

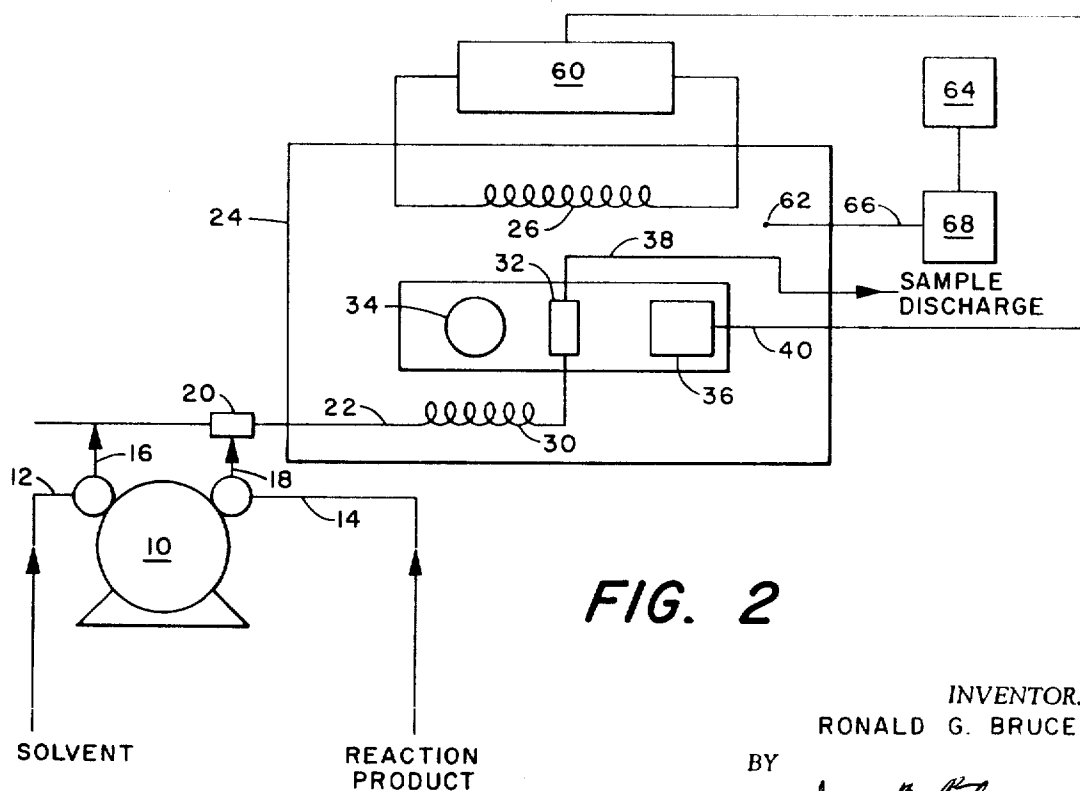
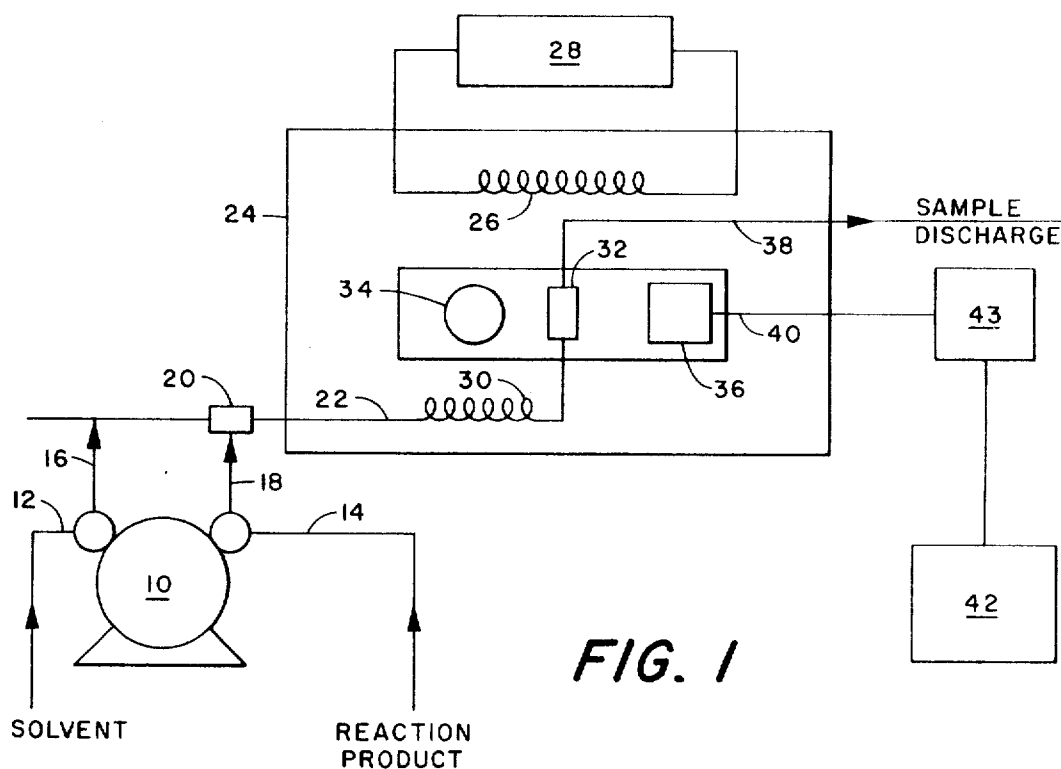
July 13, 1971

