

ALIEN PROPERTY CUSTODIAN

PROCESS OF PURIFYING CONVERSION PRODUCTS OF RUBBER

August Amann and Arthur Greth, Wiesbaden, Germany; vested in the Alien Property Custodian

No Drawing. Application filed September 14, 1940

This invention relates to the purification of the raw isomerization products produced by the process of Patent Number 2,200,715 and of our concurrently filed copending application involving heating together rubber, phenol and acidic agents at normal or superatmospheric pressure.

It has been found that the crude rubber conversion product can be freed from the phenol in a technically easier and more commercial way by first dissolving it in an aromatic or aliphatic hydrocarbon or a mixture of both and successive washing of this solution with water and aqueous phenol. It has been found that two or three washings reduce the phenol content of the rubber conversion product so far, that the last traces can be easily removed by a simple steam distillation.

As to the aliphatic or aromatic hydrocarbons which are to be used as solvents for the rubber conversion product, it is advisable to use in first line those whose solvent action on phenol is less than that of water or aqueous phenol. It is therefore advisable to use preferably aliphatic hydrocarbons or aromatics with the addition of more or less aliphatic hydrocarbons. It is also advisable to use hydrocarbons which are generally used as lacquer varnish solvents, e. g. mineral spirits, white spirit, toluene, xylene, decalin etc, since such purified solutions can be used directly for the manufacture of coating materials.

The aqueous phenol, which is used for the washing of the rawproduct, shall contain such much water, that the phenol does not crystallize at room temperature. The water contact shall be about 25% of the phenol-water mixture, however it can be also lower or higher without loosing the washing reaction. However it has been found, that the best separation the rubber conversion product solvent layer and the phenol-water layer is obtained fastest and best by using the above proportion of phenol : water in the washliquor. The separated phenol-water layer can be repeatedly used for the washing after dilution with water or crystallisation of the phenol.

After one or two washings with aqueous phenol the phenol-content of the rubber conversion prod-

uct is so low that the last traces can be easily removed by distillation after addition of a small amount of water.

The resulting solution of the rubber conversion product can be used for the preparation of coating compositions as it is or after addition of suitable solvents. It is compatible with gums, synthetic resins, celluloseesters, plasticizers, drying or nondrying vegetable oils as e. g. linseed, china-wood, castor oil and other rawmaterials, used in the coating industry. The rubber conversion product can be obtained also in a flaky-pulverized state by precipitation from its solution with a nonsolvent for the rubber conversion product.

Example

800 parts of the rubber conversion product, prepared by heating under pressure 50 parts of pale crepe rubber and 100 parts of phenol to 210° are dissolved in 1200 parts of mineral spirits to a 40% solution. 200-175 parts of water are added in order to liquify the crystallized phenol. After the separation of the two layers, the phenol solution, containing 9-10% of the phenol of the rawproduct, is separated from the benzine solution. By using more water or by trying to remove also the last traces of phenol from the benzine solution, emulsions are formed, which cannot easily be separated. Therefore the further purification is carried out with a phenol-water mixture, which surprisingly gives a fast and clean separation of the two layers. The benzine solution, 1740 parts, resulting after the first treatment with water is washed twice with each 445 parts of 75% aqueous phenol, by which the phenol content is reduced from 16-17% to 3-4%. This remaining phenol is easily removed by distillation at atmospheric or reduced pressure after addition of some water. According to the amount benzine distilled off, solutions of the rubber conversion products with various percentages of solids are obtained, which can be used as they are or diluted with other lacquer solvents.

AUGUST AMANN.
ARTHUR GRETH.