



HAL
open science

Pitfalls of a structure determination: The structure of closo-9-[4-(dibenzylamino)phenyl]-1,2-dicarbadoecaborane(12)

Max Milewski, Anne-Marie Caminade, Evamarie Hey-Hawkins, Peter Lönnecke

► To cite this version:

Max Milewski, Anne-Marie Caminade, Evamarie Hey-Hawkins, Peter Lönnecke. Pitfalls of a structure determination: The structure of closo-9-[4-(dibenzylamino)phenyl]-1,2-dicarbadoecaborane(12). Graciela Diaz de Delgado; Sean Parkin. Modern approaches and tools for teaching crystallography, Art. 6 (Issu de Acta Crystallographica 2022, E78 pp. 1145-1150), IUCr, 2023, 10.1107/S205698902201043X . hal-04195359

HAL Id: hal-04195359

<https://hal.science/hal-04195359>

Submitted on 4 Sep 2023

HAL is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L'archive ouverte pluridisciplinaire **HAL**, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d'enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.



Distributed under a Creative Commons Attribution 4.0 International License

Pitfalls of a structure determination: The structure of *closo*-9-[4-(dibenzylamino)phenyl]-1,2-dicarbadodecaborane(12)

Max Milewski,^{a,b} Anne-Marie Caminade,^b Evamarie Hey-Hawkins^a and Peter Lönnecke^{a*}

Received 4 October 2022

Accepted 30 October 2022

Edited by V. Jancik, Universidad Nacional Autónoma de México, México

Keywords: pitfalls; structure determination; pseudo translation symmetry (PTS); disorder; crystal structure.

CCDC reference: 2216569

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

^aFaculty of Chemistry and Mineralogy, Institute of Inorganic Chemistry, Leipzig University, Johannisallee 29, 04103 Leipzig, Germany, and ^bCentre National de la Recherche Scientifique (CNRS), Laboratoire de Chimie de Coordination, 205 Route de Narbonne, 31077 Toulouse Cedex 4, France. *Correspondence e-mail: peter.loennecke@uni-leipzig.de

Automated X-ray data recording and data reduction strategies are straightforward and powerful these days, as well as semi-automated routines to solve standard structures without, or at least with only little manual intervention and within a relatively short time. The structure determination of the aminocarborane *closo*-9-[4-(dibenzylamino)phenyl]-1,2-dicarbadodecaborane(12), C₂₂H₂₉B₁₀N, is an example that automated routines still always have to be revised and that it may be necessary to be aware of possible problems of a structure determination.

1. Chemical context

Derivatives of *closo*-dicarbadodecaboranes (carboranes) have been the focus of interest of many research groups over the past few years as building blocks for medicinal chemistry, supramolecular assemblies and ligands in metal complexes (selected references: Stockmann, 2019; Andrews, 1999 and Grimes, 2016). In particular, the readily accessible 1,2-disubstituted *closo*-dicarbadodecaboranes (*ortho*-carboranes) are a class of versatile compounds with attractive properties such as a flexible C—C bond. Unlike the C—H groups, the B—H units are much less polar and, depending on their position within the cluster, they exhibit different electronic properties, requiring alternative methods for regioselective substitution. Following the procedures of Zakharkin and co-workers (Zakharkin, 1982), *closo*-9-[4-(dibenzylamino)phenyl]-1,2-dicarbadodecaborane(12) was prepared (see Scheme below). After removal of the benzylic protecting groups, the resulting aniline derivative is a suitable functionalized building block for various modifications.

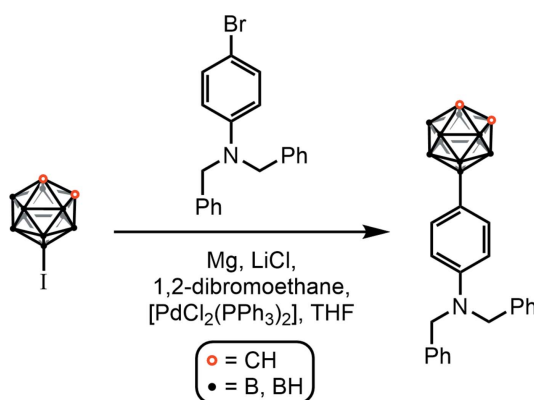
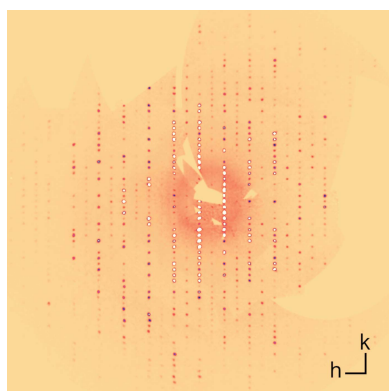


Table 1

Initial unit cell (\AA , $^\circ$, \AA^3) derived after the automated data reduction with *CrysAlis PRO* (Rigaku OD, 2020).

Standard cell		Non-standard cell ^a	
a_1	6.5043 (1)	a_2	6.5043 (1)
b_1	27.1414 (5)	b_2	27.1414 (5)
c_1	13.2456 (3)	c_2	13.9094 (3)
β_1	98.101 (2)	β_2	109.477 (2)
V	2314.99 (8)	V	2314.99 (8)

Note (a): Unit-cell transformation matrix by rows: $\bar{1} 0 0 0 \bar{1} 0 1 0 1$.

2. Structural commentary

To quote Anthony Linden (Linden, 2020), ‘With a nice graphical user interface (GUI), there is great temptation to click quickly through all the steps of, for example, a structure refinement and the task is complete within minutes, but is the result correct? Is it the best possible result? How can one be sure? Nothing is foolproof (yet!) and users should not rely 100% on automated tools.’

The structure determination of *closo*-dicarbadodecaborane derivatives based on a 12-vertex icosahedron is frequently hindered, if not prevented, by rotational disorder, especially of the CH and BH vertices. This is a consequence of the approximately spherical shape of a carborane unit and, of course, its relatively weak intermolecular interactions. Up to now, the structures of the pure *ortho*-, *meta*- and *para*-*closo*-dicarbadodecaboranes are still unknown. Only co-crystallized solvent associates have been published and these lead to precisely defined non-disordered cluster arrangements (Fox & Hughes, 2004). Considering these facts, a carborane structure may easily be associated with a disorder of the carborane unit.

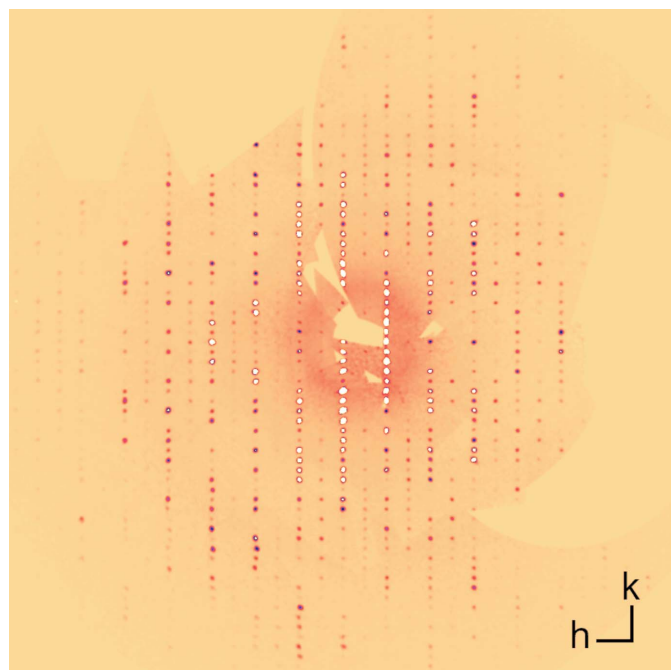


Figure 1
Reconstructed $(hk\bar{2})$ -layer.

Table 2

Experimental details.

Crystal data	
Chemical formula	$C_{22}H_{29}B_{10}N$
M_r	415.56
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	130
a, b, c (\AA)	13.0128 (2), 27.1423 (4), 13.9114 (3)
β ($^\circ$)	109.485 (2)
V (\AA^3)	4632.07 (15)
Z	8
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.06
Crystal size (mm)	0.40 \times 0.35 \times 0.25
Data collection	
Diffractometer	Xcalibur, Sapphire3, Gemini
Absorption correction	Multi-scan (<i>CrysAlis PRO</i> ; Rigaku OD, 2020)
T_{\min} , T_{\max}	0.997, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	76840, 15117, 9789
R_{int}	0.054
$(\sin \theta/\lambda)_{\text{max}}$ (\AA^{-1})	0.745
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.056, 0.160, 1.02
No. of reflections	15117
No. of parameters	827
H-atom treatment	All H-atom parameters refined
$\Delta\rho_{\text{max}}$, $\Delta\rho_{\text{min}}$ ($e \text{\AA}^{-3}$)	0.36, -0.25

Computer programs: *CrysAlis PRO* (Rigaku OD, 2020), *SHELXT2018-2* (Sheldrick, 2015a), *SHELXL2018-3* (Sheldrick, 2015b), *DIAMOND* (Crystal Impact GbR, 2022) and *WinGX* (Farrugia, 2012).

Taking the data of this structure determination automatically generated by the *CrysAlis PRO* software routine (Rigaku OD, 2020), the structure solution (monoclinic cell; space group $P2_1/n$; Table 1) provided a disordered carborane unit, which can only be refined with a very limited accuracy and with unlocalizable carbon atoms of the carborane unit. Nevertheless, the final structure parameters for this cell setting (R_1 and wR_2 for all observed reflections: 0.0543 and 0.1268), the space group $P2_1/n$ and its associated extinction conditions didn't look suspicious, and for the remaining non-disordered fraction of the molecule, even all hydrogen atoms could be localized in the final step of the refinement. However, disordered structures should always be checked thoroughly, as disorder may be feigned and a result of an incorrect space group, twinning, an erroneous unit-cell determination, *etc.* For these reasons, the original recorded frames had been manually inspected as well. This inspection of the original data revealed a small number of additional very weak reflections, which had been ignored by the automated analysis and integration routine. As a representative, the reconstructed $(hk\bar{2})$ -layer is given in Fig. 1. Analyzing all reflections the intensity distribution can be quantified by:

$$\frac{\sum_{h \neq 2n} F_o^2(hkl)}{\sum_{h=2n} F_o^2(hkl)} = 0.091$$

These additional reflections ($h \neq 2n$) represent approximately 8% of the total intensity. Taking these weak reflections into

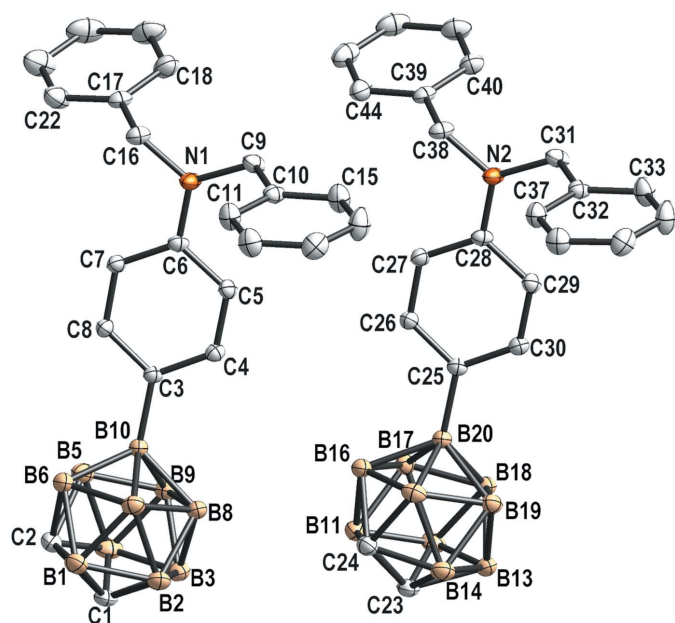


Figure 2
The two molecules of the asymmetric unit (displacement ellipsoids are drawn at the 50% probability level; hydrogen atoms are omitted for clarity).

account, the *a*-axis is doubled and a new modified unit cell is obtained, with parameters summarized in Table 2. To compare this larger unit cell with the initial cell of the automated data reduction routine given above, the cell setting of the small standard cell a_1, b_1, c_1, β_1 may be better transformed into its non-standard setting a_2, b_2, c_2, β_2 (Table 1).

