

CONSTITUTION AND PROPERTIES OF THE  $8,8'\text{-}\mu\text{-H}_2\text{N}\text{>}\text{O}$   $(1,2\text{-C}_2\text{B}_9\text{H}_{10})_2\text{-3-Co}$   
BRIDGED COBALT BORANE

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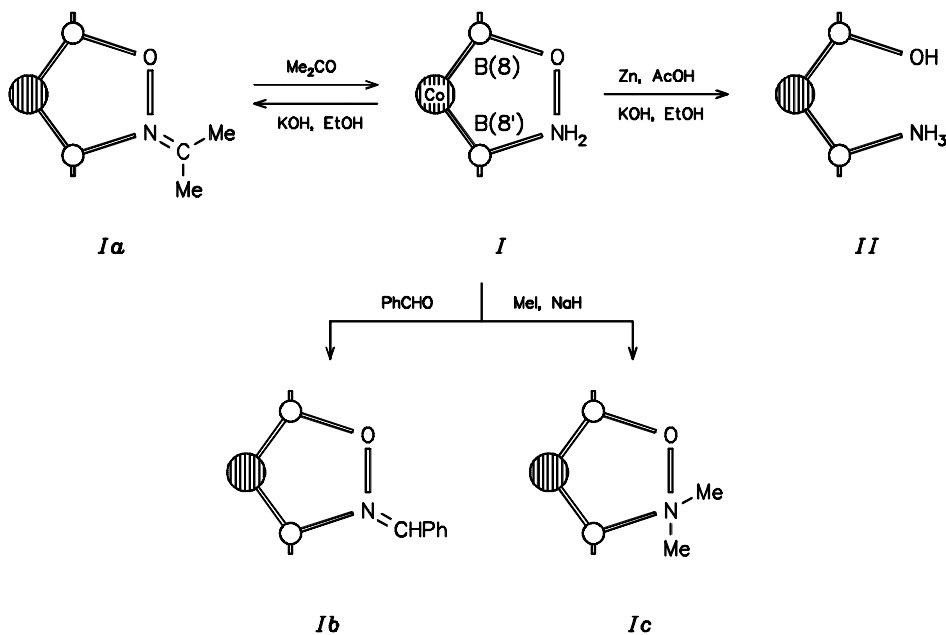
Constitution of  $8,8'\text{-}\mu\text{-H}_2\text{N}\text{>}\text{O}$   $(1,2\text{-C}_2\text{B}_9\text{H}_{10})_2\text{-3-Co}$  sandwich has been elucidated 17 years after its first synthesis, via high resolution NMR methods supplemented by mass spectrometry. The species gives relevant "oximes" with acetone and benzaldehyde and is quantitatively methylated to the  $N,N'$ -dimethyl derivative, which shows an interesting redox disproportionation to a triatomically bridged sandwich with a  $8,8'\text{-}\mu\text{-O-CH=N(Me)-}$  bridge between both ligands. Several other "peculiar" reactions of this dimethyl derivative are discussed. On reductive cleavage of the parent hydroxylamine the non-bridged  $8\text{-HO-8-H}_3\text{N}(1,2\text{-C}_3\text{B}_3\text{H}_{10})_2\text{-3-Co}$  zwitterion is obtained. Mono-, di- and trimethylated derivatives of this non-bridged species are characterized.

