May 20, 1958 E. C. MILLER 2,835,116

INFRARED ANALYZER AND PROCESS CONTROL

Filed June 9, 1955

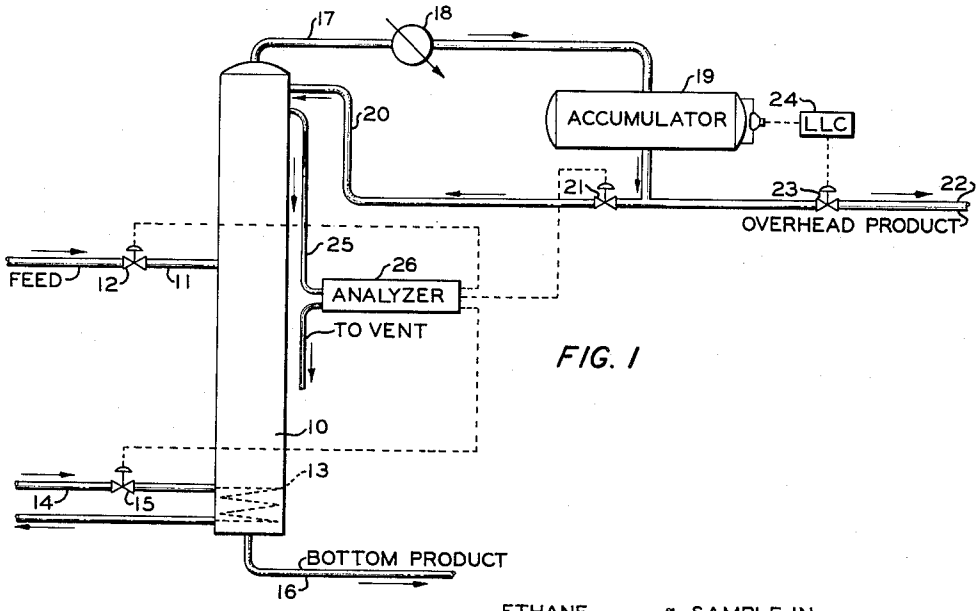


FIG. 1

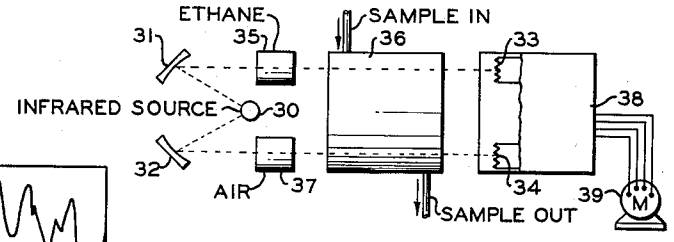


FIG. 2

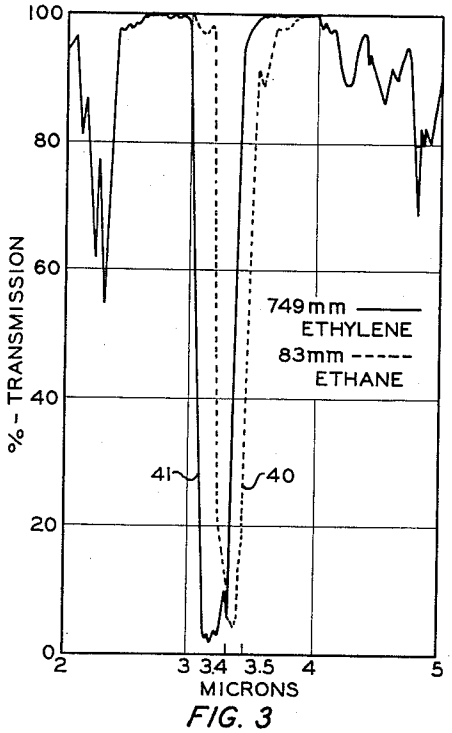


FIG. 3

INVENTOR.
E. C. MILLER

BY

Hudson & Young
ATTORNEYS.

United States Patent Office

2,835,116

Patented May 20, 1958

2,835,116

INFRARED ANALYZER AND PROCESS CONTROL

Elmer C. Miller, Bartlesville, Okla., assignor to Phillips Petroleum Company, a corporation of Delaware

Application June 9, 1955, Serial No. 514,345

5 Claims. (Cl. 62—175.5)

This invention relates to an infrared analyzer. In another aspect, it relates to a method of controlling an ethylene fractionator.

