

March 8, 1966

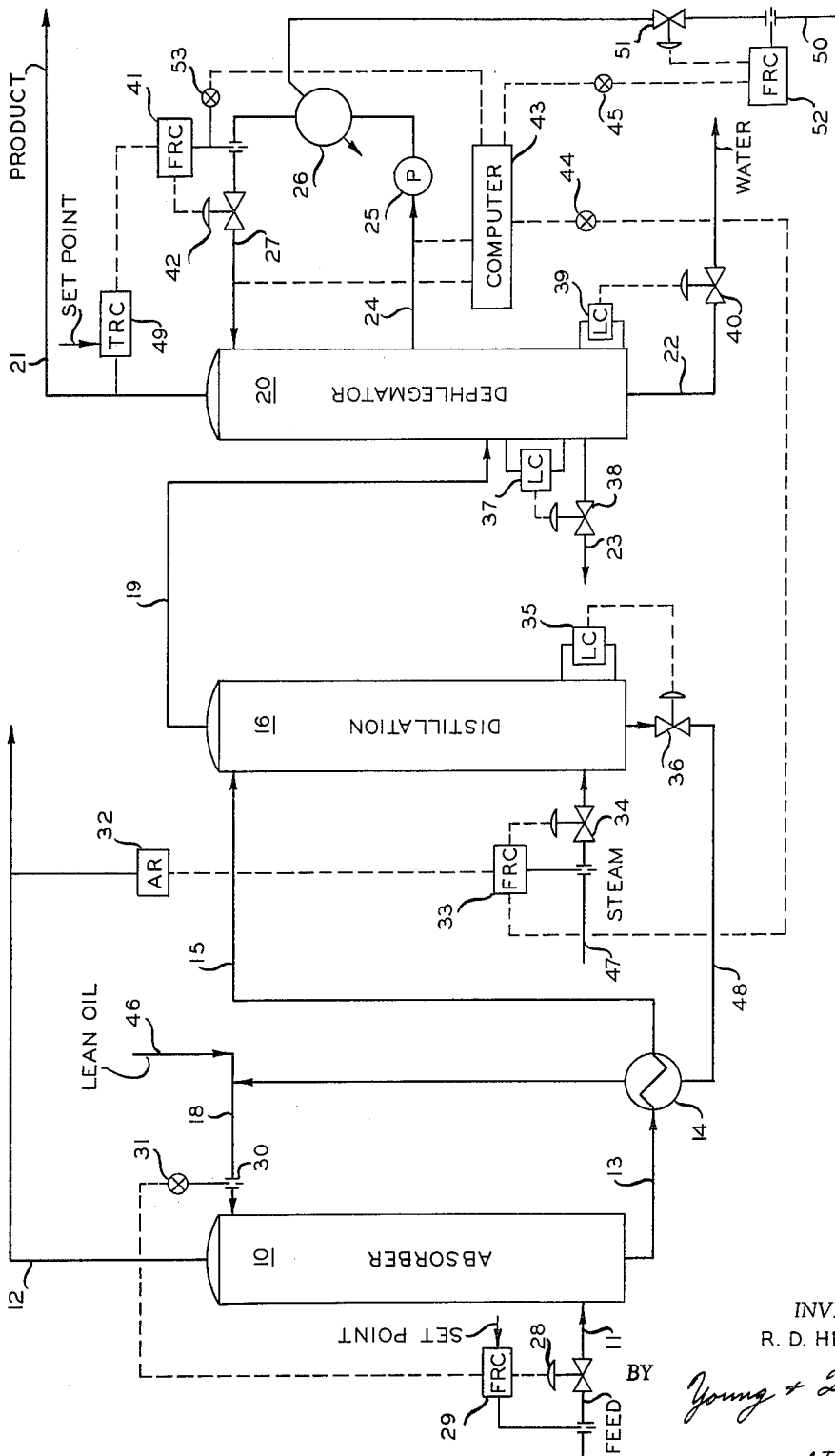
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3,239,457

