April 30, 1946. D. R. DOUSLIN ET AL 2,399,334

COOLING AGENT DISPENSER

Filed July 26, 1943 2 Sheets-Sheet 1

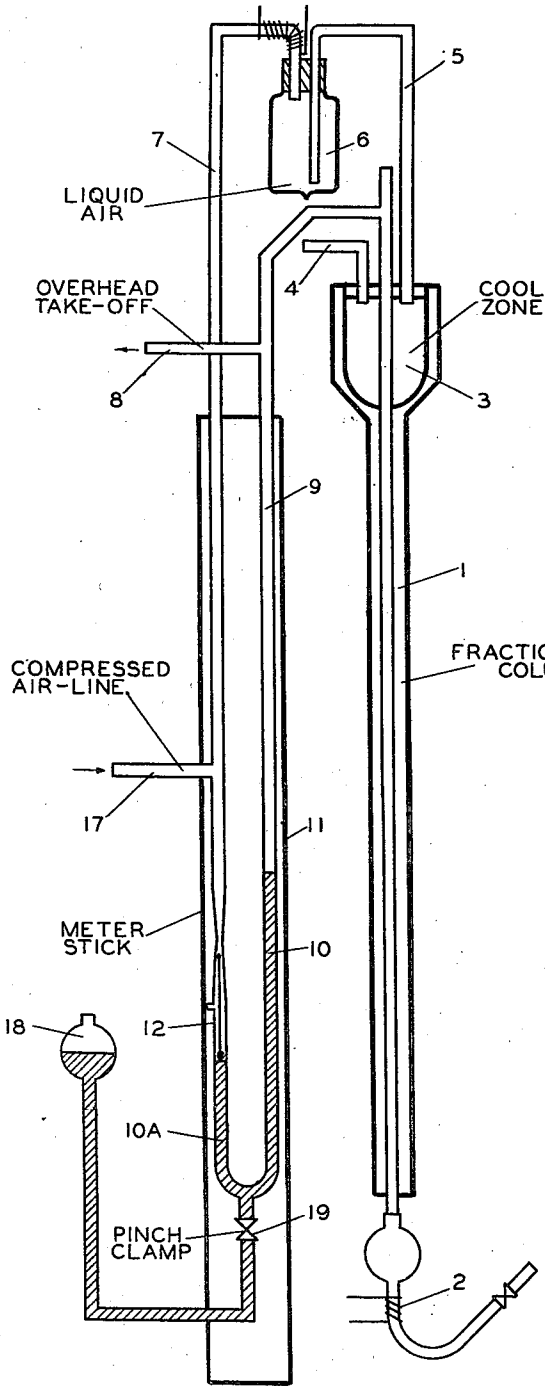


FIG. - 1

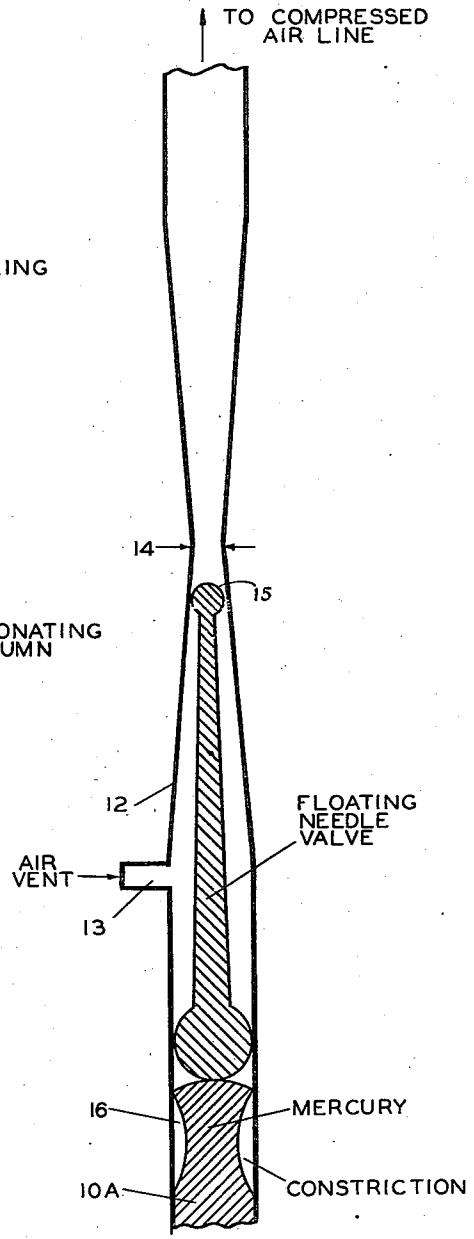


FIG. - 2

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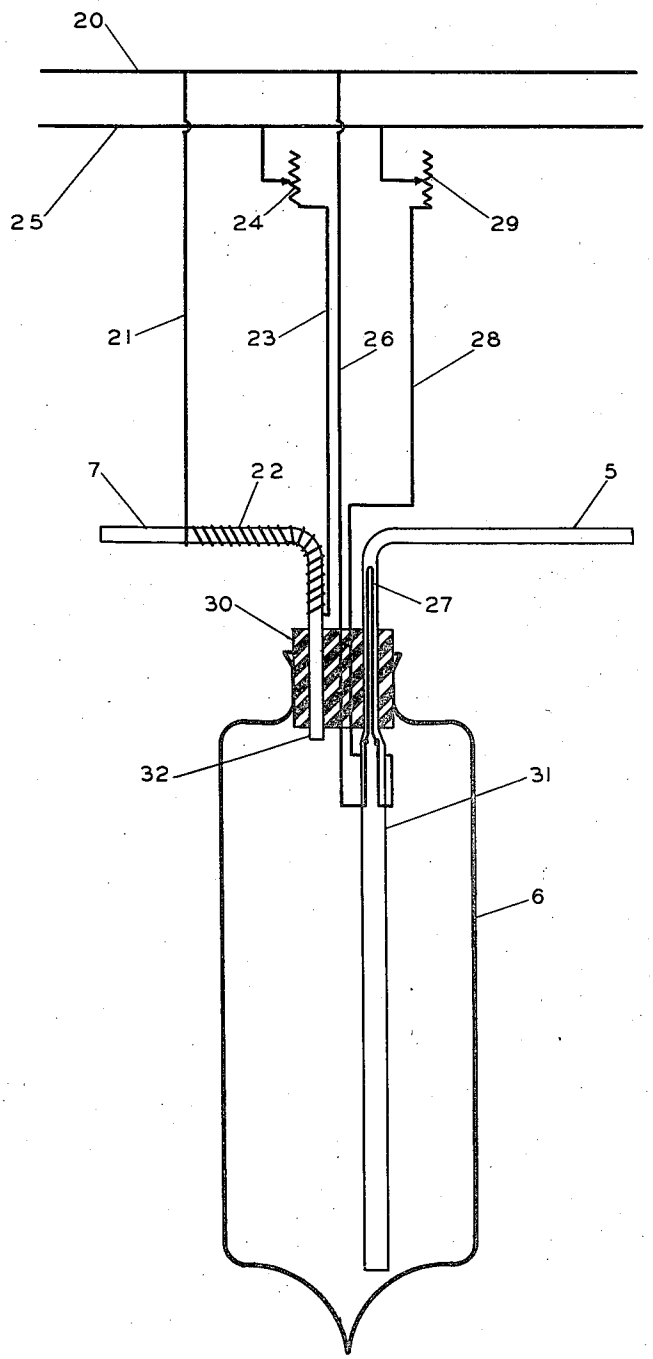


FIG. - 3

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2,399,334

COOLING AGENT DISPENSER

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Application July 26, 1943, Serial No. 496,204

5 Claims. (Cl. 202—160)

This invention relates to method of and apparatus for dispensing a cooling agent, and has particular relation to dispensing a cooling agent in connection with automatic pressure control of a low-temperature low-pressure analytical fractionating column.

In some respects, the present invention is an improvement on the patent to W. J. Podbielniak No. 1,967,258, issued July 24, 1934. The arrangement described by Podbielniak includes a control device which is adapted to maintain a constant pressure in the column. A manometer which is connected with the vapor outlet tube by a side arm is provided with electrodes leading to an electrical circuit which includes a solenoid valve. The valve controls the effective pressure applied from a source of constant air pressure to a body of liquid air in a flask. In the operation of the Podbielniak arrangement, pressure increase in the fractionating column is registered on the mercury column of the manometer, closing the circuit through the solenoid valve and applying pressure to the liquid air which is forced into the cooling section of the column, thereby reducing the fractionation pressure. Our invention is a manifest improvement over the arrangement of the patent in providing fully automatic control of the fractionating pressure in an advantageous and expedient manner and with the elimination of involved electrical circuits, solenoid valves and other relatively expensive equipment which have heretofore been considered essential to the operation of such devices.

This application is a continuation in part of the subject matter of application, Serial No. 468,148, filed December 7, 1942, by Donald R. Douslin in that liquid air container and dispenser 6 has been improved and this improvement is in effect also an improvement of the entire combination.

One of the difficulties of the prior art method and apparatus was that the liquid cooling agent would vaporize all along the tube and form a moving vapor layer and a liquid layer that would not move; and only vapor would flow along the tube leaving the liquid behind.

