

Precision in Practice: Comparative Analysis of Gravimetric and Volumetric Methods for Nickel(II) Chloride Quantification

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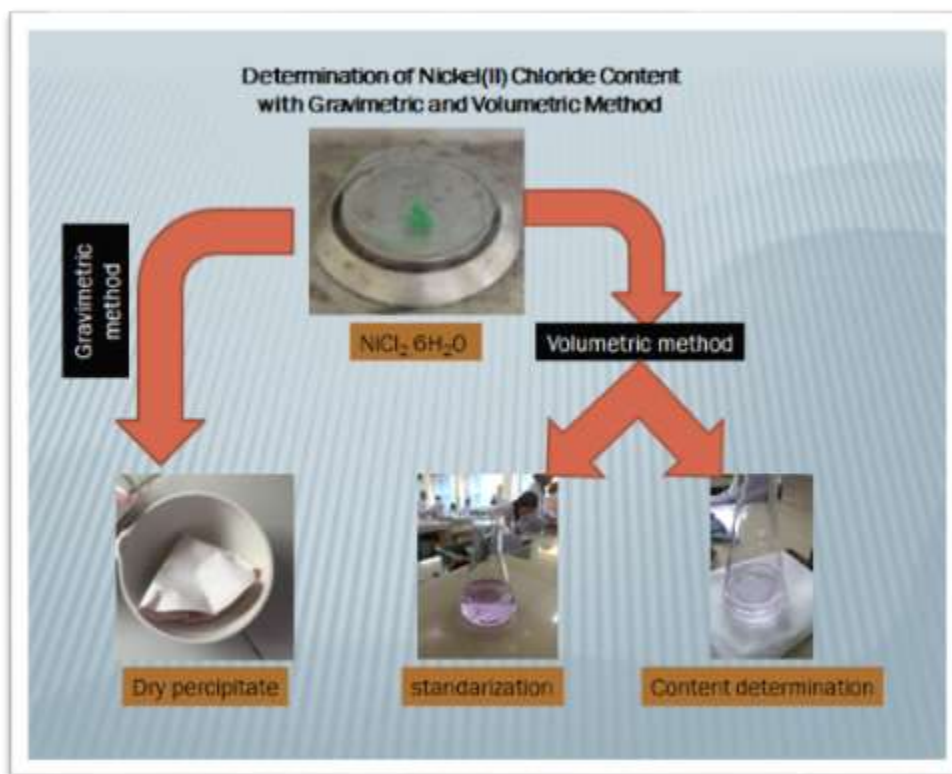
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Abstract: This study was conducted to determine the content of Nickel(II) Chloride In $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ by gravimetric and volumetric methode. In gravimetric method the content of Nickel(II) Chloride was determined using repety of heating and weighing sample with several kinds of laboratory equipment such as, watch glass, beaker, steam bath, oven desicator, etc. in gravimetric we using Chloride Acid 6M, H_2DMG 1% solution, and NH_4OH dilution for the materials. In gravimetric method the content of Nickel(II) Chloride is 21,95%. while in volumetric method the content of Nickel(II) Chloride was determined using titration with $\text{Hg}(\text{NO}_3)_2$ solution with diphenyl carbazone indicator in sample the tools are important in this method is Burrete and titration flask. the first step is standarization of $\text{Hg}(\text{NO}_3)_2$ with NaCl to determine concentration of titrate that is **0,0205 M**. from volumetric method we got the content of Nickel(II) Chloride is 82,64%. So in this studied we can conclude that volumetric method more accurate than gravimetric method.

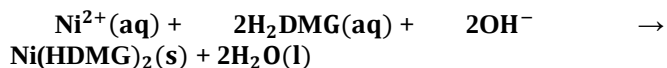


Keywords—component; content determination, gravimetric method, volumetric method

1. INTRODUCTION

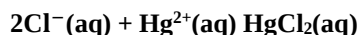
Gravimetric Experiment: In gravimetric analysis, the substance to be analyzed (analyte) is isolated from solution through a precipitation process. Analytes are generally

precipitated by adding reagents that form compounds that are difficult to dissolve with the analyte. In determining the NiCl_2 in the sample solution, nickel ions were precipitated by adding dimethyl glyoxime ions (in ethanol) to form a red precipitate of $\text{Ni}(\text{HDMG})_2$ according to the reaction:



The usual precipitate is obtained in slightly alkaline conditions (ammonia) or using ammonium acetate buffer with acetic acid. The precipitate is weighed after drying at a temperature of 110-120°C. Use of H_2DMG should only be a little excess, because if there is too much excess there is a possibility that H_2DMG itself will precipitate because its solubility in water is low and the solubility of the precipitate in the ethanol-water mixture is greater than in water.