R. G. BRUCE
APPARATUS FOR CONTINUOUSLY MONITORING THE
PROCESS OF CERTAIN REACTIONS

3,592,607

Original Filed March 2, 1966

3 Sheets-Sheet 1



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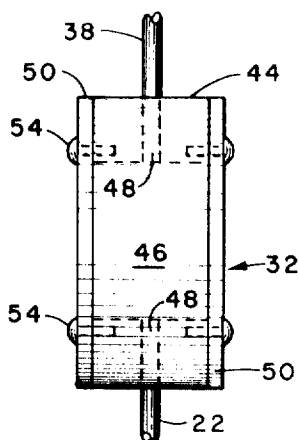


FIG. 3

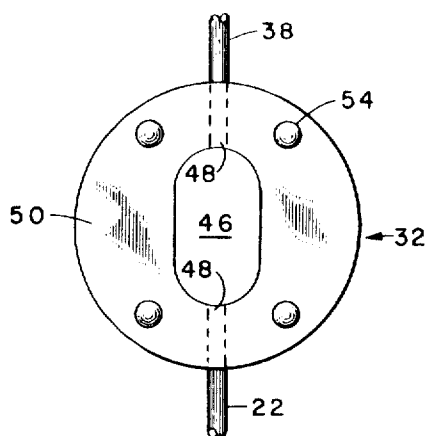


FIG. 4

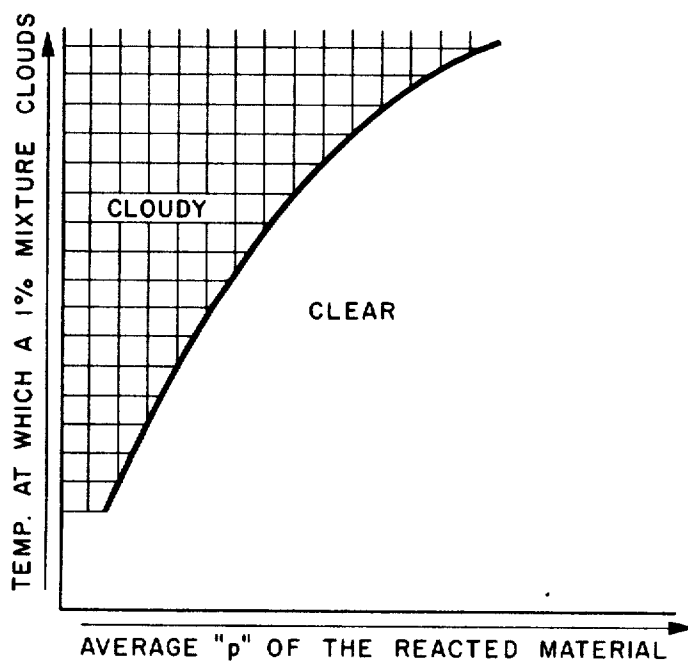


FIG. 5

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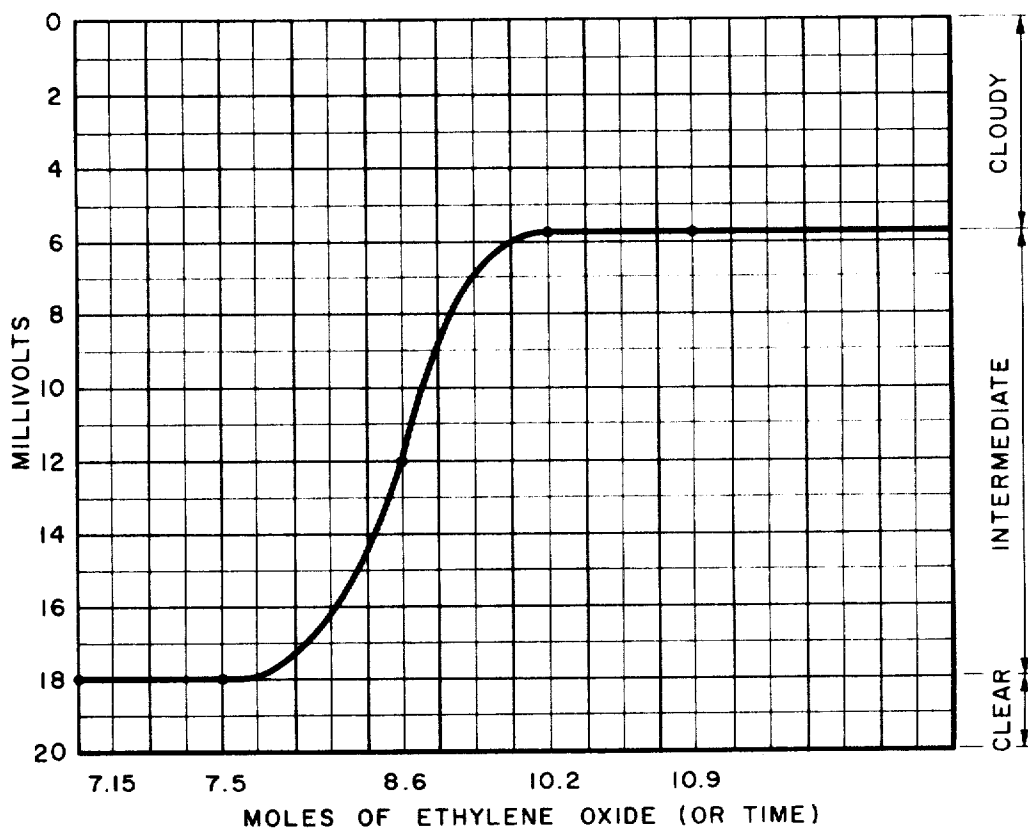


