





中国现代自然科学家选集丛书

侯德榜选集

中国南方图书公司北京编辑部 组编

李祉川 选 陈歆文 编



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加的损失，25%以上的盐被浪费掉。结果，现存的氨碱厂都有价钱便宜、数量充足的原盐供应，由于这种情况，从蒸馏废液中回收盐往往是不合算的。可是，这样回收的盐纯度很高，可与采用纯碱-石灰法精制与水制取的盐相比美。回收盐是和制取氯化钙连在一起。

我们将讨论几种从蒸馏废液中已经工业规模生产的几个主要产品，即再制盐、氯化钙、硫酸钙和氯化铵。

再制盐和氯化钙：

氨碱生产希望蒸馏废液的体积越小越好，特别是废液中某些物质要进一步加工时更是这样。要做到这一点，碳化塔内的分解率要尽可能高，以使滤液的体积尽可能小；在保证纯碱中的盐分在允许范围内的前提下，过滤机洗水要尽可能少；应用含CaO每升250克（比重1.21）的浓石灰乳，或在预灰桶内加固体石灰进行蒸馏。含有少量过量游离石灰的液体，通入一些废CO₂气进行碳化。通过澄清，沉淀出的CaCO₃和液体中的全部固体物质被分离掉。含有CaCl₂、NaCl及少量溶解的CaSO₄的清液，在三效蒸发器中浓缩，直到NaCl及少量的CaSO₄开始析出结晶。盐的结晶在离心机中滤干，往盐的滤饼上喷少量的水，以洗掉盐中的CaCl₂。从离心机下来的盐，不需进一步干燥即可直接用桶包装。

液体的蒸发一直要进行到未效蒸发器中的液体的比重达到42Be'。在这样的情况下，差不多全部盐可以析出。加入一点“氯化石灰”将铁氧化，再进行澄清。澄清液在铸铁锅中进一步浓缩，直到熔融物质冷却时凝固，得到称为75%氯化钙的产品，对应的分子式是CaCl₂·2H₂O。这个产品被舀进铁桶中，每桶约装600磅，铁桶要密封。这个产品的典型成分如表9所示：

75%CaCl₂的成分(%)

表 9

CaCl ₂	73.6	不溶物	0.08
NaCl	1.4	水	24.92

制取无水CaCl₂，并经常和HCl。HCl气95%的氯化钙。

做衬里的木桶，

氯化钙溶液75%的固体氯化钙用作碳化钙的温度下反应。

硫酸钙：

晶态CaSO₄

(Crown Filler)

氨塔废液沉降，游离石灰，即碱性，可再加一些机过滤，再用高盐。所得石膏晶样。硫酸钙用作

尚在190℃以做模型、墙粉，则水分全部逸出，含有微量的铁质，制造某种牙膏和

除去沉淀的到制碱工厂制成

氯化铵：

氯化铵的制CO₂气作用。为器中将溶液浓缩被过滤掉，氯化钙

，现存的氨碱厂都有价
情况。从蒸馏废液中回
盐纯度很高，可与采用
回收盐是和制取氯化钙

业规模生产的几个主要
铵。

好，特别是废液中某些
一点，碳化塔内的分解
；在保证纯碱中的盐分
可能少；应用含CaO每
预灰桶内加固体石灰进
通入一些废CO₂气进行
体中的全部固体物质被
的CaSO₄的清液，在三
SO₄开始析出结晶。盐
少量的水，以洗掉盐中
步干燥即可直接用桶包

中的液体的比重达到
可以析出。加入一点
清液在铸铁锅中进一步
为75%氯化钙的产品，
被舀进铁桶中，每桶约
成分如表9所示：

表 9

物	0.08
	24.92

制取无水CaCl₂，是将75%的CaCl₂在反射炉或搅动炉中进行加热，并经常把动熔融的物质，直到部分CaCl₂分解生成CaO和HCl。HCl气体逸出，剩下的是熔化的多孔的CaCl₂，称为95%的氯化钙。冷却后的无水氯化钙，稍带荧光，装到铁桶或纸做衬里的木桶，并密封。

