

PATENT SPECIFICATION



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COMPLETE SPECIFICATION.

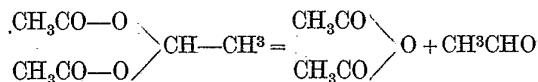
Process of Manufacture of Acetic Anhydride and Aldehyde.

We, SOCIÉTÉ CHIMIQUE DES USINES DU RHONE, of 21, rue Jean-Goujon, Paris, France, a company, organised under the laws of France, do hereby declare the

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nature of this invention and in what manner the same is to be performed, to be particularly described and ascer-

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In all the known processes having for their purpose the realisation of this decomposition, secondary reactions occur which give birth, particularly, to notable quantities of acetic acid and tar. These secondary reactions are due to the too energetic condensing action of the catalysts employed for the decomposition.

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We have found that halogenated zinc derivatives, as well as compounds capable of giving these derivatives in the presence of zinc (for instance, acetyl chloride or bromide), in quantities even inferior to 1% of the weight of the diacetate employed, allowed this decomposition to be performed in an almost quantitative manner, according to the above reaction.

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We have found, moreover, that in order that the decomposition of the diacetate should take place in a regular manner, it is necessary to withdraw, as they are formed, the anhydride and the aldehyde from the reacting medium.

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We have, besides, noticed that, during the reaction, the halogen is gradually carried away by the anhydride and aldehyde vapours which are evolved, and, in order to obviate this destruction of the catalyst, we feed the reacting medium with ethylidene diacetate containing in dissolution a little of the selected halogenated derivative.

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Besides, we hold back the greatest por-

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tained in and by the following statement:—

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The present invention relates to a process of manufacture of acetic anhydride and aldehyde by the decomposition of ethylidene diacetate, according to the reaction:

tion of the halogen which escapes during the decomposition, by causing the distilling vapours to pass through a column full of zinc presenting a large surface (shavings, turnings, threads, etc.).

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Moreover, we operate the fractionated condensation of the distilled vapours, which allows one to obtain directly pure acetaldehyde and raw acetic anhydride containing a quantity more or less small of diacetate and of acetaldehyde.

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When the decomposition is performed in accordance with these indications, the decomposed diacetate separates only into acetic anhydride and aldehyde, and the quantity of tar formed is quite small.

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EXAMPLE:

0.5 gramme of anhydrous zinc chloride is dissolved, with heat, into 5 grammes of acetic acid, and this solution is incorporated with 300 grammes of ethylidene diacetate.

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80 grammes of this mixture are caused to pass into a flask on which is mounted a column, 10 cms. in height, filled with zinc millings and heat is applied. As soon as the temperature of the liquid reaches 145° C., aldehyde begins to be evolved; it is caused to pass through a second column, where it is freed from the anhydride which has been carried away, and it is collected in a flask immersed in a mixture of ice and common salt.

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The temperature of the liquid in the flask, which had risen to 165° C., returns to 150° C., owing to further formation of anhydride which lowers the boiling point of the mixture. The heating is regulated in such a manner that the temperature at the top of the columns of zinc millings is maintained at about 130° C. The supply of ethylidene diacetate containing zinc chloride is regulated so as to maintain constant the level in the flask until the 300 grammes have been introduced.

The apparatus is maintained at the prescribed temperature until the end of

the operation, which is terminated at the end of two and a half hours.

56.80 grammes of acetaldehyde are collected in the cooled flask. The distilled raw anhydride contains 28% diacetate carried off, and about 2% acetaldehyde. There remains in the flask (returned back from the column) a mixture of anhydride and of undecomposed diacetate. By fractionating the raw anhydride and the diacetate remaining in the flask, after having stopped the action of the catalyst by the addition of a suitable compound, such as potassium acetate, one obtains:

	5.05 grammes of aldehyde dissolved in raw anhydride,
151.5	„ „ pure anhydride,
77.8	„ „ recovered ethylidene diacetate,
2.02	„ „ tar from the reaction flask.

These figures show how the decomposition has taken place regularly. The yields are excellent: The 222.2 grammes of diacetate have given 61.85 grammes of aldehyde and 151.5 grammes of anhydride, instead of 66.9 and 155.2 grammes, which are the theoretical yields respectively.

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. Process for the manufacture of acetic anhydride and aldehyde by decomposition of ethylidene diacetate, characterised by the use as decomposition catalyst of small quantities of a halogenated zinc derivative or of bodies adapted to give these derivatives in the presence of zinc, the said catalyst being capable of being used even in quantities inferior to 1% of the weight of the diacetate.

2. Process for the manufacture of acetic anhydride and aldehyde, as claimed in Claim 1, further characterised

in that ethylidene diacetate containing a halogenated zinc derivative is heated to its boiling point or to a slightly lower temperature, the anhydride and acetaldehyde produced being separated as they are formed.

3. A process of manufacture of acetic aldehyde and anhydride as claimed in Claims 1 and 2, further characterised in that the lost halogen is recovered and the loss of catalyst made up by causing the vapours of aldehyde and anhydride produced by the decomposition of ethylidene diacetate to pass upon zinc shavings, turnings or ribbons, and by feeding continuously in the decomposition apparatus a halogenated derivative capable of compensating the loss due to the catalyst carried off.

4. The acetic aldehyde and anhydride, when manufactured by the processes claimed in the preceding claims or their obvious chemical equivalents.

Dated this 22nd day of April, 1925.
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