SEPARATION PROCESS CONTROL METHOD

Filed Feb. 5, 1962

3 Sheets-Sheet 1



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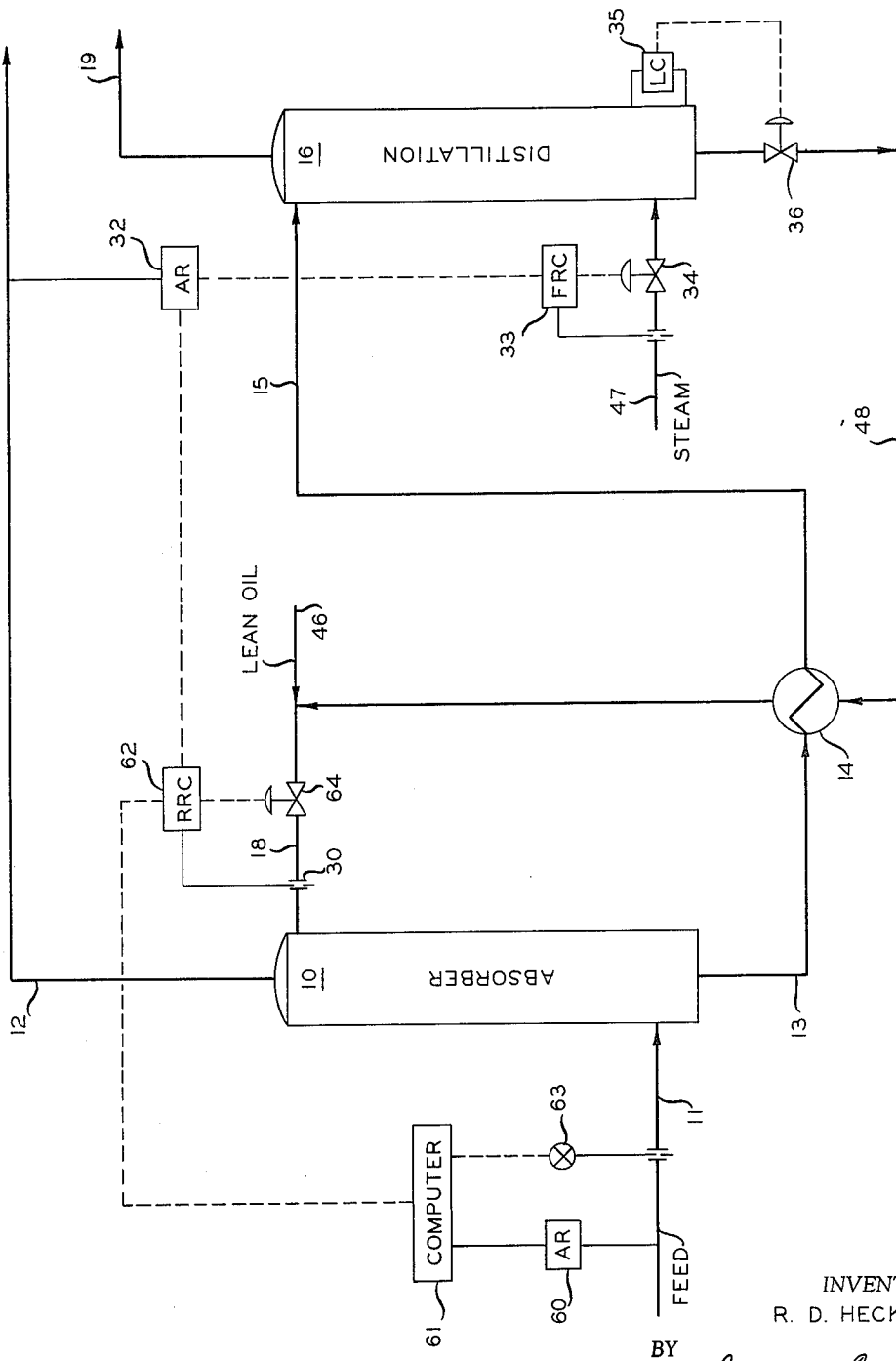
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Filed Feb. 5, 1962

