

March 5, 1929.

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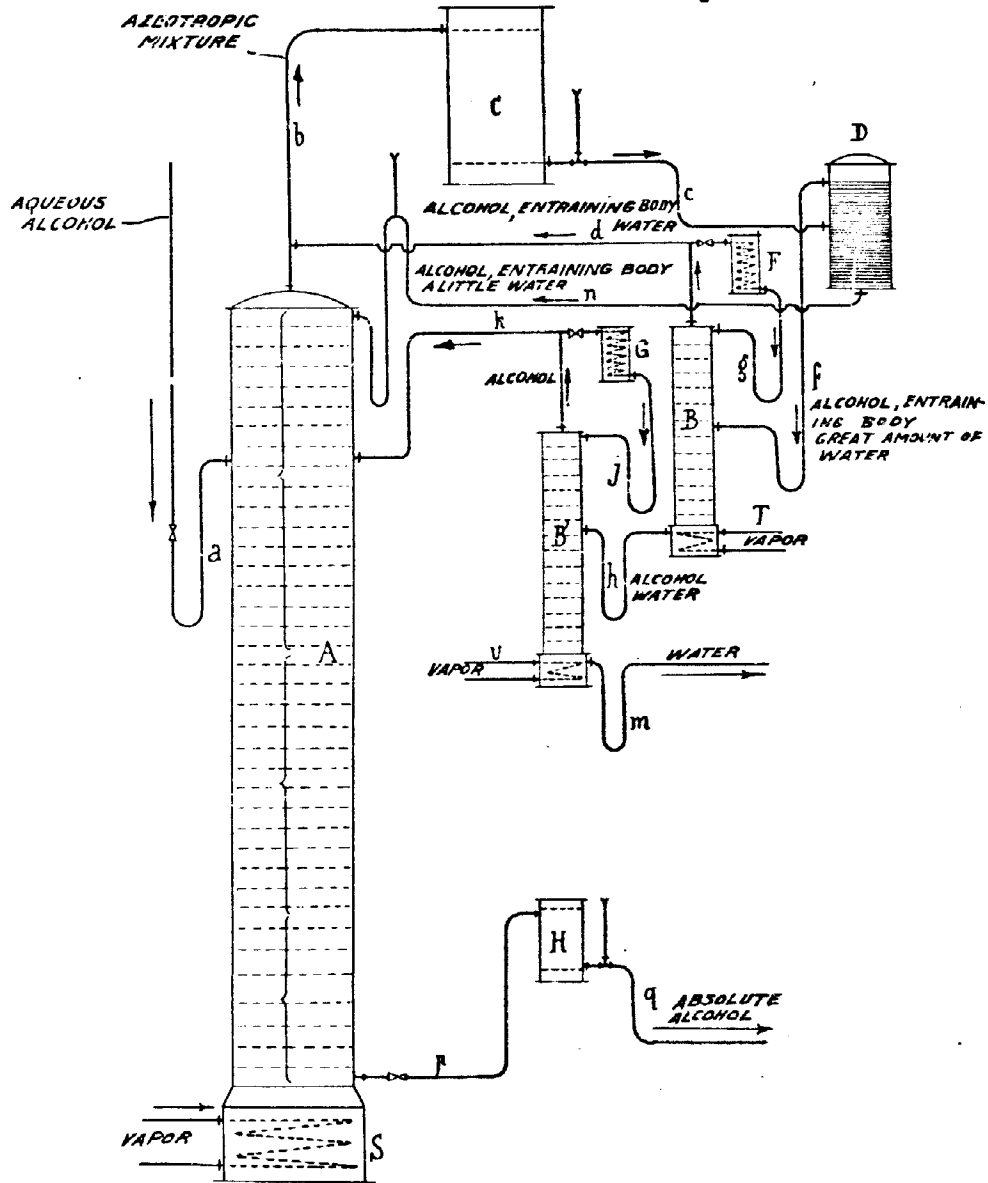
1,704,213

