May 19, 1970 A. J. BRANDEL 3,513,220

SPECIFIC GRAVITY ANALYZER FOR CONTROL OF ALKYLATION PROCESS

Filed Jan. 19, 1968 2 Sheets-Sheet 1

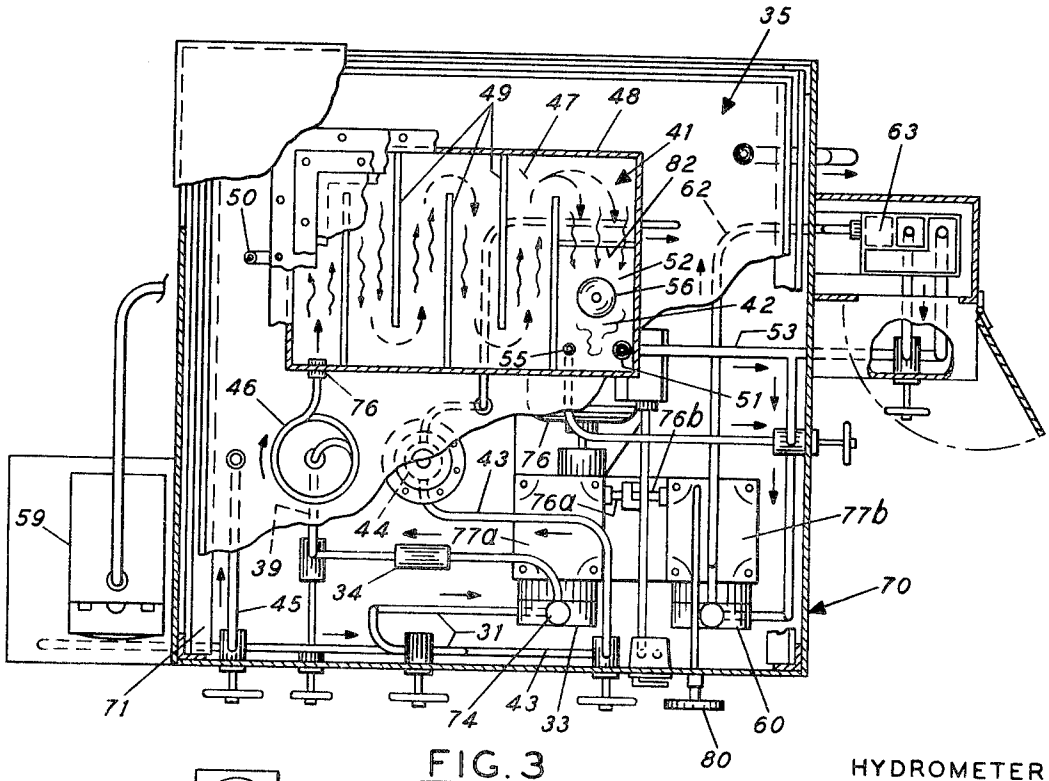


FIG. 3

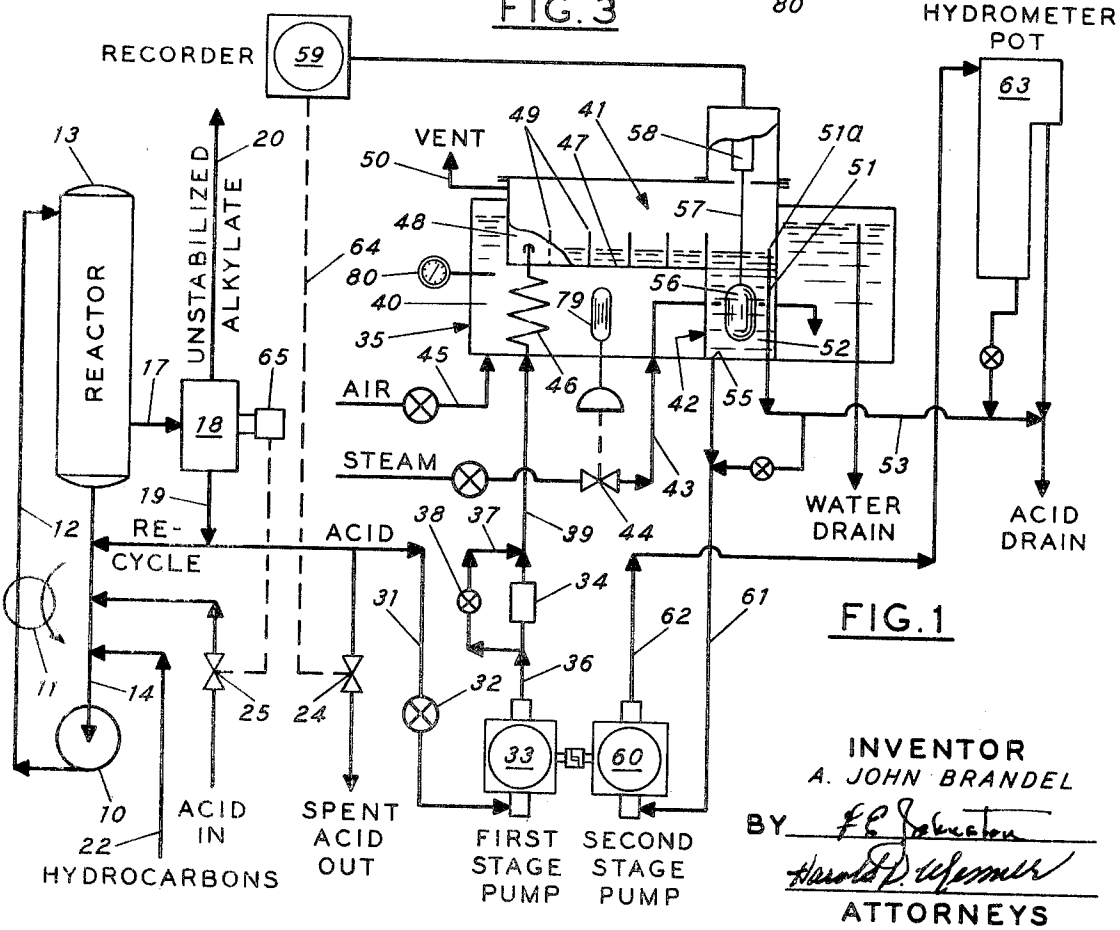


