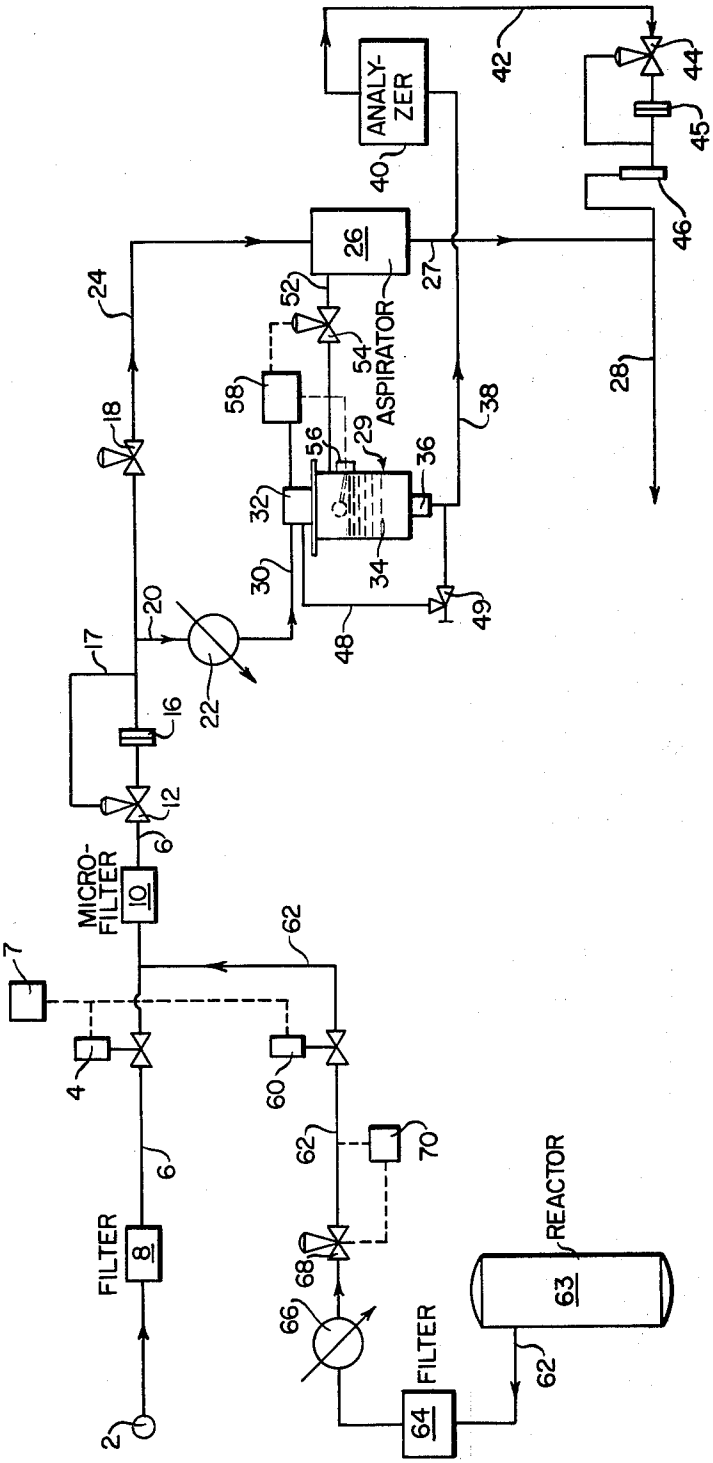
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APPARATUS FOR OBTAINING BUBBLE-FREE SAMPLE, AND METHOD FOR OBTAINING SAME

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APPARATUS FOR OBTAINING BUBBLE-FREE SAMPLE, AND METHOD FOR OBTAINING SAME

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This invention relates to the apparatus and method for obtaining a substantially bubble-free sample of liquid for analysis. In another of its more specific aspects, this invention relates to the apparatus and method for obtaining a substantially bubble-free sample of volatile hydrocarbon liquid for analysis on a spectrometer.

In the chemical and petroleum fields, for example, analysis is required of the several streams at various stages of the operation. Various automatic analyzers are commonly employed which provide accurate and rapid analysis. Suitable analyzers in wide use include, for example, infrared and ultraviolet spectrophotometers.

