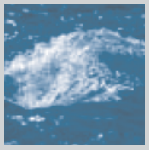
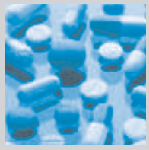
DENVER INSTRUMENT





Hyrdogen Peroxide Determination (by manganimetric titration)

Introduction

Discovered in 1818 by a French chemist, L.J Thenard, Hydrogen peroxide (H_2O_2) is a common oxidising agent used in papermaking as well as in the textile or pharmaceutical industry. It is used for disinfecting and colour removal. The dismutation reaction of hydrogen peroxide corresponds to the following equations:

$$H_2O_2 + 2H^+ + 2\,e \rightarrow 2H_2O$$

$$H_2O_2 \rightarrow O_2 + 2H^+ + 2\,e$$

The global reaction corresponds to:
$2H_2O_2 \rightarrow 2\,H_2O + O_2$ (1)

Principle

H_2O_2 determination generally uses a redox titration using potassium permanganate as oxidising agent according to the following reaction:

$$5H_2O_2 + 2MnO_4^- + 6H^+ \rightarrow 5O_2 + 2Mn_2+ + 8H_2O$$

As indicated, the reaction takes place in acidic media (generally in sulphuric media).
The H_2O_2 titration is run using potentiometry with a combined platinum/reference electrode. Results are generally expressed as a % of H_2O_2 or in volumes of oxygen by volume of solution. The titre as a % indicates the weight of H_2O_2 for 100 g of solution. The titre expressed as the volume of oxygen (in litres) freed by 1 litre of solution.

According to the reaction (1) it is possible to write:

Titre (in volumes) = (10 * titre (in %) / d) * 11.2 / 34

34 = molar weight of H_2O_2

11.2 = volume of ½ mole of O_2 under normal pressure and temperature conditions

d = H_2O_2 density

If density is supposed equal to 1:

Titre (in volumes) = Titre (in %) * 3.294

Electrode and reagents

MC3051Pt-9 Metal Electrode combined, platinum (part no. E31M003) with CL114 cable (part no. A94L114)

25% v/v H_2SO_4 solution in distilled water

Dilute 250 ml of concentrated sulphuric acid in 750 ml of distilled water. This operation is very exothermic, so perform the dilution very slowly and respect laboratory safety regulations. Let the solution cool to room temperature.n This solution is approximately 9N or 4.5M.

$KMnO_4$ solution (M/50 or 0.02M; note this solution is N/10). Using a volumetric flask, dilute 3.160 g of potassium permanganate in 500 ml of distilled water, add 10 ml of 25% sulphuric acid solution, leave to cool to room temperature and complete to 1000 ml with distilled water.

Store in a brown flask.

Note that this solution is commercially available.

Distilled water

Inflection Detection settings

Burette volume:	25 ml **(see Working range)**
Stirring speed:	650 rpm **(see Stirring)**
Working mode:	mV
Start timer:	15 s
Maximum volume:	25 ml
Stop point:	1250 mV
Direction:	Increasing mV
Predose:	10.0 ml **(see Predose note)**
Minimum speed:	0.2 ml/min
Maximum speed:	4.00 ml/min
Smoothing parameter:	5
Minimum ordinate:	750 mV
Maximum ordinate:	1100 mV
Stop at last IP:	YES
Sample unit:	g
Sample amount:	11 (see Sample amount note)
Dilution:	YES **(see Working range)**
Final dilution amount:	1000 ml
Aliquot:	10 ml
Result number:	1
Result:	%
Molar weight:	34 g/mol
Reaction:	5Smp + 2Titr
Equation number:	1
Equation unit:	conc. (volumes)
Equation:	R1*3.294 **(see Equation note)**

Procedure

Dilute the sample according to the dilution factor described under Working range.

In the titration beaker pipette the recommended sample aliquot (see Working range).

Add a sufficient quantity of distilled water and 10 ml of the 25% v/v sulphuric acid solution.

Immerse the combined platinum/ reference electrode.

Run the titration.

Results

Results can be expressed in two different units

R(%) = (5 * Ctitr * Vtitr / 2 * Vsmp) * (34/10)

5 and 2 = Reaction coefficients

Ctitr = Titrant concentration

Vtitr = Used titrant volume

Vsmp = Used sample volume

34 = Molar weight of H_2O_2

10 = factor to express result as a %

R(volumes) = R(%) * 3.294 / d

d = Sample density (in g/ml or kg/l)

Experimental results on commercial hydrogen peroxide

4 determinations

Result as a %

Mean:	29.75%
Standard deviation:	0.022%

Result in volumes

Mean:	89.09 vol.
Standard deviation:	0.066 vol.

Working range

Using a 25 ml burette and according to the expected concentration of the hydrogen peroxide, refer to the following table to choose the dilution factor and sample aliquot for the titration:

% w/w	Titre 9(vol.)*	Molarity	F. dilution	Aliquot
6	19.8	1.8	10	5 ml
30	99	8.8	100	10 ml
40	130	11.8	200	10 ml

(*) Approximate value with d = 1

Notes

Sample amount

10 ml are pipetted and weighed.

Stirring speed

Use a sufficiently high stirring speed to allow the oxygen bubbles generated during the reaction to leave the solution.

Procedure

Ensure you add sufficient acid solution to obtain a well-shaped titration curve.

Predose

The predose function can be used to save time. Modify this volume according the expected volume of titrant

Equation

If H2O2 density is known modify to the equation as follows

Equation = R1*3.924/d

d = H_2O_2 density

Curve