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ABSTRACT

In the present article are analyzed some very common consumer products as paperclips, electrical wire fitting clips and disposable needles of injection syringe. This analysis is chosen because, these articles are not rusting easily or rapidly although their major constituent is iron. Gravimetric method is used for analysis of iron, whereas volumetric method is used for determination of chromium. The spectrophotometric method is tried for estimating nickel. This analysis is of interest as it is done for knowing whether these articles are made up of stainless steel or a similar material as that of razor blades, and whether and how much do they contain chromium and nickel along with iron. To design an experiment for iron content one can use paperclips or electrical fitting pins. For estimating iron and chromium a needle of injection syringe can be used. Being used-up or low-cost these may be useful as samples for analysis. The student may later think about the relationship between the properties and composition of the material also. This is an attempt for designing an experiment for analytical chemistry which may be necessarily incorporated in the practical curriculum for post graduate students of chemistry.

KEYWORDS: Chemistry Education, Low Cost Experiment, paperclips, electrical wire fitting clips, disposable needles of injection syringe.

1. INTRODUCTION

In the present age of Science and Technology, the iron metal and iron-containing alloys have been finding wider use in making number of items from them, ranging from pins to huge and heavy equipments. The paper clips, electrical wire fitting clips and disposable needles of injection syringe are used generally everywhere and in the offices, houses and hospitals. Their demand has grown by leaps and bounds. Today making of such consumer products happens to be one of the major industries of the world. Although iron is a hard workable metal, the rusting or corrosion of it is, however, a problem. This is solved either by electroplating the article or by using an alloy of iron for making the article. The role of chromium and nickel in alloys is to increase the resistance to corrosion. Nickel also finds its use in increasing elasticity and hardness of the material. The composition of material that makes the articles is useful in deciding its properties. Therefore, the analysis of it is very much important. The composition of the product of a specific manufacturer does not vary much from batch to batch.

In the present article are analyzed, some very common consumer products as paperclips, electrical wire fitting clips and disposable needles of injection syringe (CAUTION : To avoid the dangerous infectious diseases due to accidental needle sticks, proper precautions should be taken while handling the used-up injection-needles). This analysis is chosen because, these articles are not rusting easily or rapidly although their major constituent is iron. This analysis is of interest as it is done for knowing whether these articles are made up of stainless steel or a similar material, as that of razor blades, and whether and how much do they contain chromium and nickel along with iron.

The paper clips are generally nickel electroplated as is mentioned by the manufacturers. The clips for electrical fitting seem to be of similar type because they remain intact for long period without much affect on them. Some are reported to be tin-plated. It is mentioned in literature that needles of injection syringes are made up of stainless steel [1]. A similar consumer product is the razor blade too. It is the martensitic stainless steel, a ternary

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alloy containing iron, chromium and carbon (18 % Cr and 0 - 7% C) [2] which is useful for cutlery, cutting tools and razor blades. The presence of carbon permits heat treatment for producing a fine cutting i.e. wear-resistant and corrosion-resistant edge. A major advance in razor blade technology was the use of stainless steel [3, 4] instead of carbon steel. It is also thought, naturally, that whether the used-up blade (although considered as a waste) may be useful for any other purpose. It has been considered previously as a source of iron for determining iron by redox titrations like Fe (II)-Cr (VI) reaction using HgCl_2 - SnCl_2 , method [5] or TiCl_3 method [6]. There was also an investigatory project on corrosion and rusting of different brands of blades [7]. There is, however, no report on detailed study of chemical composition of paperclips, electrical wire fitting clips and disposable needles of injection syringe. In the present work the paperclips, electrical wire fitting clips and disposable needles of injection syringe available in the market have been analyzed for their quantitative chemical composition, especially for their important constituents - iron, chromium and to a less extent nickel. Gravimetric method is used for analysis of iron, whereas volumetric method is used for determination of chromium. The spectrophotometric method is tried for estimating nickel. This is, thus, an attempt for designing an experiment for Analytical Chemistry and this work will add some products in the list of those useful for experiments in Analytical Chemistry.

Preparation of sample, suitable for disintegration

The used-up paperclips, electrical wire fitting clips and disposable needles of injection syringe may have certain material deposited on it. The adhering material may be waxy material or, dust particles, etc. Before starting analysis the samples are first cleaned with water, followed by organic solvent such as alcohol or acetone [5, 6]. Then it is dried and weighed.

Disintegration of sample [5]

The weighed sample of paperclips, electrical wire fitting clips and disposable needles of injection syringe is disintegrated by heating it with hydrochloric acid (1:1) when all the constituents i.e. metals get converted to their chlorides which remain in soluble form. It is diluted on cooling to a known volume.