FIG. 1

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SPECIFIC GRAVITY ANALYZER FOR CONTROL OF ALKYLATION PROCESS

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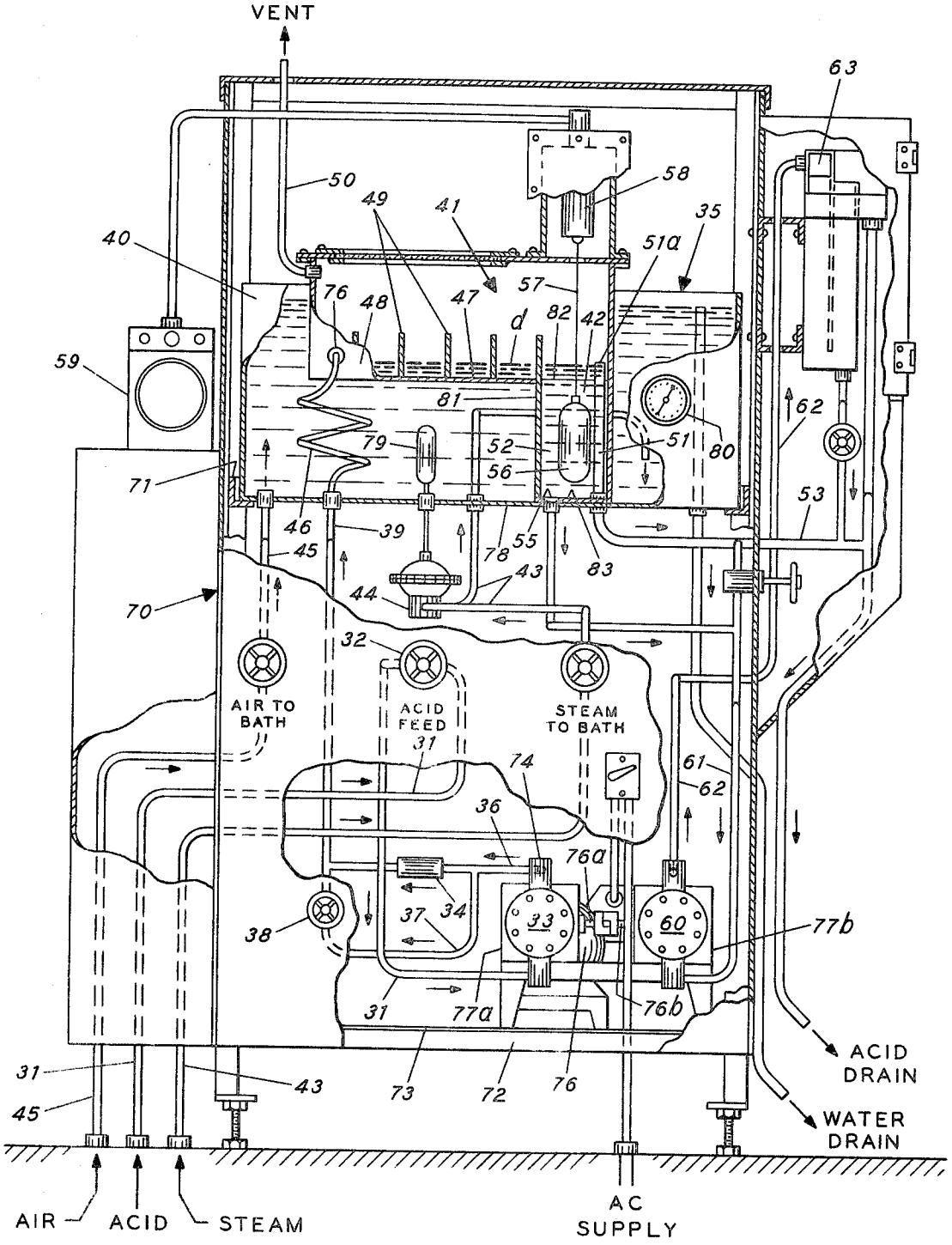


