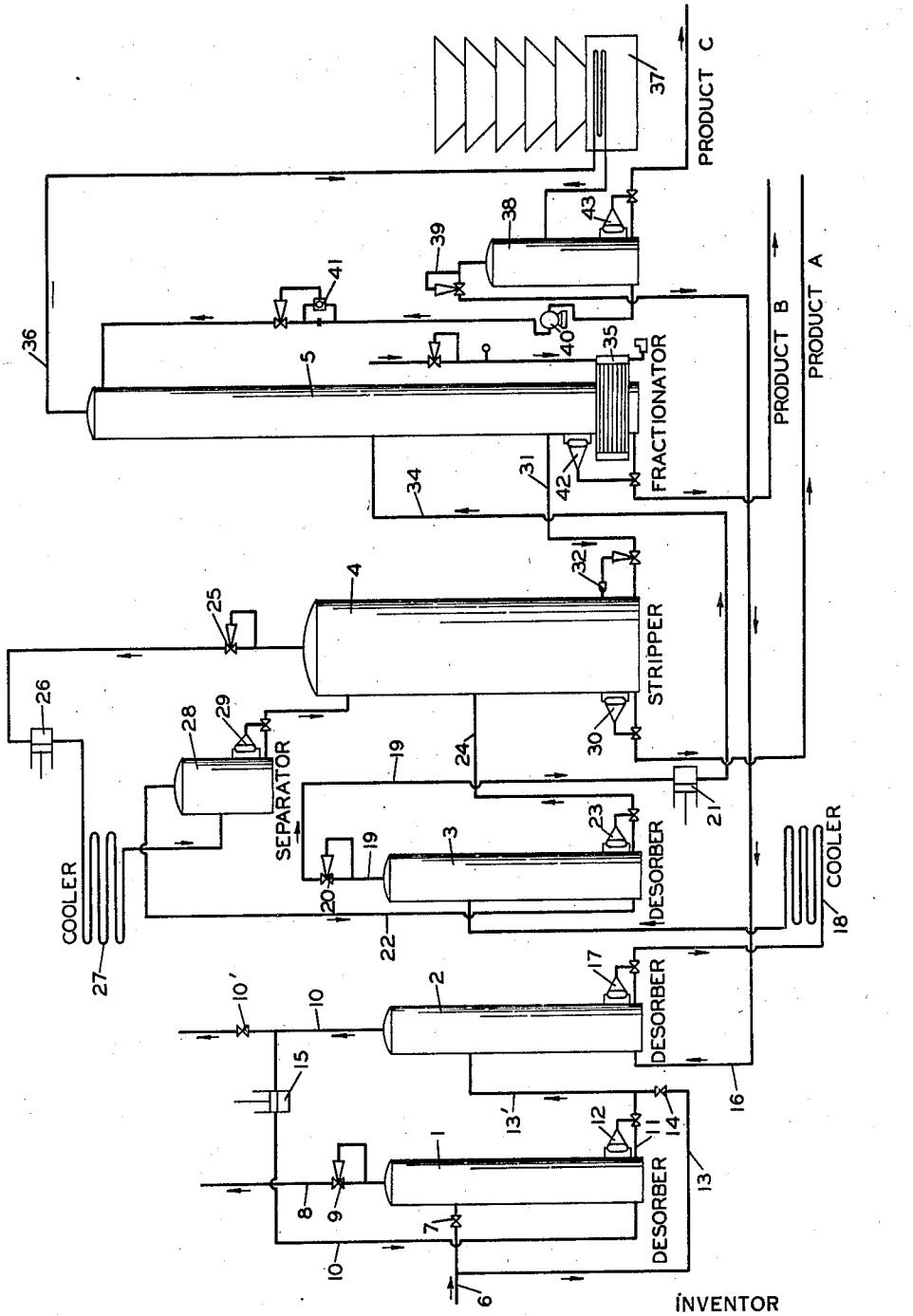
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DISTILLATE RECTIFICATION

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DISTILLATE RECTIFICATION

Samuel C. Carney, Bartlesville, Okla., assignor to Phillips Petroleum Company, a corporation of Delaware

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The present invention relates to a method of and apparatus for separating gases, vapors, and liquids. More specifically, this invention relates to the removal of undesirable gases from a mixture.

