Experiment 3 — Synthesis and analysis of $[Mo(CO)_4(PPh_3)_2]$

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Synthesis

Part 1 — Synthesis of $[Mo(CO)_4(morpholine)_2]$

Using oven dried equipment and under an atmosphere of dinitrogen $[Mo(CO)_6]$, (0.80 g), dry morpholine (9.00 cm^3), and dry heptane (20.0 cm^3) were heated under reflux. The $[Mo(CO)_6]$ dissolved to give a pale yellow solution. After *circa* 40 minutes the solution turned dull and then *circa* 50 minutes yellow precipitate started to form. After *circa* 60 minutes precipitate ceased to form. The hot mixture was filtered via vacuum and washed with dry heptane (20 cm^3). The yellow product was dried. (1.04 g, 89 %)

Part 2 — Synthesis of $[Mo(CO)_4(PPh_3)_2]$

With the same setup as in Part 1 $[Mo(CO)_4(morpholine)_2]$ (0.62 g), triphenylphosphine (1.00 g) and dry dichloromethane (30.0 cm^3) were heated to reflux for *circa* 15 minutes. The solution was cooled to room temperature and then filted under gravity. The solvent was removed under reduced pressure (until *circa* 8 cm^3) and methanol added (15 cm^3). The solution was cooled in an ice bath and the yellow crytals collected and dried via vacuum filtration. (0.60 g, 51 %)

Analysis

The IR spectrum of $[Mo(CO)_4(PPh_3)_2]$ in a solution of DCM was collected.

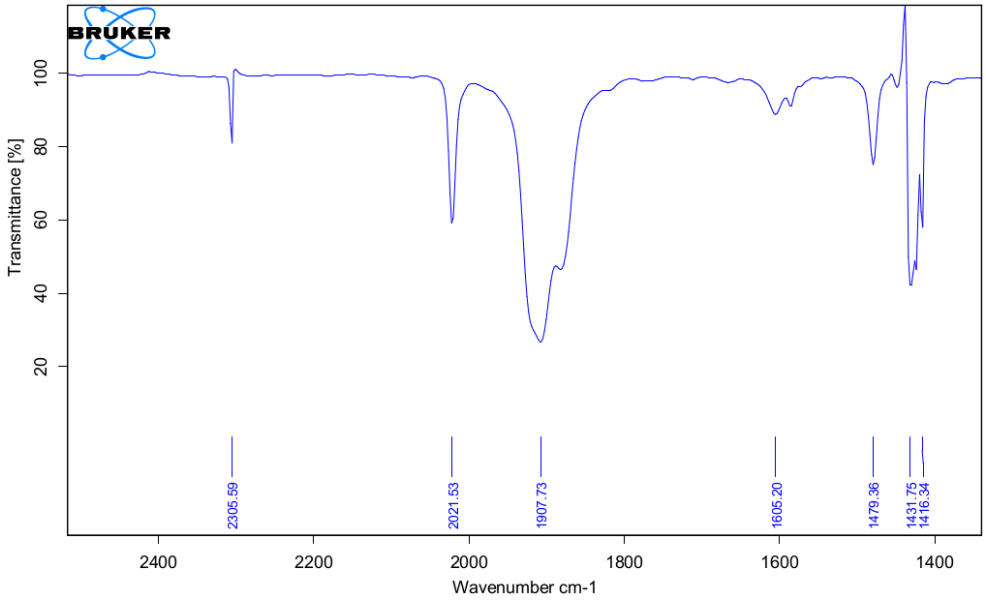

Figure 1: IR spectrum of $[Mo(CO)_4(PPh_3)_2]$