

REDOX TITRATIONS

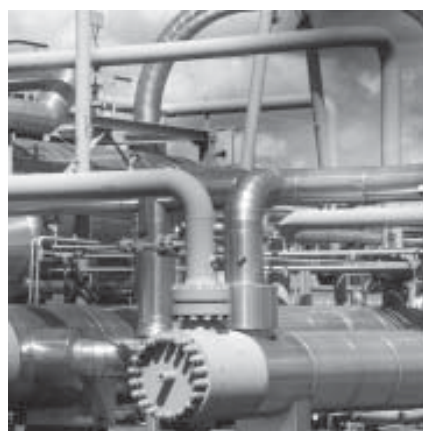
Introduction

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

Redox titrations are frequently used in many industries and involve potentiometric titrations with or without imposed current. Instructions for performing some of the most commonly used applications are given in this booklet together with calibration procedures for the corresponding titrants and electrodes. Certain modifications may be needed to take into account specific regulations or standards in force in certain countries, in particular regarding results presentation.



Radiometer Analytical produces other technique-based applications booklets as well as a range of applications dedicated to particular sectors. Ask your local representative for the following booklets:

| <i>Technique</i> | <i>Part No.</i> |
|----------------------------------|---------------------|
| Acid-base titrations | D41T009 |
| Precipitation titrations | D41T010 |
| Complexometric titrations | D41T011 |
| <i>Dedicated</i> | <i>Part No.</i> |
| Food and beverage analysis | D41T004 |
| Plating bath analysis | D41T005 |
| Water and environmental analysis | D41T006 |
| Chemical industries | D41T007 |

Our Applications Laboratory is continually developing new applications.

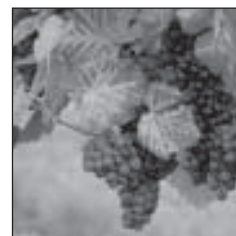
For the latest updates visit us at www.titration.com.

REDOX TITRATIONS

| | |
|-----------------------|---|
| TTEP01.09MIN | Operational Qualification (End Point Titration Manager) |
| TTIP01.01MIN | Operational Qualification (Inflection Point Titration Manager) |
| TTEP01.03MIN | Calibration of a Ferrous Solution |
| TTEP01.04MIN | Calibration of an Iodine Solution |
| TTEP01.08MIN | Calibration of a Thiosulphate Solution |
| TTEP01.03ENV | Chemical Oxygen Demand of Water |
| TTEP01.02PLA | Analysis of a Chromic Passivation Bath |
| TTEP01.07AFD | Free Sulphur Dioxide in Wine |
| TTEP01.06MIN | ClO ⁻ Determination in Bleach - Direct Titration with NaAsO ₂ |
| TTEP01.07MIN | ClO ⁻ Determination in Bleach - Indirect Titration |
| TTIP01.02PET | Bromine Number |
| TTEP-IP02.01AFD | Vitamin C in Iodometry |
| TTIP02.03PLA | Cu in Electroless Copper Baths Redox |
| TTIP02.01AFD | Peroxide Number of Edible Oils |
| TTIP02.01MIN | Hydrogen Peroxide Determination |
| TTEP-IP02.01ENV | Dissolved Oxygen in Water |
| TTEP-IP02.02AFD | Iodine Value of Animal and Vegetable Fats and Oils |

End Point Titration Manager

Operational Qualification



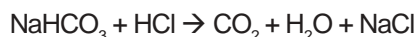
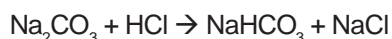
Introduction

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



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

End Point Titration Manager Operational Qualification

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 pH2011-8 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 Na₂CO₃

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 P₂O₅ 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 Na₂CO₃

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 Na₂CO₃

Use a maximum volume of 10 ml

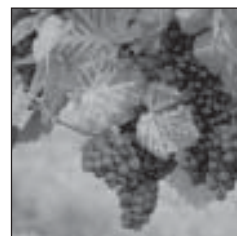
No predose.

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%.

Inflection Point Titration Manager Operational Qualification



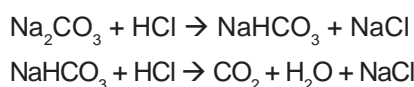
Introduction

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:



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

Inflection Point Titration Manager Operational Qualification

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 pH2011-8 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 Na₂CO₃

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 P₂O₅ 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 Na₂CO₃.

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 Na₂CO₃. Use a maximum volume of 10 ml.

With a 5 ml burette capacity

Weigh exactly approximately 20 mg of Na₂CO₃. 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.

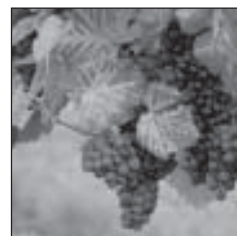
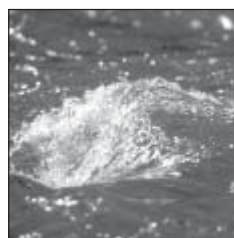
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Calibration of a Ferrous Solution Fe(II)

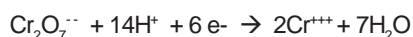


Reagent preparation

The most common Fe(II) salt is Mohr's salt: $(\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ (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 $\text{K}_2\text{Cr}_2\text{O}_7$ (molecular weight 294.19 g/mol). Potassium dichromate solutions are very stable. The oxidation power of potassium dichromate is based on the reaction



A 0.1 eq/l potassium dichromate solution contains 1/60 mole of dichromate.

To prepare 1000 ml of 0.1 eq/l $\text{K}_2\text{Cr}_2\text{O}_7$ 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

$\text{K}_2\text{Cr}_2\text{O}_7$ 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 $\text{K}_2\text{Cr}_2\text{O}_7$ 0.1 eq/l standard.

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

Calibration of a Ferrous Solution Fe(II)

Start method by pressing the RUN key.

Results

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

$$\frac{\text{Vol(FeII salt)} \times \text{C(FeII salt)}}{\text{Vol(K}_2\text{Cr}_2\text{O}_7) \times \text{C(K}_2\text{Cr}_2\text{O}_7)}} =$$

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 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

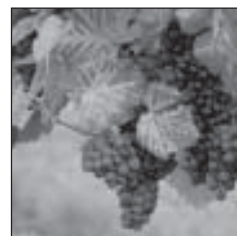
Standard unit: mg
Standard amount: xx.x
Concentration unit: %
Concentration: 100
(or purity of the standard)
Molecular weight: 294.19

And in the RESULT screen ENTER

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

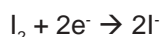
Note that for FeSO₄, concentration in eq/l is the same as in mol/l.

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



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

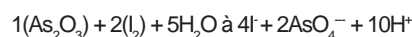
To prepare a 0.1 eq/l iodine solution, dissolve 40 g of potassium iodide in 50 ml of distilled water then add 12.69 g (235.8/20) of analytical grade iodine, wait for it to dissolve completely and complete to 1.000 ml with a volumetric flask.

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



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 |

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

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

Notes

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.

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

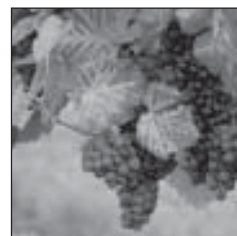
Results

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

$$\text{Vol}(\text{As}_2\text{O}_3) * C(\text{As}_2\text{O}_3) = \text{Vol}(\text{I}_2) * C(\text{I}_2)$$

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

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 $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ 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.

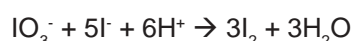
Look at the solution from time to time and filter again if necessary.

Solutions with a concentration below 0.01N (or 0.01M) are not stable.

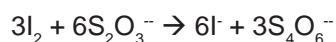
$\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ has a molecular weight corresponding to 248.181 g/mol.

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



The thiosulphate ion reacts with I_2 according to the reaction



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)

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:

$$\text{Vol}(\text{S}_2\text{O}_3^{2-}) * \text{C}(\text{S}_2\text{O}_3^{2-}) = \text{Vol}(\text{IO}_3^-) * \text{C}(\text{IO}_3^-)$$

With $\text{C}(\text{IO}_3^-)$ 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

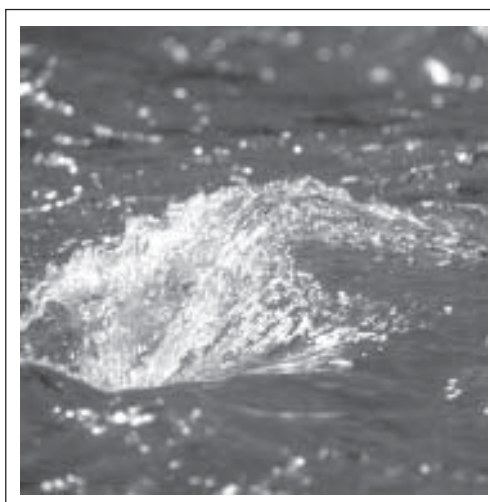
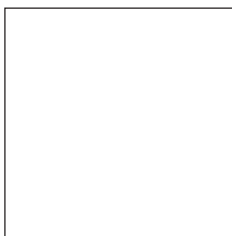
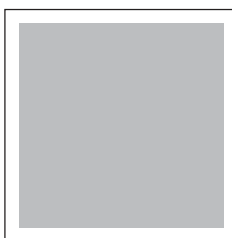
3) Instead of an aqueous solution of KIO_3 , you can use solid state KIO_3 . In this case and for the application note conditions, exactly weigh approximately 60 mg of KIO_3 (for 20 ml of sodium thiosulphate solution) and in the STANDARD screen ENTER

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

Chemical Oxygen Demand of Water

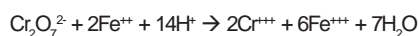


Introduction

Chemical Oxygen Demand (COD) is an important parameter for wastewater or surface water testing and plants. This determination gives information about the degree of water pollution by organic material. This application note concerns COD determination by potassium dichromate. Another method using potassium permanganate is used for low COD determinations.

Principle

Organic matter contained in a water sample is oxidised in 50% sulphuric acid, by a known excess of potassium dichromate. After digestion, remaining unreduced dichromate is determined by potentiometric titration using a Fe(II) solution according to the following reaction:



A blank titration with distilled water as sample is run for every cycle.

The result is expressed as oxygen equivalent.

