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Applications of Microscale Technique in Gravimetric Analysis of Iron to Create Eco-Friendly Atmosphere in the Laboratory

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Abstract: The present process of conducting the laboratory experiments particularly in Chemistry are based on quantitative analysis by conventional techniques are becoming more expensive due to increasing prices of chemicals and apparatus required at large scale, requires minimum six to eight hours for its completion ie time consuming, involves wastage of chemicals and energy, requires concentrated acids at large scalet, requires large volume of ammonia, creates polluted atmosphere in the laboratory. The special efforts are taken in this study to carry out the same experiment by applying microscale techniques. So gravimetric analysis of Fe as a Fe₂O₃ in presence of Copper was carried out by using microanalysis technique. Ferrous ammonium sulphate with little sulphuric acid was used as a source of Fe and it contains a contamination of CuSO₄.Copper is precipitated as a CuS is separated and removed by filtration and the Fe was estimated as a Fe₂O₃ by precipitation as Fe(OH)₃. The study have shown that, there is no conceptual deviation from the results as well as principles of analysis was observed. It has reduced the expenditure on chemicals to a great extent. The apparatus used are of smaller size. This technique requires minimum time, save the energy of a student while performing the experiments, also offers economy of fuel and also minimizes the pollution hazards. The creation of eco-friendly environment in the laboratory was observed which is a need of present world.

Keywords: Gravimetric analysis, micro scale technique, cost, time, energy, pollution, economy.

Introduction: Students as well as teacher commonly we call it as Chemists when works in the chemical laboratory which consists of equipments, glassware, hazardous chemicals, inflammable liquids etc. The chemist should be safer and comfortable while working with these materials. The creation of eco-friendly environment in the laboratory is need of present world. Students as well as teachers should aware about the chemical waste generated in a Chemical laboratory like water soluble, insoluble chemicals, solvents, acidic, alkaline material etc. and solid waste such as filter papers, drying solvents, broken glassware, Cotton, metal foil etc. to minimize the waste and make the laboratory processes more economical present need is to start with using micro analysis techniques in laboratory instead of conventional methods. The technique has not only its own advantages but also is a new technique for modern

advanced laboratory. Most of the chemicals are dangerous, toxic, hazardous, and inflammable and if not handled rightly can lead to uncontrolled processes. Micro analysis avoids the excess use of the chemicals as very little amount is required at a time. This technique is safe for flammable, fire hazard chemicals and reagents. The direct contact of fire has been avoided in the Micro analysis method. Overheating, bumping, splashing of liquids and vapors are prevented. The utilization of water and gas is also less in this technique. Thus Micro analysis is safe and environmental friendly under green roof.

Material and Method: All the chemicals were used was pure and Glassware were calibrated.

Whatman filter papers -40, 41, 42 glass fannels, silica crucibles with lids, Asbestos sheets, wire gauze, droppers, measuring cylinders 25 ml and 50ml, Beakers, 250 ml and 100 ml glass rods, metallic cones, oven etc.

We have adopted a scheme of using micro scale technique in gravimetric experiment. This has been done without any conceptual deviation of the principle of experiments in chemistry where quantities were used on gram scale, we used it on milligram scale. The apparatus used is of smaller size.

Our Initiative: Our initiative in scaling down the quantities began as a curiosity. We tried this experiment on a group of 25 students of B.Sc, who have taken Chemistry as the principle subject.

Experimental Method: The scaling down of quantities was done for the experiments gravimetric analysis, If the same solution to be determined is taken in smaller quantity then the precipitating agent, washing liquid, other reagents and chemicals are also required in smaller quantities. This results in economy of time, and consumption of chemicals and develops eco friendly atmosphere in the laboratory.

Gravimetric analysis is a process of converting an element or a compound to be determined, into a form which is suitable for weighing. This form must be stable and have a definite composition.





Gravimetric analysis can be done by using organic precipitants e.g. Ni as as Ni DMG or by using inorganic precipitants e.g. Fe as fe (OH)₃ and then as fe₂O₃. The precipitates obtained by using organic precipitants are usual Voluminous bulky and need only drying while the precipitate obtained by using inorganic precipitants are crystalline or gelatinous or colloidal. They require heating at high temperature for drying and ignition. The process of precipitation using inorganic precipitants involves many steps and is a laborious and time consuming process.

