

CHAPTER I

INTRODUCTION

1.1 Background

In determining the levels of insoluble compounds, certain methods are applied which involve the principle of deposition. Precipitation is a very important method in separating a sample into its components. The process involved is a process where the substance to be separated is used to form a solid precipitate. A precipitate is a substance that separates itself as a solid phase out of solution. Analysis with the depositional principle has been used extensively in analytical chemistry, especially in argentometric and gravimetric methods.

Argentometry is a volumetric quantitative analysis with standard AgNO_3 solutions based on deposition. Argentometry is used to determine the content of an element in titrations involving silver salts with a suitable indicator. Argentometric titration can not only be used to determine halide ions, but can also be used to determine mercaptans (thioalkohols), fatty acids, and some divalent anions. There are three methods that can be used in argentometric analysis, including the Mohr method, the Fajans method, and the Volhard method. The end point of the titration is determined by the formation of a colored solution or the appearance of the first turbidity. The use of argentometric analysis in this practicum is to determine the levels of halogenides, for example Cl^- , contained in the sample so that it is useful for oceanography, food, and industry. Chemical engineering bachelors work a lot in the field, so it's expected that chemical engineering bachelors can do argentometric analysis.

1.2 Experiment Objectives

1. Analyzing Cl^- levels using the Mohr method.
2. Analyze Cl^- levels with the Fajans method.

1.3 Experiment Benefits

1. Students understand and have skills in analyzing Cl^- levels using the Mohr method.
2. Students understand and have skills in analyzing Cl^- levels with the Fajans method.

CHAPTER II

LITERATURE REVIEW

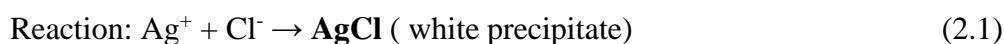
Argentometry comes from Latin “Argentum” which means silver. Argentometry is a volumetric quantitative analysis to determine the levels of halogen in a sample using standard AgNO_3 solutions in a certain setting. The argentometry method is also called the deposition method because in argentometry there is the formation of relatively insoluble compounds or sediments. AgNO_3 solution is a secondary standard solution so that it must be standardized with the primary standard solution first. The primary standard solution is a solution that has a certain or constant level, while the secondary standard solution is a solution whose levels are not fixed or can change. Primary standard solution commonly used to standardize AgNO_3 solutions is NaCl . In argentometric analysis, the end point of the titration is determined by the formation of a colored solution or the appearance of the first turbidity. There are three methods that can be used in argentometric analysis, namely, the Mohr method, the Fajans method, and the Volhard method.

2.1 Argentometric Analysis Method

The following are the methods used in Argentometric analysis:

a. Mohr's method

Used to determine the levels of halogen ions carried out in a neutral atmosphere with the K_2CrO_4 indicator and the standard solution of AgNO_3 . Chromate ions will react with silver ions to form a red brown precipitate of silver chromate (Dedi Sholeh Effendi, 2012).



The basis of the titration with this method is a stepwise deposition of AgCl and after all settles, the Ag_2CrO_4 deposits occur. For more details, we can see the following example.

For example, in a 0.1 M NaCl solution there is an indicator K_2CrO_4 which has a concentration of 0.01 M, then the Ag^+ concentration to precipitate Cl^- and CrO_4^{2-} countable.

- To precipitate the Cl^-

At this point of equality occurred. Neither chloride ion nor silver ion is excess, and each concentration is the square (of) K_{sp} . On the titration curve this

point is called the equivalence point (TE), which is the point on the curve indicates the number of grams equivalent of the titrant is equal to the number of grams equivalent of the substance being titrated.

