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Abstract

This experiment is aim to study the feasibility of electro-chemical catalysis for the reaction of chlorides with unsaturated hydrocarbons.

Due to the development of the industry technology, more and more environmental problems are exploded which at the same time, the concept of green chemistry were born as the increase awareness of the environment protection.

Chlorination, obviously, a reaction in which chlorine atoms are injected into a compound to form chloride is called chlorination, and in organic synthesis there are usually two reactions: displacement chlorination and addition chlorination. In Inorganic Chemistry, the reaction of an element or compound with chlorine is also called chlorination. In metallurgical industry, the extraction of metal with chlorine gas or chloride is also called chlorination, the chlorine gas is excited by High Energy and reacts to form chloride.

However, we can see that normal industrial conditions often require very high temperature or strong light, high energy environment, at the same time, in industry due to the use of a variety of hydrocarbons, it is pretty easy to overheat the reaction, it will cause a combustion reaction, and a lot of raw materials are wasted, which is very uneconomical. Or some heavy metal containing catalysts are used cause the requirements of the reaction, unreasonable treatment of by-products will lead to serious environmental pollution

ELECTROCATALYTIC reactions can be expressed as electron transfer reactions that are facilitated or always occur at the electrode by the presence of modifiers on the electrode surface or in the permanent night phase under the action of a power plant, a class of chemical reactions in which the surface of the electrode or the modifiers in the solution themselves do not change. ELECTROCATALYSIS is a kind of electro-chemical reaction in which the direction and rate of the reaction can be controlled by modifying the electrode material and the electrode potential. The electro-chemical reaction of the electrode material becomes electro-catalysis because it can not be modified. The base electrode of electro-catalysis can only be used as an electron conductor, and can also be used as an electron conductor with catalytic function.

And through Anastas P. T (from the American Institute of Green Chemistry) : 12 Principles

1. Prevention of pollution is better than final treatment.
2. Atomic Economy
3. Try not to use, do not produce toxic substances.
4. Production process moderate (T, P) , low energy consumption
5. Use renewable materials whenever possible.
6. Reduce avoid derivative steps (shield group, protection and repair, temporary change of physical chemistry process)
7. The use of highly selective catalysts is superior to that of stoichiometric reagents

The advantages of ELECTROCATALYTIC REACTIONS:

Clean electrons complete the REDOX, decrease the waste from the source of chemical source.

Energy efficient reaction conditions at high Atom economy

Relatively gentle reaction condition, such as room temperature and atmospheric pressure

The overall energy consumption is low, the voltage between electrodes is very low, which adapt to the requirements of thermodynamics

Easy to control, the speed of the reaction can be mastered by adjusting the current, easy to achieve automatic continuous operation

Small-scale effect, and the reaction is easy to enlarge the production.

Therefore, the use of electro-chemical methods to carry out chlorination reaction is undoubtedly a significant advantage. So, there is no doubt that the chlorination of unsaturated hydrocarbons by the method of electro-chemical has obvious advantages. The main content of this paper is the experiment of electro-catalytic chlorination of unsaturated hydrocarbons.

In this experiment, 6-chlorimidazolo [1,2-b] pyridazine, cerium chloride were used as raw materials, and the products were obtained by changing the conditions of solvent, electrolyte and electrocatalysis.

The solvent is mainly Acetonitrile and a small amount of methanol, the electrolyte is mainly lithium perchlorate, and the base consists mainly of magnesium chloride hexahydrate. In the presence of nitrogen, a derivative of 6-chlorimidazolo

[1,2-b] pyridazine was obtained by dehydrogenation. Reaction has many advantages, for instance, environmental friendly, which in line with the concept of green chemistry aforementioned, and high efficiency, which means that it can be used in industry. As far as the reaction is concerned, the catalysts, bases, electrolytes, solvents and so on are all obtained through screening, and the reaction conditions are the most suitable ones after many experiments.

The results show that the chlorination of unsaturated hydrocarbons can be carried out by Electrocatalysis, which proves that the synthesis of unsaturated hydrocarbons is efficient and environmental friendly.

Chapter 1 introduction

1.1 Background

Since the Chemistry was born, we have achieved the agreement that experiment and study are the significant usage of nowadays research. People try to obtain the more exactness data by lab working, data collecting, and observing the direct phenomenon and repeating the experiment. It can be said that the experiment is one of the most important study methods of the chemical learning. Especially, The research and development of chlorination synthesis mentioned in this paper. The experiment has the advantages of intuitionistic, repeatable and effective, but it also has the disadvantages of pollution.

While chemistry created wealth for the mankind, but it also brings danger. It is well known that the development of each since always filled with exploration and progress, due to the uncertainty, chemists will synthesize some unknown substance by chance and they can not avoid. The only thing to do is try to apply and do the research on the new substance in a long term to try to figure out the quality of it, however during the researching time, the compounds had already make some influence on the environment and the human life.