FIG. 6

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3,592,607

APPARATUS FOR CONTINUOUSLY MONITORING THE PROCESS OF CERTAIN REACTIONS

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Division of application Ser. No. 684,578, Sept. 25, 1967, now Patent No. 3,478,111, which is a continuation of application Ser. No. 531,084, Mar. 2, 1966, which in turn is a continuation-in-part of application Ser. No. 180,682, Mar. 19, 1962. This application Feb. 18, 1969, Ser. No. 800,176

Int. Cl. G01n 21/28

U.S. Cl. 23—253

2 Claims

ABSTRACT OF THE DISCLOSURE

Apparatus for determining the progress of a reaction between reactants which produce a product whose miscibility in a solvent is related to the extent of reaction. As the reaction proceeds, part of the reaction product is continually mixed with a solvent and flowed through a translucent cell mounted between a light source and a photoelectric cell. The temperature of the sample in the cell is either maintained constant during the reaction or is maintained at the changing miscibility temperature during the reaction by signals from the photoelectric cell transmitted to a temperature controller, and is particularly adapted to the oxylation of aliphatic alcohols, alkylphenols, and fatty acids.

DISCLOSURE

Cross-references to related applications

This application is a divisional application of my copending application entitled "Method and Apparatus for Continuously Monitoring the Progress of Certain Reactions," Ser. No. 684,578, filed Sept. 25, 1967 (now U.S. Pat. 3,478,111 issued Nov. 11, 1969), which in turn was a continuation of my then copending application entitled "Method and Apparatus for Continuously Monitoring the Progress of Certain Reactions," Ser. No. 531,084, filed Mar. 2, 1966, now abandoned, which in turn was a continuation-in-part of my application entitled "Method and Apparatus for Continuously Monitoring the Progress of Certain Reactions," Ser. No. 180,682, filed Mar. 19, 1962, now abandoned.

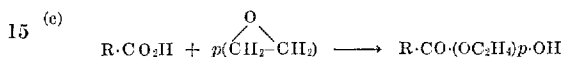
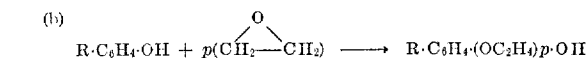
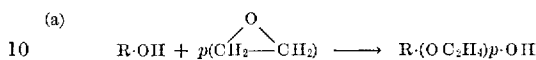
Background

This invention relates to apparatus for continuously monitoring the progress of certain chemical reactions and for automatically determining when certain types of addition reactions have proceeded to a desired end point. More particularly, but not by way of limitation, the present invention relates to novel apparatus which may be used to determine when a desired number of moles of an alkylene oxide have been added to compounds of certain types to produce alkylene oxide adducts having an optimum number of ether groups in each molecule thereof.

It is known that the products of some chemical reactions will vary in the extent to which they are miscible with a solvent according to the extent to which certain functional groups derived from one of the reactants has been incorporated in, or added to, the second reactant to yield the reaction product. Stated differently, in some types of chemical reactions, the number of moles of one reactant which has entered into the reaction by combining with, or adding to, but a single mole of a second reactant is directly related to the miscibility in one or more solvents of the product produced by the reaction. As examples of such reactions may be cited the

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reactions of alkylene oxides (or epoxides), particularly ethylene oxide and propylene oxide, with water, alkyl phenols, aliphatic polyhydric and monohydric alcohols (particularly the fatty alcohols), and fatty acids. In the latter three instances, the reactions may be expressed, respectively, by the following equations when ethylene oxide is the alkylene oxide employed:



In each of reactions (a), (b) and (c), R is an aliphatic radical and p is the number of moles of ethylene oxide which is added to the alcohol, phenol and fatty acid reactants. In each instance, the magnitude of p will depend upon the extent to which the reaction has been allowed to proceed. The miscibility of the products with water is related to the number of ethoxy groups incorporated therein.

Other types of reactions which yield a product which varies in its miscibility with water according to the extent which the reaction has proceeded are those reactions by which sulfonyl groups ($-SO_2-$) are added to the product to yield various types of sulfonic adducts. Yet other types of reactions by which products are yielded which vary in their miscibility with one or more solvents according to the extent to which the reaction has continued will be recalled by those skilled in the art.

