Titration with Potassium Bromate

Potassium bromate is primary standard.
Potassium bromate can be used as oxidising agent for inorganic reducers determination. For direct titration with potassium bromate are used specific redox indicators or various azo-dyes, which are destroyed by bromate ion surplus (disappearance colour).

Mixture of potassium bromate and potassium bromide in acidified solution applies for back-titration of organic compounds. This mixture produces bromine, which react with aryl radicals in organic compounds structure. End point in bromatometric back-titration is established iodometric-like – titration with sodium thiosulfate, with starch serving as the indicator.

I. Preparation the primary standard solution (0,1 N potassium bromate)

1. Calculate a primary standard sample (m_{PBT}) for preparation 250 ml of solution:
   \[ m_{PBT} = \frac{N \cdot V \cdot \text{meq}_{PB}}{1000} \]
   \text{M.m. KBrO}_3 = 167,001 \text{ g;}
   \text{mass of potassium bromate equivalent meq}_{PB} = M/6.
2. Weight potassium bromate sample on hand balance with 0,1 g accuracy and put powder into weighting bottle.
3. Weight weighting bottle with potassium bromate on analytical balance.
4. Transfer the potassium bromate into volumetric flask and weight empty weighting bottle on analytical balance. Calculate the potassium bromate sample weight m_{PBP}.
5. Add 40-50 ml of distilled water to volumetric flask, dissolve potassium bromate and establish exact volume of solution with water.
6. Calculate precision normal concentration of prepared standard solution of potassium bromate: \[ N_{PB} = \frac{m \cdot 1000}{V \cdot \text{meq}_{BP}} \]
7. Calculate the correction factor for prepared 0,1 N potassium bromate solution: \[ \text{CF}_{PB} = \frac{m_{PBP}}{m_{PBT}} \]

II. Sodium Salicylate Determination

In acid solution sodium salicylate transfer to phenol:

\[
\begin{align*}
\text{OH} & \quad \text{COONa} \\
\text{2} & \quad + \text{H}_2\text{SO}_4 \rightarrow \text{OH} & \quad \text{COOH} \\
& \quad \text{COOH} \\
\text{2} & \quad \text{Na}_2\text{SO}_4 \\
\end{align*}
\]

\[
\begin{align*}
\text{2} & \quad \text{OH} \quad \text{COOH} \\
\text{H}_2\text{SO}_4 \rightarrow \text{2} & \quad \text{OH} \\
& \quad \text{2CO}_2 + \text{H}_2\text{O}
\end{align*}
\]

Formed phenol reacts with bromine that produced by bromate-bromide mixture:
KBrO₃ + 5KBr + 3H₂SO₄ = 3Br₂ + 3K₂SO₄ = 3H₂O

Bromine surplus is titrated with sodium thiosulfate after potassium iodide addition:

\[ \text{Br}_2 + 2KI = 2KBr = I_2 \]

\[ I_2 + 2\text{Na}_2\text{S}_2\text{O}_3 \rightarrow 2\text{NaI} + \text{Na}_2\text{S}_4\text{O}_6 \]

1. Load a burette with 0.1 N standard sodium thiosulfate solution.
2. To conical flask pour in 10 ml aliquot of sample (V_S), add pipette using 10 ml of prepared 0.1 N potassium bromate standard solution (V_PB), add 10 ml of 10% potassium bromide solution, and add 10 ml of 1 M H₂SO₄ solution.
3. Cover the flask with a stopper and put into dark place at 10 min.
4. Add 10 ml of 10% potassium iodide solution and titrate the sample with thiosulfate until the solution becomes pale yellow. Introduce 5 drops of starch indicator, and titrate with constant stirring to the disappearance of the blue colour.
5. Read the burette mark (V_ST).
6. Repeat titration also two times. Calculate the median volumes of used sodium thiosulfate standard solution.
7. Calculate sodium salicylate percentage in sample:

\[ x_{\text{SodSal}} = \frac{(V_{PB} \cdot N_{PB} - V_{ST} \cdot N_{ST}) \cdot \text{meq}_{\text{SodSal}}}{V_S} \cdot 100\% \]

M.m. Sodium salicylate = 160.11; \text{meq}_{\text{SodSal}} = M/6.