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Research Paper

# Iodisation of Lake Katwe Salt, Kasese District

Chemistry

KEYWORDS : Rocksalt, iodization, iodate, iodine retention, lake katwe, iodized

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## ABSTRACT

*The raw rock salt was analysed for its chemical composition in terms of selected chemical species. It exhibited a large quantity of chemical impurities with sulphate at 20.7% w/w, carbonate 31.7 %, magnesium 945 ppm, iron 168 ppm, calcium 74 ppm and zinc at 18 ppm with chloride anion at 1.54% w/w. Titrimetry was used for determination of chloride and carbonates, sulphate was determined gravimetrically. ASS was used for determination of cations. Several methods were used for purification of the raw salt namely; fractional crystallization, precipitation with barium chloride, sodium state, potassium permanganate and ammonium sulphide. The purified salt had the following composition; carbonate 1.42% w/w, sulphate 3.83% chloride 50% w/w, iron 7.4±0.25, magnesium 7.64±0.20, calcium 4.40±0.30 and zinc 4.22±0.15. The refined salt was iodised at 100ppm by addition of (0.0356g) potassium iodate to 200 g salt. The level of the added iodine was determined by iodometric titration. It was found constant for two weeks (the experimental period).*

## INTRODUCTION

A program of pitting and drilling of the salt crust at Lake Katwe bed outlined reserves of approximately 22 million metric tons of mixed salt (Project UGA/89/001). The lake salt largely comprises of the following minerals: halite (NaCl), hanksite ($9Na_2SO_4.2Na_2CO_3.KCl$), burkeite ($Na_2CO_3.2Na_2SO_4$), and trona ($Na_2CO_3.\ NaHCO_3.2H_2O$). Salt has been extracted from the lake since time immemorial however, todate the salt production methods at the lake are primitive and labour intensive with little control over the quantity and quality of products.

Salt is essential for life. It is the principal constituent of extra cellular body fluids i.e. fluids in tissues, blood serum, and saliva. At the beginning of life, the human foetus floats in a saline solution and the amount of salt increases to reach approximately 230 grams in the body of an adult human (Hughes., 2003). The human blood is about 0.9% salt and 0.25% by weight of our body total weight (Abraham *et al*., 2002).

Iodine is an essential micronutrient for human and animal health and it is often inadequate in our diet. In the body it helps in the production of thyroid hormones which is essential for development and metabolism of many human tissues. Other functions of iodine includes: helping to regulate moods, preventing cancer among others (Micheal 2005). It was also demonstrated that iodine tends to be antibacterial, antiviral, antiparasitic and antifungal and that it enhances immune system. In was also shown that iodine helps in the excretion of toxic elements including lead, mercury, cadmium, bromide and fluoride from the body (Jonathan *et al.,* 2005). The lack of iodine in our daily diet results to what is called Iodine Deficiency Disorders (IDD) which includes; irreversible mental retardation, goiter, reproduction failures, increased child mortality, reduced attention span and socio economic compromise (Hetzel *et al*., 1989).

The inadequate iodine content of food supply is supplemented by the addition of iodine to salt worldwide (World Bank Paper, 1993). This is a result of universal salt iodization (USI). For over seventy years, in an effort to introduce iodine regularly into daily diet, several foods have been considered as possible vehicles. Among these, salt has become the most commonly accepted due to its availability and long shelf life (Bagepalli *et al*., 1993). It is a program aimed at elimination of Iodine Deficiency Disorders in the world by the year 2000 (WHO 1994).

UNICEF Uganda supported the Universal Salt Iodisation Program (USI) from 1994 through the production of iodized salt at Lake Katwe which officially started on May 2002. In that process a concern aroused because the iodized salt presented a special problem in that the added iodine was not detected in the salt there after. It was assumed that the iodine is ***"lost"*** on impact with the salt (Lorenzo 2003). It was then decided to have an external review to investigate what the problems were.

Iodine readily sublimes at ambient temperature. Consequently the effectiveness of salt iodisation programme depends on the stability of iodine carrier in the salt typically potassium iodate (Diosady *et al*., 1995). Studies have shown that stability of iodine in iodised salt also depends on packaging or exposure of packaged salt to prevailing climatic conditions. This is because moisture plays a critical role in the stability of iodine in salt. It was also shown that the control of moisture content in iodised salt throughout the manufacture and distribution, by improved processing, better packaging and storage is critical to the stability of added iodine. Zbigniew (1989) established that the following inorganic salts are strong reducing agents commonly found in common salts: ammonium sulphite hexahydrate, iron(II) sulfate heptahydrate, tin(II)chloride dihydrate, sodium disulphide, and sodium sulfite. It has also been noted that the presence of common anions like sulphite and thiosulphate, and sulphurous acid strongly reduce iodate to molecular iodine which readily sublimes.