It is frequently of considerable importance to know the extent to which reactions of the above-discussed types have proceeded. For example, each of the ethylene oxide reactions set forth in equation form above may be utilized to produce ethoxylated products having substantial utility as detergents. These ethoxylated products show a negative solubility coefficient in aqueous solution, especially if the degree of ethoxylation is low (where p is about 6 or less). This causes emulsions containing such products to become increasingly unstable with increasing temperatures. Other important properties, such as freeze point, sudsing ability, and dermatological mildness, are also closely related to the number of moles of ethylene oxide which is reacted, and which enters each of the products as an ethoxy group. In order to obtain the degree of product quality control which is required, it is thus necessary to in some manner be constantly apprised of the number of ethoxy groups which has been incorporated in the product.

One of the methods which has previously been employed for determining the number (p) of certain functional groups which have been incorporated in a particular product of the type described consists of periodically taking samples of the product, mixing the product with a suitable solvent, and observing the cloud point in each of the mixtures. The cloud point, of course, may be defined as the temperature at which a miscible mixture of the product with a certain solvent suddenly becomes an immiscible mixture. In the case of the reaction of ethylene oxide with aliphatic alcohols to produce aliphatic ethoxylated alcohols, samples of the ethoxylated product are periodically taken and are each added to water in the ratio of about 1 percent of product to 99 percent of water. Each sample is then heated until it suddenly becomes cloudy or immiscible. The temperature (cloud point) is noted. Each cloud point is directly related to the number of ethoxy groups which has been incorporated in the product, so that when a desired num-

ber of ether functional groups characterizes the product, a specific cloud point may be observed for the product.

Several disadvantages characterize the described method of determining when the desired end point of an epoxylation reaction has been reached. In the first place, it is laborious and time-consuming to take a series of samples of the reaction product and transport them to the laboratory for cloud point determinations. Also, a plurality of personnel including sample carriers and testers or chemists are required. A more important disadvantage, however, is the difficulty of correlating the observed cloud points with the extent to which the reaction has proceeded. This is because of the time lag which necessarily must occur between the time of sampling and the time of cloud point observation. By the time the cloud point corresponding to the desired product characteristic has been observed, the product has changed, and a greater number of epoxy groups has been incorporated in the product than desired. Only by extrapolation, with its attendant inherent errors, can this "overshooting" be avoided.

A major object of the present invention is to provide apparatus which automatically indicates when a desired reaction end point has been reached in those reactions which result in the sequential incorporation of a varying number of miscibility-affecting functional groups in the reaction product.

A further object of the present invention is to provide relatively inexpensive apparatus for monitoring the cloud point of a mixture of liquids in order to determine when a particular cloud point occurs as a result of variation in the chemical composition of one of the mixture constituents.

Another object of the invention is to provide an automatic cloud point-determining apparatus which is novelly adapted for use in determining the extent to which certain chemical reactions must proceed in order for the miscibility of the reaction product to reach a given value.

A further object of the invention is to provide an apparatus for automatically controlling the duration of a chemical reaction in which the miscibility of the reaction product in a solvent depends upon the extent to which the reaction has proceeded.

Another object of the present invention is to provide apparatus for automatically permitting only an optimum number of moles of ethylene oxide to react with a dihydric or monohydric aliphatic alcohol according to the desired properties of the reaction product.

Yet another object of the present invention is to provide an inexpensive, yet durable, apparatus for automatically indicating by a cloud point determination, when certain types of reactions have reached a desired end point.

Other objects and advantages of the invention will become apparent upon a further reading of the following detailed description of the invention in conjunction with a perusal of the accompanying drawings which illustrate my invention.

Summary of the invention

In one of its broader aspects, the present invention comprises automatically and continuously mixing a sample of the reaction product (which is continuously bled from the reaction chamber) in a constant predetermined proportion with a solvent in which the reaction product displays a cloud point when mixed in that predetermined proportion. Preferably, the proportion of reaction product to solvent is selected so that the cloud point which will characterize the mixture as to the number of miscibility-affecting functional groups in the product is within a range of conveniently attained and easily maintained temperatures. After the product and solvent are mixed, the mixture is continuously circulated through a conduit which passes through a controlled temperature chamber to a photoelectric testing system disposed within the chamber. Depending upon the manner in which the method is to be practiced, the temperature of the temperature chamber

either is maintained constant or is increased steadily during the course of monitoring. The photoelectric testing system consists of the usual photoelectric cell, source of electromagnetic radiation, and sample cell positioned between the latter two elements. For a more complete understanding of systems of this type, the reader is referred to U.S. Pat. 2,498,941 and German Pat. 666,557.

As the sample consisting of the product-solvent mixture flows through the controlled temperature chamber toward the sample cell, its temperature is altered to that prevailing in the chamber. As the sample enters the sample cell, it will be either a miscible or immiscible mixture, depending upon whether the reaction has proceeded beyond or, alternatively, has not yet reached a point where addition compounds containing sufficient miscibility-affecting functional groups have been formed to render the mixture immiscible at the temperature prevailing in the chamber. Depending upon its state of miscibility, the product-solvent mixture will attenuate the light beam passing there-through to a lesser or greater extent, which will in turn be reflected by the output from the photoelectric cell.