The traditional chemical industry do make a great contribution to the environmental pollution, and at present, the deleterious substance produced every year can be 3 billions to 4 billions, which will pollute the environment and threatening the every existence of human race. The strict reality forced every country to find a way to protect the environment and build an sustainable industry. Is it possible for the chemical industry to produce the environmental harmless products? Or even some crafts without the by-product? The concept of the green chemistry was put forward in America which is barely developed due to the chemical industry. In 1990, the United States passed a act named "pollution prevention action" . After 1991, "Green Chemistry" was proposed by the American Chemical Society (ACS) and became the central slogan of the United States Environmental Protection Agency.

In the recent years, with the enhance of the awareness of the environment protection, mankind realize that the environment protection and the pollution governance are non negligible, and the green chemistry was born under this background.

The Green Chemistry can also be called environmental friendly chemistry or friendly environmental chemistry, which aim at decrease the usage of the harm instance and the hazard compounds. The green chemistry which includes the organic synthesis and analytical synthesis and so on is mainly promote the decreasing usage of the harm chemical technology and raw material so that to achieve the target of environment protection, pollution diminishing and human health protection. The green chemistry is originated from the USA. Although the EPA in 1984 pointed to the idea of "waste minimization, " the goal is to achieve green chemistry by minimizing waste. However, waste minimization does not fully encapsulate the concept of Green Chemistry. It's just one direction, one branch. In recent years, many people have proposed Green Chemistry and worked hard in this field. As an efficient, non-toxic, harmless and pollution-free renewable energy, electric power has been used more and more widely. The use of electrocatalysis is a means of conforming to the concept of green chemistry. Before we carried out the alkylation of unsaturated compounds, a large number of excellent scientific researchers have carried out related experiments. The reason for this experiment is that as an excellent intermediate, 6-chlorimidazo [1,2-b] pyridazine needs to be synthesized in the simplest and most effective way.

In addition to electrocatalysis, there are some obvious problems in other synthetic methods, such as the use of heavy metal catalysts, or not suitable for large-scale production, too low yield, harsh reaction conditions and so on.

1.2 Electrochemical Overview and Development

In 1663, German physicist Otto von Guericke created the first generator that generate static electricity through friction in the machine. The generator puts a large sulfur ball into a glass ball and fixed to a shaft. Turning the sphere by shaking the crankshaft, electrostatic sparks occur when a liner develops friction with a rotating ball. The sphere can be disassembled and can be used as a source of electrical testing. In the middle of the 17th century, French chemist, Charles François de Cisternay du Fay has found two different electrostatic types, Different charges are mutually exclusive and different charges are mutually attractive. Du Fay said that electricity consists of two different liquids: "vitreous" (Latin "glass"), Or positive electricity; And the "resinous", Or a negative power. This was the double-liquid theory of electricity, which was denied by the single-liquid theory of Benjamin Franklin in

the late 17th century. Charles in 1781. Charles-Augustin de Coulomb developed the law of electrostatic suction in his attempt to study the law of charge repulsion proposed by British scientist Joseph Priestley, In 1791 Galvani published "animal electricity" where metal retracted the frog leg muscles, generally regarded as the origin of electrochemistry. In 1799 Volta invented a "reactor" composed of different metal sheets with wet paper, now called "Voltpile". This is the young type of chemical power supply. Prior to the invention of the DC motor, various chemical power supplies were the only ones that provided a constant steady current. The discovery of Faraday's Law of Electrolysis in 1834 laid the quantitative foundation for electrochemistry. In the second half of the 19th century, Helmholtz and Gibbs Jobs, Battery "starting power" (now called "electric potential") with a clear thermodynamic meaning; In 1889 Nernst derived by thermodynamics the relation between the material concentration and the electrode potential involved in the electrode reaction, That is, the famous Nernst formula; In 1923 Debye and Hückel proposed the widely accepted theory of strong electrolyte dilute solution, greatly promoted the development of electrochemistry in theoretical exploration and experimental methods, after the 1940 s, Application and development of electrochemical transient technology, the combination of electrochemical methods and optical and surface technologies, Enables one to study rapid and complex electrode reactions, Information on the molecules on the electrode interface can be provided. Electrochemistry has always been an active branch of physical chemistry. Its development and the development of solid physics, catalysis, and life science. Electrochemistry is the only discipline based on large industry. Its application mainly includes: electrolytic industry, in which chlorine-alkali industry is second only to inorganic base of synthetic ammonia and sulfate; smelting aluminum and sodium, copper and zinc refining is electrolysis; surface finishing of parts, electro dialysis can remove cyanide ions and chromium ions; chemical power supply; metal corrosion prevention, most metal corrosion is electrochemical corrosion; many life phenomena such as muscle movement and nerve information transmission involve electrochemical mechanism. Various electrochemical analysis methods developed by applying the electrochemistry principle have become an indispensable means of laboratory and industrial monitoring.

