

## UNITED STATES PATENT OFFICE

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## PRODUCTION OF ALCOHOL FREE FROM FUSEL OIL

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14 Claims. (Cl. 202—67)

This invention relates to the production of alcohol free from fusel oil.

In order to remove the fusel oil from crude spirit, almost all the known methods of operation make use of the fact that by a process of rectification by means of ordinary rectifying apparatus a distillate that is entirely free from fusel oil can be obtained if the vapours are concentrated to such an extent that, after condensation, they contain 94.5 to 95 per cent by weight of spirit (compare Foth, Handbuch der Spiritusfabrikation, 1929, page 737, fourth paragraph). In the case of known processes and by ordinary rectification this concentration is attained only by the consumption of considerable quantities of steam and cooling water whereas by the present invention it is possible to carry out, with a considerably reduced consumption of steam and cooling water, a direct removal of the fusel oil from aqueous alcohol containing, for example, 80 to 90 per cent by weight of alcohol.

The essence of the present invention resides in the employment of suitable salts or mixtures of salts in the carrying out of the process of fusel oil removal. The vapour phase of aqueous alcohol containing fusel oil has in the presence of suitable substances, especially salts such as alkali or earth alkali salts of the fatty acids, a higher concentration of alcohol than in the absence of these salts. Since the content of fusel oil in alcohol is essentially occasioned by the fact that, together with the alcohol vapour, a considerable amount of water vapour passes into the vapour phase, the addition of the substances mentioned causes the quantity of water vapour in the vapour phase to be reduced to such an extent that all the fusel oils remain behind, without it being necessary, as in the case of the ordinary rectification, to employ tall columns and/or to work with large reflux apparatus and a correspondingly high consumption of steam.

The process can be carried out for example by running into a distilling vessel one of the substances that have been defined above with regard to their function—e. g., a mixture of potassium acetate and sodium acetate—and then running in the alcohol to be purified. The vapours are passed through a simple rectifying column which is worked with a relatively small reflux attachment. Alternatively the alcohol vapour containing the fusel oil, such as is obtained by vaporization or as occurs in the concentration of mash, may be passed through one or more vessels charged with a suitable salt solution or suspension, whereupon the vapours also pass through a

short column; in such a way alcohol that is entirely free from fusel oil is obtained with quite small consumption of steam and cooling water. The action of such an arrangement can be substantially improved by causing suitable solutions or suspensions of appropriate salts to flow actually in a column in counter-current to the vapours; by suitably dimensioning the column and the quantities of salts, the purifying operation can be performed entirely with the aid of this simple device—e. g. a suitable washing column filled with Raschig rings. Furthermore, this purification can be utilized for bringing the alcohol simultaneously to a higher concentration than is necessary for merely removing the fusel oil in accordance with the foregoing description; for example, by treatment with a solution or suspension of the almost anhydrous salts—e. g. of a mixture of potassium acetate and sodium acetate in absolute alcohol—an alcohol of at least 99.8% by weight and at the same time entirely free from fusel oils is directly obtained.

*Example I*

A mixture of 175 parts of potassium acetate and 75 parts of sodium acetate, 150 to 200 parts of water and 50 parts of ethyl alcohol are placed in a distilling vessel and alcohol containing fusel oil is run in. The distilling vessel is provided with a short column. The distillation gives an alcohol of about 95% by weight that is free from fusel oil. Towards the end of the operation, the alcohol contained in the still can even be distilled off until the quantity of fusel oil in the still amounts to about 40% of the alcohol that is still present, without any products containing fusel oil reaching the distillate.

*Example II*

For the purpose of carrying out the separation of the fusel oil in the manner described in Example I entirely continuously, the mixture in the still must be drawn off continuously or discontinuously in separate fractions and be freed from the concentrated fusel oil and water. For example, when employing an alcohol of 80% by weight, it is necessary to remove again and again the quantities of water that remain in the still owing to the concentration to 95% by weight, in order that the dehydrating action of the contents of the still shall be maintained. Advantageously, the mixture that is continuously or discontinuously drawn off is freed from the fusel oil and from the attendant water in a second distilling device and then returned into the still. The

mixture of alcohol, fusel oil and water that is driven off can, owing to the great concentration of fusel oil in the contents of the still that is occasioned by the process, and consequently in relation to the alcohol, be very readily worked up into fusel oil and an alcohol which is poor in fusel oil and which is returned into the process.

### Example III

10 For continuously obtaining anhydrous ethyl alcohol from crude spirit containing fusel oil, the process described in Example II can be developed by washing the rectifying column with an alcoholic solution or suspension of salts, for  
15 example, potassium acetate and sodium acetate, in such quantity that the alcohol vapours coming off at the top end of the column are practically anhydrous. The working up of the salt solution in this case is effected by completely removing the residual water from the mixture of salts after  
20 driving off alcohol and fusel oil and dissolving the molten salt mixture, that is left, in absolute alcohol in this condition and again supplying it to the dehydrating column.

25 What I claim is:—

1. A process of producing ethyl alcohol of greater than 95% concentration free from fusel oil comprising contacting the vapor of an aqueous alcoholic solution containing fusel oil and  
30 at least 10% water with an absolute alcoholic solution of a dehydrating fatty acid salt, drawing off and condensing the alcohol vapors free from fusel oil and retaining the fusel oil with the salt solution.

35 2. A process of producing ethyl alcohol of greater than 95% concentration free from fusel oil comprising contacting the vapor of an aqueous alcoholic solution containing fusel oil and at least 10% water with a water-free solution inert to alcohol and fusel oil of a dehydrating fatty  
40 acid salt, drawing off and condensing the alcohol vapors free from fusel oil and retaining the fusel oil with the salt solution.