In one embodiment of the invention, the chamber temperature is preset and maintained at a value that corresponds to the cloud point temperature of the sample mixture when the reaction product has the desired number of miscibility-affecting functional groups incorporated therein. Thus, as the sample enters the sample cell, it will have attained this preset temperature, and will be either a miscible or immiscible mixture, depending upon whether the reaction has proceeded beyond, or alternatively, has not yet reached the desired end point. By commencing the flow of the sample mixture simultaneously with, or shortly after, the commencement of the reaction, the incorporation of the predetermined desired number of miscibility-affecting functional groups in the reaction product will be evidenced by a relatively sharp change in the sample mixture from a state of miscibility to a state of immiscibility, or vice versa. This change in turn is reflected by a change in the output from the photoelectric cell. The signal from the photoelectric cell may be utilized to drive a recorder, or to operate appropriate process control instruments to interrupt or stop the reaction. In this embodiment, the number of miscibility-affecting functional groups incorporated in the reaction product is not indicated at any time prior to the point where a maximum desired number of groups has been added.

In another embodiment of the invention, the chamber temperature is continually increased to maintain a temperature corresponding to the cloud point temperature of the mixture currently passing through the sample chamber. At the beginning of the reaction, the mixture of reaction product and solvent flowing through the sample cell will have a minimum cloud point temperature. As the reaction continues, the cloud point temperature will continuously increase and the temperature in the temperature chamber is automatically increased to correspond with this cloud point temperature. This is brought about by utilizing the output from the photoelectric cell, which determines the state of miscibility of the mixture, to regulate through intermediate means the temperature within the temperature chamber. At any time, then, the progress of the reaction is shown by the cloud point or miscibility temperature prevailing in the temperature chamber. The signal generated by a temperature indicator can be utilized to drive a recorder or to operate appropriate process control instruments to interrupt or stop the reaction.

DESCRIPTION OF THE DRAWINGS

In order to more completely describe the invention in its various aspects, reference is had to the following drawings of which:

FIG. 1 is a schematic view illustrating one embodiment of the apparatus which is utilized in practicing the method of the present invention.

FIG. 2 is a schematic view illustrating a second em-

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bodiment of the apparatus which is utilized in practicing the method of the present invention.

FIGS. 3 and 4 are detailed views of the sample cell which is utilized in the apparatus of the invention.

FIG. 5 is a graph illustrating the relationship between the number of moles of alkylene oxide which is reacted in one of the heretofore described reactions, and the cloud point of the reaction product.

FIG. 6 is a drawing illustrating the trace which is obtained upon the recording device of the apparatus as the reaction proceeds through a desired end point.

Detailed description

Referring now to the drawings in detail, and particularly to FIG. 1, reference character 10 designates a proportioning pump or other suitable mixing device which may be utilized for mixing two liquids in a predetermined proportion. For a better understanding of the structure and operation of a proportioning pump, the reader may refer to Lindsay United States Patent 2,656,845. Reference characters 12 and 14 designate conduits for conducting a solvent and reaction product, respectively, to the proportioning pump 10 from the solvent source and the reactor (not shown). Reference characters 16 and 18 designate conduits for discharging the solvent and reaction product, respectively, to the common mixing coupling 20. Conduit 22 connects to the discharge end of coupling 20, passes into controlled temperature chamber 24, coils into a plurality of convolutions 30, and connects with sample cell 32, which is positioned between an incandescent lamp 34, or other suitable source of electromagnetic radiation, and a photoelectric cell 36. Discharge conduit 38 connects to the opposite side of the sample cell 32. Suitable heating elements 26 are connected to a temperature control device 28, such as a thermostat, which permits the temperature within the controlled temperature chamber 24 to be altered as desired. Electrical leads 40 connect the photoelectric cell 36 to various types of meters, and to suitable control devices, such as that represented by reference character 43 in FIG. 1, for controlling the reaction to terminate it at any desired end point if the use of such devices is desired. A recorder 42 can also be connected by electrical leads in order to permit the current generated by the photoelectric cell to be registered in the form of visible indicia upon the recorder chart.

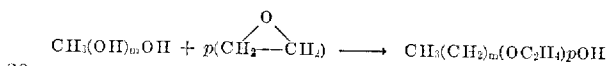
In FIG. 2, a second embodiment of the apparatus is presented. Reference characters 10 to 24, and 30 to 38 designate portions of the apparatus identical to those of FIG. 1. In this embodiment, however, electrical leads 40 connect the photoelectric cell 36 to a heater controller 60 which is controlled by signals from the photoelectric cell 36 and which, in turn, is connected to and controls the heating element 26. A change in the signal from the photoelectric cell 36 to the heater controller 60 is reflected by a decrease or increase in the electrical energy supplied to the heating elements 26, which in turn causes the temperature in the controlled temperature chamber to rise or fall. A thermocouple or other temperature indicating device 62 is mounted within the controlled temperature chamber and is connected by electrical leads 66 to various types of control meters such as designated by reference character 68 in FIG. 2 for controlling the reaction to terminate it at any desired cloud point if the use of such devices is desired. A recorder 64 can also be connected by electrical leads in order to permit the current generated by the thermocouple 62 to be registered in the form of visible indicia upon the recorder chart.

