

**Content and Language Integrated
Learning (CLIL) Materials in
Chemistry and English:
Iodometric Titrations.
Teacher's material**

LIDIA BARREIRO & TERESA NAVÉS

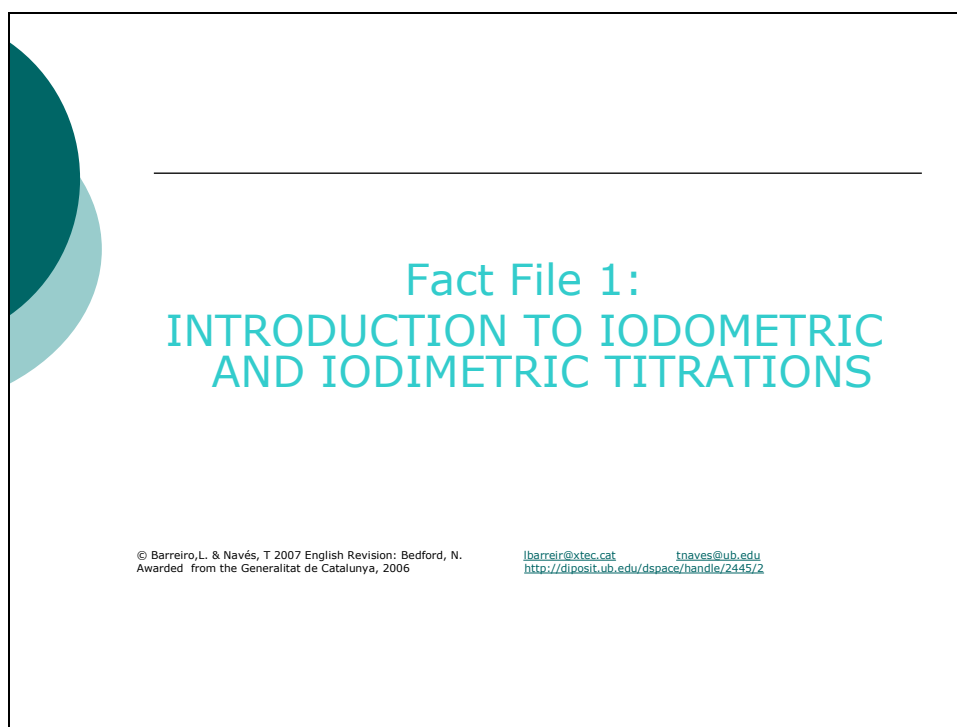
English Revision by Nick Bedford

TABLE OF CONTENTS: Teacher's material

Module	Teacher's material
Module 0. Introduction	MP3 Introduction Aims (CD)^(*) MP3 Input Source 0 (CD)^(*) MP3 Key Terms (CD)^(*)
Module 1. Getting ready for titrations	
Module 2. Iodometric and Iodimetric Titrations	Power Point Fact File 1 (CD)^(*) Fact File 1: Introduction to Iodometric and Iodimetric Titrations
Module 3. Iodometric Titration of Copper	
Module 4. The Sample: Copper wire	
Module 5. The Procedure for the Iodometric Titration of Copper	Power Point Fact File 2 (CD)^(*) Fact File 2: The Procedure for the Iodometric Titration of Copper
Module 6. The Experiment	

(*) Available in Teacher's material CD

Fact File 1: Introduction to Iodometric and Iodimetric Titrations

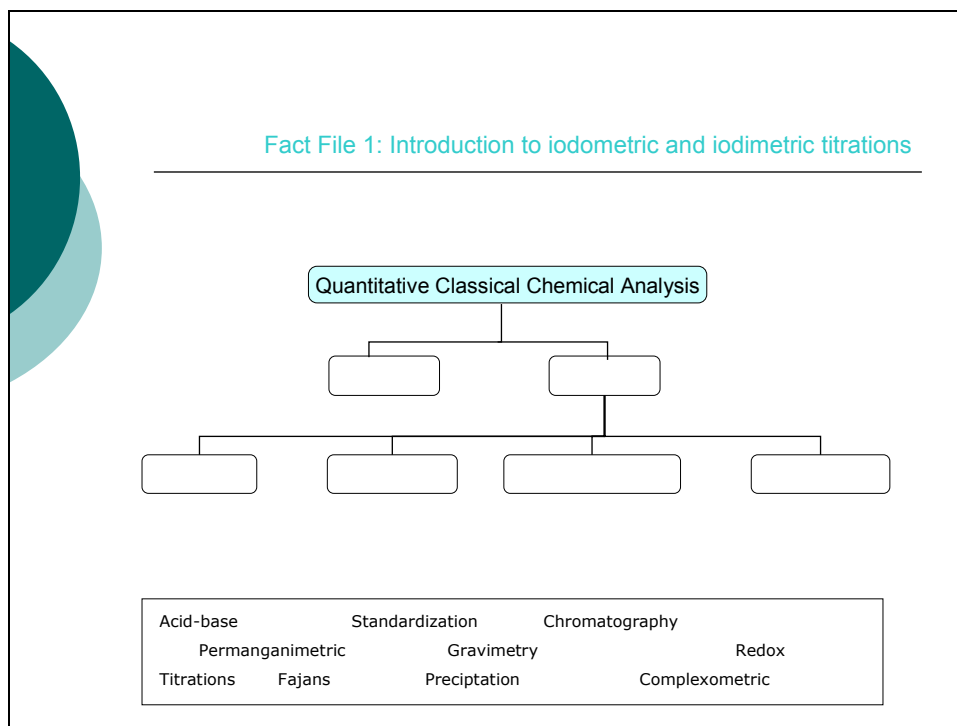


Slide 1:

- This presentation is an introduction to iodometric and iodimetric titrations.
- We are going to see the differences between both of the methods, the reactions which are involved and the specific titrants for the two types of titrations. Before seeing the differences between Iodometric and Iodimetric titrations, we are going to review some aspects about titrations and redox concepts.
- Your questions are welcome, feel free to interrupt.
- During the presentation I will ask you some questions and you will perform some short activities. Also, at the end you will do some activities to check that you have understood the essential information.

