No. 896,434. PATENTED AUG. 18, 1908.

H. O. CHUTE.

DISTILLATION APPARATUS.

APPLICATION FILED DEC. 3, 1904.

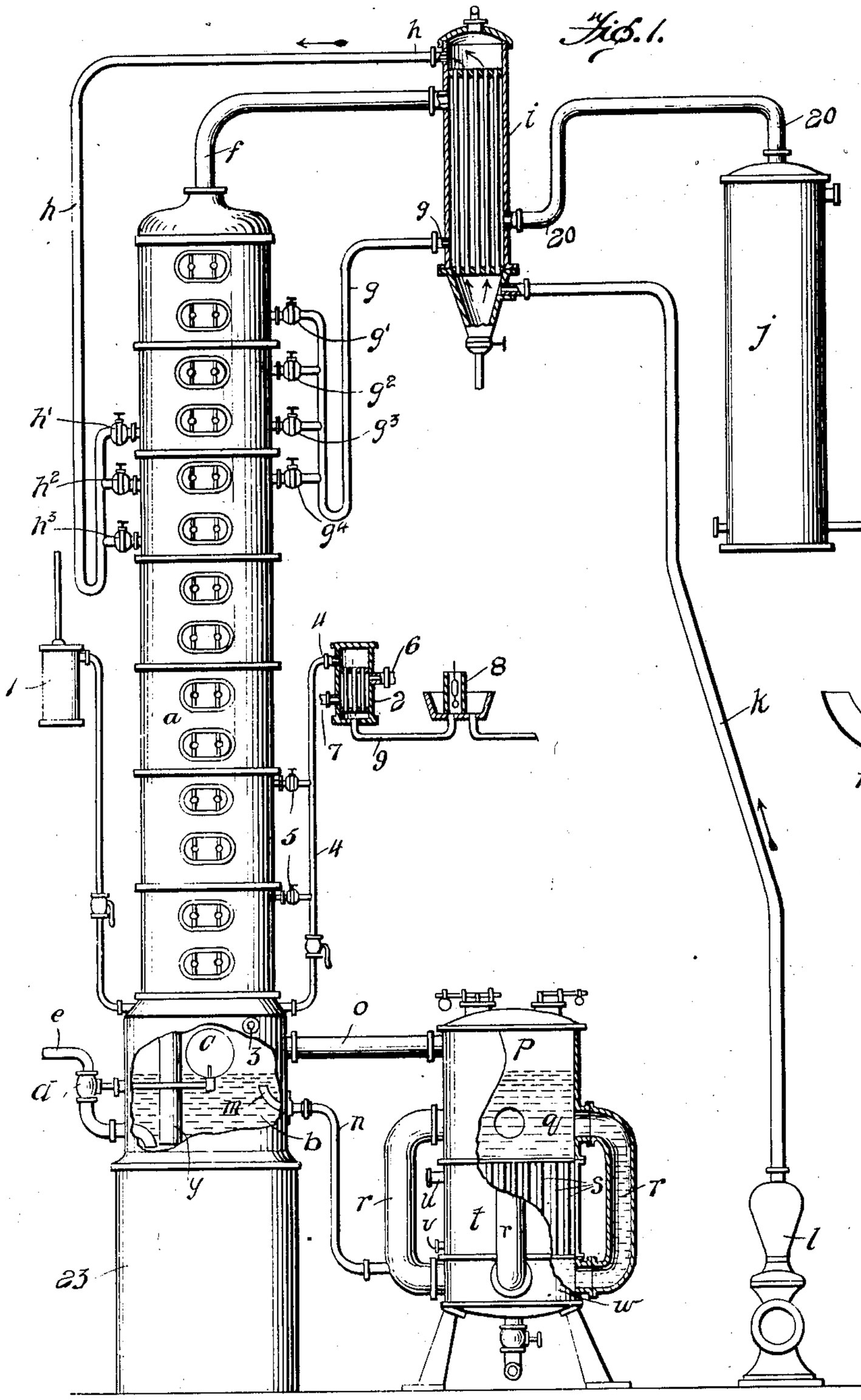

Fig. 1.