With the progress of science and technology, electrochemistry has become a very popular method of synthesis in today's society, although there are still many

people do not understand the advantages of electrochemical synthesis, or even do not believe in electrochemical synthesis. Electrochemistry is the study of the charged interface between two kinds of conductors and the changes that take place on it. The interaction between electricity and chemistry can be accomplished by a battery or by a high-voltage electrostatic discharge (such as the conversion of oxygen to ozone through a silent discharge tube) , collectively known as electrochemistry, a branch of electrochemistry known as discharge chemistry. As a result of discharge chemistry has a special name, therefore, electrochemistry often refers specifically to "the science of batteries" . Although many chemists have long recognized that electrochemistry can catalyze the halogenation of some organic compounds, the use of electrochemistry in complex molecular synthesis is relatively rare. In the past, few people have used technical terms like Cyclic voltammetry, reduction potential, and current density in synthesis. Electrochemical synthesis has been developed rapidly in the direction of synthesis, although different from the conventional Kecheng, but electrochemical synthesis as a conventional technology has aroused the interest of some synthetic groups. Although over the years electrochemical synthetic methods have generally been considered after all other methods have been exhausted, the literature on the use of electrochemical methods has grown explosively.

Electrochemistry can be divided into oxidation reactions and reduction reactions, which include substrates, electrodes, electrolytes, solvents, and various additives, oxidation at the prototype of the battery, reduction at the Cathode, many of the ionization reactions are very strange, because they occur on the surface of the electrode, they produce highly reactive intermediates such as free radical cations and free radical anions. Electrolytes occur in electrochemical reactors, called batteries for short. The term battery is used to describe any device that includes an electrically active substance, a solvent, an electrolyte, and at least two electrodes. A wide range of solvents are available, including methanol, Acetonitrile, dichloromethane, Oxolane, and many of the currently unused electrolytes, Acetonitrile, are widely used in electrochemistry, including, in general, lithium perchlorate, tetrabutylammonium, tetrafluoroborate, tetraethylammonium toluene sulfonate, tetrabutylammonium Acetate, or other tetraalkylammonium salts. As long as the electrolyte is stable under certain conditions, the electrode can be made from any material that allows electron transfer and exists in the solution, usually platinum, platinum wire, Carbon Rod, magnesium, stainless steel or reticulated glassy carbon (RVC) . RVC is a unique type

of glassy carbon that combines the properties of carbon and glass. Its High Pore efficiency, high specific surface area and chemical resistance make it a powerful choice for electrochemical conversion. In addition, many electrochemical reactions use sacrificial electrodes, which are usually made of metals such as lead or zinc, and are consumed in the reaction. Some reactions are sensitive to the current density on the surface of the electrode, in which case the electrode provides an easy way to determine the surface area, such as commonly used carbon rods or steel plates. As with any chemical reaction, the selection of solvents, electrolytes, electrodes, and current densities often needs to be optimized because these parameters have a significant effect on the result of the projection.

An important feature of electrochemical reactions is that we can tightly control the reaction site in the presence of an over functional group, thus affecting the selectivity of the redox. Controlled potential experiments will allow selective oxidation or reduction if the point must be reached or to hell with only one in a particular electrochemical oxidation or reduction process. The potential of the battery can be set to a given value relative to the reference electrode, and only those electrically active substances at the hell set point can participate in the oxidation or reduction reaction. Since the point position is set relative to the reference electrode, a third electrode is required as a reference electrode in addition to the anode and Cathode. While it doesn't take much work to build a reference electrode, many are commercially available and reusable at relatively low cost. The Standard Reference electrode keeps the electrode surface point at a set value, the controlled point experiment allows selective oxidation or reduction, however, the consumption of the electroactive substance reduces the current when the current drops, it takes longer to oxidize or reduce the material. Therefore, electrochemical research is mainly focused on two aspects, one is the study of the electrode, including the equilibrium characteristics of the electrode and the strength of the polarization characteristics, in other words, the electrochemical behavior of the interface between the electrode and the electrolyte, electroshock therapy and electrode research will involve Chemical thermodynamics, chemical kinetics, and structural materials.

1.3 Electrochemical and Chlorination applications

Electrochemistry is the study of the relationship between electricity and chemical reactions. The interaction between electricity and chemistry can be accomplished by a battery or by a high-voltage electrostatic discharge (such as the conversion of oxygen to ozone through a silent discharge tube) , collectively known as electrochemistry, a branch of electrochemistry known as discharge chemistry. As the discharge chemistry has a special name, therefore, electrochemistry often refers specifically to the science of the battery.

