# CHEMICAL INDUSTRIES
Introduction

The complete applications package

At Radiometer Analytical, we put applications first. When you order one of our new generation of titration workstations with a dedicated application package, you have everything you need to get started right away: electrodes, specific accessories, standards, maintenance solutions and, of course, methods and application notes.

Application booklets

The term "Chemical industry" is extremely vast. This booklet contains a few specific applications together with calibration procedures for the corresponding titrants and electrodes but other closely related applications can be found in our other booklets. Certain modifications may be needed to take into account specific regulations or standards in force in certain countries, in particular regarding results presentation.

In addition to dedicated application booklets, Radiometer Analytical also produces technique-based applications. Ask your local representative for the following booklets:
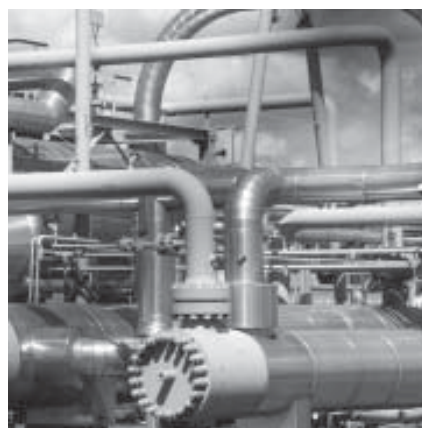
Dedicated	Part No.
Food and beverage analysis	D41T004
Plating bath analysis	D41T005
Water and environmental analysis	D41T006

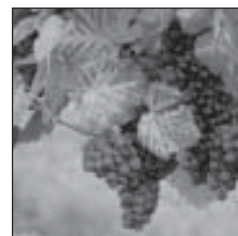| Technique	Part No.
Acid-base titrations	D41T009
Precipitation titrations	D41T010
Complexometric titrations	D41T011
Redox titrations	D41T012

Our Applications Laboratory is continually developing new applications.

For the latest updates visit us at www.titration.com.

CHEMICAL INDUSTRIES

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# Inflection Point Titration Manager Operational Qualification

Introduction

The operational qualification procedure demonstrates that an instrument runs according to the operational specifications in the selected environment. For a titration manager using automatic inflection point determination, this operation can consist of two steps:

- Calibration and checking of a pH measurement electrode system,

- Titration of anhydrous sodium carbonate standard using a commercial standard solution of HCl.

Principle

The reaction has 2 steps corresponding to 2 different inflection points:

$$Na_2CO_3 + HCl \rightarrow NaHCO_3 + NaCl$$

$$NaHCO_3 + HCl \rightarrow CO_2 + H_2O + NaCl$$
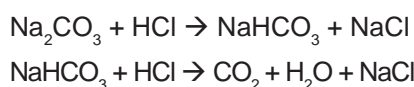
The operational qualification takes into account the second equivalence point (close to pH 4.00)

corresponding to the complete neutralisation of sodium carbonate.

The Titration Manager settings allow the complete titration curve to be seen.

Electrode and reagents

pHC2011-8 Combined pH Electrode (part no. E16M317)

T201 Temperature Sensor (part no. E51M001)

IUPAC Series pH standards

pH 4.005 (part no. S11M002)

pH 7.000 (part no. S11M004)

pH 10.012 (part no. S11M007)

100 g bottle of Merck "certified" sodium carbonate (part no. 1.06394.0100) or 500 g bottle of "pro analysis" Merck sodium carbonate (part no. 1.06392.0500)

1 l of "ready to use" Merck HCl 0.1 mol/l (part no. 1.09060.1000)

Distilled water

Continuous IP titration settings

Electrode
pH:	pHC2011-8
Calibration request:	YES
Number of cycles:	2
Number of buffers:	3
Measurement:	Stirring
Temperature:	probe

Calibration parameters
Stability:	15 mpH/min
Acceptation time:	2.00 min
Max. stab. Time:	5.00 min
Acceptation criteria:	YES
Iso. pH:	6.65 pH

Calibration solutions
1:	4.005 (IUPAC)
2:	7.000 (IUPAC)
3:	10.012 (IUPAC)
Min. zero pH:	5.8 pH
Max. zero pH:	7.5 pH
Min. sensitivity:	95%
Max. sensitivity:	103%

Titrant
ID:	HCl
Unit:	M
Titre:	Entered

![Radiometer analytical]

Method

Stirring speed:	550 rpm
Predose:	0 ml
Start timer:	45 sec
Max. volume:	**(see notes)**
Stop point:	2.000 pH
Smoothing parameter:	8
Number of IP:	2
Min. speed:	0.2 ml/min
Max. speed:	5.00 ml/min
Direction:	decreasing pH
Inflection 1	
Min. ordinate:	7.00 pH
Max. ordinate:	10.00 pH
Inflection 2	
Min. ordinate:	2.80 pH
Max. ordinate:	6.00 pH
Sample unit:	mg
Sample amount:	**(see notes)**
Results	
Results by:	cumulate
Number of result:	1
Acceptation criteria:	YES
Result 1	
Result unit:	%
Molar weight:	105.99
Reaction:	1 smp + 2 titr
Calculate with IP:	2
Minimum value:	99
Maximum value:	101

Procedure

Electrode calibration and checking

If the electrode is new, condition it by immersion in distilled water for at least 1 hour.
Connect the pHC2011-8 electrode and the T201 Temperature Sensor Using the above-mentioned settings, RUN an electrode calibration with 2 or more cycles.
At the end of the last cycle, calibration results should be accepted Ensure that the temperature of the standards does not differ by more than 2°C.

Operation qualification in continuous IP

Fit the burette of the titration manager with HCl 0.1 M as titrant and install the titrant

Preparation of Na_2CO_3

As indicated, dry approximately 5 g of anhydrous sodium carbonate in an oven for 4 hours at 250°C. Let it cool to room temperature in a desiccator with P_2O_5 or another humidity adsorber.

