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Polysubstituted derivatives of 3-(η^5-Cp)-4-SMe$_2$-3,1,2-FeC$_2$B$_9$H$_{10}$. X-ray structure of 3-(η^5-Cp)-4-SMe$_2$-7,8-(CF$_3$COO)$_2$-12-HgCl-3,1,2-FeC$_2$B$_9$H$_7$

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Abstract

The electrophilic mercuration of 3-(η^5-Cp)-4-SMe$_2$-3,1,2-FeC$_2$B$_9$H$_{10}$ (**1**) was investigated. The reaction of **1** with the excess of mercury trifluoroacetate in dichloromethane at 20 °C gives complexes containing from two to four substituents depending on the ratio of reagents. All new compounds were separated and characterized by elemental analysis and NMR spectra. The positions of substitution were established by ^{11}B-NMR and ^{11}B-^{11}B COSY NMR spectra. X-ray structure of 3-(η^5-Cp)-4-SMe$_2$-7,8-(CF$_3$COO)$_2$-12-HgCl-3,1,2-FeC$_2$B$_9$H$_7$ was determined.

Keywords: Ferracarboranes; Mercuration; X-ray diffraction

1. Introduction

Electrophilic mercuration of icosahedral metallacarboranes can proceed at 9, 8 and 12 position of the carborane cage depending on the nature of metallacarborane and on reaction conditions [1–5]. We have recently investigated electrophilic mercuration and bromination of ferracarborane 3-(η^5-Cp)-4-SMe$_2$-3,1,2-FeC$_2$B$_9$H$_{10}$ (**1**) [6]. The interaction of **1** with one equivalent of mercury trifluoroacetate under mild conditions was found to result in formation of monomercurated derivative 3-(η^5-Cp)-4-SMe$_2$-8-HgX-3,1,2-FeC$_2$B$_9$H$_9$ (**2**, X = CF$_3$COO; **3**, X = Cl) containing mercury atom at the position 8. In this paper the mercuration of **1** by the excess of mercury trifluoroacetate is described and polysubstituted products formed are studied. Earlier a few polymercurated derivatives of cobaltacarboranes were investigated [1,3].

2. Results and discussion

We found that the treatment of ferracarborane **1** with mercury trifluoroacetate at a 1:3 ratio in dichloromethane at 20 °C resulted in the mixture of compounds, from which four compounds were isolated by repeated chromatography on SiO$_2$. Trifluoroacetoxymercuro-derivatives formed were found to be unstable in solution and were converted into chlomercuro-derivatives under the treatment with NaCl in water–acetone mixture (Scheme 1).

The ^{11}B-NMR spectra of all these compounds contain three singlets that correspond to three substituents in the carborane cage, one of them is Me$_2$S-group. Therefore, the substitution takes place at two boron atoms at non-equivalent positions. The positions of substitution in all these compounds were established by comparison of ^{11}B-NMR spectra of the new compounds synthesized with spectra of compound **1** and monosubstituted derivative **3** [6] and from the COSY ^{11}B-^{11}B-NMR spectra of the new compounds. In the spectra of all compounds signal corresponding to the B(4) atom connected with SMe$_2$-group was observed in the range of −4– −7 ppm. The substitution at boron atom by the CF$_3$COO-group shifts its signal to low field for 15–20