Activity 1

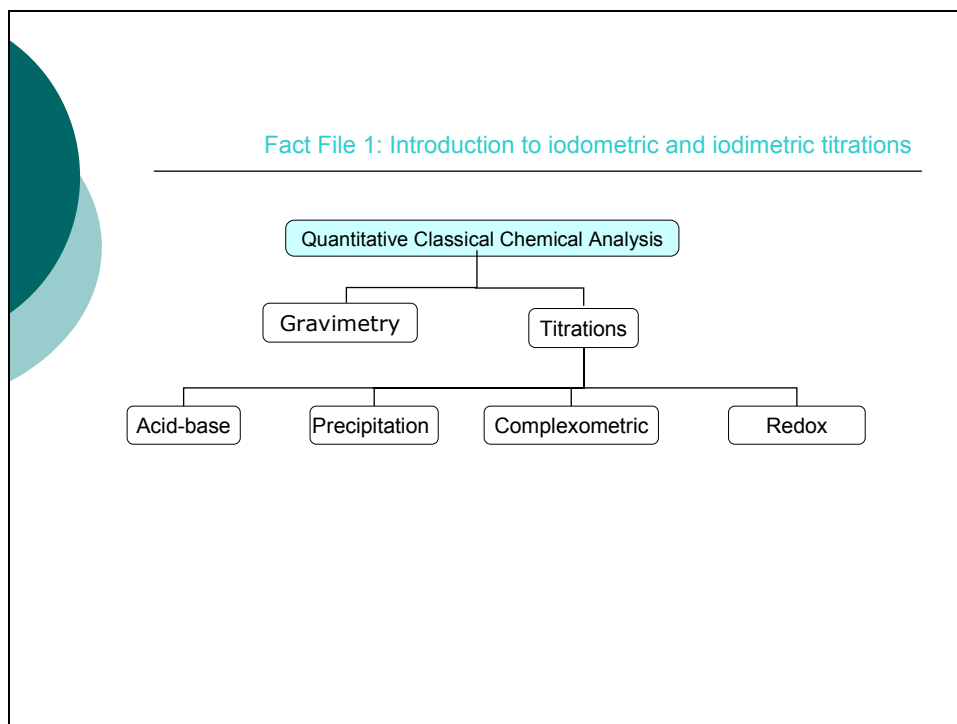
1. Work in pairs.
2. What is the aim of this presentation?

**Slide 2:**

- Before starting we are going to remember some tips about Quantitative Classical Chemical Analysis.

Activity 2

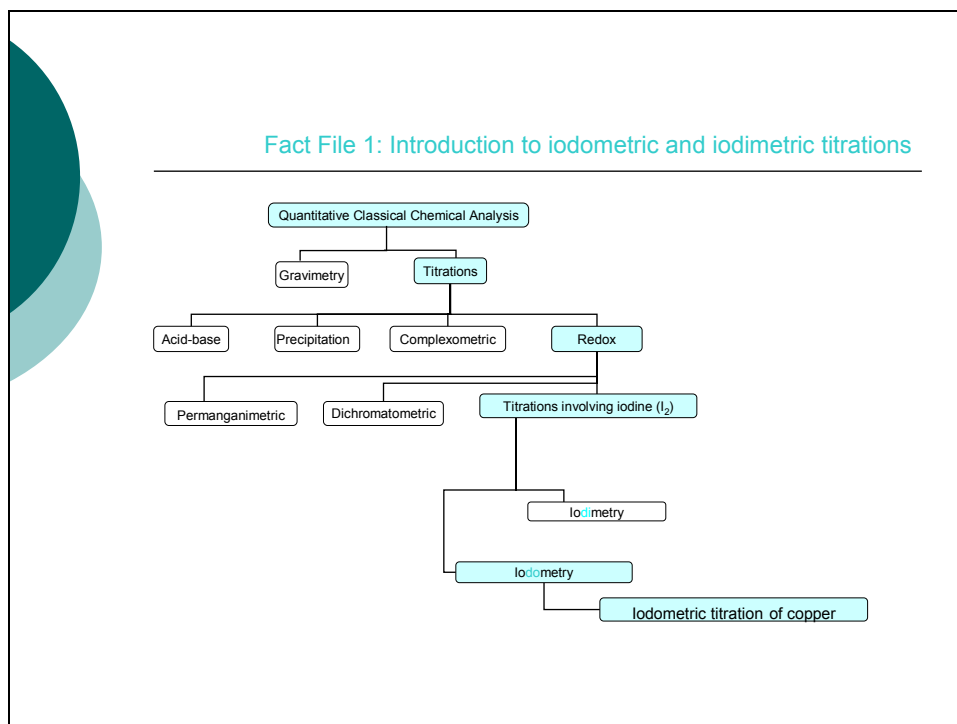
1. Work in pairs.
2. On the slide you can see an incomplete flow-chart.
3. Complete this flow chart with the most appropriate key term from the box. Pay attention, you only need 6 and there are 10!
4. Now, compare your answer with another pair. Do you have any differences?
5. What does this flow chart represent? Discuss it with your partner.

**Slide 3:**

- This flow chart represents the classification of Classical Quantitative Analysis.
- As you know, there are two types of Classical Quantitative Analysis: Gravimetry and titrations.
- And, what are the four types of titrations?
- Yes, remember the four types are:
- Firstly, acid-base titrations, secondly complexometric titrations, thirdly precipitation titrations and fourthly redox titrations.
- In textbooks about Titrations you will find first acid-base titrations, second precipitation titrations, third complexometric titrations and finally redox titrations.
- In this lecture we will be looking at one new type of redox titrations, titrations that create or consume iodine (I_2), that are widely used in quantitative analysis. We will see some examples of titrations involving iodine later.
- Talking about redox titrations:

Activity 3

1. Work in pairs.
2. What are permanganometric and dichromatometric methods?
3. Keep a record of your answer.
4. You have just 2 minutes.

