

Direct high yield synthesis of $[\text{Ir}(\text{OH})(\text{CO})(\text{PPh}_3)_2]$ from Vaska's compound by phase-transfer catalysis

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Abstract

Treatment of Vaska's compound with 50% aqueous sodium hydroxide using benzyl(triethyl)ammonium chloride as phase-transfer catalyst gave $[\text{Ir}(\text{OH})(\text{CO})(\text{PPh}_3)_2]$ in high yield.

Keywords: Iridium; Vaska's compound; Phase transfer catalysis

1. Introduction

Although complexes of the type $[\text{MX}(\text{CO})(\text{PPh}_3)_2]$ ($\text{X}=\text{F}$, Br, I, OCl_3 , N_3 or NCS) can be prepared directly from Vaska's compound by simple metathesis [1–6], this route does not seem to be practicable for the synthesis of $[\text{Ir}(\text{OH})(\text{CO})(\text{PPh}_3)_2]$. Instead, $[\text{IrCl}(\text{CO})(\text{PPh}_3)_2]$ must first be converted to the unstable fluoro-compound $[\text{IrF}(\text{CO})(\text{PPh}_3)_2]$, which is then treated with MOH ($\text{M}=\text{Li}$, Na or K) [1]. Another method for the preparation of $[\text{Ir}(\text{OH})(\text{CO})(\text{PPh}_3)_2]$ involves treatment of *trans*- $[(\text{MeO})\text{Ir}(\text{CO})(\text{PPh}_3)_2]$ with water [7].

We report here a convenient high yield one-step synthesis of $[\text{Ir}(\text{OH})(\text{CO})(\text{PPh}_3)_2]$ direct from Vaska's compound by metathesis making use of phase-transfer catalysis.

2. Results

Refluxing for 2 h of a mixture of $[\text{IrCl}(\text{CO})(\text{PPh}_3)_2]$ in benzene with 50% aqueous sodium hydroxide and benzyl-(triethyl)ammonium chloride as phase-transfer catalyst gave $[\text{Ir}(\text{OH})(\text{CO})(\text{PPh}_3)_2]$ in 73% yield. The product was identified by its physical data properties, including melting point, IR and ^{31}P - $\{^1\text{H}\}$ NMR spectra, and elemental analysis. The IR spectrum showed $\nu(\text{CO})$ at 1930 and $\nu(\text{OH})$ at 3612 nm (Nujol mull). The ^{31}P - $\{^1\text{H}\}$ NMR spectrum in CDCl_3 showed a single band at $\delta = 26$ ppm. The data are in agreement with those published previously [1,7].

3. Experimental details

The general procedure was the same as that described in recent papers from this laboratory [8]. In a typical reaction a mixture of $[\text{IrCl}(\text{CO})(\text{PPh}_3)_2]$ (0.59 g, 0.75 mmol), benzene (90 cm^3), 50% aqueous sodium hydroxide (30 cm^3), and benzyl(triethyl)ammonium chloride (0.34 g) was heated under reflux for 2 h under argon. The mixture was cooled to room temperature, water (50 cm^3) was then added, and the organic layer was separated and dried over MgSO_4 . The solvent was then evaporated to leave the product as a yellow solid (yield 0.42 g, 73%), melting point 176–177°C. Anal. Found, H 4.27; C 58.5; Calc, H 4.1, C 58.33.

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