Notes

To determine the necessary amount of sodium carbonate

With a 25 ml burette capacity

Weigh exactly approximately 85-90 mg of Na_2CO_3

This weight corresponds to

1.6-1.8 meq or 16-18 ml of HCl 0.1M. Use a maximum volume close to 20/22 ml

With a 10 ml burette capacity

Weigh exactly approximately 40 mg of Na_2CO_3. Use a maximum volume of 10 ml

With a 5 ml burette capacity
Weigh exactly approximately 20 mg of Na_2CO_3. Use a maximum volume of 5 ml

Dissolve the weighed sodium carbonate quantitatively in the titration beaker with 50 to 80 ml of freshly distilled water.

Immerse the electrode and the delivery tip in the solution.

Using the above-mentioned settings, run a titration on 3 different tests or replicates. Results should be accepted between 99 and 101%.

Calculations are programmed to give a result according to the delivered titrant volume at the inflection point situated in the acceptance range 2.80-6.00 pH

Dynamic IP Titration settings

This application note can be used with incremental addition of titrant (Dynamic IP)

Dynamic IP

Speed:	5.00 ml/min
Dynamic dose:	30
Maximum dose:	0.3 ml
Stability:	100 mpH/min
Acceptation:	10 s

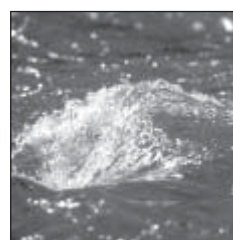These settings were tested with a 10 ml burette. The others settings are unchanged.

End Point Titration Manager
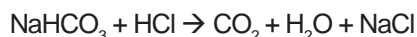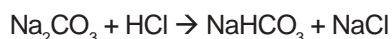# Operational Qualification

Introduction

The operational qualification procedure demonstrates that an instrument runs according to the operational specifications in the selected environment. For an end point titration manager, this operation can consist of two steps:
- Calibration and checking of a pH measurement electrode system
- Titration of anhydrous sodium carbonate standard using a commercial standard solution of HCl.

Principle

The reaction has 2 steps

$$Na_2CO_3 + HCl \rightarrow NaHCO_3 + NaCl$$

$$NaHCO_3 + HCl \rightarrow CO_2 + H_2O + NaCl$$

corresponding to 2 different end points.
The operational qualification takes into account the second equivalence point (pH 3.90) corresponding to the complete neutralisation of sodium carbonate.

Electrode and reagents

pHC2011-8 Combined pH Electrode (part no. E16M317)

T201 Temperature Sensor (part no. E51M001)

IUPAC Series pH standards pH 4.005 (part no. S11M002), pH 7.000 (part no. S11M004) and pH 10.012 (part no. S11M007)

100 g bottle of Merck "certified" sodium carbonate (part no. 1.06394.0100) or 500 g bottle of "pro analysis" Merck sodium carbonate (part no. 1.06392.0500)

1 l of "ready to use" Merck HCl 0.1 mol/l (part no. 1.09060.1000)

Distilled water

End Point titration settings

Electrode
pH:	pHC2011-8
Calibration request:	YES
Number of cycles:	2
Number of buffers:	3
Measurement:	Stirring
Temperature:	probe

Calibration parameters
Stability:	15 mpH/min
Acceptation time:	2.00 min
Max. stab. Time:	5.00 min
Acceptation criteria:	YES
Iso. pH:	6.65 pH

Calibration solutions
1:	4.005 (IUPAC)
2:	7.000 (IUPAC)
3:	10.012 (IUPAC)
Min. zero pH:	5.8 pH
Max. zero pH:	7.5 pH
Min. sensitivity:	95%
Max. sensitivity:	103%

Titrant
ID:	HCl
Unit:	M
Titre:	Entered

Method
Predose:	see below
Start timer:	45 sec
Max. volume:	see below
Number of EP:	1
Min. speed:	0.2 ml/min
Max. speed:	5.00 ml/min
Direction:	decreasing pH
End point:	3.90 pH
Delay:	10 sec
Proportional band:	3.00 pH

Results

Sample unit:	mg
Number of result:	1
Acceptation criteria:	YES
Result unit:	%
Molar weight:	105.99
Reaction:	1 smp + 2 titr
Minimum value:	99
Maximum value:	101

Procedure

Electrode calibration and checking

If the electrode is new, condition it by immersion in distilled water for at least 1 hour

Connect the pHC2011-8 combined pH electrode and the T201 Temperature Sensor

Using the above-mentioned settings, RUN an electrode calibration with 2 or more cycles

At the end of the last cycle, calibration results should be accepted

Ensure that the temperature of the standards does not differ by more than 2°C.

End point titration operation qualification

Fit the titration manager with HCl 0.1 M as titrant and install the titrant.

Preparation of Na_2CO_3

As indicated, dry approximately 5 g of anhydrous sodium carbonate in an oven for 4 hours at 250°C. Let it cool to room temperature in a desiccator with P_2O_5 or another humidity adsorber.

End point titration

To determine the necessary amount of sodium carbonate

With a 25 ml burette capacity

Weigh exactly 85-90 mg of Na_2CO_3

This weight corresponds to 1.6-1.8 meq or 16-18 ml of HCl 0.1M. Use a predose corresponding to 10 ml and a maximum volume of 25 ml

With a 10 ml burette capacity

Use the same settings as the 25 ml burette capacity

With a 5 ml burette capacity

Weigh exactly 40-45 mg of Na_2CO_3

Use a maximum volume of 10 ml

No predose.

Dissolve the weighed sodium carbonate quantitatively in the titration beaker with 50 to 80 ml of freshly distilled water.

Immerse the electrode and the delivery tip in the solution.

Using the above-mentioned settings, run a titration with 3 different samples. Results should be accepted between 99 and 101%.