As an illustration we consider here gravimetric estimation of fe as fe2O3 in presence of copper, Usually ferrous ammonium sulphate with little sulphuric acid is given as source of fe and it contains a contamination of CuSO₄.

Principle: Copper is precipitated as Cus separated and removed by filtration and then fe is estimated as Fe₂O₃ by precipitation as Fe(OH)₃

The conventional techniques of gravimetric analysis requires six to eight hours and Involves wastage of chemicals and energy (Fuel), Use of concentrated acids in large amounts and requires a lot of ammonia for precipitation which results in polluting the laboratory atmosphere.so it is necessary to carry out the gravimetric analysis on small scale.In the study of gravimetric analysis

of Fe as a Fe_2O_3 in presence of Copper was carried out by using microanalysis technique. Ferrous ammonium sulphate with little sulphuric acid was used as a source of Fe and it contains a contamination of $CuSO_4$. Copper is precipitated as a CuS is separated and removed by filtration and the Fe was estimated as a Fe_2O_3 by precipitation as $Fe(OH)_3$. The study have shown that, there is no conceptual deviation from the results as well as principles of analysis was observed and also reduced the consumption of chemicals.

Procedure:

- 1. Taking a stock solution: 10 ml of the stock solution was taken and diluted it to 100 ml. using distilled water. 25 ml of this diluted solution was taken by a burette in a 250 ml beaker for estimation of iron. Normally 25 ml of the stock solution is taken and it is diluted to 250 ml using distilled water and 50 ml of the diluted solution is taken for estimation of iron. we have reduced here the volume of the stock solution from 25 ml to 10 ml and the volume of the diluted solution from 50 ml to 25 ml)
- **2. Dilution:** Add 25 ml (equal volume) of distilled water (Normal procedure describes addition of

double the volume of water. We avoided here unnecessary dilution sine it is troublesome at a later stage)

- **3. Acidification:** Added 5 ml of dilute H₂SO₄ (not the dilute HCl). The solution should be acidic because copper is precipitated as CuS in acidic medium. (If HCl is added in place of H₂SO₄, it creates difficulty in removing chloride ions at the end). Warmed the solution for 2 minutes.
- **4) Precipitation of Copper:** Pass H₂S for 3 minutes. It proves to be enough in excess.

Advantage: Copper is precipitated as CuS in one lot

Disadvantage. Time taken for removal of H₂SO₄ is more but marginally)

- 5) Warming the Solution: The solution was Warmed for 2 minutes. If colloidal precipitate is formed, the particles get coagulated due to warming and settles down.
- **6) Filtration:** Filtered through ordinary filter paper by decantation method. washed the ppt. in the beaker itself by not water 3 to 4 times. Used small aliquots of 10 to 15 ml for each washing.

Advantage:

- i) Time required is less
- ii) The Fe2+ ions adsorbed on the precipitate of CuS and filter paper are removed in just 3 to 4 washing
- 7) Boiling the solution: The filtrate was boiled for about a minutes (In normal procedure, the large volume (about 500 to 600 ml) is reduced to 150 ml by boiling. In our procedure, the volume is already less, so the solution is boiled to remove (It was tested with lead acetate paper)
- 8) Oxidation of Fe^{2+} to Fe^{3+} The Fe^{2+} Salts usually do not get precipitated completely. So they are converted to Fe to Fe^{3+} salts. Usually do not get precipitated completely. So they are converted to Fe3+ salts before precipitation. Addition of conc. HNO₃ was done drop by drop till the color of the solution becomes distinct yellow. About 2 to 3 ml conc. HNO₃ was found to be enough. If concenterated HNO₃ is added more in amount ammonia required for neutralization of HNO₃ and precipitation of Fe as a Fe (OH)₃ ions mole If Conc. HNO₃ is added less in amount complete oxidation of Fe^{2+} to Fe^{3+} does not take place
- 9) Addition of NH₄CI: This is a crucial step.

