.2591 (2)	0.41882 (13)	0.0159 (5)
141	B1A	0.67343 (15)	-0.0740 (2)	0.38806 (15)	0.0149 (5)

142 *Atomic displacement parameters (\AA^2)*

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}	
144	O1B	0.0139 (8)	0.0119 (8)	0.0206 (9)	0.0005 (7)	0.0001 (7)	0.0023 (7)
145	O2B	0.0137 (8)	0.0144 (8)	0.0194 (9)	-0.0010 (6)	-0.0018 (7)	0.0016 (7)
146	N1B	0.0115 (10)	0.0142 (9)	0.0218 (11)	0.0004 (8)	0.0030 (8)	0.0028 (8)
147	C1B	0.0183 (13)	0.0203 (12)	0.0245 (15)	-0.0017 (10)	-0.0016 (11)	-0.0009 (11)
148	C2B	0.0194 (14)	0.0215 (13)	0.0206 (13)	-0.0021 (11)	0.0031 (11)	0.0012 (10)
149	C3B	0.0142 (13)	0.0135 (11)	0.0205 (13)	-0.0017 (10)	0.0030 (10)	0.0013 (10)
150	C4B	0.0115 (12)	0.0112 (10)	0.0213 (13)	-0.0010 (9)	-0.0012 (10)	-0.0023 (10)
151	C5B	0.0187 (13)	0.0162 (12)	0.0218 (14)	-0.0021 (10)	-0.0029 (10)	0.0006 (10)
152	C6B	0.0174 (13)	0.0157 (12)	0.0360 (16)	0.0007 (10)	-0.0082 (12)	0.0023 (11)
153	C7B	0.0144 (13)	0.0196 (12)	0.0487 (19)	0.0027 (11)	0.0015 (13)	-0.0046 (12)
154	C8B	0.0196 (14)	0.0191 (13)	0.0328 (16)	-0.0020 (11)	0.0108 (11)	-0.0065 (11)
155	C9B	0.0173 (13)	0.0168 (12)	0.0232 (13)	-0.0015 (10)	0.0015 (10)	-0.0014 (10)
156	C10B	0.0132 (12)	0.0174 (11)	0.0147 (11)	0.0022 (9)	0.0007 (9)	0.0013 (10)
157	C11B	0.0146 (12)	0.0169 (11)	0.0152 (12)	0.0014 (10)	0.0027 (10)	0.0006 (10)
158	C12B	0.0133 (12)	0.0120 (11)	0.0173 (12)	0.0018 (9)	0.0002 (10)	-0.0012 (10)
159	C13B	0.0182 (13)	0.0161 (12)	0.0172 (13)	0.0000 (10)	0.0021 (10)	-0.0014 (10)
160	C14B	0.0217 (14)	0.0217 (13)	0.0195 (13)	0.0023 (11)	-0.0053 (11)	0.0007 (11)
161	C15B	0.0143 (13)	0.0208 (12)	0.0281 (15)	0.0023 (10)	-0.0057 (11)	-0.0022 (11)
162	C16B	0.0144 (12)	0.0178 (12)	0.0244 (14)	-0.0007 (10)	0.0032 (10)	-0.0005 (10)
163	C17B	0.0177 (12)	0.0163 (11)	0.0169 (12)	0.0007 (10)	0.0008 (10)	0.0008 (10)
164	C18B	0.0171 (12)	0.0124 (11)	0.0124 (11)	-0.0024 (10)	0.0031 (9)	0.0000 (9)
165	C19B	0.0140 (13)	0.0202 (13)	0.0191 (13)	0.0031 (10)	0.0011 (10)	0.0001 (10)
