PRACTICAL ORGANIC CHEMISTRY

PURIFICATION METHODS DISTILLATION TECHNIQUES

Tuper: (A) SIMPLE DISTILLATION (B) FRACTIONAL Condittons DISTILLATION (ii) When liquid sample has non-When hip. volatile impurities difference is 10%. tall. When boiling point, difference Examples is 30 K or more. (ii) Crude oil in Examples meteoderies. (ii) Mixture of chloroform Service Street, or (BP = 334K) and Andine IIII Acetone (329 K) (BP - 45790)and Methyl Mischane of Ether alcohol (XXXXX) (b.p. - 308K) & Toluene (b.p. - 384K) (iii) Hexane (342)0; and Toursiers of RRANCI (C) DISTILLATION UNDER REDUCED PRESSURE (Vectores distillation) When liquid boils at higher temperature and it may decompose before b.p. is attained. Di Concentration of sugar juice Example (iii) Recovery of glycerol from spent lye. Dust Cidwoorroit (D) STEAM DISTILLATION $P = P_1 + P_2$ When the substance is Vapour Vapour Vapour imministible with water and priesture priesture pressure steam voletile. 100 and recollect Example : **Oversion** 60 Andline is separated Buttered from water (iiii) Notro Hermene (in) Turpentine oil (iv) Broma Benzene (v) Naghthalene (wt) a-Nitrophenol

LASSAIGNES METHOD (detection of elements)

Element	Sedium	Confirmed test
Nitrogen	Na + C - N △↓ NaCN	(NaCN+FeSO,+NaOH) bed and seed *FeCi,+conc. HCl -+Fe,FelCNi,j, frames the other
Sulphur	2Ne + 5 ∆↓ Ne,S	III Na_S = Na_Fe(CN)_NO() Na_Fe(CN)_NOS Alexa reference UP Na_S-CH_COOH = CH_COOH,Pb Alexa ref. Place
Halogen	Na + X A L NaX	NaX = HNO, - AgNO, 0 White ppt soluble in ag NH, confirms Cl. in Yellow ppt partially soluble in aq NH, confirms Sc. in Yellow ppt insoluble in aq. NH, confirms I.
Nitrogen and sulphur together	Na+C+N+S A NaCNS	As in test for nitrogen, instead of green or hise colour, blood red colouration confirms presence of N and S both.

ESTIMATION OF NITROGEN

QUANTITATIVE ANALYSIS OF ORGANIC COMPOUNDS

Estimation of carbon and hydrogen - Leebig's method

$$C_sH_s + O_{2s} \longrightarrow xCO_s + y/2 H_sO$$

 $\%$ of $C = \frac{12}{44} \times \frac{\text{wt. of } CO_s}{\text{wt. of org. compd}} \times 100$
 $\%$ of $H = \frac{2}{18} \times \frac{\text{wt. of } H_sO}{\text{wt. of org. compd}} \times 100$

Note: This method is suitable for estimation if organic compound contains. C and H only. In case if other elements e.g., N, S, halogens are also present the organic compound will also give their resides which is being absorbed in NO94 and will increase the percentage of carbon and therefore following modification should be made.

Duma's method:

The nitrogen containing organic compound yields nitrogen gas on heating it with copper (II) oxide in the presence of CO₂ gas. The mixture of gases is collected over potassium hydroxide solution in which CO₂ is absorbed and volume of nitrogen gas is defermined.

% of N =
$$\frac{28}{22400}$$
 = $\begin{pmatrix} Vol. of N_s \\ collected \\ st N.T.P. \\ Wt. of organic organical \\ organic description $V$$

Note: This method can be used to estimate nitrogen in all types of organic compounds.

Kjeldahl's method:

In this method nitrogen containing compound is heated with conc. H_iSO₄ in presence of copper sulphate to convert nitrogen into ammonium sulphate which is decomposed with excess of alkali to liberate ammonia. The ammonia evolved is

Moste: This method is simpler and more convenient and is mainly used for finding out the presentage of netrogen in food stuffs, soil, fertilities and various agricultural products. This method carevot be used for compound having nitro groups, see group (-N = N-inand nitrogen in the ring (pyridine, quincile etc.) Since nitrogen in these compounds is not quantitatively gonverted in to ammonium sulphate.

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Roman Wölfel

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