Calibration of an Acidic Solution
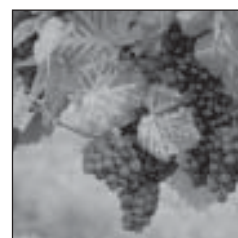
Reagent preparation

In aqueous media, 2 acids are mainly used as titrants: HCl (concentrated commercial solution is around 12M) or H_2SO_4 (18M or 36N). It is also possible to use HNO_3 (concentrated commercial solution is nearly 12.8M). To prepare 1000 ml of 0.1 eq/l strong acid solution, dilute X ml of concentrated acid (see table) in 200 ml of distilled water and, by means of a volumetric flask, dilute to 1000 ml. **CAUTION:** These operations are highly exothermic. Observe laboratory safety regulations.

Acid	Conc. % w/w	Density g/l	Conc. M (mol/l)	X = Vol (ml)
HNO_3	60%	1.35	12.8	7.8
H_2SO_4	96%	1.83	18	2.8
HCl	37%	1.18	12	8.3

Standard preparation

To calibrate acid solution, use analytical grade $Na_2B_4O_7,10\ H_2O$ as standard (molecular weight 381.4 g/mol); it reacts with H^+ ions according to

$$B_4O_7^{--} + 2H^+ + H_2O \rightarrow 4HBO_2$$
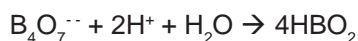
A 0.1 eq/l borax solution contains 0.05 mol/l (or 1/20 mol/l) of borax. To prepare 1000 ml of 0.1 eq/l of standard, weigh exactly 19.070 g of analytical grade $Na_2B_4O_7,10\ H_2O$ and dilute to 1000 ml with a volumetric flask.

A 0.1 eq/l solution can be stored for 1 month.

Electrode and reagents

pHC2401-8 Combined pH Electrode (part no. E16M336)

Freshly distilled water

0.1 eq/l borax standard solution

IUPAC Series pH standards pH 4.005 (part no. S11M002) and pH 10.012 (part no. S11M007)

End Point titration settings

Burette volume:	25 ml
Stirring speed:	400 rpm
Working mode:	pH
Number of end points:	1
End point:	5.10 pH
Stirring delay:	10 seconds
Minimum speed:	0.2 ml/min
Maximum speed:	6.0 ml/min
Proportional band:	3.0 pH
End point delay:	5 seconds
Direction:	Decreasing pH
Sample unit:	ml
Standard amount:	20
Standard conc.:	0.1 eq/l
Result:	eq/l

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Procedure

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

Calibrate the pHC2401-8 electrode using IUPAC standards.

Do not forget to fill the reagent bottle absorption chamber with absorbent.

Pipette exactly 20 ml of borax 0.1 eq/l.

Complete to 100 ml with distilled water.

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:

Vol(acid sol) * C(acid sol) = Vol(borax sol) * C(borax sol)

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) It is possible to calibrate an acid titrant by weighing an amount of borax.
With a 25 ml burette cylinder capacity.
Exactly weigh approximately 380 mg of borax.

In the STANDARD screen ENTER
Standard unit: mg
Standard amount: xx.x
Concentration unit: %
Concentration: 100
          (or purity of the standard)
Molecular weight: 381.4

And in the RESULT screen ENTER
Result: eq/l

For HCl or HNO_3 (result in eq/l or mol/l)
Coefficients: 1 Standard + 2 Titrant

For H_2SO_4 (result in eq/l)
Coefficients: 2 Standard + 2 Titrant

For H_2SO_4 (result in mol/l)
Coefficients: 1 Standard + 1 Titrant

Calibration of an Alkaline (NaOH or KOH) Solution

Reagent preparation

There are several ways to prepare free carbonate NaOH or KOH solution.
To prepare 0.1 eq/l NaOH or KOH solution the easiest way is to:

Take a new bottle of NaOH or KOH pellets and quickly weigh 4.00 g of NaOH or 5.60 g of KOH

(NaOH has a molecular weight of 40 g/mol and KOH 56 g/mol)

Using a conical flask, dissolve the pellets in 200 ml of hot (40°C approx.) freshly boiled distilled water, cover the flask with plastic film and leave to cool to room temperature.

Using a volumetric flask, quickly complete to 1000 ml with the same freshly boiled distilled water.

For long storage, use a polythene flask.

Standard preparation

To calibrate NaOH solution, use oxalic acid $H_2C_2O_4$, $2H_2O$ as standard (molecular weight 126.0 g/mol). As in aqueous media, the 2 acid functions are titrated together; **a 0.1 eq/l oxalic solution contains 0.05 mol/l (or 1/20 mol/l) of oxalic acid.**

To prepare 1000 ml of 0.1 eq/l of standard.
Weigh exactly 6.300 g (126.0/20) of oxalic acid. Using a volumetric flask, dissolve to 1000 ml with freshly boiled distilled water.

Electrode and reagents

pHC2401-8 Combined pH Electrode (part no. E16M336)

Freshly boiled distilled water

0.1 eq/l oxalic acid standard solution

IUPAC Series pH standards
pH 4.005 (part no. S11M002) and
pH 10.012 (part no. S11M007)

End Point titration settings

Burette volume:	25 ml
Stirring speed:	400 rpm
Working mode:	pH
Number of end points:	1
End point:	8.55 pH
Stirring delay:	10 seconds
Minimum speed:	0.2 ml/min
Maximum speed:	6.0 ml/min
Proportional band:	3.0 pH
Direction:	Increasing pH
End point delay:	5 seconds
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 NaOH solution as titrant.

Calibrate the pHC2401-8 electrode using IUPAC standards.

Do not forget to fill the reagent bottle absorption chamber with absorbent CO_2.

Pipette exactly 20 ml of oxalic acid 0.1 eq/l.

Complete to 100 ml with distilled water.

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

Vol(NaOH sol) * C(NaOH sol) = Vol(oxalic acid sol) * C(oxalic acid sol)

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) It is possible to calibrate a basic titrant by weighing an amount of oxalic acid.

