The structures of two scorpionates: thallium tetrakis(3-phenyl-1*H*-pyrazol-1-yl)borate and potassium tetrakis(3-cyclopropyl-1*H*-pyrazol-1-yl)borate

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The introduction of poly(1*H*-pyrazolyl)borate anions, better known as scorpionates, as negatively charged ligands for a great diversity of metal cations has had a tremendous influence in coordination chemistry. The structures of two salts of tetrakispyrazolylborate, namely [tetrakis(3-phenyl-1*H*-pyrazol-1-yl)borato]thallium(I), [Tl(C₃₆H₂₈BN₈)], and *catena*-poly[potassium- $[\mu_2$ -tetrakis(3-cyclopropyl-1*H*-pyrazol-1-yl)borato]], [K(C₂₄H₂₈BN₈)]_n, have been determined at 296 K in the monoclinic *P*2₁/*c* and *C*2/*c* space groups, respectively. In their crystal structures, the thallium salt presents discrete molecular motifs, while the potassium salt shows infinite polymeric chains. The ¹³C and ¹⁵N CPMAS (cross polarization magic angle spinning) NMR spectra of these compounds were recorded and the chemical shifts compared with theoretically calculated ones at the GIAO/B3LYP/6-311++G(d,p) level. Both techniques are complementary and mutually consistent.

1. Introduction

The introduction of scorpionates, *i.e.* poly(1*H*-pyrazolyl)borate anions (mainly tris), by Trofimenko in 1966 (Trofimenko, 1966) as negatively charged ligands for a great diversity of metal cations has had a tremendous influence in coordination chemistry, an influence that in spite of reviews (Trofimenko, 1972, 1993, 2004; Yap, 2013; Reglinski & Spicer, 2015; Sallmann & Limberg, 2015) and books (Trofimenko, 1999; West *et al.* 2008), is still insufficiently acknowledged. This paper aims to contribute to the recognition of Swiatoslaw (Jerry) Trofimenko as one of the most significant chemists of the 20th century. The chemistry of scorpionates has been mainly extended in three directions: (i) replacing the B–H atom of tris(1*H*-pyrazolyl)borate anions by B–*R* groups (Vitze *et al.*, 2016); (ii) replacing the pyrazole rings by other azoles, such as indazoles (Muñoz *et al.*, 2014; Kammerer & Rapenne, 2016), 1,2,4-triazoles (Janiak *et al.*, 1996) *etc.*; (iii) replacing the central B atom by other atoms, for example, C atoms, such as in neutral poly(1*H*-pyrazolyl)methanes (Juliá *et al.*, 1982; Pettinari & Pettinari, 2005; Alkorta *et al.*, 2013; Moreno-Lara *et al.*, 2015).

Our past contribution to this field has been centred mainly on the study of structural properties, using as cations ‘innocent’ ones such as K⁺ or Tl⁺, *via* negative-ion fast-atom bombardment mass spectra of pyrazolylborates (Aubagnac *et al.*, 1991); the structures of bis-, tris- and tetrakispyrazolyl-

Table 1
Experimental details.

	(1)	(2)
Crystal data		
Chemical formula	[Ti(C ₃₆ H ₂₈ BN ₈)]	[K(C ₂₄ H ₂₈ BN ₈)]
<i>M_r</i>	787.85	478.45
Crystal system, space group	Monoclinic, <i>P</i> ₂ /c	Monoclinic, <i>C</i> 2/c
Temperature (K)	296	296
<i>a</i> , <i>b</i> , <i>c</i> (Å)	9.9552 (6), 13.1693 (8), 24.7993 (14)	19.547 (8), 8.391 (3), 17.153 (8)
β (°)	91.463 (2)	117.111 (12)
<i>V</i> (Å ³)	3250.2 (3)	2504.2 (18)
<i>Z</i>	4	4
Radiation type	Cu <i>K</i> α	Cu <i>K</i> α
μ (mm ^{−1})	9.86	2.08
Crystal size (mm)	0.06 × 0.02 × 0.02	0.19 × 0.06 × 0.06
Data collection		
Diffractometer	Bruker APEXII CCD	Bruker APEXII CCD
Absorption correction	Multi-scan (<i>SADABS</i> ; Bruker, 2013)	Multi-scan (<i>SADABS</i> ; Bruker, 2013)
<i>T</i> _{min} , <i>T</i> _{max}	0.218, 0.754	0.511, 0.754
No. of measured, independent and observed [<i>I</i> > 2 σ (<i>I</i>)] reflections	45124, 6614, 5748	21173, 2126, 1361
<i>R</i> _{int}	0.045	0.085
(<i>sin</i> θ / λ) _{max} (Å ^{−1})	0.625	0.596
Refinement		
<i>R</i> [<i>F</i> ² > 2 σ (<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.029, 0.072, 1.07	0.051, 0.135, 1.03
No. of reflections	6614	2126
No. of parameters	470	155
No. of restraints	28	0
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained
$\Delta\rho_{\max}$, $\Delta\rho_{\min}$ (e Å ^{−3})	0.42, −1.27	0.14, −0.24

Computer programs: *APEX2* (Bruker, 2013), *SAINT* (Bruker, 2013), *SIR2014* (Burla *et al.*, 2015), *SHELXL2014* (Sheldrick, 2015), *Mercury* (Macrae *et al.*, 2008) and *ORTEP-3 for Windows* (Farrugia, 2012).

borates in the solid state by X-ray crystallography, and in solution by NMR (López *et al.*, 1990); determination of the protonation site of tetrakis(pyrazol-1-yl)borate in the solid

state and in solution (López *et al.*, 1994); structural characterization of paramagnetic octahedral homoscorpionate cobalt complexes by ¹H and ¹³C NMR spectroscopy and by FAB–mass spectrometry (Aubagnac *et al.*, 1995); the fluxional behaviour of molybdenum pyrazolylborates (Santa María *et al.*, 2007); several multinuclear magnetic resonance studies both in solution and in the solid state (López *et al.*, 1995; Sanz *et al.*, 1996; Claramunt *et al.*, 2004*a,b*), where, amongst other things, some ¹³C–¹¹B and ¹³C–²⁰⁵Tl residual coupling constants were measured. We also published a theoretical study of all possible pyrazolylborates derived from the parent pyrazole (Alkorta *et al.*, 2010).

The crystal structures of two salts of tetrakispyrazolylborate, namely [tetrakis(3-phenyl-1*H*-pyrazol-1-yl)borato]-

thallium(I), (1), and *catena*-poly[potassium- $[\mu_2$ -tetrakis(3-cyclopropyl-1*H*-pyrazol-1-yl)borato]], (2), have been determined and are presented here, together with a comparison of the ¹³C and ¹⁵N CPMAS (cross polarization magic angle spinning) NMR spectra of these compounds with theoretically calculations.

2. Experimental

2.1. Materials and crystallization

Compound (1) was prepared as described in López *et al.* (1995), while compound (2) is new and was obtained in a similar manner to other potassium scorpionates (Trofimenko, 1970). In both cases, crystals were grown from toluene.

2.2. Refinement

Crystal data, data collection and structure refinement details for (1) and (2) are summarized in Table 1. All H atoms in both structures were placed in calculated positions and constrained as riding, with C–H = 0.93 Å and *U*_{iso}(H) = 1.2*U*_{eq}(C) of the parent atom.

2.3. Computational details

The geometries of the title molecules have been fully optimized with the hybrid HF/DFT B3LYP (Becke, 1988, 1993; Lee *et al.*, 1988) computational method at the B3LYP/6-

Figure 1
The molecular structure of (a) compound (1) and (b) compound (2). Displacement ellipsoids are drawn at the 30% probability level for non-H atoms and H atoms are represented as spheres of 0.1 Å radii.

311++G(d,p) level (Ditchfield *et al.*, 1971; Frisch *et al.*, 1984). Frequency calculations have been carried out at the same computational level to verify that the structures obtained correspond to energetic minima. These geometries have been used for the calculations of the absolute chemical shieldings with the GIAO (gauge-including atomic orbital) method (Ditchfield, 1974; London, 1937). All calculations were carried out using the *GAUSSIAN09* package (Frisch *et al.*, 2009).

The following equations have been used to transform absolute shieldings into chemical shifts:

$\delta^1\text{H} = 31.0 - 0.97 \sigma^1\text{H}$ (reference TMS, 0.00 p.p.m.) (Silva *et al.*, 2008);

$\delta^{13}\text{C} = 175.7 - 0.963 \sigma^{13}\text{C}$ (reference TMS, 0.00 p.p.m.) (Blanco *et al.*, 2007);

$\delta^{15}\text{N} = -152.0 - 0.946 \sigma^{15}\text{N}$ (reference external neat MeNO_2 , 0.00 p.p.m.) (Blanco *et al.*, 2007).

Figure 2
The crystal packing along the *a* axis for compound (1). Benzene rings *d* (the disordered model has been omitted for clarity) are displayed in green and benzene rings *a* are displayed in yellow.

3. Results and discussion

3.1. Crystal and molecular structure

The X-ray crystal structure of compound (1) shows one molecule in the asymmetric unit (Fig. 1*a*). One of the four benzene rings (atoms C4–C9) is disordered over two positions, with a site-occupation factor of 0.60 (2) for the major-occupied site. The atoms in benzene ring *a* display large displacement parameters; however, we were unable to build a satisfactory disorder model for them. The benzene and pyrazole rings are not coplanar, displaying torsion angles (N1–C3–C4–C5) of -4.9 (5), -25.4 (5), -18.5 (4) and 6.6 (9)/ -30.9 (9)° for pyrazole derivatives *a*, *b*, *c* and the disorder models of *d*, respectively. The disordered benzene rings, *d*, and those with large displacement parameters, *a*, form tubes along the *a* axis (Fig. 2), suggesting a less compacted section in the crystal structure.

