COMPLETE ANALYSIS OF CAUSTIC SODA FLAKES

Done at

Travancore
Cochin Chemicals
Limited

A dessertation submitted to the
MAHATMA GANDHI UNIVERSITY, KOTTAYAM

In partial fulfillment of Degree
Bachelor of Science in Industrial Chemsirty

By
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DEPARTMENT OF CHEMSIRY
(Bsc Chemistry Model II -Industrial
Chemistry)
ST PAUL'S COLLEGE,KALAMASSERY
2017-2020

<u>CERTIFICATE</u>

This is hereby to certify that the original and genuine investigation work has been carried out to investigate about the subject matter and the relative data collection and investigation has been completed solely, sincerely and satisfactorily by Elloy Antony , regarding his project titled 'COMPLETE ANALYSIS OF CAUSTIC SODA FLAKES " in partial fulfillment for the Bachelor of Science in Industrial Chemistry from   St.Paul's College, Kalamassery in the academic year 2017-20.
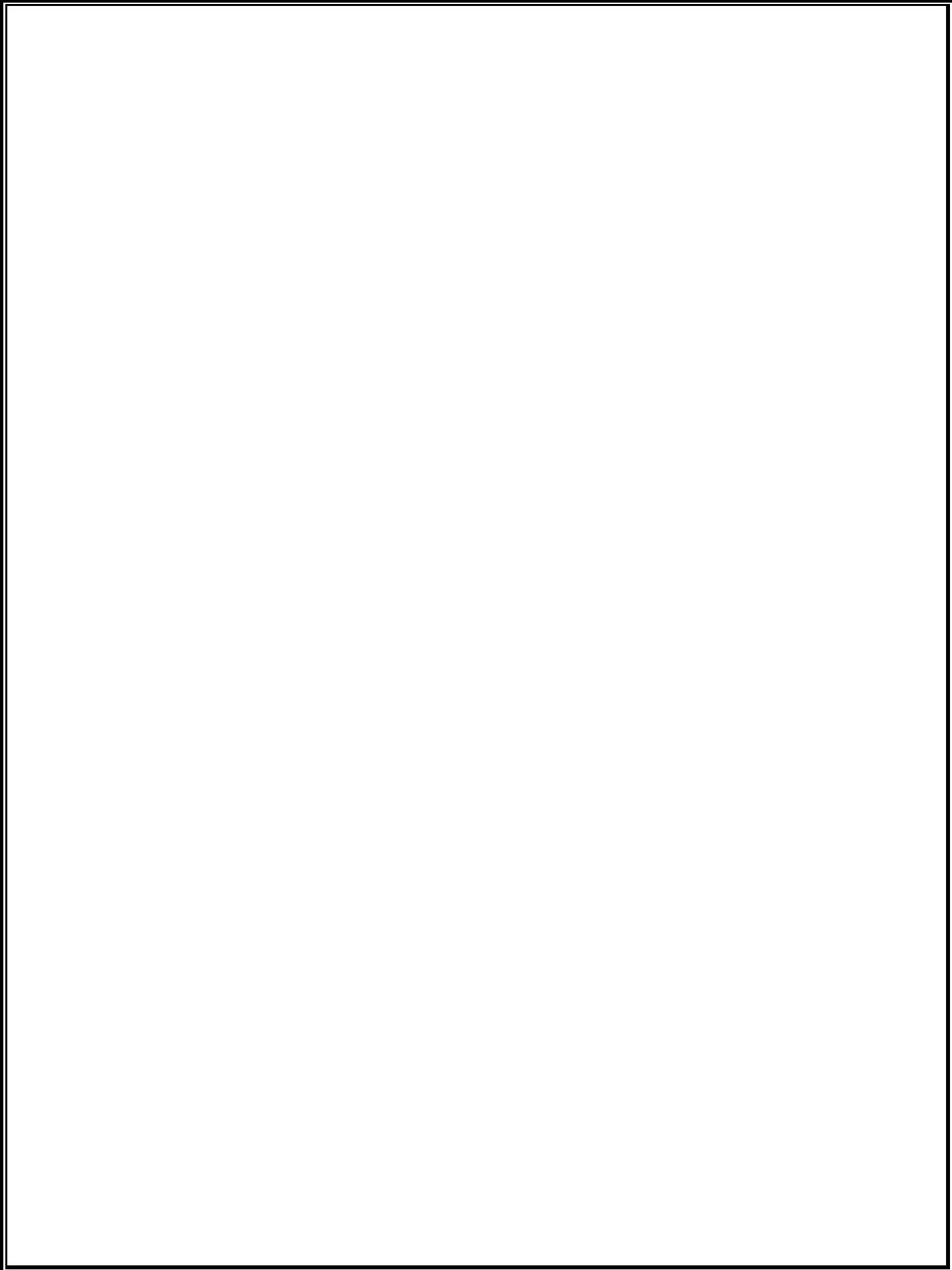
Examiner   Head of the Department   Project Guide

Date :

Place :

DECLARATION

I hereby declare that the dissertation entitled **"A STUDY OF COMPLETE ANALYSIS OF RAW SALT USED FOR CAUSTIC SODA PRODUCTION"** is an authentic record of the project work done under the guidance and supervision Mr.Texin Joseph, Head of the Department, Department of Chemistry, and Ms.Meera Jacob, Assistant professor, St.Paul's College Kalamassery.

Elloy Antony

Name of Candidate   Signature of candidate

Register number : 170021040565

ACKNOWLEDGEMENT

The satisfaction that accompany the successful completion of any task would be incomplete without mentioning the people, whose constant guidance and encouragement have served as a beam of light & crowned our efforts with success.

We would like to express our sincere thanks to Mr.Texin Joseph, Head of the Department , Department of Chemistry , St.Paul's College Kalamassery who gave   me an opportunity to carry out the thesis work in one of the most prestigious institute.