One object of the invention is to automatically dispense cooling agent in a positive manner which avoids forming vapor and liquid layers, but which positively delivers liquid to the condenser of a fractionating column when the dispenser is activated by a device designed to automatically control the pressure within the column.

Another object of the invention is to provide finer control on the amount of cooling agent delivered.

A further object is to provide constant flow of small slugs of cooling agent as distinguished from the tendency of prior art devices to suddenly surge over an excessively large slug. The result of constant flow of small slugs is obviously a more constant temperature in the cooling zone and thereby a more constant pressure in the fractionating column; and it is an object of the invention to provide this more constant pressure and to avoid any violent surges in fluids, in temperatures and in pressures.

It is also an object of the invention to maintain the pressure wtihin the cooling agent container, as opposed to the quick pressuring of the prior art, which quick pressuring is productive of large slugs.

Still another object is to prevent moisture from freezing and plugging the intake line 7.

Further objects are to provide a simple foolproof embodiment of the invention; other objects will be apparent to those skilled in the art from the construction of this illustrative embodiment of the invention as described in the specification, claimed in the claims and shown in the drawings, in which:

Figure 1 is a general view of an analytical fractionating column and manometer incorporating our improvement.

Figure 2 is a sectional detail of the valve control shown in Figure 1.

Figure 3 is an elevational view mainly in cross section of an illustrative embodiment of the improved liquid air dispensing container forming part of the invention.

With specific reference to Figure 1, 1 is an analytical fracionating column of the general type described in Podbielniak Patent 1,967,258, having heating element 2 and a cooling zone 3 forming a receptacle at the top of the column provided with an outlet 4 and inlet conduit 5 connected with a supply 6 of liquid air or nitrogen, which is adapted to be forced by compressed air pressure through conduit 7 into the cooling section in an amount proportionate to pressure variation in the fractionating column in a manner hereinafter described. The overhead vapors from the fractionating process pass through conduit 8 to an accumulator not shown. Conduit 8 is provided with a side arm 9 which connects with a mercury manometer 10 fixed to the meter stick 11. The arm 10A of the manometer connects with conduit 7 and is provided with a floating needle valve designated generally

at 12 in Figure 1 and shown in detail in Figure 2. As clearly shown in Figure 2, arm 10A is vented to the atmosphere through air vent 13 and has an elongated relatively restricted section 14 through which the head 15 of the needle valve will barely pass. The needle valve may be formed of any suitable material either solid or hollow according to the specific gravity of the manometer fluid, the main prerequisite being that it floats on the body of fluid in the column. In the present application we prefer to construct the needle valve of solid glass with an enlarged lower portion for contact with the manometer fluid. A constriction 16 is provided in the arm 10A to limit the downward movement of the needle valve.

Any low boiling point liquid, or other known liquid cooling agent may be employed if it will produce the desired degree of low temperature.

The arm 10A is provided with a side arm 17 connected with a source of air under constant pressure. It will be understood that the inside diameter of the restricted portion 14 of the arm 10A is slightly greater than the outside diameter of the needle valve head 15, so as to allow a continuous small flow of air through the vent, the magnitude of which is dependent on the position of the needle valve 12. A leveling bottle 18 having a pinch clamp 19 is connected with the manometer tube 10 at the bend thereof.

In operation, heat is applied to the bottom of the fractionating column and overhead vapors pass through the line 8 to the accumulator, not shown. The pressure variation in the fractionating column is registered on the manometer column fluid which causes the needle valve to rise and fall relative to the restricted portion 14. The quantity of air under constant pressure from air line 17 passing head 15 to the vent 13, and likewise the pressure imposed on the liquid air supply 6, is therefore, a function of the pressure in the fractionating column. It is therefore obvious that increase of pressure in the fractionating column will force an increased quantity of liquid air into the cooling zone 3 reducing the pressure in the column. During normal operation and while the pressure in the column is constant a small quantity of liquid air is continuously forced into the cooling zone. By regulation of the compressed air pressure admitted at 17 and by adjusting the mercury level with the leveling bottle 18, it is possible to fractionate under any desired constant pressure automatically and without attention of the operator.

Figure 3 is an enlarged view of the liquid cooling agent container and dispenser 6 and related parts of Figure 1. Figure 3 shows in detail the electrical connections and heating elements 22 and 27 preferably employed in practicing the invention.

Container 6 may be a double-walled Dewar flask with a vacuum chamber between the walls and we prefer to use any inexpensive thermos bottle as this container. A single-walled container could be used and container 6 is so shown for purposes of simplicity.

