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Crystal structure of potassium triethylhydridoborate ('superhydride')

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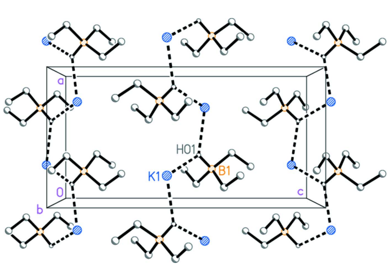
In the title compound, formally $K^+\cdot C_6H_{16}B^-$, the contact sphere of potassium consists of eleven hydrogen atoms from three different anions, assuming an arbitrary cut-off of 3 Å. The shortest interaction, 2.53 (2) Å, involves the hydridic hydrogen H01, which fulfils a bridging function in the formation of chains of $KHBEt_3$ units parallel to the *a* axis [K1—H01^{i} 2.71 (2) Å, K1—H01—K1^{ii} 126.7 (9)°, operators $x\mp1/2, -y+\frac{3}{2}, -z+1$].



1. Chemical context

The title compound $KHBEt_3$ was first prepared by Ziegler and Lehmkuhl from $NaBEt_3H$ and potassium amalgam (Ziegler & Lehmkuhl, 1963), but a more convenient approach was reported a few years later using KH and BEt_3 in toluene (Binger *et al.*, 1968). Alternatively, the latter reaction may also be performed in THF (Brown & Krishnamurthy, 1978). Since its original synthesis this so-called 'superhydride' reagent has found widespread applications, *e.g.* as a reducing reagent in organic synthesis (Brown & Hubbard, 1979; Ito *et al.*, 1985; Yoon *et al.* 1987, 1989), for the generation of low-valent transition-metal complexes (Bönnemann & Korall, 1992), and as a hydride transfer reagent resulting in well-defined metal–hydride complexes (Smith *et al.*, 2003; Pfirrmann *et al.*, 2008; Walter *et al.*, 2011; Maekawa *et al.*, 2012). Despite it being a reagent in frequent use, the structure of $KHBEt_3$ has so far remained elusive. The few reported examples of structures containing $KHBEt_3$ include its adducts with polydentate amines such as *N*,*N*,*N*′,*N*′-tetramethylethylenediamine (TMEDA) and *N*,*N*,*N*′,*N*",*N*"-pentamethyldiethylenetriamine (PMDETA) (Haywood & Wheatley, 2009). During our study on the coordination chemistry of enantiomerically pure constrained-geometry complexes of the rare-earth metals bearing a dianionic *N*-donor functionalized pentadienyl ligand, we accidentally obtained crystals of solvent-free $KHBEt_3$ unsupported by any further ligands (see *Synthesis and crystallization*) and here report its structure.

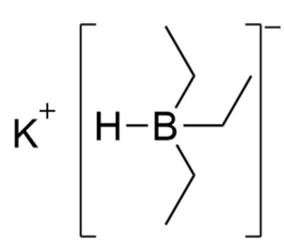


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Table 1
Selected geometric parameters (Å, °).

K1—C1^{i}	3.103 (2)	K1—H2B^{iii}	2.83
K1—B1	3.205 (2)	K1—H3B^{ii}	2.93
K1—C5	3.310 (2)	K1—H3*A*	2.93
K1—C3^{ii}	3.387 (2)	K1—H5A^{ii}	2.94
K1—C5^{ii}	3.396 (2)	K1—H3A^{ii}	2.97
K1—B1^{i}	3.465 (2)	K1—H5B^{ii}	2.99
K1—H01	2.53 (2)	B1—C3	1.640 (3)
K1—H5*B*	2.69	B1—C5	1.640 (3)
K1—H01^{i}	2.71 (2)	B1—C1	1.640 (3)
K1—H1A^{i}	2.76	B1—H01	1.20 (2)
K1—H1B^{i}	2.75		
C3—B1—C5	109.90 (16)	C1—B1—H01	106.1 (10)
C3—B1—C1	112.16 (16)	K1—H01—B1	113.5 (13)
C5—B1—C1	111.40 (16)	K1—H01—K1^{ii}	126.7 (9)
C3—B1—H01	107.6 (10)	H01—K1—H01^{i}	104.0 (4)
C5—B1—H01	109.5 (10)		

Symmetry codes: (i) $x-\frac{1}{2}, -y+\frac{3}{2}, -z+1$; (ii) $x+\frac{1}{2}, -y+\frac{3}{2}, -z+1$; (iii) $-x+\frac{1}{2}, -y+1, z-\frac{1}{2}$.