3 Sheets-Sheet 2



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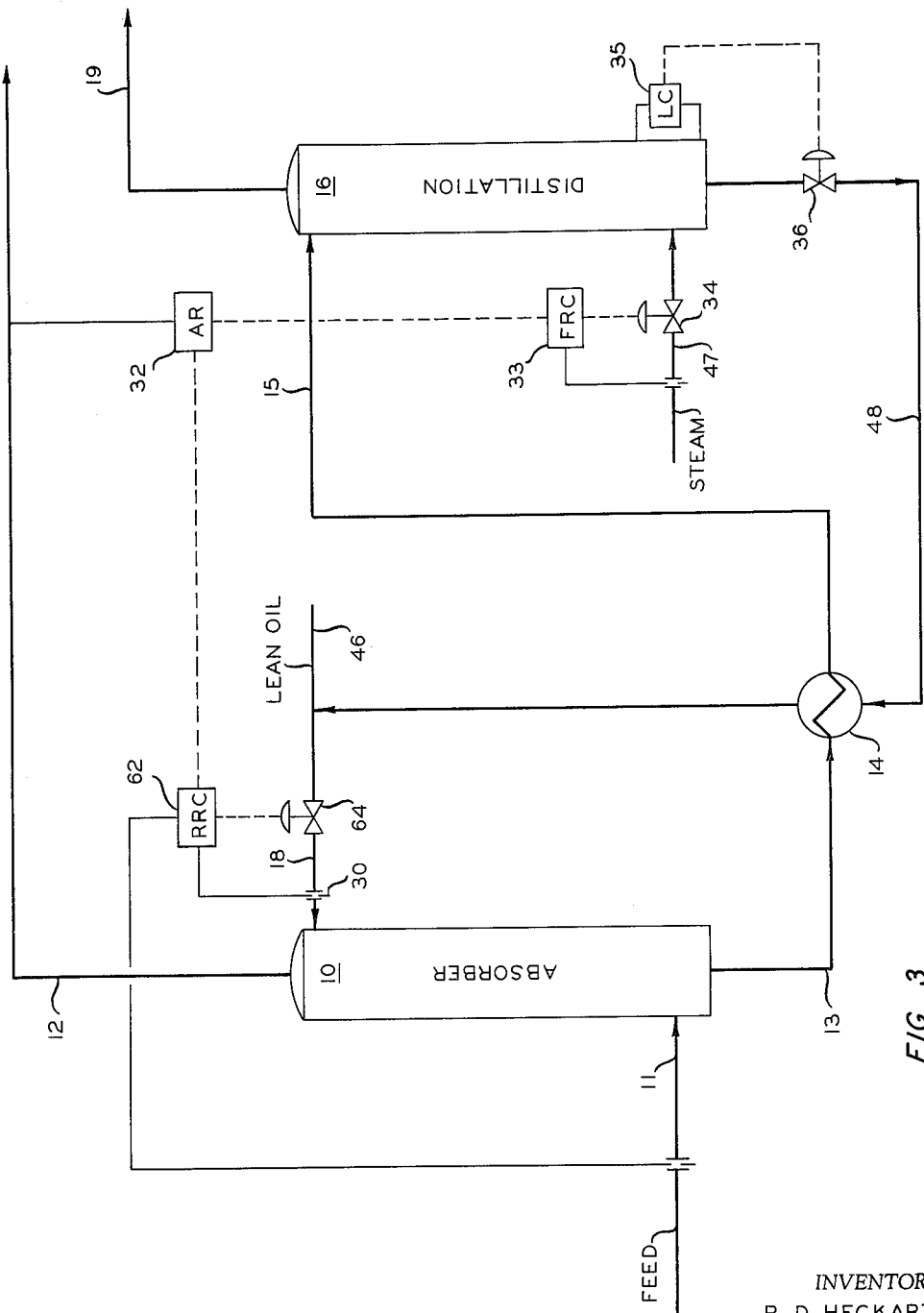
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SEPARATION PROCESS CONTROL METHOD