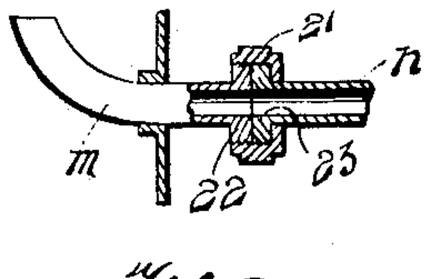

Fig. 2.

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DISTILLATION APPARATUS.

No. 896,434. Specification of Letters Patent. Patented Aug. 18, 1908.

Application filed December 3, 1904. Serial No. 235,280.

To all whom it may concern:

Be it known that I, HARRY O. CHUTE, a citizen of the United States, and a resident of the city of Cleveland, county of Cuyahoga, State of Ohio, have invented certain new and useful Improvements in Distillation Apparatus, of which the following is a full, clear, and exact description.

This invention relates to apparatus of the kind known as continuous stills, and relates particularly to stills employed for the separation of wood alcohol from pyroligneous acid, though of course such stills may be used for other purposes.

The invention consists in the combination, with a column still, of a separate boiler or heater of improved construction; in means employed for avoiding the feeding of tarry or other insoluble matter into this heater; in means for regulating the point of entry into the still of the crude liquor and the returns from the exchanger; in means for admitting said returns at a point above that at which the crude liquor is admitted; in novel means for testing the quality of the vapor in the still; and in various other features, as hereinafter more fully described and particularly pointed out in the claims.

The objects of my invention are, to increase the efficiency of fractional distilling apparatus, such as may be employed for the distillation of pyroligneous acid; to avoid the dilution of the liquor within the still by steam or like vapor employed for heating; to improve the means employed for applying heat in the still; to prevent the entry of tarry matter into the heater; to provide improved means for regulating the action of the still; to provide means for testing at will the vapors in the still; and generally to make the apparatus simple, durable and highly efficient.

I will now proceed to describe my invention with reference to the accompanying drawings, illustrating diagrammatically one form of apparatus embodying my invention, and will then point out the novel features in claims. The said drawing shows a side elevation of the apparatus, parts of which have been broken away or sectioned to show the internal construction.

In this showing, Figure 1 illustrates the apparatus as a whole, partly in elevation and partly in vertical section; and Fig. 2 illustrates, on an exaggerated scale, the connection of pipes *m* and *n* of Fig 1.

In my apparatus, I preferably employ a continuously acting column chambered still of the type known as "beer stills", in which the raw or crude liquor fed into the same at or near the top and descending therethrough is subjected to the action of the ascending heated vapors produced by vaporizing a selected portion of the liquor removed from that remaining after the evaporation of the alcohol. The boiler used for so vaporizing this selected portion is preferably structurally separate from the still and comprises upper and lower chambers and an intermediate heating chamber, through which pass heating tubes connecting the upper and lower chambers, said tubes being heated by live steam or other heating fluid circulating around them in said heating chamber. The heating chamber has no fluid connection with the liquid chambers. Said boiler further comprises exterior circulating tubes connecting the upper and lower liquid chambers.

The lower compartment or chamber of the still constitutes a settling chamber of capacity sufficient to permit the tarry and other insoluble products to settle, automatic means being provided for drawing off these tarry products with the liquor in said chamber and for maintaining a substantially uniform level of liquid in said chamber. A pipe having an adjustable end piece, is provided for drawing off a portion of substantially tar-free or clear liquor from this chamber, and conducting such liquor to the boiler before mentioned; and another pipe conducts the vapor from this boiler back into the still. The liquor to be distilled enters the upper portion of the still, after first passing through a suitable heat-exchanger wherein there is an exchange of heat between the outgoing vapors from the still and the incoming liquor. By means of suitable valves, the incoming liquor may be admitted to the still at any one of a plurality of chambers of said still; and by similar means the vapors condensed in the exchanger, may be returned to the still at any one of a plurality of points. Preferably inflowing liquid is introduced at a point in the still where the average alcoholic richness of the liquid on the plates in the chamber is not widely different from that of such inflowing liquid. In connection with the still, I preferably employ what I term a "tester" consisting of a small condenser arranged to be connected at will, to any one of a number of the lower chambers of the still, and to draw off vapors from the