Compound (2) crystallizes with higher symmetry showing half a molecule in the asymmetric unit (Fig. 1*b*). The B and K atoms are on a twofold axis and the molecule grows through a crystallographic binary axis.

While compound (1) presents discrete molecules in its crystal structure, compound (2) displays infinite polymeric chains where every B atom is bonded to four pyrazole N atoms and every K atom is also coordinated to four pyrazole N atoms (Fig. 3).

3.2. Comparison with related scorpionates found in the CSD

X-ray crystal studies of scorpionates concern almost exclusively tris derivatives (4028 hits); the bis derivatives are not so common (284 hits) and the same is true for the tetrakis derivatives, where there are 171 hits corresponding to 164 compounds reported in the Cambridge Structural Database

Figure 3
Diagram showing (a) a polymer chain and (b) the crystal packing along the *b* axis for compound (2).

(CSD, Version 5.37, November 2015 update; Groom & Allen, 2014). For more details, see the *Supporting information*.

Each of the 164 tetrakispyrazolylborate structures in the CSD contains at least one of the 11 motifs represented in Fig. 4. In all cases, the N1 atoms of the pyrazole rings are coordinated to the B atom of the tetrakis derivative. The second N atom from the pyrazole rings, *i.e.* N2, is uncoordinated in motifs **A** and **B**, is coordinated with a metal atom in motif **E** and, for the remaining motifs, some of the N2 atoms are coordinated and some are not, depending on the motifs displayed.

In Table 2, we show the frequency of these motifs present in the analyzed crystal structures. The two most frequently appeared motifs (**C** and **D**) are those where two or three pyrazole rings from the scorpionate ligand are coordinated to the metal atom, leaving the remaining pyrazole rings free, and the coordination sphere of the metal atom is completed by a ligand different from the tetrakis scorpionate ligand.

Compound (1) contains the most frequently occurring motif (**D**), with the Tl^I atom tricoordinated to pyrazole N atoms. There is a thallium complex in the CSD (refcode JARRUQ; Adams *et al.*, 2005), where 3-phenylpyrazolyl is replaced by 3-pyridylpyrazolyl. This structure also shows the **D** motif; however, it is expanded into a two-dimensional coordination polymer due to the extra coordination of the Tl^I atom with the N atom of the pyridine rings.

Motifs **D**, **H**, **I**, **J** and **K** show the metal cations coordinated to three of the pyrazole rings in the scorpionate ligand. The B–N–N–*M* torsion angles observed for these tetrakis scorpionates are in most cases less than 30°, with two exceptions, *viz.* the thallium complex JARRUQ (Adams *et al.*, 2005), with max(B–N–N–Tl) = 58°, and a sodium complex (refcode TEHSOP; Chisholm *et al.*, 2006), with max(B–N–N–Tl) = 53° (see Fig. S2 in the *Supporting information*). The B–N–N–Tl torsion angles of the three pyrazole rings (*b*, *c* and *d*) coordinated to the Tl^I atom in (1) are 18.7 (3), 20.6 (3) and 57.3 (3)°, respectively. This is in agreement with the values observed for thallium complex JARRUQ.

The structure of compound (2) corresponds to a one-dimensional coordination polymer extending along the *b* axis. These polymer chains are formed by repeating units of **E** motifs (Fig. 3). This is the only example of a tetrakispyrazolylborate where the scorpionate ligand and the metal extend into polymeric chains.

Motifs **C**, **E**, **F**, **G**, **J** and **K** present the metal cations coordinated to two of the pyrazole rings in the scorpionate ligand. The B–N–N–*M* torsion angles observed for these tetrakis scorpionates range from 0 to 40° (see Fig. S2 in the *Supporting information*). In compound (2), the B–N–N–K torsion angles are 79.2 (2) and 81.4 (2)°, and are very distorted in comparison with those reported in the CSD. A possible explanation for this unusual behaviour is the fact that this compound crystallizes forming polymeric chains.

3.3. Comparison with NMR properties

Compound (1) (a Tl⁺ salt) was reported in López *et al.* (1995) and its corresponding Na⁺ salt was reported in Trofi-

Figure 4

The 11 motifs observed for tetrakis scorpionate derivatives. Pyrazole rings are represented by the letter N, metal atoms by a grey sphere, the lines represent the coordination between a pyrazole N atom (N1) and the B atom (B) or between a pyrazole N atom (N2) and the metal atom, and the triple dashed lines represent extra coordination in the metal.

Table 2

Frequency of the 11 motifs found in the CSD for tetrakis scorpionate derivatives (the title compounds have also been included).

Motif	A	B	C	D	E	F	G	H	I	J	K
No. of structures	2	3	37	73	14	2	7	19	5	2	2

menko *et al.* (1987). Compound (2), as a K^+ salt, is reported for the first time; its Tl^+ salt, (16), was described by Rheingold *et al.* (1997) and its NMR properties were described by us (Claramunt *et al.*, 2004a).

In the *Supporting information*, we have gathered all the NMR properties of compounds (1), (2) and (16), both in solution and in the solid-state [CPMAS (cross polarization magic angle spinning) technique], reported in previous papers or determined anew for the present publication, together with the experimental details.

The coordination to the Tl^+ and K^+ ions modifies considerably the conformation of the scorpionate ligands. Compound (1) has a type **D** motif, with three pyrazoles coordinated to the Tl^I atom and the remaining one free. Thus, we expect a 1:3 situation, but the ^{15}N CPMAS spectrum (Fig. 5) displays three signals for each N atom of 1:2:1 intensities.

However, if we look at the B—N—N— Tl torsion angles of the three linked pyrazole rings, with values of 20.6 (3), 18.7 (3) and 57.3 (3)°, we see that two pyrazoles have similar confor-

Figure 5

The ^{15}N CPMAS NMR spectrum of compound (1).

Figure 6

The ^{13}C CPMAS NMR spectrum of compound (1).

mations while the third is quite different. Thus, 1:2:1 relative intensities are consistent with the X-ray structure.

In the ^{13}C CPMAS NMR spectrum (Fig. 6), there are several signals that are split in a 1:3 ratio, allowing the assignment of the uncoordinated pyrazole to the 1 intensity signals: 154.6, 139.6 and 109.0 p.p.m..

Compound (2) presents an **E** motif, which corresponds to a single type of pyrazole coordination. The B–N–N–K torsion angles of the four pyrazole rings are two of -79.2 (2°) and two of 81.4 (2°), so that two similar signals in the CPMAS NMR spectrum for each atom are expected. The ^{13}C spectrum is very complex, but the ^{15}N spectrum (Fig. 7) agrees with the presence of four different pyrazoles, two of them having atom N1 coupled with the ^{11}B atom (they should be quartets with four signals of the same intensity, but only the two central bands are observed). The $^1J(^{11}\text{B}-^{15}\text{N}) \simeq 27$ Hz values are close to those reported previously; see the *Supporting information* and Claramunt *et al.* (2004a). The fact that only two N1 atoms are coupled with the ^{11}B atom is probably related to conformational differences. The signal of atom N2 also corresponds to four different pyrazoles, two of them having the same chemical shift (intensity 2:1:1).

3.4. Comparison with theoretical calculations

We have carried out B3LYP/6-311++G(d,p) calculations of the anions (1) and (2).

Unsubstituted tetrakis(1*H*-pyrazol-1-yl)borate, when isolated, has two minimum energy conformations, of D_2 and S_4 symmetries, the latter being the most stable by 34.8 kJ mol $^{-1}$ (Alkorta *et al.*, 2010). The same happens with the title compounds: 31.9 kJ mol $^{-1}$ for (1) and 35.3 kJ mol $^{-1}$ for (2).

We have calculated the ^1H , ^{13}C and ^{15}N chemical shifts in the gas phase for the isolated anions from GIAO absolute shieldings and compared them with the experimental values measured in DMSO- d_6 . The S_4 values are slightly better than the D_2 ones:

Experimental (p.p.m.) = $-(1.0 \pm 0.6) + (1.016 \pm 0.006)$; calculated S_4 (p.p.m.), $n = 26$, $R^2 = 0.9992$;

Experimental (p.p.m.) = $-(1.4 \pm 0.8) + (1.021 \pm 0.008)$; calculated D_2 (p.p.m.), $n = 26$, $R^2 = 0.9984$.

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Figure 7

The ^{15}N CPMAS NMR spectrum of compound (2).

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Computing details

For both compounds, data collection: *APEX2* (Bruker, 2013); cell refinement: *SAINT* (Bruker, 2013); data reduction: *SAINT* (Bruker, 2013); program(s) used to solve structure: *SIR2014* (Burla *et al.*, 2015); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *Mercury* (Macrae *et al.*, 2008) and *ORTEP-3 for Windows* (Farrugia, 2012).

(comp1) [Tetrakis(3-phenyl-1*H*-pyrazol-1-yl)borato]thallium(I)

Crystal data

[Tl(C₃₆H₂₈BN₈)]
 $M_r = 787.85$
 Monoclinic, $P2_1/c$
 Hall symbol: -P 2ybc
 $a = 9.9552$ (6) Å
 $b = 13.1693$ (8) Å
 $c = 24.7993$ (14) Å
 $\beta = 91.463$ (2)°
 $V = 3250.2$ (3) Å³
 $Z = 4$

$F(000) = 1544$
 $D_x = 1.61$ Mg m⁻³
 Cu $K\alpha$ radiation, $\lambda = 1.54178$ Å
 Cell parameters from 9879 reflections
 $\theta = 3.6\text{--}74.3^\circ$
 $\mu = 9.86$ mm⁻¹
 $T = 296$ K
 Prism, colourless
 $0.06 \times 0.02 \times 0.02$ mm

Data collection

Bruker APEXII CCD
 diffractometer
 φ and ω scans
 Absorption correction: multi-scan
 (SADABS; Bruker, 2013)
 $T_{\min} = 0.218$, $T_{\max} = 0.754$
 45124 measured reflections

6614 independent reflections
 5748 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.045$
 $\theta_{\max} = 74.5^\circ$, $\theta_{\min} = 3.6^\circ$
 $h = -12 \rightarrow 12$
 $k = -16 \rightarrow 16$
 $l = -30 \rightarrow 30$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.029$
 $wR(F^2) = 0.072$
 $S = 1.07$
 6614 reflections
 470 parameters
 28 restraints

Hydrogen site location: inferred from
 neighbouring sites
 H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0358P)^2 + 1.7721P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.003$
 $\Delta\rho_{\max} = 0.42$ e Å⁻³
 $\Delta\rho_{\min} = -1.27$ e Å⁻³

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 ($\text{\AA}^2$)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
Tl1	0.14310 (2)	0.04696 (2)	0.13345 (2)	0.05693 (6)	
B1	0.0505 (3)	0.1455 (3)	0.26229 (12)	0.0426 (7)	
N1	−0.0280 (2)	0.14475 (18)	0.35851 (9)	0.0439 (5)	
N2	0.0314 (2)	0.19608 (19)	0.31732 (9)	0.0442 (5)	
C1	0.0931 (3)	0.2809 (3)	0.33641 (13)	0.0555 (8)	
H1	0.1411	0.3272	0.3161	0.067*	
C2	0.0728 (4)	0.2868 (3)	0.39028 (13)	0.0589 (8)	
H2	0.1027	0.3373	0.4139	0.071*	
C3	−0.0029 (3)	0.2005 (2)	0.40278 (11)	0.0452 (6)	
C4	−0.0498 (3)	0.1690 (2)	0.45605 (11)	0.0484 (7)	
C5	−0.1287 (5)	0.0862 (4)	0.46254 (16)	0.0809 (12)	
H5	−0.1581	0.0498	0.4324	0.097*	
C6	−0.1662 (6)	0.0548 (4)	0.5134 (2)	0.0971 (16)	
H6	−0.2172	−0.0039	0.517	0.117*	
C7	−0.1289 (5)	0.1093 (4)	0.55790 (16)	0.0828 (13)	
H7	−0.1568	0.0898	0.5919	0.099*	
C8	−0.0515 (6)	0.1914 (4)	0.55210 (15)	0.0964 (16)	
H8	−0.0243	0.2283	0.5825	0.116*	
C9	−0.0113 (5)	0.2225 (3)	0.50173 (14)	0.0821 (13)	
H9	0.0423	0.28	0.4986	0.098*	
N1A	−0.0851 (3)	0.0801 (2)	0.18346 (9)	0.0472 (6)	
N2A	−0.0821 (2)	0.10520 (19)	0.23705 (9)	0.0442 (5)	
C1A	−0.2042 (3)	0.0907 (3)	0.25772 (13)	0.0523 (7)	
H1A	−0.2273	0.103	0.2932	0.063*	
C2A	−0.2885 (3)	0.0548 (3)	0.21775 (14)	0.0559 (8)	
H2A	−0.3787	0.0376	0.2206	0.067*	
C3A	−0.2107 (3)	0.0495 (2)	0.17183 (12)	0.0462 (7)	
C4A	−0.2507 (3)	0.0167 (2)	0.11696 (12)	0.0494 (7)	
C5A	−0.1837 (4)	0.0510 (3)	0.07217 (14)	0.0622 (9)	
H5A	−0.1131	0.0968	0.0765	0.075*	
C6A	−0.2211 (4)	0.0177 (4)	0.02101 (15)	0.0776 (11)	
H6A	−0.1749	0.0409	−0.0087	0.093*	
C7A	−0.3251 (5)	−0.0487 (4)	0.01375 (17)	0.0845 (13)	
H7A	−0.3494	−0.0711	−0.0207	0.101*	
C8A	−0.3933 (4)	−0.0823 (4)	0.05747 (17)	0.0779 (11)	
H8A	−0.4642	−0.1277	0.0526	0.093*	
C9A	−0.3582 (4)	−0.0494 (3)	0.10894 (15)	0.0619 (9)	
H9A	−0.4066	−0.0716	0.1382	0.074*	
N1B	0.1877 (2)	−0.00706 (19)	0.23031 (9)	0.0421 (5)	

N2B	0.1539 (2)	0.05854 (17)	0.27058 (9)	0.0420 (5)	
C1B	0.2282 (3)	0.0377 (2)	0.31547 (12)	0.0501 (7)	
H1B	0.2235	0.0717	0.3482	0.06*	
C2B	0.3117 (3)	−0.0417 (3)	0.30479 (13)	0.0551 (8)	
H2B	0.3737	−0.072	0.3284	0.066*	
C3B	0.2844 (3)	−0.0680 (2)	0.25089 (12)	0.0442 (6)	
C4B	0.3480 (3)	−0.1468 (2)	0.21797 (12)	0.0452 (6)	
C5B	0.2896 (3)	−0.1823 (2)	0.17041 (14)	0.0528 (7)	
H5B	0.2059	−0.1576	0.1592	0.063*	
C6B	0.3536 (4)	−0.2541 (3)	0.13897 (15)	0.0621 (9)	
H6B	0.3145	−0.2756	0.1065	0.075*	
C7B	0.4759 (4)	−0.2932 (3)	0.15651 (17)	0.0705 (10)	
H7B	0.5193	−0.3414	0.1358	0.085*	
C8B	0.5335 (4)	−0.2610 (3)	0.20452 (17)	0.0704 (10)	
H8B	0.6142	−0.2894	0.2168	0.084*	
C9B	0.4720 (3)	−0.1867 (3)	0.23473 (14)	0.0587 (8)	
H9B	0.5136	−0.1633	0.2664	0.07*	
N1C	0.2122 (2)	0.20837 (19)	0.19175 (10)	0.0464 (5)	
N2C	0.1052 (2)	0.22658 (19)	0.22339 (9)	0.0443 (5)	
C1C	0.0520 (4)	0.3186 (2)	0.21191 (14)	0.0563 (8)	
H1C	−0.0226	0.347	0.228	0.068*	
C2C	0.1258 (4)	0.3627 (3)	0.17292 (14)	0.0599 (8)	
H2C	0.1124	0.4262	0.1573	0.072*	
C3C	0.2261 (3)	0.2923 (2)	0.16144 (13)	0.0526 (7)	
C4C	0.3363 (12)	0.3032 (13)	0.1237 (4)	0.064 (5)	0.397 (18)
C5C	0.422 (2)	0.226 (2)	0.1133 (8)	0.059 (5)	0.397 (18)
H5C	0.4134	0.1643	0.1307	0.071*	0.397 (18)
C6C	0.525 (2)	0.2413 (19)	0.0756 (9)	0.069 (5)	0.397 (18)
H6C	0.5829	0.1888	0.0673	0.083*	0.397 (18)
C7C	0.5359 (18)	0.3295 (17)	0.0528 (8)	0.092 (6)	0.397 (18)
H7C	0.6045	0.3394	0.0286	0.11*	0.397 (18)
C8C	0.4540 (14)	0.4057 (18)	0.0626 (8)	0.110 (7)	0.397 (18)
H8C	0.4636	0.4671	0.0446	0.132*	0.397 (18)
C9C	0.3533 (12)	0.3942 (14)	0.1000 (8)	0.087 (6)	0.397 (18)
H9C	0.2984	0.4487	0.1085	0.104*	0.397 (18)
C4D	0.3282 (10)	0.2899 (8)	0.1192 (4)	0.064 (3)	0.603 (18)
C5D	0.4437 (15)	0.2330 (12)	0.1254 (6)	0.058 (3)	0.603 (18)
H5D	0.4574	0.196	0.157	0.069*	0.603 (18)
C6D	0.539 (2)	0.2290 (13)	0.0867 (8)	0.082 (4)	0.603 (18)
H6D	0.6148	0.1886	0.0922	0.098*	0.603 (18)
C7D	0.5227 (14)	0.2870 (9)	0.0373 (5)	0.080 (3)	0.603 (18)
H7D	0.5882	0.2864	0.0113	0.096*	0.603 (18)
C8D	0.4087 (13)	0.3422 (10)	0.0305 (5)	0.108 (5)	0.603 (18)
H8D	0.3953	0.3794	−0.0011	0.13*	0.603 (18)
C9D	0.3122 (12)	0.3443 (9)	0.0691 (5)	0.091 (4)	0.603 (18)
H9D	0.2344	0.3819	0.0627	0.109*	0.603 (18)

