



Calibration of a Thiosulphate Solution

Standard Preparation

To prepare a 0.1 eq/l (or 0.1 mol/l) 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



The thiosulphate ion reacts with I₂ according to the reaction



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 pre-set end point 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

| | |
|-----------------------|------------------|
| Burette volume: | 25 ml |
| Stirring speed: | 400 rpm |
| Working mode: | mV (with i>0) |
| Imposed current: | 10 µA (AC or DC) |
| Predose: | 15 ml (see note) |
| Number of end points: | 1 |
| End point: | 200 mV |
| Stirring delay: | 60 seconds |
| Minimum speed: | 0.1 ml/min |
| Maximum speed: | 2.5 ml/min |
| Proportional band: | 200 mV |
| End point delay: | 5 seconds |
| Sample unit: | ml |
| Standard amount: | 20 |
| Standard conc.: | 0.1 eq/l |
| Result: | eq/l |
| Titration: | Increasing mV |

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:

$$\text{Vol}(\text{S}_2\text{O}_3) * \text{C}(\text{S}_2\text{O}_3) = \text{Vol}(\text{IO}_3^-) * \text{C}(\text{IO}_3^-)$$

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 KIO_3 , you can use solid state KIO_3 . In this case and for the application note conditions, exactly weigh approximately 60 mg of KIO_3 (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