

**DIMETHYL SULFATE INDUCED NUCLEOPHILIC SUBSTITUTION OF THE [BIS(1,2-DICARBOLLIDO)-3-COBALT(1-)]ATE ION. SYNTHESSES, PROPERTIES AND STRUCTURES OF ITS 8,8'- $\mu$ -SULFATO, 8-PHENYL AND 8-DIOXANE DERIVATIVES**

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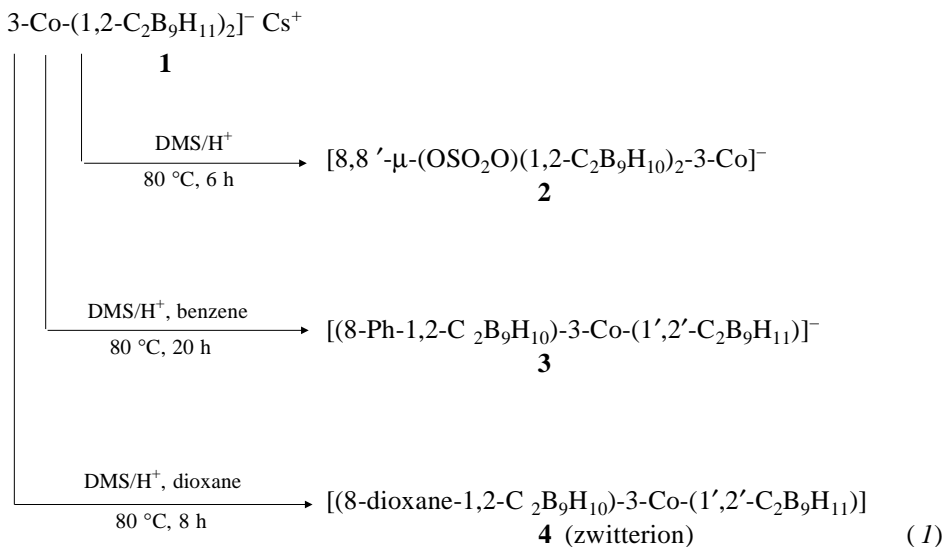
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Syntheses and structures of  $[8,8'\text{-}\mu\text{-(OSO}_2\text{O)(1,2-C}_2\text{B}_9\text{H}_{10}\text{)}_2\text{-3-Co}]^-$  and  $[(8\text{-Ph-1,2-C}_2\text{B}_9\text{H}_{10}\text{)-3-Co-(1',2'\text{-C}_2\text{B}_9\text{H}_{11})}]^-$  ions and of  $[(8\text{-dioxane-1,2-C}_2\text{B}_9\text{H}_{10}\text{)-3-Co-(1',2'\text{-C}_2\text{B}_9\text{H}_{11})}]^0$  zwitterion are described. All compounds result on dimethyl sulfate induced nucleophilic B-substitution of the parent cobalta-carborane sandwich ion under acid catalysis. A general mechanism is proposed. Constitutions of all compounds are inferred from the mass and multinuclear NMR spectroscopy and all structures are established by X-ray diffraction. Selected NMR and X-ray data are presented.

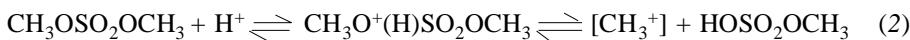
**Key words:** Electrophile induced nucleophilic substitutions; Cobaltacarborane sandwich complexes; X-Ray structures; Multinuclear NMR;  $-\text{OSO}_2\text{O}-$  bridge.

The  $[3\text{-Co-(1,2-C}_2\text{B}_9\text{H}_{11}\text{)}_2]^-$  ion<sup>1</sup> is notoriously known to react with various nucleophilic reagents in the presence of strong acids under formation of B(8,8') substituted derivatives of **1** or zwitterions. For these curious reaction sequences we coined<sup>2</sup> the designation "Electrophile Induced Nucleophilic Substitution" (EINS), which is characterized by the following processes: (i) the electrophilic particle removes the terminal hydrogen from the most electron-rich B-H vertex; (ii) the transient vacancy at this B-vertex is then filled by the most nucleophilic or most abundant particle present in the reaction medium.