Electrode and reagents

For titration in a beaker

M231Pt Metal Electrode, platinum (part no. E31M002) with CL114 cable (part no. A94L114)

REF601 Reference Electrode, mercurous sulphate (part no. E21M012)

For titration directly in digestion flask

MC602Pt Metal Electrode, combined platinum/mercurous sulphate (part no. 945-360)

Titrant $(\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ (Mohr's salt) 0.15 eq/l (MW = 392.14 g/mol)

Weigh and dissolve 58.82 g of Mohr's salt 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

Calibrate as indicated in separate application note

Potassium dichromate 0.25 eq/l solution or 0.0417M ($\text{K}_2\text{Cr}_2\text{O}_7$)

(A 0.1 eq/l $\text{K}_2\text{Cr}_2\text{O}_7$ solution contains 1/60 mole of $\text{K}_2\text{Cr}_2\text{O}_7$ that has a molecular weight of 294.19 g/mol)

Dry the potassium dichromate (analytical grade) for 2 hours at 120°C and let it cool to room temperature

Weigh exactly 12.258 g and dissolve exactly with freshly distilled water to 1000 ml using a volumetric flask

Sulphuric acid reagent

Add 5.5 g of Ag_2SO_4 to 1 kg of concentrated H_2SO_4 and leave to stand for 2 days to dissolve the Ag_2SO_4

Solid mercuric sulphate HgSO_4

End Point titration settings

Back titration with blank

| | |
|-----------------------|---------------|
| Burette volume: | 25 ml |
| Stirring speed: | 400 rpm |
| Working mode: | mV (with i=0) |
| Back titration: | Manual |
| Number of end points: | 1 |
| End point: | 300 mV |
| Stirring delay: | 30 seconds |

Chemical Oxygen Demand in Water

Minimum speed: 0.1 ml/min
Maximum speed: 2.5 ml/min
Proportional band: 350 mV
End point delay: 5 seconds
Sample unit: ml
Sample amount: 20
Blank volume:

determined by blank titration

Excess: 0.0 ml
Titration: Decreasing potential

Results

Results number: 1
Result unit: mg/l

Procedure

Blank preparation and titration

Using distilled water as sample, prepare and titrate a blank with the same procedure and reagents (see below)

Accept the obtained titrant volume as blank volume

The theoretical blank volume can be calculated according to the following formula:

Vol. of blank = $10 \times 0.25 / 0.15 = 16.67$ ml

10 = volume of $K_2Cr_2O_7$ solution in ml

0.25 = Concentration of $K_2Cr_2O_7$ in eq/l

0.15 = concentration of Mohr's salt solution in eq/l

Sample preparation

Place 20 ml of water in a 250 ml refluxing flask or in a special COD tube. Add 0.4 g of $HgSO_4$ and glass beads and very slowly 2 ml of sulphuric reagent while mixing. Then add 10.00 ml of dichromate solution and 28 ml of sulphuric acid reagent and connect to condenser. Mix thoroughly and boil under reflux during 2 hours.

After rinsing condenser with 5/10 ml of distilled water, transfer to titration beaker using 25/30 ml of distilled water to rinse. Total volume does not exceed 100 ml.

Sample titration

Prepare the titration manager with 25 ml burette and 0.15 eq/l ferrous solution as titrant.

Connect M231Pt electrode via the CL114 cable and the REF601 reference electrode or, if necessary, the MC602Pt combined electrode.

Dip electrodes and delivery tip in the sample beaker.

Start method by pressing the RUN key.

Results

Expressed as mg/l of oxygen equivalent according to

COD (in mg/l of oxygen) = $(V_{bl} - V_{tit}) \times C \times 8 \times 1000 / V_{sample}$

V_{bl} = titrant volume consumed during blank titration (close to 16.67 ml for application note conditions)

V_{tit} = titrant volume consumed during sample titration

$(V_{bl} - V_{tit})$ = directly calculated by TIM8xx

C = titrant concentration in mol/l or equivalent/l

8 = equivalent weight for oxygen (16/2)

V_{sample} = sample volume in ml

For a result in mg/l of oxygen

Enter

The sample amount in the SAMPLE screen

The titrant concentration in the TITRANT screen

1 Titrant and 1 Sample in the COEFFICIENTS display

8 as equivalent weight for oxygen

The Titration Manager gives a result according to the above formula, as $(V_{bl} - V_{tit})$ is directly calculated by the Titration Manager.

For 5 determinations

Mean: 450 mg/l oxygen
Standard deviation: 15 mg/l
Rel. standard deviation: 3%

Working range

With application note conditions, titrant concentration of 0.15 eq/l and sample volume of 20 ml

$(V_{bl} - V_{tit}) = 1$ ml corresponds to a COD of 60 mg/l

$(V_{bl} - V_{tit}) = 15$ ml corresponds to a COD of 900 mg/l

Notes

The Titration Manager uses the same electrodes for titration and reagent calibration. For titrant calibration with the electrodes in this note, you can use the application note TTEP01.03MIN with a change in the end point potential.

The latest standard NFT 90.101(2001) uses the same procedure but uses as reagents

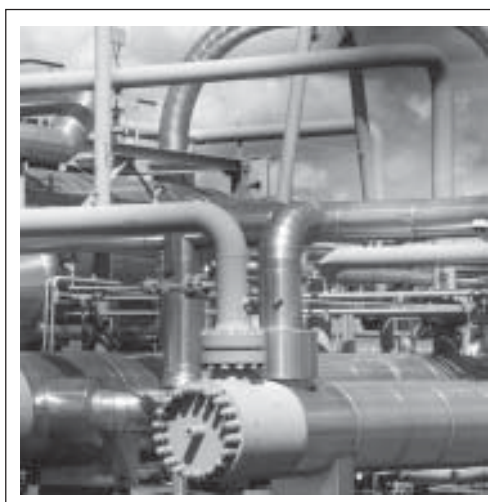
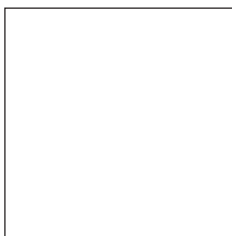
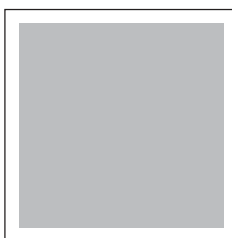
$(NH_4)_2Fe(SO_4)_2 \cdot 6H_2O$ (Mohr's salt)
0.12 mol/l
Potassium dichromate solution or
0.040 mol/l ($K_2Cr_2O_7$)

And works with 10 ml of sample

Bibliography

EPA method number 410.1
Standard methods for water and wastewater 18th edition (1992) 5-6 part 5220
ISO 6060 (1989)
NFT 90-101 (2001)

Analysis of a Chromic Passivation Bath

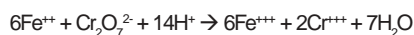


Introduction

In the car industry, the analysis of a chromic passivation bath consists of a determination of the concentration of dichromate ion by redox titration with ferrous sulphate as titration reagent. The result is expressed as "points", i.e. the number of millilitres of 0.1 eq/l titrant used for a given amount of sample, generally 50 or 100 ml.

Principle

The titration reaction occurs according to the following reaction



This reaction takes place in strong acidic media.

Electrode and reagents

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

Ferrous sulphate solution 0.1 eq/l

Weigh and dissolve 39.21 g of $(\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ (Mohr's salt) 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.

50% H_2SO_4 in water

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

End Point titration settings

| | |
|-----------------------|------------|
| Burette volume: | 25 ml |
| Stirring speed: | 400 rpm |
| Working mode: | mV |
| Number of end points: | 1 |
| End point: | 690 mV |
| Stirring delay: | 30 seconds |
| Minimum speed: | 0.1 ml/min |
| Maximum speed: | 2.5 ml/min |
| Proportional band: | 250 mV |
| End point delay: | 5 seconds |

| | |
|----------------|---------------|
| Sample unit: | ml |
| Sample amount: | 100 |
| Result: | ml |
| Titration: | Decreasing mV |

Procedure

Prepare the Titration Manager with a 25 ml burette and 0.1 eq/l ferrous solution as titrant.

Connect the electrode to the input by means of the CL114 cable.

Pipette the required amount of sample (generally 100 ml).

Add 25 ml of 50% H_2SO_4 solution for 100 ml of sample.

Dip electrode and delivery tip in the solution.

Start method by pressing the RUN key.

Results

Expressed as ml of 0.1 eq/l titrant for 100 ml of sample

$R = V(\text{titr})$

Analysis of a Chromic Passivation Bath

If the titrant used is not exactly 0.1 eq/l in concentration and if the sample amount is not 100 ml, correct as follows:

$$R = V(\text{titr}) * 100 * C(\text{titr}) / V(\text{smp}) * 0.1$$

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

-V(smp) = current sample amount

-C(titr) = exact concentration of the titrant

5 determination results in points or ml of titrant

Mean: 8.50 ml
Standard deviation: 0.076 ml
Rel. standard deviation: 0.9%

Working range

As the result for a 100 ml sample volume and a 0.1 eq/l titrant is directly the delivered volume of titrant, the working range for the best possible accuracy and reproducibility is between 8.75 and 25 ml for the application note conditions.

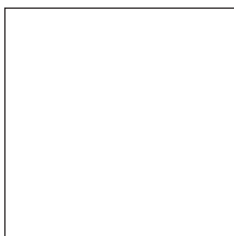
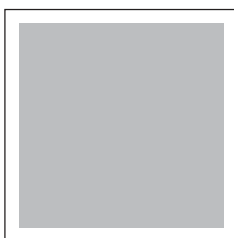
Notes

- 1) Remember that strong acidic media are necessary for this redox titration (see the reaction formula)
- 2) 50% sulphuric acid can be replaced by 50% phosphoric acid
- 3) The 30-second stirring delay is necessary to allow the platinum electrode to reach a stable potential
- 4) If the titrant used is not 0.1 eq/l, you need to multiply the result obtained by a factor, F determined as follows:

F = current titre (expressed in eq/l) / 0.1

Free Sulphur Dioxide in Wine

(Modified Ripper method)



Introduction

Sulphur dioxide is present in wines as free SO_2 and also bound to acetaldehyde. The sum gives "total" sulphur dioxide. Bound SO_2 is released by addition of NaOH 1M to the wine sample.

This application is dedicated to free SO_2 measurement.

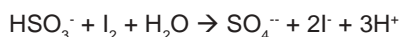
As a rule, determination of SO_2 in wines by the modified Ripper method uses a coloured indicator to determine the equivalence point, but it is also possible to use a pre-set end point titration with imposed current potentiometry.

With this end point method, many chemical functions present in the wine can modify the electrode behaviour and the reaction kinetics.

This application note gives a modified procedure suitable for many wines (white, red or rosé). We have tested various different wines but not all those available worldwide. This method has not been tested on special types of wine such as sparkling or sweet wines.

Principle

SO_2 is determined by titration with iodine solution according to the reaction



Free SO_2 is measured directly in acidic media.

The titration is run according to a pre-set end point titration with imposed current potentiometry and a double platinum wire electrode.

Results are expressed as SO_2 in mg/l.

Electrode and reagents

M231Pt2 Metal Electrode, double platinum wire (part no. E32M001) with adapter part no. A94P801 or M241Pt2-8 Metal Electrode without adapter (part no. E32M002)

Iodine solution **0.01 mol/l** (0.02 eq/l)

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 highly exothermic so perform the dilution very slowly and respect laboratory safety regulations. Let the solution cool down to room temperature. This solution is approximately 9N or 4.5M.

KI 5%

Dilute 50 g of potassium iodide in 1000 ml of distilled water.