The construction of the sample cell 32 which is utilized in the apparatus of the invention may be best understood by reference to FIGS. 3 and 4. As illustrated in these figures, the sample cell comprises an annular, disc-shaped block 44 having an axial bore 46 therethrough, and provided with a pair of radial passageways 48 communicating with the axial bore and provided for the purpose of permitting the sample line 22 and discharge line 38 to be

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connected to the sample cell. The annular block 44 has a pair of opposed transparent, parallel faces 50 which form a pair of cell windows and are held on said block by screws 54 or other suitable fastening elements.

Before commencing a description of the operation of the first-described embodiment of the apparatus of the present invention, it is thought that it would be useful in imparting a better understanding of the invention to explain in greater detail the basic principle upon which the invention is bottomed. A typical reaction of the type which has previously been described, and which may be utilized in explaining such principle, is the reaction of ethylene oxide with an aliphatic monohydric alcohol to produce an ethoxylated aliphatic alcohol. The postulated reaction may be represented by the following equation:



Where m defines the length of the aliphatic monohydric alcohol used as one of the reactants, and p is the number of moles of ethylene oxide which is added to the alcohol and enters the product as ethoxy groups. Alcohol-ethylene oxide reactions of this general type are described in greater detail in Carter, U.S. Pat. 2,870,220. Products of this type have a variety of uses, and in many instances such uses will depend upon a specific value or range of values of the number of ethoxy groups which are incorporated in the product. Thus, in order to satisfy the requirements of a particular customer with respect to the extent of ethoxylation of the product, the manufacturer of the ethoxylated alcohol must be constantly apprised of the number of moles of ethylene oxide which has reacted with the aliphatic alcohol in order that he may terminate the reaction at the desired point.

As illustrated by the graph shown in FIG. 5, for a given mixture of reaction product and a solvent, such as water, a correlation between cloud point and the number of epoxy groups in the product exists, and may be shown by plotting the cloud point against the average p of the product. It is known in advance of the cloud point tests which specific cloud point corresponds, for a particular product-solvent mixture, to a specific number of moles of alkylene oxide which has reacted and which appears in the product as the same number of epoxy groups. Thus, when any sample of product displays the particular cloud point which corresponds to a predetermined number of moles of alkylene oxide which has entered into the reaction, the reaction can be terminated so that the product yielded does not contain an excessive number of epoxy groups.

By the apparatus of the present invention, the disadvantages which have characterized the previous method of determining the end point of reactions by which miscibility-affecting groups are incorporated in the reaction product have been overcome, and a considerably more economical and accurate method for attaining this control is contemplated. In the first embodiment of the apparatus (FIG. 1), a small portion of the reaction product is continuously bled from the reactor and passed into the proportioning pump 10 through the conduit 14. Simultaneously, a suitable solvent in which the reaction product is miscible, at least in certain proportions, is charged to the proportioning pump through a second conduit 12. The proportioning pump 10 is adjusted so that its output of reaction product and solvent through the conduits 18 and 16, respectively, is such as to give a mixture of predetermined proportions when these materials are mixed in the mixing valve 20. For example, when the aliphatic alcohol-ethylene oxide reaction discussed above is the reaction of concern, the proportions in the mixture which is produced may range between 1 percent of the product in 99 percent of water to 25 percent of product in 75 percent of water.

After the product and solvent are mixed in the mixing valve 20, the mixture is passed through the sample line 22 into the controlled temperature chamber 24 where the sample mixture is heated to the temperature of the chamber as it passes through the convolutions 30. The temperature of the controlled temperature chamber 24 has previously been adjusted to a temperature equal to the cloud point which characterizes the sample system when the reaction product includes a desired number of miscibility-affecting functional groups. Thus, prior to the time the sample mixture is introduced to the sample cell 32, the sample has attained a temperature at which the miscibility of its components will change more or less drastically when the reaction has reached a stage such that the desired number of miscibility-affecting functional groups have been incorporated into the reaction product. The process of bleeding a portion of the reaction product from the reactor and passing it through the apparatus of the present invention will, of course, have been commenced at the time the reaction is commenced, or shortly thereafter, so that there is no danger of overshooting or bypassing the reaction end point which it is the function of the incandescent lamp 34 and photoelectric cell 36 to observe.

In the early stages of the reaction, the solvent and reaction product will be either immiscible and therefore characterized by a cloudy appearance, or will be miscible and clear. In the case of the alcohol-ethylene oxide reaction, the mixture is initially immiscible and cloudy. The degree of miscibility of the reaction product in water increases as the number of ethoxy groups incorporated in the product is increased. As the mixture of product and solvent approaches miscibility as a result of the further inclusion of miscibility-affecting functional groups in the product, the mixture approaches a cloud point corresponding to the temperature of the controlled temperature chamber 24 and also, of course, of the photoelectric detecting unit enclosed in the chamber.

At the instant when the predetermined desired number of miscibility-affecting functional groups is incorporated in the reaction product, the previously immiscible sample system will undergo a relatively drastic change in the miscibility of its components. This will result in a sharp increase in the amount of light or other electromagnetic radiation which is passed by the sample in the sample cell 32 and which impinges upon the photoelectric cell 36. The sharp change in the intensity of radiation acting upon the photoelectric cell 36 will in turn be reflected by a clearly discernible change in movement of the pen of the recorder 42. The output signal from the photoelectric cell 36 may also be utilized to operate control equipment of any suitable type for shutting off the flow of ethylene oxide to the reactor and thus terminating the reaction, dumping the finished batch of reaction product, or any other control function which may be desired. After passing out of the sample cell 32 in the discharge line 38, the sample may be disposed of in any suitable manner.

