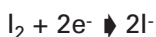




Calibration of an Iodine Solution

Reagent preparation

To prepare 0.1 eq/l iodine solution which can be used in a very wide pH range, we can dissolve iodine (I₂) in a solution of potassium iodide. Iodine in redox reaction reacts according to



A 0.1 eq/l iodine solution contains 0.05 mol/l or 1/20 mol/l of iodine I₂ has a molecular weight corresponding to 235.8 g/mol

To prepare a 0.1 eq/l iodine solution, dissolve 40 g of potassium iodide in 50 ml of distilled water then add 12.69 g (235.8/20) of analytical grade iodine, wait for it to dissolve completely and complete to 1.000 ml with a volumetric flask.

Store the solution in a brown glass bottle.

Standard preparation

To calibrate iodine solution, use As₂O₃ as standard. It has a molecular weight of 169.87.

The redox reaction between iodine and As(III) is



A 0.1 eq/l As III solution contains 1/40 As₂O₃ mol/l.

To prepare the 0.1eq/l As III standard solution.

Weigh 4.2467 (169.87/40) g of pure As₂O₃ and dissolve it in about 20 ml of NaOH 10M. You can gently heat the solution to dissolve it faster.

Add about 200 ml of pure water and H₂SO₄ 1M until the pH reaches 8.00.

Leave the solution to reach room temperature.

Complete to exactly 1000 ml with 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 equivalent point.

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

Distilled water

Na₂CO₃ saturated solution in water or pH 10.00 buffer solution (part no. S11M014)

End Point titration settings

Burette volume:	25 ml
Stirring speed:	400 rpm
Working mode:	mV with i > 0
Imposed current:	5µA AC or DC
Number of end points:	1
End point:	50 mV
Stirring delay:	60 seconds
Minimum speed:	0.1 ml/min
Maximum speed:	6.0 ml/min
Proportional band:	500 mV
End point delay:	10 seconds
Direction:	Decreasing mV
Sample unit:	ml
Standard amount:	20
Standard conc.:	0.1 eq/l
Result:	eq/l

Procedure

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

Connect the M231Pt2 electrode via the adapter part no. A94P801.

In a beaker, pipette 60 ml of pH 10.00 buffer solution or the same quantity of saturated Na₂CO₃ solution.

Pipette exactly 20 ml of As₂O₃ 0.1 eq/l standard.

Start method by pressing the RUN key.

Results

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

$$Vol(As_2O_3) * C(As_2O_3) = Vol(I_2) * C(I_2)$$

The calibration result can be accepted if 5 determinations give a result with a relative standard deviation of less than 0.5%.

Notes

a) Using a solution as standard, it is best to use a standard concentration close to the titrant concentration. This allows closed volumes for titrant and standard.

For the best result accuracy, pipette a standard volume corresponding to a delivered titrant volume greater than 50% of the used burette cylinder.

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

 Predose: 2 ml
 Maximum volume: 8 ml

c) If you calibrate an iodine solution using DC or AC imposed current you must use this titrant using the same imposed current procedure.