$$K_{sp} \text{ AgCl} = 1,0 \times 10^{-10}$$

$$[\text{Ag}^+] = [\text{Cl}^-]$$

$$[\text{Ag}^+]^2 = 1,0 \times 10^{-10}$$

$$[\text{Ag}^+] = 1,0 \times 10^{-5}$$

- To precipitate the CrO_4^{2-}

$$K_{sp} \text{ Ag}_2\text{CrO}_4 = 2 \times 10^{-12}$$

$$[\text{Ag}^+]^2 [\text{CrO}_4^{2-}] = 2 \times 10^{-12}$$

$$[\text{Ag}^+]^2 [10^{-2}] = 2 \times 10^{-12}$$

$$[\text{Ag}^+]^2 = 2 \times 10^{-10}$$

$$[\text{Ag}^+] = 1,4 \times 10^{-5}$$

From the example above, it can be seen that the number of silver ions needed to precipitate chromate ions is greater than that needed to precipitate chloride ions. So when TAT occurs, practically all of the chloride ions have precipitated, so that the silver chromate only settles after all the chloride ions have precipitated to form silver chloride.

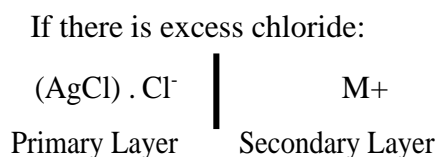
Things to consider in using the Mohr method:

1. Good for determining chloride and bromide ions but not suitable for iodide and thiocyanide ions.
2. Titrate in neutral or slightly alkaline conditions, pH 7 - 10.5.
3. Not suitable for the titration of colored solutions, such as CuCl_2 (blue), CaCl_2 (silver), NiCl (green) because it will be difficult to observe during TAT.
4. It is not possible for the Cl and Br salts which are hydrolysed because unexpected deposits are formed. For example, salts of Cl or Br with Al, Fe, Bi, Sn, Sb, and Mg cations.
5. The solution must not contain CO_3^{2-} , SO_4^{2-} , PO_4^{3-} , $\text{C}_2\text{O}_4^{2-}$ because it will settle with Ag.
6. The solution must not contain Pb^{2+} ions and Ba^{2+} because it will settle as a colored chromate salt. Removed by adding Na_2CO_3 saturated.

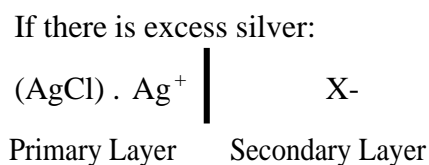
b. Fajans' Method

In this method an adsorption indicator is used. If an organic compound the color is adsorbed on the surface of a sediment, there can be modification of its organic structure, and the color can change greatly and can become older. This symptom can be used to detect the end point of a silver salt deposition titration.

The mechanism at which such an indicator works is different from any of the mechanisms discussed so far. Fajans discovered that fluorescein and some substituted fluoresceins can act as indicators for silver titrations. When silver nitrate is added to a sodium chloride solution, the very fine silver chloride particles tend to grab onto the surface (adsorb) the excess amount of chloride ions present in the solution. These chloride ions are said to form the primary adsorbed layer and thus cause the colloidal silver chloride particles to be negatively charged. These negative particles then tend to attract positive ions from the solution to form a loosely bound secondary adsorption layer.

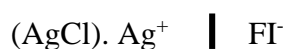


If silver nitrate is added continuously until excess silver ions, these ions will replace chloride ions in the primary layer. Then the particles become positively charged, and the anions in the solution are attracted to form a secondary layer.



Fluorescein is a weak organic acid which can be denoted by HFI. When fluorescein is added to the titration flask, its anion, FI⁻, is not absorbed by colloidal silver chloride as long as excess chloride ions are present. But if the silver ion is excess, FI⁻ ions can be attracted to the surface of the positively charged particles.

At the end point of the titration:



The resulting aggregate will be pink, and that color is strong enough to be used as a visual indicator. Figure 2.1 shows the molecular structure of the fluorescein

indicator. Figure 2.2 shows the events that occurred on when titrating the Fajans method.

