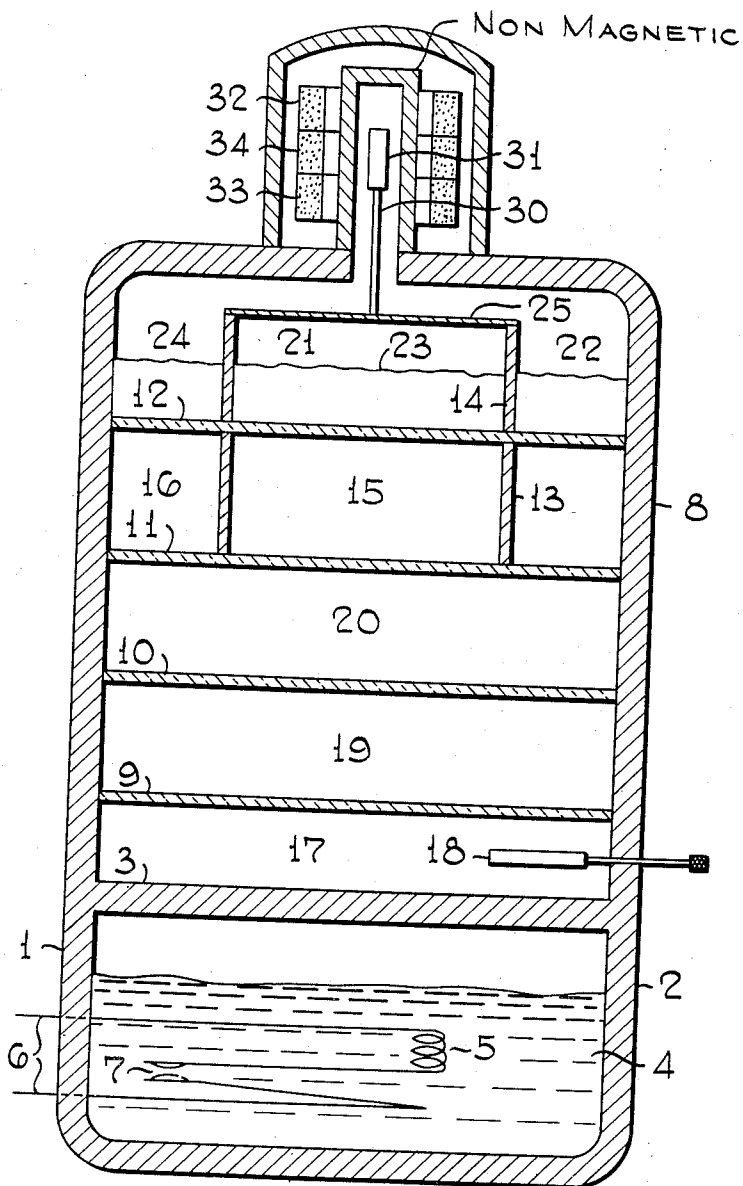


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INDUSTRIAL INFRARED ANALYZER

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INDUSTRIAL INFRARED ANALYZER

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This invention relates to improved infra-red analyzing apparatus. The analyzer of this invention is of particular application for commercial scale process control. The device is characterized by simplicity and ruggedness of construction, yet at the same time is capable of providing accurate analytical results.

At the present time the application of physical methods of analyses for plant control purposes is rapidly expanding. This expansion is particularly associated with continuous processes which are now in general use which preferably require precise, continuous analyses of reactant or product streams associated with the processes. A useful type of analytical control method which may be applied depends upon the determination of infra-red absorbing or transmitting characteristics of process streams. Particularly in the petroleum industry, this method is convenient and effective to determine small differences in hydrocarbon compositions by determining differences in infra-red absorbing characteristics of the compositions to be tested. However, a difficulty in applying infra-red analytical methods to actual plant control problems has heretofore been the inadequacy of conventional forms of infra-red analyzers. It is, therefore, the principal object of this invention to provide an improved type of infra-red analyzer better adapted to fulfill plant control requirements.

By way of example, present infra-red analyzers require expensive voltage regulation apparatus to avoid fluctuations in output which would otherwise result due to changes in line voltage, or frequency. Consequently, it is one object of this invention to overcome the necessity for use of voltage regulation, although avoiding any errors due to changes in voltage, or frequency.