A variety of 8,8'-bridged derivatives of **1** has been prepared *via* this EINS-type route, e.g. O (refs<sup>3-5</sup>), S (refs<sup>3,6,7</sup>), Se (ref.<sup>3</sup>), Te (ref.<sup>3</sup>), NR<sub>2</sub> (refs<sup>3,8</sup>) and I (refs<sup>9,10</sup>) monatomically bridged species; *o*-arylene<sup>9-11</sup>, bis(*o*-arylene)<sup>11</sup>, S<sub>2</sub> (ref.<sup>6</sup>) and  $(-\text{ONH}_2-)$  (ref.<sup>12</sup>) derivatives with diatomic bridges;  $(-\text{OC}(\text{CH}_3)\text{O}-)$  (ref.<sup>13</sup>) and  $(-\text{SCHS}-)$  (refs<sup>13,14</sup>) derivatives with triatomic bridges. Here we report on the acid catalyzed reactions of the ion **1** with dimethyl sulfate (DMS) without solvent, in benzene and in dioxane.



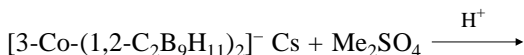
The structures of the compounds isolated are shown in Figs 1–3. For a successful reaction, acid catalysis seems to be essential and its role might be explained as follows.

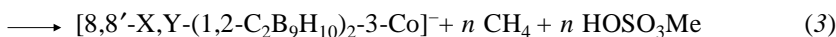


The methylium ion then acts as a hydride ion scavenger in the initial phase of the EINS type reaction. An excess of DMS leads to the ion **2** (apparently *via* the 8-CH<sub>3</sub>OSO<sub>2</sub>O<sup>-</sup> intermediate); with a small amount of DMS in benzene the ion **3** becomes the main product, and the zwitterion **4** results from the reaction with dioxane.

Because HOSO<sub>2</sub>OCH<sub>3</sub> is liberated on consumption of the CH<sub>3</sub><sup>+</sup> ion, the reaction might be autocatalytic; however, it is questionable whether this acid is strong enough to induce DMS activation. We have therefore eliminated this autocatalytic potentiality by using a small excess of H<sub>2</sub>SO<sub>4</sub> (or AlCl<sub>3</sub>) in all the cases investigated.

Both positions B(8) and B(8') in **1** are equivalent. Several reaction pathways have to be considered in the reactions below.





Solvent	X	Y	Code
None	MeOSO <sub>2</sub> O-	H-	<b>A</b>
	-O-SO <sub>2</sub> -O-	-O-SO <sub>2</sub> -O-	<b>2</b>
	MeOSO <sub>2</sub> O-	MeOSO <sub>2</sub> O-	<b>B</b>
Benzene	Ph-	H-	<b>3</b>
	Ph-	MeOSO <sub>2</sub> O-	<b>C</b>
	Ph-	Ph-	<b>D</b>
Dioxane	O(CH <sub>2</sub> CH <sub>2</sub> ) <sub>2</sub> <sup>+</sup> -	H-	<b>4</b>

With DMS alone, the species **A**, **B** and **2** might result. On treatment of the reaction mixture with Na<sub>2</sub>SO<sub>3</sub> or ammonium hydroxide, **A** and **B** are converted to water-soluble salts of the respective HOSO<sub>2</sub>O- derivatives, whereas **2** survives intact. The amazing stability of **2** is apparently due to the fact that the B-O bond is approximately as strong as the S-O bond and that S<sub>N</sub>1 or S<sub>N</sub>2 reactions are sterically impossible with deltahe-dral leaving groups.

If benzene is used as solvent, compounds **A-D**, **2** and **3** might result; on treatment with Lewis bases as above only **2**, **3** and **D** shall survive. In this case, however, **3** is actually the main product and its MeN<sub>4</sub><sup>+</sup> salt crystallizes in the form of well developed prisms. In fact, we have no evidence on the formation of **D** so far.