166	C20B	0.0236 (14)	0.0186 (12)	0.0171 (13)	0.0061 (11)	0.0014 (11)	0.0014 (10)
167	C21B	0.0277 (14)	0.0125 (11)	0.0172 (13)	0.0003 (11)	0.0044 (11)	-0.0002 (10)
168	C22B	0.0217 (13)	0.0174 (11)	0.0147 (12)	-0.0032 (10)	0.0000 (10)	-0.0025 (9)

169	C23B	0.0158 (12)	0.0167 (11)	0.0127 (11)	0.0028 (10)	-0.0025 (9)	-0.0023 (10)
170	B1B	0.0128 (13)	0.0191 (13)	0.0129 (13)	-0.0004 (11)	0.0016 (11)	-0.0006 (11)
171	O1A	0.0134 (9)	0.0138 (8)	0.0258 (10)	-0.0012 (7)	0.0003 (7)	0.0027 (7)
172	O2A	0.0139 (9)	0.0146 (8)	0.0237 (10)	-0.0005 (7)	0.0028 (7)	0.0023 (7)
173	N1A	0.0116 (10)	0.0147 (10)	0.0220 (11)	0.0002 (8)	-0.0036 (8)	0.0030 (8)
174	C1A	0.0161 (13)	0.0221 (13)	0.0304 (15)	0.0042 (11)	0.0001 (11)	0.0005 (12)
175	C2A	0.0128 (12)	0.0150 (11)	0.0251 (14)	0.0015 (10)	-0.0032 (10)	0.0033 (10)
176	C3A	0.0154 (14)	0.0273 (14)	0.0254 (14)	0.0027 (11)	-0.0061 (11)	0.0047 (11)
177	C4A	0.0121 (12)	0.0119 (11)	0.0175 (12)	0.0009 (9)	0.0005 (10)	-0.0018 (9)
178	C5A	0.0157 (12)	0.0142 (10)	0.0151 (12)	0.0010 (9)	0.0010 (10)	-0.0004 (9)
179	C6A	0.0160 (12)	0.0214 (12)	0.0223 (14)	0.0021 (10)	-0.0038 (10)	-0.0035 (11)
180	C7A	0.0155 (13)	0.0179 (12)	0.0366 (16)	-0.0054 (11)	0.0001 (12)	-0.0006 (11)
181	C8A	0.0198 (14)	0.0182 (12)	0.0300 (15)	-0.0014 (11)	0.0034 (11)	0.0066 (11)
182	C9A	0.0175 (13)	0.0173 (12)	0.0198 (13)	0.0018 (10)	-0.0009 (10)	0.0033 (10)
183	C10A	0.0129 (12)	0.0171 (12)	0.0172 (12)	-0.0036 (10)	-0.0012 (9)	0.0010 (10)
184	C11A	0.0125 (12)	0.0146 (11)	0.0138 (12)	0.0000 (9)	-0.0014 (9)	0.0005 (9)
185	C12A	0.0143 (12)	0.0123 (11)	0.0164 (12)	-0.0009 (9)	0.0007 (10)	-0.0042 (10)
186	C13A	0.0162 (12)	0.0143 (11)	0.0170 (13)	0.0002 (10)	0.0011 (10)	0.0002 (10)
187	C14A	0.0140 (12)	0.0166 (12)	0.0247 (14)	0.0000 (10)	-0.0032 (10)	-0.0033 (10)
188	C15A	0.0152 (13)	0.0220 (12)	0.0255 (14)	-0.0017 (10)	0.0056 (11)	-0.0028 (11)
189	C16A	0.0229 (14)	0.0215 (13)	0.0182 (13)	-0.0034 (11)	0.0064 (11)	0.0000 (11)
190	C17A	0.0165 (12)	0.0167 (12)	0.0166 (12)	-0.0003 (10)	-0.0014 (10)	-0.0008 (10)
