T-LEVELS | Institute for Apprenticeships & Technical Education

T Level Technical Qualification in Science

Occupational specialism assessment (OSA)

Laboratory Sciences

Assignment 2 - Part A

Assignment brief

v2.1: Specimen assessment materials
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Part A

Contents

Experimental practical

Scenario

A commercial dairy produces a large amount of wastewater from manufacturing processes. The wastewater is contaminated with milk products and therefore has a high biological oxygen demand (BOD).

Wastewater is treated before discharge to a local river. The BOD of treated wastewater is regularly measured to check the effectiveness of treatment.

To calculate BOD, the oxygen content of the water is measured initially and after 5 days. Oxygen concentration can be measured using a Winkler titration.

Task 1

You have three hours to complete tasks 1(a) and 1 (b).

1(a): Carry out a Winkler titration to determine the initial oxygen content in the samples of wastewater provided following the standard operating procedure (SOP) and the safety information provided. During this activity, you will be observed by an assessor to make judgements on your practice. (23 marks)

1(b): Record your results in a suitable table and carry out any necessary calculations. (12 marks)

Information for assessors

Setting up the assessment

Before the task

The assessor must:

- remind the student that all health and safety procedures must be followed during the assessment (the student may see the checklist below)
- ask the student to locate all relevant safety equipment and emergency procedures specific to the laboratory where the task is taking place

After the task

The assessor must complete the following checklist prior to marking the rest of the task. All criteria must be checked and signed by the assessor. In addition to the below checklist, an observation checklist is completed to show the allocation of marks against student practice.

In the event that a student performs a task in an unsafe manner, the assessor may stop the assessment, and the student will not be able to complete the assessment at this time.

Please note that in the event of one minor incident where the assessor can see that there is no immediate safety concern, and where the assessor can intervene, the assessor may provide a prompt to the student.

An example of this would be if a student lifts their goggles onto their forehead in order to see a pipette reading more clearly, and then forgets momentarily to place the goggles back over their eyes. The assessor should not stop the assessment in this instance and may remind the student to put their goggles back over their eyes. They should inform the student that if they make the same error again, that they would need to stop the assessment.

Assessor checklist

The student:

☐	can locate all relevant safety equipment and emergency procedures specific to the individual laboratory
☐	used appropriate personal protective equipment (PPE) correctly and effectively throughout the practical procedure, for example, laboratory coat fastened, splash proof eye protection and gloves worn correctly at all times
☐	followed all appropriate safety guidelines and procedures when handling materials, disposing of waste materials and during clean-up of any spills
☐	cleaned up the bench and work surfaces satisfactorily at the end of the task

Standard operating procedure

Process title: The Winkler's method for dissolved oxygen

Introduction

The Winkler's method is a standard method for the determination of dissolved oxygen concentrations in water and wastewater. This SOP covers the fixing of dissolved oxygen within stoppered bottles, the subsequent liberation of iodine and final titration with sodium thiosulphate. It does not include the preparation of the reagents (note that the preparation of some of these reagents is potentially hazardous).

Dissolved oxygen within the sample is fixed by the addition of Mn(II) under alkaline conditions, resulting in a brown precipitate of manganic hydroxide ($MnO(OH)_2$). Prior to analysis, the sample is acidified and the precipitated hydroxides dissolve in a series of reactions liberating iodine. The iodine is titrated with thiosulphate in which iodine is reduced to iodide and the thiosulphate is oxidized. A starch indicator is added to determine the end point.

Risk assessment

Substance, equipment, or procedure	Hazard	Risk	Control(s)
Concentrated sulfuric (VI) acid	Corrosive	Severe burns to skin, eyes, and respiratory tract. Strong exothermic reaction with water.	Wear nitrile gloves and splash proof goggles. Immediately dispose of gloves that get any acid on them. Do not add water to bottle.
Solution A - Manganese (II) sulphate solution	Health hazard	Organ damage with long term exposure. Risk to environment.	Wear eye protection and protective gloves. Dispose of waste or spillages as advised.
Solution B - Alkaline potassium iodide solution	Corrosive	Strong alkali. Causes severe skin burns and eye damage.	Wear splash proof goggles. Wear protective gloves.
Sodium thiosulphate solution	Low	Produces toxic gas with acids.	Do not mix directly with strong acid. Do not inhale gases.
Filling of burette	Low	Risk of spills and splashes.	Wear eye protection, follow good practice for filling burettes.

Procedure

Reagents:

Solution A - Manganese (II) sulphate solution (approx. 2.3M)	
Solution B - Alkaline potassium iodide solution (contains 8M sodium hydroxide and potassium iodide)	
Concentrated sulfuric (VI) acid	
Low hazard sodium thiosulphate solution (0.1M)	
Low hazard starch indicator solution	

1) Wear splash proof goggles, protective gloves, and laboratory coat.

2) Collect a 250 cm^3 sample of water in a 250 cm^3 stoppered glass bottle, ensuring there is no air trapped inside.

3) Use a 1 cm^3 pipette (or a marked transfer pipette) to transfer 1 cm^3 of solution A (manganese (II) sulphate solution) to the water sample. Inject solution A below the surface of the water, taking care not to introduce any air into the water.

4) Use a similar technique to transfer 1 cm^3 of solution B (alkaline potassium iodide solution) to the water sample.

5) Replace the stopper on the bottle (without trapping air inside). Agitate the bottle to mix the reagents and then allow the bottle to stand for 10 minutes. Brown manganese (III) hydroxide will precipitate.

