

# UNITED STATES PATENT OFFICE

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## PURIFICATION AND RECOVERY OF CRYSTALS OF METAL SALTS

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This invention relates to the formation, purification and recovery of crystals and more particularly to the formation, transformation, purification and recovery of crystals of metal salts which are capable of combining with water in molecular form and forming a plurality of hydrates.

An object of this invention is to purify crystals of metal salts capable of existing as crystals in two or more stages of hydration in the presence of a saturated solution of the metal salt.

Another object of this invention is to form and recover crystals of metal salts of substantially any desired size economically and efficiently.

Another object of this invention is to recover from minimum volume, the maximum yield of crystalline materials which are capable of crystallizing from a water solution in a plurality of hydrates.

Another object of this invention is to form crystals of a metal salt, from a mixture containing the metal salt and small insoluble particles, of a size sufficient to form interstitial spaces when formed in a bed for the passage of the insoluble materials therethrough.

Another object of this invention is to separate crystallizable substances capable of forming a plurality of hydrates from a liquid containing the crystallizable substances and insoluble, relatively finely divided materials.

A more particular object of this invention is to obtain a maximum amount of zinc in the form of zinc sulfate crystals from a fume containing zinc or zinc salts.

One of the methods most frequently used for the purification of crystals of metal salts is to prepare a saturated solution of the metal salt at the temperature of maximum solubility and cool the solution to room temperature. For example, zinc sulfate crystals are customarily purified by preparing a saturated solution of the zinc sulfate at a temperature of about 60° C. and cooling the saturated solution to room temperature whereby a relatively large yield of crystals per unit volume of mother liquor is obtained.

In accordance with this invention, crystals of metal salts which form a plurality of hydrates are purified to substantially the same degree or greater and with yields of 3 to 4 times per unit volume of mother liquor, than those resulting from the practice of purification by the conventional method of crystallization.

The method of purification of this invention is particularly applicable to the recovery and purification of crystals of metal salts which are contained in mixtures of finely divided insoluble

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materials, such as those involved in the recovery and purification of zinc sulfate obtained from bag house fumes. The method of purification of crystals of metal salts in accordance with this invention comprises heating a magma containing the metal salt crystals in a higher hydrate and a saturated solution of the metal salt to a temperature above the transition temperature at which the metal salt is converted from one hydrate to another hydrate. The period of heating is sufficient to convert substantially completely the crystals of the higher hydrate to the lower hydrate. In this step, the crystals of the higher hydrate of the metal salt are dissolved in the saturated solution of the metal salt with the simultaneous crystallization of the lower hydrate of the metal salt. The magma is then cooled to a temperature below the transition temperature, the rate of cooling through the transition temperature being such that the transformation from the lower hydrate of the metal salt to the higher hydrate of the metal salt is accomplished by dissolution of the lower hydrate accompanied by simultaneous crystallization of the higher hydrate from the solution. The net result of this second step is to reconvert the lower hydrate of the metal salt to the higher hydrate of the metal salt, and during this second step of the process the lower hydrate of the metal salt is substantially completely dissolved and the higher hydrate of the metal salt is simultaneously crystallized. In this process of conversion of the higher hydrate to the lower hydrate with the subsequent reconversion to the higher hydrated form, a material amount of the impurities are removed from the crystals of the metal salt initially heated. After the metal salts in the higher hydrate have been reconverted, they may be separated from the mother liquor by any conventional method such as filtration, decantation or centrifugation. When the crystals of the metal salts are contained in a magma having material amounts of finely divided small particles, the crystals may be separated by the method described in applicant's copending application S. N. 529,038, filed March 31, 1944, now abandoned.

When crystals are separated by the process described in this copending application, they are desirably relatively uniform and of a size which, when in the form of a bed on a screen, provide interstitial spaces sufficiently large for the insoluble materials to pass through. Desirably, too, such crystals are as small as possible so that the maximum amount of occluded insoluble are contained in them.