191	C18A	0.0185 (13)	0.0154 (11)	0.0127 (12)	-0.0018 (10)	-0.0012 (9)	0.0004 (9)
192	C19A	0.0151 (13)	0.0228 (13)	0.0211 (13)	-0.0022 (11)	-0.0002 (10)	0.0028 (11)
193	C20A	0.0236 (14)	0.0209 (13)	0.0190 (13)	-0.0103 (11)	-0.0021 (11)	0.0036 (10)
194	C21A	0.0294 (15)	0.0155 (12)	0.0215 (13)	-0.0025 (11)	-0.0005 (11)	0.0047 (10)
195	C22A	0.0216 (14)	0.0191 (12)	0.0237 (14)	0.0030 (11)	-0.0002 (11)	0.0019 (11)
196	C23A	0.0137 (12)	0.0178 (12)	0.0164 (12)	-0.0037 (10)	0.0009 (10)	0.0009 (10)
197	B1A	0.0126 (13)	0.0177 (13)	0.0143 (13)	-0.0006 (11)	0.0002 (11)	-0.0026 (11)

198 *Geometric parameters (Å, °)*

199	O1B—C18B	1.385 (3)	O1A—C18A	1.382 (3)
200	O1B—B1B	1.399 (3)	O1A—B1A	1.402 (3)
201	O2B—C23B	1.385 (3)	O2A—C23A	1.384 (3)
202	O2B—B1B	1.398 (3)	O2A—B1A	1.396 (3)
203	N1B—C3B	1.488 (3)	N1A—C2A	1.487 (3)
204	N1B—C4B	1.425 (3)	N1A—C4A	1.421 (3)
205	N1B—C10B	1.365 (3)	N1A—C10A	1.365 (3)
206	C1B—H1BA	0.9800	C1A—H1AA	0.9800
207	C1B—H1BB	0.9800	C1A—H1AB	0.9800
208	C1B—H1BC	0.9800	C1A—H1AC	0.9800
209	C1B—C3B	1.523 (4)	C1A—C2A	1.523 (4)
210	C2B—H2BA	0.9800	C2A—H2A	1.0000
211	C2B—H2BB	0.9800	C2A—C3A	1.515 (4)
212	C2B—H2BC	0.9800	C3A—H3AA	0.9800
213	C2B—C3B	1.520 (3)	C3A—H3AB	0.9800
214	C3B—H3B	1.0000	C3A—H3AC	0.9800

215	C4B—C5B	1.389 (3)	C4A—C5A	1.390 (3)
216	C4B—C9B	1.385 (4)	C4A—C9A	1.390 (3)
217	C5B—H5B	0.9500	C5A—H5A	0.9500
218	C5B—C6B	1.382 (3)	C5A—C6A	1.386 (3)
219	C6B—H6B	0.9500	C6A—H6A	0.9500
220	C6B—C7B	1.379 (4)	C6A—C7A	1.390 (4)
221	C7B—H7B	0.9500	C7A—H7A	0.9500
222	C7B—C8B	1.382 (4)	C7A—C8A	1.383 (4)
223	C8B—H8B	0.9500	C8A—H8A	0.9500
224	C8B—C9B	1.387 (4)	C8A—C9A	1.382 (4)
225	C9B—H9B	0.9500	C9A—H9A	0.9500
226	C10B—H10B	0.9500	C10A—H10A	0.9500
227	C10B—C11B	1.369 (3)	C10A—C11A	1.370 (3)
228	C11B—C12B	1.489 (3)	C11A—C12A	1.491 (3)
229	C11B—B1B	1.521 (4)	C11A—B1A	1.519 (3)
230	C12B—C13B	1.399 (3)	C12A—C13A	1.394 (3)
231	C12B—C17B	1.398 (3)	C12A—C17A	1.397 (3)
232	C13B—H13B	0.9500	C13A—H13A	0.9500
233	C13B—C14B	1.389 (4)	C13A—C14A	1.392 (3)
