Industrial Inorganic Chemistry
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Lecture - 24
Hydrogen Fluoride and Aluminium Fluoride

So, welcome back to this class once again, where we are talking about the production of fluorine gas and our by product was hydrogen gas also. So, we have to use the electrolysis cell and the corresponding structure of that particular electrolysis cell we have already discussed.

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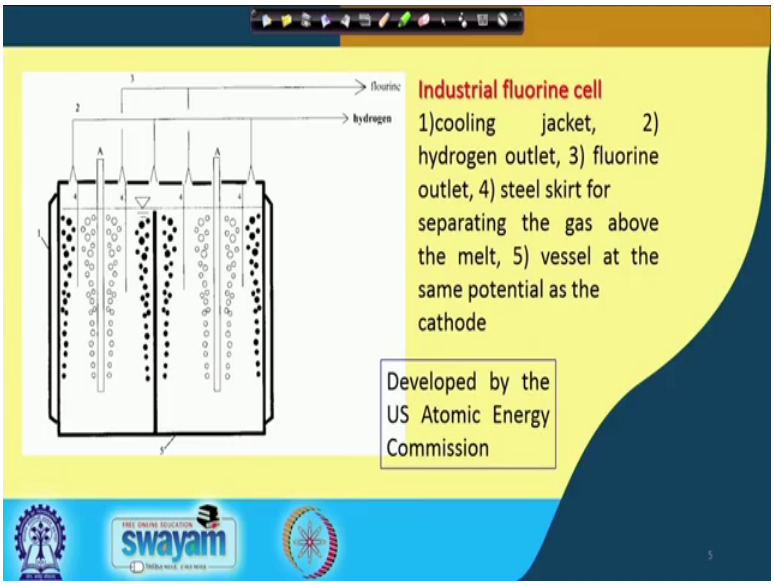

Industrial fluorine cell

1)cooling jacket, 2) hydrogen outlet, 3) fluorine outlet, 4) steel skirt for separating the gas above the melt, 5) vessel at the same potential as the cathode

Developed by the US Atomic Energy Commission

So, now we will see the diagram, the flow diagram for it. So, if you have a simple basically these are all big chambers, the you have the monell or the steel made container, and you have the corresponding cathode material of it. And as I told you that these spaces are basically separated. So, you have multiple numbers of cathodes and anodes over there. So, if your the corresponding hole material of this particular cell is one type, then you can have the corresponding anodes over there. So, it is basically a flow diagram for your industrial flourine cell where we can utilize the corresponding electrolysis of your h a present in a melt. So, one basically the number wise is you have the cooling jacket, because you have to cool it because sometimes during the production your temperature may go rise.

Then, you have number 2 is your hydrogen outlet. So, hydrogen outlet from this particular these electrodes which are very close to your steel skirts number 4. So, you have only two outlets one outlet is for fluorine, and another outlet is for hydrogen. So, you are happy that you can take out these two quantities what we are producing through electrolysis from one is from the anode and another is from the cathode.

Then, number 3 is fluorine outlet; number 4 is the steel skirts these lines, these four number 4 lines, all these lines are steel skirts for separating the gas above the melt because you have thing. So, the two bubbles basically the hollow and the solid bubbles, one is for the cathode and another is for anode. So, from bottom, these are the bottom of the melt. And through the side of these electrodes, so these are basically is connected to the body of this electrolysis cell is one type. So, this is one type. And this is the body, and this is the body. So, you have the three black one and two lighter one, the anodes.

So, these bubbles are coming out and when they are accumulating basically these bubbles or the gases have to leave the melt. So, when it is going up basically these steel skirts are hanging from the top when it is the vacant space where the gases are being stored. So, they are not getting contaminated if they are separated nicely. So, this separation basically giving you this one. So, this particular chamber giving all these gases and you have only one outlet.

Similarly, for this one also you have all these gases around this electrode, but you have one outlet. Similarly, from the side of the electrolysis cell, this side and this side, you have this and only you have the electrode outlet that is your hydrogen. So, hydrogen from the cathode and fluorine from the anode will be produced, and this particular one originally developed by US Atomic Energy Commission because in our country as well as everywhere there will be a constant demand of your fluorine supply that we will also see for the uranium enrichment, uranium hexafluoride is also one of the good species. So, like our beers in our country. So, United States Atomic Energy Commission has also developed this particular cell, because most of these materials are sometimes is patented. So, is the US patent you can have all the time.