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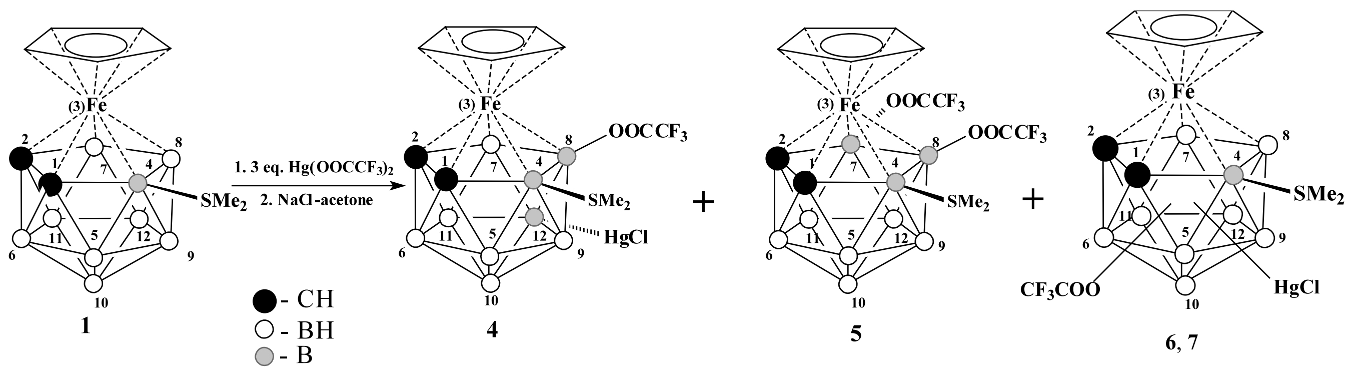


Scheme 1.

ppm in comparison with the signal of the unsubstituted boron atom. Introduction of the HgCl-substituent leads to the low field shift of the signal for 6–14 ppm.

The main product—3-(η^5-Cp)-4-SMe_2-8-(CF_3COO)-12-HgCl-3,1,2-$FeC_2B_9H_8$ (**4**) was obtained in 52% yield. The ^{19}F-NMR spectrum of the complex **4** contains one singlet at 2.46 ppm. Formation of trifluoroacetoxy-derivatives could be explained by oxidation of B–H or by mercuration followed by Hg elimination. Similar formation of trifluoroacetoxy derivatives was observed earlier under thallation of metallacarboranes [3,7].

The ^{11}B-NMR spectrum of the complex **4** contains three singlets at 14.8, −3.5 and −6.4 ppm. The signal at −3.5 ppm with B–Hg ($J_{B-Hg} = 2472$ Hz) coupling constant corresponds obviously to the mercurated boron atom, the singlet at −6.4 ppm corresponds to the B(4) atom connected with SMe_2-group, and the low field signal at 14.8 ppm corresponds to the B atom bonded to the CF_3COO-group. The analysis of the ^{11}B-^{11}B COSY NMR spectrum of **4** was based on the observation that the signal of B(4) correlates with a singlet at 14.8 ppm. This singlet has the cross-peaks to other four signals; one of those is a signal of the B atom connected with the Hg atom. Therefore, the B atom connected with the Hg atom has no connection to B(4) atom, but bonded with the B atom bearing the CF_3COO-group (Fig. 1).

Besides, a small amount of compound contained three substituents in the open face of the carborane cage: 3-(η^5-Cp)-4-SMe_2-7,8-$(CF_3COO)_2$-3,1,2-$FeC_2B_9H_8$ (**5**) as well as two regioisomers contained HgCl- and CF_3COO-groups were isolated. The position of substituents in the last two compounds was not established. In the ^{19}F-NMR spectrum of the compound **5**, two singlets of two trifluoroacetoxy groups were observed. The ^{19}F-NMR spectra of other two complexes 3-(η^5-Cp)-4-SMe_2-CF_3COO-HgCl-3,1,2-$FeC_2B_9H_8$ (**6**, **7**) consist of one singlet. The ^{11}B-NMR spectra of the complexes **6** and **7** do not allow to determine accurately the position of substituents. In compound **5**, as in the dibromo-derivative 3-(η^5-Cp)-4-SMe_2-7,8-Br_2-3,1,2-$FeC_2B_9H_8$, prepared earlier by bromination of **1** with the excess of bromine [6], both substituents are located in open face of the carborane cage in spite of possible steric hindrances.