Atomic displacement parameters ($\text{\AA}^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Tl1	0.07118 (10)	0.06286 (10)	0.03707 (8)	0.00501 (6)	0.00786 (6)	−0.00129 (5)
B1	0.0456 (16)	0.0458 (17)	0.0367 (14)	0.0003 (14)	0.0067 (12)	−0.0027 (13)
N1	0.0494 (13)	0.0463 (13)	0.0363 (11)	−0.0030 (11)	0.0051 (9)	−0.0047 (10)
N2	0.0484 (13)	0.0464 (13)	0.0380 (11)	−0.0046 (11)	0.0058 (9)	−0.0057 (10)
C1	0.0639 (19)	0.0528 (18)	0.0498 (16)	−0.0143 (16)	0.0055 (14)	−0.0040 (14)
C2	0.071 (2)	0.059 (2)	0.0467 (16)	−0.0150 (17)	0.0038 (14)	−0.0147 (14)
C3	0.0479 (15)	0.0490 (16)	0.0386 (14)	−0.0007 (13)	0.0009 (11)	−0.0046 (12)
C4	0.0531 (16)	0.0540 (18)	0.0380 (14)	0.0051 (14)	0.0019 (12)	−0.0029 (12)
C5	0.103 (3)	0.094 (3)	0.0460 (19)	−0.033 (3)	0.0108 (19)	−0.0048 (19)
C6	0.112 (4)	0.119 (4)	0.060 (3)	−0.038 (3)	0.017 (2)	0.013 (2)
C7	0.107 (3)	0.094 (3)	0.048 (2)	0.016 (3)	0.018 (2)	0.011 (2)
C8	0.174 (5)	0.079 (3)	0.0362 (18)	0.007 (3)	0.003 (2)	−0.0063 (18)
C9	0.141 (4)	0.064 (2)	0.0409 (17)	−0.012 (3)	−0.002 (2)	−0.0039 (17)
N1A	0.0525 (14)	0.0545 (14)	0.0348 (12)	0.0006 (12)	0.0017 (10)	−0.0046 (10)
N2A	0.0484 (13)	0.0489 (14)	0.0355 (11)	0.0019 (11)	0.0035 (9)	−0.0043 (10)
C1A	0.0491 (16)	0.0642 (19)	0.0441 (15)	−0.0030 (15)	0.0110 (12)	−0.0087 (14)
C2A	0.0460 (16)	0.073 (2)	0.0489 (17)	−0.0018 (15)	0.0049 (13)	−0.0057 (15)
C3A	0.0533 (16)	0.0450 (16)	0.0401 (15)	0.0040 (13)	−0.0026 (12)	−0.0006 (12)
C4A	0.0553 (17)	0.0519 (17)	0.0408 (15)	0.0071 (14)	−0.0044 (12)	−0.0007 (13)
C5A	0.064 (2)	0.077 (2)	0.0451 (17)	−0.0010 (18)	−0.0044 (14)	0.0051 (15)
C6A	0.081 (3)	0.112 (3)	0.0392 (17)	0.005 (3)	−0.0019 (16)	0.0031 (19)
C7A	0.088 (3)	0.117 (4)	0.048 (2)	−0.003 (3)	−0.0152 (19)	−0.012 (2)
C8A	0.078 (3)	0.093 (3)	0.062 (2)	−0.012 (2)	−0.0157 (19)	−0.010 (2)
C9A	0.065 (2)	0.071 (2)	0.0488 (18)	−0.0037 (17)	−0.0073 (15)	−0.0019 (15)
N1B	0.0445 (12)	0.0427 (12)	0.0392 (12)	0.0030 (10)	0.0050 (9)	−0.0002 (10)
N2B	0.0473 (13)	0.0441 (13)	0.0348 (11)	−0.0008 (10)	0.0043 (9)	−0.0007 (9)
C1B	0.0527 (17)	0.0601 (19)	0.0375 (14)	−0.0016 (15)	0.0009 (12)	0.0020 (12)
C2B	0.0549 (18)	0.066 (2)	0.0447 (16)	0.0078 (16)	−0.0024 (13)	0.0067 (14)
C3B	0.0407 (14)	0.0472 (16)	0.0450 (15)	−0.0044 (12)	0.0056 (11)	0.0059 (12)
C4B	0.0409 (14)	0.0444 (15)	0.0507 (15)	−0.0008 (12)	0.0085 (11)	0.0068 (12)
C5B	0.0443 (16)	0.0493 (17)	0.0647 (19)	0.0016 (14)	0.0018 (14)	−0.0023 (15)
C6B	0.060 (2)	0.064 (2)	0.063 (2)	−0.0008 (17)	0.0062 (15)	−0.0111 (16)
C7B	0.061 (2)	0.068 (2)	0.084 (3)	0.0122 (18)	0.0167 (18)	−0.013 (2)
C8B	0.0521 (19)	0.078 (3)	0.081 (2)	0.0204 (18)	0.0037 (17)	−0.004 (2)
C9B	0.0499 (17)	0.066 (2)	0.0597 (18)	0.0075 (16)	−0.0015 (14)	0.0029 (16)
N1C	0.0442 (13)	0.0448 (13)	0.0506 (13)	0.0022 (11)	0.0089 (10)	0.0061 (11)
N2C	0.0475 (13)	0.0446 (13)	0.0410 (12)	0.0037 (11)	0.0069 (10)	0.0009 (10)
C1C	0.0631 (19)	0.0504 (18)	0.0558 (18)	0.0138 (16)	0.0107 (15)	0.0041 (14)
C2C	0.067 (2)	0.0494 (18)	0.0632 (19)	0.0087 (16)	0.0083 (16)	0.0136 (16)
C3C	0.0474 (16)	0.0499 (17)	0.0607 (18)	0.0003 (14)	0.0059 (13)	0.0127 (14)
C4C	0.049 (9)	0.072 (10)	0.070 (10)	−0.005 (8)	−0.007 (7)	0.042 (7)
C5C	0.051 (7)	0.074 (8)	0.052 (9)	−0.009 (6)	0.001 (6)	0.000 (6)
C6C	0.049 (7)	0.090 (10)	0.071 (9)	0.004 (7)	0.023 (6)	0.009 (7)
C7C	0.062 (7)	0.128 (18)	0.087 (12)	−0.018 (11)	0.018 (7)	0.022 (11)
C8C	0.089 (9)	0.127 (15)	0.115 (14)	−0.011 (10)	0.030 (8)	0.063 (13)

C9C	0.075 (7)	0.090 (10)	0.097 (11)	0.013 (7)	0.025 (7)	0.049 (9)
C4D	0.058 (7)	0.052 (5)	0.083 (7)	0.000 (5)	0.028 (5)	0.014 (5)
C5D	0.055 (6)	0.069 (5)	0.049 (5)	0.008 (4)	0.006 (4)	0.008 (4)
C6D	0.077 (7)	0.084 (6)	0.085 (7)	0.018 (5)	0.016 (5)	0.020 (5)
C7D	0.085 (7)	0.072 (6)	0.087 (7)	−0.004 (5)	0.050 (5)	0.002 (5)
C8D	0.115 (8)	0.118 (9)	0.095 (7)	0.031 (7)	0.055 (6)	0.059 (7)
C9D	0.089 (6)	0.093 (7)	0.092 (7)	0.032 (6)	0.045 (5)	0.042 (6)

Geometric parameters (Å, °)

TI1—N1B	2.533 (2)	C1B—C2B	1.365 (5)
TI1—N1C	2.652 (3)	C1B—H1B	0.93
TI1—N1A	2.652 (3)	C2B—C3B	1.401 (4)
B1—N2	1.535 (4)	C2B—H2B	0.93
B1—N2A	1.541 (4)	C3B—C4B	1.473 (4)
B1—N2C	1.547 (4)	C4B—C5B	1.383 (4)
B1—N2B	1.550 (4)	C4B—C9B	1.396 (4)
N1—C3	1.339 (4)	C5B—C6B	1.391 (5)
N1—N2	1.371 (3)	C5B—H5B	0.93
N2—C1	1.354 (4)	C6B—C7B	1.382 (5)
C1—C2	1.358 (4)	C6B—H6B	0.93
C1—H1	0.93	C7B—C8B	1.375 (6)
C2—C3	1.402 (5)	C7B—H7B	0.93
C2—H2	0.93	C8B—C9B	1.384 (5)
C3—C4	1.472 (4)	C8B—H8B	0.93
C4—C5	1.355 (5)	C9B—H9B	0.93
C4—C9	1.380 (5)	N1C—C3C	1.346 (4)
C5—C6	1.387 (6)	N1C—N2C	1.360 (3)
C5—H5	0.93	N2C—C1C	1.350 (4)
C6—C7	1.361 (7)	C1C—C2C	1.360 (5)
C6—H6	0.93	C1C—H1C	0.93
C7—C8	1.338 (7)	C2C—C3C	1.398 (5)
C7—H7	0.93	C2C—H2C	0.93
C8—C9	1.384 (5)	C3C—C4C	1.467 (13)
C8—H8	0.93	C3C—C4D	1.479 (9)
C9—H9	0.93	C4C—C9C	1.348 (18)
N1A—C3A	1.339 (4)	C4C—C5C	1.35 (2)
N1A—N2A	1.369 (3)	C5C—C6C	1.42 (2)
N2A—C1A	1.344 (4)	C5C—H5C	0.93
C1A—C2A	1.367 (5)	C6C—C7C	1.30 (2)
C1A—H1A	0.93	C6C—H6C	0.93
C2A—C3A	1.395 (4)	C7C—C8C	1.32 (2)
C2A—H2A	0.93	C7C—H7C	0.93
C3A—C4A	1.473 (4)	C8C—C9C	1.390 (16)
C4A—C5A	1.386 (5)	C8C—H8C	0.93
C4A—C9A	1.390 (5)	C9C—H9C	0.93
C5A—C6A	1.385 (5)	C4D—C5D	1.378 (15)
C5A—H5A	0.93	C4D—C9D	1.438 (15)