Volumetric: In the volumetric method, the level of NiCl_2 is determined from the level of chloride ions present in the solution. The determination was made by titration with Hg^{2+} according to the reaction



This complex formation reaction proceeds through several stages, with the most stable form being $\text{HgCl}_2(\text{aq})$. The end point of the titration is determined using the diphenyl carbazone indicator which will form a purple complex with Hg^{2+} . This method is known as the mercurimetric method, which can be used for the determination of chloride ions in very dilute solutions (0-100 ppm). Bromide, thiocyanate and cyanide ions can also be determined by this method. (Tim Kimia Analitik, 2022)

Precipitation is the most frequently used method in qualitative analysis practice. The appearance of precipitate as a result of a certain reaction can be used as a certain ion. However, precipitation can also be used for separation. To do this a suitable reagent is added, which forms a precipitate (precipitates) with only one or a few ions present in solution. After adding the appropriate amount of reagent, the precipitate was filtered and washed. The ease with which a precipitate is filtered and washed depends in large part on the morphological structure of the precipitate, namely on the shape and size of the crystals. A very important problem in gravimetric is the formation of a pure and filterable precipitate. Further elaboration of this problem can be obtained by studying the deposition rate at which the particles turn into agglomerates large enough to separate from the solution as a precipitate. (Mulyati, 2020)

Are titration that involve the formation of a precipitate of an insoluble salt between the titrant and the analyte. The basic requirements of this type of titration are the achievement of a rapid build. Up equilibrium each time the titrant is added to the analyte, no interference to interfere with the titration, and an easily observable titration end point. One type of precipitation titration that has long been known involves the precipitation reaction between halide ions and silver Ag^+ ions. This titration

is usually referred to as, namely the titration of the determination of analytes in the form of halide ions using a standard solution of silver nitrate. Titration can not only be used to determine mercaptants, fatty acids, and several divalent anions such as phosphate ion and arsenate ion. With analytes, an example that is widely used is titration for the determination of NaCl in which ions from the titrant react with Cl^- to form a salt that is not. (Ratnasari, 2021)

Gravimetry in chemistry is a known method of quantitative analysis of a substance or a component by measuring the weight of a component in its pure state after going through a separation process. Gravimetry analysis is the process of isolating and measuring the weight of particular element or compound. The gravimetry method is time consuming, the presence of impurities in the constituent can be tested and if necessary correction factors can be used.

Nickel is a hard, silver – metal. Nickel is ductile, malleable and very tough. This metal melts at 1445°C, and is slightly magnetic, stable salts, colored of nickel (II), derived from nickel (II) oxide, NiO , which is a green substance. Dissolved nickel salts, colored green, are due to the color of the hexaamminenickelate (II) complex. A very special reaction of Ni^{2+} which can be used for both qualitative and quantitative analysis is the formation of a neutral complex. In which a bright red precipitate is produced. In addition to coordinating bonds between N and Ni^{2+} , there are also hydrogen bonds in this complex compound. (Rustaman, 2017).

2. MATERIALS AND METHODS

2.1 Materials

1. First, DMG solution 1%
2. HCl 6M
3. NH_4OH Dilution
4. AgNO_3 solution
5. $\text{Hg}(\text{NO}_3)_2$ 0,01 M
6. NaCl (p.a)
7. Diphenyl Carbazone
8. HNO_3 2M

2.2 Method

a. The Gravimetric Method

In the gravimetric method we use analytical balance to weigh the sample and precipitate, and then we dilute the sample in beaker and we adding with 5 mL HCl 6M and we heating the solution. In 70-80°C adding 35-40 mL H_2DMG 1% and ammonia dilution until from red precipitate and we leave in steam bath 20-30 minutes. Then the precipitate cooling in 1 hour, filtering and washing the precipitate - test filtrate with

AgNO₃ until the filtrate colorless- and we dry in oven for 45-55 minutes ,then after cooling the precipitate, weigh the precipitate occurs.

b. Volumetric Method

In standarizatin using 25 mL NaCl dilution , 25 mL aqua DM, 1 mL HNO₃ 2 M and few drops diphenyl carbazone indicator in titration flask and Hg(NO₃)₂ for titrate, and titrate until the purple color occurs. And then for determination content. The sample we dilute with 100 ml of water and we put 25 mL of sample, 25 mL aqua DM, 1 mL HNO₃ 2M, and few drops dhiphenyl Carbazone in titration flask, and titrate until the purple color occurs. Do duplo for both experiment