With a 25 ml burette cylinder capacity

Exactly weigh approximately 120 mg of oxalic acid

In the STANDARD screen ENTER
Standard unit: mg
Standard amount: xx.x
Concentration unit: %
Concentration: 100
 (or purity of the standard)
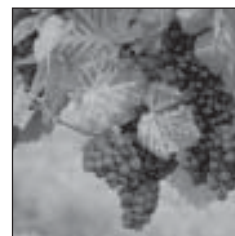Molecular weight: 126.0

And in the RESULT screen ENTER

Result: eq/l
Coefficients: 1 Standard + 2 Titrant

Note that for NaOH and KOH, concentration in eq/l is the same as in mol/l.

d) For alkaline solutions with higher concentration (1M or 1N for example), use the same procedure but take 40 g of NaOH, or 56 g of KOH, for 1000 ml of solution and calibrate the solution by exactly weighing approximately 1200 mg of oxalic acid.

Calibration of a Ferrous Solution Fe(II)

Reagent preparation

The most common Fe(II) salt is Mohr's salt: $(NH_4)2Fe(SO_4)_2$, $6H_2O$ (molecular weight 392.14 g/mol)
To prepare a 0.1 eq/l 1000 ml solution, proceed as follows:
Weigh 39.21 g of Mohr's salt and dissolve in 500 ml of distilled water
Add 100 ml of sulphuric acid approximately 1N
Using a volumetric flask, dilute to 1000 ml with distilled water

Standard preparation

One of the most common standards in redox analysis is potassium dichromate $K_2Cr_2O_7$ (molecular weight 294.19 g/mol).
Potassium dichromate solutions are very stable.
The oxidation power of potassium dichromate is based on the reaction

$$Cr_2O_7^{--} + 14H^+ + 6 e- \rightarrow 2Cr^{+++} + 7H_2O$$
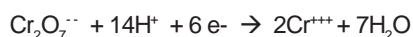
A 0.1 eq/l potassium dichromate solution contains 1/60 mole of dichromate.
To prepare 1000 ml of 0.1 eq/l $K_2Cr_2O_7$ proceed as follows:

Dry the potassium dichromate (analytical grade) for 2 hours at 120°C and leave it to cool at room temperature in a desiccator.

Weigh exactly 4.90317 (294.19/60) g and dissolve with freshly distilled water to exactly 1000 ml using a volumetric flask.

Electrode and reagents

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

50% H_2SO_4 in water

Dilute the same volume of concentrated sulphuric acid in a given volume of distilled water. As the operation is very exothermic, make this dilution very slowly according to laboratory safety regulations.

Freshly distilled water

$K_2Cr_2O_7$ 0.1 eq/l standard solution

End Point titration settings

Burette volume:	25 ml
Stirring speed:	400 rpm
Working mode:	mV with i = 0
Number of end points:	1
End point:	690 mV
Stirring delay:	30 seconds
Minimum speed:	0.1 ml/min
Maximum speed:	5.0 ml/min
Proportional band:	350 mV
End point delay:	5 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 ferrous solution as titrant.

Connect the MC3051Pt-9 electrode to input E1 via cable CL114.

Pipette exactly 20 ml of $K_2Cr_2O_7$ 0.1 eq/l standard.

Add 10 ml of 50% H_2SO_4 solution and complete to 100 ml with distilled water.

Start method by pressing the RUN key.

Results

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

Vol(FeII salt) * C(FeII salt) = Vol($K_2Cr_2O_7$) * C($K_2Cr_2O_7$)

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

Notes
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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) It is possible to calibrate the ferrous titrant by weighing an amount of potassium dichromate.

With a 25 ml burette cylinder capacity

Exactly weigh approximately 95 mg of potassium dichromate

In the STANDARD screen ENTER

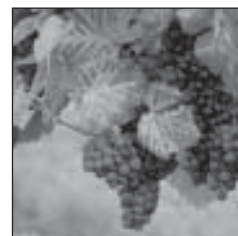Standard unit: mg
Standard amount: xx.x
Concentration unit: %
Concentration: 100
       (or purity of the standard)
Molecular weight: 294.19

And in the RESULT screen ENTER

Result: eq/l
Coefficients: 1 Standard + 6 Titrant

Note that for $FeSO_4$, concentration in eq/l is the same as in mol/l.

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

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

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 preparation

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

$$1(As_2O_3) + 2(I_2) + 5H_2O \rightarrow 4I^- + 2AsO_4^{--} + 10H^+$$
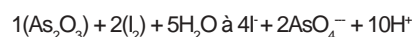
A 0.1 eq/l As III solution contains 1/40 As_2O_3 mol/l.

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 H_2SO_4 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

Na_2CO_3 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

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Calibration of an Iodine Solution

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 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:

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

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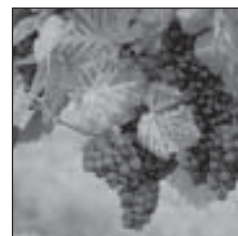
For the best result accuracy, pipette a standard volume corresponding to a delivered titrant volume greater than 50% of the used burette cylinder.

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.

_

Alkalinity of Bleach
(NaClO Solution)

Introduction

A bleach solution contains mainly NaClO plus basic products such as NaOH and Na_2CO_3.
The concentration of basic products is generally expressed as NaOH concentration, which is around 8 g/l NaOH, i.e. 0.2N concentration.
The alkalinity determination uses an acid/base titration.

Principle

The OH^- content is simply determined by an acid/base titration using a 0.1 eq/l strong acid as titrant.
Depending on the bleach solution, the titration curve generally shows 2 inflections. The most common method involves a titration of all the basic functions by a predetermined end point titration at pH 4.00. Before this titration, it is necessary to reduce the ClO^- ions present in the solution.

Electrode and reagents

pHC2011-8 Combined pH Electrode (part no. E16M317)

H_2O_2 30% (volume)

Hydrochloric acid 0.1 eq/l (or 0.1 mol/l)

Slowly add 8.3 ml of concentrated hydrochloric acid to 500 ml of distilled water and dilute to exactly 1000 ml. Calibrate the titrant versus $Na_2B_4O_7$, 10 H_2O (sodium borate) as standard.

