

[54] **METHOD AND APPARATUS FOR THE ANALYSIS OF A DISPERSED PHASE CAPABLE OF TRANSMITTING AND FOCUSING LIGHT**
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[63] Continuation-in-part of Ser. No. 309,644, Nov. 27, 1972, abandoned.
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[51] Int. Cl. **G01n 21/22**
[58] Field of Search 250/218, 218 X, 217 SS, 250/573, 576; 356/128, 201, 205, 206, 207, 208, 102, 103, 104

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[57] **ABSTRACT**

A method and apparatus for the analysis of a dispersed phase capable of transmitting and focusing light in a continuous fluid phase capable of transmitting light wherein a light beam of varying intensity is passed through the dispersed phase contained in the continuous phase whereby the light is focused by the dispersed phase to increase the intensity of light striking a light sensing means to increase the signal of the sensing means and the variation is subtracted from the signal of the sensing means to provide a more discernible difference between signals induced by the presence of the dispersed phase and signals induced by the presence of opaque contaminants.

31 Claims, 6 Drawing Figures

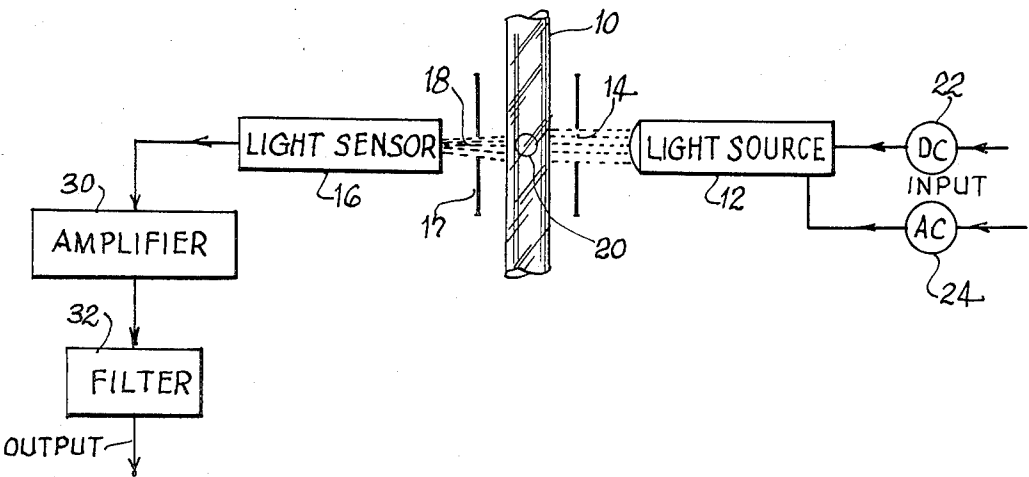


FIG. 1

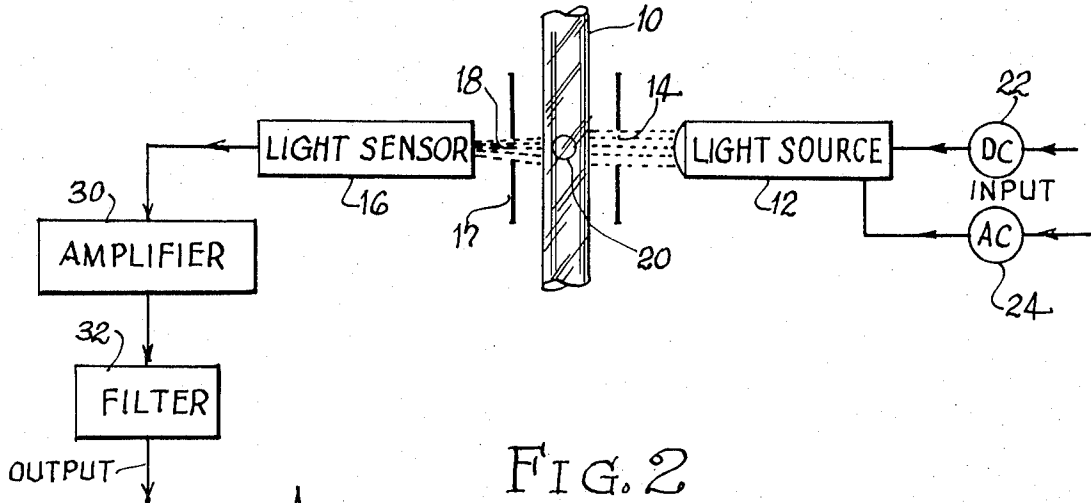


FIG. 2

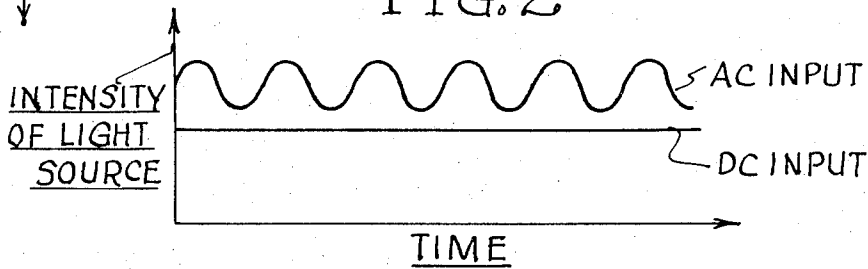


FIG. 3

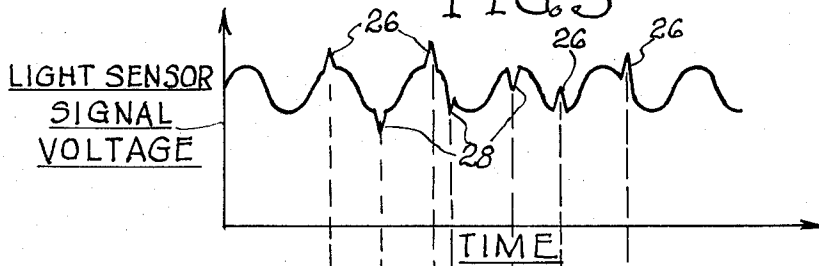


FIG. 4

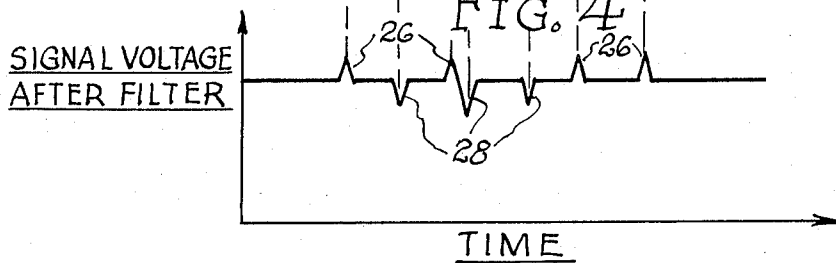


FIG. 5

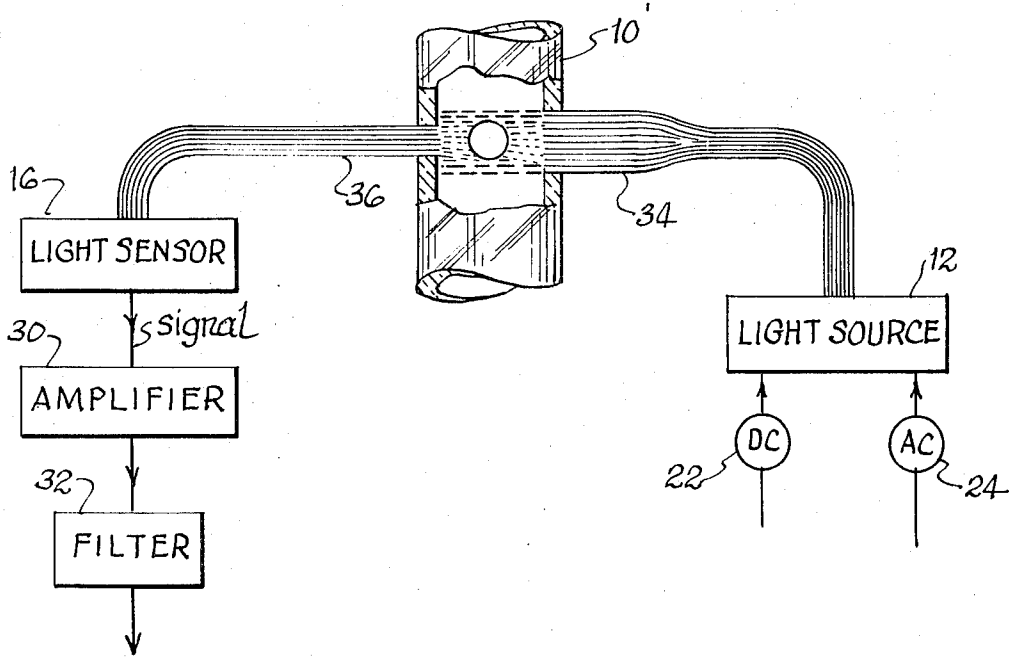
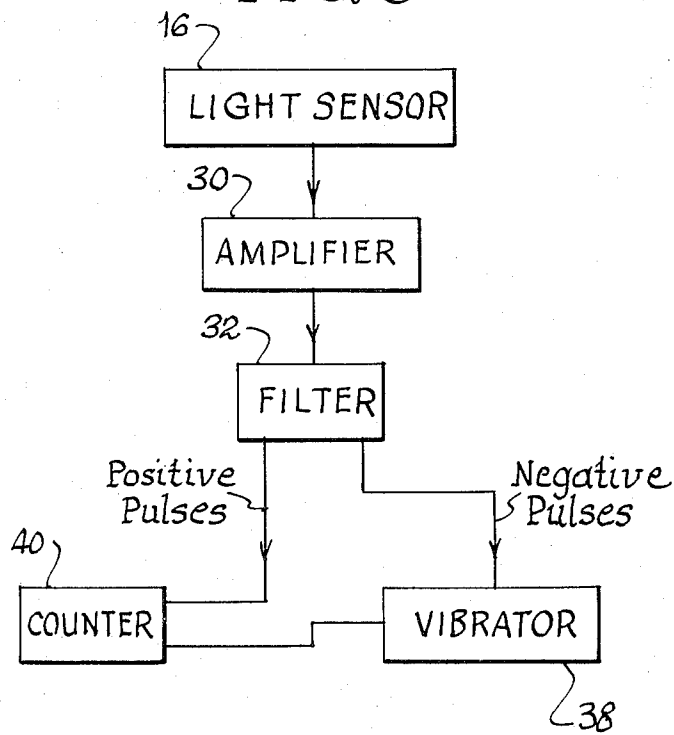


FIG. 6



METHOD AND APPARATUS FOR THE ANALYSIS OF A DISPERSED PHASE CAPABLE OF TRANSMITTING AND FOCUSING LIGHT

This application is a continuation-in-part of copending application Ser. No. 309,644, filed Nov. 27, 1972, and now abandoned.

This invention relates to an improved method and apparatus for the analysis of liquid systems, and more particularly to a method and apparatus for the analysis of a liquid dispersed in another liquid which is immiscible with the dispersed liquid.

A number of analytical techniques have been employed in the analysis of liquid systems formed of droplets or globules of a liquid dispersed in a continuous phase of another immiscible liquid. A typical liquid system of this type is an oil-in-water system in which the oil is dispersed throughout the water in the form of fine droplets or globules generally having a spherical configuration. Analysis of such systems is usually carried out with UV absorption or light scattering techniques.

However, such techniques have not been altogether satisfactory for they require costly and complex analytical apparatus. Even with such complex equipment, the precision of such analytical techniques is quite limited.