2. Structural commentary

The asymmetric unit of $KHBEt_3$ is shown in Fig. 1. Selected interatomic distances and angles are shown in Table 1. The shortest contact involving the potassium atom is K1—H01 at 2.53 (2) Å, but K1—H5*B* (not drawn explicitly) is not much longer at 2.69 Å. If the neighbouring asymmetric units generated by the 2_1 screw axis parallel to the *a* axis (see next section) are considered, there are a total of eleven K1—H distances shorter than 3 Å, with no clear limit as to what might be considered a 'bonding' distance. One further such distance involves the 2_1 screw axis parallel to the *c* axis. The environment of the potassium atom is shown in Fig. 2. For comparison, one may note the K—H distance of 2.85 Å in potassium hydride (Kuznetsov & Shkrabkina, 1962), which, however, is regarded as an essentially ionic compound, crystallizing in the NaCl lattice type with coordination number 6 (*cf.* the ionic formulation of the title compound in Table 2, which is certainly a considerable oversimplification). Some K···H contacts of *ca* 2.8–2.9 Å, involving methyl hydrogen atoms, have been postulated as structurally significant in a TMEDA complex of potassium diisopropylamide (Clegg *et al.*, 1998).

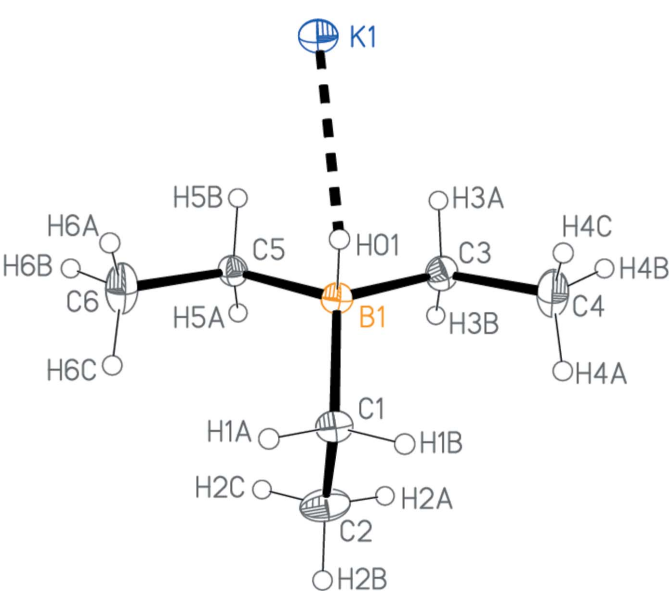

Figure 1
The asymmetric unit of $KHBEt_3$. Ellipsoids are drawn at the 50% level. Only the shortest K1—H contact is drawn explicitly.

