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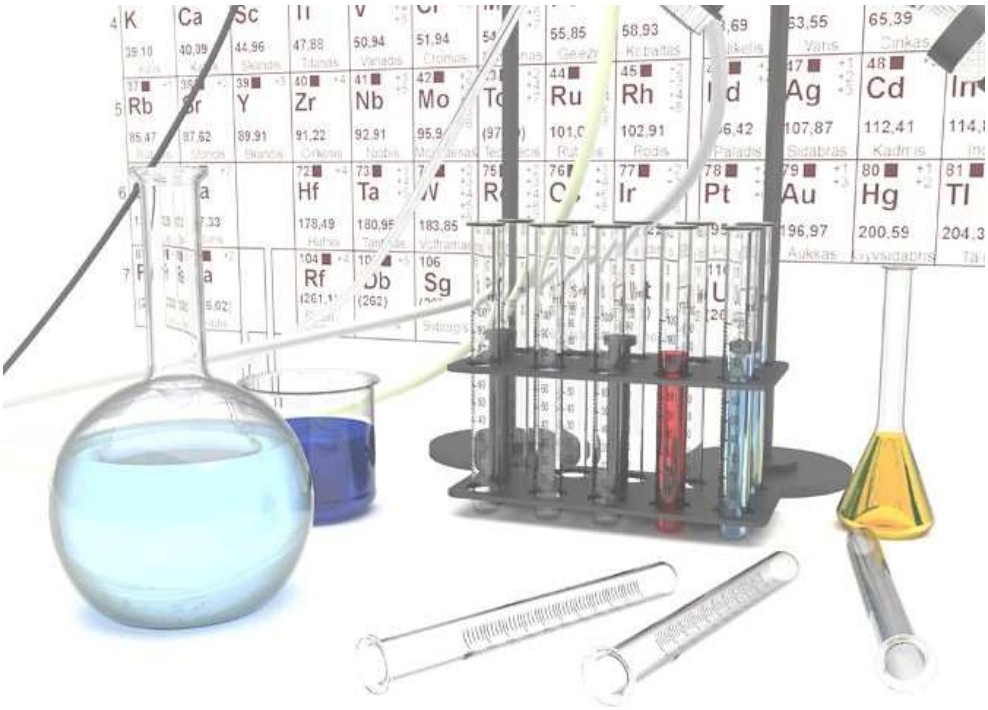


INDUSTRIAL COPPER EXTRACTION

Including

Exp. 3.1 LEACHING THE COPPER ORE.
Exp. 3.2 VISIBLE LIGHT SPECTROSCOPY
Exp. 3.3 COPPER SOLVENT EXTRACTION
Exp. 3.4 COPPER TITRATION
Exp. 3.5 COPPER ELECTROWINNING

Student booklet

Name: _____

Teacher: _____

EXPERIMENT 3.1

LEACHING THE COPPER ORE

AIM:

To simulate the leaching of oxidised copper ores using dilute sulphuric acid.

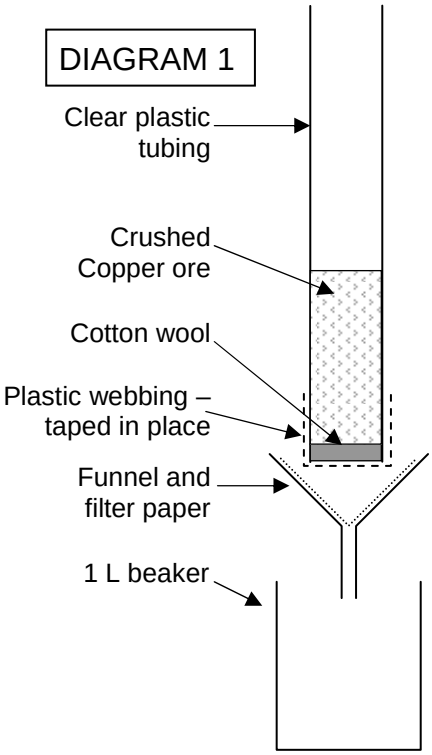
MATERIALS:

- Rocks of Copper ore
- 1 metre clear plastic 35mm tubing
- Mortar and Pestle
- 1 L and 500 mL beakers
- Filter paper and funnel
- Electronic balance (up to 200 gm)
- 3 small (~ 20 mL) sample bottles
- 12 cm piece of plastic webbing (eg shadecloth)
- Strong adhesive tape
- 500 mL measuring cylinder
- Labels

METHOD:

1. Crush 200 g of the copper ore with the mortar and pestle. You will probably need to do this in 3 parts due to the size of the mortar and pestle. A refinery would have a ball mill grinding the ore to a small granular size.
2. Arrange the tubing, 1 litre beaker, cotton wool, plastic webbing, filter paper and funnel, 1 litre beaker, and crushed ore as shown in diagram 1.
3. Slowly pour 500 mL of 0.01 M sulphuric acid into the top of the tubing. Observe the dilute acid moving through the ore, leaching the copper from the ore as soluble copper sulphate. The entire leaching process will take several hours before your first leach solution has collected in the beaker below the ore.
4. Collect a sample of this leach solution in one of your sample bottles (need less than 5 mL). Label this sample appropriately.
5. Pour the leach solution back into the top of the tubing and repeat the process. Again after several hours or overnight, collect a sample of your second leach solution.
6. Repeat step 5 one more time – obtaining you third and final sample and your final "leach solution". Place this in a sealed and labelled bottle for the third experiment.
7. Take you apparatus apart, retaining the used ore in a sealed plastic bag clearly labelled with your groups name.

DIAGRAM 1



EXPERIMENT 3.2

VISIBLE LIGHT SPECTROSCOPY - COLOURIMETRY

AIM:

To determine the concentration of copper sulphate in each of the leach solutions.

INTRODUCTION:

Spectroscopy is a means of finding unknown concentrations of chemicals in aqueous solution. Visible light spectrophotometers are often referred to as colourimeters as the use "normal" coloured light to measure concentration.

Any solution which is coloured will absorb one of the wavelengths of visible light which reflect the others – thus giving it a colour. For example, copper sulphate which is a blue solution actually absorbs light in the red to orange range (~ 700 nm). Because the red colours are absorbed and not reflected the copper sulphate solution looks blue.

The spectrophotometer/colourimeter simply shines a light with a certain wavelength through the copper sulphate solution and measures how much of the wavelength is received the other side of the sample. The missing light is said to be absorbed by the solution, and is called the "absorbance". The more concentrated the solution the greater amount of light is absorbed – the greater the absorbance reading (this is known as Beer's Law).

The absorbance can range between zero and one. Using a colourimeter allows you to measure the absorbance of an unknown sample. By comparing this value to a range of known values (called standards) the concentration of the unknown solution can be worked out.

Making up accurate standards (solutions of known concentrations) is an important part of colourimetry. Plotting a graph of absorbance versus concentration for the standards results in a linear relationship. You can use this relationship to interpolate any absorbance value within the range of your standards. Extrapolation is not consider accurate methodology with colourimetric data. If your unknown has an absorbance above the range of your standards then accurately dilute the unknown to provide a lower absorbance value that is within the range of your standards.

NB: Copper sulphate absorbs most effectively at ~700nm. The colourimeter you will be using measures only at discrete wavelengths, the closest of which is 625nm. This does not affect the way the colourimetry is performed however it means that the absorbance of very dilute solutions may not be accurately detected. Copper sulphate solutions with concentrations lower than 0.01 M will have little absorbance at 625 nm and therefore effectively read as zero absorbance and thus zero concentration. At 700 nm, where copper sulphate has its strongest absorbance, concentrations of 0.01M and lower could be measured effectively.

MATERIALS:

For colourimetry:

- 3 sample bottles of leach solution
- Dataquest and colourimeter
- Cuvette and tissue
- Plastic pipette

For Dilutions of leach (unknown) soln:

- 1 x 80 mL beakers
- 10 mL graduated pipette
- Distilled water
- Safety bulb filler for pipette
- 1 Ml graduated pipette with plastic pipette as safety bulb

For making standards:

- 2 x 80 mL beakers
- Copper sulphate (pentahydrate)
- Distilled water
- 100 mL volumetric flask
- 2 x 50 mL Burette
- Electronic balance
- Spatula
- 5 small sample bottles with label

METHOD:

Making standard solutions:

Use the appartatus to make copper sulphate (pentahydrate solutions) of 0.25 M, 0.2 M, 0.15 M, 0.1 M, and 0.05M. You will use distilled water as a zero concentration – a sixth "standard".

The volume of each standard should not exceed 20 mLs. You will need les than 5 mL for each absorbance reading, and need a little more than that in case repeat readings are necessary (you make mistakes!).

The most time effective way to do this is make a 1.0 M sample of copper sulphate and add this to one of the burettes. Place distilled water in the other burette. Add precise volumes from each burette to make serial dilutions to each of the required concentrations.

Store each standard in a labelled sample bottle.