In my copending application Ser. No. 309,644, filed Nov. 27, 1972, and now abandoned, there is described an improved method and apparatus for the analysis of fluid systems containing droplets or globules of a liquid dispersed in the continuous phase of another liquid in which a light beam is passed through the continuous phase containing the droplets whereby the droplets exert a lens effect to focus the light beam on a light sensor. Each time the light beam passes through a droplet or globule, the intensity of light falling upon the sensor is increased to thereby provide an increased signal from the light sensor. The frequency of the increased signals or pulses from the light sensor thus serves to indicate the number of droplets present in the continuous phase and consequently the relative amount of the dispersed liquid contained in the continuous phase.

While the invention disclosed and claimed in the above application represents a significant advance in the art, the presence of contaminants which do not transmit light in the continuous phase effects the accuracy of the analysis. This effect can be reduced somewhat by a feedback from the light sensor to the light source as described in the above application. However, it is frequently quite difficult to detect the positive pulses resulting from the presence of the dispersed phase because of their small amplitude and to distinguish between the positive signal of the light source caused by the presence of a droplet and the negative signal caused by the presence of dirt particles which interrupt the light beam.

It is accordingly an object of the present invention to provide an improved method and apparatus for the analysis of a liquid dispersed in an immiscible liquid which overcomes the foregoing disadvantages and is capable of providing reliable analytical data even when the liquid system contains contaminants opaque to light.

It is a more specific object of the invention to provide a method and apparatus for the analysis of a liquid dispersed in an immiscible liquid in which the signal caused by the presence of the dispersed liquid is more

clearly discernible from the signal caused by the presence of contaminants which do not transmit light.

It is a further object of the invention to provide a method and apparatus for the analysis of a liquid dispersed in an immiscible liquid in which a light beam is based through a fiber optics system for passage through the immiscible liquid containing the dispersed phase and the light beam is received by a fiber optics system for passage to a light receiving means to detect increases in light intensity as the light beam is focused by the dispersed phase.

These and other objects and advantages of the invention will appear more fully hereinafter and, for purposes of illustration but not of limitation, embodiments of the invention are shown in the accompanying drawings in which:

FIG. 1 is a schematic illustration of the apparatus of the present invention;

FIG. 2 is a graph of the input voltage to the light source shown in FIG. 1 with time;

FIG. 3 is a graph of the output voltage of the light sensor of FIG. 1 with time;

FIG. 4 is a graph of the output voltage of the filter of FIG. 1 with time;

FIG. 5 is a schematic illustration of an alternative embodiment of the apparatus of FIG. 1; and

FIG. 6 is a schematic diagram of means to correct the analysis as a result of contamination.

The concepts of the present invention reside in the discovery that, when a light beam is passed through a continuous fluid phase containing a dispersed fluid immiscible with the continuous phase which is capable of transmitting and focusing a beam of light and containing contaminants which are at least opaque to light, the positive pulses of increased light intensity are more readily discernible from negative pulses of increased light intensity where the intensity of the light beam passed through the continuous phase is varied and the resulting signal from the light sensor or sensing means is filtered to remove the variation intensity. In addition to providing a more clearly discernible difference between such negative and positive pulses as described, the concept of varying the intensity of the light beam likewise provides a signal in which problems of drift and DC coupling are substantially eliminated.

In the preferred practice of the invention, the light source employed is a light emitting diode which is energized with an encoded signal, such as an AC signal, a saw-tooth signal or a square-wave signal superimposed upon a DC signal to thereby provide a source of light in which the intensity of light varies in accordance with the encoded signal with time. In general, the effective voltage of the encoded signal superimposed upon the DC signal is small in magnitude compared to the magnitude of the DC signal. While not critical to the practice of the invention, it has been found that best results are usually achieved when the effective voltage of the encoded signal is from two to 50 and preferably five to 15 times the expected increase in light intensity due to the presence of the dispersed phase in the continuous phase.