234	C14B—H14B	0.9500	C14A—H14A	0.9500
235	C14B—C15B	1.387 (4)	C14A—C15A	1.381 (4)
236	C15B—H15B	0.9500	C15A—H15A	0.9500
237	C15B—C16B	1.385 (4)	C15A—C16A	1.389 (4)
238	C16B—H16B	0.9500	C16A—H16A	0.9500
239	C16B—C17B	1.386 (3)	C16A—C17A	1.385 (3)
240	C17B—H17B	0.9500	C17A—H17A	0.9500
241	C18B—C19B	1.374 (3)	C18A—C19A	1.372 (3)
242	C18B—C23B	1.382 (3)	C18A—C23A	1.381 (3)
243	C19B—H19B	0.9500	C19A—H19A	0.9500
244	C19B—C20B	1.401 (3)	C20A—H20A	0.9500
245	C20B—H20B	0.9500	C20A—C21A	1.381 (4)
246	C20B—C21B	1.391 (4)	C21A—H21A	0.9500
247	C21B—H21B	0.9500	C21A—C22A	1.401 (4)
248	C21B—C22B	1.393 (4)	C22A—H22A	0.9500
249	C22B—H22B	0.9500	C22A—C23A	1.375 (3)
250	C22B—C23B	1.374 (3)		
251				
252	C18B—O1B—B1B	105.28 (18)	C18A—O1A—B1A	105.46 (18)
253	C23B—O2B—B1B	105.43 (18)	C23A—O2A—B1A	105.50 (18)
254	C4B—N1B—C3B	118.34 (18)	C4A—N1A—C2A	117.83 (18)
255	C10B—N1B—C3B	119.02 (19)	C10A—N1A—C2A	118.9 (2)
256	C10B—N1B—C4B	122.6 (2)	C10A—N1A—C4A	123.2 (2)
257	H1BA—C1B—H1BB	109.5	H1AA—C1A—H1AB	109.5
258	H1BA—C1B—H1BC	109.5	H1AA—C1A—H1AC	109.5
259	H1BB—C1B—H1BC	109.5	H1AB—C1A—H1AC	109.5
260	C3B—C1B—H1BA	109.5	C2A—C1A—H1AA	109.5
261	C3B—C1B—H1BB	109.5	C2A—C1A—H1AB	109.5
262	C3B—C1B—H1BC	109.5	C2A—C1A—H1AC	109.5

263	H2BA—C2B—H2BB	109.5	N1A—C2A—C1A	111.9 (2)
264	H2BA—C2B—H2BC	109.5	N1A—C2A—H2A	107.0
265	H2BB—C2B—H2BC	109.5	N1A—C2A—C3A	111.5 (2)
266	C3B—C2B—H2BA	109.5	C1A—C2A—H2A	107.0
267	C3B—C2B—H2BB	109.5	C3A—C2A—C1A	112.1 (2)
268	C3B—C2B—H2BC	109.5	C3A—C2A—H2A	107.0
269	N1B—C3B—C1B	111.5 (2)	C2A—C3A—H3AA	109.5
270	N1B—C3B—C2B	111.6 (2)	C2A—C3A—H3AB	109.5
271	N1B—C3B—H3B	107.2	C2A—C3A—H3AC	109.5
272	C1B—C3B—H3B	107.2	H3AA—C3A—H3AB	109.5
273	C2B—C3B—C1B	111.9 (2)	H3AA—C3A—H3AC	109.5
274	C2B—C3B—H3B	107.2	H3AB—C3A—H3AC	109.5
275	C5B—C4B—N1B	120.3 (2)	C5A—C4A—N1A	119.6 (2)
276	C9B—C4B—N1B	119.9 (2)	C5A—C4A—C9A	119.7 (2)
277	C9B—C4B—C5B	119.8 (2)	C9A—C4A—N1A	120.6 (2)
278	C4B—C5B—H5B	120.0	C4A—C5A—H5A	120.1
279	C6B—C5B—C4B	120.1 (2)	C6A—C5A—C4A	119.9 (2)
280	C6B—C5B—H5B	120.0	C6A—C5A—H5A	120.1
