Sr. No. in Scope NABL / NON NABL

**Flow Chart for Analysis of Sulphur in formulation**

|  |  |
| --- | --- |
| **Date of Analysis**  |  |

|  |  |  |  |
| --- | --- | --- | --- |
| **Sl. No.** | **Step** | **Execution** | **Executed By** |
| 1. | Sample No. |  |  |  |
| 2. | Name of Sample |  |  |  |
| 2.1 | Sample Description |  |  |  |
| **3.** | **Preparation of Standard solutions (0.1 N):** | **R1** | **R2** |  |
| 3.1 | Weigh 24.8 g of Sodium thiosulphate and dissolve in water to get 1 L solution | g | g |  |
| 3.2 | Note the Serial No. in the balance logbook |  |  |  |
| **4.** | **Standardization of Sodium thiosulphate:** |  |  |  |
| 4.1 | Weigh 0.07-0.08 g of KIO3 in a conical flask |  |  |  |
| 4.2 | Note the Serial No. in the balance logbook |  |  |  |
| 4.3 | Add 40-50 ml of water and dissolve |  |  |  |
| 4.4 | Add 2 g of KI |  |  |  |
| 4.5 | Note the Serial No. in the balance logbook |  |  |  |
| 4.6 | Add 2 ml of 5N H2SO4 |  |  |  |
| 4.7 | Titrate Immediately with Sodium thiosulphate solution in burette till pale yellow appears |  |  |  |
| 4.8 | Add 1 ml of starch indicator (Color of solution changes to blue) |  |  |  |
| 4.9 | Then again titrate with Na2S2O3 till it changes to colorless (endpoint) |  |  |  |
| 4.10 | Titre value is |  |  |  |
| **5.** | **Preparation of Iodine solution** |  |  |  |
| 5.1 | Weigh 30-40 g of KI into an Iodine flask |  g |  g |  |
| 5.2 | Note the Serial No. in the balance logbook |  |  |  |
| 5.3 | Add 50-60 ml of water and dissolve |  |  |  |
| 5.4 | Add 13 g of Iodine to 5.3 and dissolve thoroughly |  g |  g |  |
| 5.5 | Note the Serial No. in the balance logbook |  |  |  |
| 5.6 | Transfer into a amber colored bottle (Keep it for overnight) |  |  |  |
| 6. | **Standardization of Iodine solution** |  |  |  |
| 6.1 | Take 25 ml of standardized Iodine solution in a conical flask |  |  |  |
| 6.2 | Add 1 ml of starch solution |  |  |  |
| 6.3 | Titrate against sodium thiosulphate solution, end point will be colorless to blue |  |  |  |
| 6.4 | Titre value is |  |  |  |
| 7. | **Procedure** |  |  |  |
| 7.1 | Weigh accurately 0.1 g of sample (0.125 g of 80% WP) and transfer into a 500 ml GG flat bottom flask  |  |  |  |
| 7.2 | Add 10 ml alcohol to dissolve sulphur (If the formulation is DP) |  |  |  |
| 7.3 | Add 30-40 ml of water |  |  |  |
| 7.4 | Add 2 g Sodium sulphite |  |  |  |
| 7.5 | Add liquid paraffin and few glass beads |  |  |  |
| 7.6 | Fix the flak to the water cooled condenser & keep for reflux for 40 minutes |  |  |  |
| 7.7 | Cool the flask, rinse the condenser with water and remove the flask |  |  |  |
| 7.8 | Add 10 ml of formaldehyde  |  |  |  |
| 7.9 | Add 10 ml of Carbon tetrachloride  |  |  |  |
| 7.10 | Add 10 ml of Acetic acid |  |  |  |
| 7.11 | Titrate immediately with standard Iodine solution using freshly prepared starch solution as indicator |  |  |  |
| 7.12 | End point will be the first appearance of blue color throughout the solution  |  |  |  |
| 7.13 | Follow the same procedure from 7.2 to 7.12 for reagent blank |  |  |  |
| 7.14 | Titre value for sample is |  |  |  |
| 7.15 | Titre value for blank is |  |  |  |

**6. Calculation:**

Wt. of KIO3 X 1000

Normality of Na2S2O3 =---------------------------------------------------

Volume of Na2S2O3 consumed X 35.67

Normality of Na2S2O3 X volume of Na2S2O3 taken

Normality of Iodine = ------------------------------------------------------------------------------

Volume of Iodine consumed

 (mL of Iodine for sample) - (mL of Iodine for blank) X N X 3.206

Active ingredient content, = -------------------------------------------------------------------------------------

% by mass Weight of sample (g)

Where N = Normality of Iodine solution

**Result:**

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **Sl. No.** | **Name of Test** | **Result** | **Unit** | **Method of Analysis** |
| 1. | Active ingredient (Sulfur) |  | % | IS : 6444 - 1979 (Reaffirmed 2002) |
| Reference in daily workbook: |

|  |  |  |
| --- | --- | --- |
| Analyzed by | Name  |  |
| Dated signature |  |
| Checked by | Name  |  |
| Dated signature |  |