With this larger cell, the structure could be solved with *SHELXT* (Sheldrick, 2015a) without any problems, whereby the two carborane clusters of the asymmetric unit are not disordered any longer. Without any doubt, all carbon atoms of both carborane units could be identified with a bond-length and displacement-parameter analysis, and in the final step,

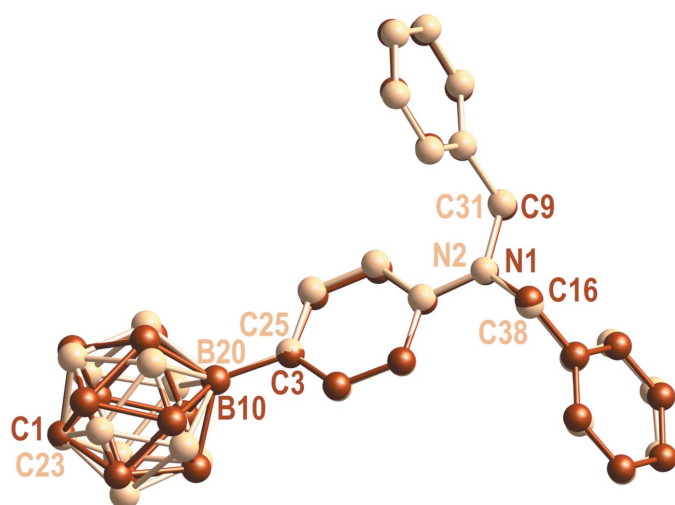


Figure 3
Overlay of molecule 1 (brown: B1–B10/C1–C22/N1) and molecule 2 (light brown: B11–B20/C23–C44/N2). Hydrogen atoms are omitted for clarity.

even all hydrogen atoms could be localized, as expected for a light-atom structure. However, there is a strong resemblance between the two molecules of the asymmetric unit (Fig. 2) and it seems to be obvious to check the structure for pseudosymmetry.

As illustrated with an overlay (Fig. 3), both molecules of the asymmetric unit represent more or less the same conformer, but the central carborane fragments C2/B1–B9 (molecule 1) and C24/B11–B19 (molecule 2) are clearly in a different position and can't be mapped to each other with a translation vector. However, these units are responsible for the enlarged unit cell, and the question arises as to the reason for these differently positioned carborane units. To answer this question, an analysis of intermolecular interactions of the two carboranes may be most expedient.

3. Supramolecular features

Numerous carborane structures have previously been analyzed and a very interesting summary on this topic was published by Fox & Hughes (2004). As a consequence of carbon and boron electronegativity differences, the CH moiety of a carborane cluster has moderate acidity, whereas the basicity is expected to be dominating for the BH fragment. Analyzing intermolecular contact distances, one of the most obvious and striking features of this structure is a relatively strong carborane–phenyl- π ($\text{CH} \cdots \pi\text{C}_6$) interaction (Fig. 4). For both molecules of the asymmetric unit, the CH moiety of the carborane in *para* position to the substituent *R*1 and *R*2 points directly towards the center of an adjacent boron-bonded phenyl ring interacting with the entire πC_6 -system. For this kind of $\text{CH} \cdots \pi\text{C}_6$ interactions, Kraka and co-workers (Zou *et al.*, 2018) calculated a bonding energy of -33 kJ mol^{-1} ($-7.9 \text{ kcal mol}^{-1}$) for an ideally centered CH donor with a centroid distance of 2.36 \AA (*i.e.* 2.74 \AA for each $\text{CH} \cdots \pi\text{C}$). This energy represents approximately one tenth of an ordinary carbon–carbon single bond. The experimentally estimated $\text{CH} \cdots \pi\text{C}$ contact distances are in the range of 2.68 (2) (d_{min}) to 2.86 (2) \AA (d_{max}) for $\text{C1H} \cdots \pi\text{C}$ and 2.68 (2) to 2.78 (2) \AA for $\text{C23H} \cdots \pi\text{C}$. It can be thus proven that all $\text{CH} \cdots \pi\text{C}$ contact distances are shorter than the sum of the van der Waals radii (H: 1.2 \AA , C: 1.7 \AA). The mean $\text{CH} \cdots \pi\text{C}$ distances of 2.76 (2) \AA for C1 and 2.73 (2) \AA for C23 are quite close to the value given by Kraka and co-workers (Zou *et al.*, 2018).

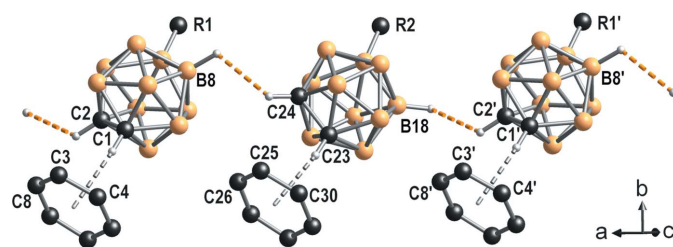


Figure 4
Intermolecular carborane–phenyl- π ($\text{CH} \cdots \pi\text{C}_6$) interactions. [*R*1 = *R*2 = $\text{C}_6\text{H}_4\text{-N}(\text{CH}_2\text{-Ph})_2$]. Symmetry codes as in Table 3 and $x - 1, y, z$.

For the remaining two N-bonded benzyl substituents only weak $\text{CH}_2 \cdots \text{C}$ π -stacking with adjacent N-bonded benzyl substituents is detectable: $\text{CH16B} \cdots \text{C22} = 2.87$ (2) Å and $\text{CH38B} \cdots \text{C44} = 2.83$ (2) Å.

The intermolecular interactions discussed so far are dominant and comparable for both molecules of the asymmetric unit; they control the packing and can thus be used as an explanation for the structurally related conformers as illustrated by the overlay shown in Fig. 3. To explain the different orientation of the central carborane fragments (C2/B1–B9 and C24/B11–B19), which is ultimately responsible for the enlarged unit cell, we analyzed intermolecular carborane–carborane interactions as well (Fig. 5). These non-conventional dihydrogen bonds $M\text{—H} \cdots \text{H—X}$, first discovered in 1996 by Robert H. Crabtree (Crabtree *et al.*, 1996), are bonds formed between a proton donor $X\text{—H}$ (X = halogen, chalcogen, pnictogen or even carbon) and a hydridic proton acceptor $M\text{—H}$ (M = metal, transition metal or boron), and it is a well-known fact by now that these dihydrogen bonds may contribute to intra- and intermolecular bonding. To quantify this kind of interaction, molecular dynamics simulations conducted by Hobza's group (Fanfrlík *et al.*, 2006) revealed bonding energies of approximately -20 kJ mol⁻¹ (-4.8 kcal mol⁻¹).

For this structure, the shortest experimental intermolecular $\text{CH} \cdots \text{HB}$ contacts are 2.49 (2) Å ($\text{H24} \cdots \text{H8X}$) and 2.61 (2) Å ($\text{H2} \cdots \text{H18X}$), both slightly longer than the sum of the van der Waals radii of 2.4 Å, indicating a quite weak and most likely electrostatic interaction. Nevertheless, this interaction provides the only striking argument for an ordered structure, as steric interactions can clearly be excluded. Considering steric arguments only, the CH fragment of the carborane moiety may take every possible position by simply rotating the entire carborane unit (rotational disorder). A theoretical calculation conducted by Hobza and co-workers (Fanfrlík *et al.*, 2006) on intermolecular interactions of biomolecules with *closo*-1-carbadodecaboranes led to the observation that carboranes favor the formation of $\text{CH} \cdots \text{HB}$ dihydrogen bonds with biomolecules, with the C–H unit of the biomolecule pointing preferably to the lower hemisphere position of the carborane, the part of the carborane cage opposite to the carbon atom. In addition, it is known that electrophiles attack carborane boron atoms preferably in an opposite *para* position to the carbon atom (Rudakov *et al.*, 2011). Considering these facts, the observed alternating interaction illustrated in Fig. 5 may be explainable: The boron atom in the *para* position to C1 and C23 bears the substituent (Fig. 4). C1–H and C23–H are strongly and highly symmetrically bonded to the adjacent π -system, fixing the carborane unit within the lattice.

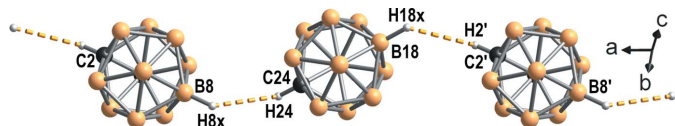


Figure 5
Intermolecular carborane-carborane interactions. Symmetry code: (') $x - 1, y, z$.

However, rotation of this unit along the C1B10 and C23B20 axis seems to be still possible so that the remaining CH units C2H and C24H can interact *via* $\text{CH} \cdots \text{HB}$ contacts with the most preferred neighboring BH units B18 and B8, as both of these boron atoms are in a *para* position to a carborane carbon atom.

A particularly interesting aspect of these non-conventional dihydrogen bonds is that linear $\text{B—H} \cdots \text{H—X}$ arrangements are an exception (Calhorda, 2000), and it is still questionable whether the hydrogen-donor bond is pointing towards the hydridic B–H bond rather than the hydrogen atom itself. For this structure, the C–H \cdots H angles for the shortest contacts ($\text{C2—H2} \cdots \text{H18X}$ and $\text{C24—H24} \cdots \text{H8X}$) range from 116 (1) to 118 (1)° (Fig. 5).