FIG. 2

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SPECIFIC GRAVITY ANALYZER FOR CONTROL OF AN ALKYLATION PROCESS

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U.S. Cl. 260—683.59 3 Claims

ABSTRACT OF THE DISCLOSURE

The acid strength of a process stream associated with an alkylation process using a mineral acid catalyst, such as sulfuric acid, can be conveniently and continuously measured by determining the specific gravity of a sample of acid taken from the main process stream. The sample is pumped under positive and fixed flow rates into and from an analysis chamber by means of first and second pumping means, connected, respectively, to the inlet and outlet of the chamber. Prior to determining the gravity of the acid within the chamber, dissolved volatile hydrocarbons and high molecular weight, normally liquid, hydrocarbons are removed from the sample by stripping and settling chambers within the analysis chamber, the high molecular weight hydrocarbons flowing from the chamber by means of an overflow tube positioned in the settling chamber. The weight percent of the carbonaceous matter removed from the sample including the high molecular weight hydrocarbons is controlled by maintaining said second pumping means at a pumping rate less than that of said first pumping means.

The present invention is concerned with a method and apparatus for the determination of the concentration of solutions. In particular, the present invention is directed to the determination and control of acid strength in chemical processes dependent on acid catalysis by means of measuring the specific gravity of the solution. More particularly, the invention is directed to the determination and control of acid strength in a sulfuric acid catalyzed alkylation reaction in which isoparaffin hydrocarbons and olefin hydrocarbons are reacted in contact with a sulfuric acid catalyst. Still more particularly, this invention relates to a method and means for measuring the concentration of spent sulfuric acid by continuously measuring the specific gravity of a sample of such acid.

The present invention may be briefly described as a method for maintaining catalyst activity in the aforementioned sulfuric acid catalyzed alkylation reaction in which the sulfuric acid gradually loses its strength for the alkylation reaction thereby requiring the withdrawal of a portion of the sulfuric acid in the system and the replacement of the withdrawn portion with sulfuric acid of a predetermined alkylation strength.

The method of alkylating isoparaffins and olefins in the presence of a concentrated sulfuric acid catalyst is well known in the petroleum refining art. Typically, in this process, isobutane and butylenes are alkylated using a concentrated sulfuric acid catalyst which may range upward in strength from about 85 weight percent H_2SO_4. The acid strength tends to decrease during the operation of the process due to dilution with carbonaceous matter such as sulfonated hydrocarbons, i.e., catalyst-hydrocarbon complexes and esters, and with water. As the acid is diluted, its acivity decreases. In the typical instance, the alkylation reaction is carried out in a series of mixing zones without the addition of acid to the materials passed through the zones. For satisfactory operation the acid strength in the last reactor must be maintained above a

certain minimum concentration. When the acid strength drops below an established minimum, the acid is discarded. It is usual to recycle a portion of the spent alkylation acid inasmuch as is possible to reduce the operating costs of the process, since the cost of acid used is important. Thus, it is desirable to minimize the amount of spent acid discarded because of the drop in acid strength. However, the determination of the acid strength is time consuming and expensive when carried out in the laboratory, and laboratory methods do not provide sufficient rapidity for close operational control of the process.

Since almost all commercial processes which utilize an acid catalyst involve continuous recycling of the acid catalyst through the reaction, it is of great importance to be able to continuously and accurately determine the acid strength. When the acid strength falls below the minimum amount necessary, additional fresh acid can be added. However, if the analytical technique is not accurate, then either too much acid may be added, resulting in a wasting of this expensive material, or else too little may be added, resulting in poor product yields.