45 3. A process of producing ethyl alcohol of greater than 95% concentration free from fusel oil comprising contacting the vapor of an aqueous alcoholic solution containing fusel oil and at least 10% water with an absolute alcoholic solution of a mixture of alkali acetates, drawing off  
50 and condensing the alcohol vapors free from fusel oil and retaining the fusel oil with the salt solution.

55 4. A process of producing ethyl alcohol of greater than 95% concentration free from fusel oil comprising contacting the vapor of an aqueous alcoholic solution containing fusel oil and at least 10% water with an absolute alcoholic solution of potassium and sodium acetate, drawing  
60 off and condensing the alcohol vapors free from fusel oil and retaining the fusel oil with the salt solution.

65 5. A process of producing ethyl alcohol of greater than 95% concentration free from fusel oil comprising contacting the vapor of an aqueous alcoholic solution containing fusel oil and at least 10% water with a solution containing about 70% potassium acetate and about 30% sodium  
70 acetate, drawing off and condensing the alcohol vapors free from fusel oil and retaining the fusel oil with the salt solution.

75 6. A process of producing ethyl alcohol of greater than 95% concentration free from fusel oil comprising contacting the vapor of an aqueous alcoholic solution containing fusel oil and at least 10% water with an absolute alcoholic solu-

tion containing about 70% potassium acetate and about 30% sodium acetate, drawing off and condensing the alcohol vapors free from fusel oil and retaining the fusel oil with the salt solution.

7. A process of producing ethyl alcohol of greater than 95% concentration free from fusel oil comprising distilling the crude spirit whereby an aqueous alcoholic solution containing fusel oil and at least 10% water in the vapor phase is obtained, then contacting the vapors with an absolute alcoholic solution of a dehydrating fatty acid salt, drawing off and condensing the alcoholic vapors free from fusel oil and retaining the fusel oil with the salt solution.

8. A process of producing ethyl alcohol of greater than 95% concentration free from fusel oil comprising distilling the crude spirit whereby the vapor of an aqueous alcoholic solution containing fusel oil and at least 10% water is obtained, then subjecting the vapor in counter-current flow to the action of an absolute alcoholic solution of a dehydrating fatty acid salt, drawing off and condensing the alcohol vapor free from fusel oil and retaining the fusel oil with the salt solution.

9. A process of producing ethyl alcohol of greater than 95% concentration free from fusel oil comprising distilling the crude spirit whereby the vapor of an aqueous alcoholic solution containing fusel oil and at least 10% water is obtained, then subjecting the vapor in counter-current flow to the action of an absolute alcoholic solution of anhydrous alkali acetates, drawing off and condensing the alcohol vapor free from fusel oil and retaining the fusel oil with the salt solution.

10. A process of producing ethyl alcohol continuously of greater than 95% concentration free from fusel oil comprising contacting the vapor of an aqueous alcohol solution containing fusel oil with an absolute alcoholic solution of dehydrating fatty acid salt, drawing off and condensing the alcohol vapors free from the fusel oil, removing the fusel oil together with the salt solution and small quantities of alcohol, removing from this mixture the fusel oil and alcohol, separating off the fusel oil from the alcohol, dehydrating the residual salt solution in a molten state and dissolving the dehydrated salt in the same molten state in absolute alcohol for reuse as contact material.

11. A process of producing ethyl alcohol continuously of greater than 95% concentration free from fusel oil comprising contacting the vapor of an aqueous alcohol solution containing fusel oil with an absolute alcoholic solution of dehydrating fatty acid salt, drawing off and condensing the alcohol vapors free from the fusel oil, removing the fusel oil together with the salt solution, separating the fusel oil from the salt solution, dehydrating the residual salt solution and dissolving the same in absolute alcohol for reuse in the process.

12. A process of producing ethyl alcohol continuously of greater than 95% concentration free from fusel oil comprising contacting the vapor of an aqueous alcohol solution containing fusel oil with a water-free solution inert to alcohol and fusel oil of alkali acetates, drawing off and condensing the alcohol vapors free from the fusel oil, removing the fusel oil together with the acetate solution, separating the fusel oil from the residual acetate solution, dehydrating the acetate

solution and dissolving the same in a solvent for reuse in the process.

13. A process of producing ethyl alcohol continuously of greater than 95% concentration free from fusel oil comprising contacting the vapor of aqueous alcohol solution containing fusel oil with an absolute alcoholic solution of potassium and sodium acetates, drawing off and condensing the alcohol vapors free from the fusel oil, removing the fusel oil together with the salt solution and small quantities of alcohol, removing from this mixture the fusel oil and alcohol, separating off the fusel oil from the alcohol, returning the alcohol separated from the fusel oil to the contacting step, dehydrating the salt solution in a molten state and dissolving the dehydrated salt solution in the molten state in absolute alcohol for reuse in the process.

14. A process of producing ethyl alcohol continuously of greater than 95% concentration free from fusel oil comprising contacting the vapor of aqueous alcohol solution containing fusel oil with an absolute alcoholic solution of 70% potassium acetate and 30% sodium acetate, drawing off and condensing the alcohol vapors free from the fusel oil, removing the fusel oil together with the salt solution and small quantities of alcohol, removing from this mixture the fusel oil and alcohol, separating off the fusel oil from the alcohol, returning the alcohol separated from the fusel oil to the contacting step, dehydrating the salt solution in a molten state and dissolving the dehydrated salt solution in the molten state in absolute alcohol for reuse in the process.

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