Reaction of the  $3\text{-Co-(1,2-C}_2\text{B}_9\text{H}_{11})_2^-$  ion<sup>1</sup> with nitrosylsulfuric acid is a source of several interesting cobaltaboranes with intramolecular bridges between both ligands<sup>2,3</sup>. So far the structure of the red  $8,8'\text{-}\mu\text{-H}_2\text{N}(1,2\text{-C}_2\text{B}_9\text{H}_{10})_2\text{-3-Co}$  zwitterion<sup>2,4</sup> and of its yellow  $8,4'\text{-}\mu\text{-H}_2\text{N}(1,2\text{-C}_2\text{B}_9\text{H}_{10})_2\text{-3-Co}$  congener<sup>3</sup> has been elucidated. But the highly polar orange main product eluded an unequivocal characterization up to now. In the first paper<sup>2</sup> we suggested, that it might be  $8,8'\text{-}\mu\text{-H}_2\text{N}\text{>}\text{O}$   $(1,2\text{-C}_2\text{B}_9\text{H}_{10})_2\text{-3-Co}$  (*I*) (Fig. 1), whereas in the last paper we proposed rather a non-bridged "dihydro" structure  $8\text{-H}_3\text{N-8}'\text{-HO}(1,2\text{-C}_2\text{B}_9\text{H}_{10})_2\text{-3-Co}$  (*II*) (Scheme 1). The difficulty with an unequivocal determination of the true constitution was first due to poor resolution of NMR devices of that time and second, due to amazing reactivity of the complex, sometimes leading to quite unique results.

In this paper we argue that the compound is indeed the hydroxylamine-bridged type *I* (Fig. 1), as proposed in our first paper<sup>2</sup>. For clarity we shall first discuss the "normal" properties and chemistry of our species, commenting upon its "peculiar" behavior later on.

The orange main product on treatment of the parent sandwich anion with nitrosylsulfuric acid shows an  $R_F$  value 0.12 (silica gel, benzene) and can be isolated in about 34% yield. The compound does not melt up to 300 °C and gives no mass spectrum due to its

low volatility. We explain this behavior as a consequence of strong intermolecular hydrogen bonds between the apparently acid amino-hydrogens and apparently basic  $-O-$  part of the bridge. So in solid state the species might be essentially polymeric, but it shows a normal NMR behavior, giving well resolved  $^1\text{H}$  and  $^{11}\text{B}$  NMR spectra compatible with structure *I*.



SCHEME 1

The “hydrogen bond” problem is absent in compounds *Ia* – *Ic* (Scheme 1). All these species show uncomparably higher  $R_F$  values on TLC due to the absence of acidic hydrogen (Table I) and both the unconventional “oximes” *Ia* and *Ib* show the expected  $m/z$  values in their mass spectra. Reaction of *I* with acetone (*Ia*) and benzaldehyde (*Ib*) is instantaneous if catalyzed by triethylamine. On the other hand, treatment of *Ia* with 10% KOH in ethanol regenerates the parent “hydroxylamine” *I* within a few seconds.  $^1\text{H}$  and  $^{11}\text{B}$  NMR spectra of *Ia* – *Ic* (Table II, Fig. 2) leave no doubt on relatedness of all of them to parent compound *I*.

Assignment of essentially all C–H,  $^{11}\text{B}$  and B– $^1\text{H}$  signals is based on the respective  $^{11}\text{B}$ - $^{11}\text{B}$  COSY 2D NMR (not shown here, available on request) and on  $^1\text{H}$  NMR selectively decoupled by  $^{11}\text{B}$ . On the NMR time scale the species *I* and *Ia* – *Ic* show one plane of symmetry but two different deltahedral ligands. Consequently we see two C–H carborane and two  $^{11}\text{B}$  singlets in all relevant spectra. If the diatomic bridge is attached

to B(8,8') as bridgeheads, we shall see two sets of patterns 1,1,2,2,2,1 one corresponding to the B–O nucleus, the other to the B–N nucleus, as is indeed the case.

The monoatomically bridged analogs 8,8'- $\mu$ -MeO and 8,8'- $\mu$ -Me<sub>2</sub>N, respectively, indicate that the span of the <sup>11</sup>B NMR spectra is by approximately 10 ppm larger in the former case<sup>2</sup>. We expect the same influence on chemical shifts also in our diatomically bridged species, and so we attribute the lowest field singlets and highest field doublets

