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## SULPHUR TREATMENT

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This invention relates to improved sulphur compositions and more particularly relates to methods and compositions for treating molten sulphur which facilitate the recovery of solid sulphur from storage.

Frequently, in industrial handling of sulphur, it becomes necessary to solidify this material from its molten state. This is usually accomplished by pouring the molten sulphur into so-called slabs or vats. The term "sulphur slab" usually refers to a single horizontal layer, up to about 10 inches thick, of sulphur solidified from its molten state. Sulphur vats constitute a convenient way of storing large amounts of solid sulphur. In modern technology, a vat is a large block of sulphur usually, but not necessarily, consisting of a number of slabs prepared by repeated pouring of molten sulphur while allowing sufficient time between the pourings for the individual layers to solidify.

It is known to those skilled in the art that when molten sulphur is poured into such vats or slabs for cooling and solidification, the resulting solid sulphur has such structural characteristics and physical strength that it is difficult to crush and break into so-called bulk or solid sulphur. This difficulty in the handling of sulphur solidified from its molten state is expensive in its consequences. Because of the sulphur's high physical strength, heavy and expensive equipment is required to break the slabs or vats into bulk sulphur.

Two methods are known in the art to control the friability of sulphur solidified from its molten state. Both, however, have serious disadvantages. One of these methods allows for a limited control over the strength of the vats by varying the thickness of the individual sulphur layers constituting a vat. It is known that the thicker these layers are, the weaker is the structure of the vat. The method of reducing physical strength by increasing layer thickness has definite disadvantages. First, the physical strength of the vat or slab is reduced only to a limited extent. Second, by pouring thicker sulphur layers the cooling rates are reduced, and therefore more time is required to produce a vat of a given size. Third, by pouring thick layers the possibility of molten sulphur pocket formation in the vat is created. These molten sulphur pockets, in which the sulphur remains at over 238.1° F., constitute a hazard to the operating personnel.

The second method known to the art consists of adding gaseous ammonia to molten sulphur prior to solidification. By an unknown mechanism the presence of small amounts of ammonia increases the friability of the solid sulphur. The disadvantage in using ammonia for this purpose lies in the necessary inconvenience of having special equipment in order to carry out the addition of a gaseous substance to molten sulphur. This equipment consists of an ammonia tank, tubing, valves, pressure gauges, etc., at many locations to which molten sulphur is delivered, such equipment is unavailable.

Thus, in the past, there was no convenient method by which the physical strength of sulphur vats or slabs could be efficiently controlled.

It is an object of this invention to provide a method

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for decreasing the physical strength and improving the handling properties of sulphur solidified from the molten state.

It is another object of the present invention to provide sulphur compositions comprising sulphur and a small amount of an additive for sulphur that is to be stored in solid vat or slab form, which renders the solidified sulphur more easily recoverable from storage by increasing its friability.

It is another object of this invention to provide an additive for sulphur that is to be stored in slabs or vats that not only increases its friability but also in many cases improves grind-ability and screen-ability, reduces the amorphous content and reduces the electrostatic charge of the sulphur.

It is a further object of this invention to provide an additive for sulphur that is to be stored in vats or slabs which has a reduced explosive hazard and reduced toxicity, compared with additives presently known, and which remains in the sulphur and in many cases upon subsequent liquefaction and solidification continues to impart improved characteristics to the sulphur through many successive meltings and solidifications.

The utility of this invention lies in the fact that molten sulphur chemically treated as described herein yields, upon solidification, a material of lower physical strength, which is more friable, and easier to crush and handle as compared to untreated sulphur.

The objects of this invention may be achieved by adding certain liquid or solid compounds or additives to molten sulphur prior to its storage and solidification. Surprisingly, it has been found that liquid and solid basic organic amines will increase the friability of sulphur if added to the molten sulphur prior to solidification.

Furthermore, the treatment of the sulphur in accordance with the invention frequently also improves the grinding and screening characteristics of sulphur, as well as reducing its electrostatic charge and amorphous sulphur content.

The compounds or additives for the sulphur which have been found to be effective in the practice of this invention belong to the class of compounds consisting of:

A. Liquid and solid, primary, secondary and tertiary, alkyl, aryl and cyclic amines having a  $K_B$  value greater than  $10^{-10}$  in which the amino nitrogen is attached to a primary, secondary or aromatic carbon atom. Such amines include: n-butylamine, tetradecyldimethylamine, aniline, cyclohexylamine, N,N-diethylcyclohexylamine, N-methylpyrrolidine, pyridine, tetraethylenepentamine, ethylenediamine, piperidine, pyrrole, piperazine, 1-amino-2-propanol, indole, 1-(2-aminoethyl)-2-(n-heptadecenyl-2)-imidazoline, n-amylamine, N,N-dimethyldodecylamine, N,N-dimethyltetradecylamine, N,N-dimethylhexadecylamine, N,N-dimethyloctadecylamine, N,N-dimethylamylamine,  $\alpha$ -methylamylamine, propylamine,  $\beta,\beta$ -dimethylpropylamine, sec-butylamine, isobutylamine, hexylamine, N,N-dimethylhexylamine, n-heptylamine, N,N-dimethylheptylamine, octylamine, etc.

B. Quaternary nitrogen compounds which decompose between 20 and 160° C. to yield ammonia. Such compounds include ammonium carbonate, ammonium bicarbonate, etc.

C. Quaternary nitrogen compounds which on heating to temperatures between 20 and 160° C. decompose to yield an organic amine having a  $K_B$  value greater than  $10^{-10}$  in which the carbon atom or atoms directly attached to the amino nitrogen is primary, secondary or aromatic. Such compounds include tetramethylammonium hydroxide, tetraethylammonium hydroxides, etc.