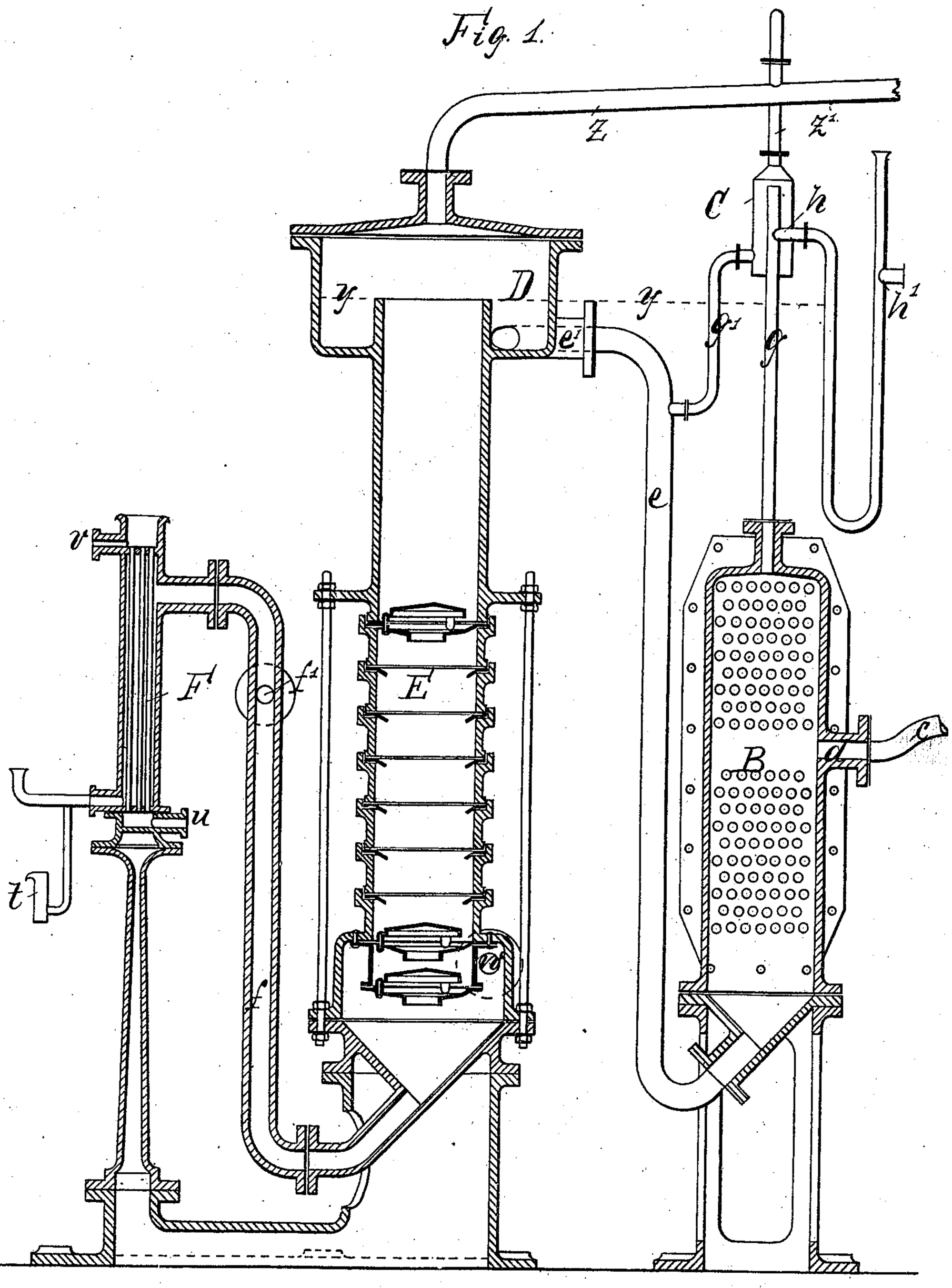
(No Model.) 2 Sheets—Sheet 1.

R. ILGES.

DISTILLING MASH.

No. 502,079. Patented July 25, 1893.

Fig. 1.



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(No Model.)

2 Sheets—Sheet 2.

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Fig. 3.

