PROCESS FOR PREPARING ANHYDROUS RARE EARTH METAL HALIDES

US Patent Publication No. 2011/0014107

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About Rare Earth Metals Halides

•Anhydrous Rare Earth Metal Halides have wide range of applications such as electrochemical reduction process for the production of metals, reduction of organometallics among others.

• Rare Earth Metals Halides occur in nature as their hydrates and comprises number of molecules of water.

•Hydrates present with rare earth metals adversely impacts the yield and purity of the desired final products.

•Removing water of hydrated rare earth metal salts at ambient temperatures leads to the formation of corresponding oxy halides.

Existing Methods & Their Disadvantages

Many methods of dehydrating rare earth halides are available. But most of them have their own disadvantages as mentioned below.

- **Reduction of rare earth metal oxy halide** present as impurity in anhydrous rare earth halide involves direct chlorination of rare earth metal oxy halide by reaction with carbon and chloride.
  - Disadvantage : Release of environmentally unacceptable chlorinated hydrocarbons and higher temperature used in this process leads to corrosion of reactors and low purity of product.
- **Direct conversion of rare earth oxide using Ammonium Chloride**
  - Disadvantage : Large amounts of Ammonium Chloride required, need to remove excess ammonium chloride, time consuming and low purity of product.

➢**Dehydrating rare earth metal halide by purging** mixture of dry air, hydrogen chloride and chlorine gas through packed bed of rare earth metal halides.

- Disadvantage : Time taking process and also requires large amounts of chlorinating agents.

➢**Preparing anhydrous cerium chloride by vacuum drying** at specified temperatures .

- Disadvantage : Inefficient at industrial scale due to formation of lumps.

Other method are also available but all of them have at least one of below mentioned disadvantage:

- Use of chlorinating agent.
- Requires high temperature
- Release of environmentally unacceptable chlorinated hydrocarbons.

- Time consuming.
- Need of solvent in which salt is soluble
- Lump formation during vacuum drying
- Corrosion problems because of high temperatures.
- Need of additional unit operation for complexation and decomplexation of ammonia and ammoniate .
- Formation of corresponding oxy halides which impacts the activity of rare earth metal halide.

As a conclusion from the above methods and their disadvantage we can say that no method provide an efficient, environment friendly, economical and industrially viable method to prepare anhydrous rare earth metal halides.

Present Method

The proposed method of Preparing anhydrous rare earth metal halide comprises

- Formation of a slurry of rare earth metal halide hydrate in an organic solvent.
- Refluxing the said slurry.
- Distillation of water from the said slurry.
- Rare Earth Metal halide hydrate is cerium (III) chloride heptahydrate and lanthanum chloride heptahydrate.

Advantages of Present Method

- Use of chlorinating agents and drying operation at high temperature not required.
- Corresponding oxy halides are not formed.
- Pure final compound.
- Formed anhydrous rare earth metals halides is efficient for industrial use.

Patent/IP Status

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Indian Application No. 1977/MUM/2008

Extremely Positive International Search Report

APPLICATIONS OF RARE EARTH METAL HALIDES:

