July 5, 1932. J. M. COAHRAN 1,865,887

METHOD OF AND APPARATUS FOR CONTINUOUSLY RECOVERING AND CONCENTRATING CHEMICALS

Filed Aug. 19, 1927 2 Sheets-Sheet 1

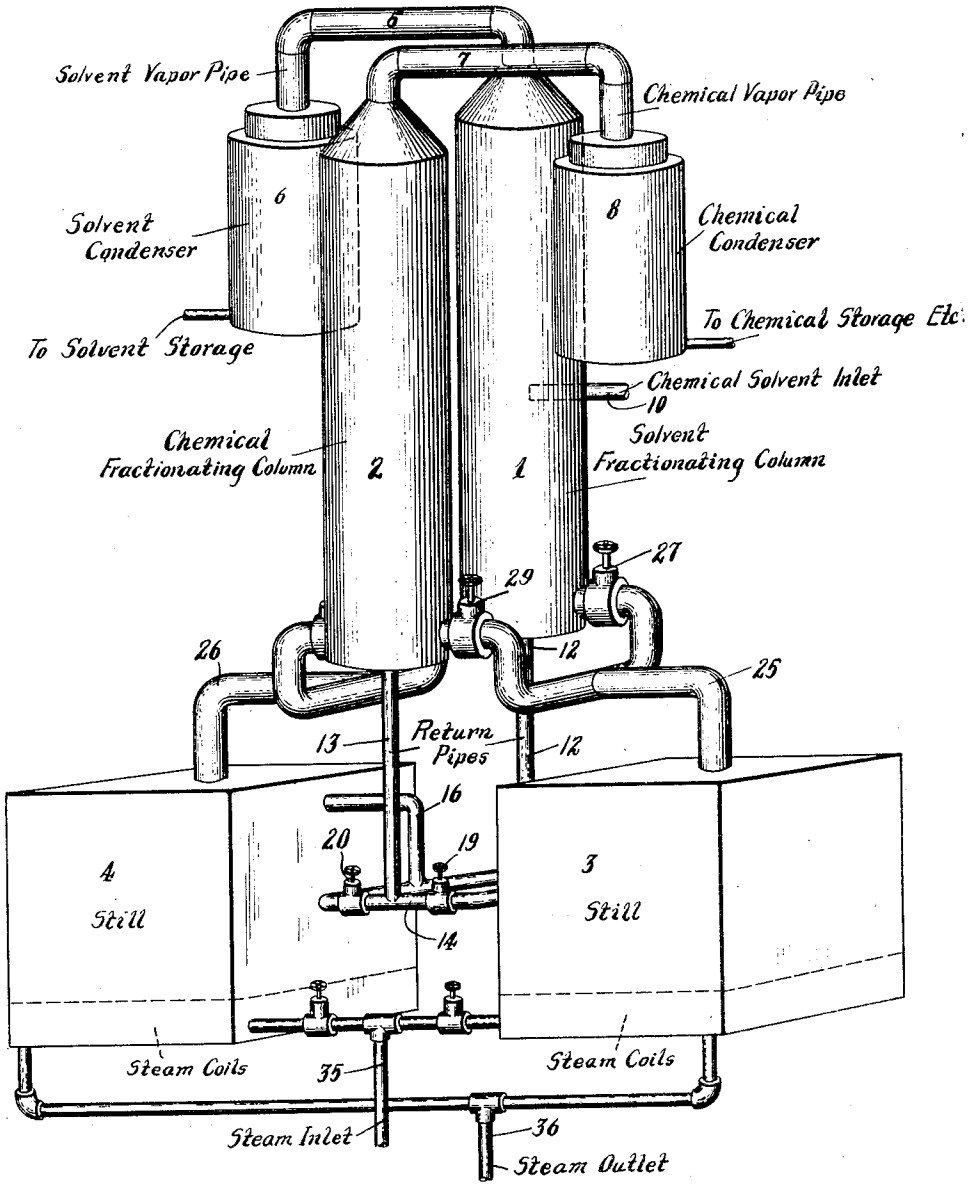

Fig. 1

Inventor:
Jesse M. Coahran.
by Wright, Brown, Quinby & May
Att'ys.

