April 7, 1959 E. C. SPANN ET AL 2,881,118

FRACTIONATION COLUMN CONTROL

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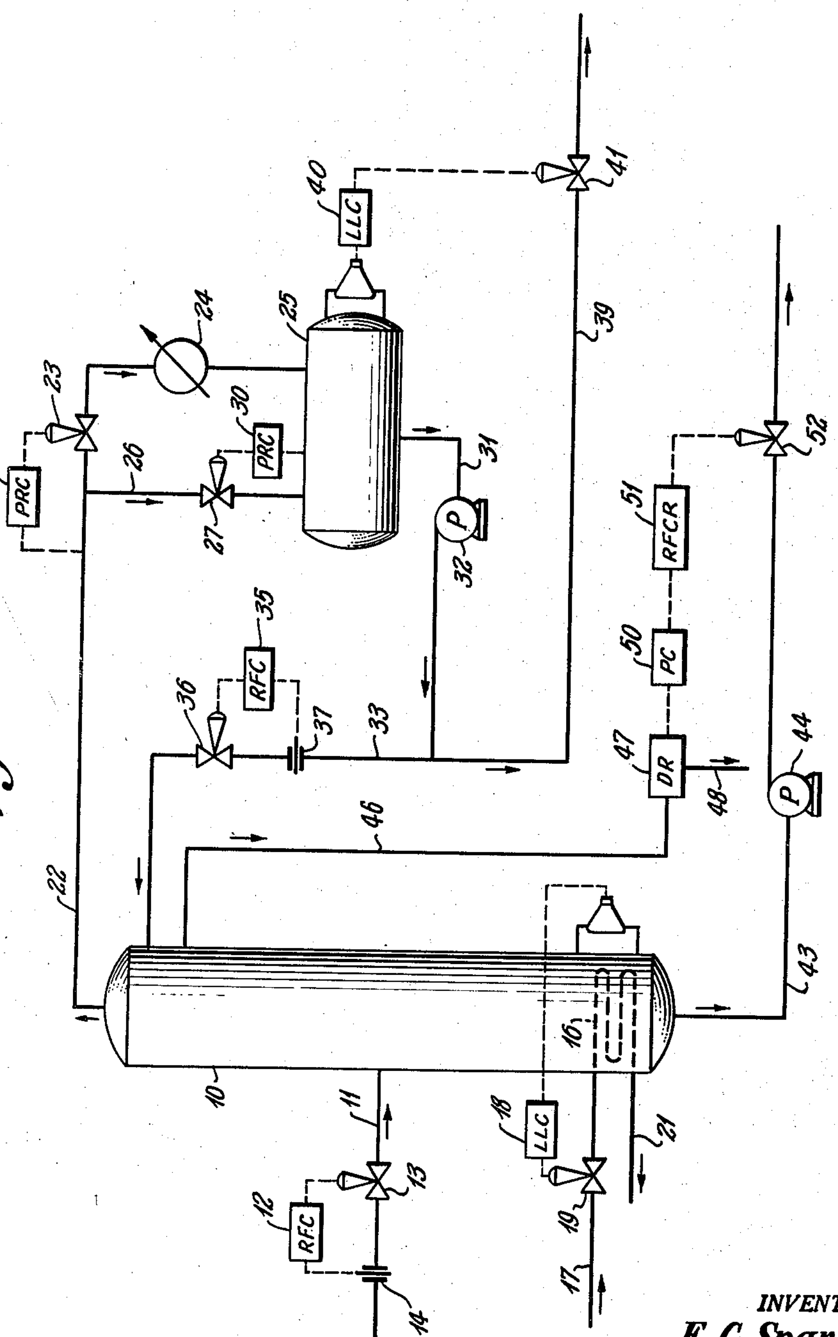
Fig. 1.