In petroleum refinery operations involving the conversion of hydrocarbons, effluent is analyzed to monitor the operation and to determine the quality of the product, for example. It is conventional practice to take a sample stream of hydrocarbon effluent which may be preliminarily treated, such as by filtering to remove foreign solids or by adjusting the temperature, and passing the sample of the effluent to a suitable analyzer. However, refinery operations are conducted at elevated temperatures and pressures, and gaseous products are usually admixed with the liquids. As a consequence, when a liquid sample of hydrocarbon effluent is passed to an automatic analyzer at lower pressure than the process pressure, gas in the liquid may form bubbles which interfere with the accuracy of the analysis. This is particularly true with spectrophotometers where bubbles in the liquid sample will cause an inaccurate reading.

It is therefore an object of this invention to provide a method and apparatus for obtaining a sample of liquid for analysis which is substantially bubble-free.

Other objects and advantages of the present invention will best be understood by referring to the following detailed specification and preferred embodiments thereof, and to the accompanying drawing which schematically illustrates the invention.

In general, the present invention for obtaining a substantially bubble-free liquid sample comprises first dividing a sample stream of the liquid into a major portion and a minor portion. The minor portion is admitted to a degassing zone which also is provided with an outlet for withdrawing liquid therefrom. As explained more fully hereinbelow, it is preferable that the level of liquid in the degassing zone is controlled to a predetermined depth providing a gas space in the degassing zone above the liquid. Simultaneously with admitting the minor portion to the degassing zone, the major portion is admitted to an aspirator. Conduit means establishes communication between the aspirator and the gas space of the degassing zone, and desirably a valve means, preferably power-actuated in response to the level of liquid in said degassing zone, is interposed in the conduit means for controlling flow between the aforesaid parts.

In operation, the minor portion of the liquid sample stream is passed to the degassing zone at a substantially constant rate to maintain the liquid at a substantially predetermined level. At the same time, the major portion of the liquid sample stream is passed to the aspirator drawing gases from the degassing zone. As a consequence, there is a substantial reduction in pressure in the degassing zone which causes the gases in the liquid to

escape. This reduces substantially the volume of gases in the liquid present in the degassing zone. The liquid withdrawn from the degassing zone can then be passed through the sample cell of an automatic analyzer for analysis at substantially the same pressure and temperature as that in the degassing zone without the formation of gas bubbles in the liquid sample.

In order to describe the invention in greater detail, reference is now made to the accompanying drawing which schematically illustrates the invention for use in a petroleum refinery operation. For purposes of illustration, the invention is shown for use in the catalytic reforming of petroleum naphtha to obtain gasolines and gasoline stocks with high octane ratings which are obtained primarily by reason of conversion of paraffins and naphthenic hydrocarbons to monocyclic aromatic hydrocarbons. The effluent sample stream is passed to an automatic analyzer such as a spectrometer. Particularly suitable for this purpose are an infrared or ultraviolet spectrophotometer. With these analyzers, a beam of infrared or ultraviolet radiation is passed through a liquid sample of the hydrocarbon effluent. The amount of radiation absorbed at a selected wave length indicates the amount present of the selected constituent under question. From this analysis, the quality of the effluent stream can be determined and the operating conditions can be controlled or adjusted.