TABLE I

Some characteristics of *N*-substituted derivatives of *I*, *II* and *III*

Compound	Substituent at nitrogen	Mass spectrum <i>m/z</i>	TLC, <i>R<sub>F</sub></i>	
			benzene	benzene–hexane <sup>a</sup>
<i>I</i>	none	–	0.12	–
<i>Ia</i>	(CH <sub>3</sub> ) <sub>2</sub> C	397	0.57	0.29
<i>Ib</i>	C <sub>6</sub> H <sub>5</sub> CH	445	0.58	0.334
<i>Ic</i>	(CH <sub>3</sub> ) <sub>2</sub>	383, 387 <sup>b</sup>	0.54	0.24
<i>II</i>	none	359	0.02	–
<i>IIa</i>	CH <sub>3</sub>	373	0.06	–
<i>IIb</i>	(CH <sub>3</sub> ) <sub>2</sub>	387	0.16	–
<i>IIc</i>	CH <sub>3</sub> O	401	0.52	0.20
<i>III</i>	OCH=NCH <sub>3</sub>	383	0.58	0.16

<sup>a</sup> 1 : 1. <sup>b</sup> Due to thermal disproportionation to *IIb* and *III*.

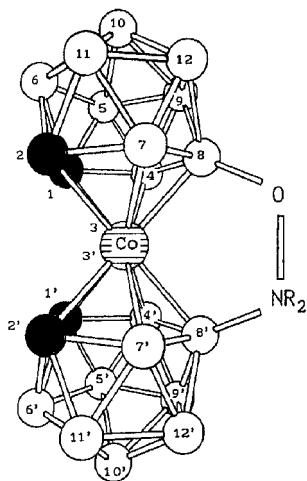


FIG. 1  
Structure and numbering of compounds with *I* framework (*I*, *Ia* – *Ic*)

in *I* and *Ia* – *Ic* to the B–O nucleus. The next higher field singlet and the next lower field doublet should then belong to the B–N nucleus, and indeed, in some 2D spectra we observe the confirming contacts. Reading upfield, the  $^{11}\text{B}$  NMR signals can be assigned to position 8, 10, 9(12), 4(7), 5(11), and 6 (antipodal to the bridgehead) in both deltahedral ligands (Table II, Fig. 2).

Easy and quantitative reductive cleavage of *I* (Scheme 1) affords the non bridged 8-HO-8'-H<sub>3</sub>N-(1,2-C<sub>2</sub>B<sub>9</sub>H<sub>10</sub>)<sub>2</sub>-3-Co zwitterion *II*. Its NMR spectra (Table III and Fig. 3) are compatible with constitution *II* and are related to those of *I* and *Ia* – *Ic*.

TABLE II  
NMR spectra of bridged complexes *I* and *Ia* – *Ic* in benzene –  $^1\text{H}$  at 500 MHz and  $^{11}\text{B}$  at 160.36 MHz. Assignment of  $^{11}\text{B}$  NMR signals is based on  $^{11}\text{B}$ - $^{11}\text{B}$  COSY 2D NMR;  $^1\text{H}(\text{B})$  signals are assigned according to selectively  $^{11}\text{B}$  decoupled  $^1\text{H}$  NMR. All values are given in ppm against conventional standards

Position	<i>I</i> <sup>a</sup>		<i>Ia</i> <sup>b</sup>		<i>Ib</i> <sup>c</sup>		<i>Ic</i> <sup>d</sup>	
	$^{11}\text{B}$	$^1\text{H}$	$^{11}\text{B}$	$^1\text{H}$	$^{11}\text{B}$	$^1\text{H}$	$^{11}\text{B}$	$^1\text{H}$
O–B ligand								
8	31.156	–	27.407	–	28.130	–	28.240	–
10	–2.815	3.611	–2.434	3.628	–2.434	3.682	–2.834	3.534
9, 12	–5.784	3.015	–5.841	3.074	–5.917	3.204	–5.441	2.986
4, 7	–9.761	3.372	–8.848	3.858	–8.810	3.972	–9.324	3.639
5, 11	–15.357	2.398	–15.242	2.448	–15.014	2.486	–14.557	<sup>e</sup>
6	–30.030	1.692	–29.421	1.740	–29.440	1.768	–29.420	1.693
Span	61.19		56.83		57.57		57.66	
N–B ligand								
8	20.099	–	20.251	–	21.545	–	25.880	–
10	–3.633	3.463	–1.910	3.626	–3.995	3.606	–2.976	3.436
9, 12	–8.372	2.254	–8.163	2.671	–7.440	2.764	–8.011	2.336
4, 7	–9.761	3.562	–8.163	3.830	–8.810	3.878	–9.819	3.670
5, 11	–16.251	2.231	–16.917	2.278	–15.775	2.408	–16.460	2.240
6	–27.594	1.706	–26.566	1.807	–27.365	1.807	–28.030	1.687
Span	47.19		46.82		48.91		53.91	