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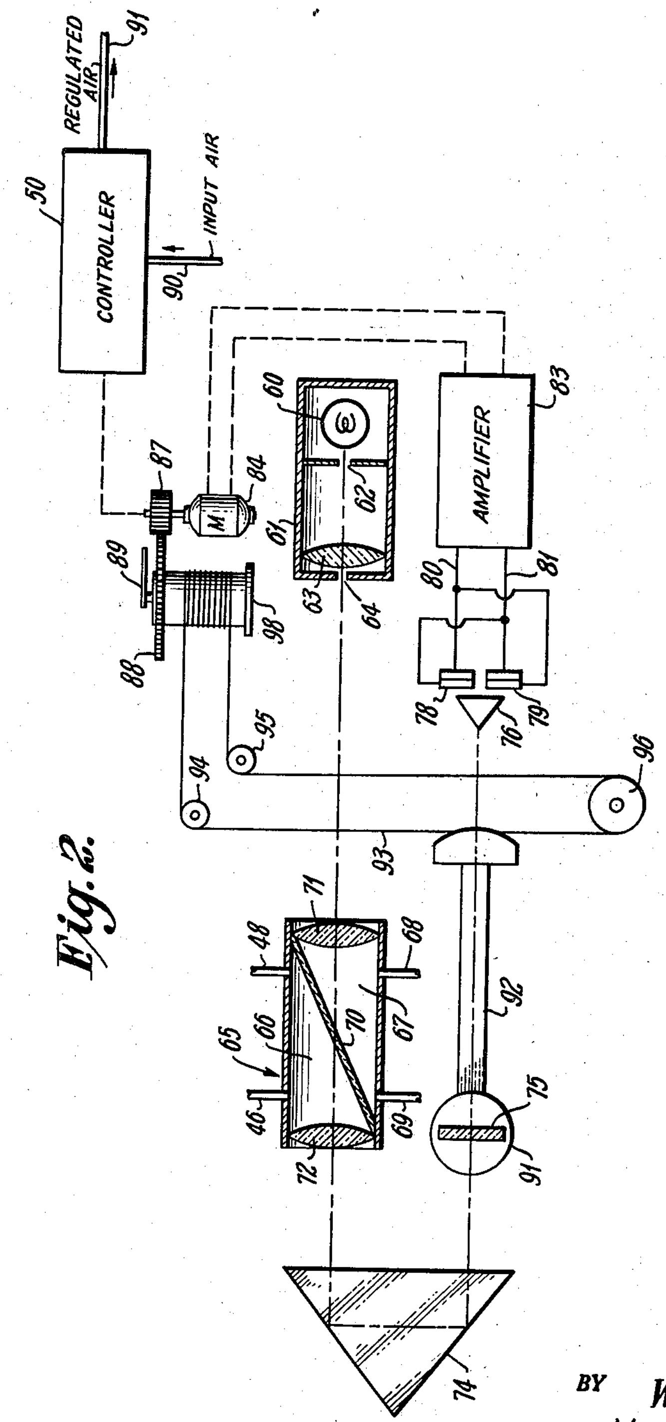

Fig. 2.

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FRACTIONATION COLUMN CONTROL

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4 Claims. (Cl. 202—160)

This invention relates to control systems for fractionation columns.

Fractionation comprises, generally, a series of vaporizations and condensations which are performed to separate a feed stream into two or more product streams by means of the difference in vapor pressure or boiling points of the constituents being separated. At each step, the vapor leaving the boiling liquid contains more of the material with the lower boiling point than does the remaining liquid. Furthermore, as this vapor is condensed the liquid that condenses first is richer in the material with the highest boiling point. A fractionation column normally comprises a series of bubble trays which are placed one above the other. These trays are designed so that vapors from a lower tray pass through the liquid in the tray next above. This action condenses a portion of the heavier materials in the vapors, and at the same time vaporizes a portion of the lighter liquid on the tray. In this manner, each tray acts as a reboiler for one distillation unit and as a condenser for the preceding unit. The liquid level on each tray is maintained by a weir placed at one edge thereof. When the liquid level on a tray increases above the weir, the liquid overflows to the next tray therebeneath. The pressure on each tray is maintained for a given throughput by the depth that the bubble cap is submerged below the surface of the liquid and by the friction of the vapors through the bubble caps. The temperature at each tray is maintained by the composition of material on the tray because each tray is at its boiling point, and the equilibrium temperature of the liquid at its boiling point is dependent upon the pressure. Thus, by controlling the pressure at one point in the column, the design of the column will regulate the pressure throughout the column.