APPARATUS FOR THE CONTINUOUS MANUFACTURE OF ABSOLUTE ALCOHOL

Filed Jan. 23, 1924

4 Sheets-Sheet 1

Fig. 1



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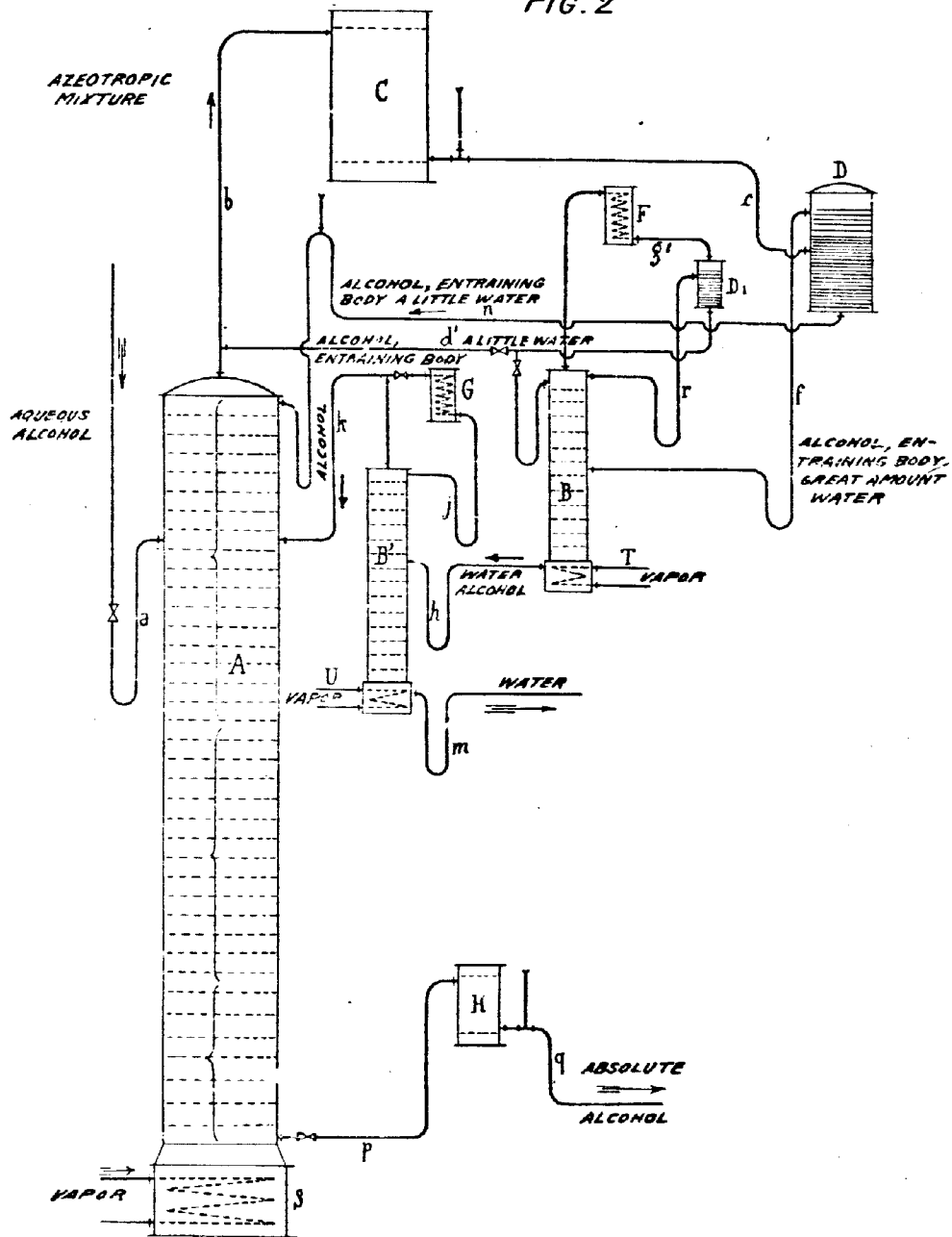
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4 Sheets-Sheet 2