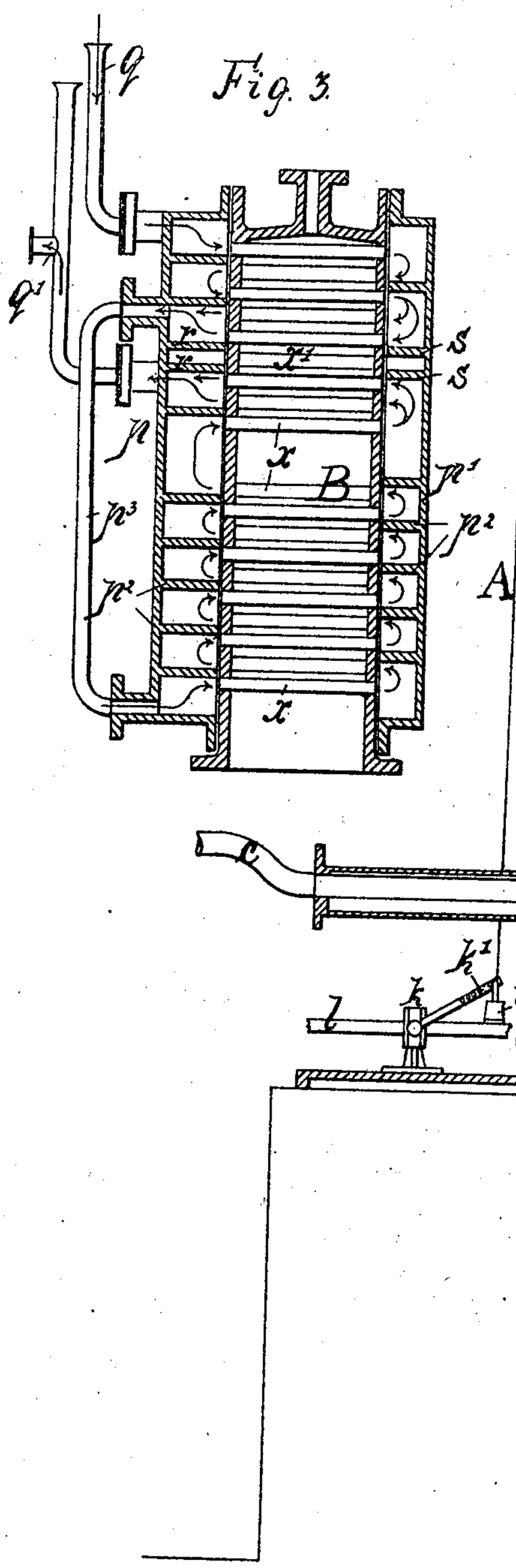


Fig. 2.

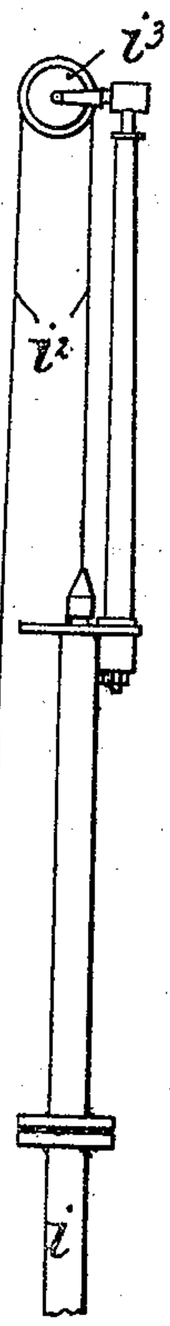
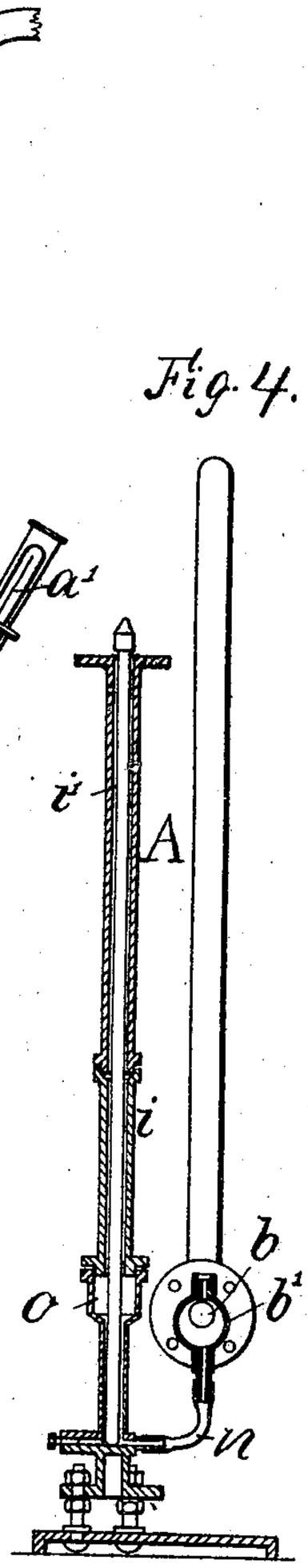


Fig. 4.