In the last case, the zwitterion **4** is the single uncharged product of the reaction and can be easily separated from by-products by extraction into benzene or methylene chloride, followed by column chromatography on silica gel. Here, however, any treatment of the reaction mixture with Lewis bases must be avoided, because otherwise the dioxane ring would open as described earlier for its 8'-iodinated analogue<sup>15</sup>.

The constitution of compounds **2-4** has been inferred from the standard combination of mass and multinuclear NMR spectroscopy; the relevant data are gathered in Tables I and II. Comparison of the chemical shifts in the "unsubstituted" parts of the molecules clearly indicates that the remote substituent distinctly alters the chemical shifts in the "unsubstituted" parts of the sandwiches. We are still far from understanding the nature of this "long range" effect. Similar peculiarities are seen in the C-H carborane and <sup>11</sup>B decoupled <sup>1</sup>H signals. Also noteworthy is the distinct extension of the span of the <sup>11</sup>B NMR spectra of the substituted C<sub>2</sub>B<sub>9</sub>H<sub>10</sub><sup>-</sup> ligands caused by any substitution at B(8).

The structures of all three compounds **2-4** have been established by the X-ray dif-fraction, and are shown in Figs 1-3. Selected bond lengths and angles are listed in

Tables III and IV\*. A notable feature is the essential parallelity of both pentagonal ligand planes along with a staggered (antiprismatic) conformation in all cases.

Noteworthy are also the different mutual arrangements of the C–H pairs in both pentagonal ligand planes in **3** and **4**. In the former compound they are opposite,

TABLE I  
Yields and some characteristics of tetramethylammonium salts of ions **2** and **3**, and of zwitterion **4**

Compound	Yield %	TLC <sup>a</sup> <i>R<sub>F</sub></i>	MS <sup>b</sup> <i>m/z</i>	<sup>1</sup> H NMR (δ, ppm)				
				C–H <sub>carb</sub> (int.)		Other CH <sub>x</sub>		
<b>2</b>	40.1	0.38	421	3.97(4)		–		
<b>3</b>	25.0	0.40	403	3.78(2)	4.58(2)	7.03(1)	7.11(2)	7.31(2)
<b>4</b>	44.7	0.48	414	4.04(2)	4.92(2)	4.71(4)	4.07(4)	

<sup>a</sup> On Silufol; compounds **2** and **3** in acetonitrile–chloroform (1 : 2), **4** in benzene; <sup>b</sup> anions **2** and **3** were measured by the FAB method, *m/z* of the zwitterion **4** was established by conventional technique. The found values agree with the calculated ones for <sup>12</sup>C<sub>4</sub><sup>11</sup>B<sub>18</sub><sup>1</sup>H<sub>20</sub><sup>16</sup>O<sub>4</sub><sup>32</sup>S<sup>59</sup>Co (**2**), <sup>12</sup>C<sub>10</sub><sup>11</sup>B<sub>18</sub><sup>1</sup>H<sub>26</sub><sup>59</sup>Co (**3**) and <sup>12</sup>C<sub>8</sub><sup>11</sup>B<sub>18</sub><sup>1</sup>H<sub>29</sub><sup>16</sup>O<sup>59</sup>Co (**4**).

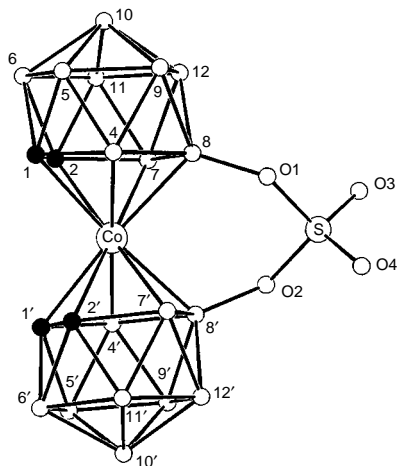


FIG. 1  
Structure of **2**

\* Complete X-ray data have been sent to Fachinformationszentrum Karlsruhe, 76344 Eggemstein, Leopoldshafen (crysdata@fiz-karlsruhe.de) (but might be also obtained on request from the authors of this paper).