(See separate application note)

Distilled water

IUPAC Series pH standards pH 4.005 (part no. S11M002) and pH 10.012 (part no. S11M007)

End Point titration settings

Burette volume:	25 ml
Stirring speed:	400 rpm
Working mode:	pH
Number of end points:	1
End point:	4.00 pH
Stirring delay:	45 seconds
Minimum speed:	0.2 ml/min
Maximum speed:	8.0 ml/min
Proportional band:	3.00 pH
End point delay:	5 seconds
Sample unit:	ml
Sample amount:	5
Titration:	Decreasing pH
Result:	g/l

Procedure

Calibrate the electrode using the two IUPAC standards

Pipette 5 ml of sample

Always dilute the sample with the same volume of distilled water (max. 50 ml)

Slowly add 5 ml of H_2O_2 for 5 ml of sample

Dip electrode and delivery tip in the solution

Start method by pressing the RUN key

 Radiometer analytical

Alkalinity of Bleach (NaClO Solution)

Results

Expressed as NaOH content (MW = 40 g/mol) in g/l

As 1 molecule of titrant reacts with 1 molecule of NaOH

$$R(NaOH) = V(titr) * C(titr) * 40 / V(smp)$$

-V(titr) = total volume of titrant to reach the end point in ml

-V(smp) = sample amount

-C(titr) = exact concentration of the titrant in eq/l

For a result in g/l

Enter

The sample amount in the SAMPLE screen

The titrant concentration in the TITRANT screen

1 Titrant and 1 Sample in the COEFFICIENTS display

40 for NaOH molecular weight

The Titration Manager gives a result according the above formula.

For a result as a %

As the Titration Manager cannot give a result in % if the sample unit is a volumetric unit, you can use the equation feature:

Equation number: 1
Equation result: % NaOH
Equation formula:
R1 / 10

R1 is the titration result calculated in g/l.

5 determinations on a commercial concentrated bleach

Mean (as NaOH):   7.25 g/l
Standard deviation: 0.07 g/l
Rel. standard deviation: 1%

Working range

Results are expressed in g/l of NaOH (MW = 40 g/mol)

1 ml of HCl 0.1 eq/l represents 4 mg of NaOH or, with a 5 ml sample volume a NaOH content corresponding to 0.8 g/l

The working range can be calculated as the following formula:

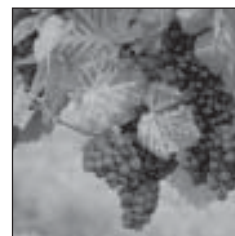Result (in g/l) =
    V(titr in ml) * 0.8 * 1000 / V(smp)

Using the conditions given in this applicatin note (5 ml sample and a 25 ml burette for titrant), it is possible to obtain results between 7 g/l (for 35% capacity of the burette) and 20 g/l (total capacity of the burette) with the best possible accuracy and reproducibility.

Notes

Addition of H_2O_2 is necessary to reduce the ClO^- ion to Cl^- before running the alkalinity titration.

# ClO$^-$ Determination in Bleach Direct Titration with NaAsO$_2$

Introduction

A bleach solution contains mainly NaClO plus basic products such as NaOH and Na$_2$CO$_3$.
ClO$^-$ can be determined directly by redox titration. The titration result is generally expressed as a % of ClO$^-$.

Principle

Direct titration of ClO$^-$ by NaAsO$_2$ uses the following redox reaction

$$AsO_2^- + ClO^- + 2OH^- \rightarrow AsO_4^{---} + Cl^- + H_2O$$
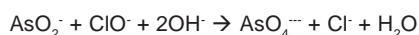
Note that in this reaction, which involves 1 mole ClO$^-$ for 1 mole AsO$_2^-$, 2 oxidation numbers are exchanged (As (III) to As (V) and Cl(1) to Cl(-1)).
This reaction needs a pH of around pH 10.00
The titration is run according to a potentiometric pre-set end point titration using a combined platinum/reference electrode.

Electrode and reagents

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

Distilled water

pH 10.00 Buffer Solution (part no. S11M014)

NaAsO$_2$ 0.1 eq/l solution in water

To prepare the 0.1 eq/l (or 0.05 mol/l) NaAsO$_2$ solution

As a 0.1 eq/l NaAsO$_2$ solution contains 1/40 As$_2$O$_3$ mol/l

Weigh 4.2467 (169.87/40) g of pure As$_2$O$_3$ and dissolve it in about 20 ml of 10M NaOH. You can gently heat the solution to have a quick dissolution

Add about 200 ml of pure water and 1M H$_2$SO$_4$ until the pH reaches 8.00

Leave the solution to reach room temperature

Dilute to 1000 ml exactly with a volumetric flask

End Point titration settings

Burette volume:	25 ml
Stirring speed:	400 rpm
Working mode:	mV
Number of end points:	1
End point:	200 mV
Stirring delay:	30 seconds
Minimum speed:	0.1 ml/min
Maximum speed:	10 ml/min
Proportional band:	375 mV
End point delay:	5 seconds
Sample unit:	ml
Sample amount:	0.5
Titration:	Decreasing potential
Result:	g/l

Procedure

Install the titration system with the NaAsO$_2$ solution

Connect the electrode

Pipette (or preferably weigh) the sample amount

Dilute the sample with 50 ml of pH 10.00 buffer solution

Dip electrode and delivery tip in the solution

Start method by pressing the RUN key

Radiometer analytical

Results

Commonly expressed as g/l of ClO- (MW = 51.43 g/mol)

Use as titrant unit: mol/l (M)

As 1 mole of titrant reacts with 1 mole of ClO- in sample:

R = V(titr) * C(titr) * 51.43 / V(smp)

V(titr) = total volume of titrant to reach the end point (in ml)

C(titr) = concentration of titrant in mol/l

51.43 = molecular weight of ClO-

V(smp) = sample volume in ml

For a result in g/l

Enter

The sample amount in the SAMPLE screen

The titrant concentration in the TITRANT screen (in mol/l)

1 Titrant and 1 Sample in the COEFFICIENTS display

51.43 as molecular weight

The Titration Manager gives a result according the above formula.