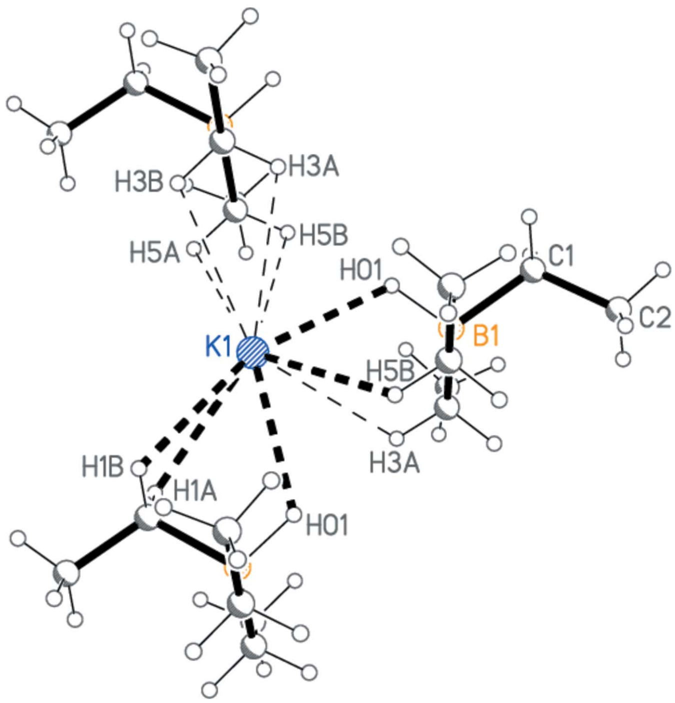

Figure 2
The environment of the potassium atom in $KHBEt_3$, showing ten of the eleven K—H contacts < 3 Å to three neighbouring hydridotriethylborate units. Radii are arbitrary. K—H distances shorter than 2.8 Å are shown as thick dashed bonds, whereas those greater than 2.9 Å are shown as thin dashed bonds. The anion on the right corresponds to the asymmetric unit; the anions at top and bottom were generated by the operators $\frac{1}{2}+x, \frac{3}{2}-y, 1-z$ and $-\frac{1}{2}+x, \frac{3}{2}-y, 1-z$, respectively. The contact to H2*B* of a fourth anion (at $\frac{1}{2}-x, 1-y, -\frac{1}{2}+z$) is omitted for clarity.

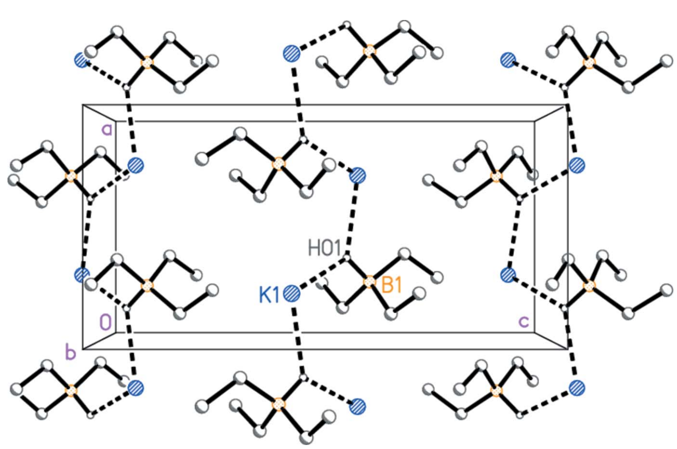

Figure 3
Simplified packing diagram of $KHBEt_3$ viewed parallel to the *b* axis. Hydrogen atoms except for H01 are omitted.

Similarly, the distances from K1 to carbon and boron atoms range upwards from 3.103 (2) and 3.205 (2) Å, respectively. The bonding to CH_n and BH moieties may involve multi-centre interactions, but we do not wish to speculate on their exact nature. The coordination geometry at the boron atom is as expected tetrahedral to a good approximation.

3. Supramolecular features

To a first approximation, ignoring all interactions at K1 except for K1—H01, the molecules are connected by the appropriate 2_1 operators to form chains parallel to the *a* axis (Fig. 3). The hydridic hydrogen atom acts as the main bridging group, with $K1—H01^{i}$ = 2.71 (2) Å, $H01—K1—H01^{i}$ = 104.0 (4)°, $K1—H01—K1^{ii}$ = 126.7 (9)°. The distance between adjacent potassium atoms in the chain is 4.6839 (6) Å.

4. Database survey

A CSD search with *ConQuest* (Bruno *et al.*, 2002) for organic hydridoborate derivatives involving K—H bonds led to the above-mentioned complexes $[K(TMEDA)Et_3BH]_2$ and $[K(PMDETA)Et_3BH]_2$ (Haywood & Wheatley, 2009, refcodes CUNNEF and CUNNIJ) with K—H distances of 2.52, 2.58 (3) and 2.64, 2.69 (3) Å, respectively, in the central K_2H_2 rings. A similar structure (refcode OZAZAR), but with 1,3,5-tri-methyl-1,3,5-triazanonane, was reported by Krieck *et al.* (2010), with K—H = 2.56, 2.59 (3) Å. Somewhat more complex structures, involving cyclic boranes and additional aromatic ligands at the potassium atom, have been reported by Grigsby & Power (1996; refcode TIZYAC, K—H = 2.54, 2.68 Å) and Chen *et al.* [2007; refcode MITWUI, K—H = 2.65–2.92 (1) Å].