As a further example of the manner in which the invention functions, the attention of the reader is invited to FIG. 6 of the accompanying drawings which shows a typical trace developed by a continuous recording instrument in response to the output from the photoelectric cell 36 when a sample of ethoxylated aliphatic alcohol in water was passed through the sample cell 32. The proportion of ethoxylated product to water was 1 to 99, or in other words, a 1% aqueous solution of the reaction product. The temperature within the controlled temperature chamber 24 was adjusted to 58° C., the temperature corresponding to the cloud point of an ethoxylated aliphatic alcohol containing an average of 8.6 ethoxy groups in each mole of product. The trace commences at the lower left corner of the chart as a product resulting from the reaction of an average of 7.15 moles of ethylene oxide with each mole of alcohol is passed through the sample cell. As the reaction proceeded, no deflection of the re-

corder pen occurred when 7.5 moles of ethylene oxide had reacted with the alcohol. However, when 8.6 moles of the ethylene oxide had reacted, a pen deflection corresponding to 6 millivolts was noted. When 10.2 moles of ethylene oxide had reacted, a further deflection of the pen of 6 millivolts occurred. After this, no further pen deflection was produced by further reaction of the ethylene oxide.

From the recorder chart shown in FIG. 6, it will be noted that a total pen deflection of 12 millivolts was produced by a difference of 2.7 moles of ethylene oxide reacted. The instrument thus may be said to display a sensitivity of about 4.5 millivolts per mole. It may be seen that the extent of reaction may be easily determined from an observation of the recorder trace.

In the second embodiment of the apparatus (FIG. 2), the signal from the photoelectric cell is utilized to maintain the temperature of the temperature chamber to correspond with the cloud point temperature of the mixture passing through the sample cell.

The flow of solvent, reaction product, and sample mixture of the two is identical to that in FIG. 1. In passing through the convolutions 30, the sample mixture is heated to the temperature of the chamber. The temperature of the controlled temperature chamber 24 is equal, or nearly so, to the current cloud point temperature of the sample mixture passing through the sample cell 32 by virtue of the control action of the photoelectric cell output signal. When the output from the photoelectric cell to the heater controller 60 indicates the presence of a cloud point in the sample mixture passing through the sample cell, the heater controller causes less electrical current to be supplied to the heater 26 to decrease the temperature in the temperature chamber and, correspondingly, the temperature of the sample mixture passing through the convolutions 30. Thus, the temperature in the controlled temperature chamber is always equal, or nearly so, to the cloud point temperature of the sample mixture in the sample cell. The progress of the reaction is thus always apparent by comparing the cloud point temperature of the sample mixture as shown by thermocouple 62 with a previous correlation of cloud point temperature with extent of reaction.

The heater controller 60 may take the form in which the signal from the photocell 36 is transmitted through a current converter (Foxboro 693, for example) and to a primary controller (Foxboro M/62, for example); simultaneously the output from the thermocouple 62 is transmitted by an electrical lead (not shown) through a current converter (Foxboro 693, for example) to a cascade controller (Foxboro M/67SRG2 with controller, for example); the difference in signals between the output from the primary controller and the current converter attached to the thermocouple 62 as measured by the cascade converter are then passed through a control device to a current regulator (such as a saturable core) device to increase or decrease the current supplied to the heater 26.

The output from the thermocouple 62 or from a current converter is passed through electrical lead 66 to a recorder 64 in order to permit the current generated by the thermocouple 62 to be registered in the form of visible indicia upon the recorder chart. The output from the thermocouple 62 or a current converter can also be directed to various types of meters and to suitable control devices such as that represented by reference character 68 for controlling the reaction to terminate the reaction at any desired end point as preferred.

The following examples are presented in further illustration of the invention.

Example 1.—A mixture of of aliphatic straight chain alcohols averaging 43 percent C₁₂, 30 percent C₁₄, 20 percent C₁₆, and 12 percent C₁₈ is reacted with ethylene oxide. It is desired to conduct the reaction to a point

where 8.6 moles of ethylene oxide per mole of alcohol is incorporated in the reaction product. The cloud point of a mixture of 1 volume of the desired reaction product in 99 volumes of water is previously determined to be 58° C. From a reaction chamber in which the alcohol mixture and ethylene oxide are reacted over a period of time, a sample stream of the reaction product is continually withdrawn. This sample stream is continually mixed with water in a ratio of 1 volume of reaction product to 99 volumes of water. This mixture of water and reaction product is then continually circulated through a temperature zone maintained at 58° C., the temperature, as just stated, corresponding to the cloud point of the ethoxylated alcohol product desired. While maintained at the 58° C. temperature, the mixture is then circulated through a photoelectric system which measures the turbidity or state of miscibility of the mixture. As the reaction continues, turbidity of the sample stream is indicated by the photoelectric cell when 7.5 moles of ethylene oxide has been reacted with the alcohol. However, when 8.6 moles of the ethylene oxide has reacted, the photoelectric cell begins indicating the presence of a miscible mixture and continues to increasingly indicate a condition of miscibility. The reaction of the ethylene oxide with the alcohol is stopped shortly thereafter.