A further object of this invention relates to the amount of energy emitted by the infra-red source, and the amount of energy which can be detected. In many applications a great deal of energy emitted from the infra-red source is absorbed by filters or infra-red absorbing fluids employed in the apparatus. For example, as little as 0.5° of the total energy emitted may be left to provide the required analytical results. This is in part due to the necessity for employing a substantially monochromatic band of infra-red energy in some analytical applications. With this factor in mind it can be appreciated that the greater the total energy emitted from the source, the more energy will be left for signal. Since the total energy available is proportional to the area of the emitting surface, it is desirable to ob-

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tain and utilize a source having as great an area as possible. The apparatus of this invention, therefore, utilizes an infra-red source having any desired area, and preferably having an area at least equal to the cross-sectional area of the cells employed in the apparatus. In a similar manner the apparatus of this invention is characterized by use of a detecting element which also has substantially the same area as the cross-sectional area of the cells employed.

A still further object of this invention which may be particularly noted, is to eliminate the effect of changes in ambient temperatures on the output of the apparatus. Thus a common type of conventional infra-red analyzer employs two differential thermopiles which are so arranged that changes in ambient temperatures may affect one of the thermopiles without affecting the other, creating errors in the analytical results obtained. The apparatus of this invention is not subject to this difficulty since any change in ambient temperature affecting one of the two detecting elements will also affect the other of the detecting elements to minimize, or eliminate any sensitivity to changes in temperature.

These and other objects of this invention, as well as the manner of achieving them may be understood by reference to the accompanying drawing which diagrammatically illustrates a preferred embodiment of apparatus of this invention. In this drawing, the apparatus is shown in cross-sectional elevational detail.

Referring to the drawing, the apparatus illustrated is generally of a cylindrical shape. Thus, essentially each of the elements of the apparatus as illustrated constitutes the elevational cross-section of a cylinder. The numeral 1 generally designates the infra-red radiation source. While the source may be of any nature adapted to provide a constant intensity of radiation over a comparatively large emitting surface, the source is preferably of the nature illustrated. Thus, the source may simply consist of a closed steel cylinder 2 of which the upper circular end 3 constitutes an infra-red emitting plate. Consequently, it is to be understood that at least the plate 3 and, if desired, the entire cylinder 2 is to be composed of a material capable of emitting infra-red radiation when heated. Steel is a suitable material of construction to achieve this object.

The emitting plate 3 must be heated in a suitable manner in order to secure infra-red radiation. It is simply requisite for a heat exchange medium or heating means to be applied to plate

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3. A convenient and effective manner of achieving this is to at least partially fill the closed cylinder 2 with a heat exchange fluid 4 such as diphenyl and or diphenyl oxide, alkyl naphthalene, or mercury. It is particularly preferred that a medium be used which is capable of vaporizing at high temperatures. By this means condensation of vapors on plate 3 will cause emission of infra-red energy. A heating coil 5 may be immersed in the liquid heat exchange medium to heat the medium to its boiling point. An electrical heating coil may be used for this purpose so that the heat exchange medium can be heated to a suitable temperature by the application of voltage across the electrical terminals 6. While various means may be used to control the temperature of a non-conducting liquid, a simple manner of achieving this is to employ a bi-metallic temperature control 7 located in the liquid heat exchange medium capable of opening or closing the heating circuit in order to maintain a desired temperature. Alternatively the temperature can be controlled by using a pressure sensitive control element positioned in the vapor space of the cylinder 2 or other means may be used. It is apparent that air or other foreign substances should be excluded from the cylinder at the time the heat exchange medium, such as mercury, is sealed in the cylinder.

By heating the liquid 4 to its boiling point, the emitting plate 3 of the cylinder will radiate infra-red energy over its entire area at a constant radiation output over each unit of its area. This infra-red radiation will travel upwardly from the emitting plate 3.