Using eq/l as titrant unit

Enter

The sample amount in the SAMPLE screen

The titrant concentration in the TITRANT screen (in mol/l)

2 Titrants and 2 Samples in the COEFFICIENTS display

51.43 as molecular weight

The Titration Manager gives a result in g/l according the above formula.

For a result as a %

As the Titration Manager cannot give a result in % if the sample unit is a volumetric unit, you can use the equation feature:

Equation number: 1
Equation result: % ClO
Equation formula:
R1 / 10

R1 is the titration result calculated in g/l

5 determinations on a commercial concentrated bleach

Mean (as ClO-): 76.5 g/l
Standard deviation: 1.2 g/l
Rel. standard deviation: 1.6%

Working range

As a commercially available concentrated bleach corresponds to a ClO- concentration in g/l of around 80 (corresponding to a total volume of titrant of around 16 ml for 0.5 ml of sample), it is possible to use the dilution procedure to facilitate pipetting or weighing 0.5 ml of sample.

For example, pipette 5 ml of sample, dilute to 100 ml with water and take an aliquot of 10 ml.

In the SAMPLE display enter

DILUTION: YES
Sample: 5 ml
Final dilution volume: 100 ml
Aliquot: 10 ml

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# ClO$^-$ Determination in Bleach Indirect Titration

Introduction

A bleach solution contains mainly NaClO plus basic products such as NaOH and Na_2CO_3.

ClO$^-$ can be determined by redox titration. Besides the direct titration by sodium arsenite; ClO$^-$ determination can be run with a two-step titration; quantitative oxidation of I$^-$ by ClO$^-$ giving I$_2$ and titration of I$_2$ by sodium thiosulphate. The titration result is generally expressed as % of ClO$^-$.

Principle

Oxidation of I$^-$ by ClO$^-$ follows the reaction

$$ClO^- + 2I^- + H_2O \rightarrow I_2 + Cl^- + 2OH^-$$
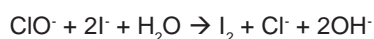
Titration of I$_2$ by sodium thiosulphate follows the reaction

$$I_2 + 2 S_2O_3^{2-} \rightarrow S_4O_6^{2-} + 2I^-$$
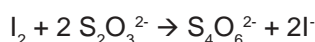
The result of these 2 equations is 1 ion ClO$^-$ corresponds to 2 ions $S_2O_3^{2-}$

The titration of I$_2$ by $S_2O_3^{2-}$ is run by pre-set end point titration with imposed current potentiometry.

Electrode and reagents

MC231Pt2 Metal Electrode, double platinum wire, with adapter part no. A94P801 (BNC/2xbanana) or M241Pt2-8 Metal Electrode

Distilled water

KI solid state

HCl pure grade

$Na_2S_2O_3$ 0.1 eq/l or 0.1 mol/l (For concentration determination see separate application note)

Weigh exactly 24.8181 g of $Na_2S_2O_3$, $5H_2O$

Dissolve in 500 ml of freshly boiled distilled water

Add 2 or 3 drops of $CHCl_3$

Dilute to 1000 ml in a volumetric flask

Wait for one day and filter if necessary

Store in a brown glass bottle

End Point titration settings

Burette volume:	25 ml
Stirring speed:	400 rpm
Working mode: mV with i = 10 µA (DC)	
Number of end points:	1
End point:	200 mV
Stirring delay:	40 seconds
Minimum speed:	0.1 ml/min
Maximum speed:	5.0 ml/min
Proportional band:	200 mV
End point delay:	5 seconds
Sample unit:	ml
Sample amount:	0.5
Titration:	Increasing potential
Result:	g/l

Procedure

Install the titration system with the $Na_2S_2O_3$ solution

Connect the electrode

Pipette (or preferably weigh) the sample amount

Dilute the sample with 50 ml of distilled water

Add 1 g of KI and 5 ml of HCl

Dip electrode and delivery tip in the solution

Radiometer analytical

Start method by pressing the RUN key

Results

Commonly expressed as g/l of ClO⁻ (MW = 51.43 g/mol)

Use as titrant unit: mol/l (M)

As 2 moles of titrant corresponds to 1 mole of ClO⁻ in sample:

R = V(titr) * C(titr) * 51.43 / 2 * V(smp)

V(titr) = total volume of titrant to reach the end point (in ml)

C(titr) = concentration of titrant in mol/l

51.43 = molecular weight of ClO⁻

V(smp) = sample volume in ml

For a result in g/l

Enter

The sample amount in the SAMPLE screen

The titrant concentration in the TITRANT screen (in mol/l)

2 Titrants and 1 Sample in the COEFFICIENTS display

51.43 as molecular weight

The Titration Manager gives a result according the above formula.

For a result as a %

As the Titration Manager cannot give a result in % if the sample unit is a volumetric unit, you can use the equation feature

Equation number: 1
Equation result: % ClO
Equation formula:
R1 / 10

R1 is the titration result calculated in g/l

5 determinations on a commercial concentrated bleach

Mean (as ClO⁻):   78.8 g/l
Standard deviation: 1.2 g/l
Rel. standard deviation: 1.6 %

Working range

As a commercially available concentrated bleach corresponds to a ClO⁻ concentration in of around 80 g/l (corresponding to a total volume of titrant of around 16 ml for 0.5 ml of sample), it is possible to use the dilution procedure to facilitate pipetting or weighing 0.5 ml of sample.