C6A—C7A	1.364 (6)	C5D—C6D	1.366 (17)
C6A—H6A	0.93	C5D—H5D	0.93
C7A—C8A	1.367 (6)	C6D—C7D	1.450 (19)
C7A—H7A	0.93	C6D—H6D	0.93
C8A—C9A	1.384 (5)	C7D—C8D	1.355 (15)
C8A—H8A	0.93	C7D—H7D	0.93
C9A—H9A	0.93	C8D—C9D	1.374 (10)
N1B—C3B	1.344 (4)	C8D—H8D	0.93
N1B—N2B	1.369 (3)	C9D—H9D	0.93
N2B—C1B	1.349 (4)		
N1B—Tl1—N1C	70.82 (8)	N2B—C1B—H1B	125.7
N1B—Tl1—N1A	74.60 (7)	C2B—C1B—H1B	125.7
N1C—Tl1—N1A	80.00 (7)	C1B—C2B—C3B	105.7 (3)
N2—B1—N2A	112.7 (2)	C1B—C2B—H2B	127.1
N2—B1—N2C	108.0 (2)	C3B—C2B—H2B	127.1
N2A—B1—N2C	107.2 (2)	N1B—C3B—C2B	109.5 (3)
N2—B1—N2B	107.5 (2)	N1B—C3B—C4B	121.6 (3)
N2A—B1—N2B	111.0 (2)	C2B—C3B—C4B	128.9 (3)
N2C—B1—N2B	110.5 (2)	C5B—C4B—C9B	118.4 (3)
C3—N1—N2	105.4 (2)	C5B—C4B—C3B	122.3 (3)
C1—N2—N1	110.3 (2)	C9B—C4B—C3B	119.3 (3)
C1—N2—B1	127.1 (3)	C4B—C5B—C6B	121.3 (3)
N1—N2—B1	120.9 (2)	C4B—C5B—H5B	119.4
N2—C1—C2	108.2 (3)	C6B—C5B—H5B	119.4
N2—C1—H1	125.9	C7B—C6B—C5B	119.3 (3)
C2—C1—H1	125.9	C7B—C6B—H6B	120.3
C1—C2—C3	105.4 (3)	C5B—C6B—H6B	120.3
C1—C2—H2	127.3	C8B—C7B—C6B	120.2 (3)
C3—C2—H2	127.3	C8B—C7B—H7B	119.9
N1—C3—C2	110.7 (3)	C6B—C7B—H7B	119.9
N1—C3—C4	121.6 (3)	C7B—C8B—C9B	120.4 (3)
C2—C3—C4	127.7 (3)	C7B—C8B—H8B	119.8
C5—C4—C9	117.6 (3)	C9B—C8B—H8B	119.8
C5—C4—C3	122.1 (3)	C8B—C9B—C4B	120.3 (3)
C9—C4—C3	120.3 (3)	C8B—C9B—H9B	119.8
C4—C5—C6	121.2 (4)	C4B—C9B—H9B	119.8
C4—C5—H5	119.4	C3C—N1C—N2C	105.7 (2)
C6—C5—H5	119.4	C3C—N1C—Tl1	112.5 (2)
C7—C6—C5	120.4 (4)	N2C—N1C—Tl1	105.06 (17)
C7—C6—H6	119.8	C1C—N2C—N1C	110.2 (2)
C5—C6—H6	119.8	C1C—N2C—B1	127.5 (3)
C8—C7—C6	119.0 (4)	N1C—N2C—B1	122.1 (2)
C8—C7—H7	120.5	N2C—C1C—C2C	108.5 (3)
C6—C7—H7	120.5	N2C—C1C—H1C	125.7
C7—C8—C9	121.1 (4)	C2C—C1C—H1C	125.7
C7—C8—H8	119.4	C1C—C2C—C3C	105.2 (3)
C9—C8—H8	119.4	C1C—C2C—H2C	127.4

C4—C9—C8	120.6 (4)	C3C—C2C—H2C	127.4
C4—C9—H9	119.7	N1C—C3C—C2C	110.3 (3)
C8—C9—H9	119.7	N1C—C3C—C4C	121.7 (8)
C3A—N1A—N2A	106.2 (2)	C2C—C3C—C4C	127.9 (8)
C3A—N1A—Ti1	131.04 (19)	N1C—C3C—C4D	117.4 (6)
N2A—N1A—Ti1	119.75 (17)	C2C—C3C—C4D	131.8 (6)
C1A—N2A—N1A	109.8 (2)	C9C—C4C—C5C	119.7 (16)
C1A—N2A—B1	131.9 (2)	C9C—C4C—C3C	118.0 (13)
N1A—N2A—B1	118.3 (2)	C5C—C4C—C3C	122.3 (15)
N2A—C1A—C2A	108.5 (3)	C4C—C5C—C6C	119.4 (19)
N2A—C1A—H1A	125.7	C4C—C5C—H5C	120.3
C2A—C1A—H1A	125.7	C6C—C5C—H5C	120.3
C1A—C2A—C3A	105.4 (3)	C7C—C6C—C5C	118.9 (19)
C1A—C2A—H2A	127.3	C7C—C6C—H6C	120.5
C3A—C2A—H2A	127.3	C5C—C6C—H6C	120.5
N1A—C3A—C2A	110.1 (3)	C6C—C7C—C8C	123 (2)
N1A—C3A—C4A	121.1 (3)	C6C—C7C—H7C	118.7
C2A—C3A—C4A	128.8 (3)	C8C—C7C—H7C	118.7
C5A—C4A—C9A	118.3 (3)	C7C—C8C—C9C	120.0 (13)
C5A—C4A—C3A	121.4 (3)	C7C—C8C—H8C	120
C9A—C4A—C3A	120.3 (3)	C9C—C8C—H8C	120
C6A—C5A—C4A	120.5 (4)	C4C—C9C—C8C	119.2 (13)
C6A—C5A—H5A	119.8	C4C—C9C—H9C	120.4
C4A—C5A—H5A	119.8	C8C—C9C—H9C	120.4
C7A—C6A—C5A	120.7 (4)	C5D—C4D—C9D	116.2 (10)
C7A—C6A—H6A	119.7	C5D—C4D—C3C	121.3 (12)
C5A—C6A—H6A	119.7	C9D—C4D—C3C	122.6 (10)
C6A—C7A—C8A	119.6 (4)	C6D—C5D—C4D	122.3 (12)
C6A—C7A—H7A	120.2	C6D—C5D—H5D	118.8
C8A—C7A—H7A	120.2	C4D—C5D—H5D	118.8
C7A—C8A—C9A	120.7 (4)	C5D—C6D—C7D	120.5 (14)
C7A—C8A—H8A	119.6	C5D—C6D—H6D	119.7
C9A—C8A—H8A	119.6	C7D—C6D—H6D	119.7
C8A—C9A—C4A	120.2 (4)	C8D—C7D—C6D	117.6 (12)
C8A—C9A—H9A	119.9	C8D—C7D—H7D	121.2
C4A—C9A—H9A	119.9	C6D—C7D—H7D	121.2
C3B—N1B—N2B	106.7 (2)	C7D—C8D—C9D	121.4 (9)
C3B—N1B—Ti1	129.37 (18)	C7D—C8D—H8D	119.3
N2B—N1B—Ti1	118.29 (17)	C9D—C8D—H8D	119.3
C1B—N2B—N1B	109.5 (2)	C8D—C9D—C4D	121.9 (8)
C1B—N2B—B1	127.5 (2)	C8D—C9D—H9D	119.1
N1B—N2B—B1	123.0 (2)	C4D—C9D—H9D	119.1
N2B—C1B—C2B	108.6 (3)		
C3—N1—N2—C1	0.8 (3)	N1B—N2B—C1B—C2B	0.2 (3)
C3—N1—N2—B1	167.0 (3)	B1—N2B—C1B—C2B	−176.7 (3)
N2A—B1—N2—C1	−140.7 (3)	N2B—C1B—C2B—C3B	0.1 (4)
N2C—B1—N2—C1	−22.5 (4)	N2B—N1B—C3B—C2B	0.5 (3)