The normal procedure recommends addition of one gram of NH₄C113





Role of NH₄Cl: – It creates common ion effect and prevents the precipitation of other cation as hydroxides.

Excess addition of NH₄Cl creates difficulties during the removal of Cl ions.

The process of washing becomes hectic and \time consuming because the precipitate of Fe(OH)₃ is colloidal in nature and shows a tendency to adsorb lions. Hence only the minimum and required quantity of NH₄Cl should be added (Refer to the calculate ions of quantitative requirements of HN₄Cl and NH₄OH)

- 10) Precipitation of ferric iron as ferric Hydroxide: The normal procedure recommends use of 1:1 ammonia (Which is nearly 7 normal) for the precipitation of Fe as Fe(OH)₃. (Sometimes written as Fe₂O₃ x H₂O ie. Hydrated ferric hydroxide). The concentration of ammonia is high. We can also add 2N ammonium hydroxide (table reagent) but then the volume required more. Add 1:1 ammonia solution drop by drop till Fe₃+ is completely precipitated as Fe (OH)₃. Initial few drops of ammonia solution are required to neutralize the HNO₃ present in the solution.
- 11) Filtration: Filtered the precipitate of Fe(OH)₃ by decantation method through whattman paper no 41.
- 12) Washing the precipitate: Washed the precipitate of Fe(OH)₃, by hot 2% NH₄NO₃ Solution. Since the precipitate is colloidal in nature, the strong electrolyte NH₄NO₃ helps to coagulate it and to remove the adsorbed Cl ions. About 4 washing of 15 ml. about each are enough to remove the adsorbed Cl ions (Test it with Ag solution). This procedure reduces the time of washing the precipitate.
- 13) Ignition of the precipitate: lignite the precipitate of Fe (OH)₃ with the ash less filter paper in a previously weighed silica crucible.. Heated the crucible in a strong oxidizing flame to about 800 °C for half an hour so that all Fe(OH)₃was converted to FeO.
- 14. Weighing the residue: The residue of Fe₂O_{3 was} Weighed and determined the amount of Fe in the give in the sample solution.

Result and Discussion:

Advantage: The gravimetric analysis of Fe ie the determination of the amount of Fe in the give sample solution was completed in minimum time and the Fe2+ ions adsorbed on the precipitate of CuS and filter paper are removed in just 3 to 4 washing.

Now, Chemistry Laboratories in academic institutions consume large amounts of chemicals due to increasing demand by students. The ever rising cost of chemicals is adversely affecting the practical exercises. The fumes and gases evolved during chemical reactions are threatening the environment. The awareness of eco- friendly experiments is becoming a global phenomenon. So efforts are taken in this study to minimize the consumption of chemicals and scaling down the size of the glass wares to reduce the atmospheric pollution which will help to maintain clean and eco-friendly atmosphere in the laboratory as well as in the college campus. The study was under taken especially for the gravimetric experiments using micro scale technique without any conceptual deviation of the principle of experiments in chemistry. It is found that students understood the practical better and developed a better skill of gravimetric analysis. The time required for the experiment was also short. Where quantities of substance were used in milligram scale instead of gram scale. This has reduced the expenditure on chemicals to a great extent. The apparatus used are of smaller size. The techniques save time, energy of a student while performing the experiments, also offers economy of fuel and also minimize the pollution hazards.

Conclusion: So it is our first attempt to change the attitude of the teachers to look at gravimetric experiments in Chemistry. Where quantities of substances were used milligram Scale. This has reduced the expenditure on chemicals to a great extent. The apparatus used is of smaller size. The scheme saves time, energy of a student while performing the experiments. The scheme also offers economy of fuel. It also minimizes the pollution hazards. The application of microanalysis in gravimetric experiments achieved a better adoption of the laboratory work to the varying needs of the students and made laboratory process more economical, and to form chemical reaction under safer and eco-friendly conditions.

We invited the feedback from the students. Everybody was positive on the application part. They also informed that they understood the practical better and developed a better skill of analysis. The time required for the experiment was also short.

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