

Franz Dornhaus and Michael  
Bolte\*Institut für Anorganische Chemie, J. W. Goethe-  
Universität Frankfurt, Marie-Curie-Strasse 11,  
60439 Frankfurt/Main, GermanyCorrespondence e-mail:  
bolte@chemie.uni-frankfurt.de

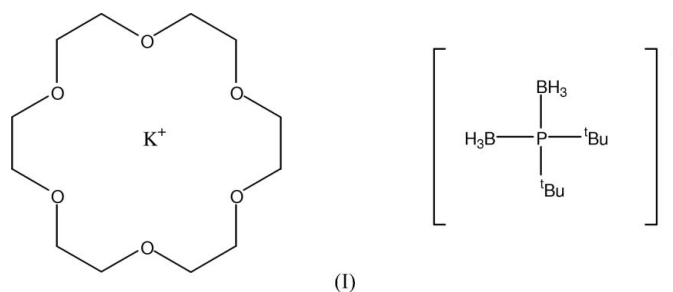
## Key indicators

Single-crystal X-ray study  
 $T = 173$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å  
 $R$  factor = 0.027  
 $wR$  factor = 0.049  
Data-to-parameter ratio = 18.6For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.(18-Crown-6)potassium di-*tert*-butyl-  
phospanylbisborohydrideThe structure of the title compound,  $[\text{K}(\text{C}_{12}\text{H}_{24}\text{O}_6)](\text{C}_8\text{H}_{24}\text{B}_2\text{P})$ , is composed of di-*tert*-butylphospanylbisborohydride anions and K cations which are coordinated by a crown ether molecule.

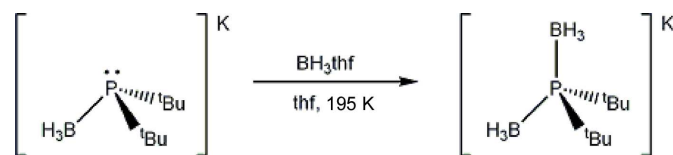
Received 21 November 2006

Accepted 21 November 2006

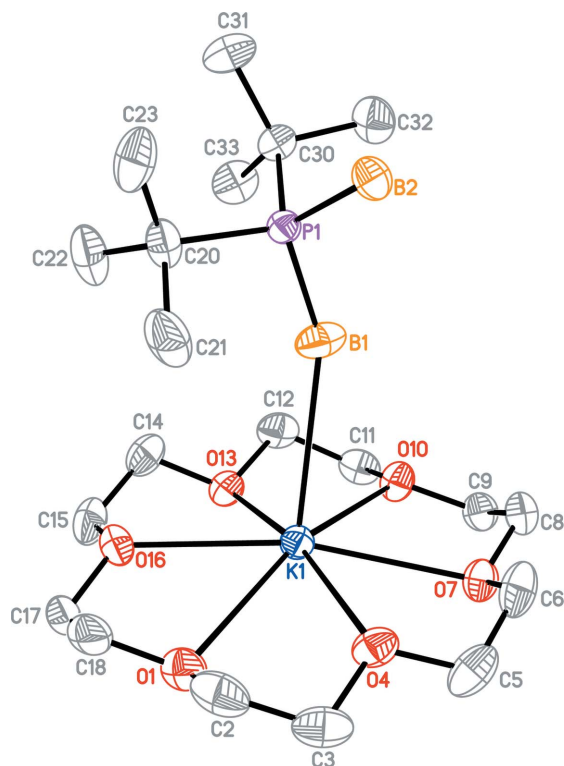
## Comment

The title compound, (I), is the *tert*-butyl derivative of the previously published compound  $[\text{K}(18\text{-crown-6})][(\text{BH}_3)_2\text{PPh}_2]$ . The structure of  $[\text{K}(18\text{-crown-6})][(\text{BH}_3)_2\text{PPh}_2]$  was discussed in a comparison of the Lewis basicities of  $[\text{BH}_3\text{PPh}_2]^-$ ,  $\text{CH}_3\text{PPh}_2$  and  $\text{HPPH}_2$  (Dornhaus *et al.*, 2006).