The treatment of ferracarborane **1** with 12 equivalents of mercury trifluoroacetate resulted in the mixture of unstable compounds with low solubility. A separation of the mixture on SiO_2 allowed to isolate only one product—3-(η^5-Cp)-4-SMe_2-7,8-$(CF_3COO)_2$-12-$HgOOCCF_3$-3,1,2-$FeC_2B_9H_7$ (**8**). The ^{19}F-NMR spectrum of the complex **8** consists of three signals at 4.09, 2.57 and 2.46 ppm corresponding to CF_3COOHg- and two CF_3COO-groups, respectively. Position of substitution in the complex **8** was established from the data of the ^{11}B and ^{11}B-^{11}B COSY NMR spectra. The ^{11}B-NMR spectrum of **8** contains four singlets at 12.3, 6.1, −6.9 (B(4)) and −9.7 ppm ($J_{B-Hg} = 2668$ Hz). In the ^{11}B-^{11}B COSY NMR spectrum of the compound **8** signal of B(4) atom has a cross-peak to the singlet at 12.3 ppm (B–$OCOCF_3$), for which the cross-peak with a singlet at 6.1 ppm was observed. Besides, the later signal has a cross-peak to the signal of the B atom bonded with the Hg atom.

The chloromercuro-derivative 3-(η^5-Cp)-4-SMe_2-7,8-$(CF_3COO)_2$-12-HgCl-3,1,2-$FeC_2B_9H_7$ (**9**) was prepared by treatment of **8** with NaCl in a water–acetone mixture (Scheme 2).

The ^{19}F-NMR spectrum of the complex **9** consists of two singlets of trifluoroacetoxy groups. In the ^{11}B-NMR spectrum of **9**, four singlets at 12.5, 6.2, −5.6 ($J_{B-Hg} =$ 2188 Hz) and −6.7 ppm were observed. The signal of the B atom connected with the Hg atom is located in the lower field in comparison with the similar signal for **8**. The similar shift of the signal of the B atom connected with the Hg atom was observed for compounds **2** and **3** [6]. The ^{11}B-^{11}B COSY NMR spectrum of compound **9** is similar to the spectrum of **8**.

The structure of **9** was confirmed by X-ray study (Fig. 2).

After isolation of **8** the other fractions were also treated by NaCl in water–acetone mixture, and compounds **9** and **10** were isolated by repeated chromatography on SiO_2. Compound **10** was eluated by a CH_2Cl_2–acetone mixture. The position of substitution