Separation of chromium, iron and nickel

An aliquot of the above-mentioned diluted solution is treated with sodium hydroxide (2 M) and hydrogen peroxide (6%) followed by heating. With this oxidizing agent, chromium (III) is converted to chromate i.e. chromium (VI), iron (II) is converted to iron (III) and alkali helps to precipitate the iron (III) hydroxide and nickel (II) hydroxide. The precipitated hydroxides are separated by filtration. The precipitate is washed with hot water till free from chloride. To the filtrate are added the washings. To this solution containing chromium (VI) is added sulphuric acid (conc.) till acidic and it is diluted to a known volume. In this, chromate on acidification gets converted to dichromate.

The precipitate containing hydroxides of iron and nickel is dissolved in hydrochloric acid (1:1). On treatment with ammonia (1:1) and ammonium chloride, iron is reprecipitated as $\text{Fe}(\text{OH})_3$ while nickel forms soluble $[\text{Ni}(\text{NH}_3)_6]^{2+}$. The precipitate is digested for half an hour, filtered, and washed with ammonium nitrate solution (1 %) till free from chloride. The filtrate and washing containing nickel as $[\text{Ni}(\text{NH}_3)_6]^{2+}$ are diluted together to a known volume.

Determination of chromium by volumetry (Redox titration)

Chromium is determined by back and blank titration. In back titration a known excess of Fe (II) solution is added to chromium (VI) solution and the excess Fe (II) reacts with standard potassium dichromate. In blank titration aliquot of Fe (II) solution same as that used for back titration is titrated against standard potassium dichromate solution. An internal indicator diphenylamine is used in Fe (II)-Cr (VI) titration and phosphoric acid is added before the titration. Phosphoric acid complexes with Fe (III) (which if present and when forms in solution) and avoids its conversion to Fe (II). By taking the difference in readings of blank and back titration and using following relation the chromium content can be calculated.

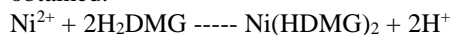
Equivalent for titrimetry: $\text{K}_2\text{Cr}_2\text{O}_7 / 6 = \text{Fe} / 1 = \text{FeSO}_4 / 1 = \text{Cr} / 3$

Determination of iron by gravimetry [8]

The precipitate of hydrated ferric oxide is ignited in a silica crucible till constant weight is obtained and then weighed as Fe₂O₃. From this the amount of iron is calculated using following relationship.
Equivalent of gravimetry: Fe₂O₃ / 2 = Fe / 1

Determination of nickel by spectrophotometry [8, 9]

When dimethylglyoxime is added to an ammoniacal solution containing nickel ions, a pink red coloration is obtained.



This reaction is made more sensitive using an oxidizing agent such as bromine water. The oxidation by hypobromite formed is complete in one-to-two minutes. The red complex formed here contains nickel in a higher valency state. This has been regarded as nickel (III)-dimethylglyoximate and also as nickel (IV) dimethylglyoximate. The complex formed which is insoluble in chloroform absorbs at 445 nm, provided absorbance readings are made within 10 minutes of mixing.

The "dimethylglyoxime oxidizing agent method" is different from the "nickel (II)-dimethylglyoxime" method. The later yields a nickel (II)-dimethylglyoximate, soluble in chloroform and this step is used initially to make nickel free from other transition metal ions. The nickel (II)-dimethylglyoximate goes in chloroform layer while other metal ions remain in aqueous phase. The chloroform layer may be decomposed by shaking with dilute hydrochloric acid. Nickel is transferred to aqueous phase, whereas dimethylglyoxime remains in the chloroform. Now the "Ni-DMG oxidizing agent method" is used. Prior to it, also, citrate is added to prevent interference of iron (if any). The color of Ni-DMG is developed again for the aqueous phase using bromine water and ammoniacal solution. The intensity of this color is measured.

The method of spectrophotometry requires the construction of a standard curve for the constituent being determined (here nickel (II)). Suitable quantities of the unknown or sample solution are taken and treated in the same way as those of standard solution for the development of color. The measurements of the optical density at the optimum wavelength are done. The optical density, is plotted against the concentration (or volume in mL), a straight line plot is obtained for standard in the range where Beer's law is obeyed. The curves or lines for standard and sample then may be used for the determination of constituent (here nickel (II)) in the sample.

2. MATERIALS AND METHODS

The setting of procedure is done by using a synthetic mixture prepared from stock standard solutions of iron, chromium and nickel. The number of sample pieces taken while doing actual analysis is varied as a single piece of consumer product is not adequate for the present complete analysis. For the analysis of paper-clips single clip was sufficient, but for other more sample-pieces are used together. The analysis of each type of product is carried out in duplicate. The work is presented as

- (a) Preparation of stock solutions of iron, chromium and nickel
- (b) Standardization of these metal stock solutions
- (c) Preparation of synthetic mixture
- (d) Analysis of this synthetic mixture
- (e) Analysis of paperclips, electrical wire fitting clips and disposable needles of injection syringe for iron, chromium and nickel.