July 5, 1932. J. M. COAHRAN 1,865,887

METHOD OF AND APPARATUS FOR CONTINUOUSLY RECOVERING AND CONCENTRATING CHEMICALS

Filed Aug. 19, 1927 2 Sheets-Sheet 2

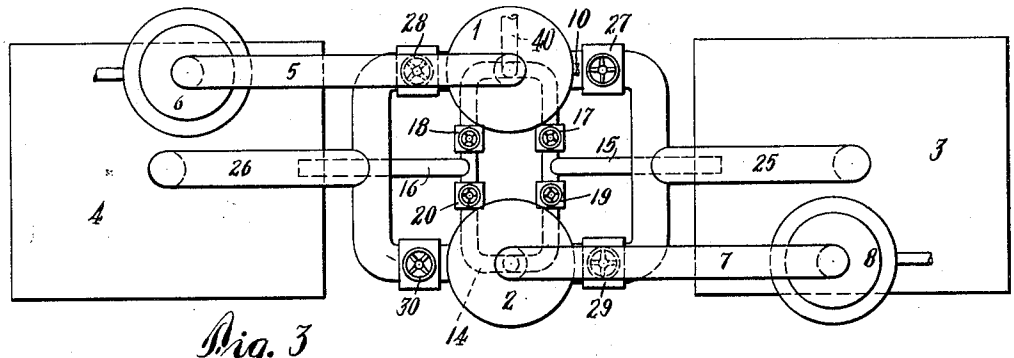

Fig. 3

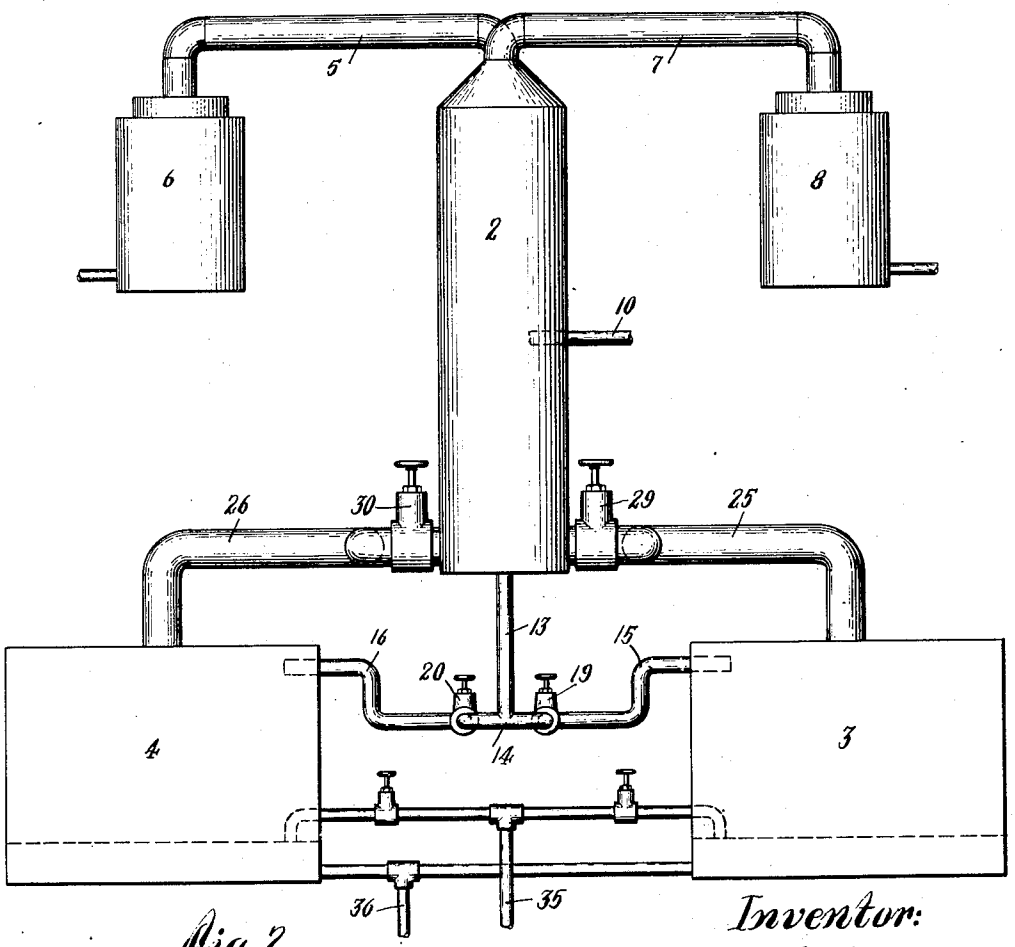

Fig. 2

Inventor:
Jesse M. Coahran.
by Wright, Brown, Quinby & Thacher
Attys.