Similarly, while not critical to the practice of the invention, best results are obtained where the frequency of the encoded signal ranges from 1/5 to 1/50, and preferably 1/15 to 1/30, times the frequency of globules or droplets of the dispersed phase. By way of illustration, it has been found that a DC signal of about 8 volts ef-

fective and an AC signal of about 100 millivolts effective and about 60 cps provide good results in the analysis of oil-in-water dispersions.

Referring now to the drawings for a more detailed description of the invention, there is shown in FIG. 1 a schematic illustration of the apparatus of the invention which is of the same general type as that disclosed and claimed in my copending application referred to above. As will be appreciated by those skilled in the art, the dimensions of the drawings have been significantly enlarged to illustrate the details of the apparatus. The apparatus includes passage means or chamber 10 through which the dispersion can be passed. A light source 12 is positioned adjacent to the chamber means 10 to pass a wide beam of light through a wide slit 14 and through the transparent chamber 10. Any source of light can be used in the practice of this invention, including an incandescent lamp, a laser, a light emitting diode or the like.

Light sensing means 16 is positioned to receive the light passed through the chamber 10. The light sensing means 16 can be any of a number of light sensitive components capable of measuring or detecting a difference intensity of a beam of light, such as a phototransistor, a photomultiplier tube, a photocell or the like. Interposed between the light sensing means 16 and the chamber 10 is a light shield 17 having a narrow slit 18 therein, with the slit 18 being aligned with the light sensing means 16, the wide slit 14 and the light source 12.

Where the light beam emanating from the light source 12 passes through the continuous phase of the dispersion contained in the chamber 10, the light beam is at least partially shielded from the light sensing means 15 by way of the shield 17, with only a portion of the light passing through the slit 18 to strike the light sensing means 16. At this time, the signal or output of the light sensing means 16 substantially corresponds to the input signal of the light source 12. However, when a discrete droplet 20 of the dispersed liquid passes through the beam of light, the droplet serves as a convex lens to focus the light beam onto the light sensing means 16, and thereby subject the light sensing means 16 to light of greater intensity to increase the signal of the light sensing means 16 to detect the presence of the droplet 20.

The relative dimensions of the wide slit 14 or breadth of the light beam from the light source 12 with respect to the width of the narrow slit 18 is not critical to the practice of the invention. It is generally sufficient that the narrow slit 18 be sufficiently small to provide a measurable difference in light intensity of the focused beam of light, that is the intensity of the beam as it is focused on the light sensing means 16 by a droplet of the dispersed liquid, as compared to the light intensity of the light beam passing through the continuous phase.

It is also preferred in the practice of the invention to space the light sensing means by a distance sufficient that the distance between a droplet in the chamber and the light sensing means is less than the focal length of the droplet. In this way, a maximum difference between the focused beam of light as compared to the non-focused beam of light is assured.

In the practice of this invention, the apparatus is provided with means to energize the light source 12 including means to provide a source of light of constant intensity and means to provide light, superimposed on the

light of constant intensity, whose intensity varies with time. In the embodiment shown in FIG. 1 of the drawing, the light source 12 is preferably a light emitting diode which is energized by a source 22 of DC voltage and a source 24 of AC voltage.

The intensity of the light emitted from light source 12 is shown graphically in FIG. 2 of the drawing. As illustrated, the light emitted as a result of the DC excitation is constant with time. Superimposed upon the light of constant intensity is light emitted as a result of the AC excitation of the light emitting diode whose intensity varies sinusoidally with time.

The light beam having the characteristics illustrated in FIG. 2 of the drawing is passed through the chamber 10 as described above, and when a droplet or globule of the dispersed phase passes through the chamber 10, the light beam is focused onto the light sensor 16 to provide an increase or pulse in the intensity of the light illuminating the sensor 16. Similarly, when a particle of a contaminant material, which is at least opaque to light, passes through the chamber, the light beam is at least partially interrupted by the particle, thereby resulting in a decrease or "negative pulse" in the intensity of the light illuminating the light sensing means 16.

