

supply an inert gas thereto and outlet means 14 to remove mixtures of the pollutant gas and the inert gas from the permeator. As is shown in FIG. 1, the apparatus also includes a casing 16 adapted to receive the permeator body 10. The casing 16 is provided with heating means 18, which are preferably electrical resistance heaters operatively connected to a control box 20 which in turn is operatively connected to a suitable power source 22. The control box 20 is preferably adjustable by means of a control 24 to control the power supplied to the heating means 18 and thereby facilitate temperature control. As will be discussed more fully hereinafter, the heating means 18 are positioned to heat the inert gas supplied to the permeator body.

As is shown in FIG. 1, the casing 16 is formed by a sleeve 34 which is surrounded by insulating material 36 in which the heating means 18 is embedded.

The inert gas for the system may conveniently be supplied by a tank 26 which is equipped with a valve 28, a pressure reducing valve 30 and, optionally, a pressure gauge 32, all of which are operatively connected to the inlet means 12 to supply the inert gas thereto.

The details of the permeator body and associated casing are more clearly illustrated in FIG. 2 of the drawing. As can be seen from this figure, the permeator body is formed of a flange element 38 integral with a barrel portion 40, with the barrel portion 40 being receivable in the sleeve 34 of the casing 16. The flange portion 38 is provided with an opening 42 adapted to receive the means 12 for supplying the inert gas to the permeator body. As illustrated in FIG. 2, the means 12 is preferably formed of a nipple portion 44, terminating in a flow constrictor 46 most preferably in the form of a small diameter needle, facilitating constant flow of the inert gas through nipple 44 into the opening 42. The small orifice of the needle also provides more accurate temperature control as the inert gas expands and is cooled on passage therethrough.

As is also shown in FIG. 2, the barrel portion 40 is provided adjacent the flange portion 38 with means 48 to secure the permeator body 10 in the sleeve 34. It is generally preferred that the barrel 40 be provided with thread means 48 whereby the upper portion of the barrel 40 is adapted to be threadedly engaged with the corresponding upper portion of the sleeve 34. The wall is also provided with a continuous groove 50 on the peripheral face thereof whereby the groove 50 defines along with the interior face 52 of the sleeve 34 a passage having a high length to cross-sectional area ratio. As is illustrated in FIGS. 2 and 4, the continuous groove 50 can most simply be formed by providing a continuous thread about the periphery of the barrel 40 whereby the path defined by the groove 50 and the interface 52 of the sleeve 34 is a continuous helix extending over the length of the barrel.

The barrel also defines an opening 54 between the inlet opening 42 and the continuous groove 50 whereby the opening 42 communicates with the continuous groove 50 to permit gas supplied to the inlet means to be passed through the needle 46 into the opening 42 and through the opening 54 to the continuous groove 50.

As will be appreciated by those skilled in the art, a number of different types of passages can be defined by the barrel portion 40 of the permeator body. It is necessary only that the passage defined by the groove 50 be of a sufficient length to permit the inert gas passed

therethrough to reach thermal equilibrium with the barrel portion 40 of the permeator body 10. In other words, the continuous groove 50 should provide a high surface area to facilitate heat transfer between the barrel 40 and the inert gas supplied thereto.

The barrel 40 of the permeator body 10 is dimensioned such that the barrel does not completely extend to the base 56 of the sleeve 34 whereby the base 58 of the barrel 40 and the base 56 of the sleeve 34 define a small opening 60 communicating with the lower portion of the continuous groove 50. In this way, the inert gas which is passed through the continuous groove 50 exits from the groove 50 into the space 60.

The permeator body 10 also defines a central opening 62 which extends over the entire length of the barrel 40 and the flange 38. At its lower portion, the central opening communicates with the space 60 at the base of the barrel whereby inert gas supplied to the space 60 is passed upwardly through the central opening 62. The central opening 62 is adapted to contain a pollutant gas permeable container or capsule 64 which can be mounted in the central opening 62 by means of a clip 66. Thus, the inert gas which is caused to pass through the continuous groove 50 into the opening 60 at the base of the barrel and then upwardly through the central opening is admixed with the pollutant gas which continuously diffuses through the walls of the capsule 64. The capsule 64 is preferably formed of a material which is permeable to the pollutant gas to permit the pollutant gas to diffuse through the walls of the capsule 64 at a slow, controlled rate. Such capsules are known in the art and are preferably formed of polymers of tetrafluoroethylene of the type referred to herein above.

The upper portion 68 of the central opening 62 defined by the flange 38 is preferably formed with thread means to threadingly engage outlet means 14 for the removal of pollutant gas admixed with the inert gas. As illustrated in FIG. 2, the outlet means 14 can be in the form of a rotometer formed of a body portion 70 having a central passage 72 formed of a tapered bore. A flow indicator such as a ball 74 can be positioned in the tapered bore to indicate the rate of flow of the mixture of the inert gas passing upwardly from the central opening 62 into the central bore 72. The body portion 70 of the rotometer preferably terminates in a nipple 76 for the attachment of suitable connecting means to pass the mixture of the SO_2 and the inert gas to the instrument to be calibrated.