<sup>a</sup> 2.466 s and 2.101 s (C–H carborane); bs (NH<sub>2</sub>). <sup>b</sup> 2.442 s and 2.255 s (C–H carborane); 1.391 (CH<sub>3</sub>); 1.091 (NH<sub>2</sub>). <sup>c</sup> 2.550 s and 2.267 s (C–H carborane); phenyl 7.047 s, 6.788 and 6.00 tripl. <sup>d</sup> 2.433 s and 2.148 s (C–H carborane); 2.461 s ((CH<sub>3</sub>)<sub>2</sub>N). <sup>e</sup> Not found; probably hidden within the (CH<sub>3</sub>)<sub>2</sub>N signal at 2.461.

It is noteworthy (and supports our explanation of non-volatility of the bridged species *I*) that compound *II* shows a normal mass spectrum (Table I) although it is by far more polar (note the  $R_F$  values in Table I) than its cyclic "dehydro" parent *I*.

The same non-bridged structure of the *II* type possess some methylated species shown in Scheme 2. Methylation of *II* with dimethylsulfate in ethanolic solution of KOH proceeds very slowly. After 24 h at ambient temperature *IIc* is formed as main product, but presence of the starting species and of the partially methylated intermediates *IIa*, *IIb* is undeniable according to TLC comparison (the  $R_F$  values of the respective species are distinctly different as shown in Table I).