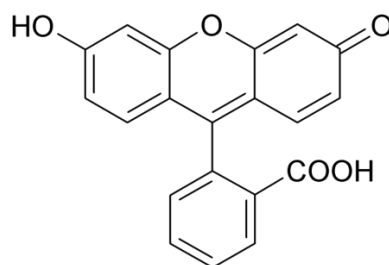


Figure 2.1 Structure of the fluorescein indicator molecule

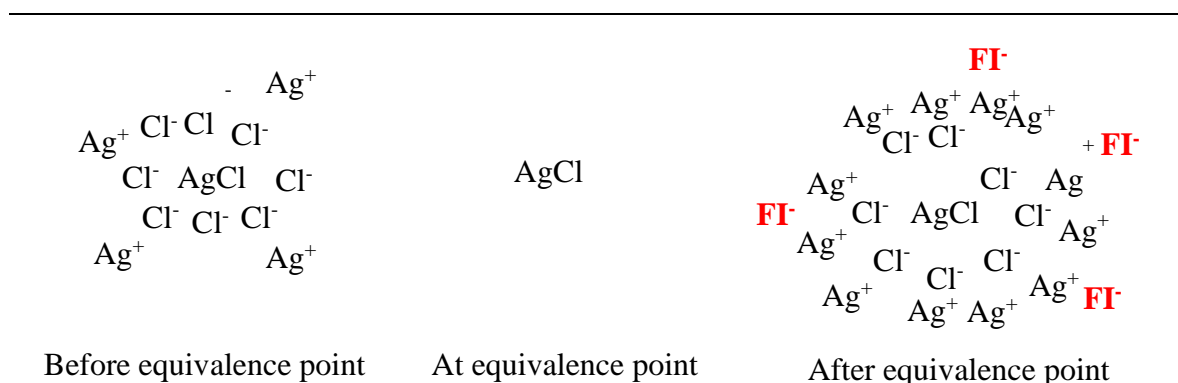


Figure 2.2 Events that occur in the Fajans method

Types of indicators commonly used include :

1. Fluorescein for chloride ions, pH 7-8 / dichlorofluorescein with pH 4.
2. Eosin for bromide, iodide, and thiocyanide ions, pH 2.
3. Bromocresol green for thiocyanide ions, pH 4-5.

Things to consider in using the Fajans method:

1. The solution should not be too dilute so that the discoloration can be clearly observed.
2. The indicator ion must be opposite to the titrant ion.
3. The sediment that occurs should be colloid so that the surface area of the absorber is large. May be added a coagulation inhibitor such as dextrans which keep the precipitate dispersed.
4. The indicator should not be adsorbed before the main ion has completely precipitated (before TE) but must be adsorbed immediately after TE has occurred.
5. The indicator that is absorbed by the bond precipitate must not be too strong because the indicator ion will be adsorbed by the precipitate before TE is reached.
6. Heating to a temperature of $\pm 80^\circ \text{C}$ is just titrated so that it supports the results of the observation.

c. Volhard's Method

This method is using *back to titration* principal, namely on the sample halogenide plus a standard AgNO_3 solution excess, then the remainder of AgNO_3 re-titrated with NH_4CNS standard solution. The indicator used is Ferric Ammonium Sulfate. In the process, the solution must be acidic in order to prevent the hydrolysis of ferric salts into ferric hydroxides, whose color disturbs TAT observations. An acidic atmosphere can be created by adding HNO_3 concentrated. But the use of HNO_3 not too concentrated because it can cause NH_4CNS is oxidized to NO and CO_2 .



In this method, the reaction mechanism will form silver chloride and silver thiocyanate.



Silver chloride is more soluble than silver thiocyanate, and it tends to re-dissolve according to the reaction.



The equilibrium constant for this reaction is determined by the ratio of the solubility product constant of silver chloride to silver thiocyanate. Since the first constant is greater than the second, the reaction tends to go from left to right. So the thiocyanate can be removed not only by the excess silver ion, but also by the silver chloride precipitate itself. If this were the case, the result would be too low a result in the chloride analysis. But this reaction can be prevented by filtering the silver chloride. For the determination of Br^- content, there is no need for filtering because the solubility product of AgBr is smaller than AgCNS , while for I^- the addition of the indicator after approaching TAT is because if I^- meet the Fe^{3+} indicator happened I_2 which often leads to titration errors.