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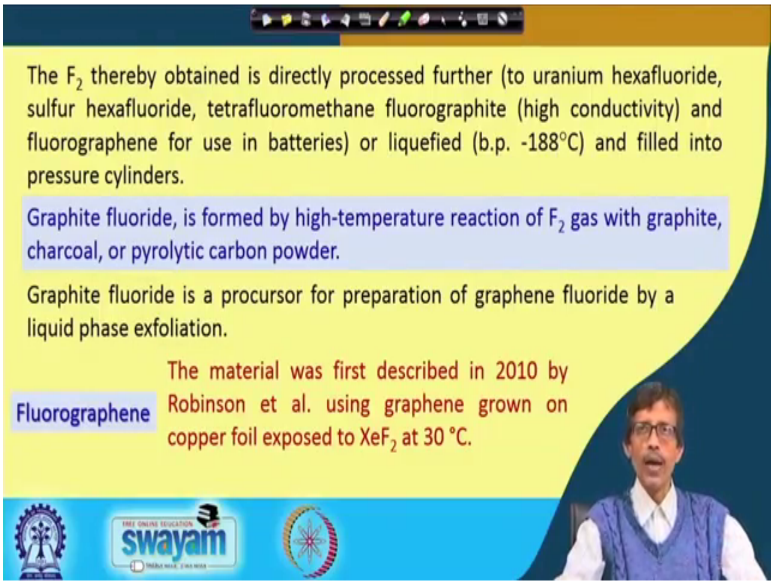

The F_2 thereby obtained is directly processed further (to uranium hexafluoride, sulfur hexafluoride, tetrafluoromethane fluorographite (high conductivity) and fluorographene for use in batteries) or liquefied (b.p. -188°C) and filled into pressure cylinders.

Graphite fluoride, is formed by high-temperature reaction of F_2 gas with graphite, charcoal, or pyrolytic carbon powder.

Graphite fluoride is a procursor for preparation of graphene fluoride by a liquid phase exfoliation.

Fluorographene

The material was first described in 2010 by Robinson et al. using graphene grown on copper foil exposed to XeF_2 at 30 °C.

So, this thing the idea is that very simple that how you get this. And when you get this particular one, so if we get fluorine and fluorine is obtained is directly processed further. So, once you get from the electrolysis cell the fluorine, so you can utilize that particular fluorine for making uranium hexafluoride just now I told you that you can make it u f 6 or sulphur hexafluoride s f 6, tetra fluoro methane, where all the hydrogen atoms of the methane can be substituted by fluorine or fluorographite. So, one good material that means the modification of the graphite material you can have because the graphite is only that carbon containing material.

But if you can have some amount of fluorine in it, so it can have the fluorographite type of thing. So, this particular one, so the carbon of that particular graphite, that means, you have the corresponding that graphite seat, we know that the corresponding benzene rings are there that means, hydrogens are there. So, those hydrogens will be substituted by fluorine will giving you the flora graphite will have a high conductivity. And another one which is a very useful material nowadays during the last 10-15 years through the work on the graphene material the production of the graphene, how you get good amount of graphene from the graphite seeds. So, these graphenes are mostly useful.

So, flora graphene we use for making the battery, the lithium ion battery, the sodium ion batteries and all these thing. So, this fluorographene, making this fluorographene is also a very useful or a challenging task. Or sometimes, you can liquefy it with a boiling point of minus 188 degree centigrade. So, the liquid fluorine gas, so liquefied fluorine gas to transport from one particular place, that means, the site of production to the site of consumption. So, is filled into pressurized cylinder, the way we pressurize the different gases like your CNG or LPG gases for our cooking. Similarly, this fluorine gas can also be filled in all those pressurized cylinders.

Then, we can have this flora graphite or graphite fluoride what we call how we make it, it is very easy to have your graphite material in your hand from any good carbon source. So, is basically the fluorination of the graphite material; the fluorination of your graphite material at high temperature reaction; that means, it is a gas phase reaction is not a thing that what we consider because we will little bit if we are able to consider that considering the fluorine, because fluorine is a very useful atomic centre for making the drug molecules, the pharmaceutical molecules also, because the activity drastically changes.