N2B—B1—N2—C1	96.7 (3)	TI1—N1B—C3B—C2B	152.9 (2)
N2A—B1—N2—N1	55.6 (4)	N2B—N1B—C3B—C4B	−178.3 (2)
N2C—B1—N2—N1	173.8 (2)	TI1—N1B—C3B—C4B	−25.9 (4)
N2B—B1—N2—N1	−67.0 (3)	C1B—C2B—C3B—N1B	−0.4 (4)
N1—N2—C1—C2	−1.0 (4)	C1B—C2B—C3B—C4B	178.2 (3)
B1—N2—C1—C2	−166.1 (3)	N1B—C3B—C4B—C5B	−18.5 (4)
N2—C1—C2—C3	0.7 (4)	C2B—C3B—C4B—C5B	163.0 (3)
N2—N1—C3—C2	−0.4 (3)	N1B—C3B—C4B—C9B	160.8 (3)
N2—N1—C3—C4	−179.2 (3)	C2B—C3B—C4B—C9B	−17.7 (5)
C1—C2—C3—N1	−0.2 (4)	C9B—C4B—C5B—C6B	−1.7 (5)
C1—C2—C3—C4	178.6 (3)	C3B—C4B—C5B—C6B	177.7 (3)
N1—C3—C4—C5	−4.9 (5)	C4B—C5B—C6B—C7B	2.2 (5)
C2—C3—C4—C5	176.5 (4)	C5B—C6B—C7B—C8B	−0.2 (6)
N1—C3—C4—C9	173.6 (3)	C6B—C7B—C8B—C9B	−2.3 (6)
C2—C3—C4—C9	−5.1 (5)	C7B—C8B—C9B—C4B	2.8 (6)
C9—C4—C5—C6	−1.8 (7)	C5B—C4B—C9B—C8B	−0.8 (5)
C3—C4—C5—C6	176.7 (4)	C3B—C4B—C9B—C8B	179.8 (3)
C4—C5—C6—C7	2.8 (9)	C3C—N1C—N2C—C1C	1.6 (3)
C5—C6—C7—C8	−2.4 (8)	TI1—N1C—N2C—C1C	−117.6 (2)
C6—C7—C8—C9	1.2 (8)	C3C—N1C—N2C—B1	176.5 (3)
C5—C4—C9—C8	0.5 (7)	TI1—N1C—N2C—B1	57.3 (3)
C3—C4—C9—C8	−178.0 (4)	N2—B1—N2C—C1C	−53.7 (4)
C7—C8—C9—C4	−0.3 (8)	N2A—B1—N2C—C1C	68.0 (4)
C3A—N1A—N2A—C1A	−0.3 (3)	N2B—B1—N2C—C1C	−170.9 (3)
TI1—N1A—N2A—C1A	−162.8 (2)	N2—B1—N2C—N1C	132.3 (3)
C3A—N1A—N2A—B1	−178.9 (3)	N2A—B1—N2C—N1C	−106.0 (3)
TI1—N1A—N2A—B1	18.7 (3)	N2B—B1—N2C—N1C	15.0 (4)
N2—B1—N2A—C1A	−13.1 (5)	N1C—N2C—C1C—C2C	−1.1 (4)
N2C—B1—N2A—C1A	−131.8 (3)	B1—N2C—C1C—C2C	−175.7 (3)
N2B—B1—N2A—C1A	107.5 (4)	N2C—C1C—C2C—C3C	0.1 (4)
N2—B1—N2A—N1A	165.1 (2)	N2C—N1C—C3C—C2C	−1.5 (4)
N2C—B1—N2A—N1A	46.4 (3)	TI1—N1C—C3C—C2C	112.6 (3)
N2B—B1—N2A—N1A	−74.3 (3)	N2C—N1C—C3C—C4C	177.0 (3)
N1A—N2A—C1A—C2A	0.6 (4)	TI1—N1C—C3C—C4C	−68.9 (4)
B1—N2A—C1A—C2A	178.9 (3)	N2C—N1C—C3C—C4D	−174.4 (4)
N2A—C1A—C2A—C3A	−0.6 (4)	TI1—N1C—C3C—C4D	−60.3 (5)
N2A—N1A—C3A—C2A	0.0 (3)	C1C—C2C—C3C—N1C	0.9 (4)
TI1—N1A—C3A—C2A	159.6 (2)	C1C—C2C—C3C—C4C	−177.5 (4)
N2A—N1A—C3A—C4A	179.8 (3)	C1C—C2C—C3C—C4D	172.4 (5)
TI1—N1A—C3A—C4A	−20.5 (4)	N1C—C3C—C4C—C9C	−171.7 (9)
C1A—C2A—C3A—N1A	0.4 (4)	C2C—C3C—C4C—C9C	6.5 (9)
C1A—C2A—C3A—C4A	−179.4 (3)	N1C—C3C—C4C—C5C	6.6 (9)
N1A—C3A—C4A—C5A	−25.4 (5)	C2C—C3C—C4C—C5C	−175.2 (9)
C2A—C3A—C4A—C5A	154.4 (3)	C9C—C4C—C5C—C6C	−2.9 (8)
N1A—C3A—C4A—C9A	155.1 (3)	C3C—C4C—C5C—C6C	178.8 (5)
C2A—C3A—C4A—C9A	−25.1 (5)	C4C—C5C—C6C—C7C	1.6 (12)
C9A—C4A—C5A—C6A	−1.7 (5)	C5C—C6C—C7C—C8C	−1.2 (17)
C3A—C4A—C5A—C6A	178.7 (3)	C6C—C7C—C8C—C9C	2.2 (17)

C4A—C5A—C6A—C7A	0.5 (6)	C5C—C4C—C9C—C8C	3.9 (11)
C5A—C6A—C7A—C8A	0.4 (7)	C3C—C4C—C9C—C8C	−177.8 (7)
C6A—C7A—C8A—C9A	0.1 (7)	C7C—C8C—C9C—C4C	−3.5 (15)
C7A—C8A—C9A—C4A	−1.4 (7)	N1C—C3C—C4D—C5D	−30.9 (9)
C5A—C4A—C9A—C8A	2.2 (5)	C2C—C3C—C4D—C5D	158.0 (8)
C3A—C4A—C9A—C8A	−178.3 (4)	N1C—C3C—C4D—C9D	147.7 (8)
C3B—N1B—N2B—C1B	−0.4 (3)	C2C—C3C—C4D—C9D	−23.4 (9)
TI1—N1B—N2B—C1B	−156.38 (19)	C9D—C4D—C5D—C6D	0.8 (12)
C3B—N1B—N2B—B1	176.6 (2)	C3C—C4D—C5D—C6D	179.5 (9)
TI1—N1B—N2B—B1	20.6 (3)	C4D—C5D—C6D—C7D	1.2 (17)
N2—B1—N2B—C1B	−8.1 (4)	C5D—C6D—C7D—C8D	−2.0 (19)
N2A—B1—N2B—C1B	−131.7 (3)	C6D—C7D—C8D—C9D	0.8 (18)
N2C—B1—N2B—C1B	109.5 (3)	C7D—C8D—C9D—C4D	1.2 (17)
N2—B1—N2B—N1B	175.4 (2)	C5D—C4D—C9D—C8D	−2.0 (13)
N2A—B1—N2B—N1B	51.8 (3)	C3C—C4D—C9D—C8D	179.3 (8)
N2C—B1—N2B—N1B	−67.0 (3)		

(comp2) *catena*-Poly[potassium- $[\mu_2$ -tetrakis(3-cyclopropyl-1*H*-pyrazol-1-yl)borato]]*Crystal data*[K(C₂₄H₂₈BN₈)] $M_r = 478.45$ Monoclinic, $C2/c$ Hall symbol: $-C\ 2yc$ $a = 19.547\ (8)\ \text{\AA}$ $b = 8.391\ (3)\ \text{\AA}$ $c = 17.153\ (8)\ \text{\AA}$ $\beta = 117.111\ (12)^\circ$ $V = 2504.2\ (18)\ \text{\AA}^3$ $Z = 4$ $F(000) = 1008$ $D_x = 1.269\ \text{Mg m}^{-3}$ Cu $K\alpha$ radiation, $\lambda = 1.54178\ \text{\AA}$

Cell parameters from 5460 reflections

 $\theta = 5.1\text{--}70.2^\circ$ $\mu = 2.08\ \text{mm}^{-1}$ $T = 296\ \text{K}$

Prism, colourless

 $0.19 \times 0.06 \times 0.06\ \text{mm}$ *Data collection*

Bruker APEXII CCD

diffractometer

 φ and ω scans

Absorption correction: multi-scan

(SADABS; Bruker, 2013)

 $T_{\min} = 0.511$, $T_{\max} = 0.754$

21173 measured reflections

2126 independent reflections

1361 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.085$ $\theta_{\max} = 66.7^\circ$, $\theta_{\min} = 5.1^\circ$ $h = -23 \rightarrow 23$ $k = -9 \rightarrow 9$ $l = -20 \rightarrow 20$ *Refinement*Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.051$ $wR(F^2) = 0.135$ $S = 1.03$

2126 reflections

155 parameters

0 restraints

Hydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2) + (0.0534P)^2 + 1.8736P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} < 0.001$ $\Delta\rho_{\max} = 0.14\ \text{e \AA}^{-3}$ $\Delta\rho_{\min} = -0.24\ \text{e \AA}^{-3}$

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 ($\text{\AA}^2$)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
K1	0	0.67908 (11)	0.25	0.0794 (4)
B1	0	1.1765 (5)	0.25	0.0448 (11)
N1A	0.04126 (13)	1.0712 (2)	0.21159 (14)	0.0460 (6)
N2A	0.08779 (14)	0.9490 (3)	0.25887 (15)	0.0562 (7)
C1A	0.11582 (17)	0.8846 (3)	0.20838 (19)	0.0524 (7)
C2A	0.0885 (2)	0.9637 (4)	0.1296 (2)	0.0726 (10)
H2A	0.0994	0.9421	0.0833	0.087*
C3A	0.0420 (2)	1.0805 (4)	0.1341 (2)	0.0659 (9)
H3A	0.0152	1.1547	0.0904	0.079*
C4A	0.16749 (19)	0.7446 (4)	0.2416 (2)	0.0720 (10)
H4A	0.1909	0.7297	0.3052	0.086*
C5A	0.1484 (3)	0.5948 (5)	0.1900 (3)	0.1159 (16)
H5A1	0.1581	0.4948	0.2217	0.139*
H5A2	0.1036	0.5949	0.133	0.139*
C6A	0.2141 (3)	0.6889 (5)	0.2005 (3)	0.0999 (14)
H6A1	0.2104	0.7471	0.1499	0.12*
H6A2	0.2648	0.6471	0.2384	0.12*
N1B	0.05869 (13)	1.2815 (2)	0.32347 (15)	0.0483 (6)
N2B	0.03531 (14)	1.4085 (3)	0.35488 (17)	0.0591 (7)
C1B	0.09807 (17)	1.4670 (3)	0.42155 (19)	0.0550 (8)
C2B	0.1610 (2)	1.3764 (4)	0.4340 (2)	0.0880 (12)
H2B	0.2116	1.3909	0.4763	0.106*
C3B	0.13353 (19)	1.2608 (4)	0.3712 (2)	0.0760 (10)
H3B	0.1626	1.1806	0.3634	0.091*
C4B	0.0927 (2)	1.6112 (4)	0.4678 (2)	0.0705 (9)
H4B	0.0428	1.6275	0.4672	0.085*
C5B	0.1331 (3)	1.7605 (4)	0.4646 (2)	0.0961 (13)
H5B1	0.1646	1.7567	0.4343	0.115*
H5B2	0.1071	1.8612	0.46	0.115*
C6B	0.1593 (2)	1.6707 (4)	0.5465 (2)	0.0797 (11)
H6B1	0.2072	1.6122	0.5669	0.096*
H6B2	0.1498	1.7165	0.5926	0.096*