Points to watch out for in the Volhard method:

1. The solution must be acidic, the aim is to avoid the hydrolysis of the ferric salt into ferric hydroxide whose color disturbs TAT observations.
2. HNO_3 which is used to provide an acidic atmosphere should not be too concentrated because it will oxidize NH_4CNS becomes NO and CO_2 . Where is

CO₂ formed can react with H₂O forms H₂CO₃ who can react with Ag⁺ and produce Ag₂CO₃ which is white so make it difficult to observe during TAT. In addition, Fe³⁺ content will decrease, so the chances of TAT going on are far away.

3. The AgCl precipitate that is formed must be filtered first, washed with water and washing water combined with the new filtrate titrated with NH₄CNS.

2.2 Physical Properties and Chemical Properties of Reagents

1. NaCl

a. Physical Properties

- BM = 58.45; BJ = 2.163 gr / cc; TD = 141.3°C; TL = 80.4°C.
- Crystal, colorless, cubic.
- Solubility in 100 parts of hot water = 39.8.
- Solubility in 100 parts of cold water = 25.7.

b. Chemical Properties

With AgNO₃ a water insoluble precipitate is formed.



2. AgNO₃

a. Physical Properties

- BJ = 4.35 g / cc; BM = 168,8; n = 1,744; TL = 213°C; TD = 244°C.
- Colorless solution.
- Solubility in 100 parts of hot water = 95.2.
- Solubility in 100 parts cold water = 22.

b. Chemical Properties

- With H₂SO₄ react to form a brown ring.



- With H₂S in an acidic / neutral atmosphere forms the Ag₂S precipitate.



- With Na₂CO₃ forming Ag₂CO₃ deposits yellowish white.



3. NH₄CNS

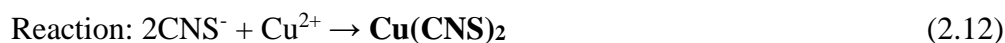
a. Physical Properties

- BM = 76.12; n = 1.685; TL = 147.6°C; TD = 170°C.
- Colorless solution.
- Solubility in 100 parts hot water = 170.

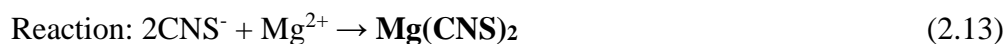
- Solubility in 100 parts cold water = 122.

b. Chemical Properties

- With CuSO_4 reacts to form $\text{Cu}(\text{CNS})_2$ precipitates.



- With $\text{Mg}(\text{NO}_3)_2$ forming a white precipitate of $\text{Mg}(\text{CNS})_2$.



- With $\text{FeCl}_3 \rightarrow$ is blood red.



4. HNO_3

a. Physical Properties

- $\text{BM} = 63.02$; $n = 1,502$; $\text{BJ} = 1.42 \text{ g / cc}$.
- Colorless solution.

b. Chemical Properties

- Turns blue litmus into red.
- Add alkaline to salt and water.



- Dissolved with nitrate salts.

5. K_2CrO_4

a. Physical Properties

- $\text{BM} = 126$; $\text{BJ} = 2,732 \text{ gr / cc}$; $\text{TL} = 97.5^\circ\text{C}$.
- Solubility in 100 parts of hot water = 75.6.
- Solubility in 100 parts cold water = 52.

b. Chemical Properties

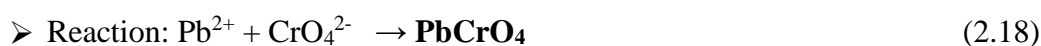
- With BaCl_2 react to form an insoluble light yellow precipitate in water but soluble in dilute mineral acids.



- With AgNO_3 forms a red brown precipitate that is soluble in nitrate acids.