As will be appreciated by those skilled in the art, the signal from the light sensing means reflects the variation in the light illuminating the sensing means 16; the signal from the light sensing means is shown in FIG. 3 of the drawing. The sine wave reflects the sinusoidally varying light emitted from the light source 12, and the positive pulses 26 detected result from the dispersed phase serving to focus the light beam to increase the intensity of light incident on the sensing means 16 whereas the "negative pulses" 28 result from the light-opaque contaminant particles serving to decrease the intensity of light incident upon the light sensing means 16.

The signal from the light sensing means 16 can then be amplified by means of a suitable amplifier 30 provided in the system. Amplification of the varying signal from the sensing means 16 permits detection of both positive and "negative" pulses which are otherwise quite small in magnitude relative to the magnitude of the DC voltage and consequently difficult to detect absent identification.

The amplified signal from the amplifier means 30 is then processed by passing the signal to a filter 32 or the like means to subtract the varying signal introduced by the excitation of the light source 12. The filter 32 thus serves to subtract the sinusoidal variation from the signal from the amplifier, leaving only the amplified positive pulses established by the dispersed phase and the "negative" pulses established by the contaminant particles present in the continuous phase.

The signal, after subtraction of the encoded signal, is represented by the graph of FIG. 4. As can be seen from this figure, the signal has a substantially constant value corresponding to the DC signal input to the light source 12, with the amplified positive and negative pulses 26' and 28', respectively, indicating the presence of the dispersed phase and the opaque contaminant, respectively. The resulting signal can be, if desired, amplified by suitable amplifier means 32 without the problems of drift and DC coupling to provide greater reliability in reading out analytical data from the electronic signal.

As will be appreciated by those skilled in the art, the filter means to subtract the varying signal can be any of the filters well-known to those skilled in the art. For example, use can be made of high pass turned filters for this purpose.

While the invention has been described above with reference to an encoded signal in the form of an AC signal superimposed on the DC signal to the light source, the invention contemplates the use of other encoded signals which vary in magnitude and/or frequency with time. As indicated above, use can be made of an encoded signal in the form of a saw tooth signal or a square wave signal.

Another embodiment of the invention is shown in FIG. 5 of the drawing. The apparatus of this embodiment is similar to that shown in FIG. 1 of the drawing, and the same elements are designated by the same reference numerals. However, instead of having the light source 12 and the light sensing means 16 in alignment with the slits 14 and 18, the light beam is transmitted through fiber optics means 34 for passage through the chamber 10', and the light beam passing through the chamber 10' is received by second fiber optics means 36 for transmission therethrough to the light sensing means. By the use of such fiber optics systems, it is possible to pass the light beam to and from the chamber 10' through a curved path and consequently reduce the size of the overall apparatus. The use of fiber optics means as described and shown in the drawings obviates the need to employ slits of the type used in FIG. 1 since the cross-sectional area of the fiber optics means 34 is dimensioned to be greater than that of the fiber optics means 36 to assure the desired measurable difference in intensity of the light illuminating fiber optics means 36 when a droplet or globule of the dispersed phase intercepts the light beam from fiber optics means 34 to fiber optics means 36.

As is now well known to those skilled in the art, such fiber optics systems, which are commercially available, are generally formed of a bundle of glass or plastic fibers which are capable of transmitting light over their entire lengths. Because the bundle is formed of a plurality of individual fibers, the bundle is flexible and is capable of conducting light through a curved path.

In the preferred embodiment of the invention, the apparatus is included with means to compensate for error in analysis due to the presence of contaminant material which is opaque to light in the continuous phase. As those skilled in the art will appreciate, the apparatus of the invention is incapable of detecting a droplet or globule of the dispersed phase present in the light beam when a light-opaque particle is likewise in the light beam. To correct for such errors in detection and analysis, the apparatus can include a monostable multivibrator operatively connected to, for example, the filter as shown in FIG. 6 of the drawing. Negative pulses constituting a portion of the signal from the filter are pulses through the vibrator, and positive pulses are supplied to counting means 40. The negative pulses are thus converted to a pulse of greater time duration by the vibrator, and the latter are counted by the counting means 40 to correct the number of positive pulses for the period in which the positive pulses cannot be counted due to the presence of the contaminant.

