Experiment No. 6

**Silylation of 2-chlorophenol and purification**

**Aim**: Silylation of 2-chlorophenol and purification

**Chemical**: 2-chlorophenol, Trimethylsilyl chloride, Imidazole, Dimethylformamide (DMF).

**Apparatus**: 50 mL round bottom flask, 250 mL conical flask, separating funnel, funnel, filter   
 paper, stir bar, hot plate.

**General procedure:**

To a 0°C solution of 2-chlorophenol (1 equivalent) in DMF (5 mL), Imidazole (1.2 equivalent) was added. The mixture stirred at 0°C for 15 min. Then, trimethylsilyl chloride (1.2 equivalent) was added into it slowly and the mixture stirred at room temperature for 3 hrs under inert atmosphere. Reaction was monitored by TLC, after completion, quenched by adding ice water. Then the residue was extracted with ethyl acetate (50 mL) and washed several times with water (50 mL) and brine solution. After that, the organic layer was dried over MgSO4, filtered, and concentrated under vacuum.



**Result**: The product obtained was:

1.

2.

3.

4.

5.

Yield (%):

IR:

TLC:

Questions:

1. Write five different examples of silyl protection of –OH group (with reaction mechanism)

2. What is the role of imidazole in this reaction?

3. Why we cannot purify the product by silica gel column chromatography?