Atomic displacement parameters ($\text{\AA}^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
K1	0.0980 (9)	0.0358 (5)	0.0960 (9)	0	0.0369 (7)	0
B1	0.057 (3)	0.028 (2)	0.050 (3)	0	0.025 (3)	0
N1A	0.0590 (15)	0.0361 (11)	0.0439 (14)	0.0011 (10)	0.0242 (12)	0.0038 (10)

N2A	0.0783 (18)	0.0400 (12)	0.0561 (15)	0.0138 (12)	0.0356 (15)	0.0043 (11)
C1A	0.065 (2)	0.0430 (15)	0.0561 (19)	0.0003 (14)	0.0332 (17)	−0.0075 (13)
C2A	0.094 (3)	0.078 (2)	0.062 (2)	0.017 (2)	0.050 (2)	0.0041 (18)
C3A	0.085 (2)	0.0629 (19)	0.055 (2)	0.0120 (17)	0.0368 (19)	0.0129 (16)
C4A	0.085 (3)	0.067 (2)	0.066 (2)	0.0195 (18)	0.036 (2)	−0.0072 (17)
C5A	0.110 (4)	0.072 (3)	0.155 (4)	0.015 (2)	0.052 (3)	−0.037 (3)
C6A	0.092 (3)	0.117 (3)	0.102 (3)	0.040 (3)	0.054 (3)	0.003 (3)
N1B	0.0518 (15)	0.0348 (11)	0.0536 (15)	0.0048 (10)	0.0200 (13)	−0.0021 (10)
N2B	0.0593 (16)	0.0399 (13)	0.0692 (18)	0.0039 (11)	0.0217 (15)	−0.0135 (12)
C1B	0.061 (2)	0.0445 (15)	0.0487 (19)	0.0011 (14)	0.0156 (17)	−0.0063 (13)
C2B	0.058 (2)	0.082 (2)	0.091 (3)	0.0097 (18)	0.005 (2)	−0.038 (2)
C3B	0.053 (2)	0.074 (2)	0.085 (3)	0.0126 (17)	0.018 (2)	−0.026 (2)
C4B	0.077 (2)	0.0542 (18)	0.076 (2)	−0.0027 (17)	0.031 (2)	−0.0197 (17)
C5B	0.154 (4)	0.051 (2)	0.079 (3)	−0.017 (2)	0.049 (3)	−0.0122 (19)
C6B	0.099 (3)	0.065 (2)	0.064 (2)	−0.0076 (19)	0.027 (2)	−0.0193 (18)

Geometric parameters (Å, °)

K1—N2B ⁱ	2.781 (3)	C4A—H4A	0.98
K1—N2B ⁱⁱ	2.781 (3)	C5A—C6A	1.448 (5)
K1—N2A	2.804 (3)	C5A—H5A1	0.97
K1—N2A ⁱⁱⁱ	2.804 (3)	C5A—H5A2	0.97
K1—C1A	3.175 (3)	C6A—H6A1	0.97
K1—C1A ⁱⁱⁱ	3.175 (3)	C6A—H6A2	0.97
K1—C1B ⁱ	3.218 (3)	N1B—C3B	1.322 (4)
K1—C1B ⁱⁱ	3.218 (3)	N1B—N2B	1.364 (3)
K1—C4B ⁱ	3.375 (4)	N2B—C1B	1.332 (3)
K1—C4B ⁱⁱ	3.375 (4)	N2B—K1 ^{iv}	2.781 (3)
K1—C4A ⁱⁱⁱ	3.387 (4)	C1B—C2B	1.378 (4)
K1—C4A	3.387 (4)	C1B—C4B	1.476 (4)
B1—N1A ⁱⁱⁱ	1.533 (3)	C1B—K1 ^{iv}	3.218 (3)
B1—N1A	1.533 (3)	C2B—C3B	1.364 (4)
B1—N1B	1.537 (3)	C2B—H2B	0.93
B1—N1B ⁱⁱⁱ	1.537 (3)	C3B—H3B	0.93
N1A—C3A	1.339 (3)	C4B—C6B	1.472 (5)
N1A—N2A	1.365 (3)	C4B—C5B	1.495 (5)
N2A—C1A	1.332 (3)	C4B—K1 ^{iv}	3.375 (4)
C1A—C2A	1.376 (4)	C4B—H4B	0.98
C1A—C4A	1.484 (4)	C5B—C6B	1.466 (5)
C2A—C3A	1.362 (4)	C5B—K1 ^{iv}	3.472 (4)
C2A—H2A	0.93	C5B—H5B1	0.97
C3A—H3A	0.93	C5B—H5B2	0.97
C4A—C6A	1.459 (4)	C6B—H6B1	0.97
C4A—C5A	1.484 (5)	C6B—H6B2	0.97
N2B ⁱ —K1—N2B ⁱⁱ	70.57 (10)	N2A—C1A—C2A	110.6 (3)
N2B ⁱ —K1—N2A	131.29 (7)	N2A—C1A—C4A	118.8 (3)
N2B ⁱⁱ —K1—N2A	131.23 (7)	C2A—C1A—C4A	130.6 (3)

N2B ⁱ —K1—N2A ⁱⁱⁱ	131.23 (7)	N2A—C1A—K1	61.81 (15)
N2B ⁱⁱ —K1—N2A ⁱⁱⁱ	131.29 (7)	C2A—C1A—K1	119.0 (2)
N2A—K1—N2A ⁱⁱⁱ	72.22 (10)	C4A—C1A—K1	85.06 (19)
N2B ⁱ —K1—C1A	106.87 (7)	C3A—C2A—C1A	105.2 (3)
N2B ⁱⁱ —K1—C1A	126.63 (8)	C3A—C2A—H2A	127.4
N2A—K1—C1A	24.76 (6)	C1A—C2A—H2A	127.4
N2A ⁱⁱⁱ —K1—C1A	91.74 (8)	N1A—C3A—C2A	108.8 (3)
N2B ⁱ —K1—C1A ⁱⁱⁱ	126.63 (8)	N1A—C3A—H3A	125.6
N2B ⁱⁱ —K1—C1A ⁱⁱⁱ	106.87 (7)	C2A—C3A—H3A	125.6
N2A—K1—C1A ⁱⁱⁱ	91.74 (8)	C6A—C4A—C1A	122.3 (3)
N2A ⁱⁱⁱ —K1—C1A ⁱⁱⁱ	24.76 (6)	C6A—C4A—C5A	58.9 (2)
C1A—K1—C1A ⁱⁱⁱ	114.20 (10)	C1A—C4A—C5A	120.0 (3)
N2B ⁱ —K1—C1B ⁱ	24.28 (7)	C6A—C4A—K1	142.8 (3)
N2B ⁱⁱ —K1—C1B ⁱ	90.51 (8)	C1A—C4A—K1	69.06 (17)
N2A—K1—C1B ⁱ	126.23 (7)	C5A—C4A—K1	84.4 (2)
N2A ⁱⁱⁱ —K1—C1B ⁱ	107.59 (7)	C6A—C4A—H4A	114.8
C1A—K1—C1B ⁱ	106.46 (8)	C1A—C4A—H4A	114.8
C1A ⁱⁱⁱ —K1—C1B ⁱ	108.50 (8)	C5A—C4A—H4A	114.8
N2B ⁱ —K1—C1B ⁱⁱ	90.51 (8)	K1—C4A—H4A	84.4
N2B ⁱⁱ —K1—C1B ⁱⁱ	24.28 (7)	C6A—C5A—C4A	59.7 (3)
N2A—K1—C1B ⁱⁱ	107.59 (7)	C6A—C5A—H5A1	117.8
N2A ⁱⁱⁱ —K1—C1B ⁱⁱ	126.24 (7)	C4A—C5A—H5A1	117.8
C1A—K1—C1B ⁱⁱ	108.50 (8)	C6A—C5A—H5A2	117.8
C1A ⁱⁱⁱ —K1—C1B ⁱⁱ	106.46 (8)	C4A—C5A—H5A2	117.8
C1B ⁱ —K1—C1B ⁱⁱ	112.87 (10)	H5A1—C5A—H5A2	114.9
N2B ⁱ —K1—C4B ⁱ	45.30 (7)	C5A—C6A—C4A	61.4 (3)
N2B ⁱⁱ —K1—C4B ⁱ	115.34 (8)	C5A—C6A—H6A1	117.6
N2A—K1—C4B ⁱ	101.71 (7)	C4A—C6A—H6A1	117.6
N2A ⁱⁱⁱ —K1—C4B ⁱ	93.99 (7)	C5A—C6A—H6A2	117.6
C1A—K1—C4B ⁱ	85.47 (8)	C4A—C6A—H6A2	117.6
C1A ⁱⁱⁱ —K1—C4B ⁱ	105.21 (8)	H6A1—C6A—H6A2	114.7
C1B ⁱ —K1—C4B ⁱ	25.74 (7)	C3B—N1B—N2B	109.5 (2)
C1B ⁱⁱ —K1—C4B ⁱ	135.58 (8)	C3B—N1B—B1	129.4 (2)
N2B ⁱ —K1—C4B ⁱⁱ	115.34 (8)	N2B—N1B—B1	120.8 (2)
N2B ⁱⁱ —K1—C4B ⁱⁱ	45.30 (7)	C1B—N2B—N1B	106.5 (2)
N2A—K1—C4B ⁱⁱ	93.99 (7)	C1B—N2B—K1 ^{iv}	96.58 (17)
N2A ⁱⁱⁱ —K1—C4B ⁱⁱ	101.71 (7)	N1B—N2B—K1 ^{iv}	114.31 (17)
C1A—K1—C4B ⁱⁱ	105.20 (8)	N2B—C1B—C2B	109.7 (3)
C1A ⁱⁱⁱ —K1—C4B ⁱⁱ	85.47 (8)	N2B—C1B—C4B	120.0 (3)
C1B ⁱ —K1—C4B ⁱⁱ	135.58 (8)	C2B—C1B—C4B	130.3 (3)
C1B ⁱⁱ —K1—C4B ⁱⁱ	25.74 (7)	N2B—C1B—K1 ^{iv}	59.14 (15)
C4B ⁱ —K1—C4B ⁱⁱ	160.58 (11)	C2B—C1B—K1 ^{iv}	124.4 (2)
N2B ⁱ —K1—C4A ⁱⁱⁱ	101.56 (8)	C4B—C1B—K1 ^{iv}	83.07 (18)
N2B ⁱⁱ —K1—C4A ⁱⁱⁱ	93.71 (8)	C3B—C2B—C1B	105.5 (3)
N2A—K1—C4A ⁱⁱⁱ	116.50 (8)	C3B—C2B—H2B	127.2
N2A ⁱⁱⁱ —K1—C4A ⁱⁱⁱ	44.89 (7)	C1B—C2B—H2B	127.2
C1A—K1—C4A ⁱⁱⁱ	136.34 (8)	N1B—C3B—C2B	108.8 (3)
C1A ⁱⁱⁱ —K1—C4A ⁱⁱⁱ	25.87 (7)	N1B—C3B—H3B	125.6

