May 17, 1949. A. A. ANDREASEN ET AL 2,470,230

DISTILLATION VAPOR LOSS DETECTOR

Filed Oct. 25, 1944

Fig. 1

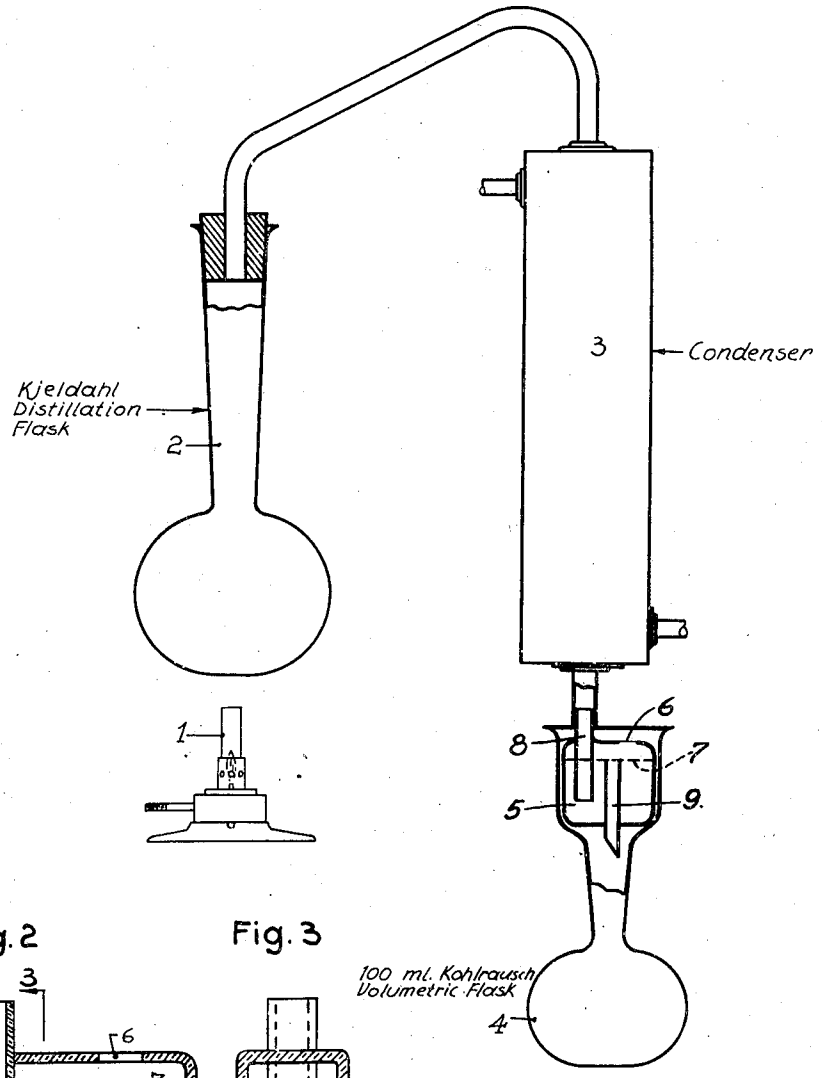


Fig. 2

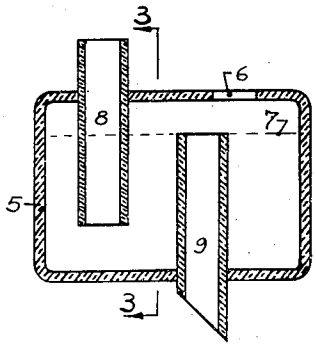
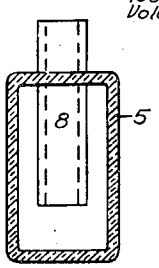


Fig. 3



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DISTILLATION VAPOR LOSS DETECTOR

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1 Claim. (Cl. 202—185)

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In distillery and other laboratories, where it is desirable from time to time to determine the alcoholic content of a given liquid such as distillers' beer, it is the practice first to distill the alcohol from a beer sample, condense the distillate and then measure it to determine the amount of alcohol originally contained in the sample, and second to repeat this procedure one or more times with one or more other samples of the same beer in order to check the results. It is desirable to perform these operations as rapidly as possible but care must be exercised to avoid conducting the distillation at a rate greater than the capacity of the condenser to condense the vapors, because the uncondensed vapors will be lost producing an erroneous (and low) indication of yield. Heretofore, a vapor loss could be avoided with certainty only by slowing up the distillation rate to a point such that the condenser operated safely below its capacity.

The present invention relates to distillations of this character and proposes a novel, simple, inexpensive and sensitive arrangement for visually indicating to the operator, at a glance, excessive distillation rates ranging upwardly from the barest minimum, thereby enabling the operator to maintain the distillation rate, by manual regulation, at an effective value, corresponding substantially to the maximum capacity of the condenser, without incurring appreciable vapor loss.

The invention is illustrated in the drawing wherein:

Figure 1 discloses an arrangement for distilling the beer, condensing the distillate and collecting the condensate in which the invention is embodied;

Figure 2 is a vertical section through the major plane of one form of a vapor loss detector used in carrying out the invention; and

Figure 3 is a section along line 3—3 of Figure 2.

The arrangement shown in Figure 1 conventionally includes: a source of distillation heat 1 in the form of a Bunsen burner; a Kjeldahl distillation flask 2; a condenser 3; and a 100 ml. Kohlrausch volumetric condensate flask 4. These parts are connected together so that the distillate from the distillation flask 2 enters the condenser 3 while the condensate from condenser 3 is collected in the volumetric flask 4.