NaHCO_3 solid form (see notes and remarks)

End Point titration settings

table {
border-collapse: collapse;
width: 100%;
}
tr {
border-bottom: 1px solid black;
}
tr {
td>Burette volume:
 10 ml |
}
tr {
 Stirring speed: | 400 rpm |
}
tr {
 Working mode: | mV with i = 1 µA (DC) |
}
tr {
 Number of end points: | 1 |
}
tr {
 End point: | 100 mV |
}
tr {
 Stirring delay: | 10 seconds |
}
tr {
 Minimum speed: | 0.2 ml/min |
}
tr {
 Maximum speed: | 5.0 ml/min |
}
tr {
 Proportional band: | 500 mV |
}
tr {
 End point delay: | 5 seconds |
}
tr {
 Sample unit: | ml |
}
tr {
 Sample amount: | 50 ml for free SO_2 |
}
tr {
 Titration: | Decreasing potential |
}
tr {
 Result: | mg/l |
}

Free Sulphur Dioxide in Wine (Modified Ripper method)

| Procedure | Using as titrant unit eq/l and 64 for molecular weight of SO ₂ | Results for free SO ₂ in red or rosé wines |
|---|--|---|
| Free SO₂ Pipette 5 ml of H ₂ SO ₄ solution 25% v/v into a low diameter beaker, add 10 ml of KI solution 5% and 50 ml of wine, and titrate quickly with 0.01M Iodine solution. Addition of NaHCO ₃ indicated in some local procedures can be avoided if titration occurs quickly. | Note that in this case, 2 eq. titrants (I ₂ corresponds to 2I) react with 1 sample. For a result in mg/l Enter The actual sample amount in the SAMPLE screen The titrant concentration in the TITRANT screen (in eq/l) 2 Titrants and 1 Sample in the COEFFICIENTS display 64 as molecular weight The Titration Manager gives a result according to the above formula. | (4 tests on each kind of wine) Grenache (red wine) Mean: 23.1 mg/l Stand dev.: 0.7 mg/l Result ⁽¹⁾ : 21.7 mg/l Merlot (red wine) Mean: 15.2 mg/l Stand dev.: 0.5 mg/l Result ⁽¹⁾ : 16.0 mg/l Syrah (red wine) Mean: 16.9 mg/l Stand dev.: 0.4 mg/l Result ⁽¹⁾ : 15.6 mg/l (Results verified free SO ₂ determination measured by aspiration method) ⁽¹⁾ (Result for aspiration method) |
| Results | Statistics | |
| Expressed as mg/l of SO₂ (MW of 64 g/mol) Using as titrant unit: mol/l (M) In this case, as 1 mole of titrant reacts with 1 mole of SO ₂ (or HSO ₃ ⁻) in the sample: $R = V(\text{titr}) * C(\text{titr}) * 64 * 1000 / V(\text{smp})$ -V(titr) = Total volume of titrant to reach the end point (in ml) -C(titr) = Concentration of titrant in mol/l 64 = Molecular weight of SO ₂ in g/mol -V(smp) = sample volume in ml For a result in mg/l with the Titration Manager Enter The actual 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 64 as molecular weight The Titration Manager gives a result according to the above formula. | For 5 determinations with a white wine Free SO₂ Mean: 35 mg/l SO ₂ Standard deviation: 0.7 mg/l SO ₂ Rel. standard deviation: 2% | Rosé wine (3 determinations) Mean: 20.3 mg/l Stand dev.: 0.2 mg/l Verification of the free SO₂ content by addition of Na₂SO₃ aqueous solution before titration (The solution S1 contains 12.0 g/l of Na ₂ SO ₃ or 6.2 mg/ml of SO ₂) 50 ml Rosé wine 20.3 mg/l 50 ml Rosé wine + 0.2 ml of S1 42.0 mg/l Theoretical 20 + 24 = 44 mg/l 50 ml of red wine 1 24.4 mg/l 50 ml of red wine 1 + 1 ml S1 126 mg/l Theoretical 24.4 + 120 = 144 mg/l 50 ml of red wine 2 29.7 mg/l 50 ml of red wine 2 + 0.1 ml of S1 41 mg/l Theoretical 29.7 + 12 = 42 mg/l 50 ml of red wine 2 + 0.2 ml of S1 51 mg/l Theoretical 29.7 + 24 = 54 mg/l |
| | Procedure for red and rosé wines With red wines, a secondary slow reactions between the iodine and tannin and other products can occur. Although addition of KI is not always necessary with white wines, it is compulsory for red and rosé wines . Addition of KI (potassium iodide) eliminates or reduces secondary slow reactions between iodine and reducing products present in red wine Titrate immediately using the titration settings above | |

Free Sulphur Dioxide in Wine (Modified Ripper method)

Note that the recovery of added SO_2 is not 100% because the wine immediately binds part of this " SO_2 ".

Working range

For free SO_2 determination, using a 0.01 mol/l titrant and 50 ml for sample volume, 1 ml of titrant corresponds to 12.8 mg/l SO_2

Automation of the free SO_2 determination

Using a sample changer (SAC80 15 position), it is not recommended to add H_2SO_4 and KI for too long before the titration. The KI/ H_2SO_4 mixture is not stable as in these conditions I^- is oxidised giving I_2 .

Add the KI solution manually to the different beakers and use a peristaltic pump for H_2SO_4 addition.

Tests were run using a Watson-Marlow Alitea 400 (040.1501.D1E) peristaltic pump fitted with silicone tubing (ext. diameter 7 mm int. diameter 4 mm). With this tubing and a speed setting of 6, it takes 12 seconds to deliver 10 ml (typical value).

Fit the silicone tubing in the head of the pump.

Dip one end of the tubing in the H_2SO_4 solution bottle and fit the other with a glass delivery tip for example.

Set the pump (potentiometer in 6 position and in CW or clockwise)

Connect the pump to the mains and let it run until the air bubbles in the tubing are eliminated

Disconnect the pump.

Connect the pump to the 5 V TTL output

Black plug of the Titration Manager to pin 8 of the external command plug

Red plug of the Titration Manager to pin 4 of the external command plug

Set the pump

Potentiometer in position 6 and in CW (clockwise) position

Connect the pump to the mains

The pump does not start as it is controlled by the titration manager.

Titration Manager settings

Use the above settings
Just add method parameters

Auxiliary output: 5 V
Aux. On for: 12 seconds

Note that use of twin head peristaltic pump allows simultaneous automatic addition of the two solutions at (H_2SO_4 25% and KI 5%) the beginning of the titration.

Notes

This method, named Ripper's method, is less accurate than the aspiration method because part of the iodine can be consumed by reducing substances other than SO_2 .

Using the aspiration method, SO_2 is removed from the sample by a stream of air through the acidified sample and oxidised in H_2SO_4 . Then H_2SO_4 is titrated by NaOH.

In some cases, the modified Ripper method can give higher results than expected with wines containing a significant amount of ascorbic acid because this acid (and some others) reacts quantitatively with iodine.

Adding NaHCO_3 forms a CO_2 blanket over the sample, which prevents oxygen interference during the titration. Addition should be avoided if the titration is run quickly in a covered beaker.

As it is easy to lose SO_2 during the preparation of the sample, make sure you titrate the sample immediately after preparation.

Imposed current values of 1 μA and 5 μA (DC) were tested without change in the results.

Titration concentrations corresponding to 0.01M and 0.05M were also tested without change in the titration speed reaction and in the results.

Bibliography

Techniques for chemical analysis and quality monitoring during wine making
Published by Patrick ILAND wines promotion Campbelltown AUSTRALIA

RADIOMETER ANALYTICAL S.A.

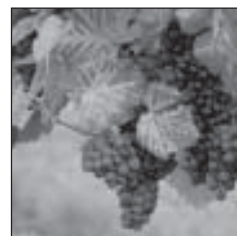
72 rue d'Alsace, 69627 Villeurbanne Cedex, France

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Tel.: +33 (0)4 78 03 38 38 - Fax: +33 (0)4 78 68 88 12

ClO⁻ Determination in Bleach

Direct Titration with NaAsO₂

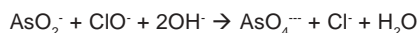


Introduction

A bleach solution contains mainly NaClO plus basic products such as NaOH and Na₂CO₃. ClO⁻ can be determined directly by redox titration. The titration result is generally expressed as a % of ClO⁻.

Principle

Direct titration of ClO⁻ by NaAsO₂ uses the following redox reaction



Note that in this reaction, which involves 1 mole ClO⁻ for 1 mole AsO₂⁻, 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)

NaAsO₂ 0.1 eq/l solution in water

To prepare the 0.1 eq/l (or 0.05 mol/l) NaAsO₂ solution

As a 0.1 eq/l NaAsO₂ solution contains 1/40 As₂O₃ mol/l

Weigh 4.2467 (169.87/40) g of pure As₂O₃ 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 H₂SO₄ 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 NaAsO₂ 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

ClO⁻ Determination in Bleach - Direct Titration with NaAsO₂

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:

$$R = V(\text{titr}) * C(\text{titr}) * 51.43 / V(\text{smp})$$

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 for-
mula.

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 commer-
cial 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 con-
centrated bleach corresponds to a
ClO⁻ concentration in g/l of around
80 (corresponding to a total vol-
ume of titrant of around 16 ml for
0.5 ml of sample), it is possible to
use the dilution procedure to facili-
tate pipetting or weighing 0.5 ml of
sample.

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

In the SAMPLE display enter

DILUTION: YES

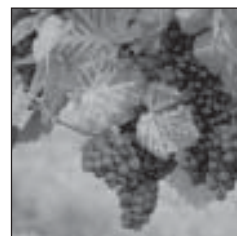
Sample: 5 ml

Final dilution volume: 100 ml

Aliquot: 10 ml

ClO⁻ Determination in Bleach

Indirect Titration

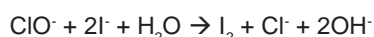


Introduction

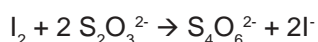
A bleach solution contains mainly NaClO plus basic products such as NaOH and Na₂CO₃. ClO⁻ can be determined by redox titration. Besides the direct titration by sodium arsenite; ClO⁻ determination can be run with a two-step titration; quantitative oxidation of I⁻ by ClO⁻ giving I₂ and titration of I₂ by sodium thiosulphate. The titration result is generally expressed as % of ClO⁻.