## MATERIALS AND METHODS

Samples of rock salt slabs (20.0 kg) were picked from the lake bed at three different positions 50 meters apart. About equal quantities of the sample from different positions were taken and mixed together, sun dried, put in polyethylene bags and transported to the Department of Chemistry, Makerere University, Kampala for laboratory work. Distilled water was used for making all solutions.

The chemical composition of the raw rock salt was determined in terms of the chemical species which are known to affect its iodine retention i.e sulphate, chloride, and carbonates as anions. And Ca, Fe, Zn and Mg were determined as cations.

Sulphate was determined by dissolving the rock salt sample (20.0 g) in distilled water (400 $cm^3$) acidified with nitric acid (10 $cm^3$) accompanied with a little heating. The resulting solution was filtered and the residue washed with the acidified distilled water. The filtrate was transferred in to a volumetric flask (1000 $cm^3$) and made to the mark with distilled water. Sulphate was determined gravimetrically by precipitating with barium chloride as described by (Wiely 1979).

The rock salt solution (20 gpl) was made and carbonate level determined by titration with hydrochloric acid (0.1 M) to the methyl orange end point. Chloride was determined by titrating fractions of the salt solution with a standard silver nitrate solution to the potassium chromate endpoint.

Atomic absorption spectroscopy was employed for determination of Mg, Ca, Zn and Fe since it is highly specific because the atomic absorption lines are narrow (0.002 to 0.005 mm) and electron transition energies are unique for each element (Gurdeep *et al.*, 2002).

To the rock salt sample (5.0 g) was added concentrated nitric acid (5.0 $cm^3$). The resulting solution was transferred in to a volumetric flask (200 $cm^3$) and made to the mark. A portion of this solution (100 $cm^3$) was used for AAS analysis. Standard solutions of Ca, Mg, Fe and Zn were each made to 200 $cm^3$: Set one: 0.2 ppm solution, Set two: 0.5 ppm solutions, Set three: 1.0 ppm solutions, Set four: 4.0 ppm solutions, Set five: 5.0 ppm solutions. During the analysis, calibration curve was prepared by adjusting the read out device to 100% transmittance with a blank (distilled water) and 0% transmittance with no radiant energy. A series of standard samples of the same element (at concentrations of 0.2, 0.5, 1.0, 2.0 and 5.0 ppm) to be determined were aspirated into the burner and percentage absorption for each concentration measured. The absorbance values were plotted against concentration. Adjustments were made until a linear calibration curve was obtained for each element under investigation. The rock sample solution was atomised and the absorbance measured under exactly the same conditions as those used when the calibration curve was prepared. The above steps were followed for each of the elements Ca, Mg, Zn, and Fe.

**Purification of the Rock Salt**

The crude rock salt (approximately 10 kg) was dissolved in distilled water (20 litres). The insoluble solids were removed by filtration, sun dried and weighed. The resulting dirty mixture was made to stand overnight and decanted.

Fractional crystallization was employed on the rock salt solution (stock prepared from previous section) by heating to boiling for an hour. The concentrated solution was made to cool and allowed to stand for two days. Brown solid sediment $S_1$ deposited at the bottom of the solution. It was decanted and filtered. A sample of the sediment (residue) was dried in an oven at 120ºC and analysed for carbonate, sulphate and chlorides using methods previously mentioned. This procedure was repeated until when clear crystalline solids formed at the bottom of the container. The solution (from which clear crystals were formed) was heated almost to dryness and made to cool slowly to form crystals. The recovered crystals (500 g) was rinsed with distilled water (200 $cm^3$) and dissolved in distilled water to make (3000 $cm^3$) solution. The solution was kept for further treatment.

A solution of sodium stearate (150 gpl) was made and added to the stock brine (from the previous procedure). The mixture was stirred strongly for 10 minutes; a thick dirty grey solid formed, made to stand for two hours and filtered. To the filtrate (the brine), extra sodium stearate solution (100 $cm^3$) was added and the mixture stirred vigorously for more 10 minutes. Lather formed on top and collapsed immediately. The mixture was filtered off and the filtrate reserved as a stock for further treatments.