particular chamber with which it may be connected at any moment. Vapors so drawn off and condensed, flow into a suitable hydrometer cup, wherein floats a hydrometer. By this means, the quality of the vapors in the various chambers of the still connected to the tester, may be determined readily whenever desired.

Pyroligneous liquor is the aqueous portion of the products from the destructive distillation of wood. It contains besides a large quantity of water, methyl alcohol—which boils at 66 degrees centigrade—, and acetone—boiling at 56 degrees centigrade—, which two latter materials together form the wood alcohol of commerce. Pyroligneous liquor also contains acetic acid—boiling at 119 degrees centigrade, and other organic volatile acids, and the liquor also contains more or less tarry products. In distilling such liquor with the apparatus ordinarily used, the alcohol, if completely distilled off from the liquor, carries with it a portion of the acids; and to so distil off practically all of the alcohol requires the evaporation of a large proportion of the liquor. My apparatus serves to separate the alcoholic or spirituous portion of the liquid in a concentrated state, substantially free from acid and delivers the acid portion undiluted, no free steam being used in heating.

Referring now to the drawing, *a* designates an ordinary column still. I do not illustrate the same in detail, as the construction of such stills is well understood. It will be understood that such still comprises a plurality of chambers through which the liquor treated descends progressively, and through which the heated vapor ascends progressively. At the base of this still there is a settling chamber *b*. Adjacent to the still is a heater or boiler *p* comprising upper and lower chambers *q* and *w* respectively, and an intermediate heating chamber *t*, which latter chamber is steam-tight and contains a large number of tubes *s* connecting chambers *q* and *w*. Steam or other suitable heating fluid may be admitted to this chamber *t* through a connection *u*, and the exhaust or condensed steam, or heating fluid, escapes through a connection *v*. Chambers *q* and *w* are further connected by external circulating pipes *r*. Pipes *r* being air-cooled cause a circulation of liquid downward therethrough.

The heater or boiler thus constructed may be made quite cheaply, and the tubes *s*, which are exposed to the steam pressure, may be made quite small, thus permitting them to withstand relatively high steam pressure even when made of relatively thin weak material. The liquor to be distilled is passed by suitable means, such as a pump *l* and pipe *k*, into an exchanger *i* of familiar form shown in section, and thence passes through a trapped pipe *h* into the column *a*. Said pipe *h* is connected with the column at a plurality of points by means of valves h^1, h^2 and h^3. The vapor from the column passes through the pipe *f* into the heating chamber of the exchanger *i* and thence passes into the main condenser *j*, and when condensed, out through pipe *x*. Any vapor which may be condensed in the exchanger may be returned to the still through a trapped pipe *g* connected with the column at a plurality of points, by means of valves g^1 g^2 g^3 and g^4.

The settling chamber *b* at the bottom of the still, is provided with a pipe *e*, the mouth of which is near the bottom of such chamber, through which pipe the tarry products, which settle to the bottom of this chamber *b*, and the acid liquor may be drawn off. Float *c*, operating a valve *d* in said pipe, regulates the level of the liquid in chamber *b*. A pipe *n* opening into the chamber at a point above the level of the draw-off pipe *e*, serves to draw off a portion of the liquor substantially free from tar or other insoluble impurities and also free from alcohol and the like from chamber *b* and to introduce the same into the boiler *p;* and, as shown, this pipe has an adjustable mouth piece *m*, whereby the level of the inlet mouth of this pipe, may be varied. In the construction shown, this mouth piece *m* is a simple bent tube, by turning which, the level of the mouth may be adjusted as desired. The vapors are returned from the boiler *p* to the upper portion of chamber *b*, above the level of the liquid therein, through a pipe *o*.