4. Database survey

To compare the contact distances with other *ortho*-carboranes, we analyzed the Cambridge Structural Database (CSD; Version 5.43, last update November 2021; Groom *et al.*, 2016) for *ortho*-carboranes with comparable intermolecular $\text{CH} \cdots \pi\text{C}_6$ contact distances. The accuracy of all intermolecular hydrogen– πC_6 distances was improved by normalizing the C–H bond distances to 1.089 Å, a value derived from neutron diffraction experiments (Allen & Bruno, 2010). The results are summarized in Fig. 6. To get more hits, the range of intermolecular interactions was expanded, so that the mean values of the $\text{CH} \cdots \pi\text{C}_6$ interactions are still smaller than the sum of the van der Waals radii. The given data represent approximately 6% of all published *ortho*-carboranes with at least one phenyl fragment in the structure. 27% of all published *ortho*-carborane-phenyl structures have at least one short contact, illustrating the particular importance of this interaction. In addition to the strength of the individual $\text{CH} \cdots \pi\text{C}_6$ interaction, represented by its contact distance, Fig. 6 illustrates its symmetry: the smaller the range of the

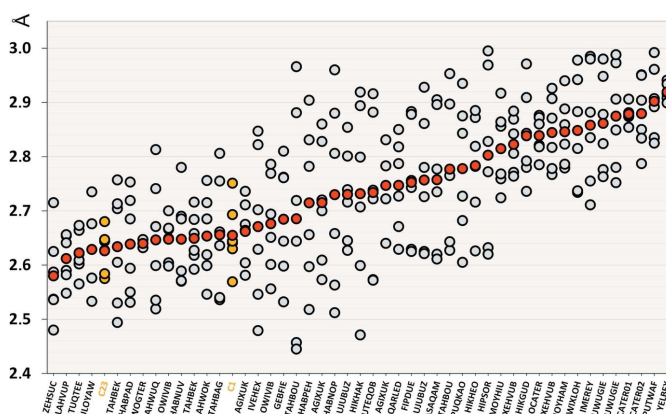


Figure 6
Normalized ($\text{C—H} = 1.089$ Å) *ortho*-carborane $\text{CH} \cdots \pi\text{C}_6$ contact distances (gray) published in the Cambridge Structural Database (CSD, version 5.43) with the corresponding CSD entry code. The experimental normalized $\text{CH} \cdots \pi\text{C}_6$ contacts for C1 and C23 are highlighted in orange, and mean $\text{CH} \cdots \pi\text{C}_6$ contacts are presented in red.

Table 3
Hydrogen-bond geometry (Å, °).

D—H...A	D—H	H...A	D...A	D—H...A
C1—H1...C3 ⁱ	0.968 (17)	2.742 (17)	3.6196 (17)	151.0 (13)
C1—H1...C4 ⁱ	0.968 (17)	2.676 (17)	3.5889 (17)	157.3 (12)
C1—H1...C5 ⁱ	0.968 (17)	2.740 (17)	3.6322 (18)	153.4 (12)
C1—H1...C6 ⁱ	0.968 (17)	2.860 (17)	3.7008 (18)	145.8 (13)
C1—H1...C7 ⁱ	0.968 (17)	2.797 (17)	3.6112 (18)	142.2 (12)
C1—H1...C8 ⁱ	0.968 (17)	2.745 (16)	3.5809 (18)	144.9 (12)
C23—H23...C25 ⁱ	0.971 (16)	2.780 (16)	3.6436 (17)	148.4 (12)
C23—H23...C26 ⁱ	0.971 (16)	2.741 (15)	3.5682 (17)	143.4 (11)
C23—H23...C27 ⁱ	0.971 (16)	2.724 (15)	3.5432 (18)	142.3 (11)
C23—H23...C28 ⁱ	0.971 (16)	2.749 (16)	3.6050 (18)	147.3 (12)
C23—H23...C29 ⁱ	0.971 (16)	2.681 (16)	3.5857 (18)	155.0 (12)
C23—H23...C30 ⁱ	0.971 (16)	2.690 (16)	3.5996 (17)	156.0 (12)

Symmetry code: (i) $x, -y + \frac{1}{2}, z - \frac{1}{2}$.

individual CH... π C₆ contact distances are, the more centered and symmetrical the π -bonded system is. Quantitatively, this can be expressed with a delta value defined as $\Delta = d_{\max} - d_{\min}$ (Fig. 7). The individual contact distances for C1 and C23 differ by only ± 0.09 Å and ± 0.05 Å from the mean value, indicating a very highly symmetrical interaction. Numerical details of the hydrogen bonds in the title structure are given in Table 3.

Finally, we compared intermolecular C—H...H—B contact distances between carboranes as well, as they are responsible for our ordered structure and the enlarged unit cell. To get more reliable values, we analyzed the CSD in a comparable manner by using normalized C—H (1.089 Å) and B—H (1.185 Å) bond lengths (Allen & Bruno, 2010). The two shortest normalized C—H...H—B contacts for our structure differ from the values given above: 2.438 (11) Å for H24...H8X and 2.539 (7) Å for H2...H18X. They are both slightly shorter as expected, but still longer than the sum of the van der Waals radii of 2.4 Å. For comparison, 21% of all *ortho*-carboranes published in the CSD exhibit much stronger interactions with contact distances ranging from 1.858 to 2.400 Å, indicating that short contacts are by no means exotic.

5. Summary

Intermolecular interactions play a crucial role in the molecular self-assembly of compounds, which is important in biological

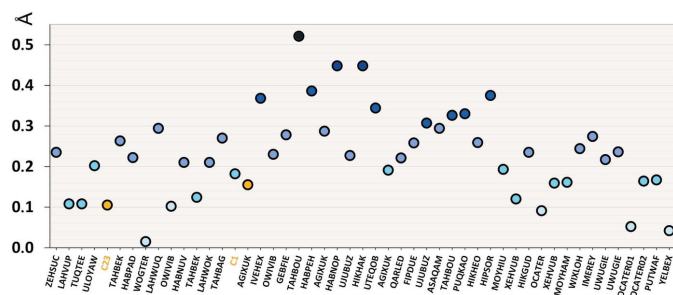


Figure 7
Delta values (blue) of *ortho*-carborane CH... π C₆ contact distances defined as $\Delta = d_{\max} - d_{\min}$. The experimental values for C1 and C23 are highlighted in orange.

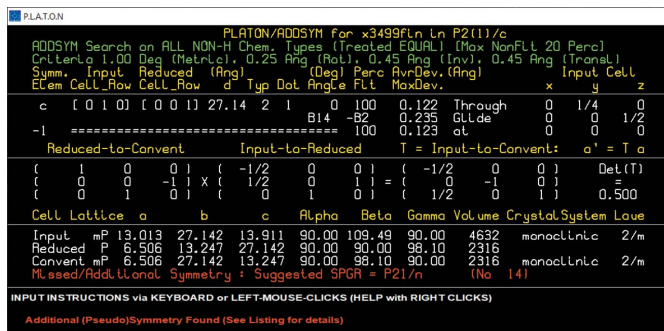


Figure 8
PLATON ADDSYM checkCIF validation alert.

systems or in catalysis. It is noteworthy that substituted carboranes can form two types of non-covalent interactions: dihydrogen bonds or stacking interactions. In particular, dihydrogen bonds of the B—H...H—X type are important for the binding of this class of compounds to biomolecules. Starting from *ortho*-carborane, promising ligands for homogeneous catalysis can be prepared by directed synthesis. The compound *closo*-9-[4-(dibenzylamino)phenyl]-1,2-dicarbado-decaborane(12) reported here can thus be used as a building block after deprotection of the amino group.

The detailed analysis of this structure determination revealed pseudo translation symmetry (PTS) and thus we can ignore the PLATON checkCIF (Spek, 2020) validation alert (Fig. 8). Without any doubt, the huge unit cell is the correct solution. The example given here clearly demonstrates that it is always important to check the original recorded data of a structure determination.

6. Synthesis and crystallization

All reactions were conducted under N₂. A solid mixture of Mg turnings (0.356 g, 14.6 mmol) and LiCl (0.364 g, 8.59 mmol) was stirred overnight to mechanically activate the Mg. 1,2-Dibromoethane (0.1 ml, 1.15 mmol) in THF (10 ml) was added slowly to further activate Mg. Then *N,N*-dibenzyl-4-bromoaniline (2.58 g, 7.33 mmol) in THF (15 ml) was added over 40 min. The reaction mixture was stirred for 18 h at rt and then slowly added to a solution of 9-iodo-*ortho*-carborane (0.592 g, 1.96 mmol) and [PdCl₂(PPh₃)₂] (0.045 g, 0.064 mmol) in THF (10 ml) over 15 min. The resulting mixture was stirred at rt for *ca* 5 d; the reaction progress was monitored by NMR spectroscopy. At the end of the reaction, the mixture was quenched with methanol (50 ml) and distilled water (100 ml), then poured into Et₂O (50 ml), and the phases were separated. The organic layer was washed with saturated sodium bicarbonate solution (20 ml), and the aqueous phase was extracted with Et₂O (4 × 50 ml). The combined organic layers were dried over MgSO₄. After evaporation of solvents, the remaining brown oil was purified by column chromatography on silica gel, using a Biotage Isolera ONE SNAP KP-SIL 100 g cartridge, 100 ml min⁻¹, *n*-hexane/ethyl acetate (97:3 to 40:60, *v/v*), yielding 0.492 g (1.18 mmol, 50%) of a colorless solid.

Crystals suitable for X-ray structure analysis were obtained from dichloromethane and *n*-pentane by slow evaporation.

R_F (*n*-hexane/ethyl acetate 85:15, *v/v*) = 0.25.

^1H NMR (400 MHz, CDCl_3 , 298 K): δ = 7.34 to 7.28 (*m*, 4H, Bz–H), 7.27 to 7.23 (*m*, 6H, Bz–H), 7.17 (*d*, 2H, J = 8.3 Hz, C_6H_4), 6.65 (*d*, 2H, J = 8.3 Hz, C_6H_4), 4.61 (*s*, 4H, CH_2), 3.56 (*s*, 1H, $\text{C}_{\text{carborane}}\text{–H}$), 3.45 (*s*, 1H, $\text{C}_{\text{carborane}}\text{–H}$), 3.14 to 1.58 (*m*, 9H, BH) ppm.

$^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, CDCl_3 , 298 K) δ = 148.9, 139.0, 133.4, 128.7, 126.9, 126.9, 111.8, 54.2, 52.9, 48.0 ppm.

$^{11}\text{B}\{^1\text{H}\}$ NMR (128 MHz, CDCl_3) δ = 8.3 (1B), –2.2 (1B), –8.7 (2B), –13.2 to –16.3 (6B) ppm.

^{11}B NMR (128 MHz, CDCl_3 , 298 K): δ = 8.3 (*s*, 1B), –2.2 (*d*, 1B, $^1J_{\text{BH}}$ = 149 Hz), –8.7 (*d*, 2B, $^1J_{\text{BH}}$ = 151 Hz), –14.7 (*m*, 6B) ppm.

HRMS (ESI+) [$\text{C}_{22}\text{H}_{29}\text{B}_{10}\text{N}$], m/z calculated: 416.3387 ($[\text{M} + \text{H}]^+$), m/z found: 416.3385 (100%).

6.1. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. All hydrogen atoms were located in difference-Fourier maps and refined isotropically. Distance restraints were applied only temporarily for hydrogen atoms with normalized B–H and C–H carborane bond lengths.

Funding information

The project leading to this application received funding from the European Union's Horizon 2020 research and innovation programme under the Marie Skłodowska-Curie grant agreement No. 860322. This paper was funded by the Open Access

Publishing Fund of Leipzig University supported by the German Research Foundation within the program Open Access Publication Funding.