In the process of separation of petroleum into commercial products, there are many instances wherein the removal of undesirable components is either necessary to or greatly assists in the separation of the mixture being processed into fractions having desirable qualities for marketing or for feed stock to other processes. For example, hydrogen, hydrogen sulfide, methane, and inert gases are common to most crude oils and must be removed prior to or during refining. In the natural gasoline industry, the product must be free from the lower boiling hydrocarbons to be stable for storage. The present invention is described herein as applied to the rectification of the condensate obtained from distillate type wells, but it is to be understood that the invention is applicable to other problems of removal of undesirable gases.

The present recycling operation on distillate wells makes the condensate in a high pressure accumulator at a pressure of 500 to 1400 pounds per square inch by reduction of the higher well pressure, and either with or without cooling by refrigeration from sources other than expansion of the gas. At present, practice is not yet settled on what is the best accumulator pressure. Condensates from the high pressure accumulators are handled in practice by stage venting in three or more flashing operations. Stage venting effects the removal of methane but a substantial amount of the higher hydrocarbon components which are valuable as polymerization feed stock are lost by flashing off with the methane. Rectification has not been used because a rectifying column cannot be made to operate on a distillate containing methane until enough methane has been removed to permit the forming of a reflux. Even after flashing, the methane and ethane remaining in the liquid, enforces the use of a relatively high column pressure and with the high column pressure the heptane and heavier present require a kettle temperature impractically high.

Even if it were practical to use the conventional rectification of the natural gasoline industry after reasonably flashing off of methane, the great loss, of polymerization feed especially, has happened during the stage venting. It is known, and practiced to some extent, to recover desired components from the flashed gas by the use of oil absorption, but even with absorption, the situation is complicated. There are usually three flashed gases at different pressures and richer in the heavier components as the pressure declines. The rich gases are so rich as to overheat an absorber, whereas the high pressure vent gas will, due to its pressure, cause considerable methane to be dissolved in the oil. In applying conventional absorption, either the three gases may be mixed in one absorber with consequent compression of the righ gases and expansion of the leaner ones, or three separate absorbers may be used combining the rich oil streams to one still. Either way this is carried out will involve a complicated system of flash tanks and absorbers to reduce a distillate to final products after such distillate has been recovered from the well stream by refrigerated retrograde condensation.

The present invention accomplishes the removal of methane without loss of polymerization feed stock, by making use of desorbers operating on distillate instead of an absorption oil and applying the principle of preferential absorption of one component over another when contacted with an absorbing medium. Desorption alone cannot be very successfully carried out on these refrigerated distillates because their ethane content is high and desorption is most efficient on methane when using ethane vapor as a desorbing medium. The high content of heptane and heavier, which may increase if a two-phase system comes into the producing reservoir, favors desorption by reason of its higher molecular weight. Heptane and heavier are an impediment to conventional rectification or oil absorption and an increase in these components would seriously disturb operation of rectifying equipment, whereas the equipment of the present invention would operate without change in case of such an increase in the percent of heavy ends.

The cycle described herein is not a substitute for conventional rectification, but a method for the entire elimination of methane at a high pressure, around 500 to 600 pounds per square inch, and the segregation of ethane at a similar pressure so that conventional rectification will deal with a liquid comprising propane and heavier. A part of the propane, together with the ethane, is delivered as a liquid for further separation or for cracking to ethylene.

One of the objects of the present invention is to provide an improved process for the removal of undesirable lighter components from a fluid mixture.

A further object of the present invention is to provide a process particularly adaptable to recti-

fication of condensate from distillate type wells whether made by reduction of pressure and cooling or as a result of high pressure oil absorption.

A still further object of the present invention is to provide a process which, when applied to rectification of condensate, will eliminate methane and at the same time retain ethane and heavier.

Other objects and advantages will be apparent from the following detailed description, and from the accompanying drawing.

The accompanying drawing is a diagrammatic elevation of an apparatus suitable for carrying out the present invention.