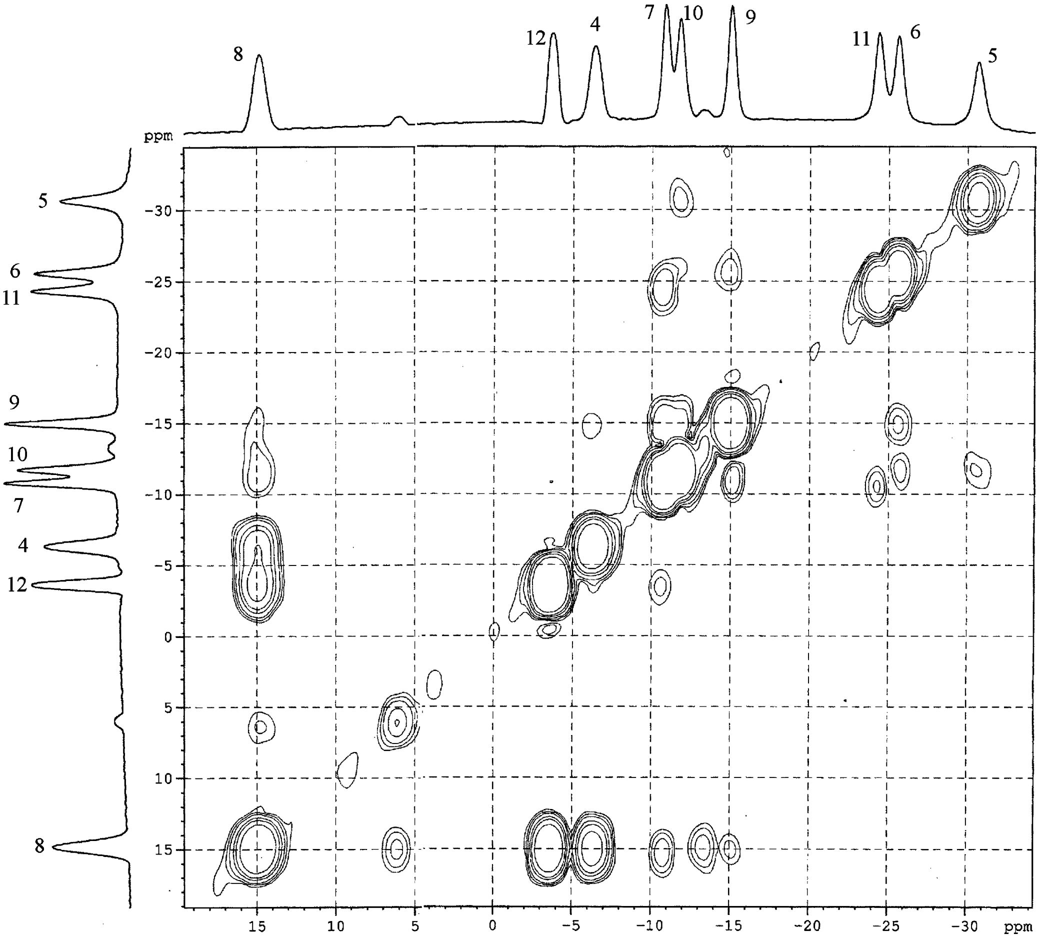
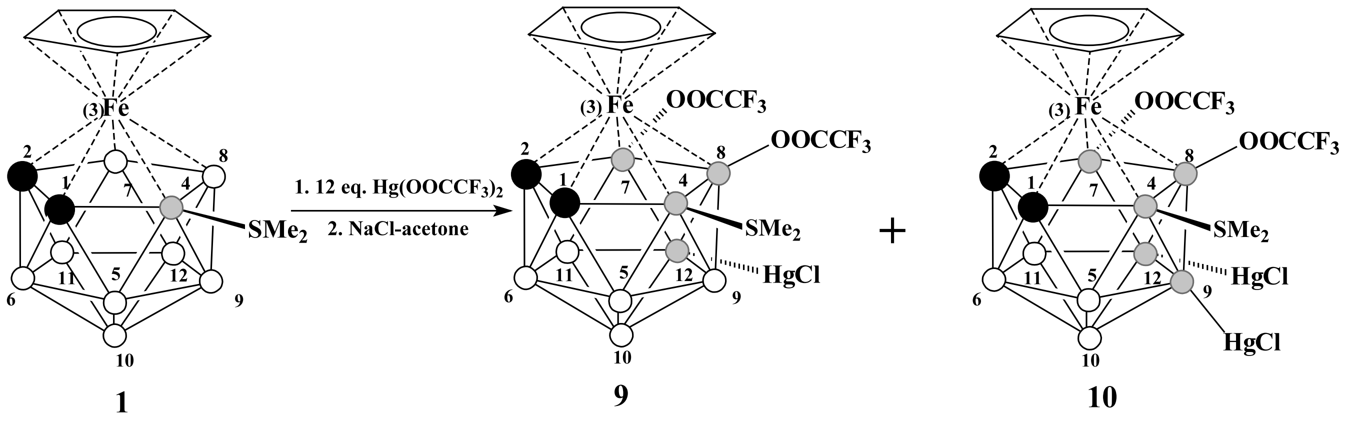


Fig. 1. ^{11}B-^{11}B COSY NMR spectrum of complex **4**.