For example, pipette 5 ml of sample, dilute to 100 ml with water and take an aliquot of 10 ml.
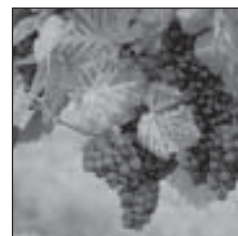
In the SAMPLE display enter

DILUTION: YES
Sample: 5 ml
Final dilution volume: 100 ml
Aliquot: 10 ml

Calibration of a Thiosulphate Solution

Standard preparation

To prepare a 0.1 eq/l (or 0.1 mol/) sodium thiosulphate solution, dissolve 24.8181 g of $Na_2S_2O_3$, $5H_2O$ in 500 ml of freshly distilled water (or freshly boiled and cooled deionised water) and 2 or 3 drops of $CHCl_3$ (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.

$Na_2S_2O_3$, $5H_2O$ has a molecular weight corresponding to 248.181 g/mol.

Standard preparation

To calibrate a thiosulphate solution, use potassium iodate KIO_3 as standard. It reacts with potassium iodide KI giving iodine I_2 according to the reaction

$$IO_3^- + 5I^- + 6H^+ \rightarrow 3I_2 + 3H_2O$$
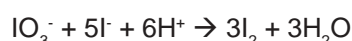
The thiosulphate ion reacts with I_2 according to the reaction

$$3I_2 + 6S_2O_3^{--} \rightarrow 6I^- + 3S_4O_6^{--}$$
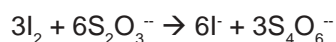
The molecular weight of KIO_3 is 214.0 g/mol. **A 0.1 eq/l iodate solution,** according to the above-mentioned reactions (1 ion IO_3^- corresponds to $3I_2$), **contains 1/60 mol/l of KIO_3.**

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)

R Radiometer analytical

Calibration of a Thiosulphate Solution

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 KIO_3 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:

$$Vol(S_2O_3^{--}) * C(S_2O_3^{--}) = Vol(IO_3^-) * C(IO_3^-)$$
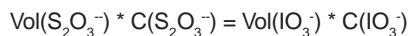
With $C(IO_3^-)$ expressed in eq/l

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

Determination of Gastric Acidity
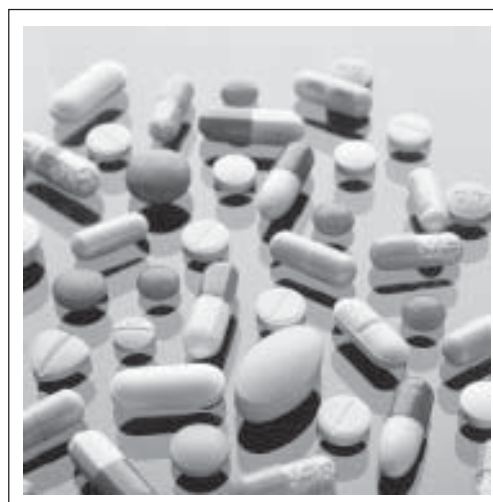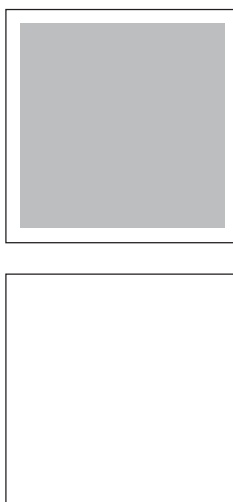
Introduction

The gastric secretions are a complex mix of HCl, pepsin, rennet, mineral chlorides (Na, K, Ca, Mg), calcium phosphate and organic material (especially mucus). Generally the acid concentration is 0.1 N, for an healthy human, but this concentration can be modified by many external factors as food or drug ingestion or stress.
In case of illness it can be necessary to determine the hyper-acidity or the acidity of the gastric secretion.

Principle

Acidity of gastric juice is determined by an end-point titration at pH 7.00 using as titrant a NaOH solution 0.1 equivalent/l. The sample amount is generally between 0.1 ml (100 µl) and 1 ml (1000 µl). The result is expressed as meq/ml (or eq/l).
As for pathological situations, expected results should be between 0.01 meq/ml and may be up to 8 meq/ml, it is very difficult to have only one titration method covering this whole range.

According to this fact the pre-programmed method "**Gastric acidity**" is suitable for orientation test and 0.1-2 meq/ml range. Copying this method, it is possible to create 2 others methods changing only few parameters.

GASTRIC AC L
(for 0.01-0.1 meq/ml range)

Minimum speed:	0.02 ml/min
Maximum speed:	0.5 ml/min
Sample amount:	500 µl
	(possibly 1000µl)

GASTRIC AC H
(for range > 2 meq/ml)

Maximum volume:	20 ml
Minimum speed:	0.1 ml/min
Maximum speed:	5.0 ml/min
Sample amount:	100 µl
	(possibly 200µl)

Electrode and reagents

PHC3101 Combined pH electrode (E16M327) with CL114 (A94L114) cable

25 mm magnetic barrels A90A410

NaOH 0.1 equivalent/l solution in distilled water (see separate application note but commercially available solution can be used). The pre-programmed titrant for the method is labelled as "**NaOH Gastric 0.1**"

pH standard IUPAC pH 4.005 (S11M002) and pH 7.00 (S11M004)

Distilled or de-ionised water

Titration Vessel PP 22-45 ml 904-489 (50 pcs) with special holder 923-172 (code number for 10 pcs)

Pipettes for 100µl, 500µl and 1 ml

End Point titration settings (Gastric Acidity)

Burette volume:	10 ml
Maximum volume:	40 ml
Stirring speed:	500 rpm
Working mode:	pH
Number of end points:	1
End point:	7.00 pH
Stirring delay:	10 seconds
Minimum speed:	0.02 ml/min
Maximum speed:	3.0 ml/min
Proportional band:	5.00 pH
End point delay:	10 seconds
Sample unit:	µl

Radiometer analytical

Sample amount: 1000
                    (see working range)
pH: Increasing

Result 1:   ml
Equation unit: meq/ml
Equation :   V1*CT*1000/SA

Procedure

Put in place the pHC3101-9 combined electrode in the suitable hole of the electrode head (see Guide to bayonet accessories booklet)

Calibrate the combined glass electrode with the above mentioned buffer solution

Place the electrode and the delivery tip in opposite positions on electrode head. The ends of electrode and delivery tip should be at the same level in the beaker.