The anion of compound (I) represents the  $\text{BH}_3$  adduct of the Lewis base  $[\text{BH}_3\text{P}^t\text{Bu}_2]^-$ . Studies concerning the structure and coordination chemistry of this anion were published recently (Kückmann *et al.*, 2007). Moreover, there are various examples of  $[\text{BH}_3\text{PR}_2]^-$  ( $R = \text{alkyl, aryl}$ ) as a ligand in transition metal chemistry (Dorn *et al.*, 2000; Gaumont *et al.*, 1999; Angerer *et al.*, 1985; Hoic *et al.*, 1996; Nakazawa *et al.*, 1997), as a building block in main group chemistry (Müller & Brand, 2003; Jaska *et al.*, 2004) and in the synthesis of organophosphorus compounds (Camus *et al.*, 2004; Wolfe & Livinghouse, 1998; Wolfe & Livinghouse, 2001; Miura *et al.*, 2000). As in the case of  $\text{K}[(\text{BH}_3)_2\text{PPh}_2]$ , X-ray quality crystals of  $\text{K}[(\text{BH}_3)_2\text{P}^t\text{Bu}_2]$  could be obtained only as the 18-crown-6 adduct. The synthesis of  $\text{K}[(\text{BH}_3)_2\text{P}^t\text{Bu}_2]$  was achieved by combination of a THF solution of  $\text{K}[(\text{BH}_3)_2\text{P}^t\text{Bu}_2]$  with a calibrated solution of  $\text{BH}_3 \cdot \text{THF}$  in THF at 195 K



A perspective view of the title compound is shown in Fig. 1. Bond lengths and angles can be regarded as normal


**Figure 1**

The molecular structure of the title compound, showing the atom numbering and displacement ellipsoids drawn at the 50% probability level. H atoms have been omitted for clarity.

(Cambridge Structural Database, Version 5.27, November 2005 update, August 2006; *MOGUL* Version 1.1; Allen, 2002). The structure of the title compound is composed of di-*tert*-butylphosphanylborohydride anions and K cations, with crown ether molecules coordinating the K ions. The K–O distances range from 2.7659 (12) to 2.8860 (14) Å. There is one short K···B distance of 3.117 (2) Å. The two P–B distances are almost the same length (Table 1).

## Experimental

Reaction and manipulations were carried out under an atmosphere of dry nitrogen using standard Schlenk techniques. Solvents were freshly distilled under argon from sodium/benzophenone [tetrahydrofuran (THF), diethyl ether, C<sub>6</sub>D<sub>6</sub>] or sodium–lead alloy (pentane) prior to use. To a stirred solution of K[BH<sub>3</sub>P'Bu<sub>2</sub>] (322 mg, 1.63 mmol) in THF (5 ml) was added a calibrated solution (1 mol l<sup>-1</sup>) of BH<sub>3</sub>·THF (1.63 ml, 1.63 mol) at 195 K with stirring. After 30 min, the cooling bath was removed and the reaction mixture was allowed to warm to ambient temperature with subsequent stirring for another 60 min. All volatiles were removed *in vacuo*; the resulting colourless solid was washed with pentane (2 × 2 ml) (yield: 316 mg, 92%). X-ray quality crystals of K[(BH<sub>3</sub>)<sub>2</sub>P'Bu<sub>2</sub>] were grown by gas-phase diffusion of diethyl ether into a solution containing (I) and an equimolar amount of 18-crown-6. The NMR spectra were recorded on Bruker AMX 250 and AV 400 spectrometers. NMR measurements were performed with K[(BH<sub>3</sub>)<sub>2</sub>P'Bu<sub>2</sub>] in undeuterated THF solution. Approximately 0.1 ml of C<sub>6</sub>D<sub>6</sub> was added to the sample to provide a lock signal. <sup>1</sup>H and <sup>13</sup>C NMR shifts are reported