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3 Sheets-Sheet 3



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SEPARATION PROCESS CONTROL METHOD
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Filed Feb. 5, 1962, Ser. No. 171,021
2 Claims. (Cl. 208—341)

This invention relates to an improved method of and apparatus for controlling a separation process. In one specific aspect, this invention relates to an improved method of and apparatus for controlling a natural gasoline recovery process. In another specific aspect, this invention relates to an improved method of and apparatus for controlling a dephlegmator.

Conventionally, in the separation of C_3 and heavier hydrocarbons from natural gas, a natural gas feed is passed to an extractive distillation zone wherein said natural gas is contacted with an absorption oil. Rich oil containing C_3 and heavier hydrocarbons is withdrawn from the extractive distillation zone. An overhead vapor stream is withdrawn from the extractive distillation zone and responsive to the analysis, for example, of C_3 and heavier hydrocarbons in the overhead product stream, the rate of flow of lean absorption oil to the extractive distillation zone or the temperature of the extractive distillation zone is manipulated.

The rich oil containing C_3 and heavier hydrocarbons is passed to a distillation or stripping zone wherein said rich oil is contacted with steam. An overhead product stream comprising steam, absorption oil, C_3 and heavier hydrocarbons is withdrawn from the distillation zone and passed to a dephlegmator. An absorption oil product stream is withdrawn from the bottom of the distillation zone and recycled to the extractive distillation zone. Control of the distillation zone conventionally comprises obtaining a sample of the bottom product stream and, in the case where a distillate hydrocarbon fraction is employed as the absorption oil, laboratory distilling the sample in order to determine the concentration of the C_3 and heavier hydrocarbons remaining in the absorption oil withdrawn from the distillation zone. Responsive to this determination, the rate of flow of steam to the distillation zone is manipulated. This method of controlling the distillation zone is unsatisfactory due to the delay involved in obtaining the sample and conducting a laboratory analysis before a process variable of the distillation zone can be manipulated to effectively control the separation within the distillation zone.

I have discovered an improved method of and apparatus for controlling a separation process which comprises measuring a property of the overhead product stream withdrawn from a solvent extraction zone representative of the composition thereof and manipulating a process variable of the distillation zone and a process variable of the solvent extraction zone responsive thereto. Solvent extraction as herein employed includes liquid-liquid extraction and extractive distillation.

Within the dephlegmator or rectifying zone, the C_3 and heavier hydrocarbons are separated from the feed stream and recovered from the dephlegmator as an overhead product stream. The absorption oil and water phases are permitted to settle into the lower region of the dephlegmator and are withdrawn from the dephlegmator as separate product streams. In the operation of dephlegmators, it has been conventional practice to control the rate of addition of water reflux to the top of the dephlegmator to maintain the temperature of the overhead vapors constant. An attempt was made in the prior art to control the overhead vapor temperature so that all the absorption oil and steam contained in the

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gasoline vapors would be condensed with a minimum amount of gasoline vapors being condensed.

I have discovered an improved method of controlling the operation of a dephlegmator wherein the ratio of steam heat input to the distillation zone to the heat removed from the circulating water reflux stream of the dephlegmator is maintained constant by manipulating the cooling of the water reflux to the top of the dephlegmator.

Accordingly, an object of my invention is to provide an improved method of and apparatus for controlling a separation process.

Another object of my invention is to provide an improved method of and apparatus for controlling a solvent extraction process.

Another object of my invention is to provide an improved method of and apparatus for controlling the operation of a dephlegmator.

Another object of my invention is to provide an improved method of and apparatus for recovering natural gasoline from a natural gas feedstock.

Other objects, advantages and features of my invention will be readily apparent to those skilled in the art from the following description and the appended claims.