281	C5B—C6B—H6B	119.8	C5A—C6A—H6A	119.9
282	C7B—C6B—C5B	120.3 (2)	C5A—C6A—C7A	120.3 (2)
283	C7B—C6B—H6B	119.8	C7A—C6A—H6A	119.9
284	C6B—C7B—H7B	120.2	C6A—C7A—H7A	120.2
285	C6B—C7B—C8B	119.7 (2)	C8A—C7A—C6A	119.6 (2)
286	C8B—C7B—H7B	120.2	C8A—C7A—H7A	120.2
287	C7B—C8B—H8B	119.7	C7A—C8A—H8A	119.8
288	C7B—C8B—C9B	120.5 (2)	C9A—C8A—C7A	120.4 (2)
289	C9B—C8B—H8B	119.7	C9A—C8A—H8A	119.8
290	C4B—C9B—C8B	119.6 (2)	C4A—C9A—H9A	120.0
291	C4B—C9B—H9B	120.2	C8A—C9A—C4A	120.1 (2)
292	C8B—C9B—H9B	120.2	C8A—C9A—H9A	120.0
293	N1B—C10B—H10B	114.4	N1A—C10A—H10A	114.0
294	N1B—C10B—C11B	131.2 (2)	N1A—C10A—C11A	131.9 (2)
295	C11B—C10B—H10B	114.4	C11A—C10A—H10A	114.0
296	C10B—C11B—C12B	125.0 (2)	C10A—C11A—C12A	125.3 (2)
297	C10B—C11B—B1B	115.2 (2)	C10A—C11A—B1A	115.1 (2)
298	C12B—C11B—B1B	119.6 (2)	C12A—C11A—B1A	119.5 (2)
299	C13B—C12B—C11B	120.9 (2)	C13A—C12A—C11A	120.8 (2)
300	C17B—C12B—C11B	121.1 (2)	C13A—C12A—C17A	118.0 (2)
301	C17B—C12B—C13B	118.0 (2)	C17A—C12A—C11A	121.2 (2)
302	C12B—C13B—H13B	119.6	C12A—C13A—H13A	119.5
303	C14B—C13B—C12B	120.9 (2)	C14A—C13A—C12A	120.9 (2)
304	C14B—C13B—H13B	119.6	C14A—C13A—H13A	119.5
305	C13B—C14B—H14B	119.9	C13A—C14A—H14A	119.8
306	C15B—C14B—C13B	120.3 (2)	C15A—C14A—C13A	120.3 (2)
307	C15B—C14B—H14B	119.9	C15A—C14A—H14A	119.8
308	C14B—C15B—H15B	120.3	C14A—C15A—H15A	120.3
309	C16B—C15B—C14B	119.4 (2)	C14A—C15A—C16A	119.4 (2)
310	C16B—C15B—H15B	120.3	C16A—C15A—H15A	120.3

311	C15B—C16B—H16B	119.7	C15A—C16A—H16A	119.9
312	C15B—C16B—C17B	120.5 (2)	C17A—C16A—C15A	120.3 (2)
313	C17B—C16B—H16B	119.7	C17A—C16A—H16A	119.9
314	C12B—C17B—H17B	119.5	C12A—C17A—H17A	119.5
315	C16B—C17B—C12B	120.9 (2)	C16A—C17A—C12A	121.0 (2)
316	C16B—C17B—H17B	119.5	C16A—C17A—H17A	119.5
317	C19B—C18B—O1B	127.9 (2)	C19A—C18A—O1A	128.7 (2)
318	C19B—C18B—C23B	122.6 (2)	C19A—C18A—C23A	122.0 (2)
319	C23B—C18B—O1B	109.4 (2)	C23A—C18A—O1A	109.3 (2)
320	C18B—C19B—H19B	122.0	C18A—C19A—H19A	121.8
321	C18B—C19B—C20B	115.9 (2)	C18A—C19A—C20A	116.4 (2)