The detailed procedure is given below and the results of analysis of synthetic mixture are given in Table 2. The results of some of the commonly used paperclips, electrical wire fitting clips and disposable needles of injection syringe used for the analysis are given in Table 3.

(a) Preparation of metal stock solutions

Iron (III) solution (10 mg/mL): 8.630 g of NH₄Fe (SO₄)₂.12H₂O is dissolved in ~ 50 mL water containing - 5 mL of H₂SO₄ (3 M) and the solution is diluted to 100 mL exactly.

[Ghoderao, *et al.*, 9(4): April, 2020]

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Chromium (III) solution (8 mg/mL): 4.099 g $\text{CrCl}_3 \cdot 6\text{H}_2\text{O}$ is dissolved in ~50 ml water containing a trace of concentrated HCl and the solution is diluted to 100 mL exactly.

Nickel (II) solution (3 mg/mL): 1.48625 g of $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ is dissolved in ~50 mL water containing ~ 1 mL of HNO_3 (1 M) and the solution is diluted to 100 mL exactly.

(b) Standardization of metal stock solutions**Standardization of Fe (III) solution**

Reagents: Sulphuric acid (3 M), Zinc dust, Phosphoric acid (85%) and Potassium dichromate (standard solution) (0.025 N), Diphenylamine indicator (1 %): This is 1% solution of diphenylamine in H_2SO_4 (conc.).

Procedure

An aliquot of 10 mL from stock solution of Fe (III) is taken in a 100 ml volumetric flask. It is diluted with water upto the mark. Then, 25 mL of this diluted solution is pipetted out in a conical flask. To it 10 mL of sulphuric acid (3 M) is added. It is heated to ~ 70°C on hot plate and - 0.5 g of zinc dust is added. The conical flask is covered with a watch glass. After 5 min, 5 mL of sulphuric acid (3 M) is added. The flask is swirled until all the zinc dust is consumed and evolution of hydrogen gas has stopped. To this solution 5 mL of sulphuric acid (3 M), 5 mL of phosphoric acid (85 %) and 3 drops of diphenylamine indicator (1 %) are added. The solution is titrated with standard $\text{K}_2\text{Cr}_2\text{O}_7$ solution (0.025 N) until persistent blue-violet color is obtained. The blank titration is carried out.

For the standardization, 25 ml of diluted iron solution gives the mean burette reading: 19.5 mL of $\text{K}_2\text{Cr}_2\text{O}_7$ (0.025 N)

1mL 1N $\text{K}_2\text{Cr}_2\text{O}_7$ = 55.85 mg Fe

19.5 mL 0.025 N $\text{K}_2\text{Cr}_2\text{O}_7$ = (55.85 x 0.025 x 19.5) mg Fe
= 27.2268 mg Fe

i.e. 25 mL aliquot of diluted solution contains 27.2268 mg Fe

Therefore, 100 mL diluted solution will contain 108.9075 mg of Fe

i.e. 10 mL of stock solution contains 108.9075 mg of Fe

i.e. 1 mL of stock solution contains 10.89075 mg of Fe

i.e. the prepared stock solution of Fe (III) is 10.89 mg/mL.

Standardization of chromium (III) solution

Reagents: H_2O_2 (6%), Ammonium ferrous sulphate (~ 0.025 N): 4.90175 g of $(\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ is dissolved and diluted to 500 mL with water.

Sodium hydroxide (2 M): 8 g of NaOH pellet dissolved and diluted to 100 mL.

Procedure

To an aliquot of 5 mL of stock solution of Cr (III) are added 15 mL NaOH (2 M) and 3 mL H_2O_2 (6%). Then, this mixture is boiled in water bath till oxygen is expelled. Then it is diluted to 250 mL exactly.

Back titration

An aliquot of 10 mL of this diluted solution is taken in a conical flask and made acidic with sulphuric acid (3 M). To this acidified solution, excess 5 mL of sulphuric acid (3M) and exactly 25 mL of ammonium ferrous sulphate (0.025 N) is added. It is titrated against standard $\text{K}_2\text{Cr}_2\text{O}_7$ solution (0.025 N), after adding 2-3 drops of diphenylamine indicator, till the color changes from faint pink to violet blue.