relative to tetramethylsilane and were referenced against residual solvent peaks (C<sub>6</sub>D<sub>5</sub>H: δ<sup>1H</sup> = 7.16, C<sub>6</sub>D<sub>6</sub>: δ<sup>13C</sup> = 128.06) (Gottlieb *et al.*, 1997). The <sup>11</sup>B NMR spectrum was referenced against external BF<sub>3</sub>·OEt<sub>2</sub>; the <sup>31</sup>P NMR spectrum is reported relative to external H<sub>3</sub>PO<sub>4</sub> (85%). Abbreviations: *d* = doublet, *qa* = quartet, *m* = multiplet, *br* = broad. <sup>1</sup>H NMR (THF, 250.13 MHz): δ 0.3 (*br qa*, <sup>1</sup>J<sub>BH</sub> = 89 Hz, 6H, BH<sub>3</sub>), 1.17 (*d*, <sup>3</sup>J<sub>PH</sub> = 10.8 Hz, 18H, CH<sub>3</sub>); <sup>11</sup>B{<sup>1</sup>H} NMR (THF, 128.38 MHz): δ -38.1 (*d*, <sup>1</sup>J<sub>PB</sub> = 69.5 Hz, BH<sub>3</sub>); <sup>13</sup>C{<sup>1</sup>H} NMR (THF, 62.90 MHz): δ 29.5 (*d*, <sup>2</sup>J<sub>PC</sub> = 1.4 Hz, CH<sub>3</sub>), 30.1 (*d*, <sup>1</sup>J<sub>PC</sub> = 21.2 Hz, CCH<sub>3</sub>); <sup>31</sup>P{<sup>1</sup>H} NMR (THF, 161.98 MHz): δ 18.3 (*m*).

## Crystal data

[K(C<sub>12</sub>H<sub>24</sub>O<sub>6</sub>)](C<sub>8</sub>H<sub>24</sub>B<sub>2</sub>P)  
*M<sub>r</sub>* = 476.27  
 Monoclinic, *P*2<sub>1</sub>/*n*  
*a* = 9.5690 (13) Å  
*b* = 15.8008 (15) Å  
*c* = 18.850 (2) Å  
 β = 102.585 (11)°  
*V* = 2781.6 (6) Å<sup>3</sup>

*Z* = 4  
*D<sub>x</sub>* = 1.137 Mg m<sup>-3</sup>  
 Mo Kα radiation  
 μ = 0.28 mm<sup>-1</sup>  
*T* = 173 (2) K  
 Block, colourless  
 0.48 × 0.43 × 0.37 mm

## Data collection

Stow IPDS-II two-circle diffractometer  
 ω scans  
 Absorption correction: multi-scan (*MULABS*; Spek, 2003; Blessing, 1995)  
*T<sub>min</sub>* = 0.878, *T<sub>max</sub>* = 0.904

12395 measured reflections  
 5075 independent reflections  
 3446 reflections with *I* > 2σ(*I*)  
*R<sub>int</sub>* = 0.031  
 θ<sub>max</sub> = 25.8°

## Refinement

Refinement on *F*<sup>2</sup>  
*R*[*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.028  
*wR*(*F*<sup>2</sup>) = 0.049  
*S* = 1.02  
 5075 reflections  
 273 parameters

H-atom parameters constrained  
*w* = 1/[σ<sup>2</sup>(*F<sub>o</sub>*<sup>2</sup>) + (0.01*P*)<sup>2</sup>]  
 where *P* = (*F<sub>o</sub>*<sup>2</sup> + 2*F<sub>c</sub>*<sup>2</sup>)/3  
 (Δ/σ)<sub>max</sub> = 0.001  
 Δρ<sub>max</sub> = 0.19 e Å<sup>-3</sup>  
 Δρ<sub>min</sub> = -0.16 e Å<sup>-3</sup>

**Table 1**

Selected bond lengths (Å).

K1–O16	2.7659 (12)	K1–O13	2.8562 (12)
K1–O10	2.7671 (12)	K1–O1	2.8860 (14)
K1–O4	2.8082 (12)	P1–B1	1.9397 (18)
K1–O7	2.8170 (11)	P1–B2	1.940 (2)

H atoms were refined with fixed individual displacement parameters [*U*<sub>iso</sub>(H) = 1.2*U*<sub>eq</sub>(C) or 1.5 *U*<sub>eq</sub>(methyl C,B)] using a riding-model with C–H = 0.99 Å for methylene and 0.98 Å for methyl and BH<sub>3</sub> H atoms, respectively. The BH<sub>3</sub> groups were allowed to rotate but not to tip.

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL97*.

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