**Slide 4:**

- Both, permanganimetric and dichromatometric are redox titrations. The difference between both is the titrant.
- In permanganimetric methods the titrant is potassium permanganate (KMnO_4).
- In dichromatometric methods the titrant is potassium dichromate ($\text{K}_2\text{Cr}_2\text{O}_7$).
- Look at the slide, as you know there are two types of Classical Quantitative Analysis: gravimetry and titrations.

Activity 4

1. Work in pairs.
2. What are the advantages of titrations in front of gravimetries?
3. Keep a record of your answer.
4. You have just 2 minutes.

- Gravimetry is not widely used as titrations.
- Titrations are more fast and they are widely used in chemical laboratories.
- As you know, there are four types of titrations: acid-base, complexometric, precipitation and redox.

- We are going to see in more detail redox titrations.
- There are a lot of redox titrations, on the slide there are the three redox titrations more used: permanganimetric, dichromatometric and Titrations involving iodine.
- Titrations involving iodine are the focus of this lecture.
- There are two types of titrations involving iodine: iodometric and iodimetric.
- In this lecture we will be looking at iodometric and iodimetric titrations in detail. At the end of this lecture you will be able to identify the type of sample, standard solution, indicator and reactions involved in idometric and iodimetric titrations.
- As an example of Iodometric analysis we are going to quantify copper in a sample.



Fact File 1: Introduction to iodometric and iodimetric titrations

Titration	Examples
Acid-base	<i>Quantification of acetic acid in vinegar</i>
Complexometric	
Precipitation	
Redox	

Slide 5:

- Before looking in detail the Iodometric and Iodimetric methods, let's review some examples of titrations.

Activity 5

1. Work in groups of three.
2. Complete the table with an example for each type of titration.
3. You have 1 minute.
4. Now, check your answer with another group. If there are new examples complete the table.

Fact File 1: Introduction to iodometric and iodimetric titrations

Titrations	Examples
Acid-base	Quantification of acetic acid in vinegar
Complexometric	Quantification of chloride (Cl ⁻) in water
Precipitation	Water Hardness (Calcium and magnesium)
Redox	Quantification of hydrogen peroxide (H ₂ O ₂)

Slide 6:**Activity 6**

1. Work in the same group of three.
2. Have you the same examples?
3. Tell the class the different ones.

Fact File 1: Introduction to iodometric and iodimetric titrations

	Titration example	Analyte	Titrant	Indicator
Acid-base	<i>Quantification of acetic acid in vinegar</i>	<i>Acetic acid (CH₃COOH)</i>	<i>Sodium hydroxide (NaOH)</i>	<i>Phenolphthalein</i>
Complexometric	<i>Quantification of chloride (Cl⁻) in water</i>			
Precipitation	<i>Water Hardness (Calcium and magnesium)</i>			
Redox	<i>Quantification of hydrogen peroxide (H₂O₂)</i>			

Slide 7:

Activity 7

1. Work in the same group of three.
2. Indicate for each example the Analyte, the Titrant and the indicator.
3. You have 2 minutes.
4. Check the answer with the class.

Comment: Maybe there is a type of titration not performed by your students.

Fact File 1: Introduction to iodometric and iodimetric titrations


	Titration example	Analyte	Titrant	Indicator
Acid-base	Quantification of acetic acid in avinegar	Acetic acid (CH ₃ COOH)	NaOH (sodium hydroxide)	Phenolphthalein
Complexometric	Water Hardness (Calcium and magnesium)	Calcium and magnesium (Ca ²⁺ , Mg ²⁺)	EDTA	Eriochrome black T Murexide
Precipitation	Quantification of chloride (Cl ⁻) in water	Chloride	AgNO ₃ (silver nitrate)	Mohr, Volhard, Fajans
Redox	Quantification of hydrogen peroxide (H ₂ O ₂)	Hydrogen peroxide (H ₂ O ₂)	KMnO ₄ (potassium permanganate)	No indicator

Slide 8:

- In this slide you can see the answer of the activity 7.

Activity 8

1. Work in pairs.
2. How could be the reaction between the analyte and the titrant?
3. Keep a record of your answer.



Fact File 1: Introduction to iodometric and iodimetric titrations

Titration:

- Direct Titrations
- Indirect Titrations
- Back Titrations
- Iodometry

Slide 9:

According to the reaction between the analyte and the titrant we can classify the titrations as:

Activity 9

1. Work in pairs.
2. Do you remember what direct titrations are?
3. Do you remember what indirect titrations are?
4. Do you remember what back titrations are?

- Direct Titration: A reaction between the analyte and the titrant.
- Indirect Titration: The analyte forms a compound with the species that reacts with the titrant.
- Back Titration: A known excess of titrant is added to the analyte. The excess of titrant is titrated with a standard solution.
- Iodometry: This type is new for you. We will see it in a minute.

Fact File 1: Introduction to iodometric and iodimetric titrations

Titration	Example	Type of reaction
Acid-base	Quantification of acetic acid in vinegar	<input type="checkbox"/> Direct Titration <input type="checkbox"/> Indirect Titration <input type="checkbox"/> Back Titration
Complexometric	Water Hardness (Calcium and magnesium)	<input type="checkbox"/> Direct Titration <input type="checkbox"/> Indirect Titration <input type="checkbox"/> Back Titration
Precipitation	Quantification of Cl in Water	Mohr Method <input type="checkbox"/> Direct Titration <input type="checkbox"/> Indirect Titration <input type="checkbox"/> Back Titration
		Fajans Method <input type="checkbox"/> Direct Titration <input type="checkbox"/> Indirect Titration <input type="checkbox"/> Back Titration
		Volhard Method <input type="checkbox"/> Direct Titration <input type="checkbox"/> Indirect Titration <input type="checkbox"/> Back Titration
Redox	Quantification of hydrogen peroxide (H ₂ O ₂)	<input type="checkbox"/> Direct Titration <input type="checkbox"/> Indirect Titration <input type="checkbox"/> Back Titration

Slide 10:

Activity 10

1. Work in the same group of three.
2. For each example tick the type of titration. If there are some examples that you don't know, guess the type of reaction: Direct Titration, Indirect Titration, Back Titration.
3. You have just 2 minutes.