C1B ⁱ —K1—C4A ⁱⁱⁱ	86.80 (8)	C2B—C3B—H3B	125.6
C1B ⁱⁱ —K1—C4A ⁱⁱⁱ	103.61 (8)	C1B—C4B—C6B	121.4 (3)
C4B ⁱ —K1—C4A ⁱⁱⁱ	91.88 (9)	C1B—C4B—C5B	120.0 (3)
C4B ⁱⁱ —K1—C4A ⁱⁱⁱ	91.26 (9)	C6B—C4B—C5B	59.2 (2)
N2B ⁱ —K1—C4A	93.71 (8)	C1B—C4B—K1 ^{iv}	71.19 (17)
N2B ⁱⁱ —K1—C4A	101.56 (8)	C6B—C4B—K1 ^{iv}	139.7 (2)
N2A—K1—C4A	44.89 (7)	C5B—C4B—K1 ^{iv}	81.1 (2)
N2A ⁱⁱⁱ —K1—C4A	116.50 (8)	C1B—C4B—H4B	115
C1A—K1—C4A	25.87 (7)	C6B—C4B—H4B	115
C1A ⁱⁱⁱ —K1—C4A	136.34 (8)	C5B—C4B—H4B	115
C1B ⁱ —K1—C4A	103.61 (8)	K1 ^{iv} —C4B—H4B	86.3
C1B ⁱⁱ —K1—C4A	86.80 (8)	C6B—C5B—C4B	59.6 (2)
C4B ⁱ —K1—C4A	91.26 (9)	C6B—C5B—K1 ^{iv}	132.9 (2)
C4B ⁱⁱ —K1—C4A	91.88 (9)	C4B—C5B—K1 ^{iv}	73.8 (2)
C4A ⁱⁱⁱ —K1—C4A	161.32 (11)	C6B—C5B—H5B1	117.8
N1A ⁱⁱⁱ —B1—N1A	109.6 (3)	C4B—C5B—H5B1	117.8
N1A ⁱⁱⁱ —B1—N1B	108.68 (12)	K1 ^{iv} —C5B—H5B1	77.2
N1A—B1—N1B	109.90 (12)	C6B—C5B—H5B2	117.8
N1A ⁱⁱⁱ —B1—N1B ⁱⁱⁱ	109.90 (12)	C4B—C5B—H5B2	117.8
N1A—B1—N1B ⁱⁱⁱ	108.68 (12)	K1 ^{iv} —C5B—H5B2	88.5
N1B—B1—N1B ⁱⁱⁱ	110.1 (3)	H5B1—C5B—H5B2	114.9
C3A—N1A—N2A	109.1 (2)	C4B—C6B—C5B	61.2 (2)
C3A—N1A—B1	129.7 (2)	C4B—C6B—H6B1	117.6
N2A—N1A—B1	121.08 (19)	C5B—C6B—H6B1	117.6
C1A—N2A—N1A	106.2 (2)	C4B—C6B—H6B2	117.6
C1A—N2A—K1	93.43 (16)	C5B—C6B—H6B2	117.6
N1A—N2A—K1	110.42 (16)	H6B1—C6B—H6B2	114.8
N1A ⁱⁱⁱ —B1—N1A—C3A	132.8 (3)	N1B ⁱⁱⁱ —B1—N1B—C3B	−141.4 (3)
N1B—B1—N1A—C3A	−107.8 (3)	N1A ⁱⁱⁱ —B1—N1B—N2B	−74.3 (3)
N1B ⁱⁱⁱ —B1—N1A—C3A	12.7 (4)	N1A—B1—N1B—N2B	165.8 (2)
N1A ⁱⁱⁱ —B1—N1A—N2A	−49.62 (18)	N1B ⁱⁱⁱ —B1—N1B—N2B	46.10 (18)
N1B—B1—N1A—N2A	69.7 (3)	C3B—N1B—N2B—C1B	1.5 (3)
N1B ⁱⁱⁱ —B1—N1A—N2A	−169.7 (2)	B1—N1B—N2B—C1B	175.4 (2)
C3A—N1A—N2A—C1A	−0.5 (3)	C3B—N1B—N2B—K1 ^{iv}	106.9 (2)
B1—N1A—N2A—C1A	−178.5 (2)	B1—N1B—N2B—K1 ^{iv}	−79.2 (2)
C3A—N1A—N2A—K1	−100.6 (2)	N1B—N2B—C1B—C2B	−1.2 (4)
B1—N1A—N2A—K1	81.4 (2)	K1 ^{iv} —N2B—C1B—C2B	−119.0 (3)
N1A—N2A—C1A—C2A	0.3 (3)	N1B—N2B—C1B—C4B	177.3 (3)
K1—N2A—C1A—C2A	112.8 (2)	K1 ^{iv} —N2B—C1B—C4B	59.5 (3)
N1A—N2A—C1A—C4A	−178.5 (3)	N1B—N2B—C1B—K1 ^{iv}	117.8 (2)
K1—N2A—C1A—C4A	−66.1 (3)	N2B—C1B—C2B—C3B	0.4 (4)
N1A—N2A—C1A—K1	−112.4 (2)	C4B—C1B—C2B—C3B	−177.8 (3)
N2A—C1A—C2A—C3A	0.0 (4)	K1 ^{iv} —C1B—C2B—C3B	−65.1 (4)
C4A—C1A—C2A—C3A	178.6 (3)	N2B—N1B—C3B—C2B	−1.3 (4)
K1—C1A—C2A—C3A	68.4 (3)	B1—N1B—C3B—C2B	−174.5 (3)
N2A—N1A—C3A—C2A	0.5 (4)	C1B—C2B—C3B—N1B	0.5 (4)
B1—N1A—C3A—C2A	178.3 (3)	N2B—C1B—C4B—C6B	174.4 (3)

C1A—C2A—C3A—N1A	−0.3 (4)	C2B—C1B—C4B—C6B	−7.5 (6)
N2A—C1A—C4A—C6A	−165.9 (3)	K1 ^{iv} —C1B—C4B—C6B	−137.4 (3)
C2A—C1A—C4A—C6A	15.5 (6)	N2B—C1B—C4B—C5B	−115.5 (4)
K1—C1A—C4A—C6A	140.1 (3)	C2B—C1B—C4B—C5B	62.6 (5)
N2A—C1A—C4A—C5A	124.0 (4)	K1 ^{iv} —C1B—C4B—C5B	−67.3 (3)
C2A—C1A—C4A—C5A	−54.6 (5)	N2B—C1B—C4B—K1 ^{iv}	−48.2 (2)
K1—C1A—C4A—C5A	70.0 (3)	C2B—C1B—C4B—K1 ^{iv}	129.9 (4)
N2A—C1A—C4A—K1	54.0 (2)	C1B—C4B—C5B—C6B	−110.9 (4)
C2A—C1A—C4A—K1	−124.6 (4)	K1 ^{iv} —C4B—C5B—C6B	−173.0 (2)
C1A—C4A—C5A—C6A	111.8 (4)	C1B—C4B—C5B—K1 ^{iv}	62.1 (3)
K1—C4A—C5A—C6A	173.7 (3)	C6B—C4B—C5B—K1 ^{iv}	173.0 (2)
C1A—C4A—C6A—C5A	−108.0 (4)	C1B—C4B—C6B—C5B	108.5 (4)
K1—C4A—C6A—C5A	−10.4 (5)	K1 ^{iv} —C4B—C6B—C5B	10.7 (4)
N1A ⁱⁱⁱ —B1—N1B—C3B	98.2 (3)	K1 ^{iv} —C5B—C6B—C4B	−9.1 (3)
N1A—B1—N1B—C3B	−21.7 (4)		

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