Referring to the drawing, the numerals 1, 2 and 3 refer to a series of desorbers in combination with a stripping column 4 and a fractionator 5. Desorbers 1, 2 and 3 may consist of plate columns containing six to eight plates and having some empty shell near the top where flashing occurs. Pipe 6 introduces feed to the system from which it may be fed through valve 7 to desorber 1, or through pipe 13 and valve 14 to desorber 2. A pipe 8 is arranged at the upper end of desorber 1 having a back-pressure regulator 9. Pipe 10 enters the lower part of desorber 1 for admission of vapor from the top of desorber 2. Connected to the base of desorber 1 is a pipe 11 connected with a float controlled valve 12 and joining pipe 13' which enters the flash section of desorber 2. Vapor from the top of desorber 2 may be withdrawn through pipe 10 and valve 10' or compressed by compressor 15 for blowing the base of desorber 1. Pipe 16 admits vapor to the base of desorber 2 while liquid from desorber 2 is withdrawn through a float controlled valve 17 and a cooling coil 18 to be admitted to the flash section of desorber 3. A vapor pipe 19 provided with a back-pressure regulated valve 20 conducts vapors from the top of desorber 3 to compressor 21. The base of desorber 3 is blown with vapor entering through pipe 22 and discharges liquid through float controlled valve 23 and pipe 24 to the feed plate of plate-type stripper 4. Vapor going overhead from stripper 4 passes through back-pressure regulator 25 to compressor 26, where it is compressed to the pressure of desorber 3, thence through cooling coil 27 to separator 28. Liquid condensed from the vapor stream by compression and cooling is admitted as reflux to stripper 4, the flow of reflux being regulated by float-controlled valve 29. Uncondensed vapor from separator 28 blows the base of desorber 3. Float-controlled valve 30 at the base of stripper 4 effects withdrawal of product A which is sent to a conventional rectifying column. Vapor from lower plates of fractionator 5 is conducted to the base of the stripper by pipe 31 where its admission is regulated by a thermostat-controlled valve 32. Vapor from desorber 3 is compressed by compressor 21 and fed hot to the feed plate of a plate type fractionator 5 through pipe 34. High pressure fractionator 5 has a steam heating unit 35 incorporated in its base. Vapors taken overhead from column 5 by pipe 36 are conducted through cooling tower 37 and sent to accumulator 38. The uncondensed vapors from accumulator 38 pass through back-pressure regulator 39 and are conducted by pipe 16 to the base of desorber 2. Reflux pump 40 returns liquid from the bottom of the accumulator to fractionator 5 as a reflux which is regulated by flow control 41. A float-controlled valve 42 regulates withdrawal of product B from the bottom of fractionator 5 which is passed to a conventional rectifying column. Float-controlled valve 43 effects withdrawal of product C from accumulator 38.

For the purpose of illustration, a typical condensate will be taken as an example showing how this condensate may be processed eliminating methane at high pressure for recycling and conserving polymerization-feed stock for further processing. Condensate from a high-pressure accumulator is delivered to the system at 1400 pounds per square inch and 78° F., having the following composition:

	Mol per cent
Methane	37.08
Ethane	15.15
Propane	7.89
Butane	8.19
Pentane	5.99
Hexane	5.39
Heptane plus	20.31
	100.00

The sum of the partial vapor pressures of the ethane and heavier in this example at 150° F. is approximately 475 pounds per square inch. In this particular example, the use of desorber 1 is not necessary to effect the complete removal of methane. The condensate is, therefore, bypassed through pipe 13 and valve 14 to desorber 2. To remove all of the methane in this desorber it is necessary to use the combined effect of adding some ethane at its base to displace methane plus the heat of solution of ethane so that the combined vapor pressure at its base in the absence of methane is equal to the vapor pressure at its top in the presence of methane. This desorber is operated at a pressure slightly in excess of the vapor pressure of the ethane and heavier or at a pressure of 500 pounds per square inch. Methane is removed through pipe 10 and valve 10' to compressors for recycling to the producing formation. Desorber 3 is operated at a pressure of about 250 pounds per square inch and is blown with the uncondensed vapor taken from separator 28 which operates at the same pressure. Stripper 4 is operated at a pressure of about 60 pounds per square inch and is refluxed by the condensate from separator 28. Vapors from the top of desorber 3 are compressed to 500 to 600 pounds per square inch and fed hot to fractionator 5 which operates in that pressure range. Fractionator 5 is a conventional rectifying column with vapor feed. Its kettle is steam heated and it is refluxed in the conventional manner. It is operated with regulated heat input at its base with a bottom product (product B) free from ethane and consisting chiefly of propane with minor amounts of butane and heavier. Its reflux at 500 pounds per square inch is about 75% ethane and 25% propane. "Excess reflux" or product C of the same composition as the reflux is withdrawn as a liquid from accumulator 38 for rectification, polymerization, alkylation, or cracking, depending upon the circumstances. Vapors from accumulator 38 containing 90 to 95% ethane, together with some propane and heavier, are sent to desorber 2 to displace methane.