Compressed air from line 17 passes through pipe 7 and out end 32 into chamber 6. The compressed air forces the liquid cooling medium into the bottom of pipe 31 and up to the heating filament 27. Heating filament 27 causes the liquid cooling agent to vaporize in flashes forcing small slugs of liquid cooling agent through pipe 5 to cooling zone 3 as will be explained in detail later.

Pipes 7 and 5 pass through a rubber stopper 30 which tightly fits the top of the container 6. Wires 26 and 28 pass through or alongside stopper 30 and are sealed by rubber stopper 30 so that the only passage for fluids is in pipe 7 and out pipe 5.

A power line 20 is tapped by wire 21 for electric current to heat resistance wire 22 wound around pipe 7. The circuit is completed by wire 23 the amount of current being controlled by rheostat 24 the electricity returning to the other power line 25.

A similar lead 26 from power wire 20 carries current through heating wire 27 and wire 28 to rheostat 29 and power wire 25.

The operation of the device is as follows:

The container 6 is filled with a liquid cooling agent and the connections at the stopper 30 of the container is made air tight. Pressuring fluid, such as compressed air, is admitted through tube 7 from device 12, 17 which controls the admittance of the pressuring fluid. The increase of pressure within container 6 causes the liquid cooling agent to rise up through tube 31 and contact heating element 27 which heating element is much hotter than the cooling agent. When this contact is made, a flash vaporization of a small amount of the cooling agent forces a small slug of unvaporized cooling agent out through tube 5 which leads to cooling zone 3 of fractionating column 1. When the cooling agent which was in contact with element 27 is forced out of tube 5 as explained, more cooling agent will rise out of container 6 through tube 31 to contact heating element 27 as before. This action as described creates a succession of small slugs of cooling agent from tube 5 the size of these slugs being determined by the quantity of heat delivered by heating element 27 and by the pressure within the container.

The quantity of heat delivered by heat element 27 is controlled by rheostat 29. The pressure within the container 6 is controlled by floating needle valve 12 and other related parts shown in Figure 2.

The heater at 22 warms the pressuring fluid as it comes in through tube 7 and prevents collection of water to any great extent from the water vapor which may be present in the compressed air and also positively prevents freezing of this water in tube 7. When other pressuring fluids are used other types of condensible and solidifiable material are prevented from forming by heater 22.

Various modifications in design of parts may be made by those skilled in the art without going outside the scope of the present invention and the foregoing explanation and accompanying drawings merely illustrate an illustrative embodiment of the invention and various changes may be made within the scope of the apparent claims without departing from the spirit of the present invention.

Having described our invention, we claim:

1. In apparatus for fractional distillation, a distilling column and means for applying heat to the lower portion thereof, means for supplying a cooling medium in heat exchange relationship with the upper portion of said column, a vapor outlet line leading from said column, means responsive to the degree of pressure change in said outlet line controlling the amount of cooling medium supplied the upper portion of said column, and direct heat exchange means in the means supplying the cooling medium for dividing the cooling medium into slugs.

2. In apparatus for fractional distillation, a distilling column and means for applying heat to

the lower portion thereof, means for supplying a cooling medium in heat exchange relationship with the upper portion of said column, a vapor outlet line leading from said column, a body of fluid responsive to the degree of pressure change in said outlet line, a valve floating on said fluid and variable in response to movement thereof to control the amount of cooling medium supplied the upper portion of said column, and direct heat exchange means in the means supplying the cooling medium for dividing the cooling medium into slugs.

3. In apparatus for fractional distillation, a distilling column and means for applying heat to the lower portion thereof, means for supplying a cooling medium in heat exchange relationship with the upper portion of said column, a vapor outlet line leading from said column, a manometer having one arm connected with said outlet line, a variable opening valve responsive to fluid movement in the other arm controlling the amount of cooling medium supplied the upper portion of said column, and direct heat exchange means in the means supplying the cooling medium for dividing the cooling medium into slugs.

4. An apparatus for dispensing slugs of a liquid cooling agent comprising in combination a Dewar flask, a source of pressure fluid comprising an inlet conduit entering said Dewar flask, an outlet conduit leaving said Dewar flask, and electrical heating means heating said outlet conduit to a degree causing slug formation, and an electrical control system, said control system comprising a power line, a circuit from the power line through said electrical heating means, and a rheostat in the circuit whereby the temperature of the heating means may be controlled.

5. An apparatus for dispensing slugs of a liquid cooling agent comprising in combination a Dewar flask, a source of pressure fluid comprising an inlet conduit entering said Dewar flask, an outlet conduit leaving said Dewar flask, electrical heating means heating said inlet conduit, and electrical heating means heating said outlet conduit to a degree causing slug formation, and an electrical control system comprising a power line, separate circuits from the power line through each electrical heating means, and a rheostat in each circuit whereby the temperature of the respective heating means may be controlled.

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