Heretofore, the acidity of the alkylation acid could be determined spectrophotometrically, but this technique suffers from several disadvantages:

(1) It requires the use of relatively expensive spectrophotometers;

(2) It requires a continuous addition of an indicator compound such as alizarin blue in known amounts to the sample. Accordingly, a complicated and expensive control and metering system for the indicator compound is needed.

In a further prior art method, the acidity function of an alkylation acid could be determined by continuously measuring the specific gravity of a sample of acid by means of a hydrometer after removal of carbonaceous matter from the sample. Experience has shown that in flowing the sample to the hydrometer clogging often occurs due to the deposit of carbonaceous matter in pressure-reducing elements of the system such as orifice restrictors.

It is therefore an object of the present invention to provide a useful and novel method and apparatus for the accurate and continuous determination of the specific gravity of reaction process streams utilizing only relatively simple and inexpensive instruments which will not require the addition of indicators or other reagents and which do not clog even though the sample contains carbonaceous matter. It is a further object to provide such a method and apparatus which can be adapted to provide automatic control over a continuous acid catalyzed chemical process so as to maintain maximum product yields with minimal acid losses.

It has been found that the specific gravity and especially changes in the specific gravity of a recycle process acid stream associated with an alkylation process using a mineral acid catalyst, such as sulfuric acid, can be conveniently and continuously measured to determine the concentration of the aforementioned recycle stream. A sample of the stream is pumped under positive and fixed flow rates into and from an analysis chamber by means of first and second pumping means, connected, respectively, to the inlet and outlet of the chamber. Prior to determining the gravity of the acid within the chamber, dissolved volatile hydrocarbons and high molecular weight, normally liquid hydrocarbons are removed from the sample by stripping and settling chambers within the analysis chamber, the high molecular weight hydrocarbons flowing from the chamber by means of an overflow tube positioned in the settling chamber. The weight percent of the carbonaceous matter removed from the sample including the high molecular weight hydrocarbons is con-

trolled by maintaining said second pumping means at a pumping rate less than that of said first pumping means.

In a specific embodiment of the invention, a continuous sample of the recycle process stream is pumped, by means of an input acid pump, to the stripping chamber. To maintain a pumping head for the pump, a pressure-sensitive relief valve may be fitted between the discharge side of the pump and the stripping chamber operated to open on the discharge stroke of the pump when a working pressure above the process pressure is achieved. The pump and sequentially operated valve provide a constant rate of flow to the separation chamber without the use of restrictors. In the stripping chamber the sample is heated to the desired temperature say, 105° F., by means of a temperature-controlled hot water bath. While within the bath, the sample flows through the stripping chamber which preferably includes a substantially horizontal extending planar base adapted to convey the input sample in open channel heat transfer contact with the bath so as to strip the volatile hydrocarbons from the sample. The volatile hydrocarbons are vented to the atmosphere from the top of the stripping chamber as vapors. Baffles on the planar base of the stripping chamber provide a sinuous flow pattern to the sample. Within the settling chamber located at one end of the base in fluid communication with the stripping chamber, the high molecular weight hydrocarbons including the alkylate are skimmed off by an overflow tube. An outlet near the bottom of the settling chamber is connected to the second fluid pumping means for removing the stripped acid sample from the chamber after the sample has passed through both the stripping and the settling chambers. Preferably, the overflow outlet tube within the stripper chamber is adapted to be positioned above the plane of the base of the stripping chamber, the amount of the projection thereby regulating the thickness of the sample stream within the stripping chamber to a predetermined relatively small magnitude whereby uniform heat contact between the stripping and the heating chamber readily occurs. Prior to removal of the stripped acid from the settling chamber, the specific gravity of the acid is determined by means of a displacemeter of constant volume in which the buoyant force is measured by means of a strain gage-type transducer so as to indicate the specific gravity of the acid sample.

By suitably controlling the pumping rates of the input and output pumping means, a ratio of fluids removed by the overflow tube and by the second fluid pumping means can be controlled which assures that substantially all volatile and high molecular weight hydrocarbons of the sample are removed prior to the gravity determinations.

If desired, the process from which the sample is taken may be controlled by utilizing the specific gravity determinations to control valves which withdraw spent acid from the system and admit new acid of correct strength to the system to maintain the strength of the acid at a proper setpoint level. When the acid strength and therefore the specific gravity falls below a preselected level, the control equipment connected to the valves may be automatically actuated in response to the deviation of the measured specific gravity until the optimum level within the system is again reached. It is thus possible to utilize such a system to maintain the acid strength equilibrating about the optimum level thereby achieving a high process efficiency.