3. RESULT AND DISCUSSION

Result

A. Gravimetric

Weight watch glass : 24,7423 grams

Weight watch glass + sample : 25,3373 grams

Weight sample : 0,5950 grams

Weight filter paper : 1,2602 grams

Weight evaporating dish : 66,7229 grams

Weight filter paper + evaporating dish + precipitate : 68,6228 grams

Weight precipitate : 68,6228 grams –
(66,7229+1,2602) grams = 0,0297 grams

B. Volumetric

- Standardization Of Hg(NO₃)₂ Solution

Weight NaCl : 0,3 grams

Weight watch glass : 20,0980 grams

Weight watch glass +NaCl: 20,3980 grams

- Determination Of Chloride Content

Dilute 10 ml NiCl₂ until 100 ml

C NiCl₂ : 0,025

Volume titran :

- V1 = 4,50 ml

- V2 = 4,20 ml

V rata-rata = 4,35 ml

3.1 CALCULATION

A. Gravimetric

➤ Theory

$$\% \text{ Ni} = \frac{\text{Ar Ni}}{\text{Mr NiCl}_{2.6\text{H}_2\text{O}}} \times 100\%$$

$$= \frac{59}{238} \times 100 \%$$

$$= 24,79 \%$$

➤ Prattice

$$\text{Gravimetric factor : } F_6 = \frac{\text{Ar Ni}}{\text{Mr Ni(HDMG)}_2} = \frac{59}{289} = 0,2042$$

Mass of precipitate : 0,6397 grams

$$\begin{aligned} \% \text{ Ni} &= \frac{\text{mass of precipitate} \times F_6 \times 100\%}{\text{mass of sample}} \\ &= \frac{0,0397 \text{ grams} \times 0,2042 \times 100\%}{0,595 \text{ grams}} \\ &= 21,95\% \end{aligned}$$

B. Volumetric

➤ Standardization Of Hg(NO₃)₂ Solution

$$\text{M NaCl} = \frac{\text{g}}{\text{Mr}} \times \frac{1000}{\text{V(ml)}} = \frac{0,3 \text{ grams}}{58,5 \text{ g/mol}} \times \frac{1000}{250 \text{ ml}} = 0,0205 \text{ M}$$

$$\text{M Hg(NO}_3)_2 = \frac{(\text{V.M})\text{NaCl}}{\text{V Hg(NO}_3)_2} = \frac{25 \text{ ml} \times 0,0205 \text{ M}}{15,9 \text{ ml}} = 0,0322 \text{ M}$$

➤ Determination Ni Content with Volumetric Method

V sample = 10 ml → 100 ml → V = 25 ml

M Hg(NO₃)₂ = 0,0322 M

V Hg(NO₃)₂ = 4,35 ml

Ar Ni = 59 g/mol

$$\begin{aligned} \text{a. } \text{Mg NiCl}_2 &= (\text{V} \times \text{M}) \text{Hg(NO}_3)_2 \times \text{Mr} \\ &= 4,35 \text{ ml} \times 0,0322 \text{ M} \times 238 \\ &= 33,47 \text{ mg} \end{aligned}$$

$$\begin{aligned} \text{Mg Ni} &= \text{V.M Hg(NO}_3)_2 \times \text{Ar} \\ &= 4,35 \text{ ml} \times 0,0322 \times 59 = 8,26413 \text{ mg} \end{aligned}$$

$$\begin{aligned} \text{b. } \text{ppm NiCl}_2 &= \frac{\text{mg}}{\text{v sample (l)}} = \frac{33,37 \text{ mg}}{0,025 \text{ l}} = \\ &= 1.334,8 \text{ ppm} \end{aligned}$$

$$\text{ppm Ni} = \frac{\text{mg}}{\text{v sample (l)}} = \frac{8,76413 \text{ mg}}{0,025 \text{ l}} = 330,5652 \text{ ppm}$$

$$\begin{aligned} \text{c. } \% \text{ Ni} &= \frac{\text{mg Ni}}{\text{v sample}} \times 100\% \\ &= \frac{8,26413}{10 \text{ ml}} \times 100\% \\ &= 82,64\%. \end{aligned}$$

4. DISCUSSION

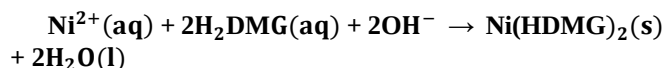
In this practicum, the determination of nickel (II) chloride content in samples was carried out using gravimetric and volumetric methods. The sample used is NiCl₂.6H₂O.