Where a stream containing ethylene and ethane is fractionated, to yield a high purity ethylene product, considerable difficulties are encountered in obtaining efficient operation of the process with production of an overhead stream of consistent purity. It has been proposed to utilize an infrared analyzer sensitized to the ethylene concentration of the overhead product. However, this has not proven satisfactory in practice because of the large concentration of ethylene in this product. Further, considerable interference is caused by the presence of light gases in the overhead product.

In accordance with this invention, a sample is withdrawn from a tray of the fractionator near the top of the column and this sample is passed through an infrared analyzer which is sensitive to the ethane concentration rather than the ethylene concentration in the overhead product. In this manner, a very efficient and accurate control is obtained, and an overhead product of the desired degree of purity is continuously produced. Moreover, the accuracy of the control and analyses is not affected by the presence of light gases, such as methane, even when they are present in substantial amounts.

The analyzer is sensitized to the ethane concentration of the overhead product by placing a filter in one beam which has a strong absorption band at wavelengths of 3.4 to 3.5 microns. A suitable material for this filter is pure ethane. When the sample passes through both beams of the analyzer, variations in ethane content affect one beam but not the other so that the output of the instrument varies as a function of the ethane content. The output of the analyzer is advantageously utilized to control an operating variable of the fractionation column such as the feed rate, reflux rate or heat supplied to the kettle so as to maintain a predetermined ethylene content in the overhead product.

Accordingly, it is an object of the invention to provide an infrared analyzer of improved construction.

It is a still further object to provide a novel system for controlling a fractionation operation wherein a high purity ethylene stream is taken overhead.

It is a still further object to provide an analyzer and control system which is very efficient in operation, and results in substantial economies processwise together with production of a product of high and consistent purity.

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

Figure 1 is a flow diagram of a process embodying the analyzer and control system of the invention;

Figure 2 is a schematic view of an analyzer constructed in accordance with the invention; and

Figure 3 is a graph illustrating a feature of the invention.

Referring now to Figure 1, I have shown a fractionation column **10** to which a feed containing ethane and ethylene is fed through a line **11** under the control of a valve **12**.

Suitable compositions for the feed, bottoms product and overhead are as follows:

	Feed			Bottom Product			Overhead Product		
	Specific Example			Specific Example			Specific Example		
	Mols per day	Mol percent	Range	Mols per day	Mol percent	Range	Mols per day	Mol percent	Range
Methane	13	.2	0–10				13	0.1	0–10
Acetylene	211	1.3	0–3				211	1.6	0–3
Ethylene	13,096	80.0	50–90	113	3.8	1–10	12,983	97.3	70–99
Ethane	2,954	18.0	10–50	2,823	94.1	90–99	131	1.0	0.1–3.0
Propylene	70	.5	0–3	70	2.1	0–5			
	16,344	100.0		3,006	100.0		13,338	100.0	

Heat is supplied to the bottom of the column by a coil **13** through which a heating medium such as steam circulated, the steam entering by a line **14** incorporating a control valve **15**. The bottoms product is withdrawn from the column through a line **16**.

Overhead product is withdrawn from the column through a line **17** and passed through a condenser **18** to an accumulator **19**.

A portion of the overhead product from the accumulator **19** is returned as reflux to the top of the column through a line **20** under the control of a motor valve **21** while overhead product is withdrawn from the accumulator through a line **22**, incorporating a valve **23** connected to and controlled by a liquid level controller **24** having a sensing element in the accumulator **19**.

Suitable operating conditions for the column are a temperature of —650° F. at the top, a bottom temperature of —20° F. and a pressure of 140 pounds per square inch gage.

In accordance with the invention, a sample is continuously withdrawn through a sample line **25** and fed to an infrared analyzer **26**. As will be explained more fully hereinafter, the analyzer produces an output which is representative of the ethane content of the overhead product and, hence of its purity. The output can be advantageously utilized and control of the process by connecting it so as to control a process variable affecting the purity of the overhead product. For example, the output of the analyzer can be utilized to control the reflux rate by connecting it to valve **21**, the feed rate by connecting it to valve **12** or the heat supplied to the bottom of the column by connecting it to valve **15**.

The construction of the analyzer is shown in more detail by Figure 2 wherein it will be noted that infrared radiation from a source **30** is reflected by mirrors **31** and **32** so as to form two radiation beams directed upon detectors **33** and **34**, respectively, which can be bolometers.

One radiation beam passes through a filter **35** and a sample cell **36** while the other beam passes through a filter **37** and the sample cell **36**, these cells being provided with suitable windows transparent to the infrared radiation.

The detectors **33, 34** are connected in a bridge circuit of a bridge and amplifier unit **38**, preferably but not necessarily of the type shown by Hutchins Patent 2,579,825, and the instrument can incorporate automatic standardization as shown in said Hutchins patent. By the operation of the bridge and amplifier circuits, the shaft of a motor **39** is caused to assume a position representative of the relative quantities of radiation incident upon the detectors **33** and **34**.

