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Crystallographic report

$[(\eta^5\text{-}C_5H_5)Fe(CO)_2]_2Ga_3Cl_3(OSiMe_2OSiMe_2O)_2$: a diiron complex of a tetracyclic trigallasiloxane

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The structure of the penta-metallic diiron trigallasiloxane, $[(\eta^5\text{-}C_5H_5)Fe(CO)_2]_2Ga_3Cl_3(OSiMe_2OSiMe_2O)_2$, reveals two distinct gallium coordination environments and Fe–Ga bond lengths (2.3258(6) Å), consistent with bonding of the iron centres to four-coordinate gallyl ligands. Copyright © 2004 John Wiley & Sons, Ltd.

KEYWORDS: crystal structure; iron; gallium; gallyl; siloxane

COMMENT

The title complex (**I**) was isolated as a minor product from the recrystallization of $(\eta^5\text{-}C_5H_5)Fe(CO)_2Ga(^s\text{Mes})Cl$ (Bunn NR, Aldridge S, unpublished results; $^s\text{Mes} = 2, 4, 6\text{-}^t\text{Bu}_3\text{C}_6\text{H}_3$) from toluene solution in the presence of adventitious silicone grease. The structure of the trigallasiloxane core (Fig. 1) is very similar to that found in the corresponding pentachloride species $Ga_3Cl_5(OSiMe_2OSiMe_2O)_2$,[1,2] with symmetry-equivalent fragments being related through a twofold rotation axis aligned along Ga(2)–Cl(2). The Fe–Ga bond lengths to the cis-orientated pendant $[(\eta^5\text{-}C_5H_5)Fe(CO)_2]$ groups are within the range expected for bonding to four-coordinate gallium centres (2.29–2.44 Å), as determined from a survey of the Cambridge Crystallographic Database.

EXPERIMENTAL AND RESULTS

A solution of $(\eta^5\text{-}C_5H_5)Fe(CO)_2Ga(^s\text{Mes})Cl$ (0.52 mmol) in toluene (5 ml) was cooled to −30 °C over a period of 1 week. Reaction with adventitious grease yielded the title compound (**I**) as colourless blocks in low yield (four or five crystals). Intensity data for **I** were collected at 150 K on a Nonius Kappa CCD diffractometer for a colourless crystal 0.15 × 0.25 × 0.25 mm^3. $C_{22}H_{34}Cl_3Fe_2Ga_3O_{10}Si_4$, $M = 998.06$, monoclinic, $C2/c$, $a = 16.338(3)$, $b = 13.349(3)$, $c = 17.854(4)$ Å, $\beta = 105.16(3)°$, $V = 3758.4(13)$ Å^3, $Z = 4$, 4224 unique data (θ_{max} 27.5°),

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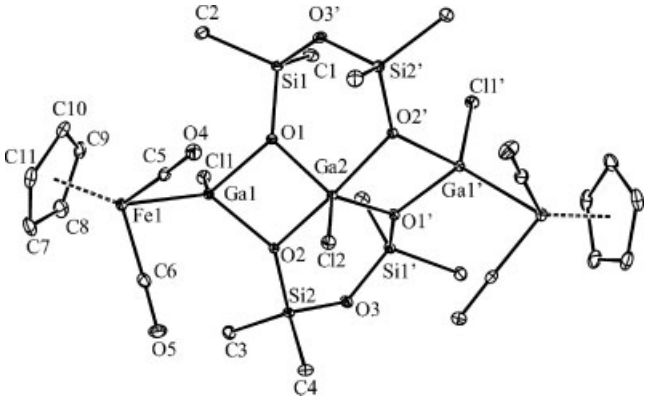

Figure 1. Molecular structure of **I**; hydrogen atoms omitted for clarity. Key geometric parameters: Ga1–Fe1 2.3258(6), Ga1–Cl1 2.2145(8), Ga1–O1 1.9760(18), Ga1–O2 1.9357(18), Ga2–Cl2 2.1828(11), Ga2–O1 1.9020(18), Ga2–O2 1.9851(18), Fe1–C5 1.756(3), Fe1–C6 1.751(3), Fe1–Cp centroid 1.718(3), Si1–O1 1.6764(19), Si1–O3′ 1.636(2), Si1–C1 1.846(3), Si1–C2 1.843(3), Si2–O2 1.6638(19), Si2–O3 1.646(2), Si2–C3 1.849(3), Si2–C4 1.846(3) Å; O1–Ga1–Cl1 103.15(6), O2–Ga1–Cl1 103.56(6), O1–Ga1–O2 79.68(8), Cl1–Ga1–Fe1 122.02(3), O1–Ga1–Fe1 118.48(6), O2–Ga1–Fe1 121.24(6), O1–Ga2–O2 80.26(8), O1–Ga2–O1′ 122.53(11), O1–Ga2–O2′ 94.20(8), O2–Ga2–O2′ 168.55(11), O1–Ga2–Cl2 118.74(6), O2–Ga2–Cl2 95.73(5), C5–Fe1–C6 95.46(14)°. Symmetry transformations used to generate primed atoms: $1 - x$, y, $3/2 - z$.

3625 data $I \geq 2\sigma(I)$, $R = 0.033$, $wR = 0.082$ (all data). Programs used: SHELXS-97, SHELXL-97, X-seed and ORTEP. CCDC deposition number: 234501.

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