Electrolytic Industry, the chlor-alkali industry is the inorganic basic industry next to synthetic ammonia and sulfuric acid, the intermediate monomer adiponitrile of Nylon 66 is synthesized by Electrolysis, and the smelting of light metals such as aluminum and sodium, copper, zinc refining are also used in Electrolysis, 2, the mechanical industry to use electroplating, electropolishing, electrophoretic coating to complete the surface finishing parts; 3, the environmental protection may use the electro dialysis method to remove the cyanide ion, the chromium ion and so on pollutant 4, the Chemical Power Source 5, the metal anti-corrosion question, most metal corrosion question is the electrochemical corrosion question; 6. Many vital phenomena, such as muscle movement and nerve transmission, involve electrochemical mechanisms 7. Various Electrochemical analysis methods developed using electrochemical principles have become indispensable tools for laboratory and industrial monitoring

Chlorination generally refers to the introduction of chlorine into compounds. In organic chemical reactions, chlorination generally includes displacement chlorination, addition chlorination and oxidation chlorination; in metallurgical industry, the use of chlorine gas or chloride to extract certain metals is also called chlorination; The process of adding chlorine or chlorine-containing oxides to water for the purposes of oxidation and disinfection is also called chlorination.

Chlorination can be used in the following ways: Thermal chlorination: the use of heat to stimulate the chlorine gas, the formation of free radicals, and then with hydrocarbon molecules to produce chloride compounds. Photoperiod: photoperiod can excite chlorine gas, generate free radicals, and then react with hydrocarbon molecules to produce chlorine compounds. UV is often used as light source. Catalytic

chlorination: The chlorination reaction using catalysts, divided into homogeneous catalysis and heterogeneous catalysis. Example of industrial induction: Thermal chlorination of Methane: Reaction Mechanism for Y_0 and chain reaction, the product is the product of four chloromethane, product composition and temperature-related, mainly depends on the proportion of chlorine and methane. Synthesis of propylene by Thermal chlorination-gas-phase Synthesis of vinyl chloride, etc.

The mixer is the key equipment of the gas-solid catalytic chlorination reaction, the mixer, the speed of vaporization raw material mixing, the mixing, the uniformity of the mixing directly affects the selectivity of the chlorination reaction and the conversion rate, the service life of the catalyst, the quality of the product. This paper introduces a new gas-gas Rapid Mixing technology—a Turbine Jet Mixer, which makes use of the flow characteristics of cross-flow jets to form two streams of gas, good principal diffusion and turbulent diffusion, to facilitate the rapid mixing of the two gases. In this paper, the concentration distribution in the mixer with different nozzle and different operating conditions is measured by using tracer method, and various parameters and momentum ratio are analyzed. Effects of the diameter, area and ratio of length to diameter of the opening on the mixing effect of the mixer. The results show that this mixer can improve the range of application of momentum and ratio, and also can get better mixing efficiency under the condition of lower momentum and ratio

The chlorination time can be greatly shortened and the reaction temperature reduced by using the chemical catalysis method. The reason is that when the catalyst is added, the activation energy of the reaction is reduced, so that the activation temperature is lowered and the reaction activity is increased. Although photochlorination can also shorten the reaction time, it does not decrease the reaction temperature. No matter what chlorination method is used in the experiment, a sudden rise in temperature occurs when the temperature reaches $56\text{ }^{\circ}\text{C}$, indicating that at that temperature active chlorine atoms are formed and begin to replace the hydrogen in the alkanes, this temperature is therefore the activation temperature of the reaction.