322	C20B—C19B—H19B	122.0	C20A—C19A—H19A	121.8
323	C19B—C20B—H20B	119.3	C19A—C20A—H20A	119.0
324	C21B—C20B—C19B	121.4 (2)	C21A—C20A—C19A	121.9 (2)
325	C21B—C20B—H20B	119.3	C21A—C20A—H20A	119.0
326	C20B—C21B—H21B	119.2	C20A—C21A—H21A	119.4
327	C20B—C21B—C22B	121.6 (2)	C20A—C21A—C22A	121.3 (2)
328	C22B—C21B—H21B	119.2	C22A—C21A—H21A	119.4
329	C21B—C22B—H22B	121.8	C21A—C22A—H22A	122.0
330	C23B—C22B—C21B	116.4 (2)	C23A—C22A—C21A	116.1 (2)
331	C23B—C22B—H22B	121.8	C23A—C22A—H22A	122.0
332	C18B—C23B—O2B	109.24 (19)	C18A—C23A—O2A	109.3 (2)
333	C22B—C23B—O2B	128.6 (2)	C22A—C23A—O2A	128.3 (2)
334	C22B—C23B—C18B	122.1 (2)	C22A—C23A—C18A	122.4 (2)
335	O1B—B1B—C11B	124.4 (2)	O1A—B1A—C11A	124.2 (2)
336	O2B—B1B—O1B	110.6 (2)	O2A—B1A—O1A	110.4 (2)
337	O2B—B1B—C11B	125.0 (2)	O2A—B1A—C11A	125.5 (2)
338				
339	O1B—C18B—C19B—C20B	-176.7 (2)	O1A—C18A—C19A—C20A	-179.5 (2)
340	O1B—C18B—C23B—O2B	0.0 (3)	O1A—C18A—C23A—O2A	-0.5 (3)
341	O1B—C18B—C23B—C22B	177.9 (2)	O1A—C18A—C23A—C22A	178.9 (2)
342	N1B—C4B—C5B—C6B	179.9 (2)	N1A—C4A—C5A—C6A	-179.4 (2)
343	N1B—C4B—C9B—C8B	-179.9 (2)	N1A—C4A—C9A—C8A	179.4 (2)
344	N1B—C10B—C11B—C12B	-9.7 (4)	N1A—C10A—C11A—C12A	-9.7 (4)
345	N1B—C10B—C11B—B1B	175.5 (2)	N1A—C10A—C11A—B1A	171.6 (2)
346	C3B—N1B—C4B—C5B	-45.6 (3)	C2A—N1A—C4A—C5A	132.7 (2)
347	C3B—N1B—C4B—C9B	132.5 (2)	C2A—N1A—C4A—C9A	-45.1 (3)
348	C3B—N1B—C10B—C11B	159.6 (3)	C2A—N1A—C10A—C11A	165.3 (2)
349	C4B—N1B—C3B—C1B	-123.7 (2)	C4A—N1A—C2A—C1A	-120.9 (2)
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352	C4B—C5B—C6B—C7B	-0.4 (4)	C4A—C5A—C6A—C7A	0.1 (4)
353	C5B—C4B—C9B—C8B	-1.8 (4)	C5A—C4A—C9A—C8A	1.6 (4)
354	C5B—C6B—C7B—C8B	-1.0 (4)	C5A—C6A—C7A—C8A	1.3 (4)
355	C6B—C7B—C8B—C9B	1.0 (4)	C6A—C7A—C8A—C9A	-1.3 (4)
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358	C10B—N1B—C3B—C1B	57.5 (3)	C10A—N1A—C2A—C1A	56.4 (3)

359	C10B—N1B—C3B—C2B	-68.4 (3)	C10A—N1A—C2A—C3A	-69.9 (3)
360	C10B—N1B—C4B—C5B	133.1 (2)	C10A—N1A—C4A—C5A	-44.5 (3)
361	C10B—N1B—C4B—C9B	-48.8 (3)	C10A—N1A—C4A—C9A	137.6 (2)