Our sincere thanks to TRAVANCORE COCHIN CHEMICALS MD. at Udyogomandol for providing opportunity and guidance to do the project work in their esteemed concern. We express our sincere thanks and deep sense of gratitude to Mr. Jomi J Kondothil Manager (T&D). Mr. Edison Francis senior officer (QC). Mr. M.P. Sonthosh Kumor, Officer (QC), Miss Jewel Joseph (QC), Mr. Haridas (QC) 8. KS. Rojendran. Chemist (QC) for their support to complete this project work successfully. Finally we would like to thank all the staff of TCC who have their support in this effort.

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Further we express our extreme gratitude to our classmates for their timely discussion and information sharing, which help to complete this project work successfully. Also we express our gratitude to our parents.

Above all we thank the God almighty to give the unlimited support and encouragement for the successful completion of this project work.

Elloy Antony

CONTENTS

INTRODUCTION

HISTORY OF THE COMPANY

The Travancore Cochin Chemicals (TCC) Is a Company Established In 1950. It has the distinction of being India's pioneer producer of Rayon grade Caustic Soda. The company was originally started as joint venture between FACT and METTURE CHEMICALS AND INDUSTRIAL CORPORATION. The purpose was to supply HCl gas to FACT for making NH_4Cl. At that time the company was named as TRAVANCORE METTURE CHEMICALS (TMC).

Soon after Inception the company faced severe financial problems and then the Travancore Cochin Government stepped In to massive financial assistance. Thus the company was renamed as the TRAVANCORE COCHIN CHEMICALS (TCC).

Commercial production was started In the year 1954 with 20 TPD Caustic Soda production capacity through the pioneering efforts of famous M/s. Sasha Sayee Brothers. The present production capacity in 175 TPD Caustic Soda by membrane cell.

The Mercury plant in TCC was stopped In July 2004 as per the May of Central Government for gradual conversion of Mercury cell plants. The membrane process plants are of ASAHI glass corporation limited, Japan and UHDE, Germany

1.2 PRODUCTS AND PRODUCTION CAPACITY

Products	Production Capacity
1. Caustic Soda Lye (100% NaOH)	57750 MT/Year
2. Caustic Soda Flakes	30000 MT/Year
3. Liquid Chlorine	23760 MT/Year
4. Commercial HCl	164182 MT/Year
5.Commercial Soda Bleach (Sodium Hypo Chlorite)	15000 MT/Year

1.CAUSTIC SODA LYE

It is clear colourless, odourless and soapy Liquid.

2.CAUSTIC SODA FLAKES

It is white deliquescent solid In flake form. Caustic soda is base alkali used in the manufacture of various articles of daily use lake Soap, Paper, Textiles, Drugs, Rubber & Petroleum industries. Caustic soda came in to being with development of the process of electrolysis.

Caustic soda is used to produce a wide variety of cleaning products like house hold bleaches, polishes and cleaning goods. Caustic soda is used in the petroleum and natural gas industry to process oil and gas in to marketable products by removing acidic contaminants. It is used to decrease corrosion of drilling equipments and to increase the solubilty of drilling mud components by maintaining an alkaline pH6.

3.CHLORINE

Chlorine is obtained as co-product In the manufacture of caustic soda. It Is important basic chemical inevitable for the manufacture of plastic, textiles,paper, insecticide etc. It is the chemical used In the purification of water. Cl^- is a greenish yellow gas with characteristic pungent smell. Liquid chlorine is amber In colour and Is one and half times as heavier than water.

2.REVIEW

2.1.USES OF NaOH

1.CHEMICAL MANUFACTURING

It Is used mainly for pH control, nutralisallon, off gas scrubbing and catalysis. It is used in the manufacture of organic intermediates, polymers and products such are propyleneoxide, poly carbonate active reagents. Large amount of caustic soda is used in all production.

2.PULP AND PAPER:

Caustic soda is used to pulp and wood chips, to extract lignin during bleaching and to neutralize acid waste streams.

3.CLE'ANING PRODUCTS:

Caustic soda is used to produce house hold bleaches, polishes, soaps, detergents and   other cleaning products.

4.PETROLEUM AND NATURAL GAS:

Caustic soda is used In the petroleum natural gas industry to process oil and gas into marketable products. especially by removing acidic contaminants. It is used to decrease corrosion of drilling equipments and to Increase the solubility of drilling components by maintaining alkaline pH-6.

5.MISCELLANEOUS CONSUMPTION:

Caustic soda is used In water treatment food processing and cotton Mercury and flue gas scrubbing. mining. glass making, textiles, dyes and adhesive preparations.

2.2.PHYSICAL PROPERTIES

Caustic soda is brittle. white, translucent crystalline solid which readily absorbs moisture and CO_2 from atmosphere. It makes a yellow stain on paper and fabric.

it is a white solid available in pellets, flakes and granules. It is also available as 32% and 50% caustic soda solution. It has a marked corrosive action on all human body tissue and hence the name caustic soda. Caustic soda means burning and caustic soda takes its name from the skin burn that it can cause. In the chemical industry it is widely known simply as caustic. Caustic soda is soluble in water and the reaction is exothermic and forms hydrates.

CHEMICAL PROPERTIES

1.With acids

Caustic soda is primarily used in neutralization reactions to form sodium salts because aqueous solution of caustic soda is highly alkaline.

$$NaOH+HCl \rightarrow NaCl+H_2O$$
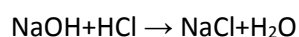
2.With elements

a) it react with metals (AI,Zn,Sn) and their oxides to form complex anions such as AIO_2, SnO_2 etc.

b) Caustic soda reacts with weak acid gases like H_2S, SO_2 etc.

$$H_2S + 2 NaOH \rightarrow Na_2S + 2 H_2O$$

$$SO_2 + 2 NaOH \rightarrow Na_2SO_3 + H_2O$$
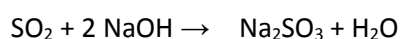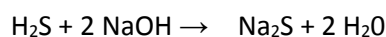
c) Organic acid react with caustic soda to form soluble salts.