Reflux to fractionator 5 is regulated by flow controlled valve 41, so balanced with heat input as to give a bottom product ethane free. Back-pressure regulator 39 is a control for the entire desorbing operation. It is set by the operator at a pressure which, at the condenser temperature, will condense the ethane in the original feed to-

gether with a part of the propane also present. If methane is not entirely removed in desorber 2, pressure will tend to rise on accumulator 38, sending more desorbing vapor to desorber 2. The operating pressure of fractionator 5 is higher than the pressure of desorber 2 to permit flow of vapor, its pressure being determined by that of the desorber.

Stripper 4 is blown by vapor, mainly propane, taken from the base of fractionator 5 by pipe 31, the adjustment being such as to give freedom of ethane to the base of the stripper. Product A from the base of the stripper is rich in the heavy components and may be sent to a lower plate of the conventional rectifying column to which product B is sent.

To avoid excessive load on the high pressure compressor 21, the pressure on the desorber which completes the removal of methane, desorber 2 in this example, should not be much higher than the sum of the vapor pressures of components other than methane at the temperature of the base of that desorber. In other words, the pressure of the high pressure desorber cannot be raised indefinitely to give a leaner gas from its top. If the character of the condensate feed is such that desorber 2 will not give good separation of methane, desorber 1 is used to remove a part of the methane at a higher pressure. In using desorber 1, valves 14 and 10' are closed and the feed admitted to the flash section of desorber 1 through valve 7. Methane is withdrawn through pipe 8 and back-pressure regulator 9 at the top of the desorber. Some of the methane of the original feed, together with the heavier components passes as a liquid through pipes 11 and 13' to the flash section of desorber 2. The final removal of methane is accomplished in desorber 2; the methane, together with some of the heavier components leaving desorber 2 through pipe 10 enters compressor 15, from which it is sent to the base of desorber 1.

Now in case of the material given as an example, the bottoms from stripper 4 will contain over 40 mol per cent of heptane and heavier. To feed such material to a rectifying column for the separation of propane as a top product will give an unreasonably high kettle temperature. Though this is no part of the invention, the bottoms of stripper 4 should be fed instead to a column whose tops are a mixture of propane and butane and to a higher feed plate in this same column should be fed liquid bottoms from column 5. This column should operate with tops totally condensed and these tops be pumped to a column at higher pressure having propane tops and butane bottoms.

By combining the methods of desorption and rectification, methane is removed at high pressure while retaining ethane in solution in the original liquid. To remove methane at a really high pressure, two things are necessary: (1) that the original liquid contains enough ethane so that its methane free vapor pressure be relatively high and these distillates answer that requirement and (2) that the displacing vapor which desorbs the methane should be very high in ethane content so that a minimum amount of it be required, thus minimizing the compressor load. The high pressure rectifying column gives this around 95% ethane vapor for use as high pressure desorbing medium for methane. It also gives a reasonably high propane vapor from its base as the final desorbing medium for ethane in the low pressure stripper.

The reason desorption alone is not well adapted to removal of ethane, is the relatively high solubility of ethane in all heavier hydrocarbons from a vapor phase rich in ethane. While it is quite simple to desorb ethane on the lower plates of a low pressure desorber, it is quite difficult and really impractical to get it out of a desorbing system through the top plates of a high pressure desorber. Of course, this fact is exactly what makes ethane so useful as a desorbent vapor for methane in solution.

This difficulty is overcome in the cycle here described by letting the ethane out of the system by way of the high pressure rectifying column. Extreme high pressure on this column is avoided by including propane as solvent for ethane in the reflux and top product. It should also be noted that if it be desired to condense pure ethane to the liquid form, this cycle provides the intermediate product, a mixture containing ethane and propane only, in the rectification of which the propane as kettle product can be boiled at reasonably low kettle temperature at the pressure necessary to form an ethane reflux.