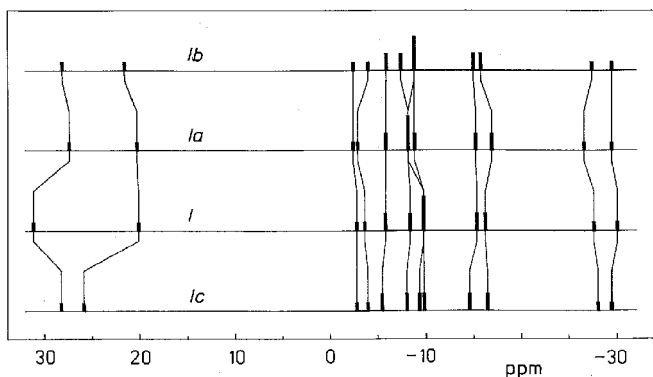


FIG. 2

$^{11}\text{B}$  correlation diagram for compounds *I*, *Ia* - *Ic*

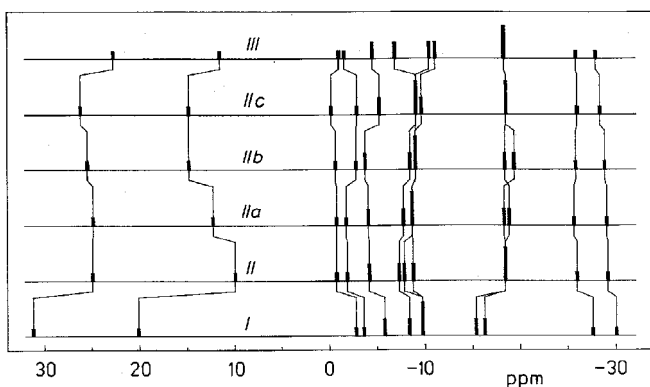
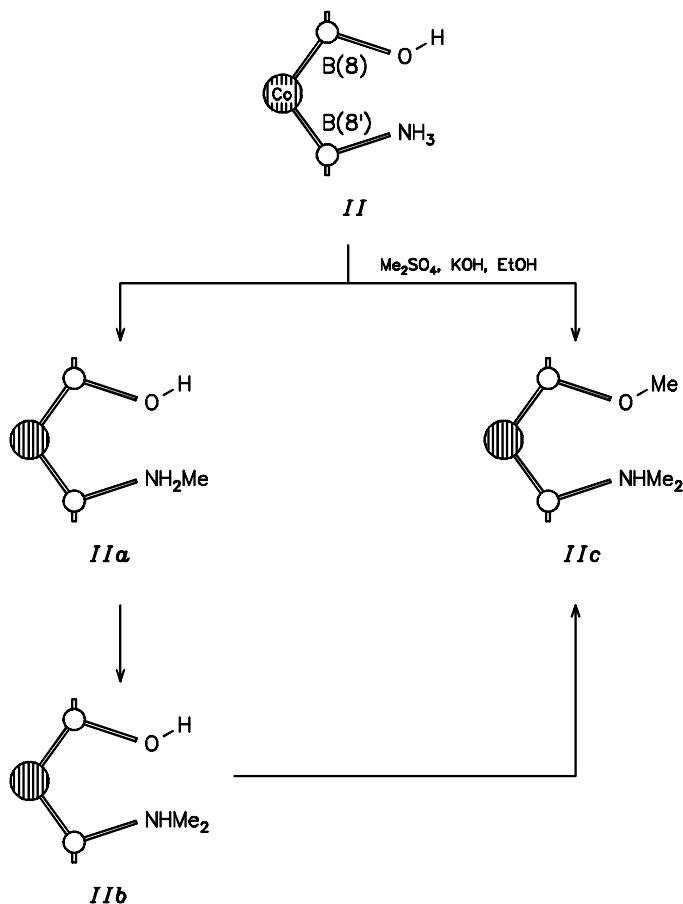


FIG. 3

$^{11}\text{B}$  correlation diagram for compounds *I* - *III*



SCHEME 2

The  $^1\text{H}$  and  $^{11}\text{B}$  NMR spectra (Table III, Fig. 3) support the constitution of *II* and *IIa* – *IIc*, leaving no doubt on their relatedness to *I* and *Ia* – *Ic*.

As shown in Fig. 3, increase in size of the bridging group as in *III*, or cleavage of the bridge as in *II*, *IIa* – *IIc* causes a systematic upfield shift of both B(8) and B(8') vertices. However, this effect is much more pronounced with nitrogen bearing B(8') vertex.

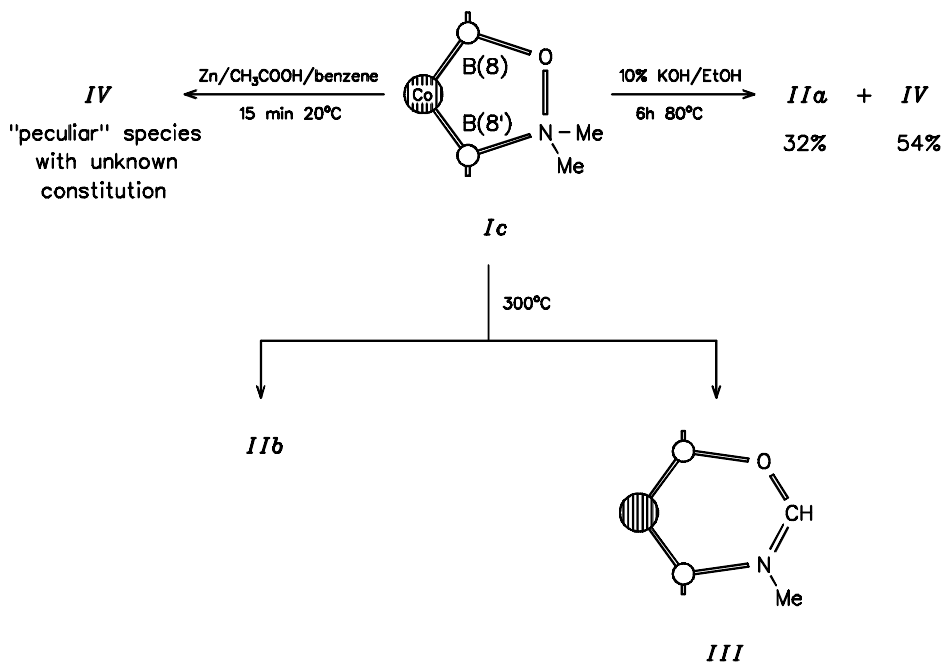
For preparation of compounds *IIa*, *IIb* the "peculiar" reactions of *Ic* are more suitable than methylation of *II*, as shown in Scheme 3.