Blank titration

To an aliquot of 25 mL of ammonium ferrous sulphate (- 0.025 N) of same stock solution are added ~ 6 mL of H_2SO_4 (3 M) and 2-3 drops of diphenylamine indicator. It is titrated against standard $\text{K}_2\text{Cr}_2\text{O}_7$ solution (0.025 N) till color changes from light green to violet blue.

For the present study, it is seen that the mean titration readings for each of the titrations are as follows:

Blank titration: 25.0 mL of $\text{K}_2\text{Cr}_2\text{O}_7$ (0.025 N)

[Ghoderao, *et al.*, 9(4): April, 2020]
 ICTM Value: 3.00

Back titration: 21.3 mL of $K_2Cr_2O_7$ (0.025 N)
 (Blank-Back) reading =: 3.7 mL of $K_2Cr_2O_7$ (0.025 N)
 1mL 1 N $K_2Cr_2O_7$ = 17.33 mg Cr
 Therefore, 3.7 mL 0.025 N $K_2Cr_2O_7$ = $(3.7 \times 0.025 \times 17.33)$ mg of Cr
 = 1.60302 mg Cr

i.e. An aliquot of 10 mL diluted solution contains 1.60302 mg Cr
 Therefore, 250 mL diluted solution will contain 40.0756 mg Cr
 i.e. 5 mL Cr (III) stock solution (~ 8 mg/mL) contains 40.0756 mg Cr

The prepared stock solution is of Cr (III) is (8.01510 mg/mL).

Standardization of nickel (II) solution

Reagents: Standard zinc solution (0.01 M), EDTA solution (0.01 M), Ammonia-ammonium chloride buffer (pH = 10) and Eriochrome black-T indicator (1 %), Hydrochloric acid (9 M): 76.2 mL of hydrochloric acid is diluted to 100 mL.

Procedure

Standardization of EDTA solution

An aliquot of 10 mL of standard zinc ion solution (0.01 M) is taken in a conical flask with pipette and neutralized with dilute ammonia (1:1). To it is added 10 mL distilled water, 2 mL buffer (pH 10) and Eriochrome Black-T indicator. The solution is titrated versus EDTA (approx.0.01 M) till the end point as wine red to blue is obtained.

The standardization of Ni (II) solution is done by indirect method or complexometry.

Blank titration: It is carried out by taking 25 mL aliquot of EDTA (approx. 0.01 M) in a conical flask and titrating it against standard Zn (II) solution.

Back titration: An aliquot of 10 mL of the Ni (II) stock solution is diluted to 25 mL with hydrochloric acid (9 M). Then 5 mL of this diluted solution is taken in a conical flask to which exact 25 mL of EDTA solution (approx. 0.01 M) from same stock solution (as used for blank) is added. After neutralizing with 1:1 ammonia are added 5 mL ammonia-ammonium chloride buffer solution (pH = 10) and 5 drops of Eriochrome Black-T indicator. The contents of flask are titrated against standard zinc solution (0.01 M) till the end point blue to wine red color appears.

For the present study the observations are as follows:

Blank titration: 25.0 mL of Zn (II) solution (0.01 M)

Back titration: 14.7 mL of Zn (II) solution (0.01 M)

(Blank-Back) reading = 10.3 mL of Zn (II) solution.

The molarity of EDTA being exactly 0.01 M i.e. similar to that of Zn (II) solution, the same volumes of EDTA solution may be taken for calculating the concentration of Ni (II) solution.

1 mL 1M EDTA = 58.71 mg Ni

Therefore, 10.3 mL 0.01 M EDTA = $(10.3 \times 0.01 \times 58.71)$ mg Ni
 = 6.0471 mg Ni

i.e. An aliquot of 5 mL diluted solution contains 6.0471 mg Ni

Therefore, 25 mL diluted solution will contain 30.2356 mg Ni

i.e. 10 mL of Ni (II) stock solution contains 30.2356 mg Ni

The prepared stock solution of Ni (II) is (3.0235 mg/mL).

(c) Preparation of synthetic mixture

Assuming the general composition of stainless steel as Fe (85 %), Cr (14 %) and Ni (1 %) the synthetic mixture for analysis has been prepared by mixing known volumes of previously standardized stock solutions of iron, chromium and nickel. The mixture containing 17.0 mL stock iron (III) solution + 2.0 mL of stock chromium (III) solution + 1.0 mL of stock nickel (II) solution is diluted to 100 mL with water in a volumetric flask and used for further analysis.

The expected amounts of iron (III), chromium (III) and nickel (II) in it can be calculated as follows:

100 mL of mixture contains,

17.0 mL of stock iron (III) (10.8908 mg/mL) solution (185.1436 mg Fe)

2.0 mL of stock chromium (III) (8.0151 mg/mL) solution (16.0302 mg Cr)

1.0 mL of stock nickel (II) (3.0235 mg/mL) solution (3.0235 mg Ni)

Therefore, an aliquot of 1 mL of above synthetic mixture contains 1.8514 mg Fe (III), 0.1603 mg Cr (III) and 0.0302 mg Ni (II).