Comment: Maybe there is a type of titration not performed by your students. But it could be interesting that they try to guess the type of reaction.

Fact File 1: Introduction to iodometric and iodimetric titrations


Titration	Example	Type of reaction
Acid-base	Quantification of acetic acid in vinegar	<input checked="" type="checkbox"/> Direct Titration <input type="checkbox"/> Indirect Titration <input type="checkbox"/> Back Titration
Complexometric	Water Hardness (Calcium and magnesium)	<input checked="" type="checkbox"/> Direct Titration <input type="checkbox"/> Indirect Titration <input type="checkbox"/> Back Titration
Precipitation	Quantification of Cl in Water	Mohr Method <input checked="" type="checkbox"/> Direct Titration <input type="checkbox"/> Indirect Titration <input type="checkbox"/> Back Titration
		Fajans Method <input checked="" type="checkbox"/> Direct Titration <input type="checkbox"/> Indirect Titration <input type="checkbox"/> Back Titration
		Volhard Method <input type="checkbox"/> Direct Titration <input type="checkbox"/> Indirect Titration <input checked="" type="checkbox"/> Back Titration
Redox	Quantification of hydrogen peroxide (H ₂ O ₂)	<input checked="" type="checkbox"/> Direct Titration <input type="checkbox"/> Indirect Titration <input type="checkbox"/> Back Titration

Slide 11:

- As you can see in the slide, all are direct titrations except the Volhard Method.
- It is time now to see in more detail what a direct titration is.

Activity 11

1. Work in the same group of three.
2. Give an example of back titration that you have performed in the laboratory?



Fact File 1: Introduction to iodometric and iodimetric titrations

There are a lot of **redox titrations** classified according to the **titrant** used.

- 1) **Permanganimetric: Titrant KMnO_4**
- 2) **Dichromatometric: Titrant $\text{K}_2\text{Cr}_2\text{O}_7$**
- 3) **Titration**s involving **iodine (I_2)**
 - **Iodimetry**
 - **Iodometry**

Titrations that create or consume I_2 are widely used in **quantitative analysis**.

Slide 13:

- Now, let's move on the aim of this lecture Iodometric and Iodimetric titrations.
- As we saw in previous slides, we classify redox titrations according to the titrant which is being used.
- There are a lot of redox titrations and the most common ones are:
- First we have Permanganimetric redox titrations where the titrant is KMnO_4 (Potassium permanganate).
- Then we have dichromatometric redox titrations where the titrant is $\text{K}_2\text{Cr}_2\text{O}_7$ (Potassium dichromate).
- And finally the titrations that interest us today are the titrations involving iodine (I_2). There are two types of titrations involving iodine (I_2).
- Iodimetry –that is, i-o-DI-metry
- Iodometry – and that is i-o-DO-metry
- As we said before titrations that create or consume iodine are widely used in quantitative analysis.

Activity 13

1. Work in pairs.
2. Which is the characteristic of Iodimetric and Iodometric Titrations?
3. Keep a record of your answer.

Fact File 1: Introduction to iodometric and iodimetric titrations

A **reducing agent** is the element or compound in a redox reaction that reduces another species. In doing so, it becomes *oxidized*, and is therefore the electron donor in the redox.

Examples of reducing agents:

The active metals sodium, magnesium, aluminum, and zinc,

NaH, CaH₂, and LiAlH₄, which formally contain the H⁻ ion.

Slide 14:


Before continue with Iodometric and Iodimetric titrations, it's time now to remember what reducing and oxidizing agents are?

Activity 14

1. Work In pairs.
2. Do you remember what a reducing agent is?
3. Give two examples of reducing agents.
4. Keep a record of your answer.

- Reducing agents lose electrons and doing that reduces the other species.

- Examples of reducing agents: The active metals, such as sodium, magnesium, aluminum, and zinc. Metal hydrides, such as NaH, CaH₂, and LiAlH₄, which formally contain the H⁻ ion, are also good reducing agents.



Fact File 1: Introduction to iodometric and iodimetric titrations

An **oxidizing agent** is the element or compound in a **redox** reaction that oxidizes another **species**. In doing so, it becomes *reduced*, and is therefore the element or compound that gain electrons.

Examples:

permanganate (MnO₄⁻), chromate (CrO₄²⁻), and dichromate (Cr₂O₇²⁻) ions, sodium hypochlorite (bleach) as well as nitric acid (HNO₃), perchloric acid (HClO₄), and sulfuric acid (H₂SO₄)

Slide 15:

Activity 15

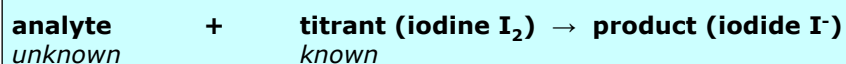
1. Work in pairs.
2. Do you remember what an oxidizing agent is?
3. Give four examples of oxidizing agents.



Fact File 1: Introduction to iodometric and iodimetric titrations

When a reducing **analyte** is titrated with iodine (the titrant), the method is called **iodimetry**.

Iodimetry: A direct titration with only 1 reaction:



Slide 16:

- A Iodimetry is a titration where the analyte is a reducing agent and the titrant is iodine.
- As we said, a reducing agent is the substance that reduces species. In this case, the reducing analyte reduces iodine to iodide, one of the products.
- Something very important to remember is that Iodimetry titrations are direct titrations. Consequently, there is only one reaction between the analyte and the titrant.
- In Iodimetric titrations the titrant is a standard solution of iodine (I₂).