Principle

Oxidation of I⁻ by ClO⁻ follows the reaction



Titration of I₂ by sodium thiosulphate follows the reaction



The result of these 2 equations is 1 ion ClO⁻ corresponds to 2 ions S₂O₃²⁻

The titration of I₂ by S₂O₃²⁻ 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

Na₂S₂O₃ 0.1 eq/l or 0.1 mol/l (For concentration determination see separate application note)

Weigh exactly 24.8181 g of Na₂S₂O₃ · 5H₂O

Dissolve in 500 ml of freshly boiled distilled water

Add 2 or 3 drops of CHCl₃

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 Na₂S₂O₃ 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

ClO⁻ Determination in Bleach - Indirect Titration

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:

$$R = V(\text{titr}) * C(\text{titr}) * 51.43 / 2 * V(\text{smp})$$

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

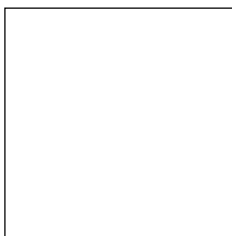
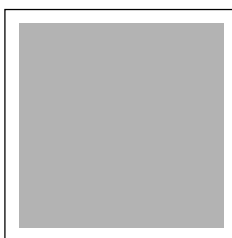
Sample: 5 ml

Final dilution volume: 100 ml

Aliquot: 10 ml

Bromine Number of Petroleum Products

(ASTM D1159-98 Electrometric titration)



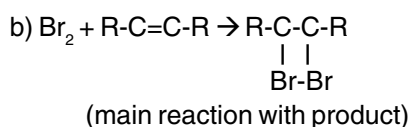
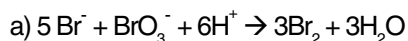
Introduction

This method covers bromine number determination of certain petroleum products indicated in ASTM standard D1159-98. The magnitude of the bromine number is simply an indication of the bromine reactive constituents, not an indication of the constituents.

The bromine number mainly indicates the concentration of double bonds present in the product.

Principle

Expressed as g of Bromine (Br_2) able to react with 100 g of product, the bromine number determination uses the reaction between a ($\text{Br}^-/\text{BrO}_3^-$) solution and the petroleum product according to the following reactions



(Note that other reactions can occur such as addition, substitution or oxidation)

To avoid secondary reactions the titration is run at low temperature (close to 5°C).

The titrant concentration, expressed as Br_2 concentration, is 0.25 mol/l (or $\frac{1}{4}$ mol/l). According to reaction (a), the titrant contains 5/12 mole of KBr and 1/12 mol/l of KBrO_3 . The molar weight of KBr is 119.9 g/mol and for KBrO_3 167.9 g/mol. Titration is run according to an inflection determination with imposed current potentiometry and a double platinum wire electrode.

Electrode and reagents

M241Pt2-8 Metal Electrode, double platinum (part no. E32M002) or

M231Pt2 Metal Electrode, double platinum (part no. E32M001) with adapter part no. A94P801

T201 Temperature sensor (part no. E51M001)

Water-jacketed titration beaker connected to a low temperature thermostat or a bath filled with ice

Bromide-Bromate titrant solution (0.25 mol/l as bromine concentration)

Dissolve 51.0 g of KBr ($5 \times 119.9/12$) and 13.92 g of KBrO_3 ($167.01/12$) in 1000 ml of distilled water using a volumetric flask.

Sulphuric acid (1+5)

Carefully mix one volume of concentrated sulphuric acid with 5 volumes of distilled water. Caution: the dilution reaction is exothermic.

Titration solvent (**see notes**)

Mix 714 ml of glacial acetic acid (CH_3COOH), 134 ml of dichloromethane (CH_2Cl_2), 134 ml of methanol (CH_3OH) and 18 ml of sulphuric acid (1+5).

Dichloromethane (CH_2Cl_2)

Warning: Reagents used in this application note are flammable. They can cause severe burns and are hazardous if swallowed, breathed or come into contact with the skin or eyes. Always respect laboratory health and safety regulations when using these reagents.

Bromine Number of Petroleum Products (ASTM D1159-98 Electrometric titration)

| Inflection Detection settings | Procedure | Results |
|--|---|--|
| CONTINUOUS IP Titration with blank Burette volume: 10 ml (see notes) Max. volume: 7 ml Stirring speed: 500 rpm Working mode: mV with $i > 0$ Current: DC Current value: 5 μ A (see notes) Blank: YES Start timer: 30 s Stop point: 0 mV Minimum speed: 0.50 ml/min Maximum speed: 2.00 ml/min Smoothing param: 8 Titration: Decreasing potential Inflection number: 1 Stop at last IP: YES Inflection 1: Min. ordinate: 200 mV Max. ordinate: 800 mV Sample Dilution: YES Sample unit: g Sample amount: 2 (see working range) Final dilution vol.: 50 ml Aliquot: 5 ml (see working range) Result 1: ml Equation 1: Equation Unit: Bromine number Equation: $(R1 \cdot CT \cdot 15.98 \cdot DA) / (SA \cdot AL)$ (see notes) | Connect the double platinum wire electrode to the corresponding input of the Titration Manager. Connect the temperature sensor to the corresponding input on the Titration Manager. For a new titration solvent batch run a BLANK titration with 115 ml of titration solvent and 5 ml of dichloromethane. The experimental blank volume is generally lower than 0.1 ml. <i>Sample preparation</i> Place 10 ml of dichloromethane in a 50 ml volumetric flask then add the weighed sample and fill the flask to the mark with dichloromethane. Add 110 ml of titration solvent to the titration beaker. Add an aliquot (generally 5 ml but not more than 10 ml) of the sample solution in dichloromethane. Dip the electrode, temperature sensor and delivery tip in the beaker and immerse the beaker in an ice bath or alternatively in a low temperature thermostatic bath. Using the electrode direct measurement function of the Titration Manager (icon ELECTRODES and "DISPLAY MEASUREMENT") allow the solution to reach the mentioned temperature (around 5°C) (see notes). Run the titration. | As indicated above, results are expressed in g of Br₂ able to react with 100 g of product . $R(\text{bromine number}) = \frac{(V_{\text{titr}} - V_{\text{blk}}) \cdot C(\text{titr}) \cdot 159.8 \cdot 100}{W(\text{smp}) \cdot 1000}$ $V_{\text{titr}} = \text{Total volume of titrant used in ml}$ $V_{\text{blk}} = \text{Blank volume used for solvent titration}$ $C(\text{titr}) = \text{Concentration of titrant in mol/l}$ $W(\text{smp}) = \text{Sample weight in g}$ 159.8 = molecular weight of Br ₂ 100 = conversion factor for 100 g of product 1000 = correction factor for result in g instead of mg For 5 determinations on cyclohexene (C₆H₁₀) Mean delivered volume: 3.42 ml Rel. standard deviation: 1.2% Corresp. Bromine number: 193 Theoretical: 194.6 Working range For a sample amount of 1g, and a calculated dilution coefficient of 10 (50 ml for final dilution volume and 5 ml for the aliquot), 1 ml of 0.25 mol/l titrant corresponds to a Bromine number of 37.5. The magnitude of the bromine number of a sample is often unknown and it is necessary to perform a preliminary test with 2 g of sample. ASTM Standard D1159 gives a table for sample amount as a function of Bromine number: |

| Bromine number | Sample amount in g | Titrant volume in ml |
|----------------|--------------------|----------------------|
| 0-10 | 20-16 | Around 4 |
| 10-20 | 10-8 | Around 3-4 |
| 20-50 | 5-4 | Around 5 |
| 50-100 | 2-1.5 | Around 4 |
| 100-150 | 1.6-0.8 | Around 3-4 |
| 150-200 | 0.8-0.6 | Around 3-4 |

Notes

Notes regarding the titration solvent

ASTM Standard D1159 gives trichloroethane as an alternative for dichloromethane. However, the use of this solvent is forbidden in many countries.

To save time, you can store the titration solvent in a refrigerator between the experiments.

Note regarding the maximum volume

To avoid a two-phase system during titration, it is not recommended to use a titrant volume higher than 10 ml.

Note regarding the imposed current

A 1 μA imposed current can give noisy curves and 10 μA gives a curve that is not well defined around the inflection point. Instead of DC imposed current it is possible to use AC imposed current. A 25 μA AC imposed current gives titration curves similar to those obtained with 5 μA DC current. In this case, also modify the "Br-Nb-Ctrl-Temp" method.

Note regarding the equation

The entered equation takes into account the programmed dilution of the sample. DA is the final dilution volume and AL the aliquot volume.

Note regarding sample handling

For sample and dichloromethane handling, you can use a glass syringe instead of a pipette.

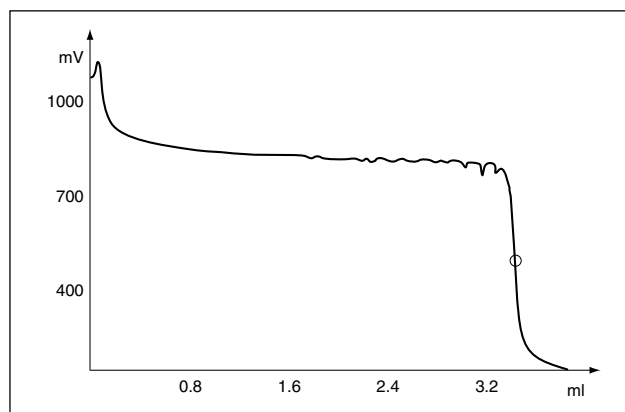
Note regarding temperature measurement

As indicated previously, you can use the DISPLAY MEASUREMENT function, but in this case you can just check the temperature of the solution (DISPLAY MEASUREMENT checks the measured potential on E1 and E2 inputs, not on Pt-Pt input). You can use this information in the pre-programmed method "Br-Nb-Ctrl-Temp" that measures the potential of the double platinum wire electrode AND the sample temperature.

Note regarding the curve shape

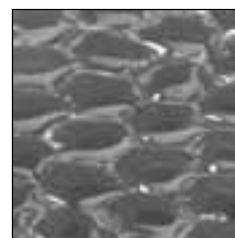
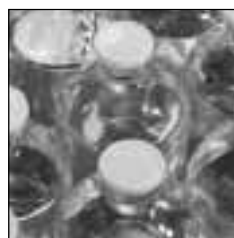
Based on experience, the starting potential may be around 1300 mV and suddenly falls to around 800 mV at the beginning of the titration. Then the curve falls very quickly to the final measured potential (generally around 100 mV) near the inflection point.

Curve



Iodometric Titration of Vitamin C

(pre-set end point and inflection detection methods)



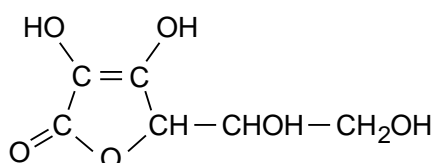
Introduction

Vitamin C (ascorbic acid or its sodium salt) is naturally present in fresh fruit juices or vegetables. It is also used in some pharmaceutical products. The food industry makes use of vitamin C as an anti-oxidation additive in cooked pork meats or canned products to avoid oxygen action (E300 for ascorbic acid or E301 for the sodium salt according to European regulations).

Summary

Many redox titrations can be used for Vitamin C determination. In some cases, depending on the media, iodometric back titration is possible, for example in fresh lemon juice or preservatives for food products.

Vitamin C formula

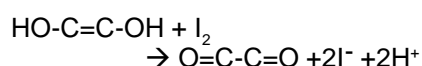


The redox titration is a two-step reaction.

First step

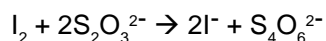
Oxidation of Vitamin C with excess iodine (I_2).