If you have a drug molecule or the medicine we having a CH bond and if you move from a CH bond to a CF bond, the reactivity pattern and all these things are drastically getting changed. So, a large number of these all medicinal molecules are available where you have carbon fluorine bonds. So, fluorination of those organic molecules will also be interesting. But here if we just try to have a material which is a graphite material, and we only just go for the fluorination. So, you use the fluorine gas either from the cylinder or insitu production that means, immediate production; that means, you must have the corresponding electrochemical cell, and you directly produce a chlorine gas and pass over there.

So, with graphite charcoal, so basically is a very raw material that we graphite thing you can have purified form or simply the charcoal-charcoal is your good carbon source or pyrolytic carbon powder. We all know that the carbon in our country also large number of companies are making carbon powders, carbon block, and all these thing. It is a very cheap quantity thing what we put in our cycle tyres, our car tyres and all these things for increasing the thermal conductivity. So, those material can also be utilized for the fluorination. So, you get the corresponding graphite fluoride or the fluorographite. So, is the precursor also for the preparation of graphene fluoride by a liquid phase exfoliation.

So, the technique which is applied, that means, layer by layer removal from your graphite block or graphite seat or whatever it is. So, the layer wise if you are able to remove one of the lair from that graphite surface it is your graphene. Graphene is nothing but a seat. So, the liquid phase exfoliation the technique is known as exfoliation that means the layer wise removal of that graphene seat from the graphite material. So, you make the graphite or the fluorographite or the graphite fluoride, and then you try to get the graphene fluoride, because the fluorographene are also very important material for industrial application.

Where we use this fluorographene? Fluorographene is the material first described not very long ago, it is only in 2010 by Robinson et al. The person who was doing is Robinson using graphene grown on copper foil, how it was discovered for the first time. Because you make a material you produce a material you never know that the material will be highly useful afterwards, but as an academic interest we do basically in the laboratory sometimes we have that excitation that why do not you make some new molecule or new material out of this.

So, he thought that it can be used from that particular one when that graphene basically is utilized for the fluorination on a copper foil because if you have the graphene on copper foil it can be deposited on the copper foil and is simply exposed to not your direct fluorine gas from the cylinder, but another molecular compound which is xenon difluoride Xe F 2 at only room temperature other than room temperature in our country we mostly most of the time we have this is our room temperature 25 to 30 degree centigrade.

So, if it is exposed to xenon difluoride, you get the corresponding fluorographene. So, this is the most useful technique not the other, fluoride compounds of xenon that means, xenon tetrafluoride or xenon hexafluoride. It is only the difluoride. Difluoride would be useful to give you the fluorographene compound.

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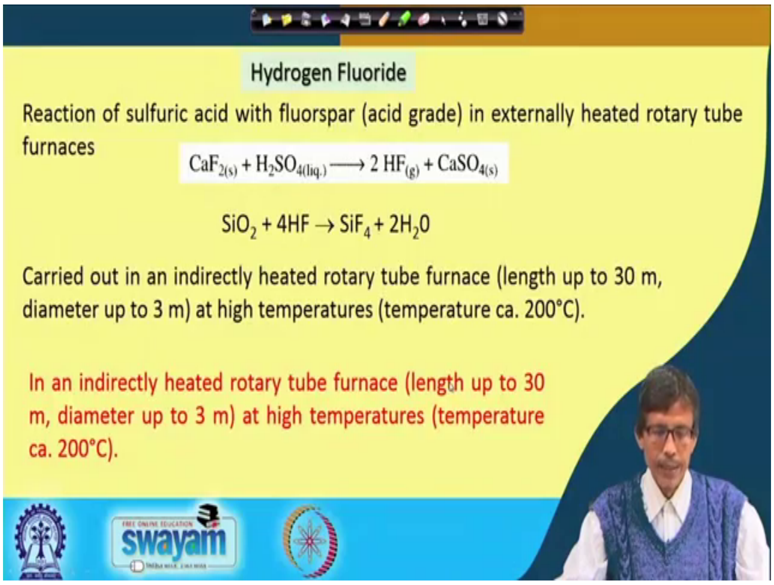

Hydrogen Fluoride

Reaction of sulfuric acid with fluorspar (acid grade) in externally heated rotary tube furnaces

$$CaF_{2(s)} + H_2SO_{4(liq.)} \longrightarrow 2\,HF_{(g)} + CaSO_{4(s)}$$

$$SiO_2 + 4HF \rightarrow SiF_4 + 2H_2O$$

Carried out in an indirectly heated rotary tube furnace (length up to 30 m, diameter up to 3 m) at high temperatures (temperature ca. 200°C).