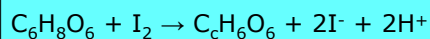
Activity 16

1. Work in pairs.
2. What is the main characteristic of the analyte in Iodimetric Titrations?
3. Keep a record of your answer?

Lecture 1: Introduction to iodometric and iodimetric titrations

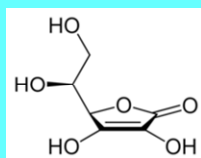
When a reducing **analyte** is titrated with iodine (the titrant), the method is called **iodimetry**.

Example: Quantification of Ascorbic Acid (Vitamin C)

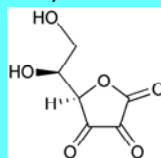


Iodine rapidly oxidizes ascorbic acid, $\text{C}_6\text{H}_8\text{O}_6$, to produce dehydroascorbic acid, $\text{C}_6\text{H}_6\text{O}_6$.

Ascorbic acid



Dehydroascorbic acid



Pictures taken from: <http://en.wikipedia.org>

Slide 17:

Activity 17

1. Work in pairs.
2. Identify the analyte, the titrant and the product of the example of an iodimetric titration: Quantification of Ascorbic Acid?
3. Keep a record of your answer.

Lecture 1: Introduction to iodometric and iodimetric titrations

Iodometry is the titration of iodine (I_2) produced when an oxidizing analyte is added to excess I^- (iodide).

Then the iodine (I_2) is usually titrated with standard **thiosulfate** solution.

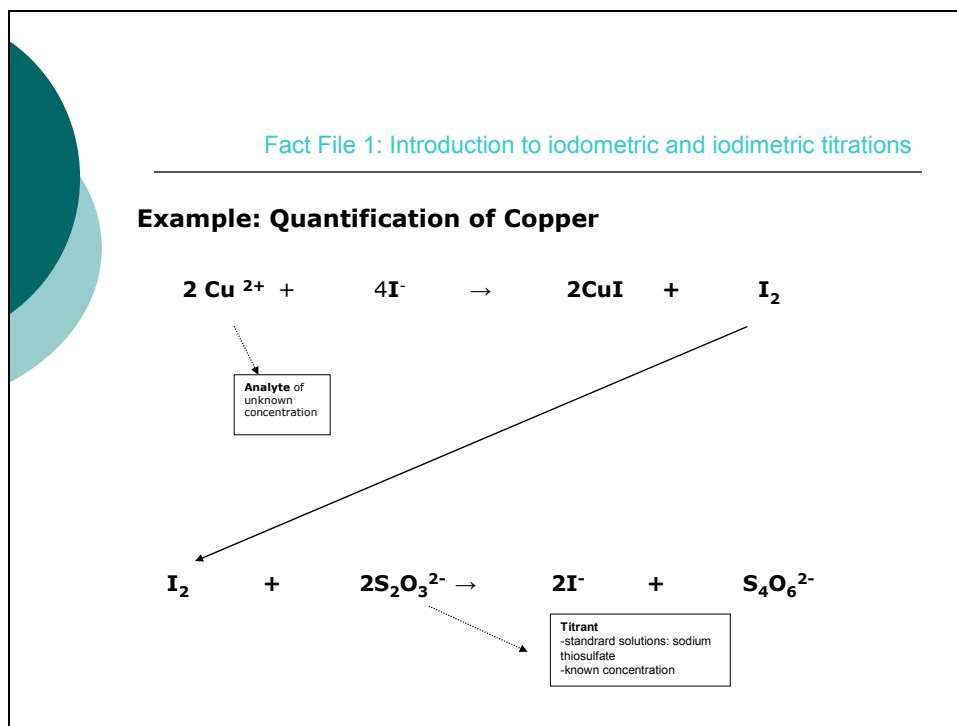
Iodometry: Not a direct titration because there are 2 reactions:

**Slide 18:**

- Now let's move on to iodometric titrations
- Unlike iodimetric titrations, in the iodometric ones two reactions are involved.
- Let's analyze these two reactions.
- In this case the analyte is an oxidizing agent. Consequently, in the first reaction the analyte oxidizes iodide to iodine.
- In the second reaction the iodine is titrated with standard thiosulfate solution.

Activity 18

1. Work In pairs.
2. What is the main characteristic of the analyte in Iodometric Titrations?
3. Keep a record of your answer?

**Slide 19:**

- The Iodometric Titration of Copper, the procedure that we are going to perform in the laboratory is our example of Iodometric titrations.

Activity 19

1. Work in pairs.
2. Identify the analyte and the titrant of this procedure.
3. Keep a record of your answer.

Fact File 1: Introduction to iodometric and iodimetric titrations

Iodimetric titrations:

- a) A reducing analyte
- b) One reaction
- c) Standard solution: Iodine (I_2)

Iodometric titrations:

- a) An oxidizing analyte
- b) Two reactions
- c) Standard solution: Sodium thiosulfate

Slide 20:**Activity 20**

1. Work in groups of three.
2. List the three main characteristics of Iodimetric and Iodometric Titrations: a) type of analyte, b) number of reactions, c) standard solution.
3. Keep a record of your answer.

So, to summarise the main information about iodimetric and iodometric titrations:

In iodimetric titrations:

- The analyte is a reducing agent.
- One reaction is involved.
- The standard solution is Iodine (I_2)

On the other hand in iodometric titrations:

- The analyte is an oxidizing agent.
- Two reactions are involved.
- The standard solution is sodium thiosulfate.