It generally is desired that fractionation columns be operated such that the composition of at least one of the product streams remains constant at a predetermined value. This control is accomplished in accordance with the present invention by removing a sample stream from a selected region of the fractionation column and analyzing the stream to determine the composition thereof. This analysis preferably is accomplished by a differential refractometer which indicates the difference between refractive indices of the sample stream and a reference material. The kettle product withdrawal rate from the fractionation column then is adjusted in response to the analysis to maintain the composition of the sample stream within predetermined limits. This can be accomplished by adjusting the column to keep the refractive index of the sample constant.

Accordingly, it is an object of this invention to provide an improved control system for fractionation columns.

A further object is to provide apparatus to control a fractionation column by regulating the kettle product withdrawal rate in response to an analysis made of a sample stream removed from the column.

A still further object is to provide a fractionation control system utilizing a differential refractometer.

Various other objects, advantages and features of this invention should become apparent from the following detailed description taken in conjunction with the accompanying drawing in which:

Figure 1 is a schematic view of the fractionation column control system of the present invention; and

Figure 2 is a schematic representation of a differential refractometer suitable for use in the control system of Figure 1.

Referring now to the drawing in detail and to Figure 1 in particular, there is shown a fractionation column **10** which is supplied with an input feed stream through a line **11** which enters an intermediate section of column **10**. This feed stream enters the column at a predetermined rate which is maintained by a rate-of-flow controller **12** which adjusts a valve **13** in line **11** in response to the pressure differential across an orifice **14** in line **11** upstream from valve **13**. Heat is supplied to column **10** by a steam coil **16** disposed within the lower portion of the column. Steam is passed into coil **16** through a line **17** at a rate which is adjusted by a liquid level controller **18** which is actuated by the level of liquid in the bottom of column **10**. Controller **18** regulates a valve **19** in line **17** to supply steam at a rate sufficient to maintain a predetermined level of liquid within column **10**. The spent steam from coil **16** is removed through an outlet line **21**.

A vapor stream is removed from the top of column **10** through a line **22** which passes through a valve **23** and a cooler **24** into a reflux accumulator **25**. A second bypass line **26** having a valve **27** therein communicates between line **22** and accumulator **25**. Valve **23** is regulated by a pressure recorder-controller **28** which adjusts valve **23** in response to the pressure in line **22**. Valve **27** is regulated by a pressure recorder-controller **30** which adjusts valve **27** in response to the pressure in accumulator **25**. Valves **23** and **27** thus control the relative amount of overhead vapor that is condensed by cooler **24** so as to maintain a desired operating pressure on the top of column **10**. The condensed vapor in accumulator **25** is removed through a line **31** having a pump **32** therein. A portion of the liquid pumped through line **31** passes through a reflux line **33** back into the upper portion of column **10**. A constant rate of flow is maintained in line **33** by a rate-of-flow controller **35** which adjusts a valve **36** in line **33** in response to the pressure differential across an orifice **37** disposed in line **33** upstream from valve **36**. The remainder of the liquid pumped through line **31** passes through an overhead product line **39**. The rate of flow through line **39** is maintained at a value which is a function of the level of liquid in accumulator **25** by means of a liquid level controller **40** which adjusts a valve **41** in line **39** in response to the liquid level in accumulator **25**.

A kettle product is removed from column **10** by a line **43** having a pump **44** therein. A sample stream is removed from the upper portion of column **10** through a line **46** which communicates with a differential refractometer **47**. From differential refractometer **47**, this sample is vented through a line **48**. Differential refractometer **47** provides an output signal that is a function of the refractive index of the sample stream circulated therethrough. This signal is applied to a pressure controller **50** which provides a proportional output air pressure to a rate-of-flow recorder-controller **51**. The output signal from controller **51** adjusts a valve **52** in line **43** to regulate the rate of withdrawal of kettle product from column **10**. This rate of withdrawal is adjusted so that the refractive index of the sample stream removed through line **46** remains constant at a predetermined value. As long as this sample stream has a constant refractive index it is known that column **10** is operating in a uniform manner.

