

Description:

Tricyclo[5.2.1.02,6]deca-3,8-diene (147 g, 150 mL, 1.11 mol, 1.00 equiv) was cracked at 180 °C in a distillation apparatus to yield cyclopenta-1,3-diene (64.0 g, 80.0 mL, 968 mmol, 87% yield). The obtained cyclopenta-1,3-diene was stored at -30 °C.

Literature:



Solvent(s): Toluene (100ml), THF (100ml)

Description:

The reaction has been conducted in dry glass ware under inert atmosphere. Sodium (3.60 g, 157 mmol, 1.02 equiv) was heated to 140 °C in dry toluene (100 mL) to finely disperse the sodium. After cooling to room temperature, the solvent was removed and tetrahydrofuran (100 mL) was added to the sodium sand. Cyclopenta-1,3-diene (10.2 g, 12.7 mL, 154 mmol, 1.00 equiv) was added dropwise to the cooled solution of sodium in THF (*strong gas evolution observable*). Afterwards, the reaction mixture was stirred at room temperature overnight whereupon no elementary sodium was left.

Literature:



Solvent(s): THF (80.0ml)

Description:

The reaction has been conducted in dry glass ware under inert atmosphere. Iron (2.68 g, 48.0 mmol, 1.00 equiv) and iron(III) chloride (15.6 g, 96.0 mmol, 2.00 equiv) were suspended in tetrahydrofuran (80.0 mL). The reaction mixture was refluxed for 4.5 hours. After cooling to room temperature, the solvent was removed under reduced pressure. Iron(II) chloride (17.1 g, 135 mmol, 94% yield) was obtained as a gray amorphous solid.

Literature:



Solvent(s): THF (100ml)

Description:

The reaction has been conducted in dry glass ware under inert atmosphere. Sodium cyclopenta-1,3-diene (11.5 g, 130 mmol, 1.00 equiv) was solved in dry tetrahydrofuran (100 mL). The reaction mixture was cooled to 0 °C and iron(II) chloride (9.13 g, 72.0 mmol, 0.55 equiv) was added in small portions. Afterwards, the mixture was slowly warmed up to room temperature and additionally stirred overnight. On the next day, a small amount of isopropanol was added to the mixture to quench the remaining unreacted sodium cyclopenta-1,3-diene. The solvent was removed under reduced pressure, the residue was extracted with pentane (50 mL), filtered through diatomaceous earth and the filter cake was washed with pentane. The solvent was removed under reduced pressure. The crude product was sublimed under static vacuum at 140 °C to yield ferrocene (7.69 g, 41.3 mmol, 64% yield) as an orange-red crystalline solid.

Type of Purification: Sublimation

Analysis:

C10H10Fe (CHMO:0000593 | 1H nuclear magnetic resonance spectroscopy (1H NMR))

¹H NMR (43 MHz, Chloroform-d [7.27 ppm], ppm) δ = 4.23 (s, 10H).

Literature: