## **Quadrant II – Transcript and Related Materials**

Programme: T. Y. B. Sc.

**Subject:** Chemistry

Paper Code: CHC 107

Paper Title: Organic Chemistry

Unit: 2- Alkaloids

Module Name: Zeisel's Method and Herzig-Meyer's Method

Module No: 10

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#### **Methods Used in Structure Determination**

**Zeisel's Method** – Method of Estimation of Methoxy group -OCH<sub>3</sub> **Herzig-Meyer's Method** – Method of Estimation of –NCH<sub>3</sub> group

Important in the field of alkaloid chemistry where such compounds are often encountered

### Zeisel's Method

This method is used to - 1) Detect the presence of methoxy group

2) Determine the number of methoxy group

**Estimated Gravimetrically** 

In this method,

1) A known weight of alkaloid is heated with hydroiodic acid at its boiling point (126 °C) – cleaves methoxy group with the formation of methyl iodide

2) Methyl iodide is absorbed by ethanolic silver nitrate – To obtain the

precipitate of silver iodide

- 3) Precipitated Silver iodide is filtered, dried and weighed
- 4) From the weight of silver iodide, the number of methoxy groups may be calculated The no. of moles of silver iodide indicate the no. of methoxy groups present in an alkaloid

# Papaverine (C<sub>20</sub>H<sub>21</sub>O<sub>4</sub>N)

$$C_{16}H_9N(OCH_3)_4$$
 + 4 HI  $\longrightarrow$   $C_{16}H_9N(OH)_4$  + 4CH<sub>3</sub>I Papaverine

$$4CH_3I + 4 AgNO_3 \rightarrow 4 AgI_{\downarrow} + 4 CH_3NO_3$$

### Herzig-Meyer's Method

This method is used to 1) Detect the presence of methyl groups attached to nitrogen atom

2) Determine the number of methyl groups attached to nitrogen atom

$$\frac{\text{HI}}{150 - 300 \, ^{\circ}\text{C}} \qquad \text{NH} \qquad + \quad \text{CH}_{3}\text{I} \qquad \frac{\text{AgNO}_{3}}{\text{C}_{2}\text{H}_{5}\text{OH}} \qquad \text{Ag I} \downarrow$$

In this method,

1) A known weight of alkaloid is heated with hydroiodic acid at about 150  $\,$  - 300  $\,$   $^{0}$ C - cleaves N-Methyl group with the formation of methyl iodide

$$\frac{\text{HI}}{150 - 300 \, ^{0}\text{C}}$$
  $\frac{\text{NH}}{\text{NH}}$  + CH<sub>3</sub>I

2) The vapours of CH3I formed is passed into alcoholic AgNO3 solution – Precipitate of silver iodide is obtained

- 3) Precipitated silver iodide is filtered, dried and weighed
- 4) From the weight of silver iodide, the number of N-Methyl groups may be calculated The no. of moles of silver iodide indicate the no. of N-Methyl groups present in the alkaloid

If the alkaloid reacts with one mole of methyl iodide to form an N-methyl derivative, it means that a secondary nitrogen atom is present For example, Coniine, C8H17N reacts with one mole of methyl iodide to form an N- methyl derivative, indicating that coniine must contain secondary nitrogen atom

## Coniine

By careful control of temperature both the groups can be estimated separately when present simultaneously in a compound

The methods can also be applied for the determination of -OEt and -NEt groups - But with higher alkyl groups, the method is not effective because of the insufficient volatility of the corresponding alkyl iodide formed