Chapter 2 Induction of Electro-catalytical Chlorination

2.1 Introduction

In this experiment, 6-chlorimidazolo [1,2-b] pyridazine and cerium chloride were used to synthesize 6-chlorimidazolo [1,2-b] pyridazine chloride derivatives. 6-chlorimidazolo [1,2-b] pyridazine is a pyridazine compound. Pyridazine is a kind of compound containing nitrogen heterocycles, which is an important intermediate in chemical industry. pyridazine compounds have various biological activities, such as INSECTICIDAL, bactericidal, antiviral, etc. , can Be used as organic liquid crystals, some pyridazine derivatives or important dye intermediates. 3-nitro-6-chlorimidazolo [1,2-b] pyridazine is an important intermediate for the synthesis of CEPHALOSPORINS. 6-chlorimidazolo [1,2-b] pyridazine can be used to prepare 3-nitro-6-chlorimidazolo [1,2-b] pyridazine. The operation is as follows: 1,000 ml single-port round-bottomed flasks containing 3-amino-6-chlorpyridazine (51.82 G, 400 MMOL) , 40% chloro-acetaldehyde aqueous solution (109.9 G, 560 MMOL) , Sodium bicarbonate (30.2 G, 360 MMOL) and 350 ml (275.1 g) acetonitrile were Magnetic stirrer, the mixture in the reaction flask was stirred at 80 ° C for 6.5 hours. After the reaction of 3-amino-6-chlorpyridazine was finished, nitric acid (40.32 G, 640 MMOL) was added and stirred for 4 hours, the intermediate 6-chlorimidazolo [1,2-b] pyridazine was determined by TLC and GC. The pure product 3-nitro-6-chlorimidazolo [1,2-b] pyridazine was obtained by recrystallization with N-hexane = 1:3. The filtrate was extracted with Ethyl Acetate, the extractant was removed by rotary distillation, and the crude product was obtained, the pure product 3-nitro-6-chlorimidazolo [1,2-b] pyridazine was obtained by recrystallization with N-hexane = 1:3. After drying, the yield was 81.70% and the purity was 99.42% (HPLC)

2.2 Experimental Section

2.2.1 Experimental raw materials

Figure2.1Raw material specifications and sources

name	formul	SP	source	amount
a		EC		

6-Chloromidazo o[1,2-b]pyridazine	C ₆ H ₄ C IN ₃	AR	Shanghai	Mylar	30.7 mg
			Chemical Technology Co., Ltd.		
Cerium trichloride	CeCl ₃	AR	Tianjin	Fuyu Fine	1.5 mg
			Chemical Co., Ltd.		
Magnesium chloride hexahydrate	MgCl ₂ • 6H 2O	AR	Shanghai	McLean	120 mg
			Biochemistry Co., Ltd.		
Perchlorate	LiClO ₄	AR	Shandong	West Asia	63.6 mg
			Chemical Co., Ltd.		
Acetonitrile	HCN	AR			6 ml
Methanol	CH ₄ O	AR			0.5 ml

2.2.2 Experimental Instruments

Figure 2.2 Instrument model and source

name	specification	source
Intelligent mixer	magnetic ZNCL-BS	Gongyi City to China Instrument Co., Ltd.
Potentiometer	DJS-292B	Shandong Haorui Instrument Co., Ltd.
Recirculating vacuum pump	water SHZ-D	Gongyi City to China Instrument Co., Ltd.
Electronic balance	FA2204N	Shanghai Haohai Instrument Co., Ltd.
Electric blast drying oven	thermostatic DHG-9070A	Shanghai Jinghong Experimental Equipment Co., Ltd.
Ultrasonic cleaner	KQ-100B	Kunshan Ultrasonic

		Instrument Co., Ltd.		
Portable ultraviolet analyzer	WFH-204BS	Hangzhou instrument Co, Ltd	qiweiwei	
Rotary evaporator	YRE-2000B	Gongyi City Instrument Co., Ltd.	China	
Constant temperature water bath	12201C	Gongyi Yingying Instrument Factory	Gaoke	
CNC ultrasonic cleaner	KQ3200DB	Kunshan instrument Co, Ltd	Ultrasonic	
Platinum electrode	10*10*0.2mm	Shanghai yueci Technology Co,Ltd	Electronic	

2.2.3 Introduction of the Electro-catalytical Chlorination

1 weigh all the solid drugs needed, such as substrates and electrolytes, place them in a reactor, place them in magnetons, and add the solvent using a pipette gun.

2 seal the reaction vessel and fill it with nitrogen as a protective gas.

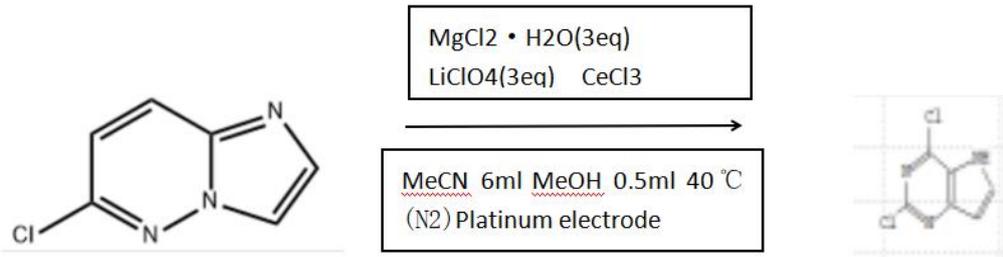
3 set up the experiment, put the reaction vessel under the suitable temperature and the reaction condition, use the potentiometer to carry on the electrocatalysis reaction, wait for the suitable time to carry on the result analysis.