Thermal redox type disproportionation shown in the bottom half of Scheme 3 is responsible for "anomalous" mass spectrum of *Ic*.

Instead of the expected value  $m/z$  385 two peaks with  $m/z$  383 and 387, respectively, are observed, corresponding to calculated values for *III* and *IIb*. This disproportionation

can be carried out preparatively and both species *IIb* and *III* are easily isolated pure via column chromatography. Their properties are gathered in Tables I, III, IV and in Fig. 3.

The top part of Scheme 3 describes a true peculiarity. Both reactions are very fast. Ethanolysis in alkaline medium leads to the “peculiar” main product *IV* along with *IIa*. Both species were separated by column chromatography. The “peculiar” species *IV* is the single product obtained on treatment of *Ic* with Zn dust and acetic acid.



SCHEME 3

There is no problem with isolation of *IV* in chromatographically pure state, but its constitution remains obscure. It is uncharged orange crystalline substance with  $m/z$  399. However, its  $^1\text{H}$  and  $^{11}\text{B}$  NMR spectra indicate total asymmetry of the molecule and are too complex for unequivocal assignment. There is no hope to reveal its structure without X-ray diffraction on a single crystal – but we were not able to grow any suitable one so far. We encountered the same problem with all species of *Ia* – *Ic* and *IIa* – *IIc* families.

The parent hydroxylamine-bridged compound *I* might be useful as a new reagent for carbonyl compounds, and eventually as a component of new polymers, resulting e.g. on polyalkylation with terminal polymethylene dihalides.

## EXPERIMENTAL

The mass spectra were measured on the Finigan MAT "Magnum" ion trap quadrupole mass spectrometer via the "heated inlet" option as developed by Spectronex AG, Basel, Switzerland. The NMR spectra ( $\delta$ , ppm) were measured with Varian Unity-500 spectrometer at 500 MHz ( $^1\text{H}$ ) and 160.4 MHz ( $^{11}\text{B}$ ) in deuteriobenzene with inserted capillary containing  $\text{B}(\text{OCH}_3)_3$  as internal standard (18.2 ppm). TLC were performed on silica gel sheets Silufol (Kavalier, Votice, The Czech Republic). The parent  $8,8'\text{-}\mu\text{-}^{\text{H}_2\text{N}}\text{O}(1,2\text{-C}_2\text{B}_9\text{H}_{10})_2\text{-3-Co}$  (*I*) has been obtained in 35% yield following the standard procedure<sup>2</sup>. Auxiliary solvents were of reagent grade and were used without further purification.

General properties of *I* were discussed in the introductory paragraphs and its NMR properties are gathered in Table II and Figs 2 and 3.

TABLE III

NMR spectra of the non-bridged complexes *II* and *IIa* – *IIc* in benzene –  $^1\text{H}$  at 500 MHz and  $^{11}\text{B}$  at 160.36 MHz. All values are given in ppm against conventional standards