TABLE II  
The  $^{11}\text{B}$  NMR and ( $^{11}\text{B}$ )-decoupled  $^1\text{H}$  NMR spectra<sup>a</sup> of tetramethylammonium salts of ions **2** and **3** and the zwitterion **4**

Position	Substituted nucleus		Position	Unsubstituted nucleus	
	$^{11}\text{B}$	( $^{11}\text{B}$ )- $^1\text{H}$		$^{11}\text{B}$	( $^{11}\text{B}$ )- $^1\text{H}$
<b>Compound 2</b>					
8,8'	22.97 s	–			
10,10'	–2.38	2.70			
4,7; 4',7'	–5.89	2.21			
9,12; 9',12'	–8.18	3.13			
5,11; 5',11'	–18.74	1.62			
6,6'	–27.23	1.51			
Span: 50.20 ppm					
<b>Compound 3</b>					
8	12.53 s	–	8'	4.97	2.51
10	–1.77	3.10	10'	–1.77	3.10
4,7	–5.38	1.93	4',7'	–3.63	2.77
9,12	–6.01	3.12	9',12'	–6.98	1.75
5,11	–18.72	1.71	5',11'	–17.41	1.48
6	–23.29	1.76	6'	–21.96	1.30
Span: 35.82 ppm					
<b>Compound 4</b>					
8	22.27 s	–	8'	8.03	3.44
10	–3.03	2.77	10'	4.51	3.22
4,7	–8.35	2.32	4',7'	–4.51	2.16
9,12	–9.68	3.21	9',12'	–4.51	2.16
5,11	–18.84	1.69	5',11'	–15.36	1.77
6	–26.87	1.54	6'	–21.27	1.78
Span: 49.14 ppm					

<sup>a</sup> The assignments are based on  $^{11}\text{B}$ - $^{11}\text{B}$  COSY 2D NMR and on ( $^{11}\text{B}$ ) decoupled  $^1\text{H}$  NMR spectra. These records are available on request. Due to several near overlaps the coupling constants  $\{J_{\text{BH}}\}$  are omitted.

whereas they are adjacent in the latter. Coulombic forces might be responsible for this difference.

In structure **3**, an attraction between the slightly positive C–H vertices of the unsubstituted deltahedral ligand and the slightly negative phenyl ring (attached to the most negative B(8) vertex) should be expected. On the other hand, in **4** there should be repulsion between the analogous C–H vertices and the positively charged oxonium

TABLE III  
Selected bond lengths (in ppm) in compounds **2–4**

Bond	<b>2</b>	<b>3</b>	<b>4</b>
C1–C2	163.0	161.8(5)	161.8
C1–B4	169.9	171.1(6)	171.4
B4–B8	182.7	178.2(6)	178.4
C1–Co	206.2	200.9(4)	203.7
B4–Co	208.3	209.9(4)	209.0
B8–Co	210.0	216.2(4)	210.3
B8–O	144.6	–	–
O–SO <sub>2</sub>	153.4	–	–
S–O	142.2	–	–
B8–Ph	–	157.7(5)	–
B8–O(diox)	–	–	151.7

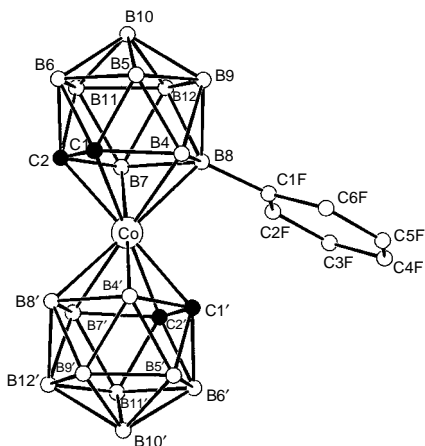


FIG. 2  
Structure of **3**

group of the dioxane component. The results presented here indicate that EINS-type substitutions promoted by the DMS–acid system might be quite general for other weak Lewis bases and other deltahedral anionic substrates.

