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PRIFYSGOL
BANGOR
UNIVERSITY

Royal Society of Chemistry

Analytical Division North West Region

Schools Analyst Competition

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Experimental Handbook

Scenario

Mr a Mrs Jones noticed a blue colour and an unpleasant metallic, bitter taste to their drinking water. They contacted their water providers as they were extremely worried about the unusual colour and taste in their water. Their water providers stated that this colour was rare and could be due to a higher amount of copper in their water which may be due to the corrosion of copper plumbing. In small amounts copper is vital for our bodies to function but high amounts of copper may cause a variety of health complications. Urgent testing needs to be done to determine the concentration of copper in Mr and Mrs Jones's drinking water! The World Health Organisation states that the concentration of copper in drinking water should not exceed 2 ppm.

Theory

The Beer-Lambert Law

The Beer-Lambert law states that the absorbance of a substance in solution is proportional to its concentration. Therefore, if the absorbance of a series of solutions of known concentrations are measured and plotted against their corresponding concentrations, the graph of absorbance versus concentration should be linear. The graph is known as a calibration graph and it may be used to determine the concentration of unknown sample solution.

The absorbance of solutions can be measured using two different spectroscopic techniques, Atomic Absorption Spectroscopy and UV-Visible Spectroscopy

Atomic Absorption Spectroscopy (AAS)

AAS is based on the absorption of UV/Visible radiation by free atoms in the gaseous phase. In this process the samples (usually liquids) are firstly vaporized and the amount of light absorbed at a specific wavelength is detected.

UV-Visible Spectroscopy

UV-Visible Spectroscopy is based on the absorption of UV/Visible radiation in the aqueous phase. Absorption of visible or UV radiation is associated with the excitation of electrons from lower to higher energy levels.

Aim

Use Atomic Absorption Spectroscopy and UV-Visible Spectroscopy to determine the concentration of copper in Mr and Mrs Jones's drinking water sample.

Health and Safety

It is essential that you read the following risk assessment, and are fully aware of the hazards associated with the materials you are using.

Hazard	Risk	Control Measures	First aid in case of accident
Harmful and toxic chemicals	Adverse health effects caused by ingestion, inhalation, or skin/eye contact with chemicals	Chemicals are used in very small quantities and are dilute. Chemicals chosen to be of low toxicity unless ingested. Gloves and eye protection worn at all times. Constant supervision by trained staff. Pipetting by mouth prohibited. Toxic chemicals used in the fume hood.	In case of contact with skin or eyes rinse the affected area with cold water. Report the incident to a member of staff
Glassware	Glassware breakage causing cuts	All glassware is pre-checked by technical staff and any damaged equipment should be returned. Care to be exercised especially when pushing rubber pipette fillers on to glass.	Contact a member of staff and obtain first aid assistance immediately in the case of an accident
Allergenic chemicals or materials	Adverse, possibly serious health effects caused by allergic reaction.	Students asked to declare any known allergy prior to the event. Students asked to report any unusual symptoms during the event. Trained first aid staff on hand. COSHH data available for all reagents	If the risk cannot be contained the student will be prohibited from performing the experiment. Rinse the affected area with cold water. Report the incident to a member of staff
Fire	Death or serious injury caused by fire	Other laboratory operations in the laboratory prohibited during event. Fire Alarms, smoke detectors and other building safety systems tested and operational at all times. Students instructed on the evacuation procedure prior to the event.	Evacuate the building.
Spectrometers	Injury caused by falling spectrometers.	Spectrometers placed on even surface and not on the end of a bench to avoid any falls.	Report the incident to a member of staff immediately.
Electrical equipment	Serious injury caused by	Electrical equipment tested regularly.	Report the incident to a member of staff

	electrical equipment		immediately.

Experimental

You are provided with two copper sulfate stock solutions, a 100 ppm solution for Part 1 and a 1000 ppm solution for Part 2.

Part 1 - Atomic Absorption Spectroscopy

Preparation of Standard Solutions

Label five 100 cm^3 volumetric pipettes with the concentrations listed in the table below. Prepare five different concentrations of copper sulfate by diluting the copper sulfate stock solution provided (100 ppm) according to the table below.

Concentration (ppm)	Stock Copper Sulfate Solution (cm^3)	Deionised Water (cm^3)
1	1	99
2	2	98
3	3	97
4	4	96
5	5	95

Preparation of the Unknown sample

Pipette 1 cm^3 of the unknown sample into a 100 cm^3 volumetric flask. Dilute up to the mark with deionised water.

Recording AAS data

Place the labelled volumetric flasks in the carrier provided and inform one of the members of staff that you are ready to record your AAS data. Members of staff will then take you, one school at a time to record the AAS data. Start Part 2 while you are waiting.

Part 2 - UV-Visible Spectroscopy

Preparation of Standard Solutions

Label five 100 cm^3 volumetric pipettes with the concentrations listed in the table below. Prepare five different concentrations of copper sulfate by diluting the stock solution (1000 ppm) according to the table below.

Concentration (ppm)	Stock Copper sulfate Solution (cm^3)	Deionised Water (cm^3)
100	10	90
200	20	80
300	30	70

400	40	60
500	50	50

Label five sample vials with the concentrations of the standard solutions. Label a 6th vial with ‘unknown sample. Transfer 5 cm^3 of each standard solutions and unknown sample (no further dilution required to the unknown sample) to separate sample vials. IN THE FUME CUPBOARD add two drops of concentrated aqueous ammonia to each vial. Do not breathe the vapours. Mix each solution.

Recording UV-Visible Spectroscopic data

IN THE FUME CUPBOARD and using a Pasteur pipette carefully transfer enough solution from each sample vial and unknown sample to two thirds fill separate disposable plastic cuvettes (one cuvette per concentration). Note the position in the rack of each cuvette to avoid confusion. Do not write on the cuvettes.

Take your cuvettes containing the standards and the sample to the spectrometer and with the assistance of a demonstrator measure the absorbance of each cuvette at a wavelength of 700 nm. Record your results. After recording your absorbances, pour the solutions into labelled waste bottles. Do not pour down the sink.

Treatment of Results

Atomic Absorption Spectroscopy Data

Use the data that you obtained to plot a graph of the absorbance against the concentration of each standard. You can then use the graph to read the concentration of copper in the water sample. Remember the dilution factor.

UV-Visible Spectroscopy Data

Use the data that you obtained to plot a graph of the absorbance against the concentration of each standard. You can then use the graph to read the concentration of copper in the water sample.

Average Concentration of water

Using the results obtained for the concentration of copper in the water sample by AAS and UV-Visible Spectroscopy calculate the average concentration of copper in the water sample.

Results Sheet

(to be handed in with your graphs)

Name of School____

Atomic Absorption Spectroscopic Data

Sample	Absorbance
1 ppm	
2 ppm	
3 ppm	
4 ppm	
5 ppm	
Unknown Water Sample	

Concentration of copper in the water sample ________________________ ppm

UV-Visible Spectroscopic Data

Sample	Absorbance
100 ppm	
200 ppm	
300 ppm	
400 ppm	
500 ppm	
Unknown Water Sample	

Concentration of copper in the water sample ________________________ ppm

Average concentration of copper in the water sample ___________________ ppm