Position	<i>II</i> <sup>a</sup>		<i>IIa</i> <sup>b</sup>		<i>IIb</i> <sup>c</sup>		<i>IIc</i> <sup>d</sup>	
	$^{11}\text{B}$	$^1\text{H}$	$^{11}\text{B}$	$^1\text{H}$	$^{11}\text{B}$	$^1\text{H}$	$^{11}\text{B}$	$^1\text{H}$
	O–B ligand							
8	24.870	–	24.819	–	25.428	–	26.170	–
10	–0.779	3.661	–0.703	3.666	–0.645	3.664	–0.208	3.699
9, 12	–4.185	2.892	–4.090	2.899	–3.729	2.926	–5.213	3.027
4, 7	–8.772	2.956	–8.658	2.257	–9.000	2.342	–9.609	3.150
5, 11	–18.402	2.270	–18.268	2.243	–19.326	2.144	–18.459	2.219
6	–29.154	1.648	–29.021	1.642	–28.793	1.609	–28.279	1.661
Span	54.02	–	53.84	–	54.22	–	54.45	–
	N–B ligand							
8	9.936	–	12.258	–	14.789	–	14.827	–
10	–1.882	3.567	–1.768	3.579	–2.796	3.512	–2.891	3.491
9, 12	–7.287	3.044	–7.744	3.012	–8.410	3.069	–9.038	3.142
4, 7	–7.820	2.270	–8.658	3.046	–9.000	3.116	–9.038	2.319
5, 11	–18.402	2.235	–18.801	2.109	–18.326	2.224	–18.459	2.137
6	–25.976	1.706	–25.634	1.711	–25.843	1.686	–25.957	1.677
Span	35.91	–	37.89	–	40.63	–	40.78	–

<sup>a</sup> 2.385 s and 2.751 s (C–H carborane); 4.615 bs ( $\text{NH}_3$ ). <sup>b</sup> 2.667 s and 2.357 s (C–H carborane); 1.436 ( $\text{CH}_3$ ), 5.532 bs ( $\text{NH}_2$  ?); 2.131 s (OH ?). <sup>c</sup> 2.602 s and 2.304 s (C–H carborane); 1.785 s ( $(\text{CH}_3)_2$ ); 1.901 bs (NH ?). <sup>d</sup> 2.651 s and 2.284 s (C–H carborane); 1.758 s ( $(\text{CH}_3)_2\text{N}$ ); 3.369 s ( $\text{CH}_3\text{O}$ ).



"Oximes" *Ia, Ib*

To a dark brown solution of *I* (1.77 g, 5 mmol) in ethanol (20 ml), acetone (3 ml) in case of *Ia* or benzaldehyde (1 ml) in case of *Ib* was added, followed by triethylamine (0.3 ml). The initial dark brown color turned light orange within 5 min and TLC monitoring showed only the spots of product *Ia* or *Ib*. Volatile solvents were stripped off in vacuum, residue was triturated with hexane ( $2 \times 10$  ml). The insoluble solids were dissolved in benzene and chromatographed with the same solvents on a silica gel column. Orange eluates were collected, concentrated to about 20 ml and this solution was carefully covered with hexane (60 ml). Over weekend orange needles separated in both cases; 1.73 g (88%) of *Ia* and 1.85 g (84%) of *Ib* were obtained. For properties inspect Tables I, II and Fig. 2.

*N,N*-Dimethyl Derivative *Ic*

To a solution of *I* (1.77 g, 5 mmol) in THF (20 ml), NaH (0.3 g, 12 mmol) was added and after vigorous evolution of hydrogen ceased methyl iodide (1 ml) was added. The initial dark brown color changed to deep orange within 5 min; TLC showed only the presence of *Ic*. Solids were sucked off and destroyed by ethanol, the filtrate was stripped off in vacuo and the residue was worked up as above with species *Ia, Ib*. Orange needles, 1.75 g (92%). Properties are recorded in Tables I, II and Fig. 2.

Ring Cleavage of *I* to *II*

To a stirred solution of *I* (1.77 g, 5 mmol) in benzene (60 ml) zinc dust (2.0 g) was added followed by acetic acid (2.0 ml). After 1 h at ambient temperature and 5 min at 80 °C the reduction was complete, according to TLC. Excess of Zn dust was dissolved by addition of 10% hydrochloric acid (50 ml). From the upper layer benzene was stripped off in vacuum leaving a yellow solid; this was triturated with hexane ( $2 \times 10$  ml) and the solid residue was dried at 80 °C at 150 Pa to constant weight; 1.70 g (95%) of TLC pure *II* was obtained. Its properties are described in Tables I, III and Fig. 3.