- With Pb acetate it forms a yellow precipitate which is insoluble in acetate acids, but soluble in HNO_3 .



2.3 Reagent Function

- a. NaCl : To standardize AgNO_3 solution.

- b. AgNO_3 : To standardize the NH_4CNS solution and to precipitate Cl^- .
- c. K_2CrO_4 : As an indicator of the Mohr's method.

CHAPTER III

RESEARCH METHODOLOGY

3.1 Materials and Tools

3.1.1 Material

1. NaCl solution 0.05 N
2. AgNO₃ solution
3. K₂CrO₄ indicator 5%
4. Fluorescein indicator
5. Sample

3.1.2 Tool

1. Burette, Statif, and Clamp.
2. Funnel
3. Erlenmeyer 250 ml
4. Beaker Glass 250 ml
5. Measuring Cup 10 ml
6. Electric Stove
7. Thermometer
8. Pipette Volume 10 ml
9. Dropper Pipette

3.2 Procedure

1. Standardization of AgNO₃ with 0.05 N NaCl
 - a. 10 ml of NaCl standard solution of 0.05 N was taken and put into the Erlenmeyer flask.
 - b. Added 0.4 ml K₂CrO₄.
 - c. The solution is titrated with AgNO₃ until the first red color appears on the shuffling. Requirement for an AgNO₃ titrant noted. Normality of the AgNO₃ solution can be calculated using equation 3.1.

$$N_{AgNO_3} = \frac{(V.N)_{NaCl}}{V_{AgNO_3}} \quad (3.1)$$

N AgNO₃ : the normality of the AgNO₃ solution (N)

V NaCl : volume of NaCl solution titrated (ml)

N NaCl : normality of the NaCl solution titrated (N)

V AgNO₃ : the volume of the AgNO₃ titrant required (ml)

2. Determination of Cl⁻ levels using the Mohr's Method

a. 10 ml of the sample solution is put into the Erlenmeyer.

b. Added 0.4 ml (8 drops) of K₂CrO₄.

c. The solution is titrated with AgNO₃ until the first red color appears lost on shuffling. The need for a titrant is noted.

Cl⁻ levels can be calculated using equation 3.3.

$$\text{Level of Cl}^- = \frac{(V.N)_{AgNO_3} BM_{Cl} 1000}{V_{sample}} \quad (3.3)$$

Level of Cl⁻ : Cl⁻ level in the sample (ppm)

V AgNO₃ : the volume of the AgNO₃ titrant required (ml)

N AgNO₃ : the normality of the AgNO₃ titrant (N)

BM Cl : molecular weight of Cl (gram / mol)

V sample : volume of sample titrated (ml)

3. Determination of Cl⁻ levels using the Fajans' Method

a. 10 ml of sample solution is put into Erlenmeyer.

b. 10 drops of fluorescein indicator were added, the pH was adjusted 7-8, then heated to ± 80°C.

c. The solution is titrated with AgNO₃ until the first pink color appears which is not lost on shuffling. The need for a titrant is noted. Cl⁻

Cl⁻ levels can be calculated using equation 3.4

$$\text{Level of Cl}^- = \frac{(V.N)_{AgNO_3} BM_{Cl} 1000}{V_{sample}} \quad (3.4)$$

Level of Cl⁻ : Cl⁻ level in the sample (ppm)

V AgNO₃ : the volume of the AgNO₃ titrant required (ml)

N AgNO₃ : the normality of the AgNO₃ titrant (N)

BM Cl : molecular weight of Cl (gram / mol)

V sample : volume of sample titrated (ml)

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CHAPTER I

INTRODUCTION

1.1 Background

In determining the levels of insoluble compounds, certain methods are applied which involve the principle of deposition. Precipitation is a very important method in separating a sample into its components. The process involved is a process where the substance to be separated is used to form a solid precipitate. A precipitate is a substance that separates itself as a solid phase out of solution. Analysis with the depositional principle has been used extensively in analytical chemistry, especially in argentometric and gravimetric methods.