FIG. 2



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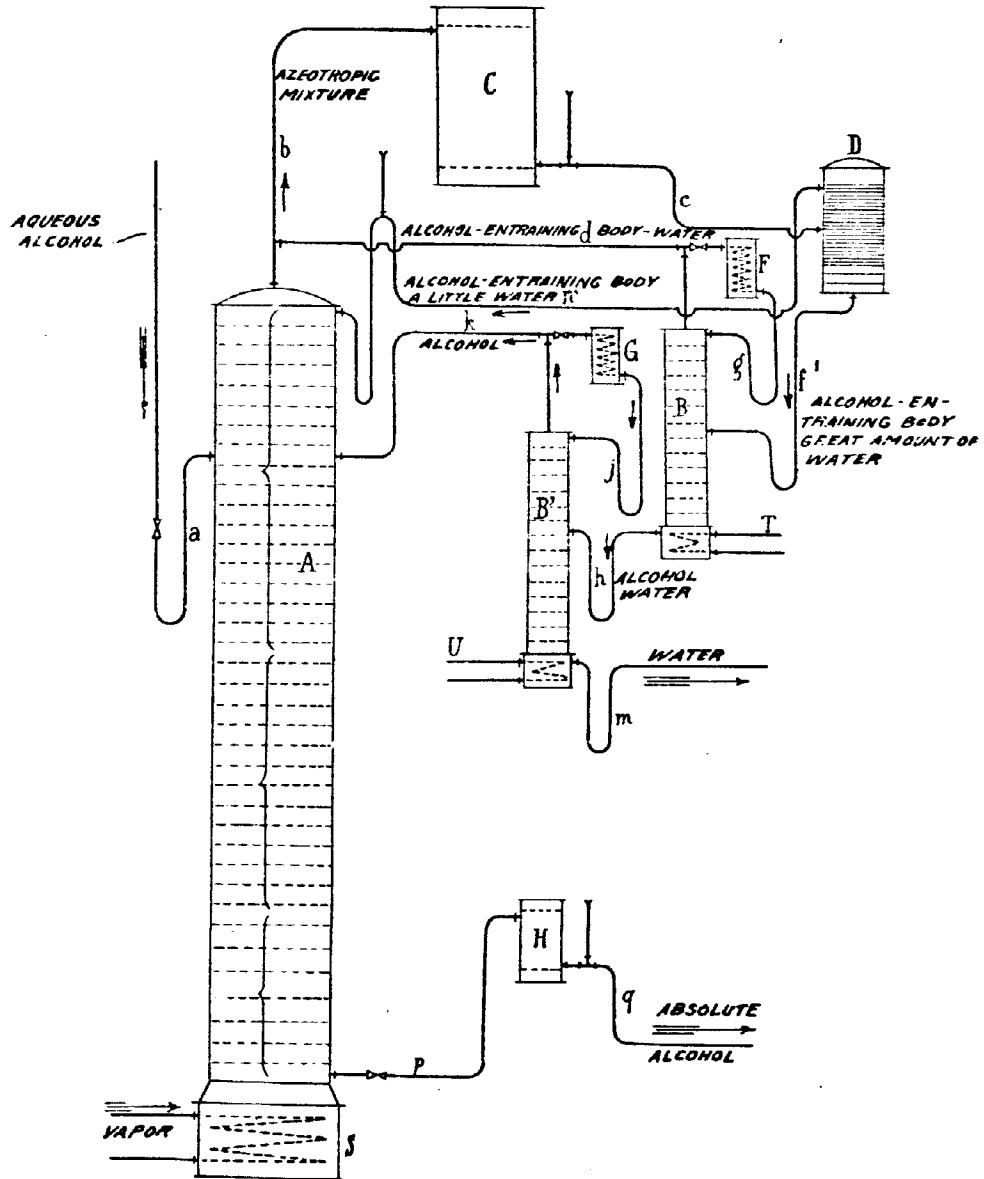
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4 Sheets-Sheet 3