4 At the end of the reaction time, the product of the reaction was taken out for the following experimental measurement.

5 The Reaction Product is injected into the column Chromatograph, the column chromatograph is ready to be carried out,

6 Through the column Chromatograph, we can get the corresponding product, and carry on the spin distillation to the product, pay attention to the flask used to carry on the weighing before the spin distillation.

7, through the Spin steaming finally we get the product, again to the flask weighing, by calculating the product quality and yield.



Electrode:platinum electrode

Speed: 400r/min

Chapter III Conclusions

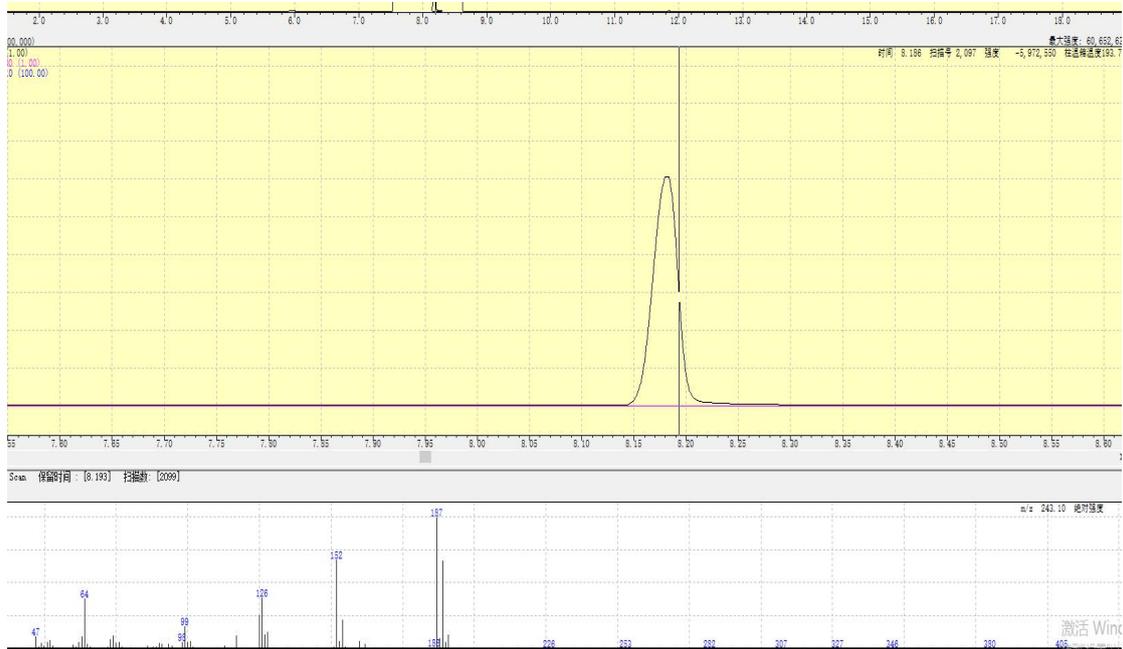


Chart 1

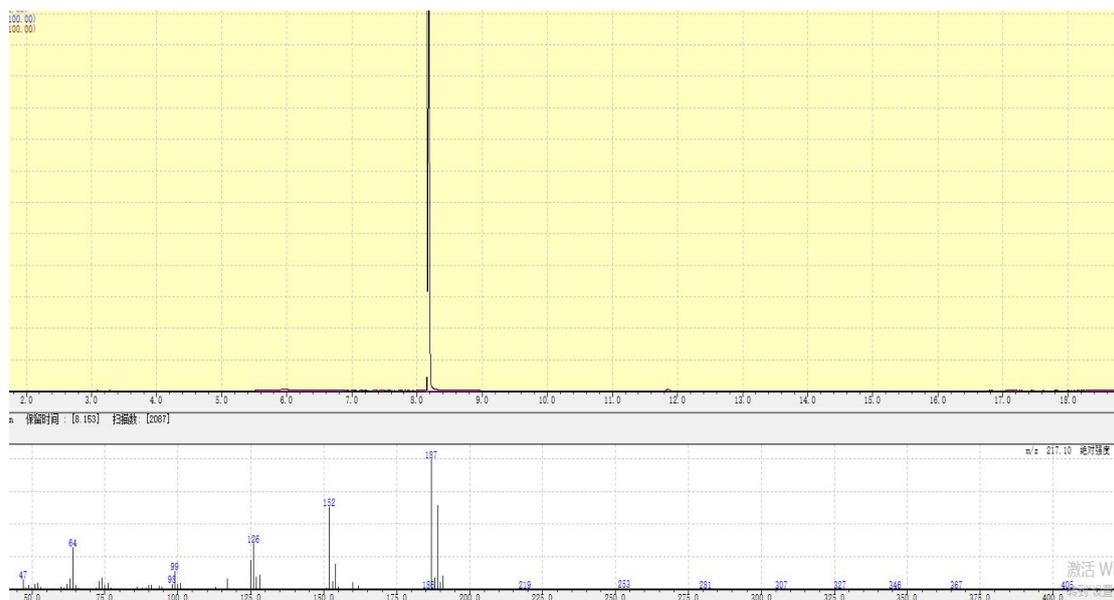


Chart 2

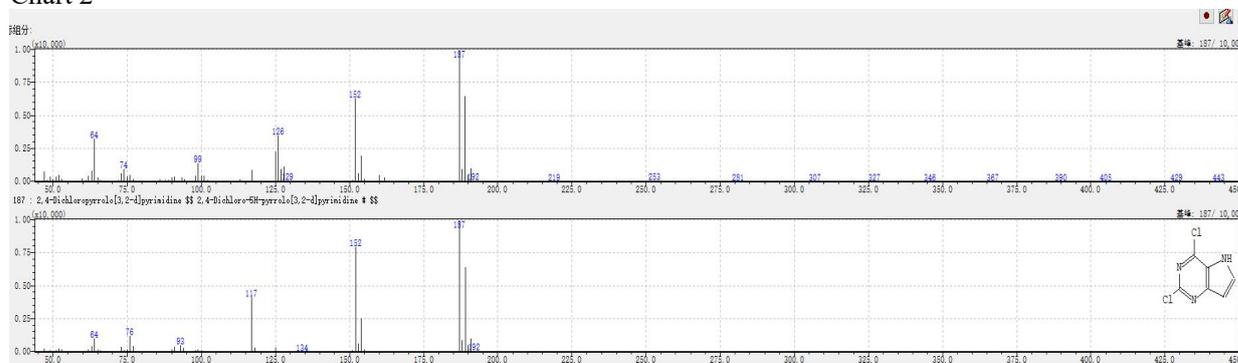


Chart 3

We can see from the chart3,the product is the product we need.In short ,we obtain the target product by the way of Electro-catalytical Chlorination

After many reactions and measurements, we took a variety of experimental times to compare, and by comparing five hours, seven hours, and twelve hours of pulling, we found that although the reactions were carried out differently, but the target product is the same, the time difference affects our production rate, does not have much influence on the final product, through the repeated data measurement, our production rate can basically be stable at around sixty-five per cent, at its peak, it can be as high as 70 per cent, and after a later period of improvement, we reduced the concentration of cerium trichloride by a fraction, and the yield was also relatively reduced, you can see that cerium trichloride has an objective effect on the output of the product, and when you adjust the CERIUM trichloride to 10 percent, the yield can be as high as seventy-seven percent. Of course, during the reaction process, we also noticed some uncertainties. For example, during the

reaction spin evaporation, there will be some subtle changes in the color of the product, the quantity of products separated by column Chromatography has a relatively large gap, but does not affect the yield and so on.