Potassium permanganate solution (50 gpl) was made. The brine (20.0 $cm^3$) obtained from the procedure above was pipetted into a conical flask and titrated with the potassium permanganate solution to the end point (when the purple colour of potassium permanganate persisted).The volume of potassium permanganate used was recorded. This titre volume was used to estimate the amount of potassium permanganate required to oxidation in the salt solution portion. To the brine potassium permanganate (35.0 $cm^3$) plus starch solution (100 $cm^3$) were added and the mixture stirred strongly. The pH of the solution was adjusted to 10.0 by addition of sodium hydroxide solution (2.0 M, 100 $cm^3$). A heavy brown precipitate formed and settled at the bottom of the container. It was decanted and filtered and the filtrate preserved for subsequent steps.

Pyrite (5.0 g) was put in a round bottomed flask with a side tube and a saturated ammonia solution (100 $cm^3$) was transferred in to a flask with a side tube. The apparatus were fitted with a rubber tube having one open end inside the ammonia solution and the other connected to the side tube of the round bottomed flask. To the pyrite, a dilute hydrochloric acid was added through a thistle funnel. The gas produced in the flask was bubbled through the ammonium hydroxide continuously for one hour. The resulting solution was ammonium sulphide. The ammonium sulphide solution was added to the brine step wise (in portion of 5.0 $cm^3$) while stirring. This was done until no dark precipitates formed on further addition of ammonium sulphide. To the resulting mixture, starch solution (100 $cm^3$) was added to induce flocculation. The mixture was made to stand for an hour and filtered off. The filtrate was preserved as the stock brine for more treatment.

To the stock solution (brine), ordinary charcoal (2 kg) was added and mixed by stirring for 5 minutes. It was made contact for three days. The mixture was filtered and the filtrate (brine) was kept for final treatments. To the brine, a saturated solution of barium chloride was added drop wise with a little heating until there was no further precipitation on additional drops. It was made to cool under natural convection and the milky suspension was filtered. To the filtrate, hydrochloric acid (2.0 M, 80 $cm^3$) was added drop wise while stirring until when the pH of the resulting solution was 7.0. The final solution was strongly heated up to a point when few crystals were observed at the bottom. It was cooled and made to stand at ordinary room temperature for one day. The crystals formed were removed, rinsed with distilled water and sun dried. The refined dry salt was packed in a polyethylene bag ready for iodisation. The chemical compositions for the relevant chemical species were determined using the methods earlier mentioned.

**Iodine Retention of the Refined Salt**

A fraction (250 g) of the refined rock salt was iodized by drying in an oven at 60°C for four hours. The dried salt crystal (200 g) was crushed to powder. To the powder, potassium iodate (0.0356 g) was added and thoroughly mixed. The resulting salt contained 100 ppm of iodine. The sample was packed in a polyethylene bag and kept in a dry place. The iodine content of iodated salt sample was analyzed using iodometric titration.

**RESULTS AND DISCUSSIONS**

**Chemical Composition of the Raw and Refined Rock Salt**

From the table 1, it can be noticed that the amount of carbonate anion is the highest in the raw salt presenting 31.7% by mass. Sulphate anion follows as the second highest constituent at 20.7% by mass of the salt. However the amount of total chloride is very low contributing to only 1.54 % by mass of the rock salt. From the metallic species analysed, magnesium is highest occurring at with 945 ppm followed by total iron at 168 ppm with calcium and zinc at 74 ppm and 18 ppm respectively. Figures for the data in Table 2, indicate a big reduction in the amounts of chemical impurities as follows; carbonate anion was reduced from the 31.7% in the raw rock salt to 1.42% w/w in refined sample, sulphate anion was reduced from 20.7% to 3.83% w/w. However the purification processes increased the ratio of chloride anions from 1.54% w/w to 50% w/w of the refined salt giving 85% w/w of sodium chloride.

**Table 1 The chemical composition of raw rock salt**

| Anion | % w/w | Cation | ppm |
|---|---|---|---|
| $CO_3^{2-}$ | 31.7 | Mg | 945±3 |
| $SO_4^{2-}$ | 20.7 | Fe | 168±2 |
| $Cl^-$ | 1.54 | Ca | 74±3 |
| | | Zn | 18±4 |

Carbonates are sometimes used as additives in salt fortified with potassium iodate to improve on its iodine retention. They work as stabilizers for iodate by buffering the reducing effects of chemical impurities. It is therefore not responsible for iodine loss in the raw rock salt. Sulphur compounds especially the sulphides, and thiosulphates are reducing agents and they reduce iodate added to salt through complex chemical reactions to free molecular iodine.

