

CHM-304

Experiment: Preparation of phosphine based metal complexes

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(18147)

7 April 2022

Aim of the Experiment:

Preparation of phosphine based metal complexes.

Chemicals:

Nickel(II) chloride hexahydrate, Acetic acid, Triphenyl phosphine

Glassware:

Beaker (100 mL x 2 nos), spatula, glass rod, measuring cylinder (10 mL & 50 mL), 2 Necked RB, Condenser, take off, filtration flask, Buchner funnel, magnetic bar, sample vial.

Theory:

Tertiary phosphines, PR_3 , are an important class of ligands because their electronic and steric properties can be altered in a systematic and predictable way over a very wide range by varying the R group(s). Tertiary phosphines also stabilize an exceptionally wide variety of metal complexes of interest to the organometallic chemist as their phosphine complexes $(R_3P)_nML$. Phosphines are more commonly spectator rather than actor ligands. Like NR_3 , phosphines have a lone pair on the central atom that can be donated to a metal. Unlike NR_3 , they are also π -acids, to an extent that depends on the nature of the R groups present on the PR_3 ligand. For alkyl phosphines, the π acidity is weak; aryl, dialkylamino, and alkoxy groups are successively more effective in promoting acidity. In the extreme case of PF_3 , the π acidity becomes as great as that found for CO. Such ligands can be classified according to their donor strength and steric bulk. These properties can be quantified by the Tolman electronic parameter and ligand cone angle, respectively. Generally, alkyl phosphines are stronger bases and σ -donors. Triphenylphosphine is a common organophosphorus compound with the formula $P(C_6H_5)_3$ - often abbreviated to PPh_3 . It is widely used in the synthesis of organic and organometallic compounds. PPh_3 exists as relatively air stable, colorless crystals at room temperature. It dissolves in non-polar organic solvents such as benzene and diethyl ether. Many PPh_3 based organometallic complex are well known as catalyst like Wilkinson's catalyst $[Rh(PPh_3)_3Cl]$, Vaska's complex $[Ir(CO)Cl(PPh_3)_2]$ etc. Ni based metal phosphine compound will be synthesised during this lab. $Ni(PPh_3)_2Cl_2$ is used as catalyst in alkyne trimerisations and carbonylations. Complex is also a catalyst in Suzuki reactions as an alternative to the traditional palladium (0) catalysts because nickel is cheaper and more abundant.

The reaction is based on the following equation:



Procedure:

Synthesis of $Ni(PPh_3)_2Cl_2$ To a solution of nickel (II) chloride hexahydrate (0.59 g, 5mmol) in water (1 mL), add glacial acetic acid (25 mL). To this add a solution of tri phenyl phosphine (1.31 g, 10mmol) dissolved in acetic acid (7.5mL). Olive green microcrystalline precipitate will be formed. Keep the solution and the precipitate in contact with the mother liquor for

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