The invention may be used to control the catalyst activity of a single or a plurality of alkylation reactors using common isoparaffin and olefin reactants in the presence of a mineral acid catalyst such as sulfuric acid, hydrofluoric acid, etc.

The present invention will be further illustrated by reference to the drawings in which:

FIG. 1 is an illustrative flow diagram of a typical hydrocarbon sulfuric acid alkylation process in which the system of the present invention can be utilized;

FIG. 2 is an elevational view partially cut away of the apparatus of FIG. 1 for processing a sample of the recycle stream in accordance with the present invention to determine the specific gravity of the sample;

FIG. 3 is a plan view, partially cut away, of the apparatus of FIG. 2 illustrating in detail the flow of a sample within the stripping and settling chambers of the apparatus of FIG. 2.

Referring to FIG. 1, there is illustrated diagrammatically a specific form of alkylation process conventionally used in the hydrofining art for the purpose of illustrating the use of the present invention. In operation, a large stream of hydrocarbons undergoes alkylation in the presence of an acid catalyst such as sulfuric acid. The hydrocarbons and catalysts are continuously circulated by pump **10**, through cooler **11**, system line **12**, reactor **13** and return line **14**. Thorough mixing of the hydrocarbons with the acid is maintained by the mixing action of the pump and also by baffling (not shown) within the reactor. A side stream of the mixture is continuously withdrawn from a convenient point of the system, say through line **17**, to settler **18** where the acid is permitted to settle from the alkylated and nonreacted hydrocarbons. The settled acid is returned to the system through line **19** and the hydrocarbons are withdrawn through line **20** for neutralization and fractionation into alkylate products as well as recycle products. Makeup hydrocarbons including the recycle products from fractionation are introduced to the system at a convenient point, as near the intake of pump **10**, as illustrated by line **22**.

The strength of the acid circulated in the system gradually decreases until a point is reached where its strength is uneconomically low for the production of good quality alkylate. If not corrected, the acid strength may become so low that various undesirable side reactions occur. To maintain the acid strength at an economic optimum, spent acid is withdrawn from time to time through valve **24** and strong makeup acid at the alkylation concentration is introduced through valve **25**. If the strength of the acid within the reactor is not accurately and continuously determinable, determinations as to the amount of spent acid to be withdrawn and the amount of alkylation acid to be added to the system cannot be made. In withdrawing a sample of the acid from the system for such determination, several factors must be considered including reducing, to a minimum, the time lag between withdrawal of the sample of the acid and the determination of the strength of the acid. Further difficulties can occur due to the pressure and temperature prevailing within the system. For example, upon release of pressure, a sample withdrawn immediately becomes foamy and must be allowed to settle. Further, volatile and high molecular hydrocarbons must be removed from the sample if the gravity determinations are to have any reproducible significance. Still further diffictulties occur in the use of orifice restrictors in withdrawing a sample of the acid from the system. Not only is the acid difficult to handle, the carbonaceous matter within the withdrawn sample frequently clogs the restrictors requiring the installation of an attendant control system for cycling steam through the sampling system.

In accordance with the present invention, the spent alkylation acid is sampled by means of sampling line **31** and flows at a constant rate through valve **32** to first stage acid pump **33** and pressure-sensitive relief valve **34**. The acid pump **33** discharges at a substantially constant flow rate through the valve **34**; typically the flow rate is low so as to be compatible with the size of the sampling system, say about 1.5 gallons per hour.

The function of the pump **33** and relief valve **34** is to dissipate system pressure in passing the sample from process line **19** where, say, a pressure of 120 p.s.i.g. exists, to analysis chamber **35** where, say, a pressure of about zero p.s.i.g. exists while simultaneously maintaining the sample at a substantially constant flow rate. Relief valve **34** is shown connected to the discharge side of pump **33** and is adapted to be biased to a full open position when a preselected discharge pressure above the system pressure

occurs in line 36. The full open position of the valve is adapted to be large enough so that carbonaceous matter in the sample does not accumulate at the valve seat. The discharge pressure of the acid pump 33 periodically rises above the setpoint level of the valve 34 to allow flow of the sample through the valve 34. The bypass line 37 which includes valve 38 is placed in parallel with the relief valve 34. Valve 38 is normally closed but can be open to facilitate additional flow from the acid pump 33.

As an alternative modification, the system pressure can also be dissipated at the suction side of acid pump 33. For example, a pressure-sensitive valve may be positioned on the suction side of the acid pump 33 and be adapted to incrementally open to maintain a given pressure drop between the valve and a pressure-sensing means on the discharge side of the pump. Accordingly, as carbonaceous matter accumulates within the valve, the valve plug is incrementally released to thereby maintain a given flow rate through the acid pump.

