CHAPTER III

Preparation of Ligand
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Preparation of 2, 4, 6 - trihydroxy acetophenone
(Phloracetophenone).

The 2, 4, 6 - trihydroxy acetophenone (phloroacetophenone, for the sake of convenience abbreviated as PAP) was prepared by Ullmann reaction (Dobner-Ullmann reaction), an extension of Ullmann aldehyde reaction. The reaction can be illustrated as follows:

\[
\begin{align*}
\text{Phloroglucinol} + \text{CH}_3\text{CN} \xrightarrow{\text{HCl}, \text{ZnCl}_2, \text{Ether}, 0^\circ C} \text{Ketimine hydrochloride}
\end{align*}
\]

PAP (Phloracetophenone)

In actual preparation 10% by weight of dry phloroglucinol (25.2 gms.), 40% anhydrous acetonitrile (16.6 gms. or 20.9 ml.), 100 ml. of sodium dried ether and 5 gms. of finely powdered and fused zinc chloride (ZnCl₂) were taken and kept in ice salt mixture. The rapid stream of dry HCl gas was passed for two hours through the solution with continuous shaking. The flask containing the material was left
for 24 hours in ice chest and again stream of dry HCl gas was passed for another two hours. The flask was again left in ice chest for 72 hours. A bulky orange-yellow precipitate of kinetin-hydrochloride is formed. The excess ether was removed by decantation and the orange-yellow solid was washed by anhydrous ether. The solid was then transferred to a large round bottom flask, provided with the reflex condenser. The dissolved yellow solution was boiled for about two hours and then allowed to cool. When the cooling is allowed about 5 gm. activated charcoal was also added. The solution is again boiled for five minutes and filtered through a Buchner funnel under suction. The yellowish needle shaped structures of chloroacetophenone (CAP) was obtained (1-3).

REFERENCES TO CHAPTER III