The redox reaction involves the two $-\text{OH}$ groups according to:



Second step

The iodine excess is then determined by sodium thiosulphate according to:



As results are generally expressed in mg/l, % or mg/kg, note that the molar weight of the Vitamin C is 176.3 g/mol corresponding to the global formula $\text{C}_6\text{H}_8\text{O}_6$. The molar weight of sodium ascorbate ($\text{C}_6\text{H}_7\text{O}_6\text{Na}$) is 198.3 g/mol.

Electrode and reagents

Electrode for pre-set end point titration

M241Pt2-8 Metal electrode, double platinum (part no. E32M002) or
M231Pt2 Metal electrode, double platinum (part no. E32M001) with adapter part no. A94P801

Electrode for inflection detection

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

Reagents

0.05 mol/l iodine solution:

Dissolve 40 g of potassium iodide in 50 ml of distilled water then add 12.69 g of analytical grade iodine, wait for complete dissolution and complete to 1.000 ml with a volumetric flask.

Store the solution in a brown glass bottle.

Iodometric Titration of Vitamin C (pre-set end point and inflection detection methods)

0.1 mol/l thiosulphate solution:

Dissolve 24.8181 g of $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ in 500 ml of freshly distilled water (or freshly boiled and cooled deionised water) and 2 or 3 drops of CHCl_3 (or 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.

These two solutions are commercially available.

Sodium acetate buffer solution

Dissolve 85 g of sodium acetate (CH_3COONa) in water, add 60 ml of glacial acetic acid and dilute to 1000 ml with freshly distilled water. This solution contains approximately 1 mole of CH_3COOH and 1 mole of CH_3COONa per litre.

Freshly distilled water.

End Point Titration settings

Burette volume: 25 ml
(for iodine solution)
Burette volume: 10 ml
(for thiosulphate solution)
Stirring speed: 400 rpm
Working mode: mV $i>0$
Current: DC
Current value: 5 μA
Start timer: 10 seconds
Maximum volume: 10 ml
Direction: Increasing mV
Minimum speed: 0.2 ml/min
Maximum speed: 2.00 ml/min

Back Titration: Automatic
(see Back titration note)
Excess reagent: I_2 0.05M
Excess volume: 15 ml

Number of end points: 1
End point: 200 mV
Proportional band: 190 mV
End point delay: 5 seconds

Sample unit: g or ml
Sample amount: Depending on the product (see below)

Result: %
Molar weight: 198 g/mol
Excess: 2Smp + 2Exc
Reaction: 1Exc + 2Titr
(see Result note)

Inflection Detection settings Continuous IP mode

Burette volume: 25 ml
(for iodine solution)
Burette volume: 10 ml
(for thiosulphate solution)
Stirring speed: 400 rpm

Working mode: mV
Start timer: 10 seconds
Maximum volume: 10 ml
Stop point: 150 mV
Direction: decreasing mV
Minimum speed: 0.2 ml/min
Maximum speed: 4.00 ml/min
Smoothing parameter: 5

Back Titration:
Automatic (see Back titration note)
Excess reagent: I_2 0.05M
Excess volume: 15 ml

Inflection No 1
Minimum ordinate: 230 mV
Maximum ordinate: 350 mV
Sample unit: g or ml
Sample amount: Depending on the product (see below)

Result: %
Molar weight: 198 g/mol
Excess: 2Smp + 2Exc
Reaction: 1Exc + 2Titr
(see note Result)

Procedure

Using a biburette Titration Manager, fill burette 1 with thiosulphate solution and burette 2 with iodine solution.

With a monoburette Titration Manager, fill the burette with thiosulphate solution, and add the iodine solution manually.

Connect the electrode.

Add 10 ml of sodium acetate buffer solution and, if necessary, freshly distilled water to the sample.

Dip electrode and delivery tip in the solution and run the titration.

Results

The general formula for result expression is:

$$C_{\text{smp}} = 1/K_{\text{smp}} * (C_{\text{exc}} * V_{\text{exc}} - (C_{\text{titr}} * V_{\text{titr}}/2))$$

- $1/K_{\text{smp}}$ is a function of the sample amount and unit

- C_{exc} concentration of the iodine solution in mol/l

- V_{exc} added volume of iodine solution in ml

- C_{titr} concentration of titrant solution in mol/l (thiosulphate solution)

- V_{titr} used volume of thiosulphate solution in ml

Using the above-mentioned coefficients for excess and reaction, the Titration Manager takes into account this formula for result calculation.

For 3 determinations with a food preservative

This is a mixture of sodium chloride, sodium acetate and vitamin C

Mean: 6.55%

Iodometric Titration of Vitamin C (pre-set end point and inflection detection methods)

Working range

Using an iodine solution with a concentration of 0.05 mol/l, 1 ml of the iodine solution reacts with 0.05 mmole corresponding to 8.81 mg of Vitamin C. Take this into account to determine the approximate amount of iodine solution necessary for the reaction and add an excess corresponding to approximately 50% of this volume.

For low concentrations, it is possible to use 0.005 mol/l iodine solution and 0.01 mol/l thiosulphate solution.

Notes

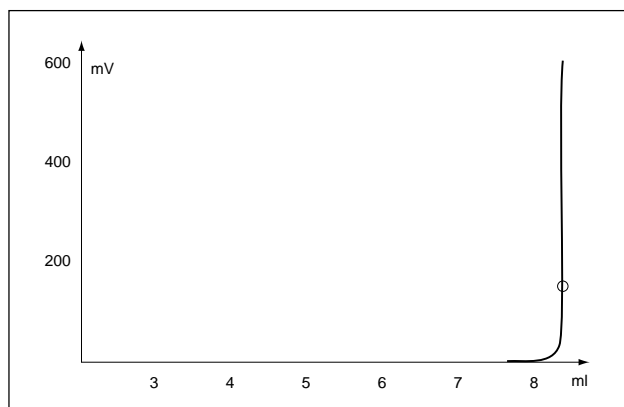
Back titration

Set "AUTOMATIC" using a bi-burette Titration Manager. With a monoburette system, set "MANUAL" and enter excess reagent concentration and volume. Other settings are unchanged.

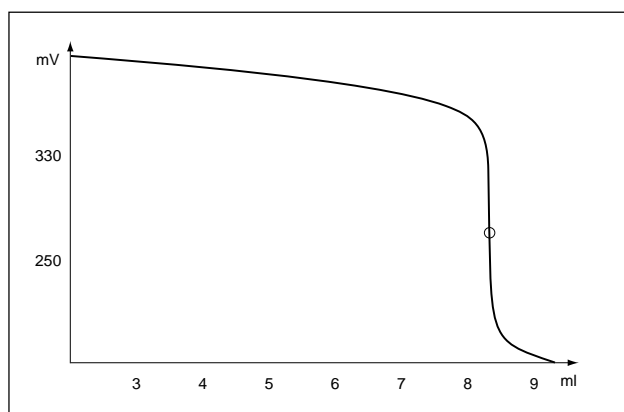
Result

Note that, in back titration, the Titration Manager asks for the SAME COEFFICIENTS for **sample and titrant**. It is therefore compulsory to set the titration system as indicated.

Curves



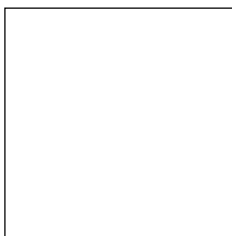
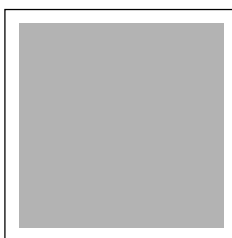
End point titration



Continuous IP mode

Copper in Electroplating Copper Baths

(Redox Titration Method)



Introduction

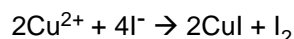
Electroless copper baths, used in particular in the manufacture of printed circuits, contain copper salts, sodium hydroxide stabilising agents and formol, but many electroplating copper baths contain acids (H_2SO_4) or cyanide ion.

For these baths, copper determination can involve a redox titration instead of E.D.T.A.

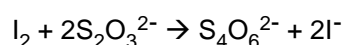
Principle

The redox titration of copper ion involves a two-step reaction:

First step: oxidation of iodide ion I^- by Cu^{2+} with iodine formation



Second step: titration of generated I_2 with thiosulphate ion



Regarding the 2 reactions, 1 mole of Cu^{2+} corresponds to 1 mole of $\text{S}_2\text{O}_3^{2-}$

The titration uses a combined platinum/reference electrode.

Electrode and reagents

MC3051Pt combined platinum/reference electrode (part no. E31M003)

CL114 connecting cable (part no. A94L114)

Potassium iodide solution

Dissolve 80 g of potassium iodide in 1000 ml of distilled water.

Glacial acetic acid

Sodium thiosulphate solution 0.1 mol/ (or 0.1N)

$\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ has a molecular weight corresponding to 248.181 g/mol

To prepare a 0.1 equivalent/l (or 0.1 mol/l) sodium thiosulphate solution; dissolve 24.8181 g of $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ in 500 ml of freshly distilled water (or freshly boiled and cooled desionised

water) and 2 or 3 drops of CHCl_3 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 and standardise again if necessary

This solution is also commercially available.

Distilled water.

Inflection Detection Settings

CONTINUOUS IP MODE

Burette volume: 10 ml
(see working range)

Stirring speed: 500 rpm

Working mode: mV
Start timer: 5 min
Maximum volume: 10 ml

Copper in Electroplating Copper Baths (Redox Titration Method)

Stop point: 170 mV
 Direction: decreasing mV
 Minimum speed: 0.1 ml/min
 Maximum speed: 5.00 ml/min
 Smoothing parameter: 5

Minimum ordinate: 200 mV
 Maximum ordinate: 320 mV
 Stop at last IP: YES

Sample unit: ml
 Sample amount: (see working range)
 Dilution: NO (see working range)

Result: g/l
 Molar weight: 159 g/mol
 Reaction: 2Smp + 2Titr

Procedure

For a sulphuric acid/Copper bath

For an expected concentration of CuSO_4 close to 75 g/l, pipette 1 ml of sample, add 10 ml of distilled water and 15 ml of CH_3COOH and complete to 60 ml with distilled water.

Stir the solution and add 5 ml of KI solution.

The solution becomes dark yellow and cuprous iodide precipitates.

Run the titration.

The start timer set to 5 minutes allows a complete reduction of Cu^{2+} .

At the end of the titration, the solution may be colourless with a white precipitate of cuprous iodide.

The complete sample preparation and titration procedure change according to the sample composition.

As a general rule, the copper reduction (first step reaction) and the iodine reduction (titration) take place in acidic media (pH 2.00/4.00).