(d) Analysis of the synthetic mixture for Fe (III), Cr (III) and Ni (II)

Reagents: HCl (1:1), Ammonia (1:1), $K_2Cr_2O_7$ standard solution (0.025 N), Ferrous ammonium sulphate (~ 0.025 N), Sodium hydroxide (2 M), Sulphuric acid (2 M), H_2SO_4 (6 %), Diphenyl amine indicator (1 %), Ammonia (conc.), Chloroform

Ammonium nitrate (1 %): 1 g solid NH_4NO_3 dissolved in water to get 100 mL solution.

Silver nitrate (0.1 g): 0.1 g $AgNO_3$ dissolved in 100 mL of water.

Citric acid (10 %): 10 g citric acid dissolved in water to give 100 mL solution.

Dimethylglyoxime (1 %): 1g of dimethylglyoxime in 100 mL of absolute alcohol.

Ammonia (1:30): Mixed 1 volume of ammonia (conc.) with 30 volume of water.

Hydrochloric acid (0.5 M): 4.4 mL of concentrated HCl is diluted to 100 mL.

Bromine water: (saturated i.e. ~ 0.2 M): 1.1 mL (3.25 g) of liquid bromine is dissolved in ~ 100 mL water.

Standard Ni (II) solution for spectrophotometry (0.01 mg/mL): It is prepared from the stock solution of Ni (II) (0.030235 mg/mL). From this solution is taken an aliquot of 33.1 mL and diluted to 100 mL exactly which will be the solution of Ni (II) (0.01001 mg/mL).

Procedure

To an aliquot of 50 mL of the diluted synthetic mixture solution, 20 mL of NaOH (2 M) and 10 mL of H_2O_2 (6 %) are added. It is heated till all oxygen is evolved. Then solution is cooled and digested for half an hour. The precipitate of hydroxides of iron and nickel is filtered through filter paper. The precipitate is washed till free from alkali. The washings and filtrate are diluted to 250 ml capacity using a volumetric flask (Filtrate 1).

The precipitate is dissolved in minimum amount of hydrochloric acid (1:1). The resulting solution is heated to boil and treated with 4 g of ammonium chloride and ammonia (1:1) to precipitate iron (III) hydroxide completely. The precipitate is digested for half an hour. It is filtered through Whatman No. 41 filter paper. The precipitate is washed with hot ammonium nitrate (1 %) solution, till the washings are free from chloride ions (as detected by silver nitrate test). The filtrate and washings are collected in a 250 mL volumetric flask and diluted up to the mark with water (Filtrate 2). While the filtration is in progress, a silica crucible with lid, is heated on burner to red heat, cooled in a desiccator and weighed (W_1). After drying the precipitate the filter paper with precipitate is folded and transferred to the weighed silica crucible. It is heated gradually, and the paper is charred. Finally, the residue is heated at a red heat till constant weight of residue. Then crucible is cooled after closing with lid in desiccator for 15 minutes and weighed as Fe_2O_3 (W_2). The difference ($W_2 - W_1$) gives weight of Fe (III) oxide residue, using which the amount of iron in the mixture can be calculated.

The filtrate 1 is used in back titration for chromium determination. The procedure of back and blank titration is same as in the case of standardization of Cr (III) stock solution.

The filtrate 2 is used for nickel determination by spectrophotometry. The detailed procedure for this experiment is as follows:

To 0.5, 1.0, 1.5, 2.0, 2.5, 3.0, 3.5, 4.0 mL each of the standard nickel (II) solution (0.01 mg/mL), 5 mL of citric acid (10 %) is added. The solution is neutralized with ammonia (conc.) and few drops are added in excess (pH ~7.5). To this slightly alkaline solution 2 mL of dimethylglyoxime (1 %) solution is added. It is extracted with three 3 mL portions of chloroform, shaking for 30 seconds at each time. The combined chloroform extract is shaken with 6 mL of ammonia (1: 30) and chloroform extract is taken out. The ammoniacal phase is again shaken with 2 mL chloroform and the chloroform phase is added to the main chloroform extract.

The nickel is returned to the ionic state by shaking chloroform extract vigorously for 1 minute with two 5 mL portions of hydrochloric acid (0.5 M). The hydrochloric acid solution is transferred to a 25 mL-volumetric flask. To it 5 mL water, 1 mL bromine water (saturated) and 2 mL of ammonia (conc.) are added. It is cooled (below

30°C) and to it 1 mL of dimethylglyoxime (1%) solution is added. The contents are diluted up to the mark with water. The optical densities are measured at 445 nm after 5 minutes.