The flange portion 38 of the permeator body is preferably provided with a shallow cavity 78 adapted to threadedly engage a colorimeter 80 which in turn is adapted to receive a thermometer 82 or like temperature measuring means to enable the temperature of the permeator body to be ascertained.

In an alternative embodiment of the invention, as shown in FIG. 5, the outlet means 14 can be provided with a T-fitting 84 having a filter 86 mounted on one branch thereof which operates to admit to the T-member ambient gas, usually air, which has been purified by way of the filter 86 to remove any significant amount of pollutant gas which may be present in the ambient air. In this embodiment, the mixture of the pollutant gas and the inert gas is withdrawn from the other branch 88 of the T-fitting 84 by passage to the instrument to be calibrated. This variation is particularly useful in the calibration of instruments in which the rate

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GAS PERMEATOR

This invention relates to a method and apparatus for use in the calibration of analytical instruments, and more particularly to an improved method and apparatus for use in the calibration of gas analytical instruments.

A number of analytical instruments for use in the detection and/or analysis of SO_2 , NO , NO_2 , etc. containing gas streams are well known to those skilled in the art. Such instruments can be based on colorimetry as described in my earlier U.S. Pat. No. 3,617,136. However, as is described in my copending application Ser. No. 172,133, filed Aug. 16, 1971, it has recently been discovered that chemiluminescence can be used as a basis for analysis of gas streams. Regardless of the mechanism by which the analysis is carried out, instruments for use in the measurement of such gases must, at some point in time, be calibrated, preferably using as a standard a gas stream containing a known quantity of pollutant gas admixed with a gas which is inert to the analytical system of the instrument.

One mode for calibrating analytical instruments involves the use of a closed tube formed of Teflon (i.e., polymers of tetrafluoroethylene) containing SO_2 , NO_2 , H_2S , butane, etc. The gas diffuses throughout the Teflon forming the tube at a fairly constant, known rate usually of the order of a few micrograms per minute. The pollutant gas which diffuses through the wall of the tube is admixed with nitrogen, air which is free from the pollutant gas, or any other inert gas, and the resulting mixture is supplied to an analytical instrument as a standard to permit the instrument to be calibrated.

One of the primary difficulties in the use of such permeable tube in the calibration of analytical instruments as described is that the rate of diffusion of the gas through the Teflon is quite sensitive to temperature, with the rate of diffusion being significantly higher at elevated temperatures. Thus, the accuracy of the calibration depends in large measure on the ambient temperature.

It has also been found that optimum rates of diffusion of such gases through the Teflon are achieved when the temperature of the system is maintained at about $31^\circ\text{--}32^\circ\text{C}$. As will be appreciated by those skilled in the art, it is quite difficult to maintain the permeator system at such temperatures, with precise temperature control within normal limits.

It is accordingly an object of the present invention to provide a method and apparatus for supplying a known quantity of pollutant gas admixed with an inert gas for use in the calibration of instruments, using gas permeator which overcomes the foregoing disadvantages and which is characterized by more uniform rates of diffusion.

It is another object of the present invention to provide a method and apparatus for supplying a pollutant gas admixed with an inert gas for use in the calibration of analytical instruments which is completely portable and can be used under varying ambient temperature conditions and yet provide a uniform mixture of the pollutant and an inert gas.

It is a more specific object of the present invention to provide a method and apparatus for supplying a pollutant gas admixed with an inert gas for use in the calibration of instruments in which a gas permeator can be maintained in a state of thermal equilibrium, independent

of ambient temperatures, and thereby at a constant temperature.

These and other objects and advantages of the invention will appear more fully hereinafter and, for purposes of illustration but not of limitation, an embodiment is shown in the accompanying drawing in which:

FIG. 1 is a schematic illustration of apparatus embodying the concepts of this invention;

FIG. 2 is a detailed view of a permeator employed in the practice of this invention;

FIG. 3 is a sectional view of the permeator of FIG. 2 taken along the line 3—3 in FIG. 2;

FIG. 4 is a detailed view of the flow path of the permeator of FIG. 2;

FIG. 5 is a cross-sectional view of an alternative outlet assembly for the permeator shown in FIGS. 1 to 4.

The concepts of the present invention reside in a method and apparatus for providing a supply of a pollutant gas in an inert gas which can be used in the calibration of gas analytical instruments in which an inert gas is passed through a path defined by a permeator body having a high ratio of length to cross sectional area to permit the inert gas to reach thermal equilibrium with the permeator body. The inert gas is then passed through a passage in the permeator body for contact with a container which is permeable to the pollutant gas and which contains the pollutant gas whereby the pollutant gas slowly diffuses through the container for admixture with the inert gas to provide a mixture of the pollutant gas and the inert gas. Because the permeator body is allowed to reach thermal equilibrium, the diffusion of the pollutant gas which occurs within the permeator body takes place at a temperature substantially independent of the ambient temperature to provide a source of pollutant gas and an inert gas in which the pollutant has is present in controlled amounts.

The temperature at which the pollutant gas diffuses through the container can be simply controlled by heating the inert gas as it is passed through the passage whereby the inert gas serves to heat the permeator body.

As used herein, the term "inert gas" is intended to refer to and include any gas which is inert to the analytical system of the instrument to be calibrated. For example, where the instrument is a colorimeter of the type described in U.