In the practice of this invention, a mixture of the immiscible liquids is just homogenized to insure that a substantially complete dispersion is obtained. The for-

mation and stability of the dispersion can frequently be enhanced by carrying out the homogenization in the presence of a surfactant. Thereafter, the dispersion is placed in and/or passed through the chamber and the positive pulses or the light sensing means are counted as an indication of the number of droplets of the dispersed liquid contained in the continuous phase.

The concepts of the invention are applicable to systems of immiscible liquids which are capable of transmitting light and which contain one liquid dispersed as droplets or globules in another fluid as a continuous phase. It has been found that the present invention is particularly well suited for use in the analysis of oil-in-water emulsions and water-in-oil emulsions. As will be appreciated by those skilled in the art, the concepts of the invention are also applicable to dispersions of balls of solids which are capable of transmitting and focusing light dispersed in a fluid continuous phase.

It will be understood that various changes and modifications can be made in the details of construction, procedure and use without departing from the spirit of the invention, especially as defined in the following claims.

I claim:

1. Apparatus for analysis of a dispersed phase capable of transmitting and focusing light contained in a continuous phase capable of transmitting light, comprising chamber means adapted to contain the dispersed phase and the continuous phase, a light source positioned to pass a wide beam of light through the chamber means, means associated with the light source to generate the light beam with a component of varying intensity, light sensing means adapted to detect differences in light intensity positioned to receive a portion of the light beam passed through the chamber means whereby the dispersed phase is passed through the beam of light to focus the beam of light onto the light sensing means to cause an increase in the intensity of the light beam striking the light sensing means to activate the light sensing means for each dispersed phase in the continuous phase.

2. Apparatus as defined in claim 1 wherein the light sensing means includes a light shield interposed between the light sensing means and the chamber means, said light shield having a narrow slit therein adapted to pass a portion of the light beam therethrough.

3. Apparatus as defined in claim 1 wherein the light source includes a light shield having a wide slit therein adapted to define the beam of light.

4. Apparatus as defined in claim 2 wherein the narrow slit is dimensioned to pass the beam of light therethrough having an intensity measurable less than the intensity of the beam focused by the dispersed phase.

5. Apparatus as defined in claim 1 wherein the light sensing means is positioned from the dispersed phase in the chamber means by a distance less than the focal length of the dispersed phase.

6. Apparatus as defined in claim 1 wherein the means associated with the light source includes means to energize the light source to provide light having a relatively constant intensity and means to energize the light source to provide light of varying intensity whereby the light beam is composed of the light of relatively constant intensity on which the light of varying intensity is superimposed.

7. Apparatus as defined in claim 6 wherein the means to energize the light source to provide light of varying

intensity is means to generate light having sinusoidally varying intensity.

8. Apparatus as defined in claim 6 wherein the means to energize the light source to provide light of varying intensity is means to generate light having intensity varying as a square wave.

9. Apparatus as defined in claim 6 wherein the means to energize the light source to provide light of varying intensity is means to generate light having intensity varying as a sawtooth wave.

10. Apparatus as defined in claim 1 wherein the light source is a light emitting diode and the means associated with the light source includes means for providing a DC signal and means for providing a signal of varying intensity superimposed on the DC signal.

11. Apparatus as defined in claim 10 wherein the signal of varying intensity is an encoded signal.

12. Apparatus as defined in claim 1 which includes means to amplify the signal from the light sensing means.

13. Apparatus as defined in claim 12 which includes filter means to subtract a signal of varying intensity corresponding to the light of varying intensity of the light beam to leave a signal corresponding to increases in intensity of the light striking the light sensing means and decreases in intensity of the light striking the light sensing means due to presence of opaque contaminants.

14. Apparatus as defined in claim 13 which includes vibrator means adapted to pulse negative signals from the filter means to correct the number of increases in intensity of light during presence of contaminants in the continuous phase.