In order to use this radiant infra-red energy for analysis purposes a plurality of infra-red transparent fluid cells or infra-red filters are positioned above the emitting plate 3. In addition a sample cell and a suitable infra-red detector are placed in line with the cells or filters above the emitting plate 3. In accordance with this invention radiation from plate 3 may be considered as being divided into two beams of radiation. A first beam of radiation is that portion of the infra-red energy concentrated in a central area above the emitting plate. A second beam of infra-red radiation is an annular beam surrounding the first beam. As will be seen, the filter cell partitions and detector cell partitions are arranged to define these two beams so that the cross-sectional area of each of the beams is substantially the same.

In order to achieve this, a cylindrical casing 8 which is closed at the upper end may be placed over the emitting plate 3. Infra-red transparent windows may be sealed to or grooved into the casing 8 at a plurality of vertically displaced points to define the different cells required. It is preferred that at least four such windows be supplied, 9, 10, 11 and 12, to divide the interior of casing 8 into five cells. As will be seen each of these five cells may be considered to be further subdivided into an interior circular cell and an exterior annular cell. Thus, cylindrical partitions 13 and 14 may be centrally positioned within the casing 8 so as to divide at least two of the five cells into the interior and exterior cells as described. It is important that the cross-sectional area of the space within the cylindrical partitions be substantially equal to the cross-sectional area of the annulus between the partitions and the casing 8. It is essential that the partition 13 form a fluid-tight cell with the infra-red windows 12 and 11 so as to provide a fluid-

tight interior cell 15 and so as to provide a fluid-tight exterior cell 16. Similarly, it is essential that the partition 14 be in fluid-tight relation with the infra-red window 12. Similar partitions may be provided in each of the other cells provided they be perforated so that the same fluid composition can be maintained in the interior and exterior cells between the windows 9 and 10, and between the window 9 and emitting plate 3. Preferably however, the partitions may be omitted as indicated in the case of the cells positioned between the emitting plate 3 and the window 9 and between windows 9 and 10 and 11 and 10. By virtue of this construction, it may be seen that rays passing vertically upward from the emitting plate 3 will consist of a central beam passing within the limits of the interior area of the vertical partitions and will have an exterior beam passing through the annular space between the vertical partitions and the case 8.

The cell 17 formed between the emitting plate 3, the window 9 and the case 8 may be evacuated or may contain an infra-red non-absorbing gas. Actually, if desired, the cell 17 may be open to air. The purpose of this cell is to provide space in which to position a radiation intensity control capable of controlling the ratio of energy in the two indicated beams. For this purpose a suitable shutter arrangement may be used or a simple opaque plate 18 supported in a variable position in the cell 17 may be used. By moving the plate 18 inwardly or outwardly, it is possible to vary the ratio of infra-red energy in the two beams.

As heretofore described, therefore, the windows 9, 10, 11 and 12, and the vertical partitions 13 and 14, define a plurality of cellular spaces within the housing 8. Cell 17 directly above the emitting plate 3 is intended to accommodate the radiation intensity control 18. Cell 19 above cell 17, and between windows 9 and 10, is intended to contain a sample of the fluid to be tested. Above cell 19 is cell 20, limited by the windows 11 and 10. Above cell 20, two cells 15 and 16 are provided so that cell 15 will define, and be exposed to the central beam of radiation coming from the emitting plate 3, and so that cell 16 will define and be exposed to the annular beam of radiation coming from plate 3. Immediately above cells 15 and 16, two cells 21 and 22 will be positioned, again defined by vertical partition 14 of the same diameter as the partition 13. Consequently, cell 21 again will be exposed only to the radiation of the central infra-red beam, while cell 22 will be exposed to the radiation of the annular infra-red beam. For the sake of simplicity, the fluid inlets and outlets to these cells have not been shown in the drawing. It is of course to be understood that suitable ducts are provided through the casing 8 to permit the introduction and removal of fluids to each of the cells identified.