5. Synthesis and crystallization

We attempted the preparation of a rare-earth metal hydride by salt metathesis between $[\{(\eta^5{:}\kappa\text{-}N\text{-pdl*SiMe}_2\text{NtBu})\text{La(thf)}\}_2(\mu\text{-Cl})]$ (Jones *et al.*, 2021) and 2 equiv. of $KHBEt_3$ (1 *M* in THF) in *n*-hexane. The standard work-up procedure included removal of the solvent under dynamic vacuum, extraction of the residue with *n*-hexane and filtration. The filtrate was concentrated and cooled to 243 K. After several days, a few pale-yellow crystals were harvested. However, in contrast to our expectations, these did not consist of $[\{(\eta^5{:}\kappa\text{-}N\text{-pdl*SiMe}_2\text{NtBu})\text{La(thf)}\}_2(\mu\text{-H})]$, but of the starting reagent $KHBEt_3$.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The BH hydrogen atom was refined freely. The methyl groups were refined as idealized rigid groups allowed to rotate but not tip (AFIX 137; C—H = 0.98 Å, H—C—H = 109.5 °). The methylene hydrogens were included using a riding model starting from calculated positions (C—H = 0.99 Å). The U_{iso}(H) values were fixed at 1.2 (for methylene groups) or 1.5 (for methyl groups) times the equivalent U_{eq} value of the parent carbon atoms.

Table 2
Experimental details.

Crystal data	
Chemical formula	$K^+ \cdot C_6H_{16}B^-$
M_r	138.10
Crystal system, space group	Orthorhombic, $P2_12_12_1$
Temperature (K)	100
a, *b*, *c* (Å)	7.4758 (3), 7.6682 (6), 14.8010 (12)
V (Å^3)	848.48 (10)
Z	4
Radiation type	Mo *Kα*
μ (mm^{-1})	0.54
Crystal size (mm)	0.3 × 0.2 × 0.15
Data collection	
Diffractometer	Oxford Diffraction Xcalibur, Eos
Absorption correction	Multi-scan (*CrysAlis PRO*; Agilent, 2013)
T_{min}, T_{max}	0.976, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	13206, 2441, 2182
R_{int}	0.060
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.704
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.036, 0.068, 1.04
No. of reflections	2441
No. of parameters	80
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.20, −0.23
Absolute structure	Flack *x* determined using 806 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons*et al.*, 2013)
Absolute structure parameter	−0.05 (3)

Computer programs: *CrysAlis PRO* (Agilent, 2013), *SHELXS97* (Sheldrick, 2008), *SHELXL2018/3* (Sheldrick, 2015) and *XP* (Siemens, 1994).

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supporting information

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Crystal structure of potassium triethylhydridoborate (`superhydride′)

Ann Christin Fecker, Matthias Freytag, Marc D. Walter and Peter G. Jones

Computing details

Data collection: *CrysAlis PRO* (Agilent, 2013); cell refinement: *CrysAlis PRO* (Agilent, 2013); data reduction: *CrysAlis PRO* (Agilent, 2013); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2018/3* (Sheldrick, 2015); molecular graphics: *XP* (Siemens, 1994); software used to prepare material for publication: *SHELXL2018/3* (Sheldrick, 2015).

Potassium triethylhydridoborate

Crystal data

$K^+ \cdot C_6H_{16}B^-$
M_r = 138.10
Orthorhombic, $P2_12_12_1$
a = 7.4758 (3) Å
b = 7.6682 (6) Å
c = 14.8010 (12) Å
V = 848.48 (10) Å^3
Z = 4
F(000) = 304
D_x = 1.081 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2443 reflections
θ = 2.8–26.1°
μ = 0.54 mm^{-1}
T = 100 K
Prism, pale yellow
0.3 × 0.2 × 0.15 mm

Data collection

Oxford Diffraction Xcalibur, Eos diffractometer
Radiation source: Enhance (Mo) X-ray Source
Graphite monochromator
Detector resolution: 16.1419 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (CrysAlisPro; Agilent, 2013)
T_{min} = 0.976, T_{max} = 1.000
13206 measured reflections
2441 independent reflections
2182 reflections with $I > 2\sigma(I)$
R_{int} = 0.060
θ_{max} = 30.0°, θ_{min} = 2.8°
h = −10→10
k = −10→10
l = −20→20

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.068
S = 1.04
2441 reflections
80 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0251P)^2 + 0.0172P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ < 0.001
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}
Absolute structure: Flack x determined using 806 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons*et al.*, 2013)
Absolute structure parameter: −0.05 (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.