Patented July 5, 1932 1,865,887

UNITED STATES PATENT OFFICE

JESSE M. COAHRAN, OF SMETHPORT, PENNSYLVANIA

METHOD OF AND APPARATUS FOR CONTINUOUSLY RECOVERING AND CONCENTRATING CHEMICALS

Application filed August 19, 1927. Serial No. 214,189.

This invention relates to the recovery of chemicals, and while not restricted to such use, has been developed with particular reference to the recovery and partial purification of acetic acid from a solvent such as ether by the use of which this acid in crude condition may have been separated from crude pyroligneous acid liquor.

The method employed is applicable to the treatment of material which may be separated by distillation into three portions of different volatilities. The portion of greatest volatility is first distilled off at a relatively low temperature and the run-back comprising the other two portions is then distilled at a higher temperature to separate out the second portion of intermediate volatility, leaving the third portion of lowest volatility as a residue. While the two distillations for any given quantity of the material are successively accomplished, according to this invention both are carried out simultaneously, the run-back from the first distillation being accumulated alternately in a pair of pools while the second distillation is carried on with the material of one pool while the other pool is being accumulated.

In the case of acetic acid recovery from solvent such as ether containing it, the solvent is the most volatile constituent, the acetic acid is the major portion of the constituent of intermediate volatility, and impurities such as tars, oils and certain acids, the constituents of lowest volatility.

One of the objects of this invention is to make possible the handling of a maximum amount of material in a minimum amount of time with a minimum amount of apparatus and the minimum expenditure of heat.

A further object is to avoid any necessity for the pumping or flowing by gravity of hot corrosive chemicals between stills and storage, and to eliminate entirely any necessity for special storage apparatus at any stage in the process. There is thus a substantial saving in initial cost, maintainance and replacement of apparatus.

These objects, as well as others which will hereafter appear, are attained by the use of two stills and two fractionating columns, one column being used continuously to separate out and fractionate the solvent, the other column being used continuously to fractionate the chemicals, and the two stills being used alternately, first to drive off the solvent into the solvent fractionating column and retain the chemical in heated condition, and then to vaporize the chemical by additional heat into the chemical fractionating column. The separation of the solvent from the chemical is thus a batch process, being carried on alternately in the two stills, while the solvent fractionation is continuous, the fractionation column drawing its supply from the stills alternately. The chemical column operates cyclically, or in batch alternately from the two stills as will later more fully appear.

For a more complete understanding of this invention, reference may be had to the accompanying drawings in which

Figure 1 is a somewhat diagrammatic perspective of the apparatus.

Figure 2 is a side elevation thereof.

Figure 3 is a top plan of the same.

Referring to these drawings, at 1 and 2 are indicated respectively the solvent and chemical fractionating columns. Beneath these are positioned the stills 3 and 4. The solvent fractionating column 1 is connected by the solvent vapor pipe 5 with the solvent condenser 6. The chemical fractionating column is connected by a similar vapor pipe 7 with a chemical condenser 8. The mixture to be separated is supplied as through the pipe 10 to the fractionating column 1. The columns 1 and 2 communicate through the return lines 12 and 13 with the end portions of a pipe loop 14. The side portions of this pipe loop communicate as by the riser pipes 15 and 16 with the stills 3 and 4, respectively. This pipe loop thus forms the lower portion of a trap between the columns and stills. Valves 17 and 18 control communication from the return line 12, and similar valves 19 and 20 control communication from the return line 13 to these stills, communication from both return lines being made through the riser pipes 15 and 16 to the respective stills. Similarly both columns 1 and 2 communicate with the upper portion of each of the stills, as

through the pipes 25 and 26, communication between the column 1 and the still 3 being controlled by the valve 27, communication between the column 1 and the still 4 by the valve 28, communication between the column 2 and the still 3 by the valve 29, and communication between the column 2 and the still 4 by the valve 30. The stills may be heated by any suitable means, as herein shown steam inlet and outlet pipes 35 and 36 being provided to supply steam coils in the stills.

