DENVER INSTRUMENT



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 (I_2) in a solution of potassium iodide. Iodine in redox reaction reacts according to

$$I_2 + 2e^- \quad 2I^-$$

A.) 0.1 eq/l iodine solution contains 0.05 mol/l or 1/20 mol/l of iodine

I_2 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 Perparation

To calibrate iodine solution, use As_2O_3 as standard. It has a molecular weight of 169.87.

The redox reaction between iodine and As(III) is

$$1(As_2O3) + 2(I_2) + 5H2O \blacktriangleright 4I^- + 2AsO_4^{---} + 10H^+$$

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 As_2O_3 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 buret 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 Na_2CO_3 solution.

Pipette exactly 20 ml of As_2O_3 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(As2O3) * C(As2O3) = Vol(I2) * C(I2)

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 buret, 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.