FIG. 3



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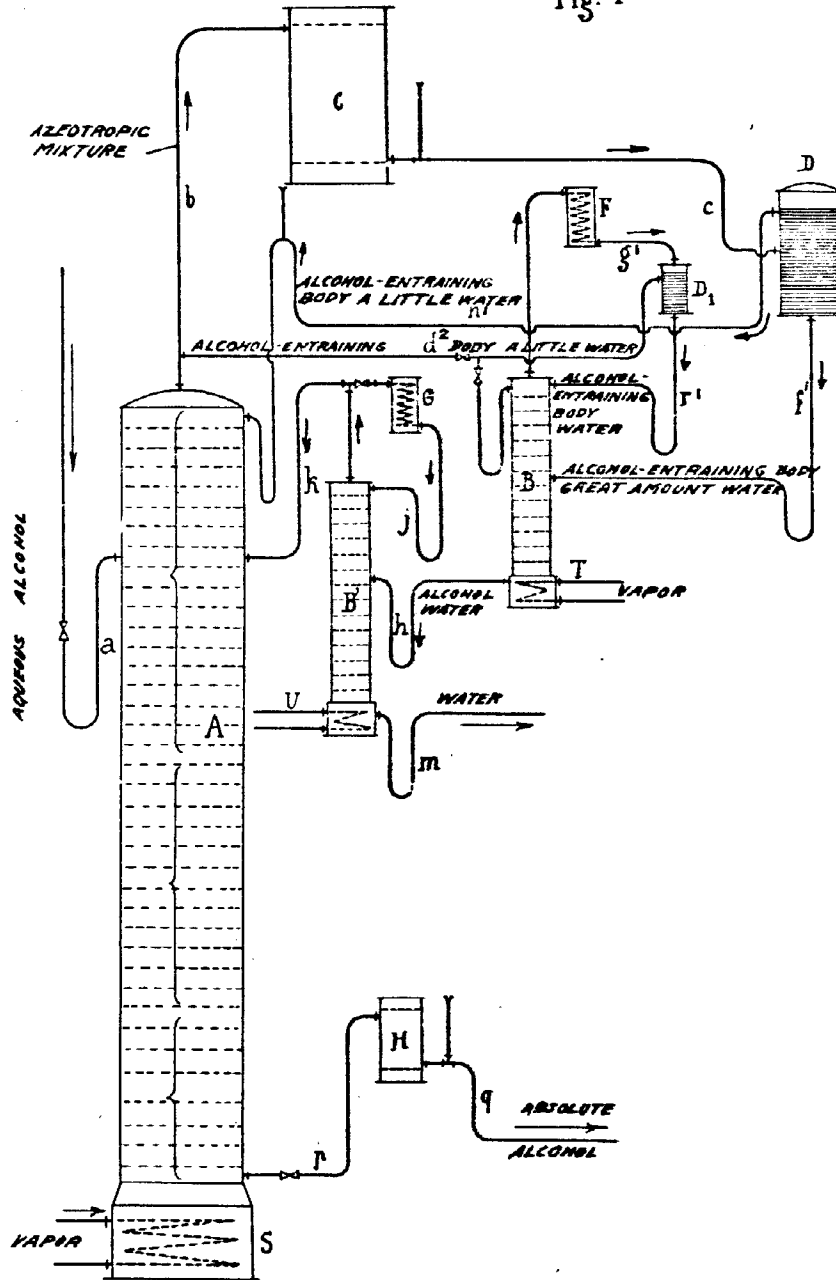
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4 Sheets—Sheet 4

Fig. 4



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UNITED STATES PATENT OFFICE.