Similarly, taking a series of aliquots from the filtrate 2, the colored solutions are prepared, following the same procedure described above, and optical densities are measured for each solution at 445 nm. A graph of volume in mL vs. optical density is plotted for standard solution and solution from synthetic mixture. The concentration of nickel in synthetic mixture is determined by using the values of slopes of lines for standard and sample as well as the value of concentration of standard and the following relation.

$$C_{\text{synthetic}} / C_{\text{standard}} = \text{Slope}_{\text{synthetic}} / \text{Slope}_{\text{standard}}$$

(e) Analysis of used-up paperclips, electrical wire fitting clips and disposable needles of injection syringe for iron, chromium and nickel

Preparation of sample suitable for analysis

The used-up paperclips, electrical wire fitting clips and disposable needles of injection syringe are thoroughly cleaned with water followed by organic solvent such as alcohol, acetone. It is dried in air and weighed exactly.

Disintegration of sample

The disintegration is carried out using 25 mL of hydrochloric acid (conc.), 5 mL of distilled water and heating the contents in a 250 mL conical flask covered with a stem-cut funnel. After disintegration, the solution is cooled and mixed with 50 mL of distilled water. The stem-cut funnel is rinsed and these washings are mixed with the solution. It is then warmed and filtered through a filter paper. The filter paper is washed with distilled water till it is free from acid. The filtrate and washings are collected together and diluted exactly to 250 mL. An aliquot of 50 mL of this solution is used for determination iron, chromium and nickel by using the procedure used for synthetic mixture.

3. RESULTS AND DISCUSSION

Determination of constituents of the synthetic mixture

Determination of Fe(III) (by gravimetry)

Weight of (crucible + lid), $W_1 = 31.838$ g

Weight of (crucible + lid + residue) after complete ignition, $W_2 = 31.970$ g

Weight of residue, $\text{Fe}_2\text{O}_3 = (W_2 - W_1)$ g of Fe_2O_3

$$= 0.132 \text{ g of } \text{Fe}_2\text{O}_3 = 132.00 \text{ mg of } \text{Fe}_2\text{O}_3$$

$\text{Fe}_2\text{O}_3 = 2 \text{ Fe}$

$$159.694 = 111.694$$

$$132.0 \text{ mg residue} = 92.324 \text{ mg Fe}$$

i.e. 50 mL aliquot of stock solution of the synthetic mixture contains 92.324 mg of Fe

Therefore, 100 mL of the synthetic mixture contains 184.648 mg of Fe

Determination of Cr(III) (by indirect method of redox titration)

Titration readings:

Blank titration: 25.0 mL of $\text{K}_2\text{Cr}_2\text{O}_7$ (0.025 N)

Back titration: 24.2 mL of $\text{K}_2\text{Cr}_2\text{O}_7$ (0.025 N)

(Blank-Back) readings = 0.8 mL of $\text{K}_2\text{Cr}_2\text{O}_7$ (0.025 N)

1 mL 1 N $\text{K}_2\text{Cr}_2\text{O}_7 = 17.33$ mg Cr

Therefore, 0.8 mL 0.025 N $\text{K}_2\text{Cr}_2\text{O}_7 = (0.8 \times 0.025 \times 17.33)$ mg Cr = 0.3466 mg Cr

i.e. An aliquot of 10 mL of filtrate 1 contains 0.3466 mg Cr

Therefore, 250 mL of filtrate 1 = 8.665 mg Cr

i.e. 50 mL of stock solution of the synthetic mixture contains 8.665 mg Cr

Therefore, 100 mL of the synthetic mixture contains 17.330 mg Cr

Determination of Ni(II) (by spectrophotometry)

From the observations are plotted the curves for standard solution and the solution obtained from synthetic mixture (Table 1).

Table 1: Optical densities of standard Ni (II) and synthetic mixture solution

Volume mL	OD for standard Ni (II) (0.01mg/mL) solution	OD for synthetic mixture solution
0.5	0.14	0.08
1.0	0.32	0.16
1.5	0.38	0.26
2.0	0.50	0.36
2.5	0.66	0.4
3.0	0.72	0.48
3.5	0.86	0.58
4.0	1.0	0.66

The slopes for standard solution (concentration as 0.01 mg/mL) and solution from synthetic mixture are 0.2686 and 0.1696 respectively.