FIGURE 1 is a schematic representation of one embodiment of the inventive control system as applied to the recovery of natural gasoline from natural gas.

FIGURE 2 is a schematic representation of a second embodiment of the inventive control system as applied to the control of the solvent extraction step of FIGURE 1.

FIGURE 3 is a schematic representation of a third embodiment of the inventive control system as applied to the control of the solvent extraction step of FIGURE 1.

Referring to FIGURE 1, a raw natural gas feed is passed via conduit means 11 and control valve means 28 to an absorber 10. Lean absorption oil, such as kerosene having a distillation range from about 350° F. to 530° F., is passed via induit means 18 to absorber 10. Within absorber 10, the raw natural gas feed is counter-currently contacted by the lean absorption oil. An overhead vapor stream is withdrawn from absorber 10 via conduit means 12. Rich absorption oil containing, for example, C_3 and heavier hydrocarbons, is withdrawn from absorber 10 via conduit means 13 and passed via heat exchange means 14 and conduit means 15 to the upper region of distillation vessel 16.

Steam is passed to distillation vessel 16 via conduit means 47 and control valve means 34. An overhead product stream containing water vapor, absorption oil, C_3 and heavier hydrocarbons is withdrawn from distillation vessel 16 via conduit means 19 and passed to a dephlegmator 20. Absorption oil is withdrawn from the bottom of distillation vessel 16 via conduit means 48 and recycled via control valve feed 36, heat exchange means 14 and conduit means 18 to the upper region of absorber 10. Additional lean absorption oil, as required, is passed via conduit means 46 to conduit means 18 and thence to absorber 10.

A reflux water stream is withdrawn from an intermediate region of dephlegmator 20 via conduit means 24, cooled via heat exchange means 26 and recycled via pump means 25, conduit means 27 and control valve means 42 as reflux to the top of dephlegmator 20. An overhead vaporous gasoline product stream is withdrawn from dephlegmator 20 via conduit means 21. Separate water and absorption oil phases settle into the lower region of dephlegmator 20. Absorption oil is withdrawn from dephlegmator 20 via conduit means 23 and control valve means 38. The absorption oil withdrawn from dephlegmator 20 can be recycled to distillation vessel 16.

Water is withdrawn from the bottom of dephlegmator 20 via conduit 22 and control valve means 40.

Having described the process flow, the inventive control system will now be described. Generally, the concentration of the constituent or constituents of interest in the overhead product stream withdrawn from absorber 10 via conduit means 12 resulting from the concentration of the constituent or constituents of interest in the feed stream flowing through conduit 11 remains substantially constant. If, for example, it is desired to maintain the concentration of C_3 and heavier hydrocarbons in the overhead product stream at a minimum control level, the concentration of C_3 and heavier hydrocarbons in the overhead product stream will not substantially change with normal changes in the composition of the raw natural gas feed flowing through conduit 11. The remainder of the C_3 and heavier hydrocarbons contained in the raw natural gas feed is withdrawn from absorber 10 with the rich oil flowing through conduit means 13.

If separation of the C_3 and heavier hydrocarbons from the absorption oil is not complete upon contact with the steam in distillation vessel 16, the absorption oil recycled via conduit means 18 to the top of absorber 10 will contain C_3 and heavier hydrocarbons. At least a portion of the C_3 and heavier hydrocarbons recycled to the top of absorber 10 will pass overhead from absorber 10 via conduit means 12, thereby increasing the concentration of C_3 and heavier hydrocarbons in the overhead product stream beyond the desired control level or control range. The concentration of C_3 and heavier hydrocarbons flowing through conduit means 12 is determined by a conventional analyzer 32 such as a chromatographic analyzer, which includes a recorder-transmitter. Instrumentation of this type is manufactured by Perkin-Elmer Corporation and others. When employing a chromatographic analyzer-recorder-transmitter, a signal representative of the concentration of C_3 and heavier hydrocarbons is transmitted to a conventional flow-recorder-controller 33. Flow-recorder-controller 33 manipulates control valve means 34, thereby adjusting the equilibrium of the distillation zone by adjusting the rate of flow of steam to distillation vessel 16 responsive to the concentration of C_3 and heavier hydrocarbons flowing through conduit means 12. The rate of flow of steam to distillation vessel 16 is thus increased with a concentration of C_3 and heavier hydrocarbons in conduit 13 above the desired control range and decreased with a concentration of C_3 and heavier hydrocarbons flowing through conduit means 12 below the desired control range.

