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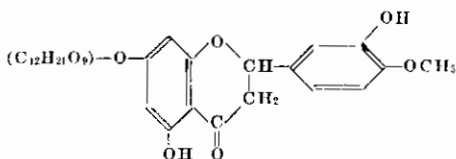
## PROCESS FOR PREPARING A DERIVATIVE OF HESPERIDIN

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This invention relates to improvements in a process for preparing a derivative of hesperidin, which is a new chemical substance, soluble in water and useful for therapeutical purpose.

Hesperidin (5-7-3' - trioxy - 4' - methoxyflavone glucoside) which has the constitutional formula

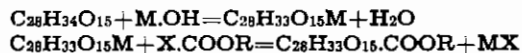


has pharmacological effects, but it is hardly soluble in water, alcohol and other ordinary solvents at room temperature so that it is very inconvenient for handling.

When an aqueous solution of an alkali, e. g. potassium hydroxide or sodium hydroxide, is added to hesperidin, the latter is dissolved and produces its alkali salt, which when treated with an alkyl halogen carbonate such as ethyl chlorocarbonate in a solvent, alcohol, benzol, etc., a reaction takes place separating halogen alkali and a carbalkoxy radical is introduced.

This product is a new chemical substance which is easily soluble in water, hot alcohol and hot ethyl acetate, and its aqueous solution shows a reddish brown colour by ferric chloride. If metallic magnesium is added to its alcoholic hydrochloric acid solution a blight red colour appears.

The chemical reactions above mentioned are shown by the following formulae.



In these formulae, the notation M represents

an alkali metal, X represents halogen atom and R alkyl radical.

### Example 1

10 parts of hesperidin are dissolved in about 150 parts of 2% aqueous solution of potassium hydroxide, to which 4 parts of ethyl chlorocarbonate are added and is kept alone for a few hours. A very small quantity of sediment produced is removed by filtration and the filtrate is evaporated under vacuum when a viscous residue is obtained.

This residue is extracted with hot alcohol so as to remove insoluble potassium chloride, and a jelly-like sediment is obtained when the alcoholic solution is cooled. This sediment is collected by filtration and is re-crystallized from alcohol. When the product is dried in vacuum at 100° C, 8 parts of slightly yellowish powder are obtained, which melts at 135° C under decomposition. It is ascertained by analysis that this is a new chemical substance having the chemical formula  $C_{31}H_{38}O_{18}$ .

This new substance is easily dissolved in a dilute caustic alkali solution and produces hesperidin, the starting material. If it is boiled with a mineral acid, sugars and aglycon are produced.

### Example 2

10 parts of hesperidin are stirred for an hour with a solution of 8 parts sodium in 500 cc. alcohol, then 4 parts of ethyl chlorocarbonate are added and the mixture is refluxed for two hours. Sodium chloride produced therein is filtered off while hot, and the alcoholic solution is evaporated until it gets to a small amount, from which jelly-like precipitates are obtained. Recrystallizing from alcohol, the same product as of the Example 1 is obtained. The yield is 7 parts.

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