Gravimetry is also a part of quantitative analysis that deals with weight measurement by separating the analyte from all other components so that the content of a substance can be determined. The initial stage of gravimetric analysis is the separation of the components you want to know from other components contained in a sample, then deposition into the form of stable and pure compounds that can be measured. Measurement in the gravimetric method is by weighing. The number of components analyzed is determined from the relationship between the weight of the sample to be analyzed, the relative atomic mass, relative molecular mass, and the weight of the precipitate the reaction results. Apart from inorganic substances, organic compounds have also been analyzed using gravimetric techniques, for example the determination of cholesterol levels in cereals and lactose in milk products.

1.2 Experiment Objectives

The purpose of this experiment was to analyze Ba^{2+} levels in the sample.

1.3 Experiment Benefits

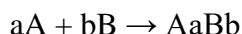
The benefit of this experiment is that students can understand and have skills in analyzing Ba^{2+} levels in a sample by a gravimetric procedure.

CHAPTER II

LITERATURE REVIEW

2.1 Gravimetric Analysis

Gravimetric analysis is a method of measuring weight by separating the analyte from all other components so that the content of a substance can be determined using a gravimetric factor. A gravimetric analysis is usually based on the reaction:



With the provision that a is the analyte A reacts with b molecule B. The result of AaBb is usually a substance with a small solubility so that it can be weighed in that form after drying or burning into another compound whose composition is known and then weighed. An excess of reagent B is usually added to suppress the solubility of the precipitate, for example in the determination of Ca^{2+} .



The requirements that must be met in the gravimetric method are :

- a. The separation must be sufficiently perfect so that the quantity of the analyte which does not precipitate the analyte is not determined.
- b. The substance being weighed must have a certain order and must be pure. Otherwise no results can be obtained.

2.2 Gravimetric Analysis Application

Gravimetry can be carried out on organic substances such as the determination of cholesterol in grains. In addition, analysis of elements and organic compounds is usually carried out in this way. For example the C in an organic compound can be determined by burning the sample in oxygen and absorbing CO_2 and H_2O is generated at suitable absorption. The absorption tube is weighed before and after combustion to obtain CO_2 and H_2O is generated.

2.3 Gravimetric Advantage

Although gravimetry has been replaced from a routine point of view with instrumentals, gravimetry is actually faster and more precise than the instruments that need to be calibrated. Tools generally provide relative measurements only and must be

calibrated on the basis of classical gravimetric or titimetric methods. If the analyte is an The first constant ($> 1\%$) the accuracy of the various thousandth parts can be expected, if the sample is not very complex. If the minority analyte is less than 1%, the gravimetric method is usually not used.

2.4 Coprecipitation and Post Precipitation Theory

Coprecipitation is the process of bringing down a substance that is usually dissolved during the precipitation of the desired precipitate. Suppose that the nitrate ion in the deposition of barium sulfate causes the precipitate to contain barium nitrate so that the nitrate is said to experience coprecipitation with sulfate or due to ion adsorption during the precipitation process. In this event, the substances that cause impurities enter the crystal side and the ions that are absorbed are dragged down during coagulation (Underwood, 1998: 74).

Procedures used to reduce coprecipitation:

- a. The method of determining these 2 reagents can be used to control the concentration of regulating substances and the electric charge carried by the primary particles of sediment in controlled by using the appropriate pH.
- b. Expansion with lumps and gelatin should be with a solution of electrolytes in a washing solution to avoid precipitation.
- c. This contamination is a great benefit of crystalline deposits, which is quite a benefit for agglomerated deposits still not used for gelatin.
- d. Re-deposition if the precipitate can easily be dissolved again, especially for hydrolysis oxidation and crystalline fatty acid salts.
- e. The impurity separation can be separated / its chemical properties changed by a wash before a precipitate is formed.
- f. The use of a requirement that leads to larger particles, that is, if the deposition is slow enough.