Flow-recorder-controller 33 transmits a signal via pneumatic-to-e.m.f. transducer 44 to computer 43, said signal representative of the steam heat input to distillation vessel 16 via the steam flowing through conduit means 47. A signal representative of the temperature of the reflux water stream flowing through conduit 27 is transmitted to computer 43. A signal representative of the temperature of the recycled water stream flowing through conduit 24 is transmitted to computer 43. A signal representative of the rate of reflux water flow is transmitted via pneumatic-to-e.m.f. transducer 53 to computer 43.

Computer 43 is capable of multiplying the difference in the signals representative of the temperatures of the water entering and leaving the dephlegmator by the signal representative of reflux flow rate and dividing the product into the signal received from flow-recorder-controller 33. An instrument capable of performing the necessary subtraction, multiplication and division is a conventional small analog computer model TR-10 manufactured by Electronic Associates, Long Branch, New Jersey. Although an electronic computer means is herein illustrated, it is within the scope of this invention to employ pneumatic instruments capable of performing the necessary subtraction, multiplication and division functions.

A signal is transmitted from computing means 43 via e.m.f.-to-pneumatic transducer 45 as a set point to flow-recorder-controller 52. Flow-recorder-controller 52 manipulates control valve 51 responsive to the set point signal received from computing means 43, thereby holding at a constant desired level the ratio of heat input to the distillation vessel 16 via the steam to the heat withdrawn from the recycled water reflux stream via heat exchange means 26. Although, as illustrated, the rate of flow of cooling medium is controlled, it is also within the scope of this invention to control the temperature of the cooling medium responsive to a set point signal transmitted by computer 43.

Conduit means 18 is provided with a rate of flow sensing means such as an orifice 30 across which a pressure differential is developed. This pressure differential pressure is transmitted by a differential pressure transmitter 31 to a conventional flow-ratio-controller 29, thereby maintaining the ratio of lean absorption oil to the raw natural gas feed substantially constant. The absorption oil level within distillation vessel 16 is noted by liquid level controller 35, liquid level controller 35 opening or closing valve 36 to thus maintain a level of absorption oil within distillation vessel 16 substantially constant.

The withdrawal of absorption oil from dephlegmator 20 can be controlled by determining the liquid level of absorption oil within dephlegmator 20 by means of a liquid level controller 37 and manipulating control valve 38 to thereby maintain the level of absorption oil within dephlegmator 20 substantially constant. The rate of withdrawal of water from dephlegmator 20 can be controlled by sensing the water level within dephlegmator 20 by means of a liquid level controller 39 and opening or closing control valve 40 responsive to the water level within dephlegmator 20.

Flow-recorder-controller 41 opens or closes control valve means 42 responsive to a set point signal transmitted by a conventional temperature-recorder-controller 49, said set point signal representative of the difference between the measured temperature of the fluid flowing through conduit means 21 and the desired fluid temperature.

Referring to FIGURE 2, the invention control system is illustrated as applied to the solvent extraction and distillation steps wherein changes in feed composition may substantially effect the concentration of the constituent or constituents in the overhead product stream withdrawn from the solvent extraction zone. For purposes of illustration it is assumed that it is desired to recover C_3 and heavier hydrocarbons from the natural gas feed and that the concentration of C_3 hydrocarbons in conduit 12 is representative of the effectiveness of the solvent stripping step in distillation vessel 16.