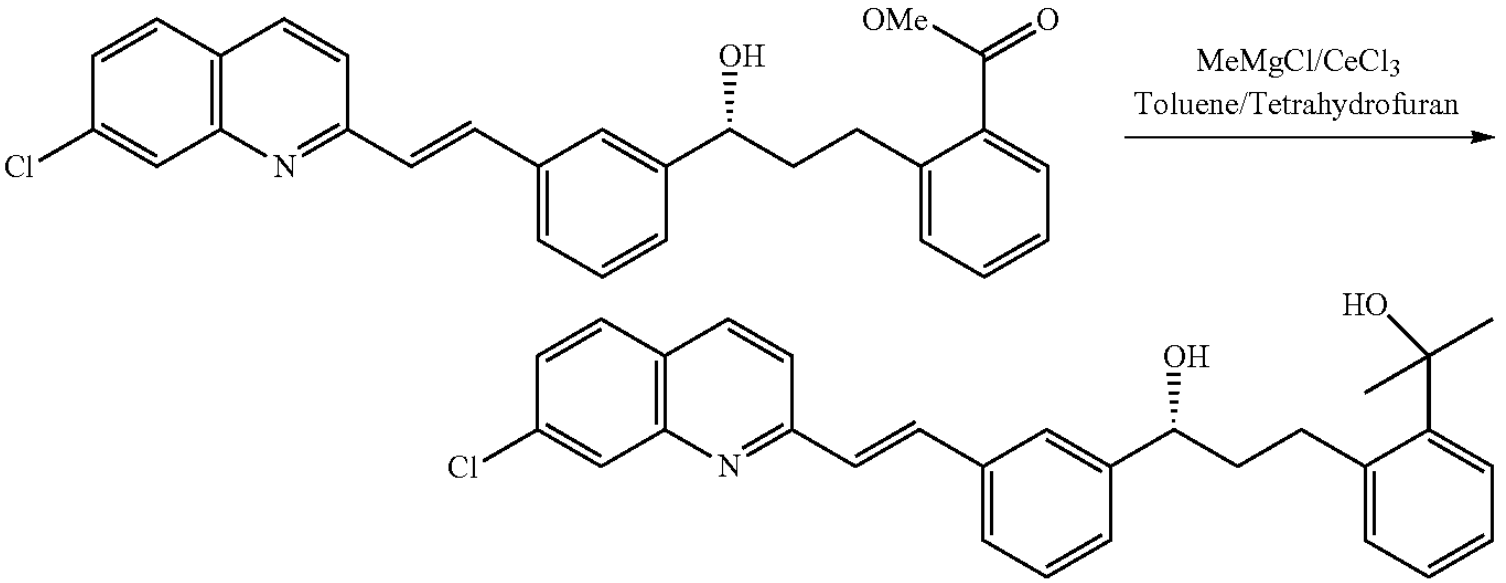
- In the production of metals by electrochemical process.
- As a catalyst in Grignard reaction.

Application of Cerium Chloride

Use of anhydrous $CeCl_3$ for the preparation of organocerium (III) reagent are less basic than organolithium or grignard reagent and react with various carbonyl compounds to give addition products in high yields even though substrates are susceptable to enolisation or metal halogen exchange with simple organo lithiums. For example

- Conversion of (S)-Hydroxy ester in to corresponding alcohol shown by following reaction.

Reaction involving use of Anhydrous Cerium Chloride

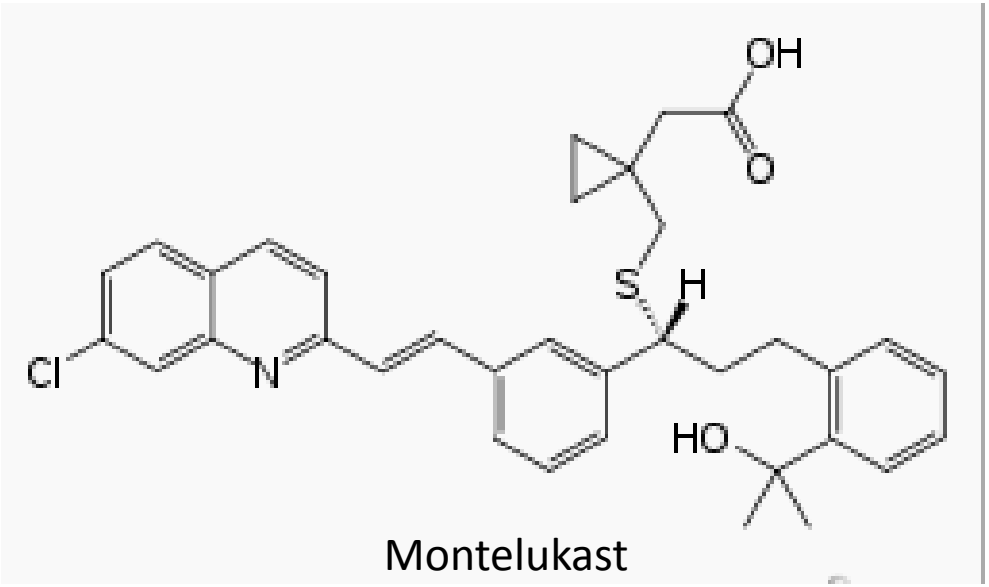


Compound A

- Compound A prepared by using cerium chloride is used as an intermediate in the preparation of **MONTELUKAST**.