References

- Allen, F. H. & Bruno, I. J. (2010). *Acta Cryst.* **B66**, 380–386.
- Andrews, P. C., Hardie, M. J. & Raston, C. L. (1999). *Coord. Chem. Rev.* **189**, 169–198.
- Calhorda, M. J. (2000). *Chem. Commun.* pp. 801–809.
- Crabtree, R. H., Siegbahn, P. E. M., Eisenstein, O., Rheingold, A. L. & Koetzle, T. F. (1996). *Acc. Chem. Res.* **29**, 348–354.
- Crystal Impact GbR. (2022). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
- Fanfrlík, J., Lepšík, M., Horinek, D., Havlas, Z. & Hobza, P. (2006). *ChemPhysChem*, **7**, 1100–1105.
- Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
- Fox, M. A. & Hughes, A. K. (2004). *Coord. Chem. Rev.* **248**, 457–476.
- Grimes, R. N. (2016). *Carboranes*, 3rd ed. London, Burlington, MA: Academic Press.
- Groom, C. R., Bruno, I. J., Lightfoot, M. P. & Ward, S. C. (2016). *Acta Cryst.* **B72**, 171–179.
- Linden, A. (2020). *Acta Cryst.* **E76**, 765–775.
- Rigaku OD (2020). *CrysAlis PRO*. Rigaku Oxford Diffraction, Wroclaw, Poland.
- Rudakov, D. A., Kurman, P. V. & Potkin, V. I. (2011). *Russ. J. Gen. Chem.* **81**, 1137–1142.
- Sheldrick, G. M. (2015a). *Acta Cryst.* **A71**, 3–8.
- Sheldrick, G. M. (2015b). *Acta Cryst.* **C71**, 3–8.
- Spek, A. L. (2020). *Acta Cryst.* **E76**, 1–11.
- Stockmann, P., Gozzi, M., Kuhnert, R., Sárosi, M. B. & Hey-Hawkins, E. (2019). *Chem. Soc. Rev.* **48**, 3497–3512.
- Zakharkin, L. I., Kovredov, A. I., Ol'shevskaya, V. A. & Shaugumbekova, Z. S. (1982). *J. Organomet. Chem.* **226**, 217–222.
- Zou, W., Zhang, X., Dai, H., Yan, H., Cremer, D. & Kraka, E. (2018). *J. Organomet. Chem.* **865**, 114–127.

supporting information

Acta Cryst. (2022). E78 [https://doi.org/10.1107/S205698902201043X]

Pitfalls of a structure determination: The structure of *closo*-9-[4-(dibenzylamino)phenyl]-1,2-dicarbadoecaborane(12)

Max Milewski, Anne-Marie Caminade, Evamarie Hey-Hawkins and Peter Lönnecke

Computing details

Data collection: *CrysAlis PRO* (Rigaku OD, 2020); cell refinement: *CrysAlis PRO* (Rigaku OD, 2020); data reduction: *CrysAlis PRO* (Rigaku OD, 2020); program(s) used to solve structure: *SHELXT2018-2* (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2018-3* (Sheldrick, 2015b); molecular graphics: *DIAMOND* (Crystal Impact GbR, 2022); software used to prepare material for publication: *WinGX* (Farrugia, 2012).

closo-9-[4-(Dibenzylamino)phenyl]-1,2-dicarbadoecaborane(12)

Crystal data

$C_{22}H_{29}B_{10}N$	$F(000) = 1744$
$M_r = 415.56$	$D_x = 1.192 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
$a = 13.0128 (2) \text{ \AA}$	Cell parameters from 15129 reflections
$b = 27.1423 (4) \text{ \AA}$	$\theta = 2.7\text{--}31.7^\circ$
$c = 13.9114 (3) \text{ \AA}$	$\mu = 0.06 \text{ mm}^{-1}$
$\beta = 109.485 (2)^\circ$	$T = 130 \text{ K}$
$V = 4632.07 (15) \text{ \AA}^3$	Prism, clear colourless
$Z = 8$	$0.40 \times 0.35 \times 0.25 \text{ mm}$

Data collection

Xcalibur, Sapphire3, Gemini diffractometer	76840 measured reflections
Radiation source: fine-focus sealed X-ray tube	15117 independent reflections
Detector resolution: $16.3560 \text{ pixels mm}^{-1}$	9789 reflections with $I > 2\sigma(I)$
ω scans	$R_{\text{int}} = 0.054$
Absorption correction: multi-scan (CrysAlisPro; Rigaku OD, 2020)	$\theta_{\text{max}} = 32.0^\circ$, $\theta_{\text{min}} = 2.2^\circ$
$T_{\text{min}} = 0.997$, $T_{\text{max}} = 1.000$	$h = -18 \rightarrow 18$
	$k = -39 \rightarrow 39$
	$l = -20 \rightarrow 19$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.056$	All H-atom parameters refined
$wR(F^2) = 0.160$	$w = 1/[\sigma^2(F_o^2) + (0.0768P)^2 + 0.5669P]$
$S = 1.02$	where $P = (F_o^2 + 2F_c^2)/3$
15117 reflections	$(\Delta/\sigma)_{\text{max}} = 0.001$
827 parameters	$\Delta\rho_{\text{max}} = 0.36 \text{ e \AA}^{-3}$
0 restraints	$\Delta\rho_{\text{min}} = -0.25 \text{ e \AA}^{-3}$
Primary atom site location: dual	

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
N1	0.80689 (8)	0.44759 (4)	0.29624 (8)	0.0207 (2)
N2	0.31192 (8)	0.44924 (4)	0.29350 (8)	0.0202 (2)
C1	0.75073 (11)	0.21628 (5)	-0.18817 (10)	0.0246 (3)
C2	0.86438 (11)	0.22701 (6)	-0.09675 (11)	0.0270 (3)
C3	0.77687 (9)	0.33326 (4)	0.07952 (8)	0.0154 (2)
C4	0.68627 (9)	0.35885 (4)	0.08718 (9)	0.0164 (2)
C5	0.69507 (9)	0.39534 (4)	0.15909 (9)	0.0170 (2)
C6	0.79726 (9)	0.40888 (4)	0.22913 (8)	0.0163 (2)
C7	0.88796 (9)	0.38137 (4)	0.22642 (9)	0.0172 (2)
C8	0.87696 (9)	0.34536 (4)	0.15272 (9)	0.0168 (2)
C9	0.71200 (11)	0.47718 (5)	0.29057 (10)	0.0219 (3)
C10	0.66716 (10)	0.50871 (4)	0.19573 (9)	0.0191 (2)
C11	0.73030 (11)	0.52328 (5)	0.13796 (11)	0.0262 (3)
C12	0.68691 (11)	0.55325 (5)	0.05303 (12)	0.0286 (3)
C13	0.58003 (11)	0.56903 (5)	0.02561 (11)	0.0271 (3)
C14	0.51617 (11)	0.55469 (6)	0.08218 (12)	0.0330 (3)
C15	0.55940 (11)	0.52455 (6)	0.16659 (11)	0.0275 (3)
C16	0.91324 (10)	0.46443 (5)	0.36173 (9)	0.0199 (2)
C17	0.95759 (10)	0.43697 (5)	0.46178 (9)	0.0199 (2)
C18	0.89638 (11)	0.43253 (6)	0.52622 (10)	0.0286 (3)
C19	0.93807 (14)	0.40840 (6)	0.61920 (11)	0.0366 (4)
C20	1.04145 (16)	0.38851 (6)	0.64884 (12)	0.0429 (4)
C21	1.10342 (15)	0.39246 (7)	0.58564 (12)	0.0426 (4)
C22	1.06112 (12)	0.41662 (6)	0.49229 (11)	0.0310 (3)
C23	0.25860 (10)	0.20807 (5)	-0.16977 (10)	0.0218 (3)
C24	0.34621 (10)	0.25254 (5)	-0.14500 (10)	0.0214 (3)
C25	0.28131 (9)	0.33029 (4)	0.08712 (9)	0.0159 (2)
C26	0.38140 (9)	0.34356 (5)	0.15898 (9)	0.0173 (2)
C27	0.39249 (9)	0.38135 (5)	0.22885 (9)	0.0172 (2)
C28	0.30168 (9)	0.40946 (4)	0.22909 (8)	0.0163 (2)
C29	0.19968 (9)	0.39532 (5)	0.16005 (9)	0.0174 (2)
C30	0.19095 (9)	0.35701 (4)	0.09171 (9)	0.0165 (2)
C31	0.21668 (11)	0.47828 (5)	0.28924 (10)	0.0217 (3)
C32	0.16852 (10)	0.50898 (4)	0.19360 (9)	0.0184 (2)
C33	0.06146 (11)	0.52514 (6)	0.16785 (11)	0.0277 (3)
C34	0.01589 (11)	0.55504 (6)	0.08342 (12)	0.0338 (3)
C35	0.07650 (11)	0.56851 (5)	0.02323 (11)	0.0275 (3)
C36	0.18253 (11)	0.55209 (5)	0.04685 (11)	0.0263 (3)
C37	0.22841 (10)	0.52246 (5)	0.13193 (10)	0.0238 (3)

