SEMESTER VI

INORGANIC CHEMISTRY

VI

Inorganic Preparations

Experiment No. 1 (3 & 6 Units)

Tris (Acetylacetonato) Iron (III), $Fe(acac)_3$

Aim: To prepare *tris* (acetylacetonato) iron (III).

Theory: This experiment is based on Green Chemistry Principles. It involves high atom economy, less hazardous chemical synthesis, environmentally clean and safe reaction at room temperature and minimises waste.

Metal acetyl acetonatos are highly efficient catalysts for a wide variety of organic transformations such as polymerisation, hydrogenation, coupling etc. They are used in rubber technology for vulcanization, for extraction and separation of metals, as fungicides, in pigments as colour stabilizers, as carbon scavengers for diesel fuels, as combustion control catalysts for rocket fuels etc.

Tris (acetyl acetonato) iron (III) is prepared directly from $Fe(OH)_3$ and acetyl acetone reagent without the use of any buffer like sodium acetate which contaminates the product.

Requirements: $FeCl_3 \cdot 6H_2O$, 1 N KOH solution, acetyl acetone reagent, buchner funnel, ice bath, fused $CaCl_2$, Whatman filter paper No. 1, vacuum desiccator, distilled water, 100 cm^3 beaker, etc.

Reaction:

$$Fe(OH)_3 + 3CH_3COCH_2COCH_3 \rightarrow Fe(CH_2COCH_2COCH_3)_3 + 3H_2O$$

Procedure:

(1) Dissolve W *g* (*i.e.* 1.5 *g* or the supplied quantity) of $FeCl_3 \cdot 6H_2O$ in 10 cm^3 of distilled water in a 100 cm^3 beaker. (Heat it if necessary).

(2) Add about 30 cm^3 of 1N KOH solution in small volumes to the above $FeCl_3$ solution. Stir the solution after each addition of KOH solution to precipitate $Fe(OH)_3$ completely.

(3) Allow the precipitate to settle down completely. Supernatant solution becomes colourless.

(4) Decant the supernatant colourless solution and wash the precipitate 2-3 times with distilled water using 5-7 cm^3 each time. Decant the solution each time after washing.

(5) Filter the precipitate of $Fe(OH)_3$ through buchner fuunel containing Whatman filter No. 1, wash it twice with distilled water.

(6) Transfer the precipitate to 100 cm^3 beaker and keep it in an icebath for 10 *minutes*.

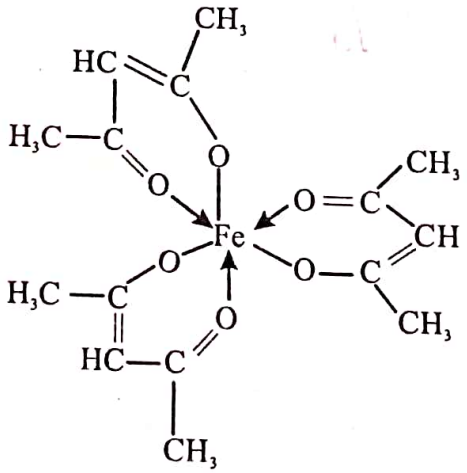
(7) Add about 4-5 cm^3 of distilled acetylacetone reagent to the cold $Fe(OH)_3$ precipitate and stir it well with a glass rod.

(8) Allow the reaction mixture to stand at room temperature for about 30 *minutes*. (Stir it occassionally with the glass rod).

(9) Shiny deep red coloured iron-acetyl acetonato complex gradually forms. Keep the beaker in an icebath for about 15 *minutes*.

(10) Filter the complex through buchner funnel containing Whatman filter paper No. 41 and dry it in vacuum over fused $CaCl_2$ in a desiccator.

(11) Weigh the complex to obtain the yield (x *g*).



Observations and Calculations:

Weight of the complex $Fe(acac)_3 = x$ *g*.

Yield of the complex:

(A) Observed Yield of the complex = x *g*.

(B) Theoretical Yield of the complex:

Now, 270.35 *g* of $FeCl_3 \cdot 6H_2O$ gives 355.85 *g* of $Fe(acac)_3$

$\therefore$ W g _____ = $\frac{355.85 \times W}{270.35}$

= 1.316 W g of $Fe(acac)_3$

(Weight of W g will be supplied).

(C) Percentage yield:

1.316 W g of $Fe(acac)_3$ corresponds to 100% yield

$\therefore$ x g _____ $\frac{100 \times x}{1.316\,W}$

= Z % yield.

Results:

Yield of the complex:

(1) Theoretical yield = 1.316 W g.

(2) Observed yield (x) = _____ g.

(3) Percentage yield (Z) = _____ %. 80%

Experiment No. 2 (6 Units Only)