was determined by ^{11}B-NMR spectrum, containing five singlets at 13.2, 6.7, −4.0, −6.2 and −8.0 ppm. Two B–Hg coupling constants ($J_{B-Hg} = 2576$ and 2650 Hz, respectively) were observed for signals at −4.0 and −8.0 ppm. The ^{19}F-NMR spectrum contains two singlets at 2.49 and 2.36 ppm of two C*F*$_3$COO-groups. According to ^{11}B- and ^{19}F-NMR data and elemental analysis, we suggest the structure 3-(η^5-Cp)-4-SMe$_2$-7,8-(CF$_3$COO)$_2$-9,12-(HgCl)$_2$-3,1,2-FeC$_2$B$_9$H$_6$ for compound **10**.

The composition and structures of the novel complexes (**4**–**10**) were also confirmed by elemental analysis and ^{1}H-NMR spectra. The ^{1}H-NMR spectra (in acetone-d_6) of all obtained complexes contain signal of protons of Cp-ring, two signals of the carborane ligand, two signals of Me-groups in the usual areas.



Scheme 2.

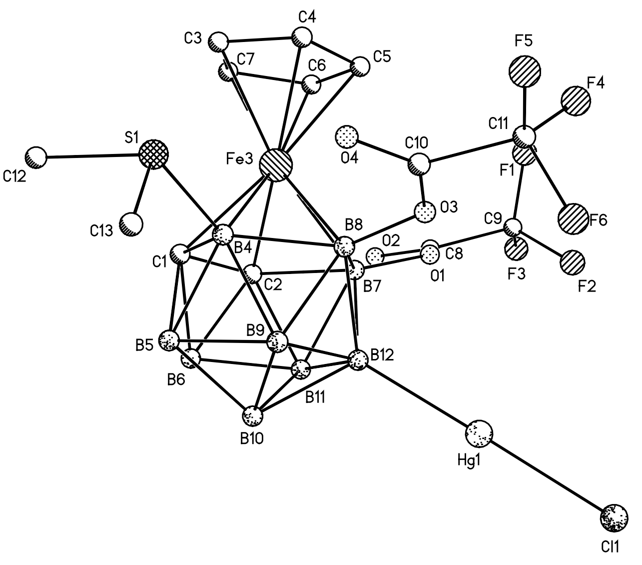


Fig. 2. Crystal structure of molecule **9**.

Complexes **4**–**10** are orange crystalline compounds, slightly soluble in CH_2Cl_2, THF, acetone, insoluble in hexane and diethyl ether. They are stable in the solid state, but their solutions are decomposed in the air.

Thus, the mercuration by the excess of mercurated agent leads to substitution at the positions 8, 12 and 7, and only in the tetrasubstituted derivative **10** at the position 9, too. This can be explained, similar to formation of the complex **2** [6], by stronger effect of the electron acceptor Me_2S-group on the position 9 of the carborane cage in comparison with other positions of the carborane ligand. It is noted that the CF_3COO-groups in all the compounds obtained are located in the open face of the carborane cage.

3. Experimental

All the reactions were carried out under inert atmosphere, using absolute solvents prepared by usual techniques. Isolation of products was performed in air. NMR spectra were obtained on Bruker WP 200-SY and Bruker AMX-400 spectrometers. Complex 3-(η^5-Cp)-4-SMe_2-3,1,2-$FeC_2B_9H_{10}$ (**1**) was obtained according to [8].

3.1. *Mercuration of 3-(η^5-Cp)-4-SMe_2-3,1,2-$FeC_2B_9H_{10}$*

(i) A mixture of 0.20 g (0.64 mmol) **1** and 0.82 g (1.92 mmol) $Hg(OCOCF_3)_2$ in 25 ml CH_2Cl_2 was stirred for 2 h at 20 °C. After filtration the solvent was removed in vacuo and solid residue was chromatographed on SiO_2 column. Eluation with CH_2Cl_2 gave two orange fractions. Further eluation with CH_2Cl_2–C_3H_6O (10:1) afforded additionally two orange fractions. The solutions of each fraction in C_3H_6O were treated with solution of NaCl in water and stirred, precipitates were separated, washed by water and dried in vacuo. Repeated chromatography and recrystallization from CH_2Cl_2–C_3H_6O mixture produced 0.22 g (52%) **4**, 0.03 g (9%) **5**, 0.05 g (12%) **6** and 0.03 g (7%) **7**.