C38	0.41834 (10)	0.46511 (5)	0.36040 (9)	0.0206 (2)
C39	0.46056 (10)	0.43727 (4)	0.46019 (9)	0.0191 (2)
C40	0.39592 (11)	0.43183 (5)	0.52134 (10)	0.0240 (3)
C41	0.43545 (13)	0.40825 (6)	0.61522 (11)	0.0301 (3)
C42	0.54040 (13)	0.38950 (6)	0.64855 (11)	0.0344 (3)
C43	0.60498 (12)	0.39415 (6)	0.58822 (12)	0.0327 (3)
C44	0.56558 (11)	0.41789 (5)	0.49431 (11)	0.0258 (3)
B1	0.81698 (12)	0.27055 (6)	-0.18993 (12)	0.0267 (3)
B2	0.67398 (12)	0.26766 (6)	-0.22070 (11)	0.0241 (3)
B3	0.64289 (12)	0.22044 (6)	-0.14670 (12)	0.0244 (3)
B4	0.76566 (14)	0.19313 (6)	-0.06946 (12)	0.0279 (3)
B5	0.84362 (12)	0.23900 (6)	0.01495 (11)	0.0246 (3)
B6	0.87445 (11)	0.28651 (6)	-0.05933 (12)	0.0236 (3)
B7	0.75088 (12)	0.31360 (6)	-0.13680 (11)	0.0215 (3)
B8	0.64304 (11)	0.28275 (6)	-0.10971 (10)	0.0201 (3)
B9	0.69944 (12)	0.23639 (6)	-0.01608 (11)	0.0222 (3)
B10	0.76666 (10)	0.29471 (5)	-0.00781 (10)	0.0162 (2)
B11	0.36914 (12)	0.20502 (6)	-0.05926 (12)	0.0228 (3)
B12	0.23609 (12)	0.18860 (6)	-0.06331 (12)	0.0234 (3)
B13	0.14027 (11)	0.22585 (6)	-0.15496 (11)	0.0231 (3)
B14	0.21202 (12)	0.26605 (6)	-0.21025 (11)	0.0238 (3)
B15	0.29526 (12)	0.30483 (6)	-0.11260 (11)	0.0221 (3)
B16	0.39070 (11)	0.26741 (6)	-0.01958 (11)	0.0208 (3)
B17	0.31821 (11)	0.22730 (5)	0.03513 (11)	0.0198 (3)
B18	0.17675 (11)	0.24009 (5)	-0.02374 (11)	0.0193 (3)
B19	0.16218 (11)	0.28806 (6)	-0.11484 (11)	0.0206 (3)
B20	0.27214 (10)	0.28968 (5)	0.00406 (10)	0.0160 (2)
H1X	0.8580 (12)	0.2750 (6)	-0.2448 (12)	0.032 (4)*
H2X	0.6239 (12)	0.2718 (6)	-0.2989 (12)	0.030 (4)*
H3X	0.5742 (13)	0.1967 (6)	-0.1808 (13)	0.038 (5)*
H4X	0.7798 (12)	0.1543 (6)	-0.0584 (12)	0.036 (4)*
H5X	0.9079 (12)	0.2265 (6)	0.0857 (12)	0.030 (4)*
H6X	0.9525 (12)	0.3046 (6)	-0.0336 (12)	0.033 (4)*
H7X	0.7472 (11)	0.3518 (6)	-0.1578 (11)	0.028 (4)*
H8X	0.5659 (11)	0.3021 (5)	-0.1150 (11)	0.022 (4)*
H9X	0.6610 (11)	0.2233 (6)	0.0401 (11)	0.027 (4)*
H11X	0.4329 (11)	0.1785 (6)	-0.0585 (11)	0.026 (4)*
H12X	0.2193 (13)	0.1499 (6)	-0.0566 (12)	0.039 (5)*
H13X	0.0655 (12)	0.2102 (6)	-0.2046 (12)	0.031 (4)*
H14X	0.1897 (11)	0.2745 (5)	-0.2911 (11)	0.026 (4)*
H15X	0.3223 (12)	0.3398 (6)	-0.1329 (11)	0.028 (4)*
H16X	0.4741 (12)	0.2797 (6)	0.0143 (12)	0.033 (4)*
H17X	0.3546 (12)	0.2139 (6)	0.1164 (11)	0.029 (4)*
H18X	0.1184 (12)	0.2351 (6)	0.0184 (12)	0.030 (4)*
H19X	0.0949 (11)	0.3153 (6)	-0.1341 (11)	0.027 (4)*
H1	0.7538 (12)	0.1918 (6)	-0.2379 (12)	0.036 (4)*
H2	0.9255 (13)	0.2086 (7)	-0.1008 (13)	0.042 (5)*
H4	0.6116 (11)	0.3516 (5)	0.0389 (11)	0.021 (4)*

H5	0.6312 (12)	0.4128 (6)	0.1600 (12)	0.025 (4)*
H7	0.9619 (11)	0.3872 (6)	0.2760 (11)	0.023 (4)*
H8	0.9449 (11)	0.3278 (5)	0.1528 (11)	0.019 (4)*
H9A	0.6543 (11)	0.4553 (5)	0.2997 (11)	0.018 (3)*
H9B	0.7361 (12)	0.4983 (6)	0.3542 (12)	0.028 (4)*
H11	0.8060 (14)	0.5123 (6)	0.1576 (13)	0.039 (5)*
H12	0.7323 (14)	0.5641 (7)	0.0159 (14)	0.045 (5)*
H13	0.5513 (13)	0.5901 (6)	−0.0343 (13)	0.034 (4)*
H14	0.4381 (17)	0.5653 (7)	0.0618 (16)	0.059 (6)*
H15	0.5134 (13)	0.5139 (6)	0.2062 (13)	0.035 (4)*
H16A	0.9674 (11)	0.4625 (5)	0.3246 (11)	0.020 (4)*
H16B	0.9041 (12)	0.5000 (6)	0.3739 (12)	0.025 (4)*
H18	0.8217 (13)	0.4463 (6)	0.5087 (13)	0.034 (4)*
H19	0.8947 (14)	0.4055 (7)	0.6620 (14)	0.043 (5)*
H20	1.0707 (16)	0.3727 (8)	0.7129 (16)	0.061 (6)*
H21	1.1814 (17)	0.3786 (8)	0.6078 (16)	0.067 (6)*
H22	1.1078 (15)	0.4198 (7)	0.4462 (15)	0.050 (5)*
H23	0.2607 (12)	0.1850 (6)	−0.2226 (12)	0.028 (4)*
H24	0.3953 (12)	0.2530 (6)	−0.1860 (12)	0.028 (4)*
H26	0.4469 (11)	0.3260 (5)	0.1611 (11)	0.020 (4)*
H27	0.4639 (11)	0.3874 (5)	0.2778 (11)	0.019 (4)*
H29	0.1364 (12)	0.4140 (6)	0.1593 (11)	0.024 (4)*
H30	0.1154 (11)	0.3494 (5)	0.0421 (11)	0.022 (4)*
H31A	0.1579 (12)	0.4560 (6)	0.2987 (12)	0.027 (4)*
H31B	0.2407 (11)	0.5004 (6)	0.3487 (12)	0.025 (4)*
H33	0.0171 (13)	0.5149 (6)	0.2090 (13)	0.037 (5)*
H34	−0.0609 (16)	0.5670 (7)	0.0671 (15)	0.051 (5)*
H35	0.0450 (13)	0.5902 (6)	−0.0366 (13)	0.036 (5)*
H36	0.2273 (14)	0.5614 (6)	0.0073 (14)	0.040 (5)*
H37	0.3018 (14)	0.5112 (6)	0.1469 (13)	0.039 (5)*
H38A	0.4750 (11)	0.4635 (6)	0.3239 (11)	0.022 (4)*
H38B	0.4126 (11)	0.5004 (6)	0.3745 (11)	0.024 (4)*
H40	0.3188 (13)	0.4444 (6)	0.5003 (12)	0.031 (4)*
H41	0.3875 (14)	0.4054 (7)	0.6553 (14)	0.045 (5)*
H42	0.5671 (14)	0.3733 (7)	0.7151 (14)	0.047 (5)*
H43	0.6804 (14)	0.3819 (7)	0.6116 (13)	0.040 (5)*
H44	0.6132 (13)	0.4216 (6)	0.4507 (13)	0.034 (4)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0218 (5)	0.0188 (5)	0.0181 (5)	0.0044 (4)	0.0019 (4)	−0.0056 (4)
N2	0.0224 (5)	0.0184 (5)	0.0160 (5)	0.0045 (4)	0.0013 (4)	−0.0040 (4)
C1	0.0259 (6)	0.0271 (7)	0.0219 (6)	−0.0025 (5)	0.0094 (5)	−0.0116 (5)
C2	0.0216 (6)	0.0330 (8)	0.0258 (7)	0.0032 (5)	0.0070 (5)	−0.0112 (6)
C3	0.0167 (5)	0.0158 (6)	0.0129 (5)	0.0002 (4)	0.0040 (4)	−0.0003 (4)
C4	0.0162 (5)	0.0160 (6)	0.0158 (5)	0.0000 (4)	0.0037 (4)	0.0002 (4)
C5	0.0169 (5)	0.0167 (6)	0.0169 (5)	0.0027 (4)	0.0049 (4)	−0.0003 (4)

C6	0.0198 (5)	0.0156 (6)	0.0123 (5)	0.0011 (4)	0.0037 (4)	0.0003 (4)
C7	0.0174 (5)	0.0184 (6)	0.0140 (5)	0.0011 (4)	0.0029 (4)	-0.0014 (4)
C8	0.0159 (5)	0.0183 (6)	0.0151 (5)	0.0017 (4)	0.0037 (4)	-0.0004 (4)
C9	0.0266 (6)	0.0218 (6)	0.0172 (6)	0.0073 (5)	0.0075 (5)	-0.0004 (5)
C10	0.0222 (5)	0.0156 (6)	0.0182 (6)	0.0017 (4)	0.0050 (4)	-0.0017 (5)
C11	0.0224 (6)	0.0281 (7)	0.0299 (7)	0.0034 (5)	0.0109 (5)	0.0053 (6)
C12	0.0297 (7)	0.0278 (7)	0.0319 (7)	-0.0003 (6)	0.0151 (6)	0.0070 (6)
C13	0.0302 (7)	0.0231 (7)	0.0261 (7)	-0.0007 (5)	0.0069 (5)	0.0079 (6)
C14	0.0231 (6)	0.0370 (9)	0.0375 (8)	0.0051 (6)	0.0081 (6)	0.0150 (7)
C15	0.0238 (6)	0.0313 (8)	0.0295 (7)	0.0047 (5)	0.0115 (5)	0.0092 (6)
C16	0.0248 (6)	0.0166 (6)	0.0161 (5)	-0.0012 (5)	0.0038 (5)	-0.0012 (5)
C17	0.0244 (6)	0.0165 (6)	0.0162 (5)	-0.0021 (5)	0.0035 (5)	-0.0035 (5)
C18	0.0268 (6)	0.0369 (8)	0.0203 (6)	-0.0060 (6)	0.0055 (5)	-0.0013 (6)
C19	0.0473 (9)	0.0419 (10)	0.0201 (7)	-0.0125 (7)	0.0106 (6)	0.0007 (6)
C20	0.0665 (11)	0.0335 (9)	0.0209 (7)	0.0075 (8)	0.0042 (7)	0.0065 (7)
C21	0.0522 (10)	0.0435 (10)	0.0262 (7)	0.0232 (8)	0.0051 (7)	0.0037 (7)
C22	0.0353 (7)	0.0334 (8)	0.0229 (7)	0.0122 (6)	0.0079 (6)	-0.0010 (6)
C23	0.0238 (6)	0.0220 (6)	0.0215 (6)	-0.0043 (5)	0.0099 (5)	-0.0083 (5)
C24	0.0225 (6)	0.0229 (6)	0.0220 (6)	-0.0044 (5)	0.0115 (5)	-0.0054 (5)
C25	0.0183 (5)	0.0157 (6)	0.0138 (5)	0.0000 (4)	0.0054 (4)	-0.0004 (4)
C26	0.0172 (5)	0.0191 (6)	0.0157 (5)	0.0014 (4)	0.0055 (4)	-0.0010 (5)
C27	0.0174 (5)	0.0182 (6)	0.0142 (5)	0.0000 (4)	0.0031 (4)	-0.0010 (4)
C28	0.0212 (5)	0.0143 (5)	0.0123 (5)	0.0015 (4)	0.0043 (4)	0.0011 (4)
C29	0.0175 (5)	0.0171 (6)	0.0171 (5)	0.0027 (4)	0.0051 (4)	0.0005 (4)
C30	0.0174 (5)	0.0164 (6)	0.0149 (5)	-0.0007 (4)	0.0041 (4)	-0.0007 (4)
C31	0.0291 (6)	0.0199 (6)	0.0160 (6)	0.0072 (5)	0.0072 (5)	-0.0004 (5)
C32	0.0219 (5)	0.0152 (6)	0.0169 (5)	0.0019 (4)	0.0050 (4)	-0.0004 (4)
C33	0.0251 (6)	0.0312 (8)	0.0303 (7)	0.0054 (5)	0.0138 (6)	0.0091 (6)
C34	0.0231 (6)	0.0385 (9)	0.0386 (8)	0.0086 (6)	0.0087 (6)	0.0157 (7)
C35	0.0271 (6)	0.0246 (7)	0.0274 (7)	0.0014 (5)	0.0047 (5)	0.0088 (6)
C36	0.0276 (6)	0.0262 (7)	0.0265 (7)	0.0006 (5)	0.0109 (5)	0.0071 (6)
C37	0.0210 (6)	0.0245 (7)	0.0259 (6)	0.0023 (5)	0.0078 (5)	0.0035 (5)
C38	0.0262 (6)	0.0160 (6)	0.0165 (5)	-0.0019 (5)	0.0032 (5)	-0.0026 (5)
C39	0.0221 (5)	0.0156 (6)	0.0160 (5)	-0.0024 (4)	0.0016 (4)	-0.0035 (4)
C40	0.0255 (6)	0.0254 (7)	0.0193 (6)	-0.0009 (5)	0.0052 (5)	0.0000 (5)
C41	0.0387 (8)	0.0306 (8)	0.0194 (6)	-0.0033 (6)	0.0077 (6)	0.0020 (6)
C42	0.0460 (9)	0.0274 (8)	0.0205 (7)	0.0012 (6)	-0.0015 (6)	0.0039 (6)
C43	0.0308 (7)	0.0274 (8)	0.0300 (7)	0.0067 (6)	-0.0029 (6)	-0.0005 (6)
C44	0.0241 (6)	0.0246 (7)	0.0254 (7)	0.0016 (5)	0.0041 (5)	-0.0035 (5)
B1	0.0263 (7)	0.0345 (9)	0.0227 (7)	-0.0052 (6)	0.0128 (6)	-0.0079 (6)
B2	0.0250 (7)	0.0307 (8)	0.0160 (6)	-0.0014 (6)	0.0061 (5)	-0.0045 (6)
B3	0.0242 (7)	0.0263 (8)	0.0229 (7)	-0.0064 (6)	0.0081 (6)	-0.0082 (6)
B4	0.0370 (8)	0.0205 (8)	0.0247 (7)	0.0018 (6)	0.0084 (6)	-0.0042 (6)
B5	0.0269 (7)	0.0243 (8)	0.0202 (7)	0.0056 (6)	0.0048 (6)	-0.0036 (6)
B6	0.0193 (6)	0.0296 (8)	0.0226 (7)	-0.0025 (6)	0.0077 (5)	-0.0082 (6)
B7	0.0249 (6)	0.0238 (7)	0.0163 (6)	-0.0028 (6)	0.0074 (5)	-0.0008 (5)
B8	0.0170 (6)	0.0257 (7)	0.0165 (6)	-0.0007 (5)	0.0042 (5)	-0.0051 (5)
B9	0.0277 (7)	0.0204 (7)	0.0202 (6)	-0.0037 (6)	0.0103 (6)	-0.0026 (6)