As shown in Fig. 2, pipe *n* is provided with a screw union consisting of a flange 22, the outside of which is threaded and rigidly attached to the pipe *m*, a flange 23 rigidly attached to pipe *n*, and collar 21 fitting over flange 23 and screwing on to the thread on flange 22. This permits pipe *m* to be turned relative to pipe *n* and therefore permits the mouth of pipe *m* to be adjusted relative to the liquid level in the settling chamber. Any other type of rotatable union however may of course be used and the type shown is merely exemplificatory.

The operation of this apparatus is as follows: The crude liquor entering through pipe *k*, is heated preliminarily, during its passage through the exchanger *i*, and thence passes into the still through pipe *h* and one or another, or two or more of the valves h^1 h^2 and h^3. Within the still, it descends successively from chamber to chamber of the still, being heated therein by the ascending vapors, the alcohol within the liquor being thereby distilled off fractionally and caused to pass off through pipe *f*, while aqueous vapor, acid vapor and the like, in the uprising vapors, are condensed and caused to descend. The liquor which collects in chamber *b* is substantially free from alcohol, and any tar therein collects in the lower portion of said

chamber disturbance of the liquor in the chamber being prevented by introducing inflowing liquid near its bottom through pipe *y* and is drawn off through pipe *e*, together with the acid. A portion of the substantially tar-free liquid from the top layers of the mass of liquid within such chamber *b* is drawn off through pipe *n* and passed through the heater or boiler *p*, whereby such liquor is evaporated and its vapor returned through pipe *o* to the still, such vapors rising through the chambers of the still and constituting the means whereby the alcohol is distilled off from the descending liquid. Both *n* and *o*, in the structure shown, being freely open the level of liquid in the settling chamber *b* and boiling chamber *p* will be the same. The valves h^1, h^2, and h^3 permit the entering liquors to be admitted to the still at different heights in order that substantially all of the alcohol shall be evaporated. The various valves g^1 g^2 g^3 and g^4 permit the condensation from exchanger *i* to be returned to the still at various points. Such condensation liquors, being exceedingly rich in alcohol, serve to condense any acid remaining in the vapors after its passage through the crude entering liquor, without depriving the uprising vapors of their alcohol. In order that this final separation may be efficient, I preferably so adjust the valves mentioned, that the crude liquor enters the still below the condensation liquor returned from the exchanger *i*. By properly adjusting these various valves, the operation of the still may be so regulated as to regulate the percentage of the alcohol in the distillate while completely separating the alcohol from the acid, all in one operation.

By use of the separate heater *p* I avoid the introduction of steam into the still, as has been done heretofore; the introduction of steam being objectionable ordinarily because it tends to dilute the acid liquor and render the separation more expensive. However, if desired, as for heating the apparatus in starting steam may be admitted through the opening 3. By means of the adjustable mouth piece *m*, I avoid drawing into the boiler, not merely the main mass of tar which collects at the bottom of the chamber *b*, but even the separate globules of tar which have not as yet coalesced. In this way I avoid the baking of the tar on the surfaces of the heating tubes of the boiler *p*, which, if it occurs, materially lowers the efficiency of the heater.

In order to be able to test the liquor in the still and determine the quality of the liquor in the various lower chambers, I employ a "tester" 2, comprising a small condenser connected to the lower portion of the still at various points, by means of a pipe 4 and valves 5, such condenser being provided with an inlet 6 and outlet 7 for cooling fluid. The vapors drawn through pipe 4 into this condenser, drop through pipe 9 into hydrometer cup 8. The hydrometer serves to indicate the average richness of the vapors in the chambers with which the tester is placed in communication. By this means may be determined the point in the still at which the liquor becomes substantially free of alcohol.

In the drawing, 1 designates a pressure gage of familiar form, comprising a cylinder connected at its upper end to the still and a pipe extending upward from such cylinder; which pipe is customarily provided with indicating means at a point above that at which the pipe is shown as broken off in the drawing.