Refinement. The compound is achiral and crystallizes only by chance in a chiral (Sohncke) space group.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	y	z	U_{iso}*/U_{eq}
K1	0.22297 (6)	0.87358 (6)	0.42934 (3)	0.01798 (12)
B1	0.2627 (3)	0.6127 (3)	0.59734 (14)	0.0140 (4)
H01	0.368 (3)	0.680 (3)	0.5467 (14)	0.020 (6)*
C1	0.3821 (3)	0.5161 (3)	0.67567 (13)	0.0159 (4)
H1B	0.457483	0.426196	0.646460	0.019*
H1A	0.463245	0.603494	0.702910	0.019*
C2	0.2753 (3)	0.4296 (3)	0.75137 (15)	0.0260 (5)
H2C	0.200842	0.517218	0.781547	0.039*
H2B	0.357773	0.377763	0.795257	0.039*
H2A	0.198637	0.338312	0.725885	0.039*
C3	0.1433 (3)	0.4737 (3)	0.53862 (14)	0.0184 (5)
H3B	0.053383	0.420846	0.579383	0.022*
H3A	0.077404	0.539343	0.491626	0.022*
C4	0.2465 (3)	0.3262 (3)	0.49238 (16)	0.0271 (5)
H4C	0.342593	0.375726	0.455348	0.041*
H4B	0.164952	0.259304	0.453846	0.041*
H4A	0.298115	0.249185	0.538342	0.041*
C5	0.1323 (3)	0.7630 (3)	0.64061 (14)	0.0150 (4)
H5B	0.067029	0.820394	0.590574	0.018*
H5A	0.042278	0.704783	0.679326	0.018*
C6	0.2249 (3)	0.9042 (3)	0.69656 (16)	0.0270 (5)
H6C	0.284544	0.850412	0.748517	0.041*
H6B	0.135728	0.988325	0.717930	0.041*
H6A	0.313644	0.964283	0.659107	0.041*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
K1	0.01299 (19)	0.0216 (2)	0.0194 (2)	0.00053 (18)	−0.00233 (19)	0.0028 (2)
B1	0.0099 (10)	0.0164 (10)	0.0156 (10)	0.0015 (10)	0.0008 (8)	0.0005 (8)
C1	0.0112 (10)	0.0187 (10)	0.0178 (11)	0.0003 (8)	0.0013 (8)	0.0028 (9)
C2	0.0195 (11)	0.0318 (12)	0.0267 (12)	0.0025 (10)	0.0045 (10)	0.0133 (9)
C3	0.0145 (10)	0.0199 (11)	0.0207 (11)	0.0033 (8)	−0.0003 (8)	−0.0018 (8)
C4	0.0236 (13)	0.0251 (11)	0.0326 (12)	0.0052 (9)	−0.0056 (10)	−0.0120 (9)
C5	0.0130 (10)	0.0163 (10)	0.0158 (10)	−0.0002 (8)	−0.0018 (8)	−0.0013 (8)
C6	0.0185 (10)	0.0248 (12)	0.0377 (13)	0.0016 (10)	−0.0033 (11)	−0.0127 (9)

Geometric parameters (Å, º)