Results

As indicated, 1 mole of Cu^{2+} corresponds to 1 mole of $\text{S}_2\text{O}_3^{2-}$

$$R(\text{g/l}) = V_{\text{titr}} * C_{\text{titr}} * 159.58 / V_{\text{smp}}$$

V_{titr} = Total volume of titrant used in ml

C_{titr} = Concentration of titrant in mol/l

V_{smp} = Sample volume in ml

159.58 = Molar weight of CuSO_4 in g/mol

3 determinations on the same bath

Mean: 74.20 g/l
 Standard deviation: 0.2 g/l

Working range

The table below can be used as a guideline according to the copper concentration:

| | | | |
|---------------------------|------|----|-----|
| CuSO_4 conc. g/l | 10 | 80 | 160 |
| Sample ml | 10 | 1 | 1 |
| Burette capacity ml | 10 | 10 | 25 |
| KI added | 5 | 5 | 10 |
| Titrant used | 6.25 | 5 | 10 |

This table takes into account the above-mentioned concentrations for reagents and titrant.

Instead of 1 ml for sample amount, it is possible to take 10 ml of sample diluted to 100 ml with a volumetric flask and use 10 ml of the diluted solution.

In this case, modify the Titration Manager settings as follows:

Sample unit: ml
 Dilution: YES
 Sample amount: 10
 Final dilution amount: 100
 Aliquot: 10

Notes

Procedure:

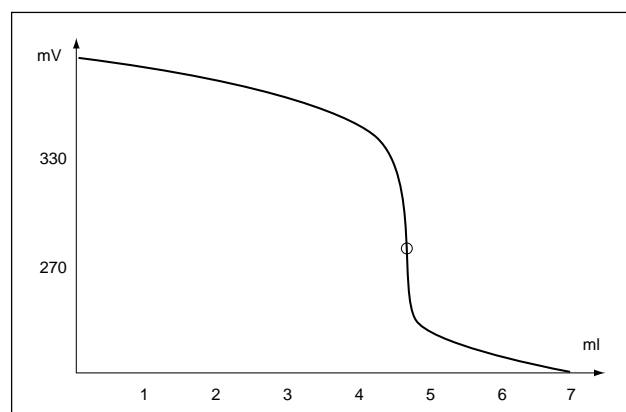
Addition of KSCN (potassium thiocyanate) avoids adsorption of I_2 on CuI and improves accuracy of the method.

Depending on the bath composition, it can be necessary to change the sample preparation. If the solution contains cyanide ion, add 3 ml of sulphuric acid and 1 ml of nitric acid to 1 ml of sample under a hood, gently

boil until white fumes are released then add water and ammonium hydroxide to pH 6.00 and then glacial acetic acid.

To obtain a well-shaped titration curve as shown in the graph and a correct result, make sure sufficient glacial acetic acid is added.

Curve



RADIOMETER ANALYTICAL SAS

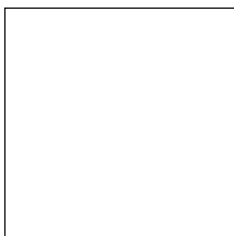
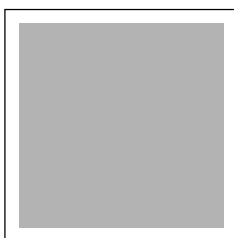
72 rue d'Alsace, 69627 Villeurbanne Cedex, France

E-mail: radiometer@analytical.com Web: www.radiometer-analytical.com

Tel.: +33 (0)4 78 03 38 38 - Fax: +33 (0)4 78 68 88 12

Peroxide Number of Edible Oils

(NF T 60-220 (1995) and ISO 3960 (2001))



Introduction

The peroxide number is a measurement of the concentration of (-O-O-) groups in edible oils. It is a measurement of the decomposition of the product and in many countries, official standards specify a maximum peroxide number beyond which the oil is unfit for human consumption. The peroxide number is therefore measured by oil manufacturers during production and after storage to check its preservation.

International standards use a redox titration in non-aqueous media, results are generally expressed in µg of peroxide (or active oxygen) per gram of product but mmoles/kg or meq of O₂/kg are also used.

The following two standards use the same titration principle but not the same solvents.

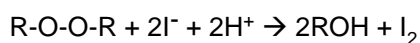
NF T 60-220 uses chloroform CHCl₃
ISO 3960 uses isooctane C₈H₁₈

According to these two standards, the equivalence point of the redox titration is determined using starch as colour indicator, but it is very easy to use potentiometric determination.

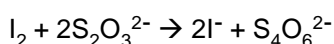
Summary

Peroxide number determination involves a two-step redox reaction:

1) Reaction of peroxide group with an excess of iodide ion according to:



2) Titration of Iodine with Na₂S₂O₃ solution (generally 0.01 or 0.02M) according to:



The titration is run according to inflection detection with continuous addition of the titrant with a combined platinum/reference electrode.

Electrode and reagents

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

Glacial acetic acid (CH₃COOH)
Chloroform (CHCl₃) (NF T 60-220)
Isooctane C₈H₁₈ (ISO 3960)

Solvent used for ISO 3960

Acetic acid/isooctane solution by mixing of 3 volumes of glacial acetic acid and 2 volumes of isooctane.

Saturated aqueous solution of potassium iodide (KI)

Note that the solubility of KI in water is approximately 150g/100 ml of water.

Do not prepare too a large volume of this solution. Store it in the dark.

Distilled water

Sodium thiosulphate solution 0.01M (or 0.02 mol/l) in water (Na₂S₂O₃)

To prepare a 0.01 mol/l (or 0.02 mol/l) sodium thiosulphate solution, dissolve 2.4818 g (or 4.9636 g) of Na₂S₂O₃ · 5H₂O in 500 ml of freshly distilled water (or freshly boiled and cooled deionised water). Add 2 or 3 drops of CHCl₃ (or 0.4 g of NaOH), as stabilising agent, and complete to 1000 ml using a volumetric flask.

Peroxide Number of Edible Oils (NF T 60-220 (1995) and ISO 3960 (2001))

Wait for one day and filter the solution if necessary (precipitation of sulphur can occur).

Stock the solution in a brown glass flask.

From time to time, look at the solution and filter or standardise again if necessary.

Na₂S₂O₃ · 5H₂O has a molecular weight corresponding to 248.181 g/mol.

As solutions with a concentration corresponding to 0.01M are not very stable, do not store this solution for more than 1 week.

As Sodium thiosulphate solution 0.1M is commercially available, you can prepare the 0.01 or 0.02M solution by dilution.

Check that solvents and reagents do not contain dissolved oxygen.

Bubbling nitrogen in the different solutions is one way to do this.

Inflection Detection settings

CONTINUOUS IP MODE

Titration with blank
Burette volume: 5 ml
(see working range)

Max. volume: 3.5 ml
Stirring speed: 500 rpm
Working mode: mV i=0
Blank: YES
Start timer: 10 s
Min. ordinate: 150 mV
Max. ordinate: 250 mV
Stop point: 0 mV

Minimum speed: 0.20 ml/min
Maximum speed: 3.00 ml/min
Smoothing parameter: 4

Titration: Decreasing potential
Inflection number: 1
Inflection 1:
Min. ordinate: 150 mV
Max. ordinate: 280 mV
Stop at last IP: YES

Sample Dilution: NO
Sample unit: g
Sample amount: 2
(see working range)

Results

Result 1:
Unit: mg/kg
Reaction: 1 sample + 2 Titrant
Molar weight: 16 g/mol
(see results)

No of equations: 2
Equation 1
Unit: mmoles O2/kg
Formula: R1/16
Equation 2
Unit: meq O2/kg
Formula: R1/8

Procedure

According to NF T 60-220

Use a stoppered titration vessel.

First run a blank titration according the following procedure but without sample.

Then run a titration.

Accurately weigh the necessary amount of edible oil.

Add 10 ml of chloroform and stir to dissolve.

Add 15 ml of glacial acetic acid and 1 ml of saturated potassium iodide solution.

Stop the titration vessel, stir for 1 minute and wait 5 minutes, keeping away from daylight.

Then add 75 ml of distilled water and titrate with 0.01 (or 0.02 mol/l) thiosulphate solution.

Procedure according to ISO 3960

The general procedure is exactly the same as for NF T 60-220.

Weigh the necessary sample amount.

Dilute with 50 ml of the isooctane/ acetic acid solution.

Add 0.5 ml of potassium iodide solution and stir for 1 minute then add 30 ml of distilled water.

Results

Expressed as µg/g (or mg/kg) of active oxygen the results corresponds to:

$$R = C_{\text{titr}} * V_{\text{titr}} * 16 * 1000 / 2 * W_{\text{smp}}$$

C_{titr} = Titrant concentration in mol/l

V_{titr} = Necessary titrant volume in ml

1000 = Constant to express the result in µg

16 = molar weight of oxygen

2 = As 2 moles of titrant correspond to 1 mole of sample

W_{smp} = Weighed amount of sample in g

Some other units can be used for result expression.

Result in mmoles/kg corresponds to: R/16

Result in meq of active oxygen/kg corresponds to: R/8

Results

3 determinations on old peanut oil

Mean: 192.8 mg/kg
(standard deviation: 2.6)

12.05 mmoles O2/kg
(standard deviation: 0.16)

24.10 meq O2/kg
(standard deviation: 0.33)

(blank determination: 0.107 ml

Peroxide Number of Edible Oils (NF T 60-220 (1995) and ISO 3960 (2001))

Working range

Use the following table which summarises the data in both standards to determine the necessary amount of sample:

| Peroxide number $\mu\text{g/g}$ or mg/kg | Peroxide number meq/kg | Sample amount (g) | Titrant volume (ml) |
|--|------------------------------------|----------------------|------------------------|
| 0-100 | 0-12 | 5.0-2.0 | 2.4 |
| 100-150 | 12-18.75 | 2.0-1.2 | 2.25 |
| 150-250 | 18.75-31.25 | 1.2-0.8 | 2.25-1.5 |
| 250-400 | 31.25-50 | 0.8-0.5 | 1.5-2.5 |
| 400-700 | 50-87.5 | 0.5-0.3 | 2.5-2.6 |

(*) Calculated with oleic acid molar weight

(**) For a 0.1M titrant

Notes

Procedure

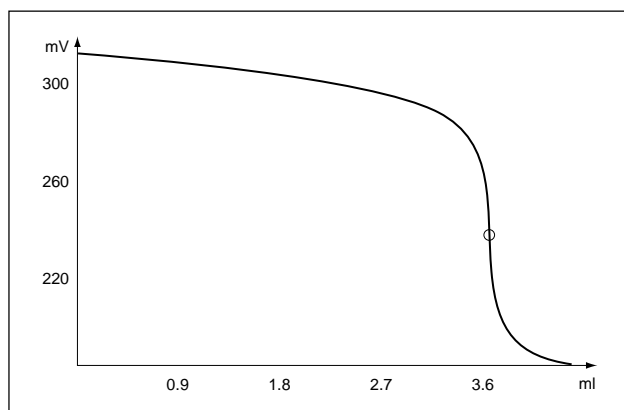
The main difficulty is to obtain reproducible results avoiding the influence of atmospheric oxygen.

Note that the saturated solution of potassium iodide is highly oxidisable. It is necessary to check this solution using the thiosulphate 0.01M solution according to the procedure described in the standards.