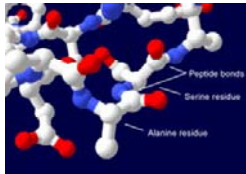
Fact File 1: Introduction to iodometric and iodimetric titrations

Analytical applications:

Iodimetric titrations:

Species analyzed (reducing analytes)

SO₂
 H₂S
 Zn²⁺ , Cd²⁺ , Hg²⁺ , Pb²⁺
 Cysteine, glutathione, mercaptoethanol
 Glucose (and other reducing sugars)




Section of a protein structure
 Source: <http://en.wikipedia.org>

Slide 21:

- Now, let's see some examples of analytes which we can quantify by iodimetric titrations.
- Some of the analytes, which are reductant agents are:
 - SO₂ (sulphur dioxide)
 - H₂S (sulphidric acid)
 - Ions like Zn²⁺ , Cd²⁺ , Hg²⁺ , Pb²⁺ (zinc, cadmium, mercury, lead)
 - Aminoacids like Cysteine
 - Peptids (units that form proteins) like glutathione
 - Proteins like mercaptoethanol
 - Note: Proteins are large organic compounds made of amino acids arranged in a linear chain and joined together by peptide bonds. In the figure you can see a protein molecular model.
- And lastly, we can also quantify reducing sugars such as glucose.

Activity 21

1. Work in pairs.
2. In which type of samples should you quantify glucose?
3. Keep a record of your answer.



Fact File 1: Introduction to iodometric and iodimetric titrations

Analytical applications:

Iodometric titrations:

Species analyzed (oxidizing analytes)


- HOCl
- Br₂
- IO₃⁻, IO₄⁻
- O₂, H₂O₂, O₃
- NO₂⁻
- Cu²⁺
- MnO₄⁻, MnO₂

Slide 22:

- With iodometric titrations we can quantify analytes, which are oxidizing agents like:
 - HOCl: Hypochlorous acid (also known as bleach)
 - Br₂ Bromine
 - IO₃⁻ (iodate ion), IO₄⁻ (periodate ion)
 - O₂ (oxygen), H₂O₂ (hydrogen peroxide), O₃ (ozone)
 - NO₂⁻ (nitrite)
 - Cu²⁺ (copper ion)
 - MnO₄⁻ (permanganate ion), MnO₂ (manganese (IV) oxide or manganese dioxide)

Activity 22

1. Work in pairs.
2. In which type of samples should you quantify copper?
3. Keep a record of your answer.



Fact File 1: Introduction to iodometric and iodimetric titrations

In this lesson: **Iodometric titration of copper**

Sample: Copper wire (solid)

First: Dissolve the sample

copper wire Cu^0 → dissolution → Cu^{2+}

Copper ion: oxidizing agent

Second: Pre-treatment of the sample

Slide 23:

- In this lesson we are going to quantify the copper present in a copper wire. As you know, copper wires are used as electric conductors.
- The procedure, an iodometric titration of copper, can be used in other samples like alloys, minerals, waters, electrolytic baths ...,
- Now these are the steps that we are going to follow in this procedure.
- Titrations must be performed in solutions. The copper wire is solid, so before carrying out the titration we need to dissolve the sample first.
- After dissolving the sample, and before the titration, a pre-treatment is required. We are going to see in more detail the pre-treatment of the sample when we explain the procedure.

Activity 23

1. Work in pairs.
2. Why is it necessary to dissolve the copper wire?
3. Keep a record of your answer.

Fact File 1: Introduction to iodometric and iodimetric titrations

Third: Iodometric titration

$$2 \text{Cu}^{2+} + 4 \text{I}^{-} \rightarrow 2 \text{CuI} + \text{I}_2$$

Analyte of unknown concentration

$$\text{I}_2 + 2 \text{S}_2\text{O}_3^{2-} \rightarrow 2 \text{I}^{-} + \text{S}_4\text{O}_6^{2-}$$

Titrant
- standard solutions: sodium thiosulfate
- known concentration


Slide 24:

- When the sample is ready, we can start the titration.
- The copper ion reacts with an excess of iodide, and generates copper iodide and iodine.
- The iodine generated is titrated by the standard thiosulfate solution.
- As we will see starch is the indicator that we will use in the Iodometric Titration of Copper.

Activity 24

1. Work in pairs.
2. Write the formula of iodine and iodide.

FACT FILE 2: THE PROCEDURE OF THE IODOMETRIC TITRATION OF COPPER



**FACT FILE 2:
THE PROCEDURE
FOR THE IODOMETRIC
TITRATION OF COPPER**

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Grant from the Generalitat de Catalunya, 2006

lbarreir@xtec.cat tnaves@ub.edu
<http://diposit.ub.edu/dspace/handle/2445/2>

Slide 1:


- This presentation is an introduction to the procedure of the iodometric titration of copper.
- We are going to see all the steps that you are going to carry out in the laboratory.
- Your questions are welcome, feel free to interrupt.
- During and after this presentation you are going to do different tasks to check that you have understood the procedure and the reactions involved.

Activity 1

1. Work in pairs.
2. Decide if these statements are True or False.

Statements	Me	My partner	Key
1. The titration that we are going to carry out in the laboratory is a Iodimetric one.			
2. Copper is the analyte.			
3. In this presentation we are going to see the main steps of the procedure.			

Key: 1. F; 2.T; 3. F



Fact File 2: The Procedure for the Iodometric Titration of Copper

- **Type of chemical analytical analysis:** _____
- **Type of titration:** _____
- **Analyte:** _____
- **Standard solution:** _____
- **Indicator:** _____

Slide 2:


○ First of all, let's remember some information about the iodometric titration of copper, the titration that you are going to carry out in the laboratory.