TABLE IV  
Selected angles (in °) in compounds 2–4

Atoms	Compound		
	2	3	4
C1–C2–B7	112.9	111.2	111.8
C2–B7–B8	104.9	105.8	103.9
B4–B8–B7	105.4	106.4	108.6
B8–Co–B8	90.6	178.7	96.7
Co–B8–OSO <sub>2</sub>	116.7	–	–
B8–O–SO <sub>2</sub>	127.6	–	–
O–SO <sub>2</sub> –O	105.8	–	–
O=S=O	116.5	–	–
Co–B8–Ph	–	116.8	–
Co–B8–O(diox)	–	–	117.6
Dihedral angle between ligand planes	3.94	–	0.74

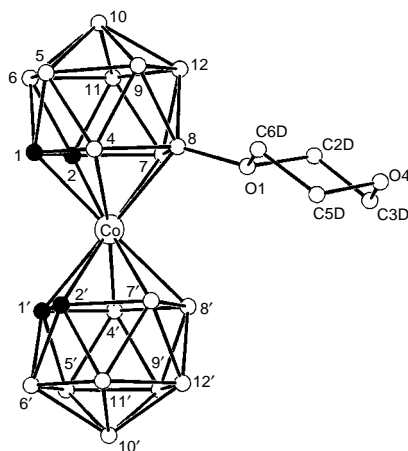


FIG. 3  
Structure of 4

## EXPERIMENTAL

### Apparatus and Chemicals

TLC was carried out on Silufol sheets (Kavalier, Votice, Czech Republic). The FAB mass spectra were recorded using a ZAB-EQ Mass Spectrometer, (VG Analytical); conventional mass spectra were taken on a Finnigan MAT MAGNUM ion trap quadrupole mass spectrometer. The NMR spectra were measured in deuteroacetone using a Varian UNITY 500 spectrometer at 160.36 MHz for  $^{11}\text{B}$  ( $\text{BF}_3\text{O} \cdot \text{Et}_2\text{O}$ ) and at 500 MHz for  $^1\text{H}$  (TMS). X-Ray diffraction was carried out on a CAD4, Enraf-Nonius device.

The bis(1,2-dicarbolliidocobaltate) cesium salt was prepared according to the literature<sup>1</sup>. Other chemicals and solvents were analytical grade from current commercial sources and were used as purchased.

### Synthesis and Isolation of **2**

A mixture of dimethyl sulfate (10 ml),  $\text{AlCl}_3$  (0.27 g, 2 mmol) and cesium salt of **1** (2.3 g, 5 mmol) was stirred for 24 h at 80 °C. The excess of dimethyl sulfate was destroyed by slow addition of a mixture of 15%  $\text{NH}_4\text{OH}$  (30 ml) and water (20 ml). Subsequently, 15% hydrochloric acid (40 ml) was added and the product was extracted as conjugate acid with diethyl ether (40 ml). After separation of the organic layer, the solvent was evaporated *in vacuo*, the residue was dissolved in dilute ethanol and tetramethylammonium chloride (1.5 g) was added. The obtained salt was crystallized from hot aqueous ethanol, yielding the tetramethylammonium salt of **2** as an orange powder (1.0 g; 40%). The last operation was repeated and the yellow needle-like crystals obtained were used for the X-ray diffraction analysis.

