UNITED STATES PATENT OFFICE.

PAUL COMMENT, OF MÜLHAUSEN, ALSACE, FRANCE, ASSIGNOR TO SOCIÉTÉ FABRIQUES DE PRODUITS CHIMIQUES DE THANN ET DE MÜLHOUSE, OF RUE DE LA MERZEAU, MÜLHAUSEN, ALSACE, FRANCE.

MANUFACTURE OF POTASSIUM SULFATE.

1,389,862. Specification of Letters Patent. Patented Sept. 6, 1921.

No Drawing. Application filed December 20, 1919. Serial No. 346,319.

To all whom it may concern:

Be it known that I, PAUL COMMENT, a citizen of the Republic of France, residing at Mülhausen, Alsace, France, have invented certain new and useful Improvements in the Manufacture of Potassium Sulfate, of which the following is a specification.

This invention relates to the manufacture of potassium sulfate of high degree of purity from sulfuric acid and potassium chlorid in accordance with the equation

$$H_2SO_4+2KCl=K_2SO_4+2HCl.$$

Potassium sulfate has been made hitherto in the manner analogous to that of sodium sulfate, that is to say by mixing sulfuric acid with the alkali metal chlorid in an ordinary salt cake furnace; this process is difficult in practice and yields a product containing an irregular proportion of chlorid, seldom less than several parts per cent. of the sulfate.

By the present invention a better result is obtained by mixing the finely pulverized potassium chlorid with a sulfuric acid of such concentration that the mixture in the proportions indicated by the foregoing equation remains during the mixing operation as pulverulent as possible, this operation being performed in a mixer such as is used for making superphosfate, or in a mechanical kneading machine of cast iron, or any other metal adapted to resist acid. The mixture thus obtained is then heated to a temperature not exceeding its melting point. The heating may be conducted in a muffle furnace kept at a temperature of about 100–120° C. or by direct heating, or by the waste heat of a muffle furnace in which the product is subjected to final calcination, as will be presently explained. During this desiccation the mixture is stirred from time to time, either with suitable tools, such as pokers, or mechanically. About half the quantity of hydrochloric acid indicated by the foregoing equation is disengaged during this operation in a state of great purity. This acid is withdrawn and condensed, as is also that which is evolved in the mixer in which the materials are mixed. This mixer is placed above the dessiccating furnace and at one end thereof. When the disengagement of acid vapor has ceased the temperature is raised to about 300° C. either in the same furnace or, more practically and for allowing continuous working of the manufacture, in the pan of an ordinary salt cake furnace. A further quantity of hydrochloric acid is liberated and at the end of about 3 hours the product, which does not contain more than a few parts per cent. of chlorid, may be calcined in a muffle furnace at a temperature of about 700°–800° C. to eliminate completely the potassium chlorid. The charge remains porous or pulverulent from beginning to end of the manufacture. This fact adapts it for easy and rapid calcination.

It has been found that the elimination of the last traces of chlorid by calcination at high temperature occurs more rapidly and more completely the less the proportion of iron salts in the mixture. To secure this the cast iron pan of the ordinary salt cake furnace may be exchanged for one of refractory brick. The cast iron mixer, in which the material is heated only to a relatively low temperature, is scarcely attacked. The potassium chlorid is eliminated from the product very nearly and often quite completely if the proportion of sulfuric acid used exceeds by some parts per cent. that required by theory. This sulfuric acid accompanies the hydrochloric acid escaping from the calcining furnace, but since the quantity of sulfuric acid vapor produced in the calcining furnace is very small as compared with the hydrochloric acid vapor, it may be condensed separately; for this purpose the excess of sulfuric acid is recovered by withdrawing the vapor through a washing tower fed with sulfuric acid of suitable strength.

There is attained a continuous manufacture wherein the heat produced under the calcining furnace is completely used by placing the desiccating furnace, provided with its mixer in succession to or by the side of the pan of the ordinary salt cake furnace, so as to cause the materials to be treated to pass successively through the mixer, the desiccating furnace, the pan and the calcining furnace. The heating process, which occurs in three stages desiccation, calcination at 300° and calcination at 700°–800°, may also be carried out in a single muffle furnace comprising several compartments at successively increasing temperature.

By way of example there may be used in the manufacture of potassium sulfate hereinbefore described, an aqueous sulfuric acid

containing 70 to 72 per cent. of sulfuric anhydrid. Up to a certain point less concentrated sulfuric acid may be used if the finely pulverized potassium chlorid is mixed with a quantity of potassium sulfate equally finely pulverized so that a moist mixture is always obtained, which, however, remains pulverulent. The proportion of sulfate varies of course with the strength of the sulfuric acid used and constitutes a part of the sulfate made, continually brought back into the manufacture.

A part of the sulfuric acid participating in the reaction may be replaced by potassium bisulfate in so far as this latter may be soluble in the sulfuric acid used.

There may be also used solutions of potassium bisulfate in which case a less concentrated hydrochloric acid is obtained.

I claim:

1. A process for making potassium sulfate (free from chlorid) and hydrochloric acid, which process consists in mixing finely pulverized potassium chlorid with sulfuric acid in such proportion that the moist mixture remains as pulverulent as possible and heating the mixture.

2. A process for making potassium sulfate (free from chlorid) and hydrochloric acid, which process consists in mixing finely pulverized potassium chlorid with a solution of potassium bisulfate in sulfuric acid in such proportion that the moist mixture remains as pulverulent as possible, and heating the mixture.

3. A process of making potassium sulfate (free from chlorid) and hydrochloric acid, which process consists in mixing finely pulverized potassium chlorid with sulfuric acid in such proportion that the moist mixture remains as pulverulent as possible, heating the mixture at first at a low temperature until acid vapors cease to be evolved and then at an increased temperature until the reaction is complete, the heating being so conducted that the mass remains porous or pulverulent throughout.

4. A process for the manufacture of potassium sulfate (free from chlorid) and hydrochloric acid, which process consists in mixing finely pulverized potassium chlorid with sulfuric acid in such proportion that the moist mixture remains as pulverulent as possible, then heating the mixture at a low temperature until evolution of hydrochloric acid ceases, collecting the hydrochloric acid thus evolved and then heating the mixture until reaction is complete.

In testimony whereof I affix my signature.

PAUL COMMENT.