To use terminology ordinarily applied to infra-red analytical apparatus, the cell 17 may be called a light trimmer compartment; the cell 19 may be called a sample cell; the cell 15 or 16 may be called a filter cell; the cell 16 or 15 may be called a compensator cell, and the cell 20 may be called an interference cell. In employing the apparatus for an analysis, the sample cell 19 may then be filled with a gas such as XAB, where X is a constituent which is to be quantitatively determined. For continuous plant application a sample of XAB may be continuously passed through the sample cell 19 so that any variation in the composition of this gas may be continuously de-

terminated. The interference cell 20 including both the interior and exterior cellular spaces may, if desired, be left empty, may be filled with air, or preferably may be filled with a selectively absorbent infra-red gas such as a dilute composition of AB. The compensator cell 16, depending upon the particular analysis will be filled with a selectively absorbent infra-red gas or may be filled with a non-absorbent infra-red gas. Thus, in some analyses the compensator cell may simply contain air or the compensator cell may contain varying compositions of A or B or A and B or other constituents. The filter cell 15 is to be filled with the constituent to be determined or X. No further description will be made of the fluids to be placed in the indicated cells as these fluids are those generally employed in conventional infra-red analytical methods employing the dual beam method of infra-red analysis.

It is to be understood, therefore, that as the infra-red radiation passes upwardly from emitting plate 3 through the cells 17, 19, 15, 16, and 20, characteristic infra-red absorption will occur so that the infra-red radiation passing the uppermost window 12 may be used to characterize the nature of the sample contained in cell 19. Thus, by virtue of the fact that a central beam of infra-red radiation passes through the central portion of the cells of the apparatus which may contain and ordinarily would contain infra-red absorbing constituents different from those contained in the portion of the cells through which the exterior beam passes, different intensities of infra-red radiation will pass through the central portion of window 12 and the annular portion of window 12. Consequently, by determining the intensity of infra-red radiation passing through the cross-sectional area of window 12 within the cylindrical partition 14 and by determining the intensity of infra-red radiation passing through the cross-sectional annular area of the window 12 between the cylindrical partition 14 and the case 3, analytical results may be obtained.

In accordance with this invention the desired determination of infra-red energy passing through the window 12 is made by employing a novel type of infra-red detector. The detector is characterized by a relatively large area substantially equal to that of the cells in which the detector is placed. Thus, a circular detecting element 23 is supported within the interior cell above the window 12, while an annular detecting element 24 is positioned in the annular space above the window 12. Alternatively elements 23 and 24 may be conical, providing a greater effective area. The detecting elements 23 and 24 are thin sheets of infra-red absorbing material such as blackened metal foil, oiled paper, or any other material capable of absorbing infra-red radiation. The detecting elements are so supported that a gas tight seal is not made with the supporting walls. At the present time it is contemplated that a preferred detecting element is aluminum foil having a thickness less than about 1 thousandth of an inch coated with a black material such as carbon black. It is critical that the detecting elements 23 and 24 should have an extremely low heat capacity. The reason for this critical limitation will be brought out hereafter. Another element of the detecting device of this invention is a diaphragm 25 fixed to the upper termination of the vertical partition 14 at some distance below the closure of the casing 8. This diaphragm may be composed of any suitable material capable of flexing in response to variations

in pressure on either side of the diaphragm. A thin metallic diaphragm may be used for this purpose. It will be seen that the diaphragm 25 has the effect of sealing off a central cylindrical volume or cell 21 bounded by the diaphragm, the partition 14 and the window 12. Further, the diaphragm forms a second fluid-tight volume or cell 22 bounded by the annular area of window 12, the external portion of partition 14, the casing 8 and the upper portion of diaphragm 25. These two fluid-tight compartments will be designated as the interior and exterior cells of the detector. Both of these cells are to be filled with a gas which does not selectively absorb infra-red radiation. This gas can therefore be air, nitrogen, or any other non-absorbing gas or may be any non-selectively absorbent gas. Each of the detector cells is preferably filled with the gas at substantially the same pressure.

The result of the indicated detector construction is that a pressure differential will be produced in the two detector cells whenever a different quantity of infra-red radiation passes to the interior and exterior detector cells. This effect occurs since infra-red radiation passing the window 12 will fall upon the absorbing detecting elements 23 and 24 so as to heat these elements. By virtue of the low heat capacity of these elements this heat will immediately be conducted to the gas filling the detector cells, heating the gas and increasing the pressure of the gas in the cells. In the case in which equal radiation passes the window 12 and passes to each of the detecting cells, the gas in these cells will be proportionately heated so that no pressure differential will exist across the diaphragm 25. However, in the case in which more radiation reaches the interior cell, the pressure within this cell will exceed the pressure in the exterior cell so that the diaphragm 25 will be moved upwardly.