15. Apparatus as defined in claim 1 which includes first fiber optics means to conduct the light beam to the chamber means for transmission therethrough.

16. Apparatus as defined in claim 1 which includes second fiber optics means to conduct light transmitted through the chamber means to the light sensing means.

17. Apparatus for a dispersed phase capable of transmitting and focusing light contained in a continuous fluid phase capable of transmitting light comprising chamber means adapted to contain the dispersed phase contained in the continuous phase, a light source adapted to emit a beam of light, first fiber optics means to conduct the beam of light emitted from the light source to the chamber means for passage of the beam of light through the chamber means, light sensing means adapted to detect differences in light intensity, second fiber optics means positioned to receive the light beam passed through the chamber means and to conduct the light beam transmitted through the chamber means to the light sensing means whereby the dispersed phase is passed through the beam of light to focus the beam of light onto the light sensing means to cause an increase in light intensity of the light beam striking the light sensing means to activate the light sensing means for each dispersed phase in the continuous phase.

18. Apparatus as defined in claim 17 which includes means associated with the light source to provide the light beam with a component of varying intensity.

19. Apparatus as defined in claim 17 wherein the light source is a light emitting diode and the means associated with the light source includes means for providing a DC signal and means for providing a signal of varying intensity superimposed on the DC signal.

20. Apparatus as defined in claim 19 wherein the signal of varying intensity is an encoded signal.

21. Apparatus as defined in claim 17 which includes means to amplify the signal from the light sensing means.

22. Apparatus as defined in claim 21 which includes filter means to subtract a signal of varying intensity corresponding to the light of varying intensity of the light beam to leave a signal corresponding to increases in intensity of the light striking the light sensing means and decreases in intensity of the light striking the light sensing means due to presence of opaque contaminants.

23. Apparatus as defined in claim 22 which includes vibrator means adapted to pulse negative signal from the filter means to correct the number of increases in intensity of light during presence of contaminants in the continuous phase.

24. Apparatus as defined in claim 17 wherein the first fiber optics means has a greater cross-sectional area than the second fiber optics means.

25. Apparatus as defined in claim 24 wherein the second fiber optics means is dimensioned to pass the beam of light therethrough having an intensity measurably less than the intensity of the beam focused by the dispersed phase.

26. In an apparatus for the analysis of a dispersed phase capable of transmitting and focusing light in a continuous fluid phase which includes chamber means adapted to contain the dispersed phase contained in the continuous phase, a light source positioned to pass a wide beam of light through the chamber means, light sensing means adapted to detect differences in light intensity positioned to receive a portion of the light beam passed through the chamber means whereby the dispersed phase is passed through the beam of light to focus the beam of light onto the light sensing means to cause an increase in the intensity of the light beam striking the light sensing means, the improvement comprising at least one fiber optics means to conduct light between at least one of the light sensing means and the light source, and the chamber means.

27. Apparatus as defined in claim 26 which includes means associated with the light source to provide the light beam with a component of varying intensity.

28. A method for measuring a discrete dispersed phase capable of transmitting and focusing light contained in a continuous fluid phase comprising the steps of passing the dispersed phase contained in the continuous phase through a transparent zone, passing a broad beam of light having a component which varies in intensity whereby the beam of light is focused by discrete portions of the dispersed phase to provide impulses of light of increased intensity as compared to the intensity of said beam, converting the impulse to an electrical signal, amplifying the signal and subtracting the portion of the signal corresponding to said light varying intensity.

29. A method as defined in claim 28 wherein the dispersed phase is a water-immiscible oil and the continuous phase is an aqueous medium immiscible with the oil.

30. A method as defined in claim 28 which includes the step of homogenizing the dispersed phase in the presence of the continuous phase to form discrete droplets of the dispersed phase in the continuous phase.

31. A method as defined in claim 30 wherein the homogenizing is carried out in the presence of a surfactant to promote the formation of droplets of the dispersed phase in the continuous phase.