Chlorides of magnesium, calcium, zinc and iron are known to be hygroscopic in nature since they absorb water from the atmosphere. As impurities in salt, they contribute to the hygroscopic nature of salt this is a major factor for its iodine loss since the absorbed water gives a medium for reactions that leads to the reduction of potassium iodate to iodine and its subsequent loss through sublimation. Iron (II) is oxidised to iron (III) depending on the chemical species in the medium; a reverse reaction, i.e. reduction can also occur. In the present of iodate, $Fe^{2+}$ is oxidised to $Fe^{3+}$ to liberate molecular iodine (Diosady *et al.*, 1997) according to the ionic equation

$$2I^{5+} + 10Fe^{2+} \rightarrow I_2 + 10Fe^{3+}$$

To obtain salt with low hygroscopic behaviour, it is important that magnesium, calcium and iron have to be removed from the s alt to a large extent. This limits the possibility of most reactions that would require water as a medium and as a source of hydrogen ions and electrons.

**Table 2 The chemical composition of the refined rock salt in terms of selected chemical species**

| Anion | % w/w | Cation | ppm |
|---|---|---|---|
| $CO_3^{2-}$ | 1.42 | Mg | 7.64±0.20 |
| $SO_4^{2-}$ | 3.83 | Fe | 7.40±0.25 |
| $Cl^-$ | 50 | Ca | 4.40±0.30 |
| | | Zn | 4.22±0.15 |

From the chemical composition of refined rock salt shown in Table 2, it can be compared to the international requirement for iodised salt (Table 3). Looking at the concentration of chemical species relevant to this research, namely calcium, magnesium and sulphates, it can be noted that, magnesium and calcium concentrations in the refined rock salt sample are below the maximum required by standards. This clearly shows that the purification methods employed are suitable for the removal of magnesium and calcium. However, sulphate level at 3.83% w/w does not conform to the standard requirement with a maximum value of 1.90% w/w. The reduced level of magnesium and calcium is positively manifested in the salt having low hygroscopic character and the low sulphate concentration depicts a reduction in the reducing property of the refined rock salt.

**Purification**

The first purification step involved fractional crystallisation. Fractional crystallisation is an equilibrium stage separation process. Is a series of repeated crystallisations and its suitability depends on differences in solubility product ($k_{sp}$) values of the salt mixture. The concentration of a given species in solution is also important because species, whose products of concentrations are above their $k_{sp}$ values, solidifies out to maintain this value.

**Table 3 The composition of crystallised fractions of the salt from fractional crystallisation process**

| Crystal fractions | Mass $CO_3$ in 20g sample(g) | % w/w of $CO_3$ | Mass $SO_4$ in 20 g sample(g) | % w/w of $SO_4$ | Mass Cl in 20 g sample (x $10^{-1}$) g | % w/w of Cl |
|---|---|---|---|---|---|---|
| S1 | 5.45±0.65 | 27.22 | 1.76±0.22 | 8.8 | 0.965±0.091 | 0.48 |
| S2 | 7.95±0.51 | 39.75 | 1.83±0.17 | 9.2 | 1.289±0.098 | 0.643 |
| S3 | 6.23±0.56 | 31.1 | 2.11±0.19 | 10.6 | 4.18±0.160 | 2.09 |
| S4 | 4.58±0.35 | 22.8 | 2.15±0.20 | 10.76 | 11.58±2.50 | 5.78 |
| S5 | 4.43±0.44 | 22 | 2.65±0.99 | 13.25 | 17.36±1.98 | 8.6 |

S1: the 1st fraction of crystals removed in the separation process
S2: the 2nd " " " " " " " "
S3: the 3rd " " " " " " " " "
S4: the 4th " " " " " " " " "
S5: the 5th " " " " " " " " "

For purification by crystallisation, the substance has to be dissolved in a suitable solvent in our case water. The choice of the solvent is very important. The solvent should not react with the compound chemically. The solubility of the components to be separated should differ at different temperature so that the right choice of temperature is made for separation. Meaning that, the impurities should be insoluble either in cold or hot solvent so that these can be removed by filtration before crystallisation. Alternatively the solubility of the impurities should be high at room temperature so as to remain in the leftover liquid (called mother liquor) during crystallization.