3.Preparation of hydroxides

From metallic salt solution precipitates the hydroxides of the metal

$$Fe(SO_4)_3 + 6 NaOH \rightarrow 2Fe(OH)_3 + 3NaSO_4$$
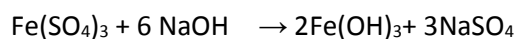
4.With ammonium salts

When heated with sodlum hydroxide ammonium salt decomposes and liberate heated ammonia.

$$NH_4Cl + NaOH \rightarrow NaCI + NH_3 + H_2O$$
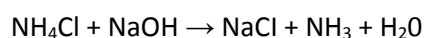
2.3 THE RAW MATERIALS USED FOR CAUSTIC SODA PRODUCTION

The main raw material used for caustic soda production is common salt NaCl. About 3700 units of electricity is consumed for about 1.6 metric tons of salt per Ion of caustic soda production.

SALT SOURCES AND METHODS OF RECOVERY

The salt of antiquity and today is obtained from various sources and ways. some sources and ways are absolute or nearly so, or are used in savages parts of the world only. The salt source in an approximate orders of the world Importance today are

1.Brine:

a) Ocean b) Salt flakes c) Natural brines.

2. Rock salt deposits

a) Artificial wellbrines b) Mines c) Quarries

3. Chemical reactions producing co-product and by product salt

4. Salt containing substance shrubs. plants. grasses

5. Salt impregnated substances peat, mud.

The methods of recovery In tentative orders of historic developments are

1.Solar evaporation of brines from various sources

2.Quarrying solid salt

3.Mining of solid salt

4.Burning of salt containing plants, using bushes as vessel.

5.Evaporation of brine on burning wood in vessel

6.Leaching of salt impregenated peat ashes and muds followed by evaporation ofbrine.

7.Sublimation of sea water or NaCl, dehydrate in cold climates.

8.Recovery of co-product or byproduct from chemical reactions.

The physical characteristics such as density, friability, natural grain size. shear strength vary from one deposit to another. In TCC the raw salt is made available from Tamil Nadu coastal regions. (Thuthukudi) & from Kutch in Gujrat.

2.4 VARIOUS PROCESESS OF MAKING CAUSTIC SODA

The caustic soda production mainly involves two stages

1. Removal of impurities present in Raw salt

2. Electrolysis.

The raw salts used for electrolysis should be analysed to estimate the amount of impurities present in it. From this. the amount of chemical needed for the removal of impurities can also be calculated.

ESTIMATION OF IMPURITIES

The raw salt contains Ca^{2+}, Mg^{2+} and SO_3^{2-} as the main impurities. The Ca^{2+} and Mg^{2+} are estimated by complexometric titration with EDTA solution and SO_4^{2-} Ions can be estimated as $BaSO_4$ by gravimetric estimation method. Cl^- is estimated by titration with $Hg(NO_3)_2$. The raw salt contains insoluble residues and moisture can also be estimated for determining the composition of the salt.

1. REMOVAL OF IMPURITIES

a) Calcium Calcium is present as calcium sulphate in the raw salt. It is removed as

carbonate by treating the brine with soda ash (Na_2CO_3) in the dissolved form.

$$CaSO_4 + Na_2CO_3 \rightarrow CaCO_3 + Na_2SO_4$$
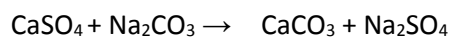
b) Sulphite Sodium sulphate in the brine is ordinariy due to the treatment of calcium sulphate with sodium carbonate. The common reactants to remove sulphates are $BaCl_2$ and $BaCO_3$

$$Na_2SO_4 + BaCl_2 \rightarrow BaSO_4 + 2NaCl$$

$$Na_2SO_4 + BaCO_3 \rightarrow BaSO_4 + Na_2CO_3$$
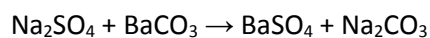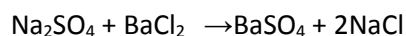
Barium carbonate is preferred over $BaCl_2$ because of the lower reagent cost. $CaSO_4$ on treatment with $BaCO_3$ gives insoluble $BaSO_4$ .The settled precipitate can be discarded. The sulphate if present is more than 6 gpl will affect the membrane used for the electrolysis.

C) Magnesium

Mg^{2+} is present as $MnCl_2$ and $MgSO_4$ in the brine solution can be precipitated by treating the caustic soda and hydrated lime. Caustic soda is preffered in T.C.C. to avoid excess Impurities problem With hydrated lime. Moreover the NaOH it used produces NaCl as a byproduct which can be reused in the plant.

$MgSO_4 + 2NaOH \rightarrow Mg(OH)_2 + Na_2SO_4$

$MgCl_2 + 2NaOH \rightarrow Mg(OH)_2 + 2NaCl$
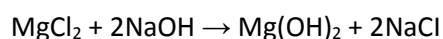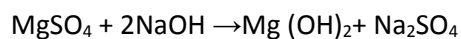
During the above treatment the Na_2CO_3 produced is removed by treating with $BaCO_3$ as said earlier. The raw brine is treated With NaOH after adding Na_2CO_3. This is because the $Mg(OH)_2$ precipitated is in the form of a large. delicate gelatinous flock with tends to settle rapidly. In so doing it traps the suspended slow setting and finely divided crystalline precipitate of $CaCO_3$ much faster and residual turbidity Is also decreased.
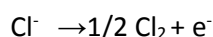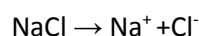
2. ELECTROLYSIS

There are' three types of cell such as mercury cell, diaphragm cell and membrane cells used for electrolysis. Now a days the mercury cells were not used because of the pollution problem. Compared to diaphragm cells. the membrane cells are more efficient and hence they are used in T.C.C for electrolysis.