About Montelukast

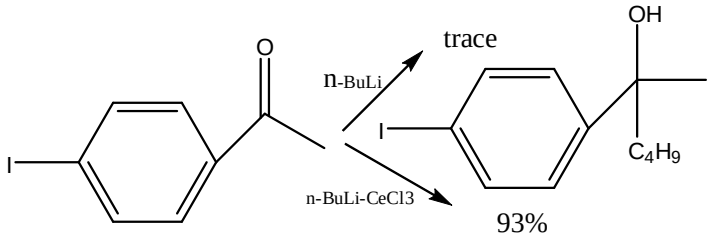
- Montelukast is a leukotriene receptor antagonist (LTRA) used for maintenance treatment of asthma and to relieve symptoms of seasonal allergies.

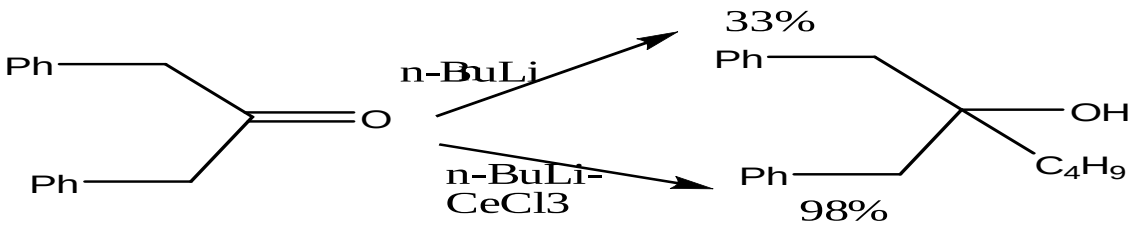


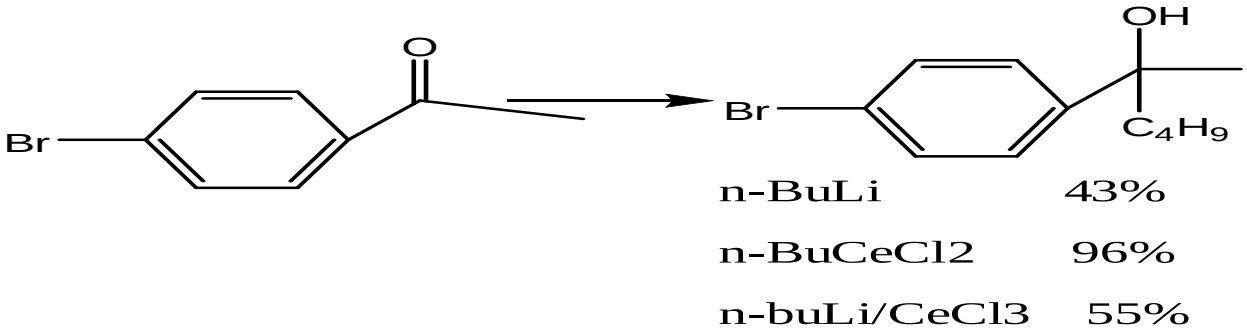
Montelukast

Reactivities of Organolithium compds

- Impact due to enolisation:







Ref.:Pure & Appl.Chem.,Vol.62,pp747-52,1990

COST COMPARISION

- Alfa Aesar (A Johnson Mathey Company)

Cerium Chloride Heptahydrate	50 gm	Rs 1083
Cerium Chloride Anhydrous	10 gm	Rs 2320

EXPECTATIONS:

- Applicant seeks to Out-Licensing the Patent Rights in India and United States on Exclusive or Non-Exclusive Terms

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