6) Transfer the contents of the bottle to a larger flask or bottle.

7) In the fume cupboard, add 1.5 cm^3 of concentrated sulfuric (VI) acid using a 2cm^3 marked transfer pipette, the precipitate will dissolve, and iodine will be liberated.

8) Titrate this solution with the standardised sodium thiosulphate solution (0.01M), adding 2 cm^3 of starch solution near the end point (using a transfer pipette).

9) The oxygen concentration of the sample in mg/dm^3 can now be calculated.

Calculating oxygen concentration

1 ‘mol’ of oxygen (O_2) is equivalent to 4 ‘mol’ of thiosulphate.

Each cm^3 of thiosulphate represents a mass of dissolved O_2 in the sample equivalent to: 1/1000 x 0.01 M x 1/4 = 0.0000025 mol O_2 (per cm^3 of 0.01M thiosulphate).

The molar mass of oxygen (O_2) is 32g. So, the mass of oxygen is 32 x 0.0000025 = 0.00008g (or 0.08mg) of O_2 per cm^3 of thiosulphate used.

Use the volume of water sample (0.25 dm^3) and the thiosulphate titration volume to calculate the dissolved oxygen concentration of the sample in mg per dm^3.

Relevant laboratory health, safety, and environmental and regulatory requirements

The Control of Substances Hazardous to Health Regulations 2002 (COSHH) - toxics, corrosives.

Emergency/spillage procedure

What to do in the event of spill

Solution A (manganese (II) salts) - manganese (II) salts at the concentration used are an environmental toxin. For spillages, ensure dilution is to below 0.25M when disposed of into a foul water drain.

Solution B (alkaline potassium iodide solution) - treat as strong alkali. Clean up drips immediately. For spills use the laboratory spills kit to absorb (inert solid) or neutralise (citric acid powder) before disposal.

Concentrated sulfuric acid - use the laboratory spills kit to absorb (using inert solid) or neutralise (using anhydrous, technical grade sodium carbonate) any spills.

Sodium thiosulphate - wipe up solution spills and rinse well.

Fixed sample solutions - wipe up spills and rinse well.

Emergency first aid procedures in event of exposure

Solution A - Manganese (II) sulphate

Eye:

- flood with gently running tap water for at least 10 minutes
- consult a medical professional

Ingested:

- wash out the mouth with water
- do not induce vomiting
- consult a medical professional

Spilt on the skin or clothing:

- remove contaminated clothing and rinse it
- wash off the skin with plenty of water
- if skin contamination is more than a small area, consult a medical professional

Solution B - Alkaline potassium iodide solution

Eye:

- rinse with water for at least 10 minutes
- remove contact lenses, if present and easy to do
- continue rinsing

In mouth:

- rinse mouth and spit out
- do not induce vomiting

If inhaled:

- remove to fresh air and keep at rest in a position comfortable for breathing
- if on skin (or hair):
  - quickly wipe off excess liquid with a dry cloth but do not delay irrigation
  - if safe to do so, remove contaminated clothing
  - rinse skin with water/shower for at least 10 minutes
  - seek medical help

Concentrated sulfuric (VI) acid:

- quickly wipe off excess liquid with a dry cloth but do not delay irrigation
- wash using copious volumes of cool water to counter the exothermic reaction of the concentrated acid with water

Eye:

- rinse with water for at least 10 minutes
- remove contact lenses, if present and easy to do
- continue rinsing

In mouth:

- rinse mouth and spit out
- do not induce vomiting

If inhaled:

- remove to fresh air and keep at rest in a position comfortable for breathing

If on skin (or hair):

- if safe to do so, remove contaminated clothing
- rinse skin with water/shower for at least 10 minutes
- seek medical help

Sodium thiosulphate

Eye:

- flood with gently running tap water for 10 minutes
- consult a medical professional if pain persists

Ingested:

- wash out the mouth with drinking water
- do not induce vomiting
- consult a medical professional

Spilt on the skin or clothing:

- rinse as necessary

Disposal procedures

Manganese (II) salts

There should be no need to dispose of waste in this procedure. Reagent should remain in labelled bottles.

Alkaline potassium iodide solution

There should be no need to dispose of waste in this procedure. Reagent should remain in labelled bottles.

Concentrated sulfuric (VI) acid

There should be no need to dispose of waste in this procedure. Reagent should remain in labelled bottles.

Sodium thiosulphate

Unused reagent from the burette should be returned to a designated receptacle.

Unused fixed water samples and used sample following Winkler titration - pour into Winkler waste bucket provided. The technician will neutralise this by adding baking soda until foaming stops. Waste will then be washed down the drain with copious amounts of water.

Any specific storage requirements

There is no requirement for storage during this procedure.

References

CLEAPSS RB 105 05/17 Winkler's method for dissolved oxygen

CLEAPSS HazCards:47B,60,91A,95A,98A

Document information

Owner: Head of Assessment Design

Change History Record

Version	Description of change	Approval	Date of Issue
v1.0	Post approval, updated for publication.		January 2021
v1.1	NCFE rebrand		September 2021
v1.2	OS review Feb 23		February 2023
v2.0	Annual review 2023 Update to task wording for clarity. 'You have three hours to complete tasks 1(a) and 1(b).' p3	June 2023	19 June 2023
v2.1	Sample added as a watermark	November 2023	20 November 2023