REACTIONS AND ENERGY CHANGES

During electrolysis of sodium chloride the following reactions occur.

At Anode

$$NaCl \rightarrow Na^+ + Cl^-$$

$$Cl^- \rightarrow 1/2\, Cl_2 + e^-$$

At Cathode

$$H_2O + e^- \rightarrow 1/2\, H_2 + OH^-$$

$$Na^+ + OH^- \rightarrow NaOH$$

Overall Reaction

$$NaCl + H_2O \rightarrow NaOH + 1/2 H_2 + 1/2 Cl_2$$
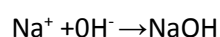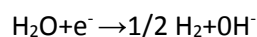
The energy consumed In the electrolysIs of brine Is the product of current flowing & the potential of the cell the theoritical or minimum voltage required for the process may be derived from the Gibbs Helmolts equation . Which express the relation between the electric energy & the heat at reaction of the system.

$$E = \frac{J\Delta H}{nF} + T\frac{dE}{dT}$$

E= Theoretical decomposition voltage.

ΔH= Enthalpy change of the reaction

T = Absolute temperature.

F = Faraday , constant Coulombs lg equivalent (96500)

n= Number of equivalent Involved.
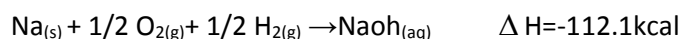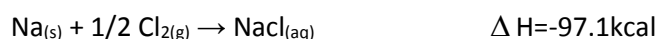
J= Electric equivalent to heat

The heat of reaction for the electrolysis of salt may be found from the heat formation of the components of the overall reaction or from the negative of these values, changes in the heat content at the system.

The Overall reaction is.

$NaCl_{(aq)} + H_2O_{(aq)}$ $NaOH_{(aq)} + 1/2\ H_{2(g)} + 1/2Cl_{2(g)}$
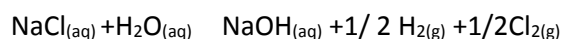
This may break down to the following reaction for formation;

$Na_{(s)} + 1/2\ Cl_{2(g)} \rightarrow Nacl_{(aq)}$ $\Delta H = -97.1 kcal$

$H_{2(g)} + 1/2\ O_{2(g)} \rightarrow H_2O$ $\Delta H = -68.4 kcal$

$Na_{(s)} + 1/2\ O_{2(g)} + 1/2\ H_{2(g)} \rightarrow Naoh_{(aq)}$ $\Delta H = -112.1 kcal$

The net change in the enthalpy(ΔH) for the overall reaction result is

    97.1 + 68.4 - 112.1 = 53.4 kcal

When this value of ΔH is substituted in the Gibbs Helmhotts equation & the change In voltage with temperature Is neglected, the value of 'F' is found to be 2.31V the omission TdE/dT Involves an error of less than 10% for most cell. The efficiency of membrane cell is found to be 95%.

PROCESS OF MAKING CAUSTIC SODA IN T CC

1. Primary Brine Purification

a. Brine saturation

b. Precipitation

c. Filtration

d. Ion exchange process

2. Secondary Brine Purification

Esstimation of calcium and Magnesium as calcium present in the sample after ion exchange process (by Hach spectra pholometer, Reagents Apparatus

3. Calibration of the Instrument :

Analysis of brine sample

4. Membrane call

Flow diagram of the membrane chlor Alkali process

Membrane cell

Ion exchange membrane cell process

Reaction in anode chamber

Reaction in cathode chamber

1.5. MEMBRANE CELL PROCESS PLANT DESCRIPTION

The process of manufacturing of Caustic Soda and chlorine through ion exchange. Membrane cell process Involves the following steps.

1.primary brine purification

2.Secondary brine purification

3.Electrolysis

4.brine dechlorination

5.Chlorine treatment

6.Hydrogen treatment

The first three processes are enough tor caustic soda production.

1.Primary Brine Purification

The primary brine purification Involves brine saturation, precipitation and filtration (purification).

a).Brine saturation

There are 2 saturatorsis In operation and other as stand by. The saturators are filled with raw salt by means of bucket elevator and feed hopper.Dchlorinated lean brine of 210 gpl sodium chloride is feed to the saturator bottom through a dip pipe. The lean brine mixed with salt and saturated brine leaves from top through a stainer. The undiscovered Impurities are removed from the bottom of the saturator periodically.

About 5-6 M^3/hr water is passed continuously through the bottom of the saturator for brine make up in the whole brine system.

b).Precipitation

Saturated brine over flows to the precipitation tanks 'A' and 'B' In series and a mixer where It is treated with $BaCO_3$, Na_2CO_3 and NaOH lye respectively. Precipitation of brine impurities such as sulphate, Calcium and magnesium takes place in the above tanks during through mixing of chemicals. Precipitated brine passes to the feed well of the clarifier through a stainer pot. A solution of 1% flocoullent prepared in the flocculent tank is added to the strainer pot by means of metering pump. About 90% of the solid are settled on the classifier and are removed from the bottom by draining to the brine sludge pitlefftuent drain.

Filtration

Four anthracite filters are provided tor the brine fitration. Normally three filters will be in service and one is stand by a centrifugal pump forces the clarified brine through the filters. The filtered brine is made acidified by mixing with HCl in a mixer. The pH is adjusted to 4-5. This is a done to supply in ionic form of calcium and magnesium to secondary purification system. In altered brine chlorine content is completely removed by adding $NaHSO_4$ to the inlet line of the filtered brine pump, also caustic soda is added to get brine of pH In between 8.5-9.5. Secondary purification unit cannot accept brine of pH below this range. Filtered brine is admitted to the secondary brine purification units.