B10	0.0154 (5)	0.0173 (6)	0.0157 (6)	-0.0002 (5)	0.0050 (5)	-0.0020 (5)
B11	0.0217 (6)	0.0224 (7)	0.0250 (7)	0.0011 (5)	0.0089 (5)	-0.0039 (6)
B12	0.0291 (7)	0.0180 (7)	0.0252 (7)	-0.0028 (6)	0.0121 (6)	-0.0040 (6)
B13	0.0196 (6)	0.0295 (8)	0.0199 (6)	-0.0045 (6)	0.0062 (5)	-0.0078 (6)
B14	0.0279 (7)	0.0267 (8)	0.0165 (6)	0.0005 (6)	0.0069 (5)	-0.0023 (6)
B15	0.0301 (7)	0.0197 (7)	0.0195 (6)	-0.0017 (6)	0.0122 (6)	-0.0015 (6)
B16	0.0191 (6)	0.0245 (7)	0.0197 (6)	-0.0029 (5)	0.0076 (5)	-0.0064 (6)
B17	0.0201 (6)	0.0190 (7)	0.0196 (6)	0.0019 (5)	0.0058 (5)	-0.0003 (5)
B18	0.0190 (6)	0.0210 (7)	0.0188 (6)	-0.0029 (5)	0.0074 (5)	-0.0038 (5)
B19	0.0216 (6)	0.0238 (7)	0.0157 (6)	0.0035 (5)	0.0053 (5)	-0.0012 (5)
B20	0.0162 (5)	0.0168 (6)	0.0148 (6)	-0.0001 (5)	0.0050 (5)	-0.0017 (5)

Geometric parameters (Å, °)

N1—C6	1.3831 (15)	C33—C34	1.3875 (19)
N1—C9	1.4529 (15)	C33—H33	0.980 (17)
N1—C16	1.4536 (15)	C34—C35	1.377 (2)
N2—C28	1.3809 (15)	C34—H34	1.002 (19)
N2—C38	1.4525 (15)	C35—C36	1.3813 (19)
N2—C31	1.4533 (16)	C35—H35	0.990 (17)
C1—C2	1.6231 (18)	C36—C37	1.3903 (18)
C1—B2	1.687 (2)	C36—H36	0.959 (18)
C1—B3	1.690 (2)	C37—H37	0.957 (17)
C1—B1	1.711 (2)	C38—C39	1.5137 (17)
C1—B4	1.716 (2)	C38—H38A	1.025 (14)
C1—H1	0.968 (17)	C38—H38B	0.987 (16)
C2—B6	1.688 (2)	C39—C40	1.3893 (18)
C2—B5	1.695 (2)	C39—C44	1.3917 (17)
C2—B1	1.710 (2)	C40—C41	1.3899 (19)
C2—B4	1.722 (2)	C40—H40	1.007 (15)
C2—H2	0.957 (17)	C41—C42	1.384 (2)
C3—C8	1.3987 (15)	C41—H41	0.968 (18)
C3—C4	1.4026 (15)	C42—C43	1.377 (2)
C3—B10	1.5751 (17)	C42—H42	0.977 (19)
C4—C5	1.3851 (16)	C43—C44	1.392 (2)
C4—H4	0.999 (14)	C43—H43	0.984 (17)
C5—C6	1.4100 (16)	C44—H44	1.007 (17)
C5—H5	0.961 (15)	B1—B7	1.754 (2)
C6—C7	1.4078 (16)	B1—B2	1.767 (2)
C7—C8	1.3889 (16)	B1—B6	1.772 (2)
C7—H7	0.992 (14)	B1—H1X	1.074 (15)
C8—H8	1.004 (14)	B2—B8	1.769 (2)
C9—C10	1.5167 (17)	B2—B7	1.772 (2)
C9—H9A	0.998 (14)	B2—B3	1.773 (2)
C9—H9B	1.013 (16)	B2—H2X	1.072 (15)
C10—C11	1.3849 (18)	B3—B4	1.764 (2)
C10—C15	1.3915 (17)	B3—B8	1.767 (2)
C11—C12	1.3897 (19)	B3—B9	1.772 (2)

C11—H11	0.976 (17)	B3—H3X	1.076 (16)
C12—C13	1.3818 (19)	B4—B9	1.761 (2)
C12—H12	0.951 (19)	B4—B5	1.778 (2)
C13—C14	1.378 (2)	B4—H4X	1.073 (17)
C13—H13	0.977 (17)	B5—B6	1.780 (2)
C14—C15	1.3870 (19)	B5—B9	1.781 (2)
C14—H14	1.00 (2)	B5—B10	1.783 (2)
C15—H15	0.983 (17)	B5—H5X	1.111 (15)
C16—C17	1.5133 (17)	B6—B7	1.770 (2)
C16—H16A	1.005 (14)	B6—B10	1.7893 (19)
C16—H16B	0.994 (16)	B6—H6X	1.077 (15)
C17—C22	1.3852 (18)	B7—B8	1.779 (2)
C17—C18	1.3888 (19)	B7—B10	1.811 (2)
C18—C19	1.389 (2)	B7—H7X	1.073 (15)
C18—H18	0.992 (16)	B8—B9	1.783 (2)
C19—C20	1.379 (2)	B8—B10	1.7837 (18)
C19—H19	0.951 (18)	B8—H8X	1.114 (14)
C20—C21	1.381 (3)	B9—B10	1.794 (2)
C20—H20	0.95 (2)	B9—H9X	1.118 (15)
C21—C22	1.394 (2)	B11—B17	1.764 (2)
C21—H21	1.03 (2)	B11—B12	1.770 (2)
C22—H22	1.024 (19)	B11—B16	1.774 (2)
C23—C24	1.6165 (18)	B11—H11X	1.097 (14)
C23—B12	1.688 (2)	B12—B18	1.771 (2)
C23—B13	1.6905 (19)	B12—B13	1.773 (2)
C23—B14	1.713 (2)	B12—B17	1.773 (2)
C23—B11	1.722 (2)	B12—H12X	1.082 (17)
C23—H23	0.971 (16)	B13—B18	1.769 (2)
C24—B15	1.690 (2)	B13—B14	1.770 (2)
C24—B16	1.6937 (19)	B13—B19	1.771 (2)
C24—B11	1.714 (2)	B13—H13X	1.075 (15)
C24—B14	1.7177 (19)	B14—B19	1.765 (2)
C24—H24	0.988 (15)	B14—B15	1.774 (2)
C25—C26	1.3988 (15)	B14—H14X	1.088 (15)
C25—C30	1.4011 (16)	B15—B19	1.781 (2)
C25—B20	1.5723 (17)	B15—B16	1.782 (2)
C26—C27	1.3873 (16)	B15—B20	1.794 (2)
C26—H26	0.969 (14)	B15—H15X	1.082 (15)
C27—C28	1.4074 (16)	B16—B17	1.770 (2)
C27—H27	0.966 (14)	B16—B20	1.7866 (19)
C28—C29	1.4074 (16)	B16—H16X	1.081 (15)
C29—C30	1.3878 (16)	B17—B18	1.7815 (19)
C29—H29	0.964 (15)	B17—B20	1.800 (2)
C30—H30	1.016 (14)	B17—H17X	1.130 (15)
C31—C32	1.5168 (17)	B18—B19	1.782 (2)
C31—H31A	1.017 (15)	B18—B20	1.7838 (19)
C31—H31B	0.985 (16)	B18—H18X	1.111 (15)
C32—C37	1.3867 (18)	B19—B20	1.7906 (19)