The composition of the feed stream flowing through conduit 11 is determined by conventional analyzer-recorder 60. As previously noted, a conventional instrument capable of determining the composition of the feed flowing through conduit 11 is a chromatographic analyzer. Analyzer-recorder 60 transmits a signal representative of the concentration of C_3 hydrocarbons to a computer 61. The rate of flow of natural gas feed through conduit 11 is determined and a signal representative of said rate of flow determination is transmitted by a conventional transmitter 63 to computer 61.

Computer 61 is an instrument capable of multiplying the input signal by another input signal and transmitting a third signal responsive thereto. Computer 61 can be a conventional pneumatic or electronic computer. Computer 61 transmits a signal representative of the rate of C_3 hydrocarbons flowing through conduit 11 to a conventional ratio-recorder-controller 62.

The concentration of C_3 hydrocarbons flowing in conduit 12 is determined by conventional analyzer 32 and a signal representative of said determination is transmitted as a reset point to ratio-recorder-controller 62. Ratio-

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recorder-controller 62 opens and closes valve means 64 responsive to the rate of flow of C_3 hydrocarbons through conduit 11 and the rate of flow through conduit 18 so as to maintain the concentration of C_3 hydrocarbons in conduit 12 substantially constant.

Analyzer-recorder 32 also determines the concentration of C_6 hydrocarbons flowing in conduit 12 and transmits a signal to flow-recorder-controller 33 responsive thereto. Flow-recorder-controller 33 opens or closes valve means 34 responsive to the signal received from analyzer 32 so as to maintain the concentration of C_6 hydrocarbons flowing in conduit 12 substantially constant.

FIGURE 3 illustrates another embodiment of the inventive control system as applied to the solvent extraction step. As in the case of FIGURE 2, analyzer-recorder 32 transmits a signal to ratio-recorder-controller 62 representative of the concentration of C_3 hydrocarbons flowing in conduit 12. Ratio-recorder-controller 62 opens or closes valve means 64 responsive to a rate of flow measurement in conduits 11 and 18 so as to maintain the concentration of C_3 hydrocarbons flowing in conduit 12 substantially constant.

Although two analyzers have been illustrated in FIGURE 2, it is within the scope of the invention to employ only one analyzer-recorder and to alternately pass samples of the feed and overhead streams to said analyzer-recorder.

The following example is presented as illustrative of the inventive control system.

Example

A raw natural gas feed at the rate of 22,608 Mscfd is passed to absorber 10, said raw natural gas feed having the following composition:

	Mol percent
C_1 -----	71.5
C_2 -----	17.2
C_3 -----	7.5
C_4 -----	2.6
$C_5 (+)$ -----	1.2
	100.0

Kerosene at the rate of 266,662 g.p.d. is passed via conduit means 18 to absorber 10. Absorber 10 is operated at a temperature of 118° F. and at a pressure of 1040 p.s.i.a. An overhead vaporous product stream containing 1.9 volume percent of C_3 and heavier hydrocarbons, as determined by a chromatographic analyzer 32, is withdrawn from absorber 10 via conduit means 12. A rich absorption oil stream is withdrawn from absorber 10 via conduit means 13, heated to a temperature of 450° F. by heat exchange means 14 and a furnace not herein illustrated and passed via conduit means 15 to distillation vessel 16.

Steam at the rate of 3500 pounds per hour is passed via conduit means 47 to distillation vessel 16, said steam representative of the addition of 4,208,000 B.t.u. per hour passed via the steam to distillation vessel 16. Kerosene at the rate of 266,436 g.p.d. is withdrawn from distillation vessel 16 via conduit means 48, cooled to a temperature of 108° F. via heat exchange means 14 and a cooler not herein illustrated, and passed via conduit means 18 to absorber 10. Additional kerosene is passed to conduit 18 via conduit means 46 at the rate of 226 g.p.d. Distillation vessel 16 is operated at a temperature of 435° F. and at a pressure of 263 p.s.i.a. An overhead product stream comprising water vapor, C_3 and heavier hydrocarbons, and kerosene is withdrawn from distillation vessel 16 via conduit means 19 and passed to dephlegmator 20.