K1—C1^{i}	3.103 (2)	B1—C1	1.640 (3)
K1—B1	3.205 (2)	B1—H01	1.20 (2)
K1—C5	3.310 (2)	C1—C2	1.527 (3)
K1—C3^{ii}	3.387 (2)	C1—H1B	0.9900
K1—C5^{ii}	3.396 (2)	C1—H1A	0.9900
K1—B1^{i}	3.465 (2)	C2—H2C	0.9800
K1—C2^{iii}	3.513 (2)	C2—H2B	0.9800
K1—C3	3.518 (2)	C2—H2A	0.9800
K1—H01	2.53 (2)	C3—C4	1.530 (3)
K1—H5B	2.69	C3—H3B	0.9900
K1—H01^{i}	2.71 (2)	C3—H3A	0.9900
K1—H1A^{i}	2.76	C4—H4C	0.9800
K1—H1B^{i}	2.75	C4—H4B	0.9800
K1—H2B^{iii}	2.83	C4—H4A	0.9800
K1—H3B^{ii}	2.93	C5—C6	1.529 (3)
K1—H3A	2.93	C5—H5B	0.9900
K1—H5A^{ii}	2.94	C5—H5A	0.9900
K1—H3A^{ii}	2.97	C6—H6C	0.9800
K1—H5B^{ii}	2.99	C6—H6B	0.9800
B1—C3	1.640 (3)	C6—H6A	0.9800
B1—C5	1.640 (3)		

C1^{i}—K1—B1	129.41 (5)	B1—C1—H1A	108.4
C1^{i}—K1—C5	112.01 (5)	K1^{ii}—C1—H1A	61.0
B1—K1—C5	29.10 (5)	H1B—C1—H1A	107.5
C1^{i}—K1—C3^{ii}	137.56 (5)	C1—C2—K1^{iv}	146.66 (14)
B1—K1—C3^{ii}	91.22 (5)	C1—C2—H2C	109.5
C5—K1—C3^{ii}	98.42 (5)	K1^{iv}—C2—H2C	96.6
C1^{i}—K1—C5^{ii}	132.18 (5)	C1—C2—H2B	109.5
B1—K1—C5^{ii}	87.74 (5)	K1^{iv}—C2—H2B	39.9
C5—K1—C5^{ii}	113.18 (5)	H2C—C2—H2B	109.5
C3^{ii}—K1—C5^{ii}	46.63 (5)	C1—C2—H2A	109.5
C1^{i}—K1—B1^{i}	28.23 (5)	K1^{iv}—C2—H2A	79.5
B1—K1—B1^{i}	101.50 (5)	H2C—C2—H2A	109.5
C5—K1—B1^{i}	84.95 (5)	H2B—C2—H2A	109.5
C3^{ii}—K1—B1^{i}	157.94 (5)	C4—C3—B1	116.29 (17)
C5^{ii}—K1—B1^{i}	150.39 (5)	C4—C3—K1^{i}	141.80 (14)
C1^{i}—K1—C2^{iii}	78.93 (6)	B1—C3—K1^{i}	101.88 (11)
B1—K1—C2^{iii}	99.69 (6)	C4—C3—K1	110.68 (14)
C5—K1—C2^{iii}	122.65 (5)	B1—C3—K1	65.47 (10)
C3^{ii}—K1—C2^{iii}	109.29 (5)	K1^{i}—C3—K1	85.41 (5)
C5^{ii}—K1—C2^{iii}	64.15 (5)	C4—C3—H3B	108.2
B1^{i}—K1—C2^{iii}	86.47 (5)	B1—C3—H3B	108.2
C1^{i}—K1—C3	109.21 (5)	K1^{i}—C3—H3B	55.0
B1—K1—C3	27.73 (5)	K1—C3—H3B	138.8
C5—K1—C3	46.18 (5)	C4—C3—H3A	108.2