2. Secondary Brine Purification

The secondary brine purification consists of ion exchange process. it removes traces level of brine impurities present ionic form and purified brine is collected in secondary brine receiving tank. The clear brine is admitted to the electrolyser anode chamber after heating in heat exchanges.

a) Ion Exchange Process

The ion exchange materials are in soluble acids and bases which when converted to salts, remain insoluble. The cation exchange materials are insoluble substances with fixed electro negative charge. These fixed charges are associated with mobile ion of opposite charge. The relative affinities account for the ion exchange process.

Most popular ion exchange materials are synthetic organic polymer in granular shape. These ion exchange resins are used In columns in which solutions containing ionic Impurities such as Ca^{2+} and Mg^{2-} is in contact with the resins. the ions to be removed are exchanged with the mobile positive ion (Na^+) in the resin and finally equilibrium ls achieved. Saturated resin can then be regenerated to its original .

Metal Anode: Both the Frame and Mesh are made of Ti coated with Ruthenium oxide

Metal Anode: The frame is made up of stainless steel and the mesh is Cu coated with Nickel and raney nickel

## FLOW DIAGRAM OF MEMBRANE CHLOR ALKALI PROCESS
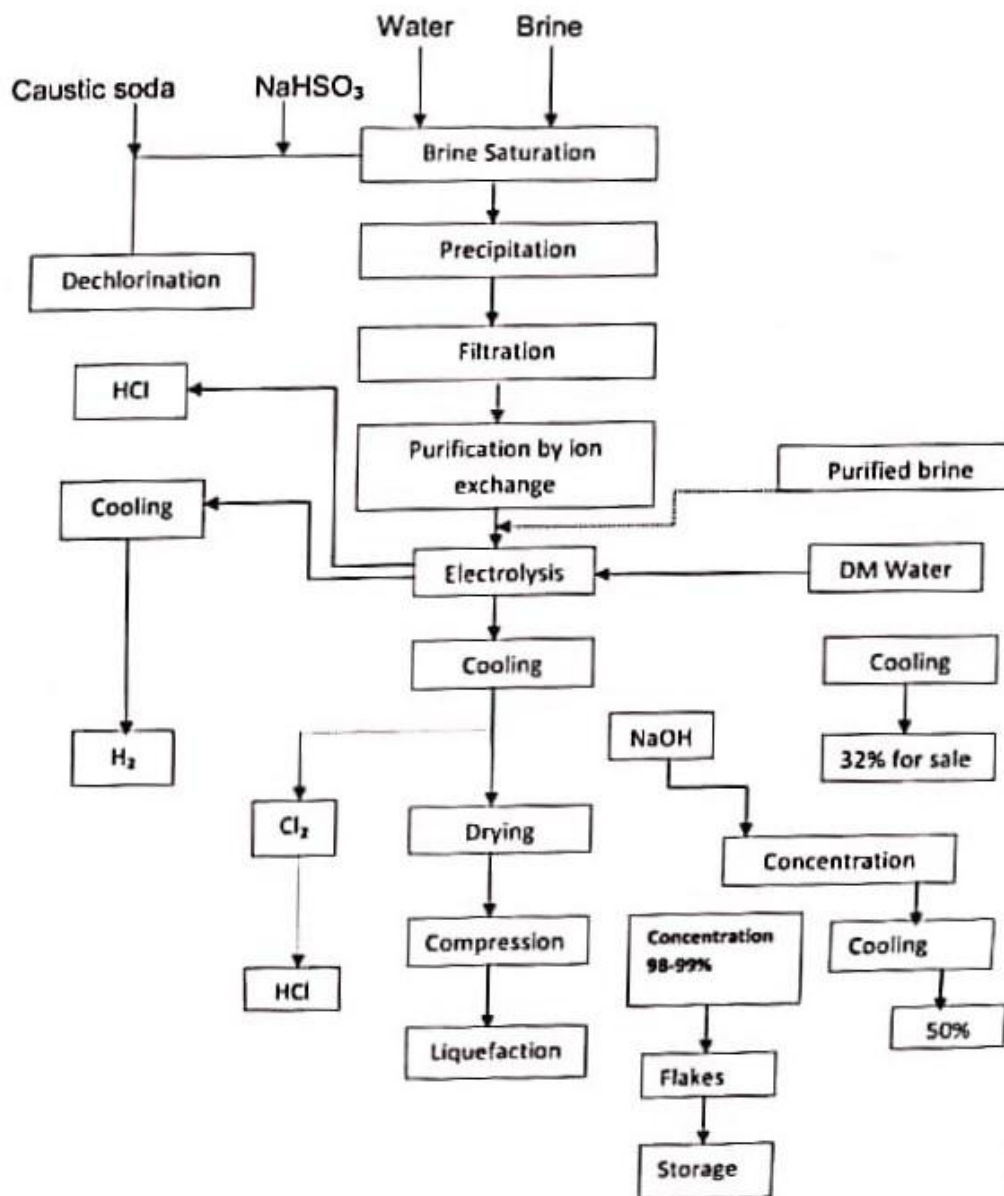
REACTION IN ANODE CHAMBER

In anode chamber electrolysios of NaCl occurs, burning Cl^- ions Into Cl_2 on the anode. The Na^+ moves to the cathode Chamber through the ion exchange membrane. In the separator and analyte is recycled through a circulation pipe.
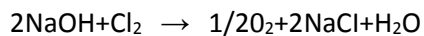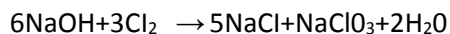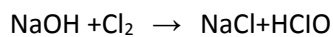
REACTION IN THE CATHODE CHAMBER

In cathode chamber decomposition of water occurs produces hydrogen gas and hydroxide ion. The OH^- ion forms caustic soda together with Na^+ ions that passed through the membrane. Generated H_2 gas and caustic soda flow up to the cathode gas separator by gas left effect. where H_2 gas is separated.