### Synthesis and Isolation of **3**

Dimethyl sulfate (5 ml), concentrated sulfuric acid (0.2 ml), benzene (40 ml) and cesium salt of **1** (4.6 g, 10 mmol) were stirred for 8 h at 80 °C. The reaction mixture was then decomposed by adding a solution of  $\text{NaOH}$  (1.4 g in 50 ml  $\text{H}_2\text{O}$ ) and of  $\text{Na}_2\text{SO}_3$  (13 g, 100 mmol). Stirring was continued for 24 h at ambient temperature. The resulting mixture was extracted with diethyl ether and the colorless aqueous layer was discarded. The ethereal layer was taken down *in vacuo*, the residue was dissolved in aqueous ethanol,  $\text{CsCl}$  (1.0 g) was added and the mixture was evaporated to dryness. The solid residue was dissolved in a mixture  $\text{CHCl}_3\text{--CH}_3\text{CN}$  (1 : 3) and poured on a silica gel column where it separated into two bands. The separated eluates were dried and extracted with acetone. The individual acetone solutions were evaporated *in vacuo*, the oily residues dissolved in aqueous ethanol,  $\text{Me}_4\text{NCl}$  (1.0 g) was added to each of them and the tetramethylammonium salts were crystallized from aqueous ethanol. The faster band afforded an orange powder (1.20 g, 25%), the slower one gave a mixture of tetramethylammonium salts of the starting anion **1**, of the  $\text{O--SO}_2\text{--O}$  bridged anion **2** and of an unidentified anion (according to NMR spectroscopy).

Crystals of tetramethylammonium salt of **3** for X-ray diffraction were obtained by slow evaporation of its solution in acetone–ethanol–water (1 : 2 : 2).

### Synthesis and Isolation of **4**

A mixture of dioxane (30 ml), cesium salt of **1** (4.6 g, 10 mmol), DMS (2 ml, 2.8 g, 20 mmol) and sulfuric acid (0.5 ml, 0.9 g, 9 mmol) was heated at 80 °C for 6 h with occasional shaking. After cooling to 20 °C, the reaction mixture was filtered and the filtrate was evaporated *in vacuo* at 35 °C. The colorless solid on the filter, containing the crystalline product **4**, was rinsed with water to



remove the precipitated  $\text{CsHSO}_4$  and the water insoluble residue was dissolved in benzene (20 ml). The benzene solution was added to the oily residue after evaporation of the original filtrate, more benzene (30 ml) was added, and the benzene layer was washed with water ( $3 \times 20$  ml). The turbid benzene layer was filtered and applied onto a dry silica gel column ( $3 \times 30$  cm) and the first yellow band eluted with benzene. The solvent was removed in vacuo and the crude residue dissolved in methylene chloride. The solution was layered carefully with a threefold volume of hexane and left to crystallize for one week to isolate orange needle dendrites of pure compound **4** (yield 1.85 g, 45%). Its  $R_f$  value, mass and NMR spectra are given in Tables I and II. Crystals for X-ray diffraction analysis were grown from a solution in a mixture  $\text{CH}_2\text{Cl}_2\text{-Et}_2\text{O}$  (1 : 1, v/v) layered with a twofold volume of hexane upon standing for a week.

#### X-Ray Analyses of Compounds **2**, **3** and **4**

*Crystal data of 2:*  $\text{C}_8\text{H}_{28}\text{B}_{18}\text{CoNO}_4\text{S}$ ,  $M_R = 487.9$ , monoclinic, space group  $P2_1/c$  (No. 14),  $a = 19.004(5)$ ,  $b = 12.674(3)$ ,  $c = 19.806(4)$  (based on the least squares refinement of 25 precisely centered reflections within the 16–200 range),  $\beta = 97.48(2)^\circ$ ,  $V = 4\,730.0(21)\text{ \AA}^3$ ,  $Z = 8$ ,  $D_c = 1.370\text{ g cm}^{-3}$ ,  $\mu = 0.832\text{ cm}^{-1}$ ,  $F(000) = 1\,984$ .

Measurement: A crystal  $0.3 \times 0.35 \times 0.4$  mm in size was measured on a CAD4 diffractometer (MoK $\alpha$  radiation,  $\lambda = 0.71969\text{ \AA}$ ). From a total of 4 611 reflections up to  $2\theta = 40^\circ$  within the  $h, k, l$  range of 0, 18; 0, 12; and  $-19, 18$ , respectively, 2 090 were regarded as "observed" according to the  $I > 2\sigma(I)$  criterion. Three standard reflections, monitored every 90 min, showed no significant fluctuations ( $<1.5\%$ ).