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So, next we will just go for the preparation of hydrogen fluoride. So, fluorine gas is gone. Now, we go for the hydrogen fluoride gas which is also like that of your hydrochloric acid gas or hydrogen chloride gas is hydrogen fluoride gas a HF which will also be very much useful material in different industries.

So, again we will take the help of fluorspar. The fluorspar will be a raw material, but now it is the acid grade fluorspar treat it with sulfuric acid. Earlier we are treating in presence of bauxite. Now, in absence of bauxite we treat it in sulfuric acid and heat it in a rotary tube furnaces. So, you take a different type of furnace now definitely since these are all industrial production, we would have to use some reactor or some furnace. So, now, you have instead of calcium fluoride sulfuric acid alumina last time the same reaction without alumina only Al 2 O 3. So, you stop it you get 2 HF plus calcium sulphate, because we are not interested to have the corresponding aluminium fluoride.

So, this when then is treated with silica that we have seen that you can have the hexa fluoro silicic acid or silicon tetrafluoride. So, when the produced HF, you utilize to treat the quartz or any other bad quality of silica also quad is the good quality of silica. So, you have this silicon dioxide that means silica which treated with HF, so giving you silicon tetrafluoride, so that is the basic or the underline principle we all know that the glass surface etching of the glass surface. If we go for the etching of these glass surfaces, we use a chip because the silica present in it on the glass will be destroyed through the formation of silicon tetrafluoride and that as a gas material will be escaped away from there.

So, how you go for this reaction the reaction is carried out in an indirectly heated rotary tube furnace. So, as I told you this a rotary not only rotary furnace it is a special type of furnace, but it is the tube furnace where the length is 30 meter and diameter is up to 3 meters, so is a huge one. So, 30 meter in length and diameter of 3 meter, but is the tube shape and a high temperature withstand of 200 degree centigrade. So, it is a very high temperature reaction to treat your fluorspar by sulfuric acid.

So, in an indirectly heated rotary tube furnace where we can use of the same dimension that means, 30 meter of length and 3 meter of diameter at high temperature, temperature is basically a 200 degree centigrade.

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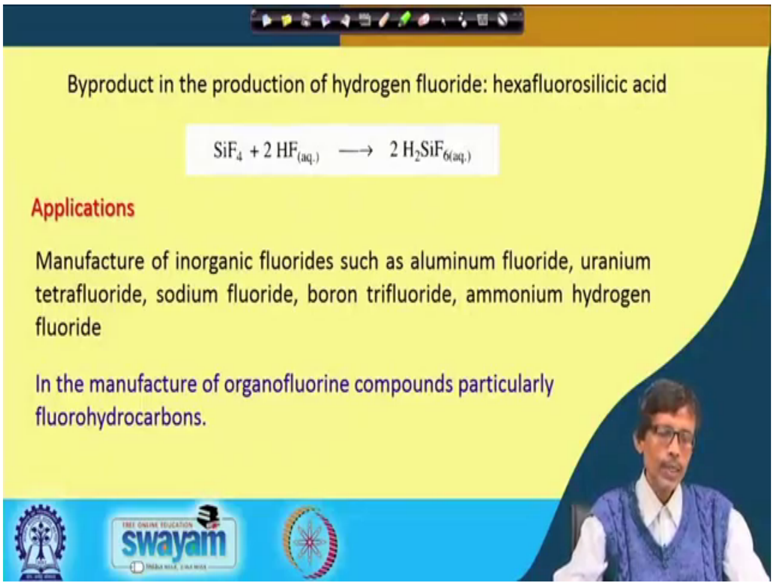

Byproduct in the production of hydrogen fluoride: hexafluorosilicic acid

$$SiF_4 + 2\,HF_{(aq.)} \longrightarrow 2\,H_2SiF_{6(aq.)}$$

Applications

Manufacture of inorganic fluorides such as aluminum fluoride, uranium tetrafluoride, sodium fluoride, boron trifluoride, ammonium hydrogen fluoride

In the manufacture of organofluorine compounds particularly fluorohydrocarbons.