The discharge of pump 33 through valve 34 is then passed by way of line 39 to the inlet of analysis chamber 35. As shown, chamber 35 is positioned within a constant temperature bath 40 and includes stripping chamber 41 and settling chamber 42. Bath 40 is employed to maintain the sample within the chamber 35 at a substantially constant temperature, say about 105° F. For this purpose, steam is passed into the bath by way of line 43 through temperature responsive valve 44. The bath is also preferably agitated by air introduced to the chamber 35 through line 45. The sample is first preheated by exchanger 46 in contact with bath 40 and is then passed into stripping chamber 41.

Stripping chamber 41 includes a planar base 47, upright walls 48 attached to the base 47 and baffle plates 49. As the sample circulates through the stripping chamber, the volatile hydrocarbons are heated to a gaseous phase. The vapors exit through vent tube 50. Discharge of the stripping chamber is by way of the settling chamber 42. Settling chamber 42 is provided with an overflow tube 51 and a receiving cavity 52. A shown, cavity 52 is positioned below the skimming level established by inlet 51A of the overflow tube 51. Within the settling chamber 42, the high molecular weight hydrocarbons in the sample are skimmed off by the overflow tube 51 and are passed from the settling chamber by way of line 53. The stripped sample passes through the cavity 52 toward outlet 55 near the floor of the settling chamber. Within cavity 52 is the displacemeter 56 weighted, i.e. with mercury, to a higher density than the maximum sample density to be measured, and connected by a wire line 57 to a buoyancy force reading transducer 58. Transducer 58 converts the magnitude of the buoyancy force acting on the displacemeter 56 to an electrical signal indicating a specific gravity of the stripped sample. The gravity-indicting signal is then recorded by recorder 59 to provide a record indicating the concentration of the alkylation acid.

Second stage acid pump 60 attaches to outlet 55 of cavity 52 for pumping the stripepd sample from the settling chamber via line 61. The discharge side of the acid pump 60 attaches via line 62 to a hydrometer pot 63. Specific gravity determinations of the stripped sample are made by a hydrometer in hydrometer pot 63 from time to time to calibrate transducer 58. Thus by monitoring the record of recorder 59, the operator may be readily advised of the strength of the acid in the alkylation system. Desired corrections in the alkylation process can be made manually or automatically by adding alkylation acid and withdrawing spent acid from the process.

To automatically control the addition of alkylation acid, recorder 59 may be provided with mechanical linkages 64 which control spent acid valve 24. As deviations from a setpoint level within the recorder-controller occur, valve 24 is adjusted to withdraw spent acid from the process. To control the admission of strong alkylation acid, to compensate for the removal of the spent acid, settler 18 may be provided with a level-indicating controller 65 for operating valve 25. Accordingly, as the acid hydrocarbon interface level within settler 18 deviates from the preselected setpoint level, the valve 25 is actuated to admit alkylation acid to the process. When an optimum concentration of alkylation acid is achieved within the alkylation process, valves 24 and 25 are biased to a closed position. Alternatively, the recorder-controller which includes recorder 59 and linkages 64 may operate spent acid valve 25 instead of valve 24. Accordingly, level-indicating controller 65 of settler 18 operates spent acid valve 24.

FIGS. 2 and 3 illustrate the use of the gravity-indicating apparatus of the present invention in marked detail. In FIG. 2, a frame 70 is seen to be fitted with lateral channel bars 71 at the upper end of the frame to which analysis chamber 35 is attached. At the lower periphery, channel bars 72 and support base 73 provide support for acid pumps 33 and 60. Hydrometer pot 63 is positioned at one side of the frame 70 while recorder 59 is positioned at an opposite side, as shown, or remotely, as in a control room.

In flowing an acid sample under pressure to the apparatus of the present invention, the first stage acid pump 33 periodically creates a discharge pressure at discharge side 74 above the setpoint bias of relief valve 34 as previously mentioned. Accordingly, acid pump 33 is preferably of the piston or diaphragm type and may be operated as shown by a motor-driven system also used to drive second stage acid pump 60. In such a drive system, a single motor 76 drives both pumps 33 and 60 through shafts 76A and 76B to separately driven gear boxes 77A and 77B within each pump. Duplex version, Model No. SL–3, Lapp Insulator Company, Inc., Le Roy, N.Y., has been found to be a satisfactory, commercially available, tandem pump.

Bath 40 of analysis chamber 35 is enclosed within a support housing 78. Water comprising the bath is placed in heat transfer contact with the pre-heat exchanger 46 as well as stripping chamber 41 and settling chamber 42. The temperature of the bath is controlled by steam entering the analysis chamber through the inlet line 43. The flow rate of the steam is controlled by temperature-sensitive diaphragm valve 44 controlled by temperature-measuring element 79. An exterior indicating dial 80 provides visual indication of the temperature of the bath.