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363	C10B—C11B—C12B—C17B	131.9 (3)	C10A—C11A—C12A—C17A	-49.3 (3)
364	C10B—C11B—B1B—O1B	-0.5 (4)	C10A—C11A—B1A—O1A	1.7 (4)
365	C10B—C11B—B1B—O2B	176.9 (2)	C10A—C11A—B1A—O2A	-179.8 (2)
366	C11B—C12B—C13B—C14B	-179.0 (2)	C11A—C12A—C13A—C14A	-179.5 (2)
367	C11B—C12B—C17B—C16B	179.3 (2)	C11A—C12A—C17A—C16A	179.9 (2)
368	C12B—C11B—B1B—O1B	-175.6 (2)	C12A—C11A—B1A—O1A	-177.2 (2)
369	C12B—C11B—B1B—O2B	1.7 (4)	C12A—C11A—B1A—O2A	1.4 (4)
370	C12B—C13B—C14B—C15B	0.2 (4)	C12A—C13A—C14A—C15A	-1.0 (4)
371	C13B—C12B—C17B—C16B	1.6 (3)	C13A—C12A—C17A—C16A	-1.6 (3)
372	C13B—C14B—C15B—C16B	0.8 (4)	C13A—C14A—C15A—C16A	-0.6 (4)
373	C14B—C15B—C16B—C17B	-0.5 (4)	C14A—C15A—C16A—C17A	1.1 (4)
374	C15B—C16B—C17B—C12B	-0.6 (4)	C15A—C16A—C17A—C12A	0.1 (4)
375	C17B—C12B—C13B—C14B	-1.3 (3)	C17A—C12A—C13A—C14A	2.0 (3)
376	C18B—O1B—B1B—O2B	1.1 (3)	C18A—O1A—B1A—O2A	-1.2 (3)
377	C18B—O1B—B1B—C11B	178.7 (2)	C18A—O1A—B1A—C11A	177.5 (2)
378	C18B—C19B—C20B—C21B	-0.9 (4)	C18A—C19A—C20A—C21A	0.1 (4)
379	C19B—C18B—C23B—O2B	-177.8 (2)	C19A—C18A—C23A—O2A	179.4 (2)
380	C19B—C18B—C23B—C22B	0.1 (4)	C19A—C18A—C23A—C22A	-1.2 (4)
381	C19B—C20B—C21B—C22B	0.3 (4)	C19A—C20A—C21A—C22A	-0.2 (4)
382	C20B—C21B—C22B—C23B	0.5 (4)	C20A—C21A—C22A—C23A	-0.3 (4)
383	C21B—C22B—C23B—O2B	176.8 (2)	C21A—C22A—C23A—O2A	-179.7 (2)
384	C21B—C22B—C23B—C18B	-0.7 (4)	C21A—C22A—C23A—C18A	1.0 (4)
385	C23B—O2B—B1B—O1B	-1.1 (3)	C23A—O2A—B1A—O1A	1.0 (3)
386	C23B—O2B—B1B—C11B	-178.7 (2)	C23A—O2A—B1A—C11A	-177.8 (2)
387	C23B—C18B—C19B—C20B	0.7 (4)	C23A—C18A—C19A—C20A	0.6 (4)
388	B1B—O1B—C18B—C19B	177.0 (2)	B1A—O1A—C18A—C19A	-178.8 (3)
389	B1B—O1B—C18B—C23B	-0.6 (3)	B1A—O1A—C18A—C23A	1.0 (3)
390	B1B—O2B—C23B—C18B	0.7 (3)	B1A—O2A—C23A—C18A	-0.3 (3)
391	B1B—O2B—C23B—C22B	-177.1 (2)	B1A—O2A—C23A—C22A	-179.7 (3)
392	B1B—C11B—C12B—C13B	124.1 (3)	B1A—C11A—C12A—C13A	-49.1 (3)
393	B1B—C11B—C12B—C17B	-53.5 (3)	B1A—C11A—C12A—C17A	129.4 (2)

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