

ALIEN PROPERTY CUSTODIAN

PROCESS FOR ISOLATING PENTAERYTHRITE FROM ITS SOLUTIONS

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The present invention relates to a process for obtaining pure pentaerythrite from its impure solutions and more particularly from impure solutions arising from its manufacture.

Pentaerythrite is industrially prepared by condensating acetaldehyde and formol in an aqueous medium in presence of a condensating agent. As is known, the reaction between these two aldehydes does not take place solely in the desired direction; secondary reactions always occur simultaneously, giving rise to other substances, in particular to dipentaerythrite, so that, at the end of the condensation, an aqueous solution of pentaerythrite containing various impurities is obtained. For separating pentaerythrite therefrom, the known processes consist in more or less highly concentrating the solution, after having eventually freed it from the condensating agent, and in allowing the concentrated solution to cool, for causing the crystallization of the pentaerythrite which is separated in the cold state.

The product obtained by this method of procedure always contains certain impurities such as dipentaerythrite, which crystallize with the pentaerythrite and which it is consequently very difficult and sometimes impossible to separate completely by recrystallization. For certain raw products, recrystallization does not even allow of obtaining an improvement in the quality.

According to another proposed technique, the raw solution is concentrated under vacuum at 35° until a content in dry substance of 70 to 75% is obtained, the solid product being then filtered or dried in the cold state. The Applicant has found that this technique offers no advantage over the preceding one as regards the quality of the product.

The difficulties, arising from the fact that certain impurities crystallize at the same time as the pentaerythrite, are well known to those skilled in the art and are mentioned in particular in the French Patent No. 786,909 dated February 14, 1935. For taking advantage of the impure raw product, said patent proposes to free it from the impurities which defile it, by a special washing process with friction.

The Applicant has found, and this is what forms the subject-matter of the present invention, that, contrarily to what was known up to now, it is possible to directly obtain by crystallization, pentaerythrite of great purity giving, in particular, in the cold state, clear aqueous solutions. It suffices, and it is essential for obtaining this result, to effect the crystallization and separa-

tion of the pentaerythrite at a temperature higher than 40°, that is to say at 45°, 80°, 100°, and even more. At these temperatures, the impurities and, in particular, dipentaerythrite, remain dissolved in the mother-water, which is not the case when the operation is effected at lower temperatures. Pentaerythrite is thus obtained which is practically free from accessory products. The temperature at which the process is carried out, for the crystallization itself as well as for the separation of the crystals, can be chosen in the conditions above defined, in particular according to the nature of the solutions treated or the facilities of execution.

A particularly advantageous method of carrying out the invention consists in causing crystallization by concentrating the solution in the hot state. In proportion as evaporation takes place, the pentaerythrite crystals deposit and their quality remains excellent, even if the concentration is continued until nearly the totality of the pentaerythrite contained in the mother-solution is separated. Thus, in a single operation, a very high output of a practically pure product is obtained.

Another particularly advantageous method of procedure for treating crystallizing mother-waters or washing waters and more generally solutions of low pentaerythrite content, consists in concentrating the solution at a high temperature, then in slightly cooling and in separating the pentaerythrite crystals deposited during the cooling; said crystals constitute a product of good purity, if they are separated at a high temperature according to the invention.

Example 1.—Condensation is effected between the acetaldehyde and the formol (4,6 molecules for 1 of acetaldehyde) in presence of lime (0,6 molecule for 1 of acetaldehyde). The lime is eliminated from the reactive liquid by treating with oxalic acid. After filtration, an impure solution is obtained containing about 10% of pentaerythrite.

100 parts of this solution are concentrated under vacuum at 85° until 84 parts of water are distilled. The crystals formed are separated from the mother-waters by draining at this same temperature, washed with a small quantity of a saturated aqueous solution of pentaerythrite, and dried. 90% of the pentaerythrite present in the initial solution is thus extracted. This very pure product gives a clear solution in cold water. Its melting point according to the Maquenne block is 262°.

If the concentration of the same solution is effected at 35° instead of 85°, the product ob-

tained is impure, even if the concentration is carried much less far. Its melting point according to the Maquenne block only reaches 242° and it gives a very turbid aqueous solution.

Example 2.—A commercial pentaerythrite giving in the cold state a turbid aqueous solution and melting at 235 to 246° (melting point taken in a capillary tube by gradually raising the temperature), is dissolved in 3 parts of boiling water. The filtered solution is concentrated when boiling at an ordinary pressure, at about 104°, with stirring, so as to eliminate 2,5 parts of water. The concentration being terminated, cool-

ing is effected, whilst continuing the stirring, to a temperature of 90°; the crystals (0,9 part) are separated at this temperature, washed and dried. Pure pentaerythrite is thus obtained giving a clear solution in an excess of cold water and the melting point of which, determined in a capillary tube in the same conditions as for the initial product, is from 253 to 255°. By evaporating the mother-water in the dry state with care, 0,1 part of very impure pentaerythrite is extracted, giving in the cold state an extremely turbid aqueous solution.

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