$H_2O + e^- \rightarrow H^+ + OH^-$

$Na^+ + OH^- \rightarrow NaOH$
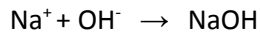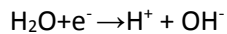
Water in the anode chamber will move to the cathode chamber through the membrane by electro osmosis. Hydroxide ion if any diffused from the cathode chamber wil produce O_2 gas, hypochlorite and chlorate by reacting.

With Cl_2 gas and O_2 gas generated in the cell during caustic soda production is tested using an orsat apparatus.

$NaOH + Cl_2 \rightarrow NaCl + HClO$

$6NaOH + 3Cl_2 \rightarrow 5NaCl + NaClO_3 + 2H_2O$

$2NaOH + Cl_2 \rightarrow 1/2O_2 + 2NaCl + H_2O$

For producing highly pure caustic soda and also to avoid the mercury pollution. mercury cell were replaced by membrane cell in TCC. The membrane cell produced from ASAHI Glass Company LTD. Japan and UHDE with 175 tons/day caustic soda production capacity.
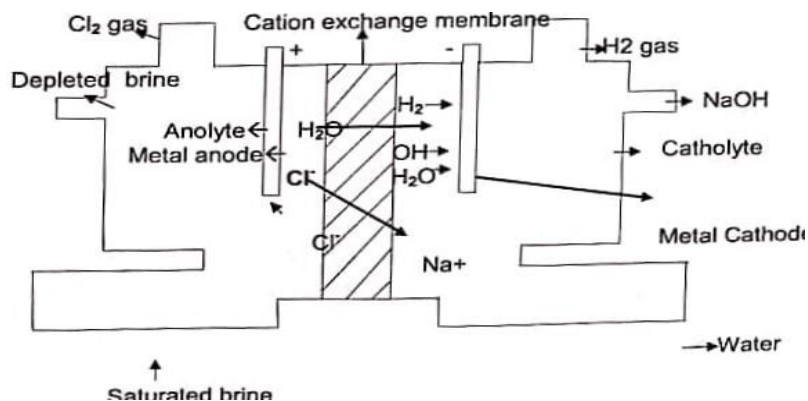
3. AIM AND SCOPE OF THE PROJECT

In TCC caustic soda is manufactured in large scale chemical industries in the country big or small depends on 10.0 for caustic soda, chlorine and hydrochloric acid.

1. The main aim of the project work Is to understand how the production of caustic soda is carried out.

2. To femiliarize with the analysts of caustic soda flakes in a chlor alkali manufacturing industry for determining the precentage of Impurities.

3. To identify the quality and to find out whether the product meet the ISI

standard.

4.EXPERIMENTAL METHODS AND PROCEDURES SAMPLING OF FLAKES :

For taking the sample scrap off the material from the top and centre. The quantity of the sample so drawn should not be less than 200 mg and the mass of the total material taken out should not exceed 1kg. Mix the quantity of the material (so called from any particular bagon a clean dry surface) rapidly. The sample should be placed on clean dry and air tight alkali glass containers. Each sample container should be sealed air tight after fulling and marked with full details of sampling, the date of sampling and batch number.
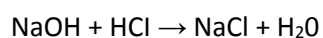
ESTIMATION OF SODIUM HYDROXIDE (NaOH AND SODIUM CARBONATE (Na_2CO_3)

Aim : Estimate the percentage of sodium hydroxide & sodium carbonate in caustic soda flakes, using standard hydrochloric acid.
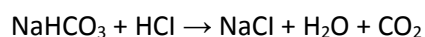
Principle :

Solid sodium hydroxide has the property of absorbing CO_2 from the air. Consequently it never obtained in the pure state. Caustic soda flakes contain the impurities like Na_2CO_3 present in it. Both sodium hydroxide and sodium carbonate can be determined simultaneously by conducting double indicator titration for that flakes is accurately weighed and titrated against standard HCl using phenolphthalein and methyl orange as indicators. When phenolphthalene is used the whole of the NaOH and half of the Na_2CO_3 will been reacted and the remaining half react when methyl orange is added. From the titre value calculations are made and percentage of NaOH and NaCl determined.

phenophthaeline:

NaOH + HCl → NaCl + H_2O

Methyl orange

Na_2CO_3 + HCl → $NaHCO_3$ + NaC|

$NaHCO_3$ + HCl → NaCl + H_2O + CO_2
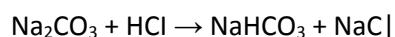
REQUIREMENTS

1.Caustic soda flakes

2.Standard hydrochloric acid

3. Indicators

   a. Phenolphthalien

   b. methyl orange

PROCEDURE

Both NaOH and sodium carbonate of the material were determined simultaneously by conducting double indicator titration. The flakes were taken in a small weighing bottle and accurately about 3 g of the material was transferred into a 250ml wide mouth conical flask containing exactly 30ml 1N HCl. Dilute with a little of freshly distilled CO_2 free water two drops of phenophthalien indicator was added.

Titrate the contents of the flask against 1N HCl, very near to the end point which is indicated by fading of pink color, note down the volume of 1N HCl is used for titration. Continue the the titration with 0.05 N HCl tilt the end point, that is disappearance of pink colour. Added methyl orange and continue the titration against N/20 HCl. End point is yellow to orange red. Noted the titre value.

Percentage of NaOH = [(30+V_1) + V_2-V_3/(20xN)] x N_1 x (40/1000) x (100/Wt Of flakes)

Where

N_1 = normality of IN HCl

N_2 = Normality of N/20 HCl

V_2 = Volume of M/20 Phenolphthalien

V_3 = Volume of M/20 methyl orange

V_1 = Volume of 1N HCl

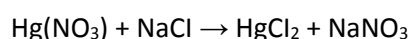Percentage Of Na_2CO_3 = (V_3 x V_1x 1/20 x 53/1000 x 100/wt)

4.3 ESTIMATION OF SODIUM CHLORIDE

Aim:

Estimate the amount of chloride as sodium chloride in caustlc soda flakes using mercuric nitrate.