氯化钙溶液，在制冰过程中用作冷冻剂，亦可作为抗冻剂；75%的固体氯化钙用作防尘剂，来减少公路上的灰尘。而无水氯化钙用作碳化钙和氮气反应制取氰氨化钙的催化剂，可以在较低的温度下反应。

硫酸钙

晶态CaSO₄·2H₂O在索尔维法产品中称为“上等填料”(Crown Filler)，远较天然石膏更为纯净。制造过程包括将蒸氨塔废液沉降，将澄清液趁热用盐饼处理。溶液中所含过量的游离石灰，即被盐饼中微量的硫酸所中和。如果所得溶液稍呈酸性，可再加一些石灰乳。沉淀的CaSO₄·2H₂O用真空转鼓过滤机过滤，再用离心机脱水，并在离心机上淋少量水，以洗去可溶盐。所得石膏晶体用低温加以干燥，大致情形象干燥碳酸氢钠一样。硫酸钙用作造纸的填充料。

倘在190℃以下干燥，便得到煅石膏CaSO₄·1/2H₂O，可供做模型、墙粉，并在建筑业有多种用途。倘在200℃以上煅烧，则水分全部逸出，但必须小心勿使硫酸盐还原成硫化物。倘其中含有微量的铁质，煅烧后被染色，呈黄色。煅烧后的硫酸钙用来制造某种牙膏和做墙和地板涂料。

除去沉淀的硫酸钙以后的滤液，即为非常纯净的盐卤，可送到制碱工厂制成饱和盐卤，供制碱用。

氯化铵

氯化铵的制造是用从蒸馏废液中得到的氯化钙溶液，与氨和CO₂气作用。为此，将蒸馏废液澄清，将固体物除去，后在蒸发器中将溶液浓缩到40 Be'时，溶液含CaCl₂约达50%。析出的盐被过滤掉，氯化钙溶液被放入带有搅拌器的罐子里，先后通入氨气

VERIFICATION

I, Wenli.Zhang of 601, Building B-2, Northern Territory, Dongsheng Technology Park, Haidian District, Beijing 100192, China, hereby declare that I am conversant with the Chinese and English languages and am a competent translator thereof. I declare further that to the best of my knowledge and belief the following 侯德榜选集 *Selected Works of Hou Debang* is a true and correct translation.

All statements made herein of my own knowledge are true and all statements made on information and belief are believed to be true. I further understand that willful false statements and the like are punishable by fine or imprisonment, or both under Section 1001 of Title 18 of the United States Code. I declare under penalty of perjury that the foregoing is true and correct.

Dated this 11 day of Nov. 2022.

Signature:

Wenli Zhang

Name:

Wenli Zhang

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Natural Scientists

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the loss, 25% or more of the salt was wasted. As a result, the existing alkali plants adopting ammonia-soda process all have the cheapest and most abundant supply of raw salt. In this case, it is often uneconomical to recover salt from waste liquor of distillation. However, the purity of the salt thus recovered is very high, which is comparable to the salts refined by soda lime method and made from water. The recovered salt is connected with the preparation of calcium chloride.

We will discuss several main products that have been produced on industrial scale from waste liquor of distillation, namely, refined salt, calcium chloride, calcium sulfate and ammonium chloride.