Using Colourimeter:

1. Turn on the Dataquest and plug the colourimeter into channel one. If this probe is not auto-recognised, select sensors, then colourimeter for channel one. The absorbance band (orange) will appear on the main screen of the Dataquest. This instructs the Dataquest to record the absorbance using the attached colourimeter.
2. The Dataquest will need to know the concentrations of the standards so select the… EVENTS WITH ENTRY STUFF!!!!!
3. Use a permanent marker to place a dot on the top of one of the cuvette supplied with the colourimeter. Fill this cuvette about ¾ full with distilled water and wipe the clear plastic sides with a dry tissue so they are as clean as possible.
4. Place this cuvette in the colourimeter so that the clear plastic sides are facing to the front and the rear of the colourimeter. Remember the direct the dot is facing –

the cuvette must always be placed in the machine in this way! Close the lid on the colourimeter.

5. Press the tiny green flag beside the green play button at the bottom of screen. This starts your experiment. The green play button changes to a snapshot icon. Any time you want to record an absorbance press this icon.
6. Press the snapshot icon now. The Dataquest will record the absorbance of the sample, and ask you the concentration. Write this in as 0.0, your zero concentration standard and press ok. The Dataquest will begin to graph your data (initially this will not appear to make sense as the Dataquest has limited information).
7. Take out the cuvette. Pour the contents into the sink. Using the plastic pipette, rinse the cuvette with some of the 0.05 m standard solution, pouring this out as well. Fill the cuvette approximately ¾ full with 0.05 M standard. Wipe the sides clean and dry with a tissue, pace back in the cuvette and close the lid.
8. Repeat steps 6 to 7 until you have recorded the absorbance of all the standards.
9. Press analyse (the graph) and choose to fit a curve to your data. Select a linear fit. The best straight line fit to the standards will be displayed along with the equation to this line. Record the equation in this booklet. Press ok.
10. Press on each of the data points on the graph. The values of absorbance and concentration are displayed (on the right) as each point is touched. Record these values this booklet.
11. Repeat step 6 to 7 for each of the leach solution samples. If the absorbance of each of these is too high (outside the range of your standards), dilute each in a known ratio until their absorbance lies within your range. Use the notes section on the dtaquest to record the absorbance of each leach solution and record it in this booklet.
12. Press analyse (the graph) and interpolate. Press anywhere on the best fit line and the absorbance and concentration on that point will be displayed. Use this tool to determine the concentration of each of your leach solutions. Record this data in this booklet.

EXPERIMENT 3.3

COPPER SOLVENT EXTRACTION

AIM:

To demonstrate the use of Copper solvent extraction.

MATERIALS:

- Copper leach solution
- 2M H_2SO_4 solution
- Organic reagent (5% extractant M5640 in Shellsol 2046 : (100ml)
- Separating funnel (250ml)
- Measuring cylinder (100ml)
- Conical flasks (100ml + 50ml)
- Safety glasses

SAFETY GLASSES MUST BE WORN

METHOD:

1. Check the tape on the separating funnel is closed. Pour 100ml of organic reagent into the separating funnel.
2. Measure out 100ml of the copper leach solution and place in the separating funnel.
3. Seal th4e separating funnel and then vigorously agitate for 5 minutes. Release the pressure in the funnel as demonstrated by your teacher. Note any colour changes.
4. Allow the two phases to separate and drain the aqueous phase (bottom layer) from the separating funnel into a 100ml conical flask. (This is referred to as the leach solution after extraction).
5. Measure out 25ml of 2M H_2SO_4 and place in the separating funnel.
6. Seal the separating funnel and then vigorously agitate for 5 minutes. Note any colour changes.
7. Allow the two phases to separate and drain the aqueous phase (bottom layer) from the separating funnel into a 50ml conical flask). This is referred to as the acid solution (i.e. after stripping the copper ions).
8. Return the original leach solution [from part (d)] to the separating funnel (with the organic reagent) and repeat the process. (i.e. shake for 5 minutes, separate, add the acid solution [from part (g)] back into the funnel, shake and separate).
9. Repeat the process a third time and by the end you should have 25ml of concentrated copper solution.
10. You now need to repeat this process with the remaining 100 ml of leach solution.
11. At the end of this stage, you will have 50ml of concentrated copper solution.
12. You could now perform a copper titration to determine the concentration of copper ions in solution or alternatively use spectroscopy to determine the concentration of copper ions.
13. Your solution is now ready for electrowinning to plate out copper.

EXPERIMENT 3.4

COPPER TITRATION

AIM:

To demonstrate the concentration of copper ions in solution.