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APPARATUS FOR THE CONTINUOUS MANUFACTURE OF ABSOLUTE ALCOHOL.

Application filed January 23, 1924, Serial No. 688,050, and in Belgium April 16, 1925.

It is observed that upon adding to commercial alcohol (ethyl alcohol containing generally from 6 to 8 per cent of water) certain liquid substances which are not miscible with water but are miscible with alcohol, mixtures are formed which distill at a lower temperature than the distillation temperatures of the constituent bodies. Such mixtures are termed "azeotropic", and I will herein designate the liquid substances producing such effects as "entraining bodies".

The vapours of these mixtures have a substantially constant composition, and when condensed they form a liquid which separates as a rule into two non-miscible layers, each layer containing the three components water, alcohol and "entraining body" in very different proportions; in fact one of the layers contains nearly all the water and the second nearly the whole amount of the entraining body.

It is evident that in this manner the small percentage of water can be removed from commercial alcohol by distilling it in the presence of an entraining body and a process for this purpose is disclosed in my copending application Serial No. 688,049, filed January 23, 1924.

With this principle as a base, I have invented an arrangement of apparatus for the continuous dehydration of commercial alcohol, comprising the following essential elements:

1. An upright distilling chamber with surface steam heating.
2. A condenser for the condensation of all the vapour produced by the said distilling chamber.
3. A decantation chamber in which the liquid from the condenser becomes separated into two layers.
4. A small upright distilling chamber with surface steam heating—provided with a condenser and optionally with a decantation chamber—adapted to receive the ternary layer having a large percentage of water which is discharged from the main decanting

chamber and to remove the entraining body therefrom.

5. Another upright distilling chamber with surface steam heating—provided with a condenser—intended to receive the binary mixture of water and alcohol issuing from the last-mentioned distilling chamber and to separate the water from the alcohol.

6. A refrigerating condenser for absolute alcohol, adapted to cool the absolute alcohol which is discharged from the bottom of the main distilling chamber.

Each of these essential elements is provided with the usual accessories, and the said elements are connected together according to the nature of the entraining body and in conformity to usual practice.

The following description, with reference to the appended drawings which are given by way of example, sets forth the present invention.

Figs. 1 to 4 shows the said apparatus, with slight modifications according to the different cases which may occur.

First example.—The layer of ternary liquid containing a large percentage of water is in this case the upper layer.

The plant shown diagrammatically in Fig. 1 consists of an upright distilling chamber A which is heated at the bottom by a steam coil S. The commercial alcohol is supplied in a regular manner by the pipe *a*. A certain amount of the entraining body is preliminarily disposed in the said distilling chamber. Under the action of the heat, the vapour produced in the upper part of the chamber will pass through the pipe *b* into the condenser C in which it is condensed. The liquid produced by this vapour passes through the pipe *c* into the decantation chamber D in which it separates into two layers. The upper layer is evacuated through the pipe *f* and enters the chamber B which is heated by the steam coil T; the liquid distilled in the latter chamber will produce vapour whereof one portion is condensed in the condenser F and returns to the chamber B through the pipe *g*, and an-

other portion passes through the pipe *d* and enters the condenser C. A binary mixture of water and alcohol flows from the bottom of the chamber B through the pipe *k* into the chamber B¹ which is heated by the steam coil U, and the resulting vapour will be condensed in part in the condenser C and will return to the chamber B¹ through the pipe *j*, whilst another part of the vapour passes through the tube *k* into the chamber A. The water is discharged from the bottom of the chamber B¹ through the pipe *m*.

The lower layer of the liquid in the decanting chamber will pass through the pipe *n* into the upper part of the chamber A. The absolute alcohol discharged as vapour or liquid from the bottom of the chamber A through the pipe *p* is cooled by the refrigerating condenser H and is evacuated through the pipe *g*.