So, this heating procedure is basically giving us the corresponding production of your hydrofluoric acid. And the by product in this production of hydrofluoric acid is that if you have as I told you that you use silica or quartz for taking up your HF production for its conversion to silicon tetrafluoride or H 2 Si F 6, but sometime your silica Si O 2 is already present in your fluorspar, your quality of flora spar is not so good that it has already silicon dioxide or the silica material in it.

So, whatever a HF you produce there, so in situ the produced a HF will attack your silicon dioxide and that silicon dioxide will produce Si F 4. And ultimately in presence of excess a HF, it will be producing H 2 Si F 6 or hexa fluoro silicic acid. So, this is the reaction. What you get over there; that means, whatever form as silicon tetrafluoride which will be your gas. The same basic reaction for etching of the glass surface will trap. So, during it is the escape passage of your silicon tetrafluoride which is also a gas material will take up the HF because you have the HF in the liquid phase aqueous HF. So, it will take and will remain there in the aqueous medium as H 2 Si F 6 instead of HF.

So, you have large amount of H 2 Si F 6 along with your HF. So, you have to separate these, that means, H 2 Si F 6 has to be separated to increase the concentration of HF or to increase the purification process or to improve the purification process of your HF, such that a good quality of HF you can have, because most of the time for our laboratory you will also HF what we are getting some aqueous solution only say 40 percent of HF we get for the day to day laboratory use. Sometime, it can have the corresponding 60 percent or 70 percent HF in the aqueous medium. So, most of the time you take that particular material, that means the raw HF which is available in the aqueous medium as the corresponding, one only thing that you have to level it you should know the corresponding concentration whether it is a 40 percent HF bearing aqua HF or a 50 percent HF bearing aqueous HF.

Then, we can utilize for the different applications one such application for making is the direct inorganic fluoride making. As we have seen earlier that directly using fluorspar and sulfuric acid we can make aluminium trifluoride or aluminium hexafluoride. Then we now use that particular one in a large scale for the manufacture of one such useful inorganic fluoride is the aluminium fluoride because for getting aluminium out of that as your cryolite because the cryolite is the naturally occurring material. Similarly laboratory prepared or the industry prepared aluminium fluoride as cryolite Na 3 Al F 6 can also be utilized for aluminum production.

Then, aluminium uranium tetrafluoride U F 4; sodium fluoride, one very useful molecule which used in the all the laboratories all the research laboratories, in all organic chemistry laboratories is the boron trifluoride B F 3. Then ammonium hydrogen fluoride ammonium by fluoride, what we use in typical anti chemistry laboratory classes also is a typical masking agent, because it reacts very quickly with the iron present in it.

So, if we try to remove iron from any medium, that means, the colour from that medium also that iron always we know in the ferric state it is basically light yellow to orange. So, to remove that particular one we as add excess fluoride as ammonium hydrogen fluoride and ammonium by fluoride, ammonium HF ammonium by fluoride. So, the entire iron can be taken away as Fe F 6 3 minus. So, it is also decolorized, and you can take out that particular one even for the titrimetric procedure for analytical chemistry you add this. And you can avoid the iron estimation, because this iron will not interfere during that estimations suppose we are analyzing iron and copper together.

Then it is utilized for the manufacture of different organofluorine compounds particularly fluoro hydrocarbons or fluoro corresponding fluora derivative of the ethylene as I told you earlier that you can make it tetra fluoroethylene for the preparation of teflon. So, the tetrafluoroethylene preparation is also a preparation of your flouro hydro carbon type of material.

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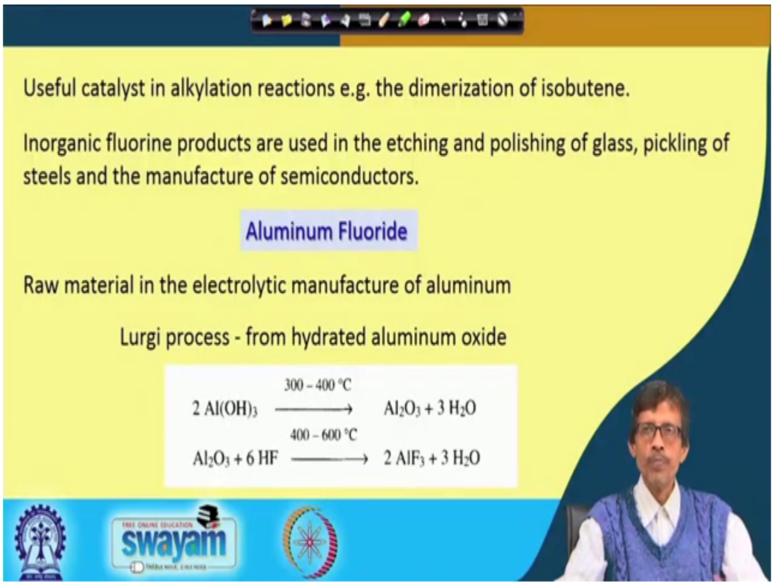