In the reforming of petroleum naphtha using a platinum-alumina catalyst, for example, the total effluent from the operation is usually treated in a high pressure separator to remove most of the hydrogen and light gaseous hydrocarbons. The resulting effluent is then passed to a stabilizer to recover the normally liquid reaction products substantially free from light hydrocarbons. As explained above, analysis of the effluent is required, and at the conditions of the hydrocarbon conversion operation, gases are present in the liquid which may form bubbles at the pressure at which the analysis is carried out and therefore interfere with the analysis. Thus, referring to the drawing, hydrocarbon effluent from a high pressure separator, not shown, is delivered through line **2** for processing in a stabilizer, not illustrated in the drawing. As exemplary of a commercial reforming operation, effluent from the high pressure separator would be at a temperature of about 110 to 115° F. and a pressure of about 450 p.s.i.g. When it is desired to take a sample from line **2**, solenoid valve **4** in line **6** is opened by electric sequence control means **7**, and a sample stream of high pressure separator effluent is passed through line **6**. The stream is passed through a filter **8** to remove foreign solids, such as carbon particles, catalyst particles, etc., and through a microfilter **10** to remove fine particles of foreign solids down to about 1 micron size. Generally, it is desirable or necessary to reduce the temperature and pressure in the sample line, for in commercial operating units, the sample streams taken for analysis are likely to be at somewhat higher temperatures and pressures than the analyzer is equipped to handle or might handle under the most favorable conditions. For example, when an ultraviolet spectrophotometer is employed to analyze the sample stream delivered to the stabilizer of the reforming operation mentioned above, the part of the sample stream passed to the analyzer may be desirably at a temperature of 100° F. and a pressure of 400 p.s.i.g. Therefore, the sample stream is passed first through a differential pressure regulator **12** in line **6** which maintains the pressure immediately downstream of the valve higher than a reference pressure by a fixed amount. Suitable differential pressure regulators are known and are commercially available. A fixed metering orifice **16** is provided downstream of differential pressure regulator **12**. A sharp edge plate orifice

with the sharp edge upstream and with a No. 55 drill opening is suitable as the metering orifice. Control line **17** connected with line **6** downstream of metering orifice **16** provides a reference pressure for differential pressure regulator **12** so that a constant pressure difference is maintained across metering orifice **16** whereby a constant volume of liquid is passed through the metering orifice. Suitably the differential pressure regulator is adjusted to give a flow of 1 gallon per minute (3785 cc. per min.) through the metering orifice. A pressure regulator **18** controls the pressure in line **6** downstream of metering orifice **16** at a constant predetermined value. A minor portion of the stream flows through line **20** and then through cooler **22** to reduce the temperature of the stream. The remainder of the stream, constituting the major portion thereof, passes through pressure regulator **18** and through line **24** to aspirator **26**. The liquid and gases leaving the aspirator are cycled to a refinery stream via lines **27** and **28** for further handling.

Hydrocarbon sample enters the degassing zone (indicated generally at **29**) through line **30** into upper chamber **32**, which is of larger cross-section than line **30**. As a consequence, the flow of fluid is decelerated abruptly thereby facilitating separation of gas from liquid. The hydrocarbon liquid is collected in chamber **34** which is of relatively large volume and serves as a liquid receiver section of the degassing zone. Chamber **34** is provided with an orifice **36** at the bottom of the chamber which regulates the flow of sample to the analyzer. Suitably a No. 60 drill size orifice (0.001256 square inch opening) will provide a rate of flow of about 100 ccs. per minute. The liquid sample flows through line **38** to the spectrophotometer indicated generally at **40** for analysis. Analysis of the liquid sample may be conducted on a continuous basis, and the liquid sample is directed from the analyzer through line **42**. A second differential pressure regulator valve **44**, and metering orifice **45**, suitably with a No. 60 drill size sharp edge orifice, controls the rate of flow of the sample through the sample cell of the analyzer. The volume of sample passing through metering orifice **45** can be determined by a suitable flow meter **46**. The sample stream is discharged through line **28**, usually back into a refinery stream.

A pressure equalizing line **48** extends from line **38** to vapor space in the degassing zone to assure a smooth, uniform flow of liquid to the analyzer **40**. Valve **49** is interposed in pressure equalizing line **48** to prevent pressure surges which could be damaging to some of the more fragile parts of the analyzer.

Conduit **52**, having valve **54** interposed therein, provides fluid communication between the aspirator **26** and the gas space of the degassing zone **28**. Chamber **34** is provided with a suitable float or displacer **56**, or other liquid level responsive device which operates liquid level controller **58** which serves to actuate valve **54** controlling the rate of withdrawal of gas from the degasser and the level of the liquid in the chamber at a predetermined level.