The temperature within the analysis chamber 35 must be maintained at a uniform temperature during operations. If the temperature remains constant, substantially the same amount of hydrocarbons are removed from the acid sample within the analysis chamber. A single bath 40 for both heating the sample to remove volatile hydrocarbons as well as maintaining the settling chamber at a constant temperature as a specific gravity characteristic of the acid is measured, has been found to be an important advantage of the present invention.

However, the temperature selected must be high enough to separate substantially all of the volatile hydrocarbons within the stripping chamber without too much troublesome foam. It should not be so high, however, as to substantially decompose the high molecular weight hydrocarbons, normally in the liquid phase, removed by skimming within the settling chamber. A selected temperature of about 105° F. has proven to be satisfactory although a range somewhere between 90–150° F. should prove to be satisfactory in the average installation.

As shown best in FIG. 2, the stripping chamber 41 is positioned at an elevated location above the floor of the bath housing 78. It is attached in the elevated position by welding its planar base 47 and sidewalls 48 to adjacent portions of the bath housing 78 in the settling chamber 42, as shown. When the level of the heater within bath housing 78 is adjusted to a level so that contact is made both with the side walls 48 and the base 47 of the stripping chamber, as shown, heating of the sample

occurs at the sides as well as the base of the stripping chamber.

FIG. 3 illustrates the flow patern of the acid sample within the stripping chamber **41** in more detail. As shown, the stripping chamber **41** is provided with a series of upright plates **49** attached to base **47** and the side walls **48**. After the acid sample has passed into the stripping chamber by way of inlet **76** and helically wound preheating tube **46**, the plates **49** direct the sample in the sinuous flow pattern as shown. The incremental time period that the sample contacts bath **40** is thus increased. Accordingly, substantially all of the volatile hydrocarbons are stripped from the sample. The vapors vent to atmosphere by way of vent tube **50**.

As shown in FIG. 2, settling chamber **42** includes side walls **81** attached to the bath housing **78** in fluid communication with the stripping chamber **48** through aperture **82** in the base **47**. The planar base **83** is attached to the floor of the bath housing. Base **83** is provided with openings by which overflow tube **51** and outlet **55** are rigidly positioned so as to contact the fluid within the settling chamber **42**. After the sample has entered the settling chamber, the high molecular weight hydrocarbons are stripped from the sample at a skimming level established at the inlet **51A** of the overflow tube **51**. The skimmed hydrocarbons are disposed to drain via line **53** attached to the overflow tube **51**. The stripped acid sample is then passed through cavity **52** below the skimming level established by the overflow tube and then is conveyed from the settling chamber by way of outlet **55** to the second stage pump **60**.

In addition to establishing a skimming level for removing the high molecular weight hydrocarbons, the overflow tube **51** also performs another important function in the operation of the gravity-indicating aparatus of the present invention. As shown in FIG. 2, the skimming level established by overflow tube **51** only slightly exceeds the plane of base **47** of the stripping chamber **41**, say by the incremental distance "*d*," as shown. The depth of the sample within the stripping chamber is, of course, equal to the incremental distance "*d*" since the pressure in the stripping and settling chambers is the same. Accordingly, the depth of the sample can be easily reduced to a relatively small magnitude, say ⅛"–½" without introducing inaccuracies in the gravity measurements later performed in the settling chamber. As the removal of the hydrocarbons from the acid sample is enhanced by the apparatus of the present invention, measurements of the specific gravity characteristic of the stripped sample within cavity **52** of the settling chamber **42** result in a true and easily reproducible indication of the specific gravity of the acid.

The pumping rates of the acid pumps **33** and **60** closely control the gravity-indicating apparatus of the present invention so that the pumping rate of the input, or first stage, acid pump **33** is always greater than that of the output, or second stage, acid pump **60**. Accordingly, not only is the original acid sample pumped under positive flow conditions into the analysis chamber **35** by means of acid pump **33**, thereby reducing clogging in the measuring system to a minimum, but the stripped sample is also seen to be pumped under positive flow conditions from the settling chamber **42** by means of second stage acid pump **60**.

Furthermore, it can be seen that the differential pumping rate established by the first stage and second stage pumps can easily be varied to values that assure that all of the hydrocarbons are removed from the acid sample prior to measuring the specific gravity characteristic of the stripped sample within cavity **52** of the settling chamber **42**. The skimming level of the fluid within the analysis chamber is preferably constructed to remain fixed during operations. Accordingly, the percentage by weight of the hydrocarbons removed from the acid sample is a direct function of the differential pumping rates of the first stage and second stage acid pumps. Stated mathematically, for the minimum case:

$$\text{Weight percentage hydrocarbons } (W_R) = PR/R_{Input}$$

where:

PR is the differential flow rate of the first stage and second stage acid pumps, and

R_{Input} is the flow rate of the first stage acid pump.