Useful catalyst in alkylation reactions e.g. the dimerization of isobutene.

Inorganic fluorine products are used in the etching and polishing of glass, pickling of steels and the manufacture of semiconductors.

Aluminum Fluoride

Raw material in the electrolytic manufacture of aluminum

Lurgi process - from hydrated aluminum oxide

$$2\,Al(OH)_3 \xrightarrow{300-400\ ^\circ C} Al_2O_3 + 3\,H_2O$$

$$Al_2O_3 + 6\,HF \xrightarrow{400-600\ ^\circ C} 2\,AlF_3 + 3\,H_2O$$

And sometime a very good quality that means your material is highly pure and a small amount can be utilized as the good catalyst. So, is a useful catalyst for alkylation reactions, if we want to put more number of alkyl functions on a particular type of carbon centre we say that we are going for alkylation reaction such that you have a methyl function which is CH 3. You can go for is a tertiary butyl function by replacing all the three hydrogen centres or the hydrogen atoms by three methyl groups.

So, is a basically a methylating process or methylation process, because it is a very useful reaction. In the biology also we know it as a bio methylation process. So, this particular methylation process is useful metal ions are also getting methylated out of this. So, in all these typical organic molecules or organic backbones, if you go for the alkyllation reactions, and once such simple example is you take isobutene. And two of the isobutene if you try to attach it, that means the dimerization of isobutene in that particular case your this material can function as a good catalyst out of that.

Then we can go for the production of large amount of inorganic fluorine products used in the etching as I told you etching of the glass surface polishing of the glass surface also. That if we have unwanted silica material over the glass material so that unwanted silica or Si O 2 can be removed by reaction with your HF as Si F 4, and that will go as Si F 4 is the gaseous material.

Then pickling of the steels basically so is a particular process of steel making material. So, one particular process of pickling is being done with this, and the manufacture of semiconductors. So, for the semiconductors also the fluorine based reactions are also utilized. So, these starting materials can function as a very good material for making for all these, and therefore, useful for the semiconductor industry as well.

Then we quickly see now that as already we have seen that how aluminum fluoride is forming. So, this aluminum fluoride can be utilized for the formation of all these things. So, raw material for electrolytic manufacture of aluminum that means now you see that Al F 3. Your Al F 3 is nothing but your typical material for aluminum production. If we are able to convert that as alumina from alumina from the bauxite to aluminum trifluoride that means you require to supply the fluorine over there, and that particular one we can get it from your first part itself.

So, now, enrichment the quantity or the amount of aluminum fluoride in a particular material should be enriched such that this can be converted to your cryolite synthetically prepared cryolite, if you are able to make it in industry not that naturally available cryolite which is Na 3 Al F 6. One is one particular process is the Lurgi process. The Lurgi process is from hydrated aluminium oxide that hydrated alumina or the bauxite you use.

And we get it for this particular purpose that since we are considering it as a hydrated aluminum oxide, we can consider it as if it is hydrated, and if you have large amount of water of hydration, what you write, you write the formula of this Al 2 O 3 as Al 2 3 dot 3 H 2 O center dot 3 H 2 O. What is that? We are not able to say it as it is typically aluminum hydroxide the way we get aluminum hydroxide precipitation from a reaction medium even in a test tube. If you have a aluminum solution from alum or aluminum chloride or aluminum sulphate, and add some dilute alkaline, you get immediate precipitation, white precipitate of aluminum hydroxide, gelatinous white precipitate of aluminum hydroxide, so that is typically aluminum hydroxide.

