

Synthesis of nickel complexes



Abstract

The purpose of this experiment was to investigate the Ligand exchange that occurs when different Nickel complexes and reacted with Triphenylphosphine.

Introduction

Triphenylphosphine was first prepared by Pfeiffer and Sauvage in 1904

Experimental

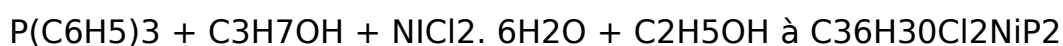
Triphenylphosphine (1.408g) was dissolved under reflux with Propan-2-ol (15cm³) forming a colourless solution to which 5 boiling chips were added. Nickel chloride hexahydrate (0.603g) was dissolved in absolute Ethanol (10cm³) and heated until a green/yellow solution formed. This solution was poured into the reaction mixture through the condenser. Immediately the solution turned dark green, the solution was refluxed for a further 6mins. The reaction was removed from the heat and allow to cool for 4mins, a dark precipitate was visible in the flask. The warm solution was filtered under suction to collect the dark blue/green crystals which were washed with ice cooled absolute ethanol (1x10cm³ + 1x3cm³). Once dry the boiling chips were removed with a spatula and the crystals were transferred to a n open sample vial and dried in the desiccator for a week.

A further solution of Triphenylphosphine (1.410g) and Propan-2-ol (15cm³) was made and set to reflux as before. Sodium thiocyanate (0.601g) was dissolved in Ethanol (11cm³), to this nickel nitrate hexahydrate (0.773g) was added. The flask was warmed until all the green solid has dissolved and a white solid has formed. Once cooled the solution was filtered under suction and the Filtrate (aqua blue liquid in Buchner flask) was added to the refluxing <https://assignbuster.com/synthesis-of-nickel-complexes/>

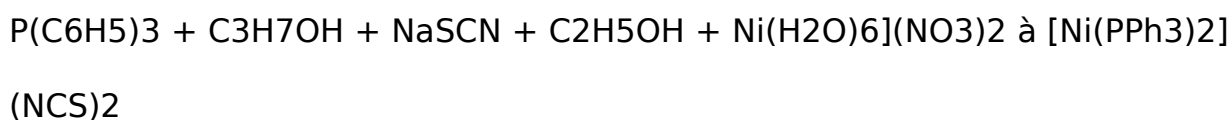
Triphenylphosphine through the condenser. Immediately the solution turned blood red/brown. The solution was refluxed for a further 7mins and then cooled for 5 mins. The warm solution was filtered under suction and the orange/ red crystals washed with ice cooled Ethanol (1x10cm³ + 1x8cm³). Once dry the boiling chips were removed as before and the crystals were transferred to the desiccator for a week.

Results and discussion

Triphenylphosphine + Propan-2-ol+ Nickle Chloride hexahydrate + Ethanol à
Bis(triphenylphosphine)nickel(II)chloride



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Equations

XM = Molar magnetic susceptibility (cgs units)

C= Calibration constant = 1. 044

L= Length of sample (cm units)

R= Balance reading with FULL samples tube

Ro= Balance reading with EMPTY sample tube

M= Relative molecular weight of sample

m= Mass of samples (units grams)

$X'M = XM -$ (diamagnetic correction)

$X'M =$ Corrected Molar susceptibility

$XM =$ Molar magnetic susceptibility (cgs units)

Diamagnetic correction = Calculated from literature table of diamagnetic corrections (X_d)

$\mu_{\text{eff}} =$ Magnetic moment (units Bohr Magneton (BM))

$X'M =$ Corrected Molar susceptibility

T= Temperature (units Kelvin (K))

Calculations

$XM = 294.0798111 \times 10^{-6}$

Diamagnetic correction (data obtained from Inorganic Chemistry CMB004 d-block Chemistry Laboratory course 2009-10 page 14)

Compound: $[\text{Ni}(\text{PPh}_3)_2\text{Cl}_2]$

Diamagnetic correction = $(-13 \times 10^{-6}) + (-46 \times 10^{-6}) + (-400 \times 10^{-6}) = -459 \times 10^{-6}$

$X'M = XM -$ (diamagnetic correction)

$X'M = 294.079811 \times 10^{-6} - (-459) \times 10^{-6} = 753.079811 \times 10^{-6}$

$$\mu_{\text{eff}} = 1.33 \text{ BM}$$

Complex B showed a magnetic moment decrease of -055 to -073 on the first measurement so I re ran the measurement.