Dephlegmator 20 is operated at a bottom temperature of 322° F., a top temperature of 200° F., and at a pressure of 261 p.s.i.a. Water is withdrawn from dephlegmator 20 via conduit means 24 at the rate of 132,450 g.p.d. and having a temperature of 250° F. The withdrawn water is cooled to a temperature of 144° F. by means of heat

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exchange means 26. The cooling of the recycled water represents a heat withdrawal rate of 4876M B.t.u. per hour. Kerosene at the rate of 5760 g.p.d. is withdrawn from dephlegmator 20 via conduit means 23.

5 Water at the rate of 9705 g.p.d. is withdrawn from dephlegmator 20 via conduit means 22. An overhead product gasoline stream is withdrawn from dephlegmator 20 via conduit means 21, said overhead product stream having the following composition:

	Mol percent
H_2O -----	2.5
C_1 -----	5.4
C_2 -----	27.4
C_3 -----	43.2
C_4 -----	15.0
$C_5 (+)$ -----	6.5

The inventive method of control has been defined as applied to the recovery of natural gasoline from natural gas. It is not intended to limit the invention thereto. The inventive method of control can be applied to other extractive distillation processes and to liquid-liquid extraction processes such as the separation of isoprene from a stream containing isoamylenes utilizing furfural as the selective solvent.

As will be evident to those skilled in the art, various modifications of this invention can be made, or followed, in the light of the foregoing disclosure and discussion without departing from the spirit or the scope thereof.

I claim:

1. In a process for recovering desired constituents from a fluid mixture comprising contacting said mixture with a solvent selective for said constituents in a dissolution zone, withdrawing residue fluid from said dissolution zone, passing enriched solvent from said dissolution zone to a distillation zone, passing steam to said distillation zone, passing lean solvent from said distillation zone to said dissolution zone, passing said constituents from said distillation zone to a rectifying zone; withdrawing water from an intermediate region of said rectifying zone, removing heat from said water and recycling the cooled water to the upper region of said rectifying zone; withdrawing said constituents from said rectifying zone, withdrawing water from said rectifying zone, and withdrawing solvent from said rectifying zone, the improvement in control which comprises determining the concentration of said constituents in said residue fluid, manipulating the flow of steam to said distillation zone in response to said determined concentration, determining the heat input to said distillation zone, and manipulating the rate of heat removal from said rectifying zone in response to said determined heat input to maintain the ratio of heat input to the heat removed substantially constant.

2. In a process for recovering natural gasoline constituents from a raw natural gas comprising contacting said gas with lean oil selective for said natural gasoline constituents in an absorber, withdrawing residue gas from said absorber, passing steam to said distillation zone, passing enriched oil from said absorber to a distillation zone, withdrawing and passing lean oil from said distillation zone to said absorber, withdrawing and passing said natural gasoline constituents from said distillation zone to a rectifying zone; withdrawing water from an intermediate region of said rectifying zone, removing heat from said water, and recycling the cooled water to the upper region of said rectifying zone; withdrawing said natural gasoline constituents from said rectifying zone, withdrawing water from said rectifying zone, and withdrawing lean oil from said rectifying zone, the improvement in control which comprises measuring the concentration of said constituents in said residue gas and manipulating the rate of steam passing to said distillation zone responsive to said measurement so as to regulate the recovery of said natural gasoline constituents from said enriched oil, passing a first signal representative of the steam heat input of said distillation zone to a computing zone, passing

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a second signal representative of the temperature of said withdrawn water to said computing zone, passing a third signal representative of the temperature of said cooled recycle water to said computing zone, passing a fourth signal representative of the rate of flow of recycle water to said computing zone, and manipulating the rate of heat removal from said rectifying zone in response to a fifth signal from said computing zone based on said first, second, third and fourth signals to improve the recovery of said natural gasoline constituents.

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