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UNITED STATES PATENT OFFICE.

ROBERT ILGES, OF COLOGNE-BAYENTHAL, GERMANY.

DISTILLING MASH.

SPECIFICATION forming part of Letters Patent No. 502,079, dated July 25, 1893.

Application filed October 11, 1892. Serial No. 448,587. (No model.) Patented in Germany February 12, 1889, No. 48,343, June 3, 1890, No. 55,666, and October 29, 1890, No. 60,399; in Austria-Hungary December 29, 1891, No. 41,556 and No. 73,555, and in Russia May 1, 1892, No. 5,314.

To all whom it may concern:

Be it known that I, ROBERT ILGES, a subject of the King of Prussia, German Emperor, and a resident of Cologne-Bayenthal, in the Province of the Rhine, Kingdom of Prussia, Empire of Germany, have invented certain new and useful Improvements in Distilling Mash for the Purpose of Obtaining Highly-Concentrated Fusel-Oil and Purified Alcohol, (for which I have obtained Letters Patent in Germany, No. 48,343, dated February 12, 1889, No. 55,666, dated June 3, 1890, and No. 60,399, dated October 29, 1890; in Russia, No. 5,314, dated May 1, 1892, and in Austria-Hungary, No. 41,556 and No. 73,555, dated December 29, 1891,) of which the following is a full, clear, and exact specification.

My invention relates to improvements in distilling mash, and more particularly to the last treatment of the singlings or phlegm left in the rectificator of a mash distilling apparatus, the object being to effect a continuous and direct separation of highly concentrated fusel-oil and purified alcohol.

My invention therefore consists in an operation performed as the last step of the distillation, and composed of three distinct parts; the first part has for its object to produce and to maintain in the singlings such a proportion of alcohol as will be most favorable for the separation of fusel-oil, and I prefer to effect this by keeping the singlings at the boiling temperature which corresponds to the desired proportion of alcohol; the second part of the process consists in cooling the boiling singlings, with a view to further reducing their capacity of dissolving fusel-oil and facilitating the separation of the latter; and lastly, by the third part of my improved process I effect the continuous removal of the accumulated fusel-oil without any simultaneous outflow of singlings.

In order to fully disclose my invention I will proceed to describe the same specifically, referring to the accompanying drawings, which represent an apparatus suited for the execution of my improved process, and in which—

Figure 1, Sheet I, is a longitudinal sectional elevation of the left part of the apparatus. Fig. 2, Sheet II, is the continuation of Fig. 1. Fig. 3 is a detail cross sectional elevation of the cooler B. Fig. 4 is a similar view of the temperature-regulator A.

Similar parts are designated by similar letters throughout the several figures.

The apparatus consists first of the temperature regulator A, composed of two horizontal tubes *b b'*, the former, which is preferably made of copper, being inclosed within the latter, which consists of wrought iron. The tube *b* connects with an upright pipe *a*, fitted with a thermometer *a'*, the tube *b'* with an upright tube *i*, the latter connection being effected by means of an india-rubber tube *n*. The tube *b'* is perfectly closed at both ends. *m* is the bed-plate to which the tube *i* is secured by means of three screws, so as to be easily adjusted to an absolutely vertical position. Tube *i* is provided with a widened portion, forming a small chamber *o*. Within this upright tube *i* I place a float *i'* which is preferably constituted by a tube of glass containing a small quantity of mercury. At the upper end of the float *i'* there is secured a string or wire i^2—preferably a silk cord—which is passed over a roll i^3 and has a weight i^4 fastened to its other end so as to counterbalance the weight of the float *i'*. This weight is also connected to the handle *k'* of a cock, damper, or throttle-valve *k* adapted to regulate the supply of water through pipe *l* to the cooler of the dephlegmator.