C3^{ii}—K1—C3	113.21 (5)	B1—C3—H3A	108.2
C5^{ii}—K1—C3	91.37 (5)	K1^{i}—C3—H3A	57.4
B1^{i}—K1—C3	84.88 (5)	K1—C3—H3A	47.0
C2^{iii}—K1—C3	76.61 (5)	H3B—C3—H3A	107.4
C1^{i}—K1—H01	148.8 (5)	C3—C4—H4C	109.5
B1—K1—H01	20.1 (5)	C3—C4—H4B	109.5
C5—K1—H01	44.7 (5)	H4C—C4—H4B	109.5
C3^{ii}—K1—H01	73.1 (5)	C3—C4—H4A	109.5
C5^{ii}—K1—H01	69.0 (5)	H4C—C4—H4A	109.5
B1^{i}—K1—H01	121.4 (5)	H4B—C4—H4A	109.5
C2^{iii}—K1—H01	97.1 (5)	C6—C5—B1	116.11 (17)
C3—K1—H01	40.9 (5)	C6—C5—K1	103.74 (13)
C3—B1—C5	109.90 (16)	B1—C5—K1	71.90 (10)
C3—B1—C1	112.16 (16)	C6—C5—K1^{i}	142.33 (14)
C5—B1—C1	111.40 (16)	B1—C5—K1^{i}	101.54 (11)
C3—B1—K1	86.80 (11)	K1—C5—K1^{i}	88.60 (5)
C5—B1—K1	79.00 (11)	C6—C5—H5B	108.3
C1—B1—K1	151.74 (13)	B1—C5—H5B	108.3
C3—B1—K1^{ii}	119.99 (12)	K1—C5—H5B	44.0
C5—B1—K1^{ii}	127.85 (13)	K1^{i}—C5—H5B	57.7
C1—B1—K1^{ii}	63.51 (10)	C6—C5—H5A	108.3
K1—B1—K1^{ii}	89.12 (5)	B1—C5—H5A	108.3
C3—B1—H01	107.6 (10)	K1—C5—H5A	143.3
C5—B1—H01	109.5 (10)	K1^{i}—C5—H5A	54.9
C1—B1—H01	106.1 (10)	H5B—C5—H5A	107.4
K1—B1—H01	46.4 (10)	C5—C6—H6C	109.5
K1^{ii}—B1—H01	42.8 (10)	C5—C6—H6B	109.5
C2—C1—B1	115.49 (18)	H6C—C6—H6B	109.5
C2—C1—K1^{ii}	156.25 (14)	C5—C6—H6A	109.5
B1—C1—K1^{ii}	88.26 (11)	H6C—C6—H6A	109.5
C2—C1—H1B	108.4	H6B—C6—H6A	109.5
B1—C1—H1B	108.4	K1—H01—B1	113.5 (13)
K1^{ii}—C1—H1B	60.2	K1—H01—K1^{ii}	126.7 (9)
C2—C1—H1A	108.4	H01—K1—H01^{i}	104.0 (4)

C3—B1—C1—C2	−66.0 (2)	K1^{ii}—B1—C3—K1^{i}	−166.41 (7)
C5—B1—C1—C2	57.7 (2)	C5—B1—C3—K1	77.09 (14)
K1—B1—C1—C2	164.9 (2)	C1—B1—C3—K1	−158.41 (16)
K1^{ii}—B1—C1—C2	−179.55 (19)	K1^{ii}—B1—C3—K1	−87.13 (10)
C3—B1—C1—K1^{ii}	113.58 (14)	C3—B1—C5—C6	−179.27 (18)
C5—B1—C1—K1^{ii}	−122.76 (14)	C1—B1—C5—C6	55.8 (2)
K1—B1—C1—K1^{ii}	−15.5 (3)	K1—B1—C5—C6	−96.78 (17)
B1—C1—C2—K1^{iv}	161.9 (2)	K1^{ii}—B1—C5—C6	−16.6 (3)
K1^{ii}—C1—C2—K1^{iv}	−17.0 (6)	C3—B1—C5—K1	−82.48 (14)
C5—B1—C3—C4	179.06 (18)	C1—B1—C5—K1	152.57 (16)
C1—B1—C3—C4	−56.4 (2)	K1^{ii}—B1—C5—K1	80.16 (12)
K1—B1—C3—C4	101.97 (17)	C3—B1—C5—K1^{i}	2.19 (17)
K1^{ii}—B1—C3—C4	14.8 (2)	C1—B1—C5—K1^{i}	−122.76 (14)

C5—B1—C3—K1^{i}	−2.19 (17)	K1—B1—C5—K1^{i}	84.67 (6)
C1—B1—C3—K1^{i}	122.31 (13)	K1^{ii}—B1—C5—K1^{i}	164.83 (9)
K1—B1—C3—K1^{i}	−79.28 (7)		

Symmetry codes: (i) $x-1/2$, $-y+3/2$, $-z+1$; (ii) $x+1/2$, $-y+3/2$, $-z+1$; (iii) $-x+1/2$, $-y+1$, $z-1/2$; (iv) $-x+1/2$, $-y+1$, $z+1/2$.