

Accepted Manuscript

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PII: S0022-328X(18)30255-9

DOI: [10.1016/j.jorganchem.2018.04.019](https://doi.org/10.1016/j.jorganchem.2018.04.019)

Reference: JOM 20411

To appear in: *Journal of Organometallic Chemistry*

Received Date: 31 January 2018

Revised Date: 11 April 2018

Accepted Date: 12 April 2018

Please cite this article as: S.A. Anufriev, S.A. Erokhina, K.Y. Suponitsky, A.A. Anisimov, J.N. Laskova, I.A. Godovikov, F. Fabrizi de Biani, M. Corsini, I.B. Sivaev, V.I. Bregadze, Synthesis and structure of Bis(Methylsulfanyl) derivatives of iron Bis(Dicarbollide), *Journal of Organometallic Chemistry* (2018), doi: 10.1016/j.jorganchem.2018.04.019.

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Synthesis and Structure of Bis(Methylsulfanyl) Derivatives of Iron Bis(Dicarbollide)¹

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Abstract. Bis(methylsulfanyl) derivatives of iron(II) bis(dicarbollide) [8,8'-(MeS)₂-3,3'-Fe(1,2-C₂B₉H₁₀)₂]²⁻ (**4**²⁻), [4,4'-(MeS)₂-3,3'-Fe(1,2-C₂B₉H₁₀)₂]²⁻ (**5**²⁻), and [4,7'-(MeS)₂-3,3'-Fe(1,2-C₂B₉H₁₀)₂]²⁻ (**6**²⁻) were prepared by the treatment of the corresponding dimethylsulfonium derivatives **1-3** with potassium butylthiolate. Their oxidation by air in aqueous solution results in the corresponding derivatives of iron(III) bis(dicarbollide) [8,8'-(MeS)₂-3,3'-Fe(1,2-C₂B₉H₁₀)₂]⁻ (**7**⁻), [4,4'-(MeS)₂-3,3'-Fe(1,2-C₂B₉H₁₀)₂]⁻ (**8**⁻) and [4,7'-(MeS)₂-3,3'-Fe(1,2-C₂B₉H₁₀)₂]⁻ (**9**⁻). The structures of (Bu₄N)₂[8,8'-(MeS)₂-3,3'-Fe(1,2-C₂B₉H₁₀)₂] and (Me₄N)[4,7'-(MeS)₂-3,3'-Fe(1,2-C₂B₉H₁₀)₂] were determined through single-crystal X-ray diffraction. In the solid state, the rotation of the dicarbollide ligands with respect to each other is hampered due to formation of weak intramolecular CH...S(Me) hydrogen bonds between the ligands resulting in stabilization of *transoid*-conformation in the case of 8,8'-isomer and *gauche*-conformation in the case of 4,4'-

¹ Dedicated to Professor Narayan Hosmane in recognition of his outstanding contribution in the field of carborane and metallacarborane chemistry and with very best wishes on the occasion of his 70th birthday.

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