In accordance with the invention, the filter **35** is constructed from a material which has a strong absorption band at wavelengths of 3.4 to 3.5 microns which is a region where ethane absorbs radiation strongly. Advantageously, the filter should transmit less than 10% of the radiation in this wavelength band, preferably 5% or less, and should be substantially transparent at adjoining wavelengths. To this end, the filter cell **35** can be filled with ethane which has the absorption characteristics indicated by the dashed line **40** of Figure 3. Also shown on the graph of Figure 3 by a solid line **41** are the absorption characteristics of ethylene. The filter cell **37** can contain air or other transparent material.

Accordingly, when a sample of the overhead product in column **10** is fed through the sample cell, the intensity of both beams is affected by changes in the ethylene concentration whereas changes in ethane content produce no effect on the upper radiation beam, although they do change the intensity of the lower radiation beam. As a result, the position of motor **39** is representative of the ethane content of the overhead product. Suprisingly, when the overhead product is controlled so as to maintain a predetermined ethane concentration, an overhead product of consistent ethylene concentration and purity is readily produced, and a much more uniform product is obtained than where the analyzer is sensitized for ethylene.

Where the sensitizing cell **35** is ½ to 1" long, and the sample cell **36** is 4" long, a full-scale reading on a conventional recorder chart can be readily obtained covering a percentage range of 0.0 to 1.5% ethane, thus giving a very sensitive control of the ethane concentration in the overhead product. Manifestly, it would be very difficult to obtain a full-scale span over a range of ethylene content of 97 to 99%.

It is a further feature of the analyzer and its accompanying control system that it is substantially insensitive to the presence of methane in the overhead product. Thus, the effect produced by methane upon the instrument reading is only 1/30 of the change produced by ethane so that a variation of 2 or 3% in the methane content does not materially change the purity of the overhead product.

While the invention has been described in connection with a present, preferred embodiment, thereof, it is to be understood that this description is illustrative only and is not intended to limit the invention.

I claim:

1. A method of controlling a fractionation process wherein a stream containing ethane and ethylene is fractionated to produce an overhead product containing 0.1 to 3.0 mol percent ethane, 0.0 to 5.0 mol percent methane and the balance substantially all ethylene, wherein a process variable is adjustable to control the purity of said overhead product, the steps which comprise continuously withdrawing a sample from the fractionation zone, passing twin beams of infrared radiation continuously through the withdrawn sample, passing one of said beams but not the other of said beams through a body of ethane, producing an output voltage representative of the relative intensities of said beams after they have passed through said sample and said ethane, and controlling said process variable in accordance with said voltage to maintain a constant purity of said overhead stream.

2. The process of claim 1 wherein said process variable is the rate of flow of reflux of material to the fractionation zone.

3. The process of claim 1 wherein said process variable is the heat supplied to the bottom of the fractionation zone.

4. The process of claim 1 wherein said process variable is the feed rate to the fractionation zone.

5. A method of controlling a fractionation process wherein a stream containing ethane and ethylene is fractionated to produce an overhead product containing 0.1 to 3.0 mol percent ethane, 0.0 to 10.0 mol percent methane and the balance substantially all ethylene, wherein a process variable is adjustable to control the purity of said overhead product, the steps which comprise continuously withdrawing a sample from the fractionation zone, passing twin beams of infrared radiation continuously through the withdrawn sample, passing one of said beams but not the other of said beams through a body of ethane, producing an output representative of the relative intensities of said beams after they have passed through said sample and said ethane, and controlling said process variable in accordance with said output to maintain a constant purity of said overhead stream.

References Cited in the file of this patent

UNITED STATES PATENTS

2,579,825	Hutchins	Dec. 25, 1951
2,600,110	Hachmuth	June 10, 1952
2,629,239	Gantt	Feb. 24, 1953
2,697,789	Skarstrom	Dec. 21, 1954

OTHER REFERENCES

Elements of Fractional Distillation (fourth edition), by Robinson and Gilliland, pages 472 to 476 relied on.

UNITED STATES PATENT OFFICE

CERTIFICATE OF CORRECTION

Patent No. 2,835,116 May 20, 1958

Elmer C. Miller

It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction and that the said Letters Patent should read as corrected below.

Column 2, line 52, for "-650° F." read -- -65° F. --; column 3, line 35, for "Suprisingly" read --Surprisingly--.

Signed and sealed this 14th day of October 1958.

(SEAL)
Attest:

KARL H. AXLINE
Attesting Officer

ROBERT C. WATSON
Commissioner of Patents