As a general result, it is observed that the commercial hydrated alcohol is supplied in a regular manner to the chamber A by the pipe *a*, and a regular discharge of absolute alcohol takes place through the pipe *g*, and of water through the pipe *m*.

Second example.—The layer of ternary liquid containing a large percentage of water is in this case the upper layer, and the plan will be accordingly modified, as shown in Fig. 2.

The operation, in the chamber A and its accessory elements is performed as in the preceding case, but it is differently performed in the chamber B. The vapours condensed in the condenser F will produce a liquid which flows through the pipe *g'* into the auxiliary decantation chamber D¹; the liquid is therein separated into two layers, and the upper layer of the azeotropic mixture, which contains the larger percentage of water, is entirely returned through the pipe *r* to the chamber B; the lower layer of said mixture containing the smaller percentage of water is discharged in part into the chamber B through the pipe *r* and in part into the chamber A through the pipe *d'*.

Third example.—The layer of ternary liquid having a large percentage of water is now the lower layer, and the plant is accordingly disposed as in Figs. 3 and 4.

In this case the lower layer produced in the decantation chamber D, Figs. 3 and 4, is discharged into the chamber B whilst the upper layer is returned to the main chamber A through the pipe *n'*.

The chamber B may be provided solely with the condenser F as in Example I (Fig. 3) or with an additional decantation chamber D (Fig. 4) as in Example II.

With respect to the three examples given above, it is pointed out that the layer containing the greater amount of water may be the upper layer or the lower one, according to the nature of the entraining body em-

ployed. The layer containing the major portion of the entraining body, when this entraining body has a high specific gravity, forms the lower layer and, in this event, the more aqueous layer is at the top. This occurs for instance with trichlorethylene and tetrachloride of carbon. When the entraining body has a low specific weight, it forms the upper layer, and the aqueous layer becomes the lower layer. This is the case, for example, with gasoline and butyl chloride.

What I claim is:—

1. An apparatus for the continuous production of absolute alcohol, comprising a main distilling column adapted for the distillation of a mixture of alcohol, water and an entraining body forming an azeotropic mixture having a minimum boiling point, means for heating the main distilling column, a condenser connected to the top of said column for the condensation of the vapors discharged from the latter, a decantation chamber connected with said condenser and adapted to separate out two liquid layers from the distillate, a second distillation column connected with the decantation chamber adapted to receive the liquid layer from the decantation chamber which contains the greater percentage of water, means for heating the second distillation column, a condenser connected to the top of said second distillation column by a duct so as to receive vapor therefrom, a third distillation column connected with the bottom of the second distillation chamber adapted to receive a liquid from the second distillation column, a pipe leading from the top of the third distillation column to the main distilling column in its upper portion, a pipe connecting with said duct to convey vapors from the second distillation column to the said azeotropic mixture, and a pipe connecting the decantation chamber with the main distilling column near the top thereof.

2. An apparatus for the continuous production of absolute alcohol comprising a main distilling column adapted for the distillation of a mixture of alcohol, water and an entraining body forming an azeotropic mixture having a minimum boiling point, means for heating said column, a condenser connected to the top of the main distilling column for the condensation of the vapor discharged from the latter, a decantation chamber connected with said condenser and adapted to separate out two liquid layers from the distillate, a second distillation column connected with the decantation chamber adapted to receive the liquid layer from the decantation chamber which contains the greater percentage of water, means for heating said second distillation column, a condenser and a decantation chamber connected with the top of the second distillation column by a duct so as to receive vapor there-

from, a third distillation column connected with the bottom of the second distillation column adapted to receive a liquid from the second distillation chamber, a pipe leading
5 from the top of the third distillation column connected with the main distilling column in its upper portion, a pipe connecting with said duct to convey vapors from the second distillation column to the said azeotropic mixture, and a pipe leading from the decan- 10 tation chambers and connected with the main distilling column near the top thereof.

In testimony whereof I have signed this specification.

ELOI RICARD.