MATERIALS AND APPARATUS:

- Copper solution
- 0.03 M sodium thiosulfate solution $Na_2S_2O_3$
- Starch
- Potassium iodide crystals, KI
- Wash bottle with distilled water
- 10ml pipette
- Pipette bulb
- 100ml beaker
- 250ml flask
- 50ml burette
- 10ml measuring cylinder
- Burette stand

PROCEDURE:

1. Pipette 10ml of copper solution into a 250ml flask.
2. Add 10ml of distilled water.
3. Use the KI spatula to add a small amount (about the size of a pea) of potassium iodide crystals. Swirl to obtain a yellow/brown colour.
4. Fill the burette with sodium thiosulfate solution and note the initial burette reading.
5. Titrate with sodium thiosulfate solution to a pale milky yellow colour and then add 5ml of starch and swirl to obtain a deep blue/black colour. Continue to titrate until the end point when the blue/black colour **just** disappears. The solution is a milky white colour.
6. Take the final burette reading.

COPPER TITRATION CHEMISTRY

Titration to determine the Cu^{2+} concentration in solution.

(a) React Cu^{2+} with I- and liberate I_2 (brown/yellow colour)

Full equation: $2CuSO_4(aq) + 4KI(aq) \rightarrow 2CuI(s) + I_2(aq) + 2K_2SO_4(aq)$

Ionic equation: $2Cu^{2+}(aq) + 4I(aq) \rightarrow 2CuI(s) + I_2(aq)$ **(1)**

blue — brown/yellow

(b) Titrate the liberated I_2 with thiosulfate solution ($S_2O_3^{2-}$)

Full equation: $I_2(aq) + 2Na_2S_2O_{3-}(aq) \rightarrow Na_2S_4O_6(aq) + 2NaI(aq)$

Ionic equation: $I_2(aq) + 2S_2O_3^{2-}(aq) \rightarrow S_4O_6^{2-}(aq) + 2I^-(aq)$ **(2)**

Brown/yellow — Clear

(c) $2Cu^{2+} = I_2 = 2S_2O_3^{2-}$ **(3)**

The end point is identified as the brown/yellow colour of the iodine disappears.

The end point can be seen more easily if starch is added. It forms a deep blue colour with iodine that disappears when the iodine is used up.

COPPER TITRATION – CALCULATIONS

From equation (1): 2 Moles Cu^{2+} liberate 1 mole I_2

From equation (2): 2 moles $S_2O_3^{2-}$ react with 1 mole I_2

Therefore from **equation (3):** number of moles $S_2O_3^{2-}$ used equals number of moles of Cu^{2+}

$$\mathbf{n}_{Cu2+} = \mathbf{n}_{S2O3-2}$$

$$\mathbf{C}_{Cu2+} \times \mathbf{V}_{Cu2+} = \mathbf{C}_{S2O3-2} \times \mathbf{V}_{S2O3-2}$$

$$\mathbf{C}_{Cu2+} = \frac{\mathbf{C}_{S2O3-2} \times \mathbf{V}_{S2O3-2}}{\mathbf{V}_{Cu}{}^{2+}}$$

EXPERIMENT 3.5

COPPER ELECTROWINNING

AIM:

To demonstrate the principles of copper electrowinning.

MATERIALS AND APPARATUS:

- Lead Anodes (2)
- Stainless steel cathode
- Beaker with stirrer
- Variable current DC power source
- Copper ion solution from solvent extraction'
- Measuring cylinder
- Safety glasses
- 2 x 20 ml sample bottles

PROCEDURE:

1. Place solution into container and commence agitation.
2. Weigh the stainless steel cathode. Record the weight.
3. Place the stainless steel cathode between the two lead anodes and ensure that the faces are aligned parallel.
4. Connect the DC power source, turn it on and adjust to approximately 2 amps. (This ensure a current density of 300 Amps/m^2). Record the time at which power was turned on.
5. Leave running for 2 – 4 hours. Check once every hour during this time as the amps may have to be adjusted slightly.
6. Turn off power source and agitator. Remove a sample of solution for later analysis. Record the time at which power was turned off.
7. Remove cathode and anodes. Wash with water and allow to dry.
8. Weigh the stainless steel cathode and deposited copper cathode. Record the result.
9. Strip copper cathode from stainless steel cathode. Weigh the copper cathode. Record the result.

ELECTROWINNING RESULTS

Mass of stainless steel cathode = _____ g

Mass of stainless steel cathode and copper cathode = _____ g

Mass of copper cathode deposited = _____ g

Average current = _____ amps

Average voltage = _____ volts

To calculate the mass of copper which should theoretically have been deposited, use:

Q = I x t Q = total charge in Coulombs

I = Average current

T = deposition time (s)

and the knowledge that 96500C deposit 0.5 mol of copper

Compare theoretical and actual copper deposited.

$$\text{Current efficiency} = \frac{\text{actual mass of Cu deposited}}{\text{Theoretical mass of Cu deposited}} \times 100$$