But if you keep that aluminum hydroxide for a longer period you can go for its corresponding removal of water molecules over it, and it can be converted to alumina the formula of it is Al 2 O 3 that means not that each and every aluminum centres will be attached to your hydroxide ion, instead of that you have a network of aluminium centres aluminum as Al 3 plus with oxide ions or the hydroxide ions. So, when these oxide and hydroxide ion giving you a typical network structure, the formula what we will would write is as the 3 water of hydration which is equivalent to that of your alumina plus 3 water.

So, if you take that hydrated aluminum oxide from any other source, there is no need to think of your level of hydration, whether it is a monohydrate, or a dehydrate, or a trihydrate. What you consider is that you take it and heat it from 300 to 400 degree centigrade, and up to that point so all the moisture content will not be there. So, moistures will be removed. Then whatever amount of hydrated alumina you have will be converted to pure Al 2 O 3 that means, water free Al 2 O 3 with 3 water which is being removed from there.

So, the dry Al 2 O 3 what is there in your hand definitely, it will be dried at this particular temperature can be utilized. So, if you make it and you transfer from one chamber to the other, what happens that it is moving from one chamber to the other, but the reaction chamber is a different one now which is kept at a higher temperature and with the introduction of your hydrofluoric acid if in your HF gas is also useful.

So, your Al 2 O 3 will now be attacked by HF at 400 to 600 degree centigrade giving you now simply Al F 3 aluminum trifluoride. And again 3 molecules of water will be removed from there for taking this as the corresponding oxide of the aluminum attached to it as water molecules.

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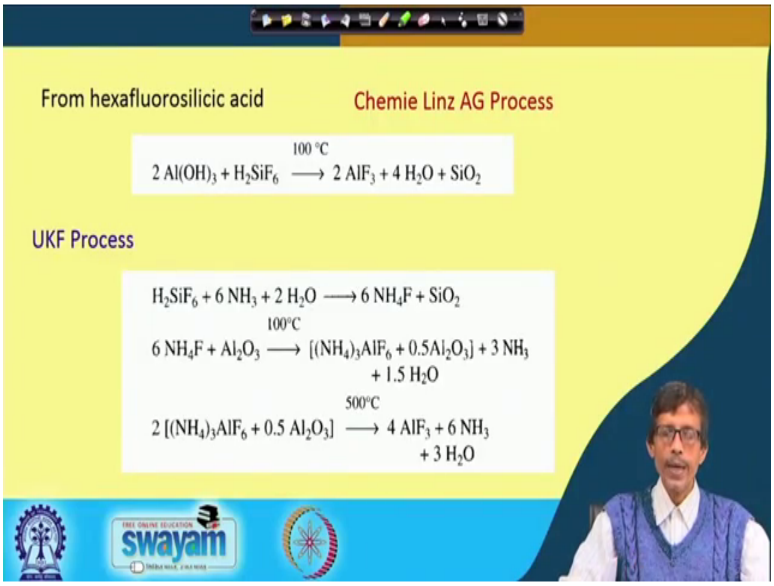

From hexafluorosilicic acid

Chemie Linz AG Process

$$2\,Al(OH)_3 + H_2SiF_6 \xrightarrow{100\ ^\circ C} 2\,AlF_3 + 4\,H_2O + SiO_2$$

UKF Process

$$H_2SiF_6 + 6\,NH_3 + 2\,H_2O \longrightarrow 6\,NH_4F + SiO_2$$

$$6\,NH_4F + Al_2O_3 \xrightarrow{100^\circ C} [(NH_4)_3AlF_6 + 0.5Al_2O_3] + 3\,NH_3 + 1.5\,H_2O$$

$$2\,[(NH_4)_3AlF_6 + 0.5\,Al_2O_3] \xrightarrow{500^\circ C} 4\,AlF_3 + 6\,NH_3 + 3\,H_2O$$

So, you get aluminum trifluoride. Then, that aluminium trifluoride can be converted to your hexafluorosilicic acid also. So, the conversion what we get it as that Al F 3, then you can convert it to your hexafluorosilicic acid corresponding one that means instead of your this thing that you get it as aluminum as your aluminum fluoride, but now you use hexafluorosilicic acid as the material. So, this hexafluorosilicic acid material which is now supplying your fluoride ions, not your HF, not your sodium fluoride, not your ammonium fluoride, as we have seen that very easily we can also make hexafluorosilicic acid.

