

PATENT SPECIFICATION



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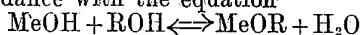
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COMPLETE SPECIFICATION.

Manufacture of Alkali Alcoholates.

We, DR. ALEXANDER WACKER GESELLSCHAFT FÜR ELEKTROCHEMISCHE INDUSTRIE G.M.B.H., a body corporate organised according to the laws of Germany, of 20, 5 Prinzregentenstrasse, Munich, Germany, do hereby declare the nature of this invention and in what manner the same is to be performed, to be particularly described and ascertained in and by the 10 following statement:—

The manufacture of alcoholates in accordance with the equation



from an alcohol and an alkali by distillation is known. Particularly in the case of 15 alcohols soluble in water there have been used in this manufacture auxiliary liquids, such as benzine, benzene etc. The chief difficulty resides in the complete separation of water. This necessitates a great expenditure of heat and requires a very effective and extensive 20 fractionation apparatus. The difficulty in converting a caustic alkali into the alcoholate increases with the degree of conversion required. This circumstance is particularly disadvantageous since for various applications of the alcoholates, for instance for condensation, products 25 free from caustic soda are necessary and even a small content of caustic alkali may produce undesired secondary reactions.

This invention relates to a process of quickly, cheaply, and continuously 35 achieving the substantially complete conversion of an alkali hydroxide into an alcoholate in a comparatively simple apparatus, and consists in bringing in contact a stream of an alcoholic alkali 40 solution with a counter-current of the alcohol vapour. This operation being conducted in a column still there is formed at the lower end of the column an alkali alcoholate containing no free 45 alkali; the water and alcohol and, if used, the auxiliary liquid passing away at the upper end of the column. An auxiliary liquid, such as benzene, which forms with water or with water and alcohol an azeotropic mixture, is to be recommended 50 when the operation is conducted with an alcohol soluble in water. As auxiliary liquids there are generally suitable

volatile substances and mixtures of them which under the given conditions have no 55 tendency to react either with the alcohol or with the alcoholate, for instance such liquids as hydrocarbons or unhydrolysable ethers or the like.

In applying the invention there is 60 advantageously used a column still which is fed at its upper part with an alcoholic solution of alkali and, if the nature of the alcohol used requires it, also with an auxiliary liquid. The alkali solution 65 flows down the column entering into reaction with the alcohol vapour which flows in the opposite direction and gives up to the latter the water produced by the reaction, so that from the lower part 70 of the column an alcoholate solution, free from caustic alkali, can be continuously withdrawn. The vapours issuing from the head of the column are condensed and the alcohol, or the auxiliary liquid 75 and the alcohol, are separated from the water and returned to the column; an additional reflux is generally superfluous.

By use of the invention there is obtained as compared with the known processes and in an unexpectedly favourable manner, an equal output in a considerably shorter time and with complete conversion to a pure product. 80

The following Examples illustrate the invention:— 85

EXAMPLE 1.

In order to make sodium ethylate by the action of ethyl alcohol on caustic soda in the presence of benzene as an auxiliary liquid, a still having a column 90 6000 mm. in height and 300 mm. in diameter is used. For starting the process the lower part of the apparatus is charged with alcohol free from water or with a solution of ethylate, preferably with addition of a small percentage of benzene. 95 The upper part of the column is charged with a quantity of benzene and alcohol sufficient for the formation of a ternary mixture of alcohol-water-benzene. At 100 the upper part of the column about 600 mm. below the head, there are supplied per hour about 20 litres of an alcoholic solution of 7—10 per cent. strength of caustic 105 soda. This latter solution may with

advantage contain a small proportion of benzene, which, however, may be separately introduced into the column. The heating is so regulated that about 60 litres per hour of a ternary mixture of alcohol benzene and water passes over. The distillate boils at 64°C ., is turbid and separates into two layers. The lower, aqueous layer, is withdrawn in a separating vessel and the upper layer, chiefly a mixture of benzene and alcohol, is returned to the column, if necessary after separation of undesired components, by any known operation. The lower layer is worked up in known manner and if necessary alcohol and benzene are restored to the cycle. The sodium ethylate solution which may be withdrawn continuously from the lower part of the column is completely free from caustic soda.

EXAMPLE 2.

For making sodium butylate the same apparatus as is described in Example 1 may be used, but the operation is conducted without an auxiliary liquid. There are supplied per hour 50 litres of a solution of 10 per cent. strength of caustic soda in butyl alcohol and the heating is so controlled that about 70 litres of aqueous butanol distil per hour. The distillate separates into two layers, of which the lower consists essentially of water and is separated, while the upper

layer, which consists chiefly of butyl alcohol, is returned to the column.

EXAMPLE 3.

For making potassium butylate the operation may be as described in Example 2, merely substituting caustic potash for caustic soda.

The alkali alcoholates of other alcohols may be made in a similar manner. In many cases it is of advantage to use either raised pressure or reduced pressure, so that the ratio of volatile constituents to each other may be affected and the consumption of heat thereby diminished.

Having now particularly described and ascertained the nature of our said invention and in what manner the same is to be performed, we declare that what we claim is:—

1. A manufacture of alkali alcoholates from alcohols and caustic alkalies by distillation, wherein an alcoholic alkali solution is caused to flow in counter-current to the alcohol vapour.

2. A manufacture as referred to in Claim 1, wherein an auxiliary liquid is used.

Dated this 25th day of April, 1932.

ABEL & IMRAY,

Agents for the Applicants,
30, Southampton Buildings, London,
W.C. 2.