The communication between the temperature-regulator A and the cooler B is effected by the bend *c* of the tube *b*, this bend being directed upward for a purpose stated hereinafter. The tube is fitted to the socket *d*, located about at medium height of the cooler B. The latter is a cast-iron vessel of square cross-section, and is provided with a series of horizontal tubes *x* which are inserted in holes made in opposite walls of the cooler.

p p' are water chambers secured to the cooler B, and are divided into a series of compartments by horizontal partitions p^2 placed above one another in steps in such a manner that the compartments of each chamber are

separated by partitions placed at a height intermediate between that of the partitions of the other chamber. Two of these partitions, *r r* and *s s*, are however placed at the same height in both chambers, so that the row of tubes lettered *x'* is entirely disconnected from the other tubes *x*. It will be seen by a glance at Fig. 3 that a continuous zig-zag path is created by the tubes *x*, the partitions p^2 of the chambers *p* and *p'*, and a tube p^3 connecting the compartment situated directly above the upper partition *r* and the undermost compartment of chamber *p*.

q is the inlet-pipe communicating with a water supply pipe or with a reservoir, and *q'* is the outlet pipe. It will be noticed that there are no tubes *x* in the middle part of the cooler B, where the socket *d* is fitted to the same. At the bottom of the cooler there is a chute from which a pipe *e* leads to a socket *e'* terminating tangentially into an annular chamber D, at the top of the distilling apparatus proper E.

g is an upright tube fitted centrally into the top of the cooler B, the upper end of this tube is inclosed within a cylindrical chamber C and connects laterally with a loop-pipe *h*, provided with an outlet socket *h'*; the latter ought to be located considerably above the level *y—y* of the annular chamber D. The top of the chamber C has an upright pipe *z'* inserted into it centrally, and communicates thereby with pipe *z*, fitted to the top of the chamber D.

E is a distilling apparatus or tower of the kind employed in the distillation of alcohol, and is provided with a pipe *w* inserted tangentially, to supply steam. At the bottom of this apparatus there is a chute from which a pipe *f*, provided with a lateral socket *f'*, leads into a cooler F constructed with vertical tubes in which water may enter from below through the socket *u* and from which it may flow off by the socket *v*.

A collecting-vessel *t* connects with the lower part of the cooler F and is adapted to receive the material having passed through the same.

I wish to have it understood that the described apparatus is combined with a mash-distilling apparatus of any suitable construction and will be substituted in such apparatuses for that part in which the phlegm left after the rectification of the alcohol is subjected to a last distillation. The pipes *a* and *z* are connected to the rectificator.

My improved process is based on the following considerations: The singlings to be treated according to that process contain alcohol and products of fermentation of minor volatility, that is to say, fusel-oil; for it may be taken for granted that the singlings as a rule do not contain highly volatile compounds, and if they do exceptionally, this is due to chemical transformations, especially of the fusel-oil, occasioned by a defective construction of the distilling apparatus. It is therefore sufficient, according to the principle of my improved method, to separate the substances of minor volatility, *i. e.* the fusel-oil, in order to obtain pure alcohol. Now in any distilling apparatus of rational construction, that is to say, effecting an energetic dephlegmation and rectification, the entire amount of fusel-oil may be easily separated from the alcohol vapors before the latter are led to the cooler; this fusel-oil is then condensed and dissolved in highly concentrated singlings. In order to separate the fusel-oil, its proportion in the singlings must be steadily increased; this is effected during the passage of the singlings through the rectificator, by the continuous vaporization, as alcohol is considerably more volatile than fusel-oil. This favorable effect is however observed only as long as the volumetrical proportion of alcohol is in excess of about seventeen per cent. of the singlings; if the proportion is less, the fusel-oil will not be dissolved in the singlings, as desired, but vaporized in the rectificator. The conclusion drawn from the above facts is that the separation of the fusel-oil succeeds best, as long as the volumetrical proportion of alcohol is in excess of about seventeen per cent. of the singlings, and, according to my experiments made hitherto, the most favorable proportion appears to be twenty-one to twenty-four per cent., while the possibility of the separation of fusel-oil is varying between about twenty and twenty-eight per cent. The first step of my improved process consists therefore in constantly keeping the proportion of alcohol contained in the singlings within the mentioned favorable limits, and I prefer to effect this in an indirect manner, by keeping the singlings at the boiling temperature which corresponds to the desired proportion of alcohol. The second part of my improved process consists in the cooling of the boiling singlings, already flown through the temperature-regulator, whereby the solubility of fusel-oil in the singlings is diminished and the separation of the fusel-oil therefore facilitated. The cooling of the singlings effects the separation of the fusel-oil already at a higher volumetrical poroportion of alcohol, and the most favorable proportion of alcohol is twenty-four to twenty per cent., a cooling to 88—68° Fahrenheit being provided, while the possibility of the separation of fusel-oil lies between about twenty and thirty-five per cent. The part of the apparatus, appertaining to this process, is the cooler B. My improved process is therefore performed as follows: The boiling singlings left in the rectificator of a mash-distilling apparatus are let in through pipe *a* and are caused to flow continuously through the tube *b*, the bend *c*, the socket *d*, cooler B, pipe *e*, socket *e'*, distilling tower E, pipe *f* and socket *f'*. Before starting the operation the tube *b'* is filled with mercury up to the tube *b*, and the remaining space with strong alcohol or any other liquid boiling at a low temperature. Now the heat of the boiling singlings flowing through tube