In operation, valve **4** in line **6** is opened, and hydrocarbon sample may be passed from the high pressure separator at the rate of about 1 gallon per minute. This rate is representative only of a typical operation, and it should be understood that the rate may be suitably adjuted as determined by the various conditions of operation. The stream in line **6** is then divided by valves **18** and **44**, as explained above, and a minor portion at the rate of about 100 cc. per minute is passed to the degassing zone, the remainder of the stream being directed to the aspirator. Gas is drawn from chamber **34** allowing the chamber to fill to a predetermined level with degassed liquid sample. The liquid in chamber **34** is maintained at a predetermined level thereby providing a space in the chamber above the liquid. As explained above, the major portion of liquid is passed to the aspirator **26** via line **24**, and, with valve **54** open, suction in line **52** occurs and the gases from the degassing zone are drawn therefrom. This creates a substantial reduction in pressure in the degassing zone which causes the gases in the liquid to escape readily. The liquid withdrawn from the degassing zone via line **38** is then substantially bubble-free, and is passed to the automatic analyzer **40** for analysis.

A rise in liquid beyond the predetermined level will tend to close valve **54**, reducing the rate of gas withdrawal from chamber **34** and permitting the pressure in the space occupied by gases above the liquid to increase. As a result, the rate of flow of sample to the degassing zone from line **6** is decreased. If the liquid level in chamber **34** decreases, the float or displacer in chamber **34** actuates valve **54** to open the valve more and permit gas to flow at a faster rate through line **52**, further reducing pressure in chamber **34** and drawing in sample from line **6** at a faster rate to re-establish the liquid level in chamber **34**.

Where desired, hydrocarbon effluent may be taken for analysis from several points in the over-all hydrocarbon conversion operation. For example, a side stream taken from the reactor in a reforming operation would be typically at about 900 to 975° F. and a pressure of about 600 to 650 p.s.i.g., which is substantially greater than the temperature and pressure for a side stream from a stabilizer. When it is desired to analyze a side stream from the reactor, electric sequence control means **7** opens solenoid valve **60** in line **62** leading from the reactor indicated generally at **63**. The sequence control means **7** thus closes valve **4** in line **6** so that streams from different units are not mixed. Effluent from the reactor is filtered at **64** to remove foreign solids, and is then passed through cooler **66** and pressure regulator valve **68**, having pressure controller **70**, to reduce the temperature and pressure of the stream. The stream from line **62** is then conducted to line **6** for further processing in accordance with the invention as described above.

Having described my invention, and certain embodiments thereof, I claim:

1. Apparatus for obtaining a substantially bubble-free liquid sample from a liquid process stream comprising in combination,

means including a first conduit for withdrawing a sample from a liquid process stream,

aspirating means having a first inlet means for admitting a first portion of said liquid process stream to create suction therein, a second inlet means for aspirating gas thereto, and an outlet means for discharging a mixture of liquid and gas therefrom;

degassing means for degassing a second portion of said process stream, said degassing means having a liquid inlet means, a degassing zone, liquid outlet means for withdrawing therefrom a liquid sample substantially free of gas, and gas outlet means;

a second conduit connecting said first conduit to said aspirating means for supplying aspirating liquid thereto;

a third conduit connecting said first conduit to said liquid inlet means of said degassing means for supplying a liquid sample thereto;

valve means controlling the volume of liquid entering said second and third conduits;

a fourth conduit connecting together said second inlet of said aspirating device and said gas outlet from said degassing zone;

and means for combining the liquid outlet means of said aspirating means with the liquid outlet means of said degassing means.

2. Apparatus in accordance with claim **1**, also comprising means to control the level of liquid in said degassing zone.

3. Apparatus in accordance with claim **1**, also comprising

means responsive to the level of liquid in said degassing zone to control the level of said liquid therein to a predetermined depth, and

valve means interposed in said fourth conduit means and operated by said level responsive means, to control flow of said gas from said gas outlet of said degassing means to said gas inlet of said aspirating means, and

means for analyzing said liquid substantially free of gas.

4. A method for obtaining a substantially bubble-free liquid sample, which comprises:

separating a stream of a liquid containing gaseous components into a major portion and a minor portion,

introducing said minor portion into a degassing zone,

separating gaseous components from said minor portion by vaporization of said gaseous components in said degassing zone,

simultaneously introducing said major portion into an aspirator,

applying suction created in said aspirator to said degassing zone and removing gaseous components therefrom,

discharging said major portion including the removed gaseous components from said aspirator, and

withdrawing liquid substantially free of gas from said degassing zone.