Principle:

When chloride ions are titrated with mercury (ll) nitrate solution In presence of sodium nitro prusside indicator, the mercury (ll) nitrate solution was converted into mercuric chloride.

$Hg(NO_3) + NaCl \rightarrow HgCl_2 + NaNO_3$

$Hg (NO_3): + Na_2 [Fe(CN)_5NO] 2H_2O \rightarrow Hg[Fe(CN)_5NO]$

The excess Hg (NO₃) solution react with sodium nitro prusside and form soluble mercuric salt. The first appearance of turbidity Is the end point of the reaction.

REQUIREMENTS:

1. Mercury (ll) nitrate Solution

About 16.29 of mercury (II) nitrate Hg(NO3)z is dissolved in 500 ml distilled water.

2.Nitric acid Solution (0.1 N)

About 3.15 gm of HNO_3 is dissolved in 500ml distilled water.

3.Sodium nitro prusside solution

About 1gm of sodium nitro prusside reagent is dissolved in 5CC HNO₃ and make up to 1 liter distilled water 10g of solid flakes sample was weighed accurately into a conical flask. Diluted It to 100ml using demineralised water. Neutralized the solution by adding Conc HNO₃ Then added 5ml of add in excess and cooled the solution. Then added sodium nitro prusside Indicator, titrated the solution against 0.0lN Hg (NO₂)₂ from burette. The end point is the appearance of the turbidity due to the formation of Hg [Fe (CN)₅ NO) noted the titre value.

Procedure:

Percentage of chloride as sodium chloride

$$= \frac{T.V \times \text{strength of } Hg(NO_3)_2 \times 58.45 \times 100}{(1000 \times \text{wt of flakes})}$$

Equivalent weight of NaCl. = 58.45

4.4 ESTIMATION OF IRON

AIM :

Estimate the amount of iron ion caustic soda flakes using pottasium thiocyanate solution.

PRINCIPLE :

Nitric acid oxidizes ion from ferrous state to ferric reacts with thiocyanate to give an intense red colored compound, Which remains in the solution. Ferrous ion does not react. The color is due to ferric thiocyanate. The solution contain the complex. $[Fe(CN)_6]^{3-}$ having used red coloration.

$Fe^{3+} + 3CNS \rightarrow [Fe(CNS)_3]$

$Fe^{3+} + 6CNS \rightarrow [Fe(CNS)_6]$
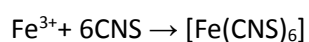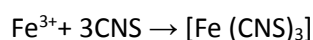
Iron is estimated colorimatrially by visual comparisons.

REQUIREMENTS

1.Sample solution

2.Con. Nitric Acid

3.Pottassiurn thiocynate

4.Nessler's cyclinder

5.Standard iron soln. : Dissoive 0.79 of ferrous ammonium sulphate.

6.$[FeSO_4 (NH_4) SO_4 6H_2O]$ in 100ml of distilled water and 10ml of concentration H_2SO_4 and dil $KMNO_4$ solution drop wise until a slight pink colour persist after stirring , then diluted with water to 1000 ml. Transfer 100ml of this solution to 1000 ml volumettic flask and dilute again to 1000 ml. 7.1 ml of this solution is equivalent to 0.01mg of iron (Fe).

Procedure:

Take 10g Rakes and neutralize it with iron tree. A R. HCl and make it acidic. Boil the solution, while boiling one or two drops of Con. Nitric acid to convert iron from the ferrous state to ferric state was added. Boil It for 10 minutes. Then cooled.

Make up this solution into 100ml and take 50ml of thus solution. Add potassium thiocynate solution with dil. HCl and diluted to 100 ml. Run a blank with standard 0.01 gpl Fe solution and compare the color.

Fe(ppm) = Tv x (0.01/1000) x (100/50) x (10^6 / Wt.Of sample)

4.5 ESTIMATION OF NICKEL

AIM :

Estimate the amount of nickel in caustic soda flakes.

PRINCIPLE:

Nickel present in the sample solution is precipitated as insoluble nickel dimethyl glyoxine from the ammoniacal solution or nickel ammoqmum sulphate by adding alcoholic solution of demethyl glyoxime.

$NiSO_4 + 2C_4 + H_2O_2N_2 \rightarrow Ni(C_4H_7O_2N_2)_2 + H_2SO_4$
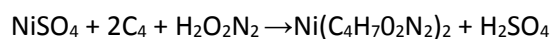
From the mass of dimethyl glyoxime complex the mass of nickel is calculated by knowing that 288.69g complex, contains 58.6g of nickel.

REQUIREMENTS:

1. Sample solution

2. Dil. hydrochloric acid

3. Conc Nitric acid

4. Pottasium bitartarate

5. Bromine water

6. Ammonia

7. Dimethyl glyoxime reagent

PROCEDURE:

10g of the flake is accurately weighed and neutralised with an HCl and added to it a few drops 01 Conc HN03, cooled and make up to 100ml. Take 50ml from the made up solution, added a little pottasium bitartrate to mark the iron ion then added a little bromine to oxide in the nickel and then neutralized with ammonia. Added dimethyl glyoxime and compared the color with standard 0.01 gpl solution.

Nickel(ppm) = T.V. x 0.01 x 100 x 10^6

1000 x 50 x Wt of Sample

4.6 ESTIMATION OF SODIUM NITRATE:

AIM :

Estimate the amount of sodium nitrate in the caustic soda flakes.

PRINCIPLE:

Sample solutions neutralized with 1:2 H_2SO_4 and add indigo as indicator, disappearance of colour shows the presence of $NaNO_2$. if $NaNO_2$ is present sample solution neutralized with 1:2 H_2SO_4 then titrated against N/10 $KMnO_4$ till

permanent pink color persist.