Bibliography

- [1] (a) H. J. Schaeffer, *Top. Curr. Chem.*, 1990, 152, 91; (b) T. Shono, *Top. Curr. Chem.*, 1988, 148, 131; (c) K. D. Moeller, *Tetrahedron*, 2000, 56, 9527; (d) R. D. Little and K. D. Moeller, *Electrochem. Soc. Interface*, 2002, 11, 4, 28; (e) M. M. Baizer and H. Lund, *Organic Electrochemistry: An Introduction and a Guide*, 2nd Edn, Dekker, New York, NY, 1983.
- [2] A. J. Fry, *Synthetic Organic Electrochemistry*, Harper & Row, New York, NY, 1972.
- [3] K. Yoshida, *Electrooxidation in Organic Chemistry*, John Wiley & Sons, Inc, New York, NY, 1984.
- [4] M. R. Rifi and F. H. Covitz, *Introduction to Organic Electrochemistry* Marcel Dekker, Inc New York, NY, 1974.
- [5] H. Lund and O. Hammerich, *Organic Electrochemistry*, 4th Edn, Marcel Dekker, Inc., New York, NY, 2001.
- [6] A. J. Bard and L. K. Faulkner, *Electrochemical Methods: Fundamentals Applications*, 2nd Edn, John Wiley & Sons, New York, 2001.
- [7] RVC is commercially available from a number of sources. See references that utilize RVC for specific commercial sources.
- [8] All substances listed as being commercially available can be purchased through either Bioanalytical Systems, Inc. (BAS <http://www.bioanalytical.com>) or through The Electrosynthesis Co., Inc. (<http://www.electrosynthesis.com>).
- [9] K. H. Leavell and E. S. Lewis, *J. Org. Chem.*, 1972, 37, 916.
- [10] T. Hudlicky, C. D. Claeboe, L. E. Brammer, Jr., L. Koroniak, G. Butora and I. Ghiviriga, *J. Org. Chem.*, 1999, 64, 4909.
- [11] M. Durandetti, J.-Y. Ne' de' lec and J. Pe' richon, *Org. Lett.*, 2001, 3, 2073
- [12] (a) B. Batanero and F. Barba, *Org. Lett.*, 2005, 7, 2567; (b) J. M. Saveant, *Acc. Chem. Res.*, 1993, 26, 455 - 61.
- [13] J. D. Parrish and R. D. Little, *Tetrahedron Lett.*, 2001, 42, 7371.
- [14] H.-J. Schaeffer and T. Pienemann, *Synthesis*, 1987, 1005.
- [15] T. Shono, H. Masuda, H. Murase, M. Shimomura and S. Kashimura, *J. Org. Chem.*, 1992, 57, 1061.
- [16] H. Maeda, T. Maki and H. Ohmori, *Tetrahedron Lett.*, 1992, 33, 1347.
- [17] H. Maeda, T. Maki, H. Ashie and H. Ohmori, *J. Chem. Soc., Chem. Commun.*, 1995, 871.
- [18] R. D. Little, D. P. Fox, L. Van Hijfte, R. Dannecker, G. Sowell, R. L. Wolin, L. Moens and M. M. Baizer, *J. Org. Chem.*, 1988, 53, 2287.
- [19] C. G. Sowell, R. L. Wolin and R. D. Little, *Tetrahedron Lett.*, 1990, 31, 485.
- [20] E. Eru, G. E. Hawkes, J. H. P. Utley and P. B. Wyatt, *Tetrahedron*, 1995, 51, 3033.
- [21] P. G. M. Wuts and C. Sutherland, *Tetrahedron Lett.*, 1982, 23, 3987.
- [22] P. L. Wong and K. D. Moeller, *J. Am. Chem. Soc.*, 1993, 115, 11434.

- [23] J. E. Leonard, P. C. Scholl, T. P. Steckel, S. E. Lentsch and M.R. Van De Mark, *Tetrahedron Lett.*, 1980, 21, 4695.
- [24] T. Shono, T. Soejima, K. Takigawa, Y. Yamaguchi, H. Maekawa and S. Kashimura, *Tetrahedron Lett.*, 1994, 35, 4161.
- [25] S. H. K. Reddy, K. Chiba, Y. Sun and K. D. Moeller, *Tetrahedron*, 2001, 57, 5183.
- [26] B. Liu, S. Duan, A. C. Sutterer and K. D. Moeller, *J. Am. Chem. Soc.*, 2002, 124, 10101.
- [27] J. S. Swenton, A. Callinan, Y. Chen, J. J. Rohde, M. L. Kerns and G. W. Morrow, *J. Org. Chem.*, 1996, 61, 1267.
- [28] A. Matzeit, H.-J. Schäfer and C. Amatore, *Synthesis*, 1995, 1432 and all references contained therein.
- [29] S. Duan and K. D. Moeller, *Org. Lett.*, 2001, 3, 2685 and all references contained therein.
- [30] D. A. Frey, N. Wu and K. D. Moeller, *Tetrahedron Lett.*, 1996, 37, 8317.
- [31] T. Siu and A. K. Yudin, *J. Am. Chem. Soc.*, 2002, 124, 530.
- [32] (a) J. B. Sperry, C. R. Whitehead, I. Ghiviriga, R. M. Walczak and D. L. Wright, *J. Org. Chem.*, 2004, 69, 3726; (b) J. B. Sperry and D. L. Wright, *J. Am. Chem. Soc.*, 2005, 127, 8034.
- [33] J. Mihelcic and K. D. Moeller, *J. Am. Chem. Soc.*, 2004, 126, 9106.
- [34] Y. Yanagisawa, Y. Kashiwagi, F. Kurashima, J. Anzai, T. Osa and J. M. Bobbitt, *Chem. Lett.*, 1996, 1043 and all references contained therein.
- [35] 王宏力,杨晓刚,孟祥平,李敏,郭歌,王启翰.C_(12~24)正构烷烃的催化氯化反应研究[J].
现代化工,2011,31(01):37-39.
- [36] 恽魁宏. 有机化学 北京: 高等教育出版社 1982: 86-103.
- [37] 李述文. 实用有机化学手册 [M]. 上海: 上海科学技术出版社
- [38] 李本祥,王良芥,罗和安. 气固催化氯化反应混合器混合特性研究[A]. 中国化工学会、化学工程专业委员会、生物化工专业委员会.第一届全国化学工程与生物化工年会论文摘要集(上)[C].中国化工学会、化学工程专业委员会、生物化工专业委员会:中国化工学会,2004:1.

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