Complex **4**, ^{11}B-NMR (C_3H_6O-d_6): δ 14.8 (s, B(8)), −3.5 (s, $J_{B-Hg} = 2472$ Hz, B(12)), −6.4 (s, B(4)), −10.8 (d, $J_{B-H} = 129$ Hz, B(7)), −11.7 (d, $J_{B-H} = 131$ Hz, B(10)), −15.0 (d, $J_{B-H} = 140$ Hz, B(9)), −24.3 (d, $J_{B-H} = 164$ Hz, B(11)), −25.6 (d, $J_{B-H} = 151$ Hz, B(6)), −30.7 (d, $J_{B-H} = 166$ Hz, B(5)). ^{19}F-NMR (C_3H_6O-d_6): δ 2.46 (s). ^{1}H-NMR (C_3H_6O-d_6): δ 4.84 (s, 5H, Cp), 4.36 (s, 1H, C–H_{carb}), 3.29 (s, 1H, C–H_{carb}), 2.86 (s, 3H, Me), 2.84 (s, 3H, Me). Anal. Calc. for $C_{11}H_{19}B_9ClF_3HgO_2S$: C, 19.97; H, 2.77. Found: C, 19.97; H, 2.90%.

Complex **5**, ^{11}B-NMR (C_3H_6O-d_6): δ 12.4 (s, B(8)), 6.2 (s, B(7)), −7.2 (s, B(4)), −12.3 (d, $J_{B-H} = 146$ Hz, B(12)), −15.8 (d, $J_{B-H} = 140$ Hz, B(9 or 10)), −16.2 (d, $J_{B-H} = 150$ Hz, B(B(10 or 9)), −23.8 (d, $J_{B-H} = 161$ Hz, B(11)), −30.2 (d, $J_{B-H} = 150$ Hz, B(5 or 6)), −30.8 (d, $J_{B-H} = 159$ Hz, B(6 or 5)). ^{19}F-NMR (C_3H_6O-d_6): δ 2.52 (s), 2.31 (s). ^{1}H-NMR (C_3H_6O-d_6): δ 4.67 (s, 1H, C–H_{carb}), 4.63 (s, 5H, Cp), 4.61 (s, 1H, C–H_{carb}), 2.85 (s, 3H, Me), 2.78 (s, 3H, Me). Anal. Calc. for $C_{13}H_{19}B_9F_6FeO_4S$: C, 29.15; H, 3.51. Found: C, 29.00; H, 3.56%.

Complex **6**, ^{19}F-NMR (C_3H_6O-d_6): δ 2.28 (s). ^{1}H-NMR (C_3H_6O-d_6): δ 4.80 (s, 5H, Cp), 4.65 (s, 1H, C–H_{carb}), 4.52 (s, 1H, C–H_{carb}), 2.89 (s, 3H, Me), 2.87 (s, 3H, Me). ^{11}B-NMR (C_3H_6O-d_6): δ 8.8 (s, 1B), 0.2 (s, $J_{B-Hg} = 2220$ Hz, 1B), −5.3 (s, 1B), −10.6 (d, $J_{B-H} = 159$ Hz, 1B), −12.4 (d, $J_{B-H} = 152$ Hz, 1B), −15.3 (d, $J_{B-H} = 143$ Hz, 1B), −21.5 (d, $J_{B-H} = 156$ Hz, 1B), −26.2 (d, $J_{B-H} = 167$ Hz, 1B), −29.5 (d, $J_{B-H} = 155$ H, 1B).