Install the titrant (**NaOH gastric 0.1N**) and enter its concentration

Pour 20 ml of distilled or de-ionised water into the 22-45 ml beaker and place it on the sample stand of the Titration Manager using a beaker holder

Add the recommended volume of sample

If the expected result is approximately known run the appropriate method

If the expected result is unknown, run, as orientation test with 100µl of sample amount the "Gastric Acidity" method and according to this first result choose the suitable method

Working ranges

Titrant volume in ml as a function of the gastric acid concentration (in meq/ml) and sample amount (in µl)

	0.01 meq/ml	0.1 meq/ml	1.0 meq/ml	2.0 meq/ml	8.0 meq/ml
100µl	(0.01 ml)	(0.1 ml)	1.0 ml	2.0 ml	8.0 ml
500µl	(0.05 ml)	0.5 ml	5.0 ml	10.0 ml	(40.0 ml)
1000µl	0.1 ml	1.0 ml	10.0 ml	(20.0 ml)	(80.0 ml)

In brackets: titration case not recommended for good accuracy or for titrant consumption , increase or decrease the sample size respectively.

Results with "Gastric Acidity" and an healthy person

Used sample: 500 µl of a mix of HCl and pepsin in de-ionised water
The result corresponds to:

$$R1 = V(titr) * C(titr) / V(smp)$$

V(titr) = Titrant volume in ml
C(titr) = Titrant concentration in eq/l
V(smp) = sample volume in ml

Results (3 determinations)
Mean: 0.1067 meq/ml
Standard deviation: 0.0004

Notes

1) The pHC3101-9 can be interesting for low maintenance level; Do not forget to store this electrode in KCl 3M solution as recommended by Radiometer Analytical. Note that this electrode is used in the pre-programmed method.

2) Place the electrode and the delivery tip in opposite positions on electrode head. Extremities of electrode and delivery tip are at the same level in the beaker.

3) As the programmed time between two electrodes calibrations is **1 day** the corresponding icon on the main menu will always be "cloudy" after a calibration. See the user's guide (D21T043) chapter 2, p. 12.

4) For quick determinations, it is possible to use an end-point Titration Manager fitted with a 25 ml burette cylinder, 1000 µl of sample volume and the pre-programmed method without change. With these conditions the working range is 0.02 meq/ml - 4.0 meq/ml.

Hydrogen Peroxide Determination
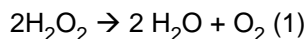## (by manganimetric titration)
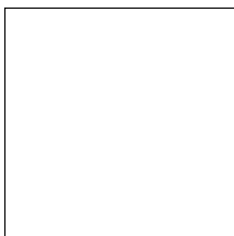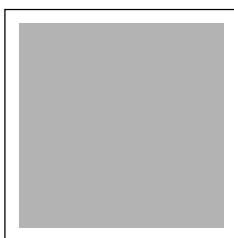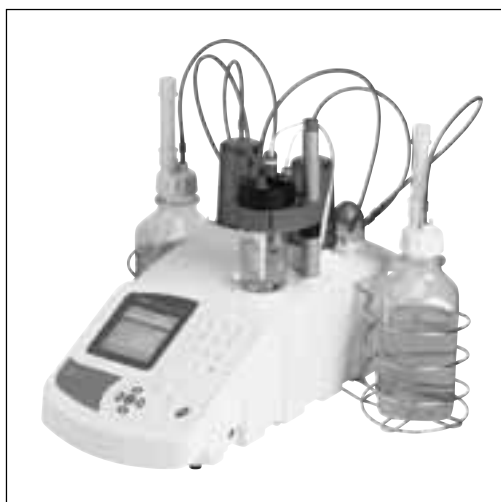
Introduction

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.

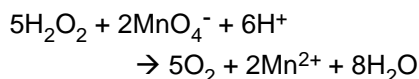The dismutation reaction of hydrogen peroxide corresponds to the following equations:

$$H_2O_2 + 2H^+ + 2\bar{e} \rightarrow 2H_2O$$
$$H_2O_2 \rightarrow O_2 + 2H^+ + 2\bar{e}$$
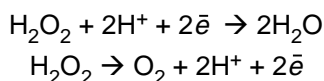
The global reaction corresponds to:

$$2H_2O_2 \rightarrow 2 H_2O + O_2 \ (1)$$

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. 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

Hydrogen Peroxide Determination (by manganimetric titration)

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

$$R(\%) = (5 * C_{titr} * V_{titr} / 2 * V_{smp}) * (34/10)$$

5 and 2 = Reaction coefficients
C_{titr} = Titrant concentration
V_{titr} = Used titrant volume
V_{smp} = Used sample volume
34 = Molar weight of H_2O_2
10 = factor to express result as a %

$$R(\text{volumes}) = R(\%) * 3.294 / d$$

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 (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
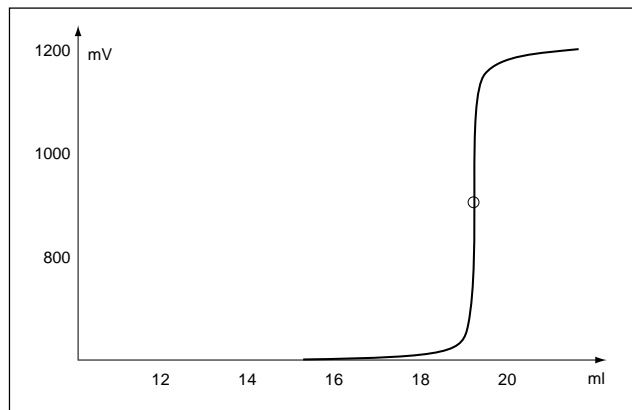
Equation
If H2O2 density is known modify to the equation as follows

Equation = R1*3.924/d

d = H_2O_2 density

Curve

Direct curve