In the conventional operation of this arrangement, exactly 200 mls. of beer are measured off in a volumetric flask and transferred to the distillation flask 2. The volumetric flask is washed with 100 mls. of water divided in three or four portions and the washings added to the contents

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of the distillation flask 2. The 100 ml. volumetric condensate flask 4 may be provided with 10 mls. of distilled water and the distillation instituted slowly and continued until the level in the condensate flask 4 reaches the 100 ml. mark. A repetition of these operations is preferably conducted in a duplicate setup to produce a duplicate 100 ml. sample. Both condensate flasks 4 are then placed in the refrigerator and cooled to the same temperature. The percentage of alcohol is then determined with a refractometer and, from the data obtained, the volume of alcohol originally contained in the 200 ml. sample of beer can be ascertained. The results from both samples should check within 0.03%.

With a constant rate of heat input, the distillation rate progressively decreases from a maximum at the beginning of the operation to a minimum at the end thereof. To maintain a more constant distillation rate, it becomes necessary to increase the rate of heat input progressively as the operation proceeds. With the conventional arrangement, however, the initial rate of heat input and the subsequent increases in the rate of heat input are matters of guesswork which often result in the use of excessive rates of heat input causing excessive distillation rates with resultant vapor loss and erroneous indications of yield. If this condition be avoided by maintaining the heat input within a range of values sufficiently low to be absolutely safe, then the time required to conduct the operation is substantially increased.

In order to avoid the possibility of appreciable vapor loss and at the same time reduce the operating time to a minimum, I propose to place, between the condenser 3 and volumetric flask 4, a vapor loss detector or visual indicator which is capable of indicating a flow of uncondensed vapors from the condenser 3. The indicator comprises: a transparent glass housing 5 having a vent opening 6 and an overflow level tube 9, the upper end of the latter defining an overflow level 7; and an inlet tube 8 extending from within the housing 5 at a point substantially below the overflow level 7, upwardly through the wall of the housing 5 to form an externally projecting inlet terminal which may be connected to the condensate outlet of the condenser. Overflow tube 9 extends from within the housing at the overflow level 7 downwardly through the bottom wall of the housing to form an externally projecting discharge terminal which may be arranged to discharge directly into the volumetric condensate flask 4.

With this arrangement, the indicator housing 5 is filled with distilled water, preferably up to the overflow level 7, as defined by the upper end of tube 9 and the distillation operation instituted as before. As the distillation proceeds, the condensate will flow into the detector and replace the water. If the heat input is too large, uncondensed vapors will bubble up through the liquid in the detector and thereby provide a visual indication of an excessive distillation rate permitting the operator to reduce the rate immediately to a point at which the bubbles disappear. As the distillation proceeds the operator can increase the heat input in order to maintain the same rate of overflow in the detector 5. In fact, the operator may, without incurring appreciable vapor loss, increase the rate until bubbles again appear and then decrease it immediately to the slight extent necessary to make them barely disappear. In this way a distillation rate corresponding substantially to the maximum capacity of the condenser may be maintained without incurring appreciable vapor loss. The operation of course continues as before until the condensate in the volumetric flask 4 reaches the 100 ml. mark. At the end of the operation the detector will be filled with liquid up to the liquid level but this liquid may be discarded since it is substantially pure water containing only a trace of alcohol.

The reliability of our arrangement in comparison with the conventional method is indicated by Tables 1 and 2 which follow:

Table 1

Conventional Method	Vapor Detector
7.11	7.21
7.12	7.22
7.12	7.22
7.12	7.22
7.14	7.22
7.18	7.24

In the above table, the figures represent the percentage yield of alcohol, identical samples being used throughout.

Table 2

Conventional Method	(1)	(2)	(3)	(4)	(5)	(6)
100–110 cc	0.02	0.05	0.05	0.04	0.04	0.03
110–120 cc	0.01	0.04	0.03	0.03	0.03	0.02
120–130 cc	------	------	0.02	0.02	0.02	0.01
130–140 cc	------	------	------	------	0.01	0.00

Vapor Detector	(7)	(8)	(9)	(10)
100–110 cc	0.05	0.05	0.05	0.05
110–120 cc	0.03	0.03	0.03	0.03
120–130 cc	0.02	0.02	0.02	0.02
130–140 cc	0.01	0.01	0.01	0.01

In obtaining the data of Table 1, twelve identical samples were distilled, six by the conventional method and six by the present arrangement disclosed in this application.

In Table 1 it will be noted that the present arrangement consistently indicates a higher yield than the conventional arrangement, and that its indications are not subject to variations as substantial as the variations obtained in the conventional method. The lower yield obtained with the conventional method may be explained only on the basis of an undetected loss.

In obtaining the data of Table 2, ten identical beer samples were provided and, from each sample, 100 ccs. of beer were distilled off. From the residue of each sample, four successive 10 cc. fractions were distilled off and the distillate of each fraction separately collected. Six residue samples were distilled using the conventional method and four using the arrangement disclosed in this application. The figures represent the percentage of residual alcohol in the several fractions. With the present arrangement, consistent, and as a matter of fact absolutely constant, results were obtained whereas with the conventional method substantial variations were encountered. As before, these variations may be explained only on the basis of undetected losses.

Having described our invention, we claim:

In the art of distilling materials and continuously condensing and collecting the condensate for the quantitative determination of volatile constituents thereof by subjecting a sample of the material to a distilling operation in an apparatus including a still and a condenser, a method of determining and maintaining a relatively constant and maximum rate of distillation substantially corresponding to the capacity of the condenser, comprising: continuously discharging all of the material from the condenser into an externally visible liquid bath at a point below the level of the liquid so that uncondensed vapors discharged into the bath will bubble visibly through the bath; and maintaining a rate of distillation in the still slightly below that rate at which bubbles appear.

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