Low values for blanks are recommended (between 0.05 and 0.1 ml according to the standards).

After addition of water, the titration medium is a two-phase solution. Check you immerse the measuring electrode in the aqueous phase. Do not use too high a titration speed, because it is necessary to free all the iodine from the solvent layer.

Curve



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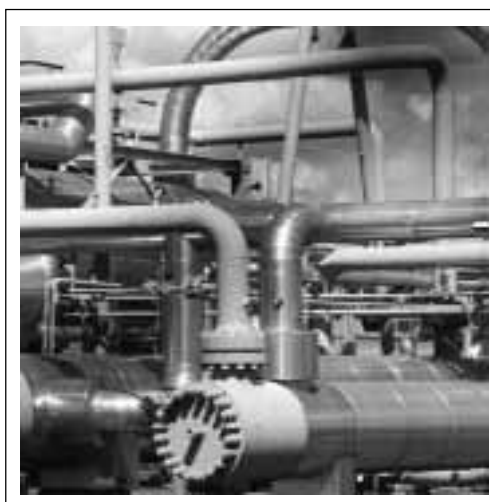
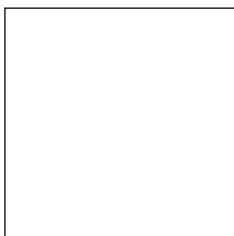
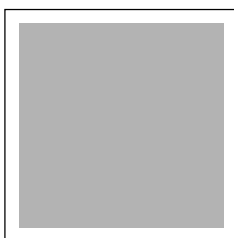
72 rue d'Alsace, 69627 Villeurbanne Cedex, France

E-mail: radiometer@analytical.com Web: www.radiometer-analytical.com

Tel. : +33 (0)4 78 03 38 38 - Fax: +33 (0)4 78 68 88 12

Hydrogen Peroxide Determination

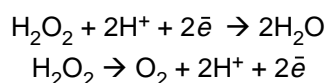
(by manganimetric titration)



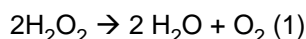
Introduction

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:

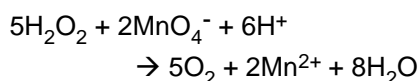


The global reaction corresponds to:



Principle

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



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:

$$\text{Titre (in volumes)} = (10 \cdot \text{titre (in \%)} / d) \cdot 11.2 / 34$$

34 = molar weight of H_2O_2

11.2 = volume of $\frac{1}{2}$ mole of O_2 under normal pressure and temperature conditions

d = H_2O_2 density

If density is supposed equal to 1:

$$\text{Titre (in volumes)} = \text{Titre (in \%)} \cdot 3.294$$

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: $R1 \cdot 3.294$
(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

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

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 %

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

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

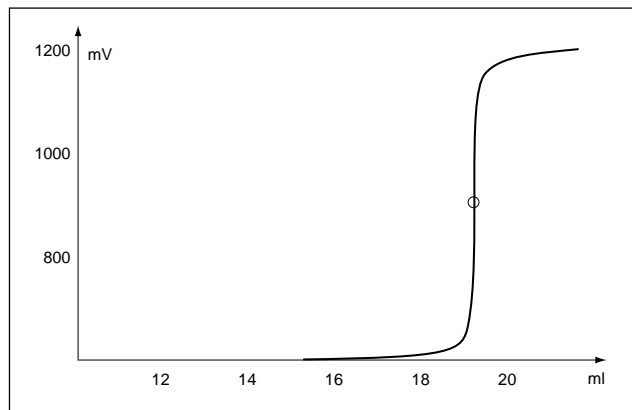
If H_2O_2 density is known modify to the equation as follows

$$\text{Equation} = R1 \cdot 3.924 / d$$

$d = \text{H}_2\text{O}_2$ density

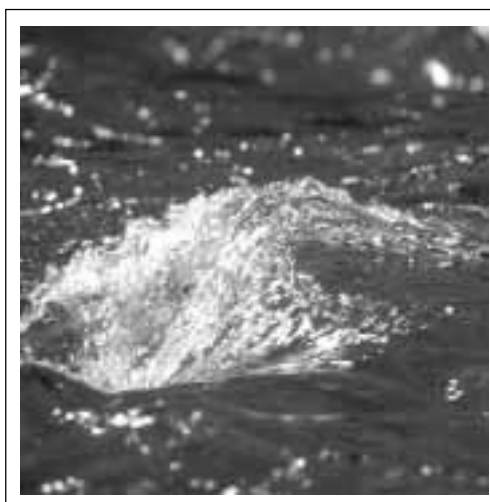
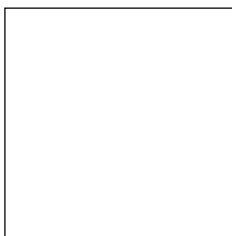
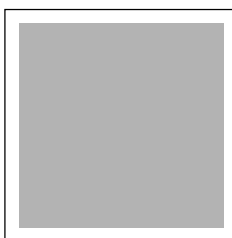
Hydrogen Peroxide Determination (by manganimetric titration)

Curve



Direct curve

Dissolved Oxygen in Water



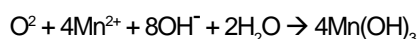
Introduction

Determination of dissolved oxygen (DO) is essential in water analysis, as oxygen is required for survival and growth by many organisms. For this determination, 2 standards are most commonly used based on the same procedure but using different reagent concentrations.

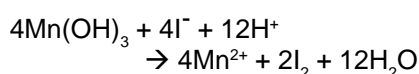
Principle

The principle used is a modified Winkler titration with a 3-step analysis.

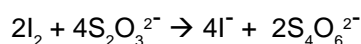
Step one: reaction between dissolved oxygen and divalent manganese Mn^{2+} according to



Step two: reduction of $Mn(OH)_3$ by I^- in acidic media according to



Step three: titration of I_2 using sodium thiosulphate according to



At the end, 1 molecule of O_2 corresponds to 4 molecules of $S_2O_3^{2-}$

The titration of I_2 by $S_2O_3^{2-}$ can be an end point titration using imposed current potentiometry and a two Pt-Pt wire electrode or an inflection

point titration with continuous addition mode.

Electrode and reagents

The reagent concentrations vary depending on the standard used (see table):

| European standard | US standard |
|---|---|
| SOL 1 $MnSO_4$, H_2O 380 g/l in water | SOL 1 $MnSO_4$, H_2O 364 g/l in water |
| SOL 2 KIO_3 0.3567 g/l in water (1/600 mol/l = 0.00167 mol/l = 0.01 eq/l) (for titrant calibration) | SOL 2 $KH(IO_3)_2$ 0.8124 g/l in water (0.0021 mol/l) (for titrant calibration) |
| SOL 3 $Na_2S_2O_3 \cdot 5H_2O$ 2.48 g/l in water (0.01 mol/l) 0.4 g/l NaOH (Titrant) | SOL 3 $Na_2S_2O_3 \cdot 5H_2O$ 6.205 g/l in water (0.025 mol/l) 0.4 g/l NaOH (Titrant) |
| SOL 4 H_2SO_4 50% vol./vol. in water | SOL 4 H_2SO_4 concentrated |
| SOL 5 NaOH 35 g NaI 27 g NaN_3 1 g In 100 ml of water | SOL 5 A B NaOH 480 g or NaOH 500 g NaI 750 g NaI 135 g NaN_3 10 g NaN_3 10 g In 500 ml of water In 1000 ml of water |

KI solid state (for titrant calibration)
Distilled water

Dissolved Oxygen in Water

Electrode for end point titration

MC231Pt2 Metal Electrode with two Pt-Pt wires with adapter part no. A94P801 (BNC 2 banana) or M241Pt2-8 Metal Electrode.

Electrode for inflection point titration

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

End Point Titration settings

Burette volume: 25 ml
Maximum volume: 25 ml
Working mode: mV with $i = 1\mu\text{A}$ (DC)
Stirring speed: 600 rpm

Number of end points: 1
End point: 200 mV
Stirring delay: 40 seconds
Minimum speed: 0.1 ml/min
Maximum speed: 2.5 ml/min
Titration: Increasing potential
Proportional band: 150 mV
End point delay: 5 seconds

Sample unit: ml
Dilution: YES
Sample amount: 300
Final dilution amount: 302
Aliquot: 200

Result1
Unit: mg/l
Reaction: 1 sample + 4 titrant
Molar weight: 32 g/mol

Inflection Detection settings Continuous IP mode

Burette volume: 25 ml
Maximum volume: 25 ml
Stirring speed: 600 rpm
Stirring delay: 10 s
Stop point: 120 mV
Smoothing parameter: 5
Inflection point number: 1
Minimum speed: 0.2 ml/min
Maximum speed: 5 ml/min
Direction: Decreasing mV
Stop at last IP: YES

Inflection 1
Min. ordinate: 200 mV
Max. ordinate: 450 mV

Sample unit: ml
Dilution: YES
Sample amount: 300
Final dilution amount: 302
Aliquot: 200

Result1
Unit: mg/l
Reaction: 1 sample + 4 titrant
Molar weight: 32 g/mol

Procedure

Set up the Titration Manager with SOL 3 as titrant.

Connect the two Pt/Pt wire or combined platinum/reference electrode.

Enter the titrant concentration (use mol/l or mmol/l) in the Titration Manager.

Add 1 ml of SOL 1 and 1 ml of SOL 5 to the sample collected in a bottle (generally 300 ml).

Stopper the bottle securely and mix the solution by turning the bottle upside down several times.

Leave the solution. When precipitate has settled sufficiently (half of the bottle) add 1 ml of SOL 4 and stopper the bottle. Mix the solution by turning the bottle upside down until dissolution is complete.

Pipette 200 ml of treated sample.

Dip electrode and delivery tip in the solution.

Start method by pressing the RUN key.

Results

Expressed as mg/l of O_2 as titrant concentration is expressed in mol/l

$$R = V(\text{titr}) * C(\text{titr}) * 32 * 1000 / V(\text{smp}) * 4$$

- $V(\text{titr})$ = Volume of titrant in ml
- $C(\text{titr})$ = Concentration of titrant in mol/l
- 32 = molecular weight of oxygen
- $V(\text{smp})$ = Actual sample volume used for titration
- 4 = Factor according to the fact that 1 molecule of O_2 corresponds to 4 molecules of $\text{S}_2\text{O}_3^{2-}$

For a result in mg/l of O_2 You need to take into account the dilution of the sample by reaction reagents as indicated in the Titration Manager settings

3 determinations on a water sample

Mean: 15.03 mg/l
Standard deviation: 0.108 mg/l
Relative standard deviation: 0.7%

Working range

Note that according to the above-mentioned formula, for **200 ml of sample** and with a **titrant concentration of 0.025 mol/l**:

1 ml of titrant corresponds to 1 mg/l of DO

The experimental limit of the method is about 2 mg/l.

Notes

SOL 5B is used for samples that are not highly saturated and SOL 5A for "supersaturated" samples. Addition of NaN_3 eliminates interference from NO_2^- ions. For other modifications necessary due to interference, refer to your local standards.