A suitable differential refractometer which can be employed in this invention is illustrated schematically in Figure 2. The refractometer comprises a source of light **60** mounted in a housing **61**. Source **60** can be an ordinary incandescent bulb emitting radiation in the visible spectrum. Light emitted from source **60** passes through a first aperture **62** and thence through a converging lens **63**. A narrow beam of light emerges from housing **61** through a second aperture **64** and is directed through a refractometer cell arrangement **65**. The purpose of aperture **62** is to reduce the total transmitted radiation from source **60** to avoid excessive heating of the cell arrangement. The filament of source **60** is near the focal point of lens **63**, but slightly therebeyond; and aperture **64** is disposed in close proximity to lens **63**. Cell **65** includes a first chamber **66** connected to inlet line **46** and outlet vent line **48** through which a sample of the liquid from column **10** is circulated. Cell **65** also includes a second chamber **67** provided with an inlet conduit **68** and an outlet conduit **69** which are adapted for filling chamber **67** with a standard liquid having a refractive index approximating the refractive index of one of the components of the mixture being separated. Chambers **66** and **67** are separated by a diagonal transverse plate **70** constructed of a material such as glass which is transparent to the light beam from source **60**. A converging lens **71** defines one opening of chamber **67** and a second converging lens **72** defines a corresponding opening of chamber **66**. The components thus far described are arranged such that the aperture **64** is at the effective principal focus of lens **71**. In this manner a narrow beam of parallel light enters chamber **67** and emerges from chamber **66** through lens **72** after passing through diagonal plate **70**.

The light beam energing from lens **72** enters a glass prism **74** disposed such that its front surface is perpendicular to the path of light. The light beam is twice reflected in prism **72** and emerges therefrom to pass through a rotatable block of glass **75** having its two surfaces substantially perpendicular to the path of radiation. From glass block **75**, the light beam passes through a second prism **76** disposed such that the light beam normally strikes the apex in a line perpendicular to its base. A radiation detector unit comprising first and second photovoltaic cells **78** and **79** is positioned such that the light beam striking the apex of prism **76** normally impinges equally upon adjacent cells **78** and **79**. The outputs of cells **78** and **79** are connected in opposition by means of electrical leads **80** and **81** so as to produce a resulting voltage proportional to the difference in total radiation incident upon the two cells. The voltage appearing between leads **80** and **81** is amplified by an amplifier **83**, the output of which is applied to a reversible motor **84**. The shaft of motor **84** carries a gear **87** which engages a second gear **88**. Gear **88** carries a pointer **89** mounted to indicate the degree of rotation of motor **84** produced by the output electrical signal from amplifier **83**.

Glass block **75** is mounted centrally on a rotatable base **91** having a pivot point at the center thereof. Base **91** is provided with an arm **92** which is attached to a cable **93**. Cable **93** passes about suitable support posts **94**, **95** and **96** and is wrapped about the shaft **98** attached to gear **88**. Thus, rotation of gear **88** in response to the output signal from amplifier **83** moves cable **93** to rotate glass block **75** about its mid point. The shaft of motor **84** is also mechanically coupled to pressure controller **50**.

If the refractive indices of the gases contained in chambers **66** and **67** are equal, the light beam emerging from cell **65** is in optical alignment with the light beam entering cell **65**. The apparatus is positioned initially such that an undeviated light beam strikes the apex of prism **76** and is thereby directed in equal intensities upon cells **78** and **79**. However, should the refractive indices of the two gases differ from one another, the emerging light beam is deviated in one direction or the other by cell **65** such that a greater intensity of radiation is incident upon either cell **79** or cell **78**. This in turn causes an unbalanced voltage, which after amplification, drives motor **84**. The rotation of motor **84** in turn drives shaft **98** to rotate glass block **75** through the connecting linkage cable **93**. This rotation of block **75** is such as to deviate the light beam in the opposite direction, and continues as long as unequal intensities of radiation are incident upon cells **78** and **79**. The degree of this rotation, as indicated by pointer **89**, is, therefore, the measure of the difference in refractive indices between the two gases in cell **65**, and this rotation also adjusts the output air pressure from controller **50**.