Post precipitation is a process in which an impurity is deposited after the deposition of the desired substance. This process is different from coprecipitation. The amount of impurity increases with the longer the desired precipitate is allowed to be in contact with the mother liquor. Mother liquor is a chemical standard solution that is made with high levels and is used to make standard solutions with lower levels. With the presence of these impurity deposits, it is certain that the weight of the deposits will increase. The fix

is to use the highest acidity possible and the precipitate is filtered one or two hours after deposition (Underwood, 1998: 77-78).

2.5 Physical Properties and Chemical Properties of Reagents

1. H_2SO_4

Physical Properties :

- Molecular weight = 98.08 gr / mol
- Specific gravity = 1.83 gr / cc
- Boiling point = 337 ° C
- Melting point = 10.44 ° C
- Solubility in 100 parts of cold water = 80
- Solubility in 100 parts of hot water = 59

Chemical Properties :

- Is a strong acid.
- If you add a base to form salt and water.

- With Pb^{2+} formed PbSO_4



- With Ba^{2+} formed BaSO_4



2.6 Reagent Function

H_2SO_4 : forming the BaSO_4 deposit

CHAPTER III

RESEARCH METHODOLOGY

3.1 Materials and Tools

3.1.1 Material

1. H_2SO_4 0.1 N sufficiently
2. H_2SO_4 very watery sufficiently
3. Aquadest sufficiently
4. Sample

3.1.2 Tool

1. Whatman filter paper
2. Stirrer
3. Funnel
4. Beaker Glass 250 ml
5. Measuring Cup 10 ml
6. Dropper Pipette
7. SHZ-D(III) Water Aspirator

3.2 Instruments



Figure 3.1 SHZ-D(III) Water Aspirator

Figure information:

1. Switch
2. Tap/Port
3. Power
4. Motor hood net
5. Vacuum gauge
6. Box



Figure 3.2 Vacuum filtration set

Figure information:

1. Filter cup
2. Rubber stopper
3. Collecting flask
4. Rubber vacuum hose
5. Connector

3.3 Procedure

3.3.1 Preparation of vacuum filtration set

1. Join the rubber vacuum hose to the connector in the collecting flask from Port I in the SHZ-D(III) water aspirator.
2. Apply the filter cup to the collecting flask with the rubber stopper.
3. Connect SHZ-D(III) water aspirator to the electricity socket
4. Turn on SHZ-D(III) water aspirator by switching up the lever upwards towards ON.

3.3.2 Filtering solution with Whatman filter paper

1. Weigh Whatman filter paper.
2. Take 10 ml of sample containing Ba^{2+} (volume taken depends on sample concentration).
3. Add H_2SO_4 0.1 N and stir well.
4. Filter BaSO_4 (white fine crystal) deposits with Whatman filter paper that is placed in the filter cup. Filtrate is collected in the collecting flask.
5. Wash the precipitate with dilute H_2SO_4 . The washing solution is then combined with the previous filtrate and add H_2SO_4 0.1 N again.

6. Steps 4 and 5 are repeated until the addition of H₂SO₄ does not create anymore sedimentation.
7. Dry the precipitate in the oven at 100-110°C.
8. After drying, cool the Whatman filter paper in the dessicator and weigh the filter paper.

Ba²⁺ concentration can be calculated using equation 3.1.

$$\text{Ba}^{2+} \text{ Concentration} = \frac{(W_2 - W_1) \times BM \text{ Ba} \times 1000}{BM \text{ BaSO}_4 \times V \text{ sampel}} \times 1000 \quad (3.1)$$

Ba ²⁺ concentration	: Ba ²⁺ concentration in sample (ppm)
W1	: Weight of filter paper in the first instance (gram)
W2	: Weight of filter paper with deposit (gram)
BM Ba	: Molecular weight of Ba (gram / mol)
BM BaSO ₄	: Molecular weight of BaSO ₄ compound (gram / mol)
V sample	: Volume of sample used (ml)

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