Patented July 18, 1944

2,353,959

UNITED STATES PATENT OFFICE

2,353,959

METHOD FOR REFINING SULPHUR

Robert C. Hills, Port Sulphur, La., assignor to Freeport Sulphur Company, New York, N. Y., a corporation of Delaware

No Drawing. Application October 18, 1941, Serial No. 415,638

7 Claims. (Cl. 23—229)

This invention relates to the refinement of sulphur and more particularly to the treatment of crude sulphurs for the removal of organic or carbonaceous impurities. The treatment, however, is ordinarily carried out in such manner that all impurities are substantially completely removed, including in addition to the carbonaceous impurities also inorganic matter, acid and moisture.

The invention is of primary or special value when the process is applied to the refinement of relatively pure crude sulphurs, namely, those containing less than about 0.05 percent organic impurities or even less than 0.03 percent of such impurities, as are obtained, for example, by Frasch mining operations. Through operation of the instant process, the carbonaceous content may be easily reduced to less than 0.00001 percent. For all practical purposes the product may be described as one hundred percent rhombic sulphur.

A specific object of the invention is to provide a process of obtaining refined sulphur of high purity from relatively pure crude sulphur at a cost sufficiently low to supply the large demand for refined sulphur at present unsatisfied due to the high cost of production. Refined sulphur is highly desirable in the manufacture of gun powder, rubber, pigments, fumigants and insecticides, and in the sterilization and preservation of many agricultural products.

According to the only process now in commercial use for the production of refined sulphur, the crude sulphur is distilled and the vapors condensed in a large masonry work chamber wherein the temperature is maintained around 100° C., producing flowers of sulphur which condense as a fine powder on the walls of the chamber and/or a liquid sulphur which condenses on the floor of the chamber. The liquid sulphur is drawn off into molds where it hardens in the form of blocks or sticks referred to in the trade as "brimstone" or "roll sulphur."

The high cost of production of the refined sulphur in accordance with the foregoing process has caused a decrease in consumption of refined sulphur over a period of years. This high cost has been due to the large sized equipment necessary to produce a comparatively small tonnage. Furthermore, the operation of the process is dangerous due to the extremely explosive and inflammable nature of the sulphur vapors. The process is also costly due to the deterioration and short life of the retorts brought about by the corrosive action of sulphur at the temperatures maintained therein, and finally, due to the necessity of removing accumulations of carbonaceous residues from the chamber and the necessity for periodically discharging the complete contents of the chamber to waste. It is an object of the present invention to provide a process of producing refined sulphur which is economical to operate and of less danger to operators of the equipment required.

Many attempts have already been made to provide a substitute for the distillation process, but as far as applicant is aware none of these processes has been economically feasible. In accordance with these proposed processes the crude sulphur has been treated with various absorbent and decolorizing agents, such as fuller's earth, zeolites, activated carbon, silica gel, diatomaceous earth, and the like, such treatment involving a subsequent filtration or decantation. Other proposed processes combine with the absorbent treatment a treatment with chemical agents, such as sulphuric acid, aluminum chloride or a combination of sulphuric acid with various oxidizing agents, which treatments are followed by washing or by filtration to remove the carbonaceous impurities. Although these prior processes remove a large proportion of the carbonaceous content of the sulphur, they are not capable of economically producing a refined sulphur of the same quality as is produced by the practice of the instant invention.

Broadly considered, the instant invention involves reacting the impurities contained in crude sulphur with chloro-sulphonic acid and separating the reaction products formed from the sulphur. The treatment may be carried out by treating the sulphur in molten condition with a small quantity of the acid and when the acid has reacted with the organic impurities to form readily separable reaction products, removing such reaction products by any of a number of processes, including filtering or washing with superheated water. A steam-jacketed agitator vessel of conventional construction may be used for the treatment. The intimate contact with the acid is best effected when the sulphur is in a non-viscous molten form, which condition lies generally between 250° and 320° F.

The reason for the high purifying power of chloro-sulphonic acid as compared with other agents such as fuming sulphuric acid has not been definitely established. The acid appears to react with the organic impurities to form soluble