**Table 4 International standard for table salt**

| Composition | Formula/Unit | Values |
|---|---|---|
| Sodium chloride | NaCl, %min | 97.00 |
| Calcium | Ca, % max | 0.40 |
| Magnesium | Mg, %max | 0.40 |
| Sulphates | $SO_4^{2-}$%max | 1.90 |
| Insoluble matters | % max | 0.20 |
| Moisture | % max | 6.00 |
| Source: UNBS | | |

The chemical composition of the fractions of salt crystallised at the different stages of crystallisation is in Table 3. The level of carbonate shot up from 27.2% w/w in the first fraction $S_1$ to 39.7% w/w in second fraction $S_2$ and reduced to about 22% w/w in the 5th fraction $S_5$. When carbonate level reached 22.8% in the fourth fraction $S_4$, both chlorides and sulphates solidified out at increasing quantity. The level of carbonate shot up from 27.2% w/w in the first fraction $S_1$ to 39.7% w/w in second fraction $S_2$ and reduced to about 22% w/w in the 5th fraction $S_5$. When carbonate level reached 22.8% in the fourth fraction $S_4$, both chlorides and sulphates precipitated at increasing quantity. At this point therefore, further crystallisation removes chloride anion at increasing rate. Chloride and sulphate are co-crystallised at increasing quantity. Further recrystallisation could not separate chloride and sulphate. Therefore this is a point to change the purification method to avoid the loss of chlorides. It can be concluded that a complete separation of these three salts (carbonate, chloride and sulphate) is not possible by the application of fractional crystallisation alone.

Ions can be separated from each other based on solubility of their salts. When solubility of two or more ions is different, they can be separated by precipitation. Separation of ions in an aqueous solution by using a reagent that forms a precipitate with one or a few of the ions is called selective precipitation. This is another equilibrium stage separation process. The formation of crystals from a saturated solution is a heterogeneous equilibrium phenomenon and it can be applied to separate various ions in solution. Sodium stearate solution was used to precipitate out insoluble metal stearate salts using this principle. It is a result of metathesis reactions where there are interchanges of ions. In this case the soluble sodium stearate reacted with the cations in solution through exchange with sodium ions on the stearate salt, these reactions are shown in chemical equations below:

$$2R\,COO^- Na^+ (aq) + Mg^{2+}(aq) \rightarrow (RCOO)_2^- Mg^{2+} (s) + Na^+(aq)$$

$2R\ COO^- Na^+ (aq) + Ca^{2+}(aq) \rightarrow (RCOO)_2^- Ca^{2+} (s) + Na^+(aq)$

$2R\ COO^- Na^+ (aq) + Zn^{+2}(aq) \rightarrow (RCOO)_2^- Zn^{2+} (s) + Na^+(aq)$

The stearate salts formed above are insoluble in water at room temperature. The separation was done by the use of separating funnel since the stearate salts are less dense and floated on the brine. The insoluble stearate salts above have been known to be nontoxic and in their pure states they exist as powder. Magnesium stearate is used as a diluent for the manufacture of medical tabs and filling agents in the manufacture of capsules. Most of them are used as plasticisers in the plastic industry and lubricants in many applications (Weiner *et al.,* 1999).

Removal of sulphate was achieved by treating the salt solution with a solution of barium chloride. Barium chloride is a soluble salt in water and therefore free barium and chloride ions are released in water allowing barium ions to react metathetically with free sulphate ions to form insoluble barium sulphate. This process is represented in equations of reaction below

**Step 1. Dissolution in water**
$BaCl_2(s) + H_2O(l) \rightarrow Ba^{2+}(aq) + 2Cl^-(aq)$

**Step 2. Precipitation of barium sulphate**
$Ba^{2+}(aq) + SO_4^{2-}(aq) \rightarrow BaSO_4(s)$

Both barium carbonate and sulphates are highly insoluble in water. The $k_{sp}$ of barium sulphate in water at room temperature is $1.1x10^{-10}$. Sulphates can therefore be effectively removed from salt mixture dissolved in water as barium sulphates.

Sulphide ion is always used to separate metal ions because the solubility of sulphide salts spans a wide range depending on pH of the solution. Base insoluble sulphides and hydroxides are precipitated out when pH of the solution is 8.0 and above. In a basic solution made by addition of ammonium sulphide, the concentration of sulphide is higher than in acidic solution. Thus the ionic products for many of the more soluble sulphides are made to exceed their $k_{sp}$ values and precipitation occurs. The metal ions precipitated at this stage are: Zn, Ni, Co and Mn. Al, Fe and Cr are precipitated as insoluble hydroxides.