Refined salt and calcium chloride:

In production using ammonia-soda process, it is hoped that the volume of waste liquor of distillation should be as small as possible, especially when some substances in the waste liquor need further processing. To this end, the decomposition rate in the carbonation tower should be as high as possible to make the volume of filtrate as small as possible; on the premise of ensuring that the salt content in soda is within the permissible range, the washing water of filter should be as little as possible; and concentrated lime milk containing 250 g CaO per liter (with the specific gravity being 1.21) should be used, or solid lime should be added into the pre-ash bucket for distillation. The liquid containing a little excessive free lime is carbonized by introducing some waste CO₂ gas. By clarification, the precipitated CaCO₃ and all solid substances in the liquid are separated. The clear solution containing CaCl₂, NaCl and a small amount of dissolved CaSO₄ is concentrated in a triple-effect evaporator until NaCl and a small amount of CaSO₄ begin to precipitate and crystallize. The salt crystals are filtered out in a centrifuge, and a small amount of water is sprayed on the filter cake of the salt to wash off the CaCl₂ in the salt. The salt from the centrifuge can be directly packed in barrels without further drying.

Evaporation of the liquid should be carried out until the specific gravity of the liquid in the last effect of the evaporator reaches 42 °Bé. In this case, almost all salt can be precipitated. A little "chlorinated lime" is added to oxide the iron before clarification. The clarified solution is further concentrated in a cast iron pan until the molten substance solidifies when being cooled to obtain a product called 75% calcium chloride, the corresponding molecular formula of which is CaCl₂•2H₂O. This product is scooped into iron drums, each of which holds about 600 pounds. The iron drums should be sealed. The typical components of this product are as shown in Table 9:

Composition of 75% CaCl ₂ (%)			Table 9
CaCl ₂	73.6	Insolubles	0.08
NaCl	1.4	Water	24.92

To prepare anhydrous CaCl_2 , 75% CaCl_2 is heated in a reverberating furnace or a stirring furnace, and the molten substance is frequently raked until part of CaCl_2 is decomposed to generate CaO and HCl . The HCl gas escapes, leaving the molten porous CaCl_2 , called 95% calcium chloride. The cooled anhydrous calcium chloride, which is slightly fluorescent, is packed in iron drums or paper-lined wooden barrels and sealed.

The calcium nitride solution can be used as a refrigerant or antifreeze in ice making process; the 75% solid calcium chloride is used as a dust suppressant to reduce dust on roads. The anhydrous calcium chloride can be used as a catalyst for preparing calcium cyanamide by reacting calcium carbide with nitrogen, where the reaction is possible at a low temperature.

Calcium sulfate:

Crystalline $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ is called "Crown Filler" in products made by Solvay process, which is far purer than natural gypsum. The manufacturing process includes settling the waste liquor from ammonia distillation tower, and treating the clarified liquid with salt cake while it is hot. The excessive free lime contained in the solution is neutralized by a trace amount of sulfuric acid in the salt cake. If the obtained solution is slightly acidic, some lime milk may be added. The precipitated $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ is filtered by a vacuum drum filter, and then dewatered by a centrifuge, on which a small amount of water is poured to wash away the soluble salt. The gypsum crystals obtained are dried at low temperature, which is roughly like drying of sodium bicarbonate. The calcium sulfate is used as a filler for papermaking.

If drying is carried out at 190°C or lower, calcined gypsum $\text{CaSO}_4 \cdot 1/2\text{H}_2\text{O}$ is obtained, which can be used for making models and wall powder, and has many applications in the construction industry. If burning is carried out at above 200°C or above, all water will escape, but care must be taken not to reduce sulfate to sulfide. If containing trace iron, it will be dyed yellow after calcination. The calcined calcium sulfate is used to make a certain toothpaste and wall and floor coatings.

The filtrate, after removal of precipitated calcium sulfate, is a very pure bittern, which may be sent to an alkali plant to be made into a saturated bittern for use in alkali manufacture.

Ammonium chloride:

Ammonium chloride is produced by making calcium chloride solution obtained from waste liquor of distillation react with ammonia and CO_2 . To this end, the waste liquor of distillation is clarified, the solids are removed, and then the solution is concentrated in an evaporator to 40°Be . Now, the content of CaCl_2 in the solution reaches about 50%. The precipitated salt is filtered out, and the calcium chloride solution is put into a jar with a stirrer, and ammonia gas is introduced...