In actual practice, the pumping differential is always adjusted so that the ratio defined above is always greater than the expected weight percentage of hydrocarbons within the sample to be removed. For example, where the acid to be monitored is thought to contain about 5 percent by weight volatile and high molecular weight hydrocarbons, the percentage of removal established by the acid pumps is set at a much higher level, say 30 percent. Accordingly, 25 percent of the stripped acid would also be removed via overflow tube **51**.

To establish optimum pumping rates for the acid pumps **33** and **60**, several operating parameters of the gravity-indicating apparatus of the present invention must be taken into consideration, among which are: the capacity of the gravity-indicating apparatus, the size of the sample stream, the maximum time lag permitted to measure the sample, as well as the weight percent of hydrocarbons within the sample. For a maximum time lag of 12 minutes, an input flow rate of 1.5 gallons per hour, a total apparatus capacity of about ½ gallon, a second stage pumping rate of about 1.0 gallons per hour provides removal of about 30 percent by weight of the input sample. Accordingly, as the weight percent of volatile and high molecular weight hydrocarbons within the sample increases, the differential flow rate of the acid pumps can be readily adjusted to facilitate their removal.

Removal of the hydrocarbons from the sample in the aforementioned manner has been found to provide stripped samples that are not only substantially free of hydrocarbons, but the hydrocarbons that remain, if any, are substantially constant. Accordingly, the gravity-indicating apparatus of the present invention has been found to provide results that can be repeated within very small tolerances.

The invention has been described above in conjunction with a specific type of alkylation process for which it was particularly designed. However, it is useful in conjunction with other forms of sulfuric acid alkylation process and also with other processes wherein strong sulfuric acid is contacted under pressure with normally gaseous hydrocarbons. In such processes it is generally desirable to withdraw a sample of the acid from time to time for control purposes. The invention provides a ready means of of obtaining such sample without the customary difficulties arising from the sudden volatilization of the hydrocarbons in the withdrawn sample.

I claim:

1. In an alkylation process including a hydrocarbon-sulfuric acid contacting vessel, an acid-hydrocarbon settling vessel and a recycle acid line for returning settled acid to said contacting vessel, control means including an inlet valve for introducing sulfuric acid into said process, means including an outlet valve for withdrawing spent sulfuric acid from said recycle line and a sampling line in communication with said recycle line, the method of controlling the acid concentration in said alkylation process, which comprises the steps of:

(1) continuously pumping at a first pumping rate a sample of said acid through said sample line to a chamber,

(2) heating said sample within said chamber to strip volatile hydrocarbons,

(3) skimming high molecular weight hydrocarbons within said sample by establishing a predetermined skimming level within said chamber after stripping of said volatile hydrocarbons from said sample, said high molecular weight hydrocarbons above said skim-

ming level being removed from said chamber by means of an overflow tube,

(4) continuously pumping at a second pumping rate said acid sample from said chamber, the second pumping rate of said sample being less than that of said first pumping rate so as to establish a differential pumping rate therebetween, said differential pumping rate being based on the amount of said volatile and high molecular weight hydrocarbons which are removed from said sample during steps (2) and (3),

(5) measuring a specific gravity characteristic of said acid sample at a known temperature to provide a continuous indication of the concentration of said acid sample,

(6) controllably varying acid flow relative to said alkylation process through control of at least one of said inlet and outlet valves in accordance with said measured specific gravity characteristic of said sample to thereby return said acid to a predetermined setpoint level.

2. Method in accordance with claim **1** in which the last-mentioned step of varying acid flow relative to said process includes controlling said outlet valve for withdrawing spent sulfuric acid from said recycle line in accordance with said measured specific gravity characteristic of said acid sample, and controlling said inlet valve for introducing said sulfuric acid into said process in accordance with the hydrocarbon-sulfuric acid interface level in said settling vessel so as to return said acid to a predetermined setpoint level within said process.

3. Method in accordance with claim **1** in which the last-mentioned step of varying acid flow relative to said process includes controlling said inlet valve for introducing sulfuric acid into said process in accordance with said measured specific gravity characteristic of said acid sample and controlling said outlet valve for withdrawing spent sulfuric acid from said recycle line in accordance with the hydrocarbon-sulfuric acid interface level in said settling vessel so as to return said acid to a predetermined setpoint level within said process.

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