TABLE IV

NMR spectra of the  $-\text{O}-\text{CH}=\text{N}(\text{Me})-$  bridged species *IIIa* ( $^1\text{H}$  at 500 MHz,  $^{11}\text{B}$  at 160.38 MHz, in benzene). All values are given in ppm against conventional standards

$^{11}\text{B}$ Position	O-B ligand		N-B ligand	
	$^{11}\text{B}$	$^1\text{H}$	$^{11}\text{B}$	$^1\text{H}$
8	22.687	–	11.516	–
10	–1.007	3.739	–1.597	3.699
9, 12	–4.490	3.139	–6.850	2.534
4, 7	–11.056	2.955	–10.428	2.851
5, 11	–18.249	2.184	–18.249	2.184
6	–27.860	1.683	–25.824	1.762
Span	50.55	–	37.34	–

$^1\text{H}$  signals: 2.127 ( $\text{CH}_3$ ); 2.468 and 2.416 (C–H carborane); 4.263 (C–H bridge).

### Methylation of *II* to *IIa* – *IIc*

To a stirred solution of *II* (1.06 g, 3 mmol) and KOH (5.0 g) in ethanol (50 ml) six portions of dimethylsulfate (à 0.5 ml) were added in 1 h intervals and the mixture was stirred overnight at ambient temperature. TLC showed the presence of still not consumed *II* and of methylated products *IIa* – *IIc*. The mixture was diluted with water (50 ml), acidified with concentrated hydrochloric acid (5 ml) and ethanol was stripped off in vacuum. The resulting slurry was extracted with benzene (30 ml), the combined extracts were concentrated until first solids began to separate, then it was soaked on a dry column of silica gel and the components were separated with benzene as eluent (compare the respective  $R_F$  values in Table I). All species were isolated by evaporation of benzene in vacuum and crystallization of the residues as described above. The yields were: 0.11 g (10%) of *IIa*, 0.19 g (17%) of *IIb* and 0.68 g (57%) of *IIc*; about 0.05 g of the nonreacted *II* was recovered from the last orange eluate.

Alternative routes to *IIa*, *IIb* are described below in the paragraphs on “peculiar” solvolysis of *Ic* and on thermal disproportionation of *Ic*, respectively.

### Thermal Disproportionation of *Ic* to *IIb* and *III*

At 150 Pa *Ic* (0.2 g) was heated in a bath to 330 °C. First sublimate appeared at 250 °C and partial melting of the residue was observed at 320 °C. After 2 h at 330 °C, the experiment was interrupted. According to TLC the sublimate as well as the glassy residue contained both species *IIb* and *III*, the latter prevailing in the sublimate. Both fractions were dissolved in benzene (20 ml), some insoluble material was filtered off and the filtrate was chromatographed on a silica gel column with benzene as eluent. The products were isolated as described in case *Ia* – *Ic* and *IIa* – *IIc*. The yield of the non-bridged *N,N*-dimethyl derivative *IIb* was 0.06 g (30%) and of the compound *III* was 0.08 g (40%).

### Solvolysis of *Ic* to *IIa* and *IV*

In a stirred mixture of ethanol (30 ml) and benzene (6 ml) *Ic* (0.76 g, 2 mmol) was dissolved, KOH (1.0 g) was added and the mixture was heated to 60 °C until the spot of *Ic* disappeared on TLC. Then benzene (30 ml) and diethyl ether (10 ml) were added, followed by water (50 ml). The upper layer was washed with water (2 × 10 ml) and volatile solvents were evaporated from the upper layer in vacuum. The residual solid foam was dissolved in benzene (20 ml) and chromatographed on a silica gel column with benzene as eluent till two bands developed; these were separated mechanically and the products were eluted with diethyl ether. Slower yellow band afforded 0.24 g (33%) of *IIa*; from the faster orange band 0.41 g of the “peculiar” species *IV* with unknown constitution was obtained. It is noteworthy, that reduction of *Ic* with Zn dust (as described above with *I*) afforded only the “peculiar” compound *IV*.

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