Now,

$$\begin{aligned} C_{\text{synthetic}} / C_{\text{standard}} &= \text{Slope}_{\text{synthetic}} / \text{Slope}_{\text{standard}} \\ C_{\text{synthetic}} &= 0.1696 \times 0.01 / 0.2686 \\ &= 0.006314 \text{ mg/mL of Ni} \end{aligned}$$

The concentration of filtrate 2 is 0.006314 mg/mL of Ni

Total nickel in 250 mL filtrate 2 will be 1.5785 mg

i.e. 50 mL aliquot of diluted synthetic mixture (from which filtrate is obtained) contains 1.5785 mg Ni.

Therefore, 100 mL synthetic mixture contains 3.157 mg Ni

The values of iron, chromium and nickel in synthetic mixture are summarized in Table 2.

Table 2: Expected and observed values of iron, chromium and nickel from a synthetic mixture

Metal	Expected (mg)	Observed (mg)
Iron	185.1436	184.6480
Chromium	16.0302	17.3300
Nickel	3.0235	3.1570

Determination of constituents of the paperclips, electrical wire fitting clips and disposable needles of injection syringe

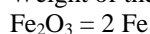
For the each type of sample analyzed in the present work, details of analysis of a representative sample are as follows and the observations are given in Table 3.

Paperclip sample

Weight of the paperclip: 0.34534 g

Determination of iron

Weight of the residue: 0.0910 g



$$159.694 = 111.694$$

Therefore, 0.0910g residue = 0.06364 g of Fe

An aliquot of 50 mL solution contains 0.06364 g of Fe

Therefore, 250 mL diluted solution will contain 0.3182 g of Fe

i.e. 0.34534 g of paperclip material contains 0.3182 g of Fe

Therefore, 100 g of paperclip material will contain 92.15 g of Fe.

Percentage of Fe is 92.15

Determination of chromium

The blank and back titration readings are same indicates that amount of chromium in paperclips is nil.



Determination of nickel

It was tried to determine nickel in paperclip by spectrophotometry. But, as the nickel content is too low, only a pink color is developed with DMG which has no measurable optical density at 445 nm. After concentrating the solution to half the volume also, the optical density is not measurable.

Electrical wire fitting clips sample

Weight of the electrical wire fitting clips: 0.34200 g

Determination of iron

Weight of the residue: 0.0959 g

$\text{Fe}_2\text{O}_3 = 2 \text{ Fe}$

$159.694 = 111.694$

Therefore, 0.0959g residue = 0.06707 g of Fe

An aliquot of 50 mL solution contains 0.06707 g of Fe

Therefore, 250 mL diluted solution will contain 0.3353 g of Fe

i.e. 0.34200 g of electrical wire fitting clips material contains 0.3353 g of Fe

Therefore, 100 g of electrical wire fitting clips material will contain 98.06 g of Fe.

Percentage of Fe is 98.06

Determination of chromium

The blank and back titration readings are same indicates that amount of chromium in electrical wire fitting clips is nil.

Determination of nickel

It was tried to determine nickel in electrical wire fitting clips by spectrophotometry. But, as the nickel content is nil no pink color is developed with DMG.

Disposable needles of injection syringe sample

Weight of the Disposable needles of injection syringe: 0.32800 g

Determination of iron

Weight of the residue: 0.0895 g

$\text{Fe}_2\text{O}_3 = 2 \text{ Fe}$

$159.694 = 111.694$

Therefore, 0.0895g residue = 0.06259 g of Fe

An aliquot of 50 mL solution contains 0.06259 g of Fe

Therefore, 250 mL diluted solution will contain 0.3129 g of Fe

i.e. 0.32800 g of disposable needles of injection syringe material contains 0.3129 g of Fe

Therefore, 100 g of disposable needles of injection syringe material contain 95.42 g of Fe.

Percentage of Fe is 95.42

Determination of chromium

The titration readings are as follows:

Blank titration: 25.0 mL of $\text{K}_2\text{Cr}_2\text{O}_7$ (0.025 N)

Back titration: 24.6 mL of $\text{K}_2\text{Cr}_2\text{O}_7$ (0.025 N)

(Blank-back) readings = 0.4 mL of $\text{K}_2\text{Cr}_2\text{O}_7$ (0.025 N)

1 mL 1 N $\text{K}_2\text{Cr}_2\text{O}_7 = 17.33 \text{ mg of Cr}$

Therefore, 0.4 mL 0.025 N mL of $\text{K}_2\text{Cr}_2\text{O}_7 = (0.4 \times 0.025 \times 17.33) \text{ mg Cr}$

$= 0.1733 \text{ mg Cr i.e. } 0.0001733 \text{ g Cr}$

An aliquot of 25 mL contains 0.0001733 g of Cr

Therefore, 250 mL diluted solution will contain 0.001733 g of Cr

i.e. An aliquot of 50 mL of stock solution contains 0.001733 g of Cr

Therefore, 250 mL stock solution from sample will contain 0.008665 g of Cr

i.e. 0.32800 g of disposable needles of injection syringe material contains 0.008665 g of Cr

Therefore, 100 g of disposable needles of injection syringe material = 2.64 g of Cr
 Percentage of chromium in disposable needles of injection syringe is 2.64

Determination of nickel

It was tried to determine nickel in disposable needles of injection syringe by spectrophotometry. But, as the nickel content is nil no pink color is developed with DMG.