A solution of potassium permanganate was used to remove insoluble impurities and odour by oxidation reactions. Potassium permanganate is used widely to control taste and odour, remove colour (Lalezary *et al.*, 1986), control biological growth and remove iron and magnesium in treatment plants. Potassium permanganate is reduced to manganese dioxide ($MnO_2$) which precipitates out of solution (Hazen *et al.*, 1992). Under alkaline conditions the oxidation half reaction is

$MnO_4^- + 2H_2O + 3e^- \rightarrow MnO_2 + 4OH^-\ E° = 0.60\ V$

The permanganate anion will oxidize iron and manganese, converting $Fe^{2+}$ to $Fe^{3+}$ state and manganese (+2) to manganese (+4) state. The classic reactions for the oxidation of iron and manganese are:

$3Fe^{+2}(aq) + K^+(aq) + MnO_4^- (aq) + 7H_2O(l) \rightarrow 3Fe(OH)_3(s) + MnO_2(s) + K^+(aq) + 5H^+(aq)$

$3Mn^{2+}(aq) + K^+(aq) + 2MnO_4^- (aq) + 2H_2O(aq) \rightarrow 5MnO_2(s) + 2K^+(aq) + 4H^+(aq)$

The oxidized forms will precipitate as ferric hydroxide and manganese (IV) oxide (American Water Works Association, 1991). These precipitates are decanted or filtered out. It is the principle used for iron removal from the brine.

In most of the processes involving separation of precipitates, starch was used as a flocculant for micro particles. Flocculants adsorb micro particles and cause destabilisation either by bridging or charge neutralization. Flocculation aids the separation of suspended matter from aqueous solution since the large flocs formed settle down. Starch is a polysaccharide originated from plants as renewable sources. Native starch consists of two different types of glucose polymers amylase and amylopectine. Starch is stored as granules in plants and the morphology and crystalinity is determined by the amylopectin fraction which constitutes about 70 to 80% of normal starch (XING Guo-xiu *et al.*, 2004). Natural starch is used as a flocculant however advances are being made to improve its flocculation efficiency (Ye Aiquian, *et al.*, 2004). Starch is a natural flocculant mostly pregelatinised hence water soluble. They are corn or potato starches.

Adsorption of odours and colours was achieved using charcoal. Generally charcoal is a carbon based material which is typically hydrophobic and nonpolar. Carbon is used for adsorption of organic substances and nonpolar adsorbates. It is the most widely used adsorbent. According to Vander *et al.* (1999), charcoal has the advantage of its large micropore and mesopore volumes and the resulting high surface area. This fact led to the choice of charcoal for this purification process.

**Iodine Retention**
From the result of iodometric titration the amount of iodine retained in the purified rock salt fell in a range of 82.3 ppm to 99.8 ppm. No remarkable loss of iodine retained in the refined salt over the two weeks was noted. However there are slight differences in the quantity of iodine analysed and that added to the salt.

**Table 5 The amount of iodine retained by the refined salt for a period of two weeks**

| Duration (days) | 1 | 3 | 6 | 9 | 12 | 15 |
|---|---|---|---|---|---|---|
| Iodine levels (ppm) | 99.8 ±9.0 | 94.1 ±5.6 | 95.3 ±12.1 | 97.6 ±7.5 | 82.3 ±3.8 | 90.9 ±7.1 |

This could be inherent in the use of solid mixtures which cannot be perfectly homogenous. The amount of iodine added to the salt was meant to give 100 ppm of iodine in the salt. The fluctuations in amounts of iodine determined do not show a general drop in iodine retained in the salt over the two weeks. The refined rock salt is non hygroscopic contrasting with the raw form which exhibits hygroscopic property.

**CONCLUSIONS**
From table 1 the results of chemical analysis confirmed the presence impurities in the raw salt and this is the main reason why the salt exhibits such characteristics consistent with those of the impurities i.e. hygroscopic and reducing. Earlier studies proved that the presence of sulphur compounds which a major constituent of the salt are are strongly reducing thereby reducing iodate to molecular iodine which readily sublimes. This explains why the salt does not retain iodine in its raw form. During fractional crystallisation, an equilibrium position was reached where sulphate and carbonate salts were co precipitated with chloride at increasing rates. Consequently, these impurities cannot be removed from chloride by employing this method singly. It is practically impossible to have 100% sodium chloride using methods employed in this piece of work. From the iodine retention the techniques, employed were proved to be effective in purifying the salt for its iodisation. Conclusively, iodisation of the raw rock salt is curtailed by the presence of impurities and their removal paves way for its effective iodisation.

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