A. Gravimetric Method

Gravimetry is a known method of quantitative analysis of a substance or component by measuring the weight of the component in its pure state after going through a separation process. (Tim Kimia Analitik, 2022)

In the experiment, the aim was to determine the nickel content of a nickel salt by precipitating nickel ions (Ni^{2+}) in the form of nickel dimethyl glyoximate. Nickel ions are precipitated in the form of nickel dimethyl glyoximate by adding a solution of dimethyl glyoxime (DMG) to the sample. In order to obtain a large precipitate, the solution must be made in an acidic environment and under hot temperature conditions. Therefore the addition of 6 M HCl and heating is carried out so that the resulting precipitate has a large size as expected. The addition of 6 M HCl, in addition to affecting the size of the precipitate formed, also aims to prevent the early precipitate of Nickel dimethyl glyoximate $[\text{Ni}(\text{HDMG})_2]$ when H_2DMG is reacted with the sample. If this early deposition occurs, H_2DMG is a white solid organic compound that is poorly soluble in water but soluble in organic reagents (Mulyati, 2020).

The addition of dimethyl glyoxime is done with a little excess, if the addition of H_2DMG is done too much it will result in the formation of H_2DMG crystals which will not dissolve in water. The crystals formed will mix with the precipitate and thus the resulting precipitate weight will be much larger than it should be. A dilute ammonia solution is added to make the solution neutral and then alkaline. Because Nickel dimethylglyoximate $[\text{Ni}(\text{HDMG})_2]$ will precipitate completely in alkaline conditions. The addition of dilute ammonia was carried out until there was no spontaneous reaction in the form of a color change from the solution when diluted ammonia was added. (reaction) :



The precipitate is dried in an oven at 105-110°C at this temperature the water component in the precipitate will evaporate completely. The coolant in the desiccator aims to absorb heat in the sample so that the weight obtained is more stable.

Based on the calculation results, it was found that the nickel content in the sample was 21,95 %, theoretically the nickel content in the sample $\{\text{NiCl}_2 \cdot 6\text{H}_2\text{O}\}$ was 24.79%. The reasons for the levels obtained were not in accordance with the literature, namely during the filtering process the precipitate still remained on the walls of the glass beaker, another possible cause was imperfect washing.

B. The Volumetric Method

In this pratikum we using volumetric method or titimetric method. Titration is a volumetric terchnique which involves the use of known volumes of a known substance to quantitatively determination the amount of a specific substance

in an unknown given sample. This the technique is one of the fundamental analytical technique in chemistry and is applied in life sciences, clinical chemistry, water polution, etc.

In volumetric using mercurymetric method. Mercurymetry means using mercury salts as the titrant and using halogen salts, namely CN^- ions and CNS^- ions as titrate, the compounds to be leveled. The indikator used in this mercurymetric titration is diphenyl carbazone (Mulyati, 2020).

Mercurymetry can be carried out directly or indirectly, depending on the titrate of the complex compound to be formed. The indirect method is used when it's difficult to observe the end point of the complex compound formed directly, so that by using the indirect method of forming complex compounds with other titrans the end point can be easily observed, because in an indirect titration two different titrans are used. In the experiment, indirect titration was used because the reseulting titrant was different and easy to observe (Ratnasari, 2021).

The standard solution of $\text{Hg}(\text{NO}_3)_2$ must first be standardized using a NaCl solution because $\text{Hg}(\text{NO}_3)_2$ is a secondary standard solution, so it must be standardized using a NaCl solution which is a primary standard solution. The purpose of standardization is to determine the actual concentration of $\text{Hg}(\text{NO}_3)_2$ and standardization is necessary, because secondary standard solutions are ususally unstable when stored for a long time. While the primary standard solution chosen usually has stable properties, if stored for a long time (Rustaman, 2017)

In this volumetric method, the NiCl_2 content is determined form the chloride ion content present in the solution. We use $\text{Hg}(\text{NO}_3)_2$, so this called mercurymetric which can be used for determination of chloride ion in very dilute solution.

We use diphenyl carbazone indicator that will from a purple complex with Hg^{2+} ion. The end point that we get in our praticum is 4,35 ml and then from calculation Ni content in 330,5652 ppm, and then % Ni from titration is 82,64%.

For standardization we use NaCl 0,0205 M and the volume of titration is 15,9 ml, so the concentration of $\text{Hg}(\text{NO}_3)_2$ is 0,0322 M. To determination content we should calculation mass in mg, so we get $\text{NiCl}_2 = 33,37$ mg and $\text{Ni} = 8,2613$ mg.

So, we compared the both method, the titrimetric or volumetric better than gravimetric. We should know in result and the time to determination content, in volumetric faster than gravimetric and the result in volumetric better than gravimetric

5. CONCLUSIONS

The conclusions that can be drawn from this experiment are:

- Gravimetry is a known method of quantitative analysis of a substance or component by measuring the weight of the component in its pure state after going through a separation process. Nickel content in the sample by gravimetric determination of 21,95%.
- Volumetric is an analytical method in which the concentration and composition of the sample is determined based on the volume of the reagent (known volume) added to the solution of the test substance to the components of the volumetric analysis. Nickel content in the sample by volumetric determination of 82,64 %

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