b will cause the mercury and alcohol placed in tube *b'* to expand; thus the mercury, the level of which was initially the same in tube *b'* and the vertical tube *i*, will rise in the latter and fill the small chamber *o* provided for this purpose. Gradually the alcohol in tube *b'* will be heated to its boiling temperature, and the vapors evolved will cause the mercury to rise in tube *i* to such a height as to lift the float *i'*. According to the fluctuations in the temperature of the singlings, the float *i'* will oscillate up and down. This movement will be transmitted to the handle *k'* of the throttle-valve *k* by means of the silk cord i^2, and the admission of water through pipe *l* to the cooler of the dephlegmator will be increased or reduced accordingly in such a manner as to regulate the temperature of the singlings and to keep it constantly at the required point. It will be obvious that thereby a constant proportion of alcohol is maintained simultaneously.

It is desirable to prevent as much as possible the heat of the singlings from being communicated to the upright tube *i*, and therefore the connecting tube *n* is made of india-rubber or any other good non-conductor of heat. It will be obvious that the same effect, viz., keeping the temperature of the singlings at a constant point, may be obtained by regulating the supply of steam or mash to the mash-distilling apparatus. It will be further obvious that, when regulating the supply of steam to the mash distilling apparatus, the passage of the throttle-valve *k* is to be narrowed when the temperature of the singlings increases, whereas it is to be widened with rising temperature when regulating the supply of water to the cooler of the dephlegmator. It will be easily understood that the regulation of the supply of mash to the mash-distilling apparatus may be effected in an analogous manner. The singlings, which already contain fusel-oil in a separated state, owing to the keeping up of a temperature which highly favors the evaporation of alcohol, flow then through the bend *c*. This bend is directed upward in order to permit the unhindered flow of the specifically lighter fusel-oil to the cooler B. If the pipe were straight, the singlings cooled in the cooler B would partially flow back in the lower half of this pipe into the temperature-regulator, *i. e.* into the tube *b*, whereby the action of this regulator would be influenced unfavorably. If the bend *c* were directed downward, a large portion of tube *b* would remain filled with fusel-oil, whereby the transmission of heat from the singlings to the mercury and alcohol in tube *b'* would be impaired. The singlings entering the cooler B by the socket *d* may spread uniformly in the middle part of the cooler, which is free of tubes, and sink gradually into the lower part of the same. During this downward movement the singlings are cooled by the water which is made to circulate through the tubes *x* and the compartments of the chambers *p* and *p'*; thereby the remainder of fusel-oil dissolved in the singlings is separated, and being specifically lighter rises to the surface of the same. Now as the upper layers of the singlings are still warm, the rising globules of fusel-oil will be partly dissolved, and it is necessary to cool them again energetically in order to effect their complete separation. For this purpose the cooling water is caused to enter at the top of the cooler B, as described, so that the upper layers of the singlings will be cooled most energetically. In order to prevent a mixture of this strongly cooled layer with the layer of boiling singlings in the middle of cooler B, a layer of medium or neutral temperature is created between the same by placing one row of horizontal tubes *x'*, out of the circuit through which water circulates. It will be seen by reference to Fig. 3 and to the arrows marked thereon that the particles rising from the hottest [boiling] layer of the singlings are cooled by the water in several rows of tubes *x* before getting to the neutral zone at the level of the tubes *x'*. In consequence of the described arrangement the globules of fusel-oil are fully separated from the singlings in the upper part of the cooler B and being in a highly concentrated state rise in the oil-tube *g*. As the socket *h* for the outflow of fusel-oil is located a considerable distance above the highest level to which the singlings ascend, *i. e.* the upper termination *y—y* of the chamber D, it will be evident that the singlings cannot mingle with the fusel-oil flowing off through the loop-pipe *h'* into a suitable closed reservoir. The singlings cooled in the cooler B rise in the pipe *e* and are let into the chamber D below the line *y—y*, *i. e.* the level of the liquid, in order to avoid changes in the pressure exerted on the surface of said liquid. This pressure is obtained by steam entering the singlings-distilling tower E through the socket *w*. It is of great importance to constantly produce an equal pressure on the surface of the liquids in the communicating tubes *g* B and *e* D. This pressure is exerted in chamber D upon the surface of the singlings, and in chamber C upon the surface of the fusel-oil accumulated in pipe *g*. The singlings in chamber D are heated directly by the steam rising in the tower E and indirectly by the heat absorbed and transmitted by the walls of the tower. In order to prevent the heated singlings from commingling with the cooled material entering through the socket *e'*, the latter is fitted tangentially to the chamber D, so that the singlings are brought to move on with a uniform rotating motion. In the other branch of the communicating tubes the same precautions must be taken for insuring a gradual heating of the cooled fusel-oil rising in pipe *g*. The steam and the alcohol vapors proceeding from the tower E enter into the chamber C through the tubes *z* and *z'* and heat the upper end of the tube *g*,