It is to be understood that the operating pressures given are approximate, and are selected for the distillate herein taken as a specific example. Optimum conditions of pressure, temperature, etc., may be calculated by methods well known to those skilled in the art, the actual operating pressures being determined by the operator. It has been stated that the pressure of the desorber which completes removal of methane should be only a little above the vapor pressure of the components heavier than methane in the original feed at the temperature on the bottom plate of the desorber. The pressure on stripper 4 which completes removal of ethane should be somewhat below the vapor pressure of propane and heavier in the original distillate, which is 65 pounds per square inch at 150° F. for the example taken herein. The pressure of desorber 3 (shown at 250 pounds per square inch) is an intermediate pressure, chosen to minimize butane and heavier in its top vapor and hence in the kettle of the high pressure rectifying column. The best values of these pressures is determined during operation as those which result in the smallest load on the compressors.

I claim:

1. The process of treating distillate or condensate from high pressure wells comprising passing the distillate through a methane desorbing zone, passing the bottoms from this desorbing zone through a second desorbing zone to remove ethane, propane and heavier vapors, passing the bottoms from this second desorbing zone to a stripping zone and removing therefrom stripped distillate or condensate as bottoms and a vaporous overhead, passing said vaporous overhead to the second desorbing zone; passing the ethane, propane and heavier vapors from the second desorbing zone to a fractionating zone, removing from the said fractionating zone an ethane-free bottoms, a vaporous side stream and a vaporous overhead, condensing said fractionator overhead to form a polymerization feed stock and passing the uncondensed portion to the methane desorption zone, and passing said vaporous side stream to the stripping zone as stripping agent.

2. The process of treating distillate or condensate from high pressure wells comprising pass-

ing the distillate through a methane desorbing zone including a plurality of methane desorbing steps of successively lower pressures, passing the bottoms from the last of the methane desorbing steps through a second desorbing zone to remove ethane, propane and heavier vapors, passing the bottoms from this second desorbing zone to a stripping zone and removing therefrom stripped distillate or condensate as bottoms and a vaporous overhead, condensing said vaporous overhead and passing the condensate to the stripping zone as reflux and the uncondensed portion to the second desorbing zone; passing the ethane, propane and heavier vapors from the second desorbing zone to a fractionating zone, removing from the said fractionating zone an ethane-free bottoms, a vaporous side stream and a vaporous overhead, condensing said fractionator overhead to form a polymerization feed stop and passing the uncondensed portion to the last of the methane desorbing steps, and passing said vaporous side stream to the stripping zone as stripping agent.

3. The process of treating distillate or condensate from high pressure wells comprising passing the distillate through a methane desorbing zone including a plurality of methane desorbing steps of successively lower pressures, passing the bottoms from the last of the methane desorbing steps through a second desorbing zone to remove ethane, propane and heavier vapors, passing the bottoms from this second desorbing zone to a stripping zone and removing therefrom stripped distillate or condensate as bottoms and a vaporous overhead, condensing said vaporous overhead and passing the condensate to the stripping zone as reflux and the uncondensed portion to the second desorbing zone; compressing the ethane, propane and heavier vapors from the second desorbing zone and passing the compressed vapors to a fractionating zone, removing from the said fractionating zone an ethane-free bottoms, a vaporous side stream and a vaporous overhead, condensing said fractionator overhead to form a polymerization feed stock and passing the uncondensed portion to the last of the methane desorbing steps, and passing said vaporous side stream to the stripping zone as stripping agent.

4. The process of treating distillate or condensate from high pressure wells comprising passing the distillate through a methane desorbing zone including a plurality of methane desorbing steps of successively lower pressures, cooling the bottoms from the last of the methane desorbing steps and passing the said cooled bottoms to a second desorbing zone to remove ethane, propane and heavier vapors, passing the bottoms from this second desorbing zone to a stripping zone and removing therefrom stripped distillate or condensate as bottoms and a vaporous overhead, condensing said vaporous overhead and passing the condensate to the stripping zone as reflux and the uncondensed portion to the second desorbing zone; compressing the ethane, propane and heavier vapors from the second desorbing zone and passing the compressed vapors to a fractionating zone, removing from the said fractionating zone an ethane-free bottoms, a vaporous side stream and a vaporous overhead, condensing said fractionator overhead to form a polymerization feed stock and passing the uncondensed portion to the last of the methane desorbing steps, and passing said vaporous side stream to the stripping zone as stripping agent.

SAMUEL C. CARNEY.