In operation the mixture to be separated is introduced through the pipe 10 into the column 1, the descending liquids being met by the hot vapors rising from one of the stills, either 3 or 4, depending on the conditions of the controlling valves. Assuming, for example, that the valves 17, 20, 27 and 30 are open and the valves 18, 19, 29 and 28 are closed, the hot vapors arise from the still 3 and meet the down-coming liquids in the column 1, the heat causing the solvent to be driven off in the column 1 through the vapor pipe and into the solvent condenser 6 from which it may be removed to the solvent storage. The less volatile constituents of the mixture, in the case of acetic acid and solvent this comprising the acetic acid together with impurities such as propionic and butyric acids, methanol, aldehydes, phenols, oils, and tars, and some water, pass down through the return pipe 12 into the still 3. This continues until the still 3 is substantially full of the crude impure acid. When the still 3 has been so supplied with a pool of this material, the valves 17, 20, 27 and 30 are closed, and valves 18, 19, 28 and 29 are opened, whereupon the column 1 receives the hot vapor supply from the still 4 and the acid passing down through the pipe 12 passes into the still 4. The still 3 which has thus been filled with acid and which is hot is then still further heated as by means of the steam coils, the vapor passing up through the pipe 25 past the open valve 29 and into the chemical fractionating column 2 where it is driven off through the pipe 7 and condensed in the condenser 8.

The constituents of the crude acid come off from the column 2 in about the following order: (1) Ether containing a very small percentage of acid. This is returned to the extractor by which the crude acetic acid was separated from the pyroligneous acid liquor, with the feed liquor. (2) Light and then heavy wood oils with very little acid which is disposed of as oil. (3) Water containing increasingly larger amounts of acid, some acetic but more propionic and butyric. (4) Acid increasing in strength from 45% to 98% and containing also some water and some propionic and butyric acid. The weaker portion of this is returned to the still which is being filled with crude acid, the return being connected to the trap coil 14 as shown at 40 in Figure 3, the stronger portion being collected for further treatment and refining. (5) Still residue comprising tar, heavy high boiling oils with about 30 to 40% of acid including acetic, propionic and butyric acids. This still residue is removed and treated to recover the tar, the remaining materials being returned to the crude acetic acid extractor.

By this means it will be seen that the liquid to be separated is supplied continuously to one of the fractionating columns, where the more volatile liquids are drawn off and concentrated, and that the less volatile liquids are collected alternately in one of two pools from which they are distilled into the other fractionating column where they are partially separated, concentrated, and recovered, the one pool being accumulated from the first fractionating column while the other is being used to feed the second fractionating column.

It will thus be seen that no pumping or gravity feed of hot corrosive liquids from or to storage receptacles is necessary and that no separate storage receptacle other than the stills by which the vaporization is effected is necessary, and that the heat content of the less volatile liquid as it passes from the first column is utilized to its full extent when it is subjected to distillation and fractionation.

From the foregoing description of an embodiment of this invention it should be evident to those skilled in the art that various changes and modifications might be made therein without departing from the spirit or scope of the invention as defined by the appended claims.

I claim:

1. The process which comprises continuously fractionating a mixture of crude acetic acid containing impurities less volatile than the acid and an acetic acid solvent more volatile than said acid so as to remove said solvent, collecting the crude acid containing said impurities alternately in a pair of pools, and distilling and fractionating the crude acid alternately from each one of said pools so as to separate out acetic acid from said one pool while the other pool is being accumulated.

2. The process of treating a liquid mixture having constituents of different volatilities, which comprises continuously supplying the mixture to a fractionating zone and removing the constituents of highest volatility, refluxing the remaining constituents alternately in a pair of differentially heated pools, and vaporizing constituents from one of said pools at a higher temperature than for removal of the constituents of highest volatility in the fractionating zone and removing therefrom the constituents of intermediate volatility, while the other pool is being accumulated.

3. An apparatus comprising a pair of fractionating columns of substantially identical

capacity, a pair of stills, means for supplying a mixture of three constituents of different volatility to be separated to one of said columns to drive off and recover the most volatile constituent, means to convey the two less volatile constituents of said mixture at will to either of said stills while said still supplies vapor to said one column, and means for distilling from the still not receiving flow at any time to the other of said columns to drive off and recover the constituent of intermediate volatility while leaving the constituent of least volatility.

4. An apparatus of the class described, comprising a pair of fractionating columns of substantially identical capacity, a pair of stills below said columns, heating means in each still, means for supplying liquid to one of said columns, and valved connections between said columns and still for directing hot vapor from either selected still to either selected one of said columns and to return the run-back from said one column to said selected still, and simultaneously therewith to direct the vapor distilled from the other of said stills to the other of said columns.

5. An apparatus of the class described comprising a pair of fractionating columns, a pair of stills below said columns, heating means in each still, means for supplying liquid to one of said columns, valved connections between said columns and stills for directing vapor distilled from either selected still into either selected column, and valved connections independent of said vapor connections and having traps therein between said columns and stills for directing the run-back from either of said columns selectively into either of said stills.

In testimony whereof I have affixed my signature.

JESSE M. COAHRAN.