Table 3 Analysis of iron, chromium and nickel** in some consumer products supposed to have constitution like 'stainless steel'*

Code number and other information	Weight of Sample, g diluted to 250 mL (number of sample pieces used)	Iron		Chromium		Nickel
		Weight of Fe ₂ O ₃ g from 250 mL aliquot	Calculated % Fe	Titer (Blank - back) mL K ₂ Cr ₂ O ₇ (0.025 N) for 25 mL aliquot	Calculated % Cr	
Paper clips Nickel electroplated Non-tear ends, triangular	0.34534 (one)	0.0910	92.15	Nil	Nil	Positive qualitative test
Clips for electrical fitting (Tin joint clips), (size 25 mm)	0.34200 (Three)	0.0959	98.06	Nil	Nil	Nil
Needles of injection syringe (Gamma sterilized)	0.32800 (Eight)	0.0895	95.42	0.4	2.64	Nil
*Chromium is present only in needles of injection syringe.						
**Paper clips contain trace amount of nickel but that amount of nickel cannot be determined by spectrophotometry. Electrical fitting clips and needles of injection syringe do not contain traces of nickel.						

On the basis of study carried out on these three consumer products following results can be drawn.

1. The needle of injection syringe and electrical fitting clip do not contain nickel as seen by negative qualitative test.
2. The needle of injection syringe may be of stainless steel of martensitic type.
3. Plastic covered paper clips are ductile in nature. The study may be carried out for them. The present work is related to ordinary pins only. (The plastic clips may contain aluminium as one of the constituents.)
4. To design an experiment for iron content one can use paperclips or electrical fitting pins. For estimating iron and chromium a needle of injection syringe can be used. Being used-up or low-cost these may be useful like razor-blade as samples for analysis.
5. If the articles are electroplated with tin (such as clip for electrical wiring) one may have to take precaution to avoid interference of tin in determination of other constituents.

4. CONCLUSION

The use of consumer products in the teaching of analytical chemistry has been incorporated into quantitative analysis and instrumental methods of analysis everywhere to achieve the following objectives [10]

- (1) to illustrate best the basic principles and techniques associated with analytical chemistry
- (2) to increase the students' awareness of the importance of chemistry of consumer products, and
- (3) to help the students learn and use "critical thinking" in solving real-world situations. The critical thinking includes the modification of method, to compute the results and to device new procedures. This not only stimulates the student's interest but bridges also the gap between academic life and real life.

In the present work, it is tried to utilize used-up consumer products for analysis. This helps in knowing the chemistry of these products, and also makes one to think of reuse of the used-up materials for recovery of chemicals or as substitute for them. The percentage of iron, chromium and nickel in used up consumer products may make one to think before throwing these products in garbage. An attempt to use these products for estimation of iron, chromium and nickel may tempt one to find out similar such consumer products for analysis. This will then be the success of the type of work presented here.

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REFERENCES

- [1] Siemer, D.D., Reeder, S.D. and Wade M.A., *J.Chem.Educ.*, 61, 467 (1988)
- [2] Long, G.L. and Leighly Jr. H.P., *J. Chem. Educ.*, 59, 953, (1982)
- [3] 'Encyclopedia Britannica', (Raynal – Sarraut), Vol. 19, 1768 (1974), p. 3
- [4] Sharma, V., *Science Today*, 7(11), 13–18, May (1973)
- [5] Fritz, J.S. and Schenk, G.H., 'Quantitative Analytical Chemistry', 4th ed., Allyn and Bacon Inc., (1979)
- [6] Klett, L.D., Black, A.H. and Parker, G.A., *J. Chem. Educ.*, 61, 1020 (1984)
- [7] Chakravarty, A.K., 'Investigatory Projects in Chemistry', Tata-McGraw Hill Publ. Co., New Delhi (1983), 44
- [8] Vogel, A.I., 'A Textbook of Quantitative Inorganic Analysis', ELBS, 3rd ed., (1973), p. 468, 472, 747, 794, 901
- [9] Snell, F.D. and Biffen, F.M., 'Commercial Methods of Analysis', D.B.Taraporvala Sons and Co., Private Ltd., Bombay, Revised ed., (1964) 99
- [10] Lieu, V.T., and Kalbus, G.E., *J. Chem Educ.*, 65, 207 (1988)