It should be stressed that the nature of the detecting elements 23 and 24 must be critically chosen to achieve these results however. By way of example, it has been found that suitable results are not obtained in the case in which blackened aluminum foil having a thickness of about 8 thousandths of an inch is employed. Apparently, the heat capacity of foil of this thickness is so great that heat transmission to the vertical partition 14 can occur at a sufficiently rapid rate so that the desired pressure differential cannot be set up.

Consequently, as brought out, the apparatus described is capable of moving the diaphragm 23 in response to variations in the amount of infra-red radiation passing through the interior and exterior portions of window 12. By fixing a rod 30 to the diaphragm, movement of this diaphragm may be detected and used to indicate changes in the nature of the sample contained in the sample cell of the apparatus. Thus, the plunger 30 may terminate in an iron armature 31 positioned centrally in a transformer arrangement consisting of the two secondary coils 32 and 33 and the primary coil 34. Consequently, movements of the diaphragm 23 will move the armature 31 upwardly or downwardly, varying the output of the secondary coils 32 and 33. Again, since the inductance transmitters of this nature are well known to the art no further description of this element of the invention will be made.

The pressure differential in the cells 22 and 21 may alternatively be determined in different manners. For example, a conventional null

balance differential pressure cell may be connected to the two cells. In this case diaphragm 25 may be rigid.

As described, therefore, the novel apparatus of this invention comprises a flat emitting plate capable of emitting a constant intensity of infra-red radiation over the entire area of the plate. A plurality of fluid cells are stacked above this plate in infra-red transparent relation so as to divide the radiation leaving the plate into a central circular beam and an external annular beam. After transmission through these cells the two beams are caused to fall upon detecting elements having a total area substantially equal to the area of the emitting plate. One detecting element is positioned in a central cell while the second detecting element is placed in an external cell so as to be positioned in the interior and exterior beams of radiation from the emitting plate. The two cells are divided by a common diaphragm so that pressure differentials set up in the cells by differential infra-red absorption by the detecting elements will cause a movement of the diaphragm. This diaphragm movement may be translated to an electrical signal by coupling the moving diaphragm to an inductance transmitter.

As described, the apparatus of this invention consists in employing a novel infra-red radiation source, and a novel infra-red detecting device in combination with a sample cell and absorption cells, or filters. While the invention has been described with particular reference to the type of infra-red apparatus employing absorption cells, it should particularly be noted that the invention can equally be applied to the type of apparatus employing selective infra-red filters to perform the same function as the absorption cells.

While the principal advantages of the apparatus described are more or less apparent, particular attention may be called to one advantage. This advantage may be said to be the lack of temperature sensitivity possessed by the apparatus. In conventional infra-red analytical apparatus, the infra-red detectors employed are generally sensitive to changes in ambient temperature. This effect is minimized, or eliminated by the apparatus of this invention. Thus, referring to the

detecting elements of the apparatus shown in the drawing, it will be noted that should ambient temperatures change outside of the housing of the apparatus, such a change would affect each of the two detecting elements proportionately. Thus, an increase in ambient temperatures would raise both the external annular detector pressure, and the internal central detector pressure so that by proper choice of design requirements, no differential change in pressure will result.

What is claimed is:

1. Infra-red analytical apparatus comprising an infra-red emitting plate, a series of fluid cells including infra-red transparent windows parallel to and adjacent the emitting plate, said cells being arranged so as to divide the radiation passing from said plate into a central beam and an annular beam substantially equal in cross-sectional area and an infra-red detector positioned in both the said central and annular beams of infra-red energy emitted from said plate, and passing through said cells.

2. The apparatus of claim 1 in which the said detector comprises two cells having a common diaphragm, each of said cells containing a thin infra-red absorbing element having an area not less than substantially that of the cross-sectional area of the beam of radiation reaching the cell, and each of said cells being filled with a gas, whereby differential radiation impinging on the said detecting elements causes the movement of said diaphragm.

3. The apparatus of claim 1 in which the said emitting plate comprises the closed end of a cylinder at least partially filled with a vaporized liquid maintained at a temperature to cause infra-red emission from the said plate.

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