Activity 2

1. On your own.
2. Complete the missing information on the slide.

- Type of chemical analytical analysis
- Type of titration
- Analyte
- Standard solution
- Indicator

Key: Answer on the next slide



Fact File 2: The Procedure for the Iodometric Titration of Copper

- **Type of chemical analytical analysis:** Titration
- **Type of titration:** Redox
- **Analyte:** Copper
- **Standard solution:** Sodium thiosulfate ($\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$) 0.1M
- **Indicator:** Starch

Slide 3:

- What type of analysis is an iodometric titration?
- To be precise, iodometric titrations are quantitative analysis and are also types of redox titrations.
- What is the sample?
- Yes, the sample is copper wire.
- What is the titrant solution?
- Yes, the titrant solution is sodium thiosulfat.
- So we will perform:
- A quantitative analysis where the analyte is copper and the sample is copper wire.
- The analysis is an iodometric titration with sodium thiosulphate as the standard solution.
- Now, step by step we are going to see the procedure for the iodometric titration of a copper wire
- You have a flow-cahrt of the procedure in Input Source 3, which you can follow.

Activity 3

Work with a partner.

1. Did you get all the answers right?
2. Do you need extra help?



Fact File 2: The Procedure for the Iodometric Titration of Copper

Steps of the procedure:

- Step 1. Weigh the samples
- Step 2. Dissolve of the samples
- Step 3. Pre-treat of the samples
(addition of reactivés in order to
prepare the sample for the
titration)
- Step 4. Titrate

(see Input Source 3: Flow-chart of the Procedure)

Slide 4:

- A procedure could be divided in different steps.
- We can divide this procedure in 4 steps: Weigh the samples; dissolve of the samples; pre-treat of the samples and titrate.

Activity 4


1. Work with a partner.
2. Keep a record of the main actions.

Key: weigh, dissolve, pre-treat and titrate

Fact File 2: The Procedure for the Iodometric Titration of Copper

STEP 1

Weigh 3 samples
 $\approx 0.500 - 0.6000 \text{ g}$



sample 1 sample 2 sample 3

Slide 5:

- First, you need to label three Erlenmeyer flask. In the each Erlenmeyer you should write the number of the sample and the name of the students. For instance, sample 1, Ana García and Luis Sunyer.
- We need three samples and weight of each sample will be approximately 0.5000 g. So, in which balances will the weighing be conducted?
- Yes, in the analytical balance.
- Then, first to label three Erlenmeyer Flasks, second weight sample 1 and write its weight in the report sheet, third weight sample 2 and write its weight in the report sheet and finally weight sample 3 and write its weight in the report sheet.

Activity 5

1. Work with a partner.
2. Make a list of the main actions of Step 1.


Key: Label the Erlenmeyer Flasks, weigh the samples, keep a record on the Report Sheet.

Fact File 2: The Procedure for the Iodometric Titration of Copper

STEP 2

- **Add** 10 ml concentrated HNO_3 and heat.
- Nitric acid is an oxidizing agent.
- The reaction:

$$3\text{Cu} + 8 \text{HNO}_3 \rightarrow 3\text{Cu}^{2+} + 6\text{NO}_3^- + 2\text{NO} + 4\text{H}_2\text{O}$$



Nitrogen dioxide
<http://en.wikipedia.org>

Slide 6:

- The second step of the procedure is the dissolution of the samples.
- The sample is solid and, as we have said, titrations are performed in a liquid medium. Consequently, the next step is to dissolve the sample. In this case we will use an oxidising acid like nitric acid.
- We will add 10ml of concentrated nitric acid to each Erlenmeyer. What safety precautions must we bear in mind in this operation?
- Exactly, work with goggles, gloves and under the hood.
- The flask will be heated on a hot plate. The sample will dissolve until the solution turns light blue.
- Pay attention, because brown nitrogen dioxide gas is generated, so it's very important to work under the hood.
- Nitrogen oxide in contact with the atmosphere turns into nitrogen dioxide.
- The ion copper (II) is blue in solution.

- The reaction which takes place between the nitric acid and copper is the following. As you can see on the slide.
- Is this reaction a redox?
- Yes.
- Why?
- Because, there is an electron transfer. Copper loses electrons and Nitrogen gains electrons.
- Now that the sample is dissolved, we will prepare the sample to be tested.

Activity 6

1. Work with a partner.
2. Answer these questions:
 - a) Which species has blue colour in this step?
 - b) Which species has brown colour?
 - c) Make a list of the main actions of Step 2.

Key: a) copper (II) ion; b) nitrogen dioxide; c) add nitric acid, heat

Fact File 2: The Procedure of Iodometric Titration of Copper

STEP 3

- **Add** 8-9 ml H_2SO_4 and **heat**: white fumes of sulphur trioxide appear.
- This eliminates HNO_3 .
- HNO_3 might later oxidize Iodide (I^-) into Iodine (I_2).

Slide 7:

- After the dissolution of the samples it is necessary to prepare the sample for the titration, this step is the pre-treatment of the sample.
- Carry out the following treatment for the samples individually. That is to say, begin with one sample and once it has been evaluated do the same for the following one
- Once the Erlenmeyer is cold, 8 to 9 ml of concentrated sulphuric acid are added
- Heat until white gases of sulphur trioxide are formed
- In this operation we are eliminating the nitric acid which can oxidise in the titration of iodide to iodine.

Activity 7

1. Work with a partner.


2. Answer this question: Why is it necessary to add sulphuric acid?

Key: Sulphuric acid eliminates nitric acid. Nitric acid could oxidise iodide in further steps of the procedure.

Fact File 2: The Procedure for the Iodometric Titration of Copper

STEP 3

- **Cool.**
- **Add** drops of 6 M NH_4OH .
- Deep blue colour complex: tetraamminecopper (II)

$$\text{Cu}^{2+} + 4 \text{NH}_4^+ \rightarrow [\text{Cu}(\text{NH}_4)]^{2+} \rightarrow$$


- Avoid an excess.
- **Add** CH_3COOH : Eliminates any precipitate.