The still is supported on a base 23, which may be of any desired shape or construction.

What I claim is:

1. In distillation apparatus, the combination with a chambered column still provided at its lower end with a quieting and settling chamber and with a draw-off outlet, of a heater having a liquid supply passage communicating with said settling chamber at a point above said outlet, said heater provided with means for returning vapor to said settling chamber near the top thereof.

2. In distillation apparatus, the combination with a chambered column still provided at its lower end with a settling chamber and with a draw-off outlet, and automatic level-regulating means controlling such outlet, of a heater having a supply passage communicating with said settling chamber at a point above said outlet, said heater provided with a connection for returning vapor to said still.

3. In distillation apparatus, the combination with a chambered column still provided at its lower end with a settling chamber and with a draw-off outlet, of a heater having a liquid supply passage communicating with such settling chamber, and provided with means for adjusting the point of communication to various heights above said outlet, said heater further provided with means for returning vapor to the still.

4. In distillation apparatus, the combination with a chambered column still provided at its lower end with a settling chamber and with a draw-off outlet, and automatic level-regulating means controlling such outlet, of a heater having a supply passage communicating with such settling chamber and provided with means for adjusting the point of communication to various heights above said outlet, said heater further provided with means for returning vapor to the still.

5. In distillation apparatus, the combination with a chambered column still provided at its lower end with a settling chamber, of a heater and means connecting the same with said settling chamber comprising a bent pipe within such chamber rotatable to vary

the level of its mouth, said heater provided with means for returning vapor to the still.

6. In distillation apparatus, the combination with a chambered column still having a quieting and settling chamber at its base, of a separate heater therefor comprising upper and lower chambers, an intermediate heating chamber, pipes passing through such heating chamber, and connecting such upper and lower chambers, and circulating passages connecting said upper and lower chambers, and located outside said heating chamber, said heater provided with supply and return connections to said still, the supply connection drawing off liquid at a point above the base of said settling chamber of the still, and the return connection delivering vapors to the settling chamber at a point near its top and above normal level of liquids therein, whereby the same normal liquid level is maintained in both heating chamber and settling chamber.

7. In distillation apparatus, the combination with a chambered column still provided with heating means, of an exchanger therefor, provided with vapor and liquor connections comprising adjustable means for admitting the heated liquor to said still at any one of a plurality of levels, and comprising also means for returning condensed liquid to said still at any one of a plurality of levels.

8. In distillation apparatus, the combination with a continuously fed chambered column still comprising heating means and means for supplying liquor to the upper portion thereof and means for withdrawing exhausted liquor from the base, of a tester comprising a condenser provided with means for connecting it to the vapor spaces of the still at any one of a plurality of intermediate levels to draw off vapor, and comprising also means for indicating the quality of the liquid condensed from the vapor so drawn off.

9. In distillation apparatus, the combination with a continuously operating chambered column still comprising heating means and means for supplying liquor to the upper portion thereof and means for withdrawing exhausted liquor from the base, of a tester comprising a condenser provided with means for connecting it to the vapor spaces of the still at any one of a plurality of intermediate levels to draw off vapor, a receptacle receiving the fluid condensed, and a hydrometer therein.

10. In distillation apparatus, a continuously operating fractionating still comprising a chambered column still, means for continuously feeding raw hot liquor into an upper chamber thereof, said means comprising a heat exchanger adapted to transfer heat from outflowing vapors to inflowing liquor, a quieting and settling chamber communicating with the column still at the base thereof and provided with automatically acting means for preserving a constant liquid level therein by drawing off regulated amounts of liquid from the base of said quieting chamber, and a heating chamber arranged to take liquid from said settling chamber at a point above the automatically controlled liquid outlet thereof and to return vapors of said liquid to a point near the top of said settling chamber and above the normal liquid level therein.

In testimony whereof I affix my signature in presence of two witnesses.

HARRY O. CHUTE. [L. S.]

Witnesses:
T. M. KREGELIUS,
H. L. PAYNE.