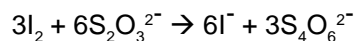
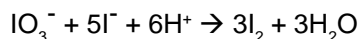
Titration calibration

If necessary, you can calibrate the sodium thiosulphate solution using the procedure described in application note TTEP01.08MIN (addition of solid state KI and concentrated acid before titration).

Dissolved Oxygen in Water

With KIO₃ (European standard)

In this case, 1 molecule of KIO₃ corresponds to 6 molecules of Na₂S₂O₃ according to



Pipette 10 ml of SOL 2, add 1 g of KI, 50 ml of distilled water and 5 ml of sulphuric acid 50% v/v solution.

Enter as standard amount: 10
Standard unit: ml
Standard conc.: 1.67 mmol/l (mM)

Result unit: mmol/l (mM)
Coefficients: 1 standard and 6 titrant

Depending on the titrant and standard concentrations indicated above, around 10 ml of sodium thiosulphate solution is required.

With KH(IO₃)₂ (US standard)

In this case, 1 molecule of KH(IO₃)₂ corresponds to 12 molecules of Na₂S₂O₃

Pipette 10 ml of SOL 2, add 1 g of KI, 50 ml of distilled water and few drops of concentrated sulphuric acid

Enter as standard amount: 10
Standard unit: ml
Standard conc.: 2.1 mmol/l (mM)

Result unit: mmol/l (mM)
Coefficients: 1 standard and 12 titrant

Depending on the titrant and standard concentrations indicated above, around 10 ml of sodium thiosulphate solution is required.

Bibliography

International standard ISO5813.
1983
European standard EN25813.
1992
Standard methods for water and wastewater 18th edition (1992)
4-98 part 4500

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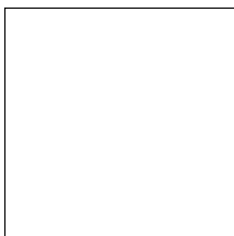
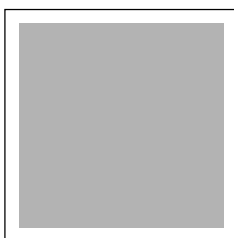
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Iodine Value of Animal and Vegetable Fats and Oils

(EN ISO 3961 (1999) and ISO 3961 (1996))



Introduction

The iodine value of animal and vegetable fats and oils measures the amount of -C=C- (double bonds) present in the product. The result is expressed as g of iodine (I_2) per 100 g of sample; the molar weight of I_2 is 253.8 g/mol. Instead of I_2 , ISO 3961 and EN ISO 3961 standards use Iodine chloride (I Cl) in acetic acid solution, also known as Wijs solution, for this determination.

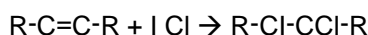
ISO and EN ISO standards use the reaction of the sample with an excess of Wijs solution followed by determination of excess of Wijs solution using a redox titration with sodium thiosulphate.

As indicated in standard ISO 3961, potentiometric determination of the equivalence point can be used.

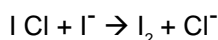
Summary

The iodine value determination involves a three-step operation:

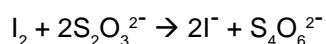
- 1) Reaction of sample with Wijs solution in excess according to



- 2) Reaction of the excess of Wijs solution with potassium iodide according to



- 3) Determination of the amount of released iodine according to



According to this three-step operation, the titration is a back titration with a blank.

This titration is run according to inflection detection with continuous addition of the titrant with a combined platinum/reference electrode, but it is also possible to work with a pre-set end point titration.

Electrode and reagents

MC3051Pt combined platinum/reference electrode (part no. E31M003)
CL114 connecting cable (part no. A94L114)

Or

M241Pt2-8 (part no. E32M002) or a M231Pt-2 Metal Electrode (part no. E32M001) with adapter part no. A94P801.

Wijs solution 0.1M (see note below)

This solution is commercially available (e.g. part no. 35 071 from Sigma Aldrich)

Titration solvent: Mix the same volumes of cyclohexane and glacial acetic acid.

Potassium iodide solution: Dissolve 100 g of potassium iodide in 1000 ml of pure water. Store the solution in a brown bottle avoiding oxidation of the iodide ion.

Sodium thiosulphate solution 0.1 mol/ (or 0.1N)

Na₂S₂O₃ · 5H₂O has a molecular weight corresponding to 248.181 g/mol.

To prepare a 0.1 equivalent/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 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/standardise again if necessary.

This solution is also commercially available.

Distilled or deionised water

Inflection Detection settings Continuous IP mode

Back titration with blank

Burette volume: 25 ml or 50 ml
(see "burette capacity" below)

Stirring speed: 700 rpm

Working mode: mV

Start timer: 20 sec

Blank: YES

Min. ordinate: 200 mV

Max. ordinate: 300 mV

Predose until:
(see "burette capacity" below)

Maximum volume: 25 ml or 50 ml

Stop point: 100 mV

Direction: decreasing mV

Minimum speed: 0.1 ml/min

Maximum speed: 5.00 ml/min

Smoothing parameter: 5

Back Titration: MANUAL
(see "back titration" below)

Excess reagent: Wijs solution

Excess volume: 20 ml

Excess titre: 0.1

Inflection1:

Minimum ordinate: 200 mV

Maximum ordinate: 300 mV

Stop at last IP: YES

Sample unit: g

Sample amount:
(see "working range" below)

Dilution: NO

Result: %

Molar weight: 253.8 g/mol

Excess: 2 Smp + 2 Exc

(see "result" below)

Reaction: 1 Exc + 2 Titr

Procedure (see notes)

For blank

For blank determination, prepare a solution in the same way as indicated below but without a sample.

For sample

Weigh the recommended amount of oil (see "working range")

Add the recommended volume of titration solvent (20 or 25 ml).

Add 20 ml (10 ml can sometimes be sufficient) of Wijs solution (see "Wijs solution" and "burette capacity").

Mix the solution and stopper the beaker (or the conical flask).

Leave the solution in the dark for 1 or 2 hours (refer to local standards) until the first step of the reaction is complete. Remember the reaction time depends on the nature of the sample and excess of Wijs solution if results are to be reproducible.

Next add 20 ml of the potassium iodide solution and 100/150 ml of deionised water.

If the procedure uses a conical flask with a ground glass stopper, it is possible to pour the solution into a beaker just before the titration, using deionised water to rinse the flask.

Titrate the blank followed by the corresponding sample.

Results

As the result is generally expressed in g of I₂ per 100 g of sample; according to the reactions, the Titration Manager calculates the result according to:

$$R = (V_{bl} - V_{smp}) * C_{titr} * 253.8 / 2 * W_{smp} * 10$$

V_{bl} = Titrant volume for blank in ml

V_{smp} = Titrant volume for sample in ml

C_{titr} = Titrant concentration in mol/l

253.8 = Molar weight of I₂

W_{smp} = Sample weight in g

2 = Stoichiometric factor of the reactions (1 mole of ICl corresponds to 1 mole of I₂ that corresponds to 2 moles of S₂O₃²⁻)

10 = Factor expressing result in %

For determination on old peanut oil

Iodine number: 80 g/100 g

Working range

Depending on the expected iodine value, look at the following table to determine the correct sample amount.

| Iodine value in % | Sample amount in g | Solvent in ml | $V_{bl} - V_{smp}$ in ml |
|-------------------|--------------------|---------------|--------------------------|
| <1.5 | 15 | 25 | 17 |
| 1.5 - 2.5 | 10 | 25 | 12 - 19 |
| 2.5 - 5 | 3 | 20 | 6 - 12 |
| 5 - 20 | 1 | 20 | 4 - 16 |
| 20 - 50 | 0.4 | 20 | 6.5 - 16 |
| 100 - 150 | 0.13 | 20 | 10 - 15 |
| 150 - 200 | 0.1 | 20 | 12 - 16 |

It is important to have a sample amount that corresponds to the volume of the Wijs solution.
The quantity of Wijs solution consumed by the sample should be close to 50% of the total amount of Wijs solution added.

Notes

Wijs solution

This solution is inflammable and corrosive.

Work under a hood

Never pipette this solution directly with the mouth, always use a pro-pipette.

Always consult the safety data sheet available from the product supplier before handling.

Burette capacity and maximum volume

According to the reaction between I_2 and I^- and titration of I_2 with $S_2O_3^{2-}$, if you use 20 ml of I_2 0.1M, the blank determination theoretically needs 40 ml of $S_2O_3^{2-}$ 0.1M. This should be taken into account when choosing the burette capacity (25 or preferably 50 ml), the maximum volume and the possible predose volume.

Use of the predose may be mandatory in continuous IP titration with a 25 ml burette capacity and saves time in end point titration

Back titration

As it is necessary to wait 1 or 2 hours for a complete reaction, do not use AUTOMATIC back titration.

Procedure

As the titration medium is a two-phase medium, it is advisable to use large diameter beakers and a stirring speed of around 700 rpm in order to obtain efficient mixing of the two phases. *Otherwise, some iodine may stay in the organic phase (becoming light violet coloured) giving a false result.* According to some publications, it is possible to shorten the reaction time by adding 10 ml of 2.5% mercuric acetate $[(CH_3COO)_2Hg]$ solution in water as catalyst after the Wijs solution.

Result

For a back titration, the Titration Manager asks for identical coefficients for Sample and Titrant.

END POINT TITRATION

As indicated in application note TTIP-EP02-01AFD (Iodometric titration of Vitamin C); it is possible to work with a M241Pt2-8 Metal Electrode (part no. E32M002) or a M231Pt-2 Metal Electrode (part no. E32M001) with adapter part no. A94P801, using imposed current potentiometry.

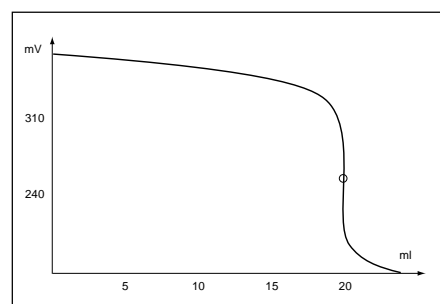
In this case, the Titration Manager settings should be modified as follows:

Working mode: mV ≥ 0
 Current value: 1 μA
 Direction: Increasing mV
 Minimum speed: 0.2 ml/min
 Maximum speed: 2.00 ml/min

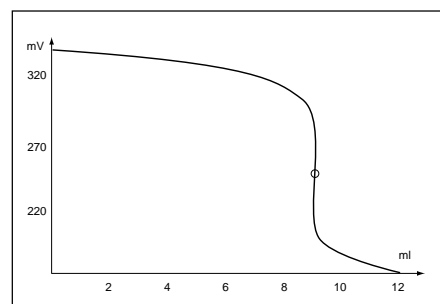
Number of end points: 1
 End point: 200 mV
 Proportional band: 190 mV
 End point delay: 5 sec

The other settings are similar to those already described.

Curves



Blank - Continuous IP mode



Sample - Continuous IP mode

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