

## Experiment No. 05

**AIM:** To prepare and submit tolbutamide from p-toluene sulfonamide and calculate its percentage yield.

### **REFERENCES:**

1. Vogel's Textbook of Practical Organic Chemistry by Brian S. Furniss, Antony J. Hannaford, Peter W. G. Smith & Austin R. Tatchell; Fifth Edition; Page No.....

### **REQUIREMENTS**

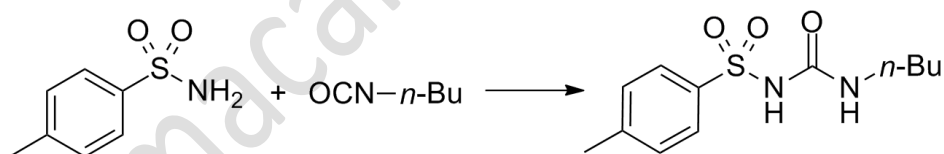
**Chemicals:** p-toluene sulfonamide, Methyl isocyanate, Sodium hydroxide, Ethanol, Ice-cold water, and recrystallization reagents (ethanol or a suitable solvent).

**Apparatus:** 250 ml round-bottom flask, magnetic stirrer, heating mantle or water bath, reflux condenser, Filtering apparatus, melting point apparatus, and balance.

### **PRINCIPLE:**

Tolbutamide is a sulfonylurea hypoglycaemic agent used to manage type 2 diabetes. It functions by stimulating insulin release from the pancreatic beta cells. The synthesis of tolbutamide involves the reaction of p-toluene sulfonamide with isocyanates to form the corresponding sulfonylurea compound.

### **REACTION:**



### **PROCEDURE:**

1. Weigh 1.0 g of p-toluene sulfonamide and place it in a 250 ml round-bottom flask. Add 10 ml of ethanol to the flask to dissolve the p-toluene sulfonamide.
2. Add 0.5 g of sodium hydroxide to create a basic environment. Slowly add 0.8 g of methyl isocyanate to the reaction mixture while stirring continuously.
3. Attach a reflux condenser to the flask and heat the mixture in a water bath at 60-70°C for 4 hours with constant stirring.
4. After the reaction, cool the mixture to room temperature. Pour the reaction mixture into a beaker containing ice-cold water to precipitate the crude product.
5. Filter the solid product using a filtration apparatus. Wash the solid with ice-cold water to remove any impurities.

6. Dissolve the crude product in a minimum amount of hot ethanol. Allow the solution to cool slowly to room temperature, then place it in an ice bath to complete the crystallization.
7. Filter and dry the recrystallized product. And calculate its percentage yield and melting point.

**CALCULATION:**

The limiting reagent is p-toluene; Hence, the yield should be calculated from the amount taken.

The molecular formula of p-toluene sulfonamide =  $C_7H_9O_2NS$

The molecular formula of Tolbutamide =  $C_{12}H_{18}O_3N_2S$

The molecular weight of p-toluene sulfonamide = 171g/mole

The molecular weight of Tolbutamide = 270g/mole

***Theoretical yield:***

171g p-toluene sulfonamide forms 270 g Tolbutamide

Therefore, 1g p-toluene sulfonamide will form .....? (X) g Tolbutamide

$$270 \times 1/171 = 1.58 \text{ g}$$

Theoretical yield = 1.58 g

Practical yield = \_\_\_\_\_ g

$$\% \text{ Yield} = (\text{Practical Yield})/(\text{Theoretical Yield}) \times 100$$

**RESULT:**

Tolbutamide was synthesized from p-toluene and submitted.

<b>Name of Compound</b>	<b>Tolbutamide</b>
Theoretical yield	.....gm
Practical yield	.....gm
% Practical yield	.....%
Melting point	.....°C