$2 KMnO_4 + 3H_2SO_4 \rightarrow K_2SO_4 + 2MnSO_4. + 3H_2O + 5[0] + 5 NaNO_2 \rightarrow 5 NaNO_3$
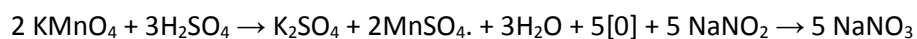
REQUIREMENTS:

1. Sample solution

2. 1:2 H_2SO_4

3. N/10 $KMnO_4$

4. Indigo as indicator

5. Reducing solution

PROCEDURE:

Weighed out 10ml of flakes and dissolved In water, neutralized with 1:2 H_2SO_4, heated to boiling added 2mL indigo indicator. if the color disappeared it show the presence of $NaNO_2$.

If there is the presence of $NaNO_2$ fresh flakes sample is dissolved In water neutralize with 1:2 H_2SO_4 cooled and then titrate against standard N/10 $KMnO_4$. End point is pink color.

$$NaNO_2(ppm) \quad = \quad \frac{T.V. \times N/10\ KMnO_4 \times 69 \times 100}{2 \times 1000 \times Wt.of\ Sample}$$

5.RESULTS AND DISCUSSION

<div align="center">ANALYSIS OF FLAKES</div>

5.1 Determination of NaOH And Na_2CO_3

Weight of Flakes	2.4912 g
Volume of 1N HCl	57.432 mL
Volume of N/2 Phenopthalien	8.7 mL
V_2	0.65 mL
N/20 Methyl Orange (V_3)	1.066 N
Normality of 1N HCl (N1)	0.0533 N
Normality of N/20 HCl (N_2)	57.432 x 1.066 x $\frac{40 x 100}{1000 x 2.4912}$ = 98.298%
Percentage of NaOH	$\frac{1.5 x 53 x 100}{20 x 1000 x 2.4912}$ = 0.129%

5.2 Determination of Chloride as NaCl

Weight of Sample	8.3448 g
Percentage of Chloride	T.V x Strength of $HgNO_3$ x$\frac{58.5\ x 100}{1000\ x\ wt\ of\ sample}$ $= \frac{23\ x\ 0.01\ x\ 100\ x\ 10^6}{1000\ x\ 100\ x\ 8.3448}$ = 0.0161 %

5.3 Determination of Iron (ppm)

Iron (ppm)	$\frac{T.V.\ x\ 0.01\ x\ 100\ x\ 10^6}{1000\ x\ 50\ x\ wt\ of\ sample}$ $= \frac{1.2\ x\ 0.1\ x\ 100\ x\ 10^6}{1000\ x\ 50\ x\ 10.1235}$ = 2.37072

5.4 Determination of Nickel (ppm)

Titerate Value	0.5
Weight of Sample	10.1235 g
Nickel in the Solution	$= \dfrac{0.01 x\ T.V\ x\ 100\ x\ 10^{\wedge}6}{1000\ x\ wt\ of\ sample\ x\ 50}$ $= \dfrac{0.01 x\ 0.5\ x\ 100\ x\ 1000}{1000 x\ 10.1235\ x\ 50}$ $= 0.9878 < 1\text{ppm}$

5.5 Determination of NaNO$_3$

NaNO$_2$ (ppm)	T.V. x KMnO$_4$ x $\dfrac{69}{2 x 1000}$ x $\dfrac{10000000}{wt.of sample}$

7 days of analysis showed NaNO$_3$ is absent in the flakes

COMPARISION OF SODIUM HYDROXIDE FLAKES FOR VARIOUS COMPONENTS

Table 1   ANALYSIS ON WET BASIS

Components	FLAKES						
	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6	Sample 7
NaOH (%) By mass	98.1	98.34	98.38	98.50	98.43	98.44	98.64
Na_2CO_3 By mass	0.159	0.170	0.169	0.170	0.165	0.173	0.169
NaCl (%) By mass	0.0161	0.022	0.024	0.022	0.023	0.024	0.022
Fe (ppm)	2.37072	4.7	4.9	5.23	4.7	5.40	4.9
Ni (ppm)	<1	<1	<1	<1	<1	<1	<1
$NaNO_2$	Nil	Nil	Nil	Nil	Nil	Nil	Nil

$$\text{Value on dry Basis} = \frac{\text{Value on Wet basis} \times 100}{a+b+c+........K+1}$$

Table 2 : ANALYSIS ON DRY BASIS

Components	FLAKES						
	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6	Sample 7
NaOH (%) By mass	99.80	99.79	99.80	99.79	99.79	99.79	99.80
Na_2CO_3 By mass	0.158	0.173	0.171	0.172	0.171	0.175	0.171
NaCl (%) By mass	0.025	0.022	0.024	0.022	0.023	0.024	0.022
Fe (ppm)	5.68	4.7	4.9	5.23	4.7	5.40	4.9
Ni (ppm)	<1	<1	<1	<1	<1	<1	<1
$NaNO_2$	Nil	Nil	Nil	Nil	Nil	Nil	Nil

6. CONCLUSION

The experiments were carried out to find the various amount of impurities present In the Caustic Soda Flakes. Experiments showed that the given caustic soda sample were above 99.5% pure and reaches international qualities.

7.BIBLIOGRAPHY

1. Kirk-Othmer encyclopedia of Chemical technology 5th edition, John Wileg &   Sons, accessed November 21,2005.

2. Heaten A (1996), An introduction to Industrial Chemistry, 3rd   edition.

3. Thaker H.Leon, Kastner, Juslin Carcas disposal, A comprehensive review, Chapter 6, National Agricultural Biosecurity Centre, Kansas State University, 2004.

4. Green Wood Norman N Eam shaw A (1997) chemistry of Elements 2nd edition.

5. "Trafiqura knew of waste dangers" BBC Newsnight 16 September 2009.

6. Operation Manual 2001, Travancore Cochin Chemicals

7. N.M. Pont and J.S.Moor House, "Modern Chloralkali Technology", Vol 4 Society of chemical industry Elseview applied science, London,1990.