Standard Preparation
To prepare a 0.1 eq/l (or 0.1 mol/) sodium thiosulphate solution, dissolve 24.8181 g of Na₂S₂O₃, 5H₂O in 500 ml of freshly distilled water (or freshly boiled and cooled deionised water) and 2 or 3 drops of CHCl₃ (or also 0.4 g of NaOH) and complete to 1000 ml using a volumetric flask.

Wait for one day and filter the solution if necessary (precipitation of sulphur can occur).
Stock the solution in a brown glass flask.

Look at the solution from time to time and filter again if necessary.

Solutions with a concentration below 0.01N (or 0.01M) are not stable.

Na₂S₂O₃, 5H₂O has a molecular weight corresponding to 248.181 g/mol.

To calibrate a thiosulphate solution, use potassium iodate KIO₃ as standard. It reacts with potassium iodide KI giving iodine I₂ according to the reaction:

\[
\text{IO}_3^- + 5\text{I}^- + 6\text{H}^+ \rightarrow 3\text{I}_2 + 3\text{H}_2\text{O}
\]

The thiosulphate ion reacts with I₂ according to the reaction:

\[
3\text{I}_2 + 6\text{S}_2\text{O}_3^{2-} \rightarrow 6\text{I}^- + 3\text{S}_4\text{O}_6^{2-}
\]

The molecular weight of KIO₃ is 214.0 g/mol. A 0.1 eq/l iodate solution, according to the above-mentioned reactions (1 ion IO₃ corresponds to 3I₂), contains 1/60 mol/l of KIO₃.

Weigh exactly 3.5667 g of potassium iodate (214/60) and dilute in 1000 ml of distilled water using a volumetric flask.

Electrode and reagents
For this reaction, the best titration procedure is a preset endpoint titration using imposed current potentiometry with a double platinum wire electrode.

The curve shape is very sharp around the equivalence point.

M231Pt2 Metal Electrode, double platinum wire (part no. E32M001) with adapter part no. A94P801 (BNC - 2xbanana) or M241Pt2-8 Metal Electrode, double platinum wire with BNC plug (part no. E32M002).

Distilled water
KI, solid state
HCl, concentrated

End Point titration settings

<table>
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<tr>
<th>Parameter</th>
<th>Value</th>
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</thead>
<tbody>
<tr>
<td>Burette volume</td>
<td>25 ml</td>
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<tr>
<td>Stirring speed</td>
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<td>Working mode</td>
<td>mV (with i&gt;0)</td>
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<tr>
<td>Imposed current</td>
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<tr>
<td>Predose</td>
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<td>Standard conc.</td>
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<tr>
<td>Result</td>
<td>eq/l</td>
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<tr>
<td>Titration</td>
<td>Increasing mV</td>
</tr>
</tbody>
</table>

Procedure

Prepare the titration system with a 25 ml burette and 0.1 eq/l sodium thiosulphate as titrant.

Connect the M231Pt2 electrode via the adapter or the M241Pt2-8 electrode directly.

Pipette 20 ml of the KIO₃ standard solution, add 30 ml of distilled water, about 1 g of solid KI. Wait for dissolution, then slowly add 5 ml of concentrated HCl.

Dip electrode and delivery tip in the solution.
Start method by pressing the RUN key.

Results

The result is expressed as eq/l concentration and based on the following formula:

\[
\text{Vol(S}_2\text{O}_3^2-) \times \text{C(S}_2\text{O}_3^2-) = \text{Vol(IO}_3^-) \times \text{C(IO}_3^-) 
\]

With C(IO₃⁻) expressed in eq/l

The calibration result can be accepted if 5 determinations give a result with a relative standard deviation below 0.5%.
Notes

1) The titration curve shows a characteristic shape with a very flat first part and a sharp second part. To save time, it is therefore advisable to use a predose and a not too high burette speed to avoid “over-titrating”.

2) The application note uses a 25 ml cylinder capacity. If you use a 10 ml cylinder for the burette, pipette 5 ml of standard, with a 5 ml burette pipette, also 5 ml of standard and modify the method as follows:

- Predose: 2 ml
- Maximum volume: 8 ml

3) Instead of an aqueous solution of KIO₃, you can use solid state KIO₃. In this case and for the application note conditions, exactly weigh approximately 60 mg of KIO₃ (for 20 ml of sodium thiosulphate solution) and in the STANDARD screen ENTER:

- Standard unit: mg
- Standard amount: xx.x
- Concentration unit: %
- Concentration: 100
  (or purity of the standard)
- Molecular weight: 214

And in the RESULT screen ENTER:

- Result: eq/l
- Coefficients: 1 standard and 6 titrants