Complex **7**, ^{19}F-NMR (C_3H_6O-d_6): δ 2.54 (s). ^{1}H-NMR (C_3H_6O-d_6): δ 5.03 (s, 5H, Cp), 4.62 (s, 1H, C–H_{carb}), 4.15 (s, 1H, C–H_{carb}), 2.89 (s, 3H, Me), 2.87 (s, 3H, Me). ^{11}B-NMR (C_3H_6O-d_6): δ 1.9 (s, 1B), −1.1 (s, $J_{B-Hg} = 2467$ Hz, 1B), −3.4 (s, 1B), −7.2 (d, $J_{B-H} = 145$ Hz, 1B), −10.6 (d, $J_{B-H} = 137$ Hz, 1B), −14.1 (d, $J_{B-H} = 144$ Hz, 1B), −22.6 (d, $J_{B-H} = 169$ Hz, 1B), −29.1 (d, $J_{B-H} = 137$ Hz, 2B).

(ii) A mixture of 0.30 g (0.95 mmol) **1** and 4.80 g (11.25 mmol) of $Hg(OCOCF_3)_2$ was stirred in 20 ml CH_2Cl_2 for 2 h at 20 °C. The solvent was removed in vacuo and solid residue was chromatographed on SiO_2 column with CH_2Cl_2–C_3H_6O (10:1) mixture as eluent to give 0.03 g (4%) of **8** and second fraction containing mixture of two compounds. Solution of **8** was stirred with a solution of NaCl in water. The precipitate was isolated, washed by water, dried in vacuo, and chromatographed on column SiO_2. Compound **9** (0.01 g) was isolated. Similar treatment of the second fraction with

NaCl followed by repeated chromatography allowed to isolate 0.03 g (4%) of **9** (eluent: CH_2Cl_2–$C_3H_6O = 10{:}1$) and 0.9 g (93%) of **10** (eluent: CH_2Cl_2–$C_3H_6O = 5{:}1$).

Complex **8**, ^{11}B-NMR (C_3H_6O-d_6): δ 12.3 (s, B(8)), 6.1 (s, B(7)), −6.9 (s, B(4), −9.7 (s, $J_{B-Hg} = 2668$ Hz, B(12)), −15.0 (d, $J_{B-H} = 141$ Hz, B(9,10)), −22.8 (d, $J_{B-H} = 164$ Hz, B(11)), −29.4 (d, $J_{B-H} = 136$ Hz, B(5 or 6)), −30.4 (d, $J_{B-H} = 149$ Hz, B(6 or 5)). ^{19}F-NMR (C_3H_6O-d_6): δ 4.09 (s), 2.57 (s), 2.46 (s). ^{1}H-NMR (C_3H_6O-d_6): δ 4.76 (s, 5H, Cp), 4.61 (s, 1H, C–H_{carb}), 4.33 (s, 1H, C–H_{carb}), 2.84 (s, 3H, Me), 2.77 (s, 3H, Me).

Complex **9**, ^{11}B-NMR (C_3H_6O-d_6): δ 12.5 (s, B(8)), 6.2 (s, B(7)), −5.6 (s, $J_{B-Hg} = 2188$ Hz, B(12)), −6.7 (s, B(4)), −15.0 (d, $J_{B-H} = 120$ Hz, B(9,10)), −22.8 (d, $J_{B-H} = 158$ Hz, B(11)), −29.3 (d, $J_{B-H} = 141$ Hz, B(5 or 6)), −30.3 (d, $J_{B-H} = 148$ Hz, B(6 or 5)). ^{19}F-NMR (C_3H_6O-d_6): δ 2.47 (s), 2.36 (s). ^{1}H-NMR (C_3H_6O-d_6): δ 5.62 (s, 5H, Cp), 4.61 (s, 1H, C–H_{carb}), 4.33 (s, 1H, C–H_{carb}), 2.87 (s, 3H, Me), 2.84 (s, 3H, Me). Anal. Calc. for $C_{13}H_{18}B_9ClF_6FeHgO_4S$: C, 20.24; H, 2.53; F, 14.07. Found: C, 20.19; H, 2.35; F, 14.74%.