Source picture: <http://www.es.fishersci.com>

Slide 8:

- Next, cool and slowly add drops of aqueous ammonia
- As a result a coordination compound between the ammonium and the copper: tetraamminecopper is formed.
- It is important to avoid an excess of ammonia, so add it slowly until the solution turns bright blue.
- If there is any precipitate this will be eliminated after adding 3ml of concentrated acetic acid
- Now we have the solution prepared for the titration.

Activity 8

1. Work with a partner.
2. Answer these questions:
 - a) Which species has deep blue colour in this step?

- b) Why is it necessary to add acetic acid?
 c) Make a list of the main actions of Step 3.

Key: a) a coordination compound between the ammonium and the copper: tetraamminecopper;
 b) eliminates any precipitate; c) cool, add aqueous ammonia, add acetic acid

Fact File 2: The Procedure for the Iodometric Titration of Copper

STEP 4

○ IODOMETRIC TITRATION:
 ○ First reaction:

$$2 \text{Cu}^{2+} (\text{aq}) + 4 \text{I}^{-} (\text{aq}) \rightarrow 2 \text{CuI} (\text{s}) + \text{I}_2 (\text{aq})$$

\downarrow
 \searrow

$[\text{Cu}(\text{NH}_4)]^{2+}$
Add 2.5 g KI

Slide 9:

- Remember that in the iodometric titrations two reactions are involved.
- The first is between the analyte and an excess of iodide which generated iodine.
- In the second reaction the iodine is titrated with the standard solution.
- On adding 2.5g of potassium iodide, iodide reacts with ion copper (II) and generates iodine. As you can see from the formula on the slide.

Activity 9

1. Work with a partner.
2. Why is it necessary to add potassium iodide?

Key: Add iodide that reacts with copper (II) ion and generates iodine, this iodine will be titrated in the next reaction of the titration

Fact File 2: The Procedure for the Iodometric Titration of Copper



STEP 4

○ **Titrate** with the **standard solution**:

$$2 \text{S}_2\text{O}_3^{2-}(\text{aq}) + \text{I}_2(\text{aq}) \rightarrow \text{S}_4\text{O}_6^{2-}(\text{aq}) + 2\text{I}^-(\text{aq})$$

Second reaction

When the brown colour disappears
add the indicator: **Starch**.


→


Source:
<http://www.csudh.edu/oliver/demos/hh-cubr/hh-cubr.htm>

Slide 10:

- Swirl and titrate immediately the iodine with sodium thiosulfate.
- The reaction is here on the slide.
- $2 \text{S}_2\text{O}_3^{2-}(\text{aq}) + \text{I}_2(\text{aq}) \rightarrow \text{S}_4\text{O}_6^{2-}(\text{aq}) + 2\text{I}^-(\text{aq})$
- Titrate until the dissolution turns into a light yellow colour. This indicates that almost all the iodine has been titrated. Iodine is brown, like the medicine that you use on wounds.
- At this moment, when the solution is light yellow, add 3 ml of starch indicator.

Activity 10

1. Work with a partner.
2. When will you add the indicator? At the beginning? Why?

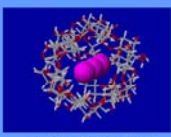
Key: Not at the beginning, when the dissolution turns light yellow

Fact File 2: The Procedure for the Iodometric Titration of Copper

STEP 4


- **Add** 3 ml starch indicator.
- Blue complex starch-iodine (I_2)

Starch - Iodine Complex

$$I_2 + I^- \rightarrow I_3^-$$


Iodine slides into starch coil to give a blue-black color

C. Gilman, v. 2002



Source: <http://www.elmhurst.edu/~chm/vchembook/548starchiodine.html>

Source: <http://www.csudh.edu/oliver/demos/hh-cubr/hh-cubr.htm>

Slide 11:

- The starch with the iodine forms a deep blue coloured complex.
- The sugar chain forms a helix around nearly linear I_6 chains.
- Continue titrating until the blue colour almost disappears.

Activity 11

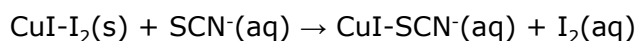
1. Work with a partner.
2. Which complex forms the blue colour?

Key: The complex that forms the starch (indicator) and iodine

Fact File 2: The Procedure for the Iodometric Titration of Copper

STEP 4

- When the blue colour almost disappears **add** 1-1.5 g **KSCN**.



copper iodide-iodine + thiocyanate ion →
copper iodide – thiocyanate + iodine

Slide 12:

- Then add 1g of potassium thiocyanate. Why is it necessary to add potassium thiocyanate?

Activity 12

1. Work with a partner.
2. Do you remember why it is necessary to add potassium thiocyanate?


- Because, there is a problem in this procedure, the CuI forms a complex with the iodine.
- This complex means that some iodine will not be titrated with sodium thiosulfate.

- The thiocyanate ion replaces the iodine in the complex $\text{CuI-I}_2(\text{s})$ (copper iodide-iodine) and forms a new complex $\text{CuI-SCN}^-(\text{aq})$, and therefore the iodine complexed is liberated.
- $\text{CuI-I}_2(\text{s}) + \text{SCN}^-(\text{aq}) \rightarrow \text{CuI-SCN}^-(\text{aq}) + \text{I}_2(\text{aq})$

Fact File 2: The Procedure for the Iodometric Titration of Copper

STEP 4

- **Titrate** drop by drop until the blue colour disappears, turns colourless and holds for 20-30 seconds.



Source:
<http://www.csudh.edu/oliver/demos/hh-cubr/hh-cubr.htm>

- **Calculate** the percentage of copper.

Slide 13:

- Now, it's time to finish the titration, add drop by drop until the blue colour disappears.
- The colourless solution must hold for 30 seconds. If the solution turns blue, you will add drops of titrant.
- Write the volume of titrant in the report sheet.
- Then, calculate the percentage of copper.

Activity 13

1. Work with a partner.
2. Why do you need to wait 20-30 seconds after reaching the end point?

Key: Because the solution could turn to blue again, it means that you haven't reached the end point.