and consequently the fusel-oil contained in the same, before coming in direct contact with the said fusel-oil. The condensed steam and vapors do not mix with the fusel-oil, but flow back to the pipe *e* through the tube *g'*. In this manner the high concentration of the fusel-oil is preserved. The singlings flowing into the tower E over the inner rim of chamber D, and freed from their contents of fusel-oil, as described, are completely freed from the alcohol which they still contain by the steam admitted through socket *w*. The remaining watery solution is let off through pipe *f* and socket *f'*. The vapors evolved from this solution, which is in a superheated condition, are led into the cooler F of a singlings testing apparatus to be condensed there. The condensed liquid flows in a small continuous stream into the collecting-vessel *t*, where its contents of alcohol are ascertained by means of an alcoholmeter. The cooling water is caused to circulate from below to above, entering through the socket *u*, and flowing off through the socket *v*. The vapors generated in the tower E, which are free from fusel-oil, are conveyed to the rectificator of the mash distilling apparatus through the pipe *z*.

By the three steps of my improved process the fusel-oil is separated from the singlings, as described, and removed from the apparatus, so that the amount of fusel-oil contained in the rectificator and dephlegmator is reduced, and decompositions of these products of fermentation prevented. Thus the ethylic alcohol contained in the mash is obtained without any admixture of impurities.

What I claim as my invention, and desire to secure by Letters Patent of the United States, is—

1. As an improvement in the distillation of mash, the herein described process for separating the fusel-oil from the singlings proceeding from the rectificator of a mash distilling apparatus, consisting in boiling said singlings at a constant temperature, which corresponds to the volumetrical proportion of alcohol and to the pressure within the distilling apparatus, and in cooling the boiling singlings thereafter for the purpose set forth.

2. As an improvement in the distillation of mash, the herein described process for separating the fusel-oil from the singlings proceeding from the rectificator of a mash distilling apparatus, which consists in keeping the said singlings at a constant boiling temperature corresponding to a volumetrical proportion of alcohol somewhat exceeding seventeen per cent., and in cooling the singlings thereafter, for the purpose set forth.

3. As an improvement in the distillation of mash, the herein described process for separating the fusel-oil from the singlings proceeding from the rectificator of a mash distilling apparatus, which consists in keeping the said singlings at a constant boiling temperature corresponding to a volumetrical proportion of alcohol somewhat exceeding seventeen per cent., and in cooling the singlings thereafter, and in utilizing the inferior specific weight of the fusel-oil for separating and drawing it off in a continuous manner, substantially as and for the purpose described.

In testimony whereof I have signed this specification in the presence of two subscribing witnesses.

ROBERT ILGES.

Witnesses:
FRITZ SCHROEDER,
EVA HAUSEN.