So, these hexafluorosilicic acid is here now material to attack your dried aluminium, that means, the hydrated aluminum also, that means, just only Al O H 3not that Al 2 O 3. So, since this can also be an acid form. So, this is also these. So, you get it as so along with the production of silicon dioxide at a very low temperature. So, is the Chemie Linz AG process is the name of a German company, so that company, they are producing it through this particular process.

So, very easily basically without maintaining a very high temperature only thing that expensive part of this reaction is your availability of your H 2 Si F 6, so that H 2 Si F 6, what is available over there, you can the raw material, that means, the hydrated alumina you can directly use for your conversion of this typical aluminum hydroxide to your aluminum fluoride. So, you get it. Then another one procedure is can be considered as a very big one or a very elaborate one, but do not be bothered of this all these thing, but only you stepwise you look at it.

So, the single sort I am showing all these three because I have to go quickly little bit at some time, because already I told you all these things, that means, instead of all these things; that means, your hexafluorosilicic acid if you now use it for its reaction with only the ammonia liquod ammonia or aqueous ammonia or ammonia gas. So, with ammonia it is simply giving you ammonium fluoride, that means, ammonia will be converted you have corresponding acidic ion from this part or a hydrogen ion from water molecule giving you ammonium ion. And that ammonium ion will be able to take your fluorides from your hexafluorosilicic acid to making you ammonium fluoride.

So, it is basically some amount of a indirect process and that indirect process is being utilized for the formation of now ammonium fluoride. Earlier what we are using, were using the corresponding direct sodium fluoride or HF or corresponding hexafluorosilicic acid, now you utilize the ammonium fluoride, but it is again we are getting from hexafluorosilicic acid. But all the different processes can have some advantages, and the different companies they are utilizing all these things because these are all industrially trade secret, industrial trade secrets, they are all patented or patent protected.

So, they are basically going through different procedures depending upon the availability the easy availability of the material and their cost. So, once this particular one, that means, hexafluorosilicic acid is converted to ammonium fluoride, then ammonium fluoride is utilized to attack your alumina, that means, your bauxite to giving you something which is basically a salt of ammonium hexa fluora aluminate ammonium hexa fluora aluminate which is basically very similar to that of your cryolite which is nothing but Na 3 Al F 6.

So, instead of Na 3 Al 3 F 6, it is ammonium al F 6, and some amount 0.05 mole of Al 2 O 3 the bauxite. So, some amount of bauxite is still remaining because sometimes the inherent character of that particular the second reaction which is taking place at 100 degree centigrade, the inherent property of this particular reaction is such that whatever amount of ammonium fluoride you increase, you may not go beyond that.

So, it is the nature of that particular reaction, it is controlled by its thermodynamic or catalytic character or the corresponding kinetic character that it will stop there. Basically you cannot remove the entire amount of Al 2 O 3. So, some amount of Al 2 O 3 will still be there and we get that particular one as the raw material and the challenge is that that you have some amount of extra fluorine because you are looking for making aluminum fluoride is only making Al F 3, but the other part which is the similar compound a similarity with that of your cryolyte which has 4, sorry 6 fluoride ions around the aluminum centre. So, you have excess fluoride.

So, if you are able to take that excess fluoride from that particular first part of the unit, that means, N H 4 whole 3 Al F 6 by this Al 2 of the alumina Al 2 O 3, then we will be able to make it as the entire amount of aluminum available as aluminum fluoride. So, the process is that though it is a very simple three step process, but again for this particular material, you increase the temperature to 500 degree centigrade. So, when you increase this temperature to 500 degree centigrade the salt that particular type of double salt type of thing when you have excess alumina in it is basically decomposed at that particular higher temperature. So, high temperature solid state reaction can now give you the corresponding very straight cut conversion of the entire amount of aluminum available in it to aluminum trifluoride.

So, the whole amount of this. So, two of this molecule heated at 500 degree centigrade is converted to aluminum trifluoride. So, Al F 3, and this Al F 3 can remove the entire amount of ammonium as NH 3 and water molecule. So, we have now in your hand the Al F 3. So, next day we just move from here that how we utilize this Al F 3 to some useful purpose whether we go for its conversion to some cryolite formation or any other things such that this fluorine bearing compound can be useful for your industrial purpose.

Thank you very much.