Example 2.—A sample stream of reaction product is continually withdrawn from a reaction chamber in which the same kind of alcohol mixture and ethylene oxide as in Example 1 is reacted over a period of time. Withdrawal of the sample stream is begun at the start of the reaction. It is desired to continually monitor the course and extent of the reaction in progress. The sample stream is continually mixed with water in a ratio of one volume of sample to 99 volumes of water. Again, it is desired to react the alcohol and ethylene oxide so that about 8.6 moles of ethylene oxide are added per mole of alcohol. This again corresponds to a cloud point temperature of about 58° C. of the reaction product-water mixture. This mixture of water and reaction product is then circulated through a temperature zone initially at a temperature of 30° C., so that the sample mixture is brought to this temperature of 30° C. The mixture, originally in a state of complete immiscibility, is continually circulated through a photoelectric cell wherein its turbidity or state of miscibility is determined. As the reaction proceeds, sufficient moles of ethylene oxide are incorporated in the reaction product to render the mixture miscible. When the photoelectric cell indicates this condition of transition between immiscibility and miscibility, the temperature in the temperature zone is increased slightly so that the mixture reverts to its original immiscible state. As the reaction between alcohol and ethylene oxide continues and the mixture of water and reaction product is circulated through the temperature zone and photoelectric cell, the cloud point temperature of the mixture continually increases. The temperature in the temperature zone is continually increased to maintain the mixture just at the threshold of the cloud point, as shown by the photoelectric cell. When the cloud point temperature reaches 58° C., the reaction has proceeded to a point where about 8.6 moles of ethylene oxide have been added per mole of alcohol and the reaction is stopped.

From the foregoing description of the invention, it will be appreciated that a relatively inexpensive apparatus for rapidly and constantly monitoring certain types of reactions has been proposed. The invention eliminates the time lag between observation of cloud point and reaction product sampling which has previously been characteristic of the methods used in monitoring and controlling such reactions.

Although reference has been made herein to certain preferred embodiments of the invention, it is to be understood that these are described by way of example and illustration only. Accordingly, it is contemplated that

modifications and variations can be made in the operating techniques described without departing from the true spirit and scope of the invention.

What is claimed is:

1. A system for monitoring an addition reaction between an alkylene oxide compound and an aliphatic alcohol to produce an addition compound containing a desired average number of molecules of said alkylene oxide compound added to each molecule of aliphatic alcohol, said desired average number increasing with length of reaction time and said addition compound when mixed with a solvent displaying a cloud point indicative of the average number of molecules of said alkylene oxide compound added to each molecule of said aliphatic alcohol, said system comprising:

- (a) a liquid proportioning pump into which said reagents are introduced for releasing therefrom in predetermined proportions;
- (b) a mixing coupling for combining said reagents to give a mixture of predetermined proportions;
- (c) a conduit connecting said proportioning pump and said mixing coupling for the passage of said reagents;
- (d) a hollow temperature chamber for heating said mixture of reagents to a desired temperature;
- (e) heating elements in said temperature chamber for bringing the temperature therein to a desired degree;
- (f) a temperature control device connected to said heating elements to manipulate the output of said heating elements thereby determining the temperature within said temperature chamber;
- (g) a convoluted conduit in said temperature chamber through which said mixture of reagents is passed to make the temperature of said mixture correspond to the temperature of said chamber;
- (h) a conduit connecting said convoluted conduit and said mixing coupling for the passage of said mixture of reagents;
- (i) a photoelectric cell positioned in said temperature chamber so as to receive a beam of light after passage thereof through said mixture of reagents;
- (j) a light source positioned opposite said photoelectric cell in said temperature chamber so as to interact therewith by emanating a beam of light which passes through said mixture of reagents and is received by said photoelectric cell to determine the degree of miscibility of said mixture at a given point of the reaction;
- (k) a sample cell in said temperature chamber positioned between said photoelectric cell and said source of light so as to allow a light beam emanating from said light source to pass through said mixture of reagents contained therein, and be recorded by said photoelectric cells, said sample cell comprising a cylindrical block having an axial bore therethrough for the containment of said mixture and having a pair of radial passageways therethrough communicating with the axial bore for the passage of said mixture therethrough, and a pair of opposed, transparent parallel faces secured to the opposite sides of said cylindrical block and covering the axial bore therethrough at each end of said bore so as to permit the transmittal of a light beam emanating from said light source through said mixture contained in said axial bore;
- (l) a conduit connected to the radial passageways in said block and to said convoluted conduit for the passage of said mixture; and
- (m) a recording device connected to said photoelectric cell to permit current generated by the receipt of said light beam passing through said mixture by said photoelectric cell to be registered in the form of visible indicia upon a recorder chart.

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2. The system of claim 1 wherein said temperature control device of (f) is also connected to the output of said photoelectric cell; and there is substituted for said recording device of (m) connected to said photoelectric cell (i) a recording device connected to said temperature chamber (d).

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