

Erratum

Erratum to “Cyclopentadienyl-functionalised polyhedral silsesquioxanes as building blocks for new nanostructured materials”
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Experimental, page 467, in 3.2. *Synthesis of Cp₁₀Si₁₀O₁₅ (1)*

It should read: “The mixture was stirred for one week.”

Experimental, page 467, in 3.4. *Synthesis of (CpC₃H₆)₈Si₈O₁₂ (3)*

It should read: “The mixture was stirred for one week at ambient temperature.”

Corrected experimental procedures for compounds **1** and **3** are below.

3.2. *Synthesis of Cp₁₀Si₁₀O₁₅ (1)*

Freshly prepared trichloro-cyclopenta-2,4-dienyl-silane (21.9 g, 0.11 M) in 100 ml of THF was carefully hydrolysed by the drop wise addition of a solution of (NH₄)₂CO₃ (2.97 g) in distilled water (70 ml) at 0 °C. The mixture was stirred for one week. The product was extracted with diethyl ether and the organic layer was separated and dried with anhydrous magnesium sulphate. The solution was concentrated in vacuum. A pale yellow solid **1** was precipitated. The precipitate was washed with diethyl ether and dried in vacuum to give 8.6 g (67%) of **1**. Anal. Calc. for C₅₀H₅₀Si₁₀O₁₅: C, 51.28; H, 4.27. Found: C, 52.03; H, 4.34%. ¹H NMR (400 MHz, CDCl₃, 22 °C) δ: 5.71 (40H, vbr, Cp, H–C=), 3.22 (10H, vbr, Cp, H–C–). ²⁹Si NMR (99 MHz, CDCl₃, 22 °C) δ: –71.50(s), –74.39(s), –77.04(s). IR (KBr, cm^{–1}): 2962(m), 1709(m), 1446 (m),

1261 (m), 1105(w sh), 845(w), 801(w), 757(m), 431(w). MS (ESI TOF, CH₃CN–H₂O): 1171 [M+H]⁺, 1105 [M–Cp]⁺, 846 [M–5Cp+H⁺]⁺, 371 [M–Cp+2H⁺]²⁺. Cryoscopy in *c*-hexane: average (out of 5) M.w. 1193.

3.4. *Synthesis of (CpC₃H₆)₈Si₈O₁₂ (3)*

(3-Cyclopentadienylpropyl)triethoxysilane was prepared from (3-chloropropyl)-triethoxysilane (60.20 g, 0.25 M) and CpNa in THF and was purified by vacuum distillation (b.p. 90–91 °C/1 mm, 41 g, 61%). The hydrolytic condensation of (3-cyclopentadienylpropyl)triethoxysilane (20 g, 0.074 M) in acetone (170 ml) was performed by the addition of distilled water (25 ml), with the traces of conc. HCl (0.5 ml) at 0 °C. The mixture was stirred for one week at ambient temperature. The solvent was removed in vacuum to give a pale yellow precipitate. The product was washed with hexane and dried in vacuum (6.94 g, 59%). Anal. Calc. for C₆₄H₈₈Si₈O₁₂: C, 60.38; H, 6.92. Found: C, 60.63; H, 6.98%. ¹H NMR (400 MHz, CDCl₃, 22 °C) δ: 6.4–5.99 (32H, br m, Cp), 3.51 (8H, br m, Cp), 1.85–0.77 (48H, vbr m, CH₂). ²⁹Si NMR (99 MHz, CDCl₃, 22 °C) δ: –68.51 (br s). IR (KBr, cm^{–1}): 2933(w), 1713(m), 1455(m), 1383(w), 1155(w m), 1050(w), 799(w), 700(m), 467(w sh). MS (ESI TOF, CH₃CN): 1272 [M]⁺, 1207 [M–Cp]⁺, 844 [M–4CpC₃H₆]⁺. Cryoscopy in *c*-hexane: average (out of 5) M.w. 1297.

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