Complex **10**, ^{11}B-NMR (C_3H_6O-d_6): δ 13.2 (s, B(8)), 6.7 (s, B(7)), −4.0 (s, $J_{B-Hg} = 2576$ Hz, B(12 or 9)), −6.2 (s, B(4)), −8.0 (s, $J_{B-Hg} = 2650$ Hz, B(9 or 12)), −13.9 (d, $J_{B-H} = 140$ Hz, B(10)), −21.4 (d, $J_{B-H} = 160$ Hz, B(11)), −28.1 (d, $J_{B-H} = 160$ Hz, B(5,6)). ^{19}F-NMR (C_3H_6O-d_6): δ 2.49 (s), 2.35 (s). ^{1}H-NMR (C_3H_6O-d_6): δ 4.74 (s, 5H, Cp), 4.73 (s, 1H, C–H_{carb}), 4.48 (s, 1H, C–H_{carb}), 2.89 (s, 3H, Me), 2.88 (s, 3H, Me). Anal. Calc. for $C_{13}H_{17}B_9Cl_2F_6FeHg_2O_4S$: C, 15.55; H, 1.84; B, 9.69. Found: C, 15.48; H, 1.70; B, 9.65%.

3.1.1. *Crystal data for 9*

The yellow crystal 3-(η^5-Cp)-4-SMe_2-7,8-$(CF_3COO)_2$-12-HgCl-3,1,2-$FeC_2B_9H_7$, $C_{13}H_{18}B_9ClFeF_6HgO_4S$ ($M = 773.51$), monoclinic, space group $P2_1/c$ (no. 14), $a = 14.487(3)$, $b = 8.864(2)$, $c = 19.593(4)$ Å, $\beta = 95.45(3)^\circ$, $V = 2504.6(9)$ Å^3, $Z = 4$, $D_{calc} = 2.051$ g cm^{-3}, $\mu = 6.957$ mm^{-1}, $F(000) = 1464$, crystal size $= 0.55 \times 0.35 \times 0.20$ mm.

Single-crystal X-ray diffraction experiments were carried out with a CAD4 Enraf–Nonius diffractometer, using graphite monochromated Mo–K_α radiation ($\lambda = 0.71073$ Å, $\theta - 5/3\theta$-scan technique, the absorption correlation was introduced using the experimental curves of azimuthal scan (18 reflections, $0 < \psi < 360^\circ$ with an interval of 10°, Tmin = 0.563 and Tmax = 0.9573), $2\theta < 50^\circ$) at 295 K. A total of 4545 reflections were measured and 4359 ($R_{int} = 0.0290$) independent reflections were used in further calculations and refinement. The structures were solved by direct method and refined by the full-matrix least-squares against F^2 in anisotropic (for non-hydrogen atoms) approximation. The H atoms of the Cp were placed in geometrically calculated positions and included in the refinement using the riding model with the Uiso(H) = 1.2Ueqv(C), where Ueqv(C) is the equivalent isotropic temperature factor of the carbon atom bonded to the corresponding H atom. All the other H atoms were located from the difference Fourier syntheses and included in the refinement with the fixed positional and thermal parameters.

The final refinements were converged to $R_1 = 0.0330$ (from 2730 unique reflections with $I > 2\sigma(I)$) and $wR_2 = 0.0727$ (from all 4359 unique reflections); the number of the refined parameters is 325. All the calculations were performed on an IBM PC/AT using the SHELXTL software [9].

4. Supplementary material

Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Centre, CCDC no. 169958 for compound **9**. Copies of this information may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (Fax: +44-1223-336033; e-mail: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk).

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