Pressure controller **50** can be a commercially available instrument such as the Brown Air-O-Line air-operated controller which is described in Catalog No. 8905 of the Brown Instrument Company, Philadelphia, Pennsylvania. Air is supplied to controller **50** by a line **90** at a constant pressure, such as twenty pounds per square inch. The input signal applied to controller **50** from motor **84**, which can be either a mechanical rotation or an electrical signal from a telemetering transmitter actuated by motor **84**, serves to vary the output air pressure of controller **50** to provide an output air pressure proportional to the rotation of motor **84**. This regulating air pressure is applied by a line **91** to the pneumatic reset rate-of-flow controller **51** which can be a commercially available instrument such as the Taylor Pneumatic Set Controller which is described in Bulletin 98159, March 1944, of the Taylor Instrument Companies, Rochester, New York.

The control system of this invention has been applied to a fractionation column having 150 trays. The input stream passed to column **10** was at a rate of approximately 40,000 gallons per day. Approximately 23,400 gallons per day of liquid were removed as the kettle product through line **43** with approximately 16,600 gallons per day of overhead product being removed through line **39**. The reflux passed back to column **10** through line **33** was at a rate of approximately 150,000 gallons, per day. The temperature at the bottom of column **10** was maintained at approximately 245° F. with a top temperature of approximately 195° F. The pressure on the top of column **10** was maintained at approximately 15 pounds per square inch gauge, the bottom pressure being approximately 36½ pounds per square inch gauge.

In the following table the composition of the mixture being separated is given for the feed stream, the kettle product and the overhead product. The refractive indices of the constituents are also given.

Component	Stream composition, liquid volume percent			Refractive index (20° C.)
	Feed	Kettle product	Overhead product	
Normal hexane	2.6	0.0	7.8	1.3749
Methylcyclopentane	27.0	10.0	60.7	1.4097
2,2-dimethylpentane	2.7	1.6	4.8	1.3822
Benzene	0.6	Trace	1.9	1.5011
2,4-dimethylpentane	5.0	4.4	6.2	1.3815
Cyclohexane	59.8	80.8	17.9	1.4262
3,3-dimethylpentane	0.5	0.8	0.0	1.3909
1,1-dimethylcyclopentane	0.2	0.0	0.7	1.4122
2-methylhexane	1.6	2.4	0.0	1.3849
	100.0	100.0	100.0	---------

The sample stream circulated through chamber 66 was removed from the 134th tray from the bottom of column 10. Chamber 67 was filled with methylcyclopentane. Controller 51 adjusted valve 52 to maintain the refractive index of the sample stream constant.

While this invention has been described in conjunction with a differential refractometer to analyze a sample stream removed from a fractionation column, other continuous analysis instruments can be employed to advantage in some applications. For example, an ultraviolet analyzer such as described in Beckman Bulletin 122B, National Technical Laboratories, South Pasadena, California, can be employed in conjunction with the separation of butadiene from 2-butenes wherein the sample stream preferably is removed from below the point of feed stream entry to the column. In the separation of furfural from 2-butenes, an infrared analyzer such as described in U.S. Patent 2,579,825 can be employed with the sample stream preferably being removed from below the point of feed stream entry to the column.

Thus, while the invention has been described in conjunction with a present preferred embodiment thereof, it obviously is not limited thereto.

What is claimed is:

1. A fractionation system comprising a fractionation column, means to supply a feed mixture to be separated to said column, a reflux accumulator, a condenser, conduit means to pass overhead vapors from said column through said condenser to said accumulator, means to maintain a predetermined pressure in said column, conduit means to return a portion of the condensed vapors from said accumulator to said column as reflux, conduit means to withdraw a portion of the condensed vapors from said accumulator as an overhead product stream, conduit means to withdraw a kettle product from said column, means to supply heat to the lower region of said column, means to measure the liquid level in the lower region of said column, means responsive to said means to measure liquid level to control the rate of heat addition to said column to tend to maintain the liquid level in said column at a predetermined height, means to withdraw a sample stream from a preselected region of said column, means to measure the refractive index of said sample stream, and means responsive to said means to measure refractive index to control the rate of kettle product withdrawal through said last mentioned conduit means to tend to maintain the measured refractive index of said sample constant.