5. A method for obtaining a substantially bubble-free liquid sample, which comprises:

separating a stream of a liquid containing gaseous components into a major portion and a minor portion,

introducing said minor portion into a degassing zone,

separating gaseous components from said minor portion by vaporization of said gaseous components in said degassing zone,

controlling the level of said liquid in said degasssing zone at a point below the top of said degassing zone,

simultaneously introducing said major portion into an aspirator,

applying suction created in said aspirator to the upper portion of said degassing zone and removing gaseous components therefrom,

discharging said major portion including the removed gaseous components from said aspirator, and

withdrawing liquid substantially free of gas from said degassing zone.

6. Apparatus for analyzing a liquid process stream which comprises, in combination,

means for withdrawing a sample stream from said process stream,

means for removing solid impurities from said sample stream,

means for metering the rate of flow of said sample stream,

means for dividing said metered sample stream into a major portion and a minor portion,

exchanger means for controlling the temperature of said minor portion,

degassing means for separating gas from said minor portion, including inlet means connected to said exchanger means for introducing said minor portion into the upper portion of said degassing means, means for collecting said separated gas in the upper portion of said degassing means, gas outlet means connected to said gas collecting means, means for collecting substantially bubble-free liquid in the lower portion of said degassing means, liquid outlet means connected to said liquid collecting means, and pressure equalizing means connecting said liquid outlet means with said upper portion of said degassing means,

liquid level control means within said degassing means for controlling the level of said liquid therein to a predetermined depth,

flow control means located in said gas outlet means of said gas degassing means and actuated by said liquid control means,

aspirating means having a first inlet for admitting said major portion of said process stream, a second inlet connecting with said gas space in said degassing zone for admitting gas thereto, and an outlet for discharging liquid and gas therefrom,

analytical means connecting with said liquid outlet means of said degassing means,

valve means interposed between said liquid outlet means and said analytical means for controlling the rate of flow of bubble-free liquid to said analytical means, and

means for returning analyzed liquid from said analytical means and liquid and gas discharged from said outlet of said aspirating means to said liquid process stream.

7. Apparatus for obtaining a substantially bubble-free liquid sample from a liquid process stream comprising in combination,

means including a first conduit for withdrawing a sample from a liquid process stream and for introducing said sample of said process stream into a degassing means, said degassing means having a liquid inlet connected to said first conduit, a degassing zone, liquid outlet means for withdrawing therefrom a liquid sample substantially free of gas, and a gas outlet means;

aspirating means having a first inlet means for admitting a first portion of said liquid process stream to create suction therein, a second inlet means for aspirating gas thereto, and an outlet means for discharging a mixture of liquid and gas therefrom;

a second conduit connecting said first conduit to the liquid inlet of said aspirating device for supplying aspirating liquid thereto;

a third conduit connecting said first conduit to the liquid inlet of said degassing means for supplying a liquid sample thereto;

valve means controlling the volume of liquid entering said second and third conduits,

a fourth conduit connecting together said second inlet of said aspirating device and said gas outlet from said degassing zone,

said degassing means including a constricted zone of relatively smaller cross-section than said degassing zone, and pressure equalizing means connecting said liquid inlet means and said liquid outlet means;

means responsive to the level of liquid in said degassing zone to control the level of said liquid therein to a predetermined depth,

flow control valve means interposed in said fourth conduit,

means operated by said liquid level responsive means, for opening said flow control valve means when said liquid level is below said predetermined depth, and for closing said flow control valve means when said liquid level exceeds said predetermined depth, and

means for returning said gas-free liquid from said degassing means and said liquid and gas discharged from said aspirating means to said liquid process stream.

8. Apparatus in accordance with claim 7, including,

analyzing means connected to said liquid outlet from said degassing zone for analyzing said liquid substantially free of gas, and

said means for returning said gas-free liquid from said degassing means, and said liquid and gas discharged from said aspirating means, to said liquid process stream connected to said analyzing means.

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