C32—C33	1.3888 (17)	B19—H19X	1.109 (15)
C6—N1—C9	119.95 (10)	C1—B4—B5	103.63 (11)
C6—N1—C16	120.99 (10)	C2—B4—B5	57.90 (9)
C9—N1—C16	118.32 (10)	B9—B4—B5	60.42 (8)
C28—N2—C38	120.85 (10)	B3—B4—B5	108.02 (11)
C28—N2—C31	120.09 (10)	C1—B4—H4X	117.2 (9)
C38—N2—C31	118.89 (10)	C2—B4—H4X	116.8 (8)
C2—C1—B2	111.41 (11)	B9—B4—H4X	132.9 (8)
C2—C1—B3	111.34 (10)	B3—B4—H4X	125.3 (8)
B2—C1—B3	63.35 (9)	B5—B4—H4X	124.1 (9)
C2—C1—B1	61.64 (9)	C2—B5—B4	59.38 (9)
B2—C1—B1	62.64 (9)	C2—B5—B6	58.07 (9)
B3—C1—B1	115.29 (11)	B4—B5—B6	108.25 (11)
C2—C1—B4	62.01 (9)	C2—B5—B9	104.32 (10)
B2—C1—B4	115.32 (11)	B4—B5—B9	59.29 (9)
B3—C1—B4	62.38 (9)	B6—B5—B9	108.14 (10)
B1—C1—B4	115.66 (11)	C2—B5—B10	104.59 (11)
C2—C1—H1	116.0 (9)	B4—B5—B10	108.27 (10)
B2—C1—H1	122.4 (10)	B6—B5—B10	60.29 (8)
B3—C1—H1	121.7 (9)	B9—B5—B10	60.44 (8)
B1—C1—H1	116.0 (10)	C2—B5—H5X	116.8 (8)
B4—C1—H1	114.7 (10)	B4—B5—H5X	117.8 (8)
C1—C2—B6	111.71 (11)	B6—B5—H5X	119.1 (8)
C1—C2—B5	111.75 (10)	B9—B5—H5X	128.5 (8)
B6—C2—B5	63.48 (9)	B10—B5—H5X	128.9 (8)
C1—C2—B1	61.71 (9)	C2—B6—B7	104.38 (10)
B6—C2—B1	62.87 (9)	C2—B6—B1	59.16 (9)
B5—C2—B1	115.77 (11)	B7—B6—B1	59.35 (9)
C1—C2—B4	61.65 (9)	C2—B6—B5	58.45 (9)
B6—C2—B4	115.45 (10)	B7—B6—B5	108.64 (10)
B5—C2—B4	62.72 (9)	B1—B6—B5	108.56 (11)
B1—C2—B4	115.42 (10)	C2—B6—B10	104.59 (10)
C1—C2—H2	114.7 (10)	B7—B6—B10	61.17 (8)
B6—C2—H2	122.5 (10)	B1—B6—B10	108.78 (10)
B5—C2—H2	122.9 (10)	B5—B6—B10	59.94 (8)
B1—C2—H2	114.4 (10)	C2—B6—H6X	120.5 (8)
B4—C2—H2	115.4 (10)	B7—B6—H6X	125.3 (9)
C8—C3—C4	115.51 (10)	B1—B6—H6X	118.8 (8)
C8—C3—B10	122.41 (10)	B5—B6—H6X	120.5 (8)
C4—C3—B10	122.05 (10)	B10—B6—H6X	126.0 (8)
C5—C4—C3	122.67 (11)	B1—B7—B6	60.40 (9)
C5—C4—H4	117.1 (8)	B1—B7—B2	60.14 (9)
C3—C4—H4	120.3 (8)	B6—B7—B2	107.92 (11)
C4—C5—C6	121.14 (11)	B1—B7—B8	107.94 (11)
C4—C5—H5	120.0 (9)	B6—B7—B8	107.22 (11)
C6—C5—H5	118.8 (9)	B2—B7—B8	59.75 (8)
N1—C6—C7	122.21 (10)	B1—B7—B10	108.64 (11)

N1—C6—C5	121.11 (11)	B6—B7—B10	59.95 (8)
C7—C6—C5	116.68 (11)	B2—B7—B10	107.82 (10)
C8—C7—C6	120.81 (10)	B8—B7—B10	59.57 (7)
C8—C7—H7	117.5 (9)	B1—B7—H7X	120.8 (8)
C6—C7—H7	121.7 (9)	B6—B7—H7X	121.1 (8)
C7—C8—C3	122.97 (11)	B2—B7—H7X	122.4 (8)
C7—C8—H8	117.3 (8)	B8—B7—H7X	123.0 (8)
C3—C8—H8	119.7 (8)	B10—B7—H7X	121.5 (8)
N1—C9—C10	115.63 (10)	B3—B8—B2	60.19 (9)
N1—C9—H9A	109.0 (8)	B3—B8—B7	108.24 (10)
C10—C9—H9A	111.0 (8)	B2—B8—B7	59.92 (8)
N1—C9—H9B	104.8 (8)	B3—B8—B9	59.86 (9)
C10—C9—H9B	111.0 (9)	B2—B8—B9	108.44 (10)
H9A—C9—H9B	104.7 (12)	B7—B8—B9	108.98 (10)
C11—C10—C15	118.56 (12)	B3—B8—B10	108.59 (10)
C11—C10—C9	122.37 (11)	B2—B8—B10	109.17 (9)
C15—C10—C9	119.06 (11)	B7—B8—B10	61.10 (8)
C10—C11—C12	120.60 (12)	B9—B8—B10	60.37 (8)
C10—C11—H11	119.2 (10)	B3—B8—H8X	121.2 (7)
C12—C11—H11	120.2 (10)	B2—B8—H8X	120.9 (7)
C13—C12—C11	120.15 (13)	B7—B8—H8X	121.4 (7)
C13—C12—H12	120.3 (11)	B9—B8—H8X	121.5 (7)
C11—C12—H12	119.5 (11)	B10—B8—H8X	121.4 (7)
C14—C13—C12	119.87 (13)	B4—B9—B3	59.91 (9)
C14—C13—H13	121.4 (9)	B4—B9—B5	60.29 (9)
C12—C13—H13	118.7 (9)	B3—B9—B5	107.57 (10)
C13—C14—C15	119.91 (13)	B4—B9—B8	107.61 (10)
C13—C14—H14	120.3 (12)	B3—B9—B8	59.62 (8)
C15—C14—H14	119.8 (12)	B5—B9—B8	107.02 (10)
C14—C15—C10	120.90 (13)	B4—B9—B10	108.59 (10)
C14—C15—H15	119.9 (10)	B3—B9—B10	107.96 (10)
C10—C15—H15	119.2 (10)	B5—B9—B10	59.84 (8)
N1—C16—C17	114.38 (10)	B8—B9—B10	59.82 (8)
N1—C16—H16A	110.3 (8)	B4—B9—H9X	118.6 (8)
C17—C16—H16A	109.3 (8)	B3—B9—H9X	120.6 (8)
N1—C16—H16B	105.6 (8)	B5—B9—H9X	121.6 (7)
C17—C16—H16B	110.6 (9)	B8—B9—H9X	124.3 (8)
H16A—C16—H16B	106.3 (12)	B10—B9—H9X	124.0 (8)
C22—C17—C18	118.44 (13)	C3—B10—B5	121.91 (10)
C22—C17—C16	120.78 (12)	C3—B10—B8	123.76 (10)
C18—C17—C16	120.77 (11)	B5—B10—B8	106.91 (10)
C19—C18—C17	120.81 (14)	C3—B10—B6	121.06 (10)
C19—C18—H18	116.9 (10)	B5—B10—B6	59.77 (9)
C17—C18—H18	122.3 (10)	B8—B10—B6	106.18 (9)
C20—C19—C18	120.15 (15)	C3—B10—B9	123.34 (10)
C20—C19—H19	120.3 (11)	B5—B10—B9	59.72 (8)
C18—C19—H19	119.6 (11)	B8—B10—B9	59.81 (8)
C19—C20—C21	119.86 (15)	B6—B10—B9	107.16 (10)

C19—C20—H20	120.3 (12)	C3—B10—B7	121.93 (11)
C21—C20—H20	119.8 (12)	B5—B10—B7	106.70 (10)
C20—C21—C22	119.78 (15)	B8—B10—B7	59.33 (8)
C20—C21—H21	120.5 (12)	B6—B10—B7	58.88 (8)
C22—C21—H21	119.7 (12)	B9—B10—B7	107.13 (9)
C17—C22—C21	120.96 (15)	C24—B11—C23	56.13 (8)
C17—C22—H22	119.6 (11)	C24—B11—B17	103.81 (10)
C21—C22—H22	119.4 (11)	C23—B11—B17	103.82 (10)
C24—C23—B12	111.31 (10)	C24—B11—B12	103.06 (10)
C24—C23—B13	111.54 (10)	C23—B11—B12	57.78 (8)
B12—C23—B13	63.30 (9)	B17—B11—B12	60.23 (8)
C24—C23—B14	62.04 (8)	C24—B11—B16	58.06 (8)
B12—C23—B14	115.49 (10)	C23—B11—B16	103.31 (10)
B13—C23—B14	62.67 (9)	B17—B11—B16	60.05 (8)
C24—C23—B11	61.69 (8)	B12—B11—B16	107.56 (10)
B12—C23—B11	62.56 (9)	C24—B11—H11X	117.1 (8)
B13—C23—B11	115.38 (10)	C23—B11—H11X	116.4 (8)
B14—C23—B11	115.88 (10)	B17—B11—H11X	133.6 (8)
C24—C23—H23	116.5 (9)	B12—B11—H11X	124.3 (8)
B12—C23—H23	121.2 (9)	B16—B11—H11X	125.7 (8)
B13—C23—H23	122.1 (9)	C23—B12—B11	59.66 (8)
B14—C23—H23	116.0 (9)	C23—B12—B18	104.29 (11)
B11—C23—H23	114.7 (9)	B11—B12—B18	108.33 (10)
C23—C24—B15	111.86 (10)	C23—B12—B13	58.43 (8)
C23—C24—B16	111.84 (10)	B11—B12—B13	108.97 (11)
B15—C24—B16	63.56 (9)	B18—B12—B13	59.89 (8)
C23—C24—B11	62.17 (8)	C23—B12—B17	104.83 (10)
B15—C24—B11	116.04 (10)	B11—B12—B17	59.69 (8)
B16—C24—B11	62.75 (9)	B18—B12—B17	60.35 (8)
C23—C24—B14	61.73 (8)	B13—B12—B17	108.40 (11)
B15—C24—B14	62.72 (9)	C23—B12—H12X	118.6 (9)
B16—C24—B14	115.56 (10)	B11—B12—H12X	117.7 (8)
B11—C24—B14	116.02 (10)	B18—B12—H12X	128.1 (8)
C23—C24—H24	115.6 (9)	B13—B12—H12X	120.1 (9)
B15—C24—H24	122.0 (9)	B17—B12—H12X	126.5 (9)
B16—C24—H24	121.8 (9)	C23—B13—B18	104.26 (10)
B11—C24—H24	114.5 (9)	C23—B13—B14	59.27 (8)
B14—C24—H24	115.5 (9)	B18—B13—B14	108.36 (10)
C26—C25—C30	115.45 (11)	C23—B13—B19	104.66 (10)
C26—C25—B20	122.01 (10)	B18—B13—B19	60.46 (8)
C30—C25—B20	122.50 (10)	B14—B13—B19	59.77 (9)
C27—C26—C25	123.06 (11)	C23—B13—B12	58.27 (8)
C27—C26—H26	117.3 (9)	B18—B13—B12	60.01 (8)
C25—C26—H26	119.7 (8)	B14—B13—B12	108.53 (10)
C26—C27—C28	120.74 (11)	B19—B13—B12	108.45 (10)
C26—C27—H27	118.1 (9)	C23—B13—H13X	118.4 (8)
C28—C27—H27	121.1 (9)	B18—B13—H13X	128.5 (8)
N2—C28—C27	121.73 (10)	B14—B13—H13X	117.3 (8)