Structure solution and refinement: direct methods<sup>16</sup> (SHELXS86), full-matrix least squares refinement<sup>17</sup> (SHELXL90), anisotropic refinement of all non-hydrogen atoms, hydrogen atoms fixed in calculated positions. Function minimized:  $w(F_o^2 - F_c^2)^2$  with  $w = 4F^2/[(F_o^2)]^2$ , final  $R = 0.0664$ ,  $wR = 0.068$ ,  $R_{\text{int}} = 0.06$ ,  $S = 1.036$ ,  $[(\Delta/\sigma)_{\text{max}}] = 0.3$ . The final difference electron density map was featureless, with extremal values of 0.493;  $-0.375\text{ e \AA}^{-3}$  near the Co atom. Program ORTEP (ref.<sup>18</sup>) was used to draw the molecules.

*Crystal data of 3:*  $\text{C}_{14}\text{H}_{38}\text{B}_{18}\text{CoN}$ ,  $M_R = 473.96$ , monoclinic, space group  $P2_1/n$  (No. 14),  $a = 12.5997(7)$ ,  $b = 10.3540(10)$ ,  $c = 20.361(2)\text{ \AA}$  (based on the least squares refinement of 25 precisely centered reflections within the 16–200 range),  $\beta = 99.762(6)^\circ$ ,  $V = 2617.8(4)\text{ \AA}^3$ ,  $Z = 4$ ,  $D_c = 1.203\text{ g cm}^{-3}$ ,  $\mu = 0.662\text{ cm}^{-1}$ ,  $F(000) = 984$ .

Measurement: A crystal  $0.2 \times 0.31 \times 0.2$  mm in size was measured on a CAD4 diffractometer (MoK $\alpha$  radiation,  $\lambda = 0.71969\text{ \AA}$ ). From a total of 4 734 reflections up to  $2\theta = 50^\circ$  within the  $h, k, l$  range of  $-14, 14$ ; 0, 12 and 0, 24, respectively, 2 745 were regarded as "observed" according to the  $I > 2\sigma(I)$  criterion. Three standard reflections, monitored every 90 min, showed no significant fluctuations ( $<1.5\%$ ).  $R = 0.0429$ ,  $wR = 0.045$ ,  $R_{\text{int}} = 0.039$ ,  $S = 1.035$ ,  $[(\Delta/\sigma)_{\text{max}}] = 0.4$ . The final difference electron density map was featureless, with extremal values of 0.564;  $-0.320\text{ e \AA}^{-3}$  near the Co atom.

*Crystal data of 4:*  $\text{C}_8\text{H}_{25}\text{B}_{18}\text{CoO}_2$ ,  $M_R = 406.79$ , monoclinic, space group  $P2_1/n$  (No. 14),  $a = 13.7827(12)$ ,  $b = 10.3102(6)$ ,  $c = 14.707(2)\text{ \AA}$  (based on the least squares refinement of 25 precisely centered reflections within the 14–200 range),  $\beta = 93.904(8)^\circ$ ,  $V = 2\,085.1(3)\text{ \AA}^3$ ,  $Z = 4$ ,  $D_c = 1.296\text{ g cm}^{-3}$ ,  $\mu = 0.825\text{ cm}^{-1}$ ,  $F(000) = 824$ .

Measurement: A crystal  $0.4 \times 0.4 \times 0.3$  mm in size was measured on a CAD4 diffractometer (MoK $\alpha$  radiation,  $\lambda = 0.71969\text{ \AA}$ ). From a total of 3 662 reflections up to  $2\theta = 50^\circ$  within the  $h, k, l$  range of  $-16, 16$ ; 0, 12 and 0, 17, respectively, 2 757 were regarded as "observed" according to the  $I > 2\sigma(I)$  criterion. Three standard reflections, monitored every 90 min, showed no significant fluctuations ( $<1.5\%$ ).  $R = 0.0414$ ,  $wR = 0.047$ ,  $R_{\text{int}} = 0.064$ ,  $S = 1.065$ ,  $[(\Delta/\sigma)_{\text{max}}] = 0.2$ . The final difference electron density map was featureless, with extremal values of 0.672;  $-0.248\text{ e \AA}^{-3}$  near the Co atom.

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