2. A fractionation system comprising a fractionation column, means to supply a feed mixture to be separated to said column, a reflux accumulator, a condenser, conduit means to pass overhead vapors from said column through said condenser to said accumulator, means to maintain a predetermined pressure in said column, conduit means to return a portion of the condensed vapors from said accumulator to said column as reflux, conduit means to withdraw a portion of the condensed vapors from said accumulator as an overhead product stream, conduit means to withdraw a kettle product from said column, means to supply heat to the lower region of said column, means to measure the liquid level in the lower region of said column, means responsive to said means to measure liquid level to control the rate of heat addition to said column to tend to maintain the liquid level in said column at a predetermined height, first and second adjacent sample cells separated by a transparent plate, means to withdraw a sample stream from a preselected region of said column and to direct same through said first sample cell, a reference material disposed in said second sample cell, means to direct a beam of radiation in the range from ultraviolet through infrared through said cells and said plate, means to measure the deviation of the radiation beam transmitted through said cells, and means responsive to said means to measure to control the rate of kettle product withdrawal through said last mentioned conduit means to tend to maintain the deviation of the radiation beam constant.

3. A fractionation system comprising a fractionation column, a first conduit communicating with said column to supply a feed mixture to be separated, a reflux accumulator, a condenser, a second conduit communicating between the top of said column and said accumulator through said condenser, means to control the flow through said second conduit in response to the pressure in said column to tend to maintain a constant pressure in said column, a third conduit communicating between the top of said column and said accumulator, a pressure controller to regulate the flow through said third conduit in response to the pressure in said accumulator to tend to maintain a constant pressure in said accumulator, a fourth conduit communicating between said accumulator and the upper region of said column to return condensed vapors to said column as reflux, a flow controller associated with said fourth conduit to tend to maintain a predetermined flow therethrough, a fifth conduit communicating with said accumulator to withdraw an overhead product stream, means responsive to the liquid level in said accumulator to control the flow through said fifth conduit to tend to maintain a predetermined liquid level in said accumulator, means to supply heat to the lower region of said column, a sixth conduit communicating with the bottom of said column to withdraw a kettle product stream, means responsive to the liquid level in the lower region of said column to control the rate of addition of heat to said column to tend to maintain a predetermined liquid level in said column, means to withdraw a sample stream from a preselected region of said column, means to measure the refractive index of said sample stream, and means responsive to said means to measure refractive index to control the rate of kettle product withdrawal through said sixth conduit to tend to maintain the measured refractive index of said sample constant.

4. A fractionation system comprising a fractionation column, a first conduit communicating with said column to supply a feed mixture to be separated, a reflux accumulator, a condenser, a second conduit communicating between the top of said column and said accumulator through said condenser, means to control the flow through said second conduit in response to the pressure in said column to tend to maintain a constant pressure in said column, a third conduit communicating between the top of said column and said accumulator, a pressure controller to regulate the flow through said third conduit in response to the pressure in said accumulator to tend to maintain a constant pressure in said accumulator, a fourth conduit communicating between said accumulator and the upper region of said column to return condensed vapors to said column as reflux, a flow controller associated with said fourth conduit to tend to maintain a predetermined flow therethrough, a fifth conduit communicating with said accumulator to withdraw an overhead product stream, means responsive to the liquid level in said accumulator to control the flow through said fifth conduit to tend to maintain a predetermined liquid level in said accumulator, means to supply heat to the lower region of said column, a sixth conduit communicating with the bottom of said column to withdraw a kettle product stream, means responsive to the liquid level in the lower region of said column to control the rate of addition of heat to said column to tend to maintain a predetermined liquid level in said column, first and second adjacent sample cells separated by a transparent plate, means to withdraw a sample stream from a preselected region of said column and to direct same through said first sample cell, a reference material disposed in said second sample cell, means to direct a beam of radiation in the range from ultraviolet through infrared through said cells and said plate, means to measure the deviation of the radiation beam transmitted through said cells, and means responsive to said means to measure to control the rate of kettle prod-

uct withdrawal through said sixth conduit to tend to maintain the deviation of the radiation beam constant.

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