N2—C28—C29	121.39 (11)	B19—B13—H13X	126.4 (8)
C27—C28—C29	116.88 (11)	B12—B13—H13X	120.4 (8)
C30—C29—C28	120.98 (11)	C23—B14—C24	56.23 (8)
C30—C29—H29	120.8 (9)	C23—B14—B19	104.03 (10)
C28—C29—H29	118.2 (9)	C24—B14—B19	103.81 (10)
C29—C30—C25	122.75 (11)	C23—B14—B13	58.05 (8)
C29—C30—H30	117.2 (8)	C24—B14—B13	103.26 (10)
C25—C30—H30	120.0 (8)	B19—B14—B13	60.16 (9)
N2—C31—C32	115.53 (10)	C23—B14—B15	103.58 (10)
N2—C31—H31A	109.9 (9)	C24—B14—B15	57.88 (8)
C32—C31—H31A	109.3 (9)	B19—B14—B15	60.44 (8)
N2—C31—H31B	105.6 (8)	B13—B14—B15	108.00 (10)
C32—C31—H31B	108.9 (9)	C23—B14—H14X	118.4 (8)
H31A—C31—H31B	107.2 (12)	C24—B14—H14X	117.7 (8)
C37—C32—C33	118.65 (12)	B19—B14—H14X	131.9 (8)
C37—C32—C31	122.31 (11)	B13—B14—H14X	125.5 (8)
C33—C32—C31	119.02 (11)	B15—B14—H14X	123.5 (8)
C34—C33—C32	120.78 (13)	C24—B15—B14	59.40 (8)
C34—C33—H33	119.5 (10)	C24—B15—B19	104.26 (10)
C32—C33—H33	119.7 (10)	B14—B15—B19	59.53 (8)
C35—C34—C33	120.01 (13)	C24—B15—B16	58.31 (8)
C35—C34—H34	120.4 (11)	B14—B15—B16	108.51 (11)
C33—C34—H34	119.6 (11)	B19—B15—B16	107.76 (10)
C34—C35—C36	119.92 (13)	C24—B15—B20	104.35 (10)
C34—C35—H35	120.6 (9)	B14—B15—B20	108.23 (10)
C36—C35—H35	119.5 (9)	B19—B15—B20	60.11 (8)
C35—C36—C37	120.03 (13)	B16—B15—B20	59.94 (8)
C35—C36—H36	121.9 (10)	C24—B15—H15X	118.4 (8)
C37—C36—H36	118.0 (10)	B14—B15—H15X	119.3 (8)
C32—C37—C36	120.59 (12)	B19—B15—H15X	128.2 (8)
C32—C37—H37	120.4 (11)	B16—B15—H15X	118.8 (8)
C36—C37—H37	119.0 (11)	B20—B15—H15X	127.2 (8)
N2—C38—C39	114.61 (10)	C24—B16—B17	104.38 (10)
N2—C38—H38A	111.0 (8)	C24—B16—B11	59.19 (8)
C39—C38—H38A	109.3 (8)	B17—B16—B11	59.67 (8)
N2—C38—H38B	107.2 (8)	C24—B16—B15	58.12 (8)
C39—C38—H38B	109.3 (9)	B17—B16—B15	108.69 (10)
H38A—C38—H38B	104.8 (12)	B11—B16—B15	108.57 (10)
C40—C39—C44	118.51 (12)	C24—B16—B20	104.53 (9)
C40—C39—C38	120.39 (11)	B17—B16—B20	60.81 (8)
C44—C39—C38	121.07 (12)	B11—B16—B20	108.88 (10)
C39—C40—C41	120.96 (13)	B15—B16—B20	60.36 (8)
C39—C40—H40	122.3 (9)	C24—B16—H16X	118.1 (8)
C41—C40—H40	116.7 (9)	B17—B16—H16X	127.1 (8)
C42—C41—C40	119.90 (14)	B11—B16—H16X	117.5 (8)
C42—C41—H41	122.1 (11)	B15—B16—H16X	119.7 (8)
C40—C41—H41	118.0 (11)	B20—B16—H16X	128.1 (8)
C43—C42—C41	119.77 (14)	B11—B17—B16	60.27 (8)

C43—C42—H42	121.5 (10)	B11—B17—B12	60.08 (8)
C41—C42—H42	118.8 (10)	B16—B17—B12	107.61 (10)
C42—C43—C44	120.38 (14)	B11—B17—B18	108.17 (10)
C42—C43—H43	120.8 (10)	B16—B17—B18	107.43 (10)
C44—C43—H43	118.8 (10)	B12—B17—B18	59.76 (8)
C39—C44—C43	120.48 (14)	B11—B17—B20	108.75 (10)
C39—C44—H44	119.2 (10)	B16—B17—B20	60.05 (8)
C43—C44—H44	120.3 (10)	B12—B17—B20	107.77 (10)
C2—B1—C1	56.65 (8)	B18—B17—B20	59.74 (8)
C2—B1—B7	104.16 (10)	B11—B17—H17X	119.9 (8)
C1—B1—B7	104.31 (10)	B16—B17—H17X	121.5 (8)
C2—B1—B2	103.74 (11)	B12—B17—H17X	121.7 (8)
C1—B1—B2	58.01 (9)	B18—B17—H17X	123.2 (7)
B7—B1—B2	60.43 (9)	B20—B17—H17X	122.5 (8)
C2—B1—B6	57.97 (9)	B13—B18—B12	60.10 (9)
C1—B1—B6	103.77 (11)	B13—B18—B17	108.20 (10)
B7—B1—B6	60.25 (8)	B12—B18—B17	59.89 (8)
B2—B1—B6	108.02 (10)	B13—B18—B19	59.84 (8)
C2—B1—H1X	118.5 (8)	B12—B18—B19	108.04 (10)
C1—B1—H1X	118.6 (9)	B17—B18—B19	108.53 (10)
B7—B1—H1X	130.9 (9)	B13—B18—B20	108.45 (10)
B2—B1—H1X	124.5 (8)	B12—B18—B20	108.60 (10)
B6—B1—H1X	124.1 (8)	B17—B18—B20	60.65 (8)
C1—B2—B1	59.35 (9)	B19—B18—B20	60.28 (8)
C1—B2—B8	104.12 (11)	B13—B18—H18X	121.5 (8)
B1—B2—B8	107.84 (10)	B12—B18—H18X	120.8 (8)
C1—B2—B7	104.55 (10)	B17—B18—H18X	121.1 (8)
B1—B2—B7	59.42 (8)	B19—B18—H18X	122.4 (8)
B8—B2—B7	60.34 (8)	B20—B18—H18X	121.8 (8)
C1—B2—B3	58.40 (9)	B14—B19—B13	60.07 (9)
B1—B2—B3	108.50 (11)	B14—B19—B15	60.03 (8)
B8—B2—B3	59.87 (9)	B13—B19—B15	107.60 (10)
B7—B2—B3	108.32 (10)	B14—B19—B18	107.98 (10)
C1—B2—H2X	117.7 (8)	B13—B19—B18	59.70 (8)
B1—B2—H2X	118.4 (8)	B15—B19—B18	107.58 (10)
B8—B2—H2X	128.6 (8)	B14—B19—B20	108.79 (10)
B7—B2—H2X	127.9 (8)	B13—B19—B20	108.03 (10)
B3—B2—H2X	119.1 (8)	B15—B19—B20	60.31 (8)
C1—B3—B4	59.55 (9)	B18—B19—B20	59.90 (8)
C1—B3—B8	104.06 (10)	B14—B19—H19X	120.2 (8)
B4—B3—B8	108.17 (10)	B13—B19—H19X	121.9 (8)
C1—B3—B9	104.97 (10)	B15—B19—H19X	121.2 (8)
B4—B3—B9	59.73 (9)	B18—B19—H19X	123.2 (8)
B8—B3—B9	60.52 (8)	B20—B19—H19X	122.0 (8)
C1—B3—B2	58.25 (9)	C25—B20—B18	124.41 (10)
B4—B3—B2	108.77 (11)	C25—B20—B16	120.92 (10)
B8—B3—B2	59.94 (9)	B18—B20—B16	106.62 (10)
B9—B3—B2	108.76 (10)	C25—B20—B19	122.50 (10)

C1—B3—H3X	118.2 (9)	B18—B20—B19	59.82 (8)
B4—B3—H3X	117.8 (9)	B16—B20—B19	107.14 (9)
B8—B3—H3X	128.5 (9)	C25—B20—B15	120.63 (10)
B9—B3—H3X	126.8 (9)	B18—B20—B15	106.94 (9)
B2—B3—H3X	119.6 (9)	B16—B20—B15	59.70 (8)
C1—B4—C2	56.34 (8)	B19—B20—B15	59.58 (8)
C1—B4—B9	104.33 (11)	C25—B20—B17	123.05 (10)
C2—B4—B9	104.06 (11)	B18—B20—B17	59.61 (8)
C1—B4—B3	58.07 (9)	B16—B20—B17	59.15 (8)
C2—B4—B3	103.42 (11)	B19—B20—B17	107.35 (10)
B9—B4—B3	60.36 (9)	B15—B20—B17	106.86 (10)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C1—H1...C3 ⁱ	0.968 (17)	2.742 (17)	3.6196 (17)	151.0 (13)
C1—H1...C4 ⁱ	0.968 (17)	2.676 (17)	3.5889 (17)	157.3 (12)
C1—H1...C5 ⁱ	0.968 (17)	2.740 (17)	3.6322 (18)	153.4 (12)
C1—H1...C6 ⁱ	0.968 (17)	2.860 (17)	3.7008 (18)	145.8 (13)
C1—H1...C7 ⁱ	0.968 (17)	2.797 (17)	3.6112 (18)	142.2 (12)
C1—H1...C8 ⁱ	0.968 (17)	2.745 (16)	3.5809 (18)	144.9 (12)
C23—H23...C25 ⁱ	0.971 (16)	2.780 (16)	3.6436 (17)	148.4 (12)
C23—H23...C26 ⁱ	0.971 (16)	2.741 (15)	3.5682 (17)	143.4 (11)
C23—H23...C27 ⁱ	0.971 (16)	2.724 (15)	3.5432 (18)	142.3 (11)
C23—H23...C28 ⁱ	0.971 (16)	2.749 (16)	3.6050 (18)	147.3 (12)
C23—H23...C29 ⁱ	0.971 (16)	2.681 (16)	3.5857 (18)	155.0 (12)
C23—H23...C30 ⁱ	0.971 (16)	2.690 (16)	3.5996 (17)	156.0 (12)
C16—H16 <i>B</i> ...C22 ⁱⁱ	0.994 (16)	2.869 (16)	3.769 (2)	151.0 (12)
C38—H38 <i>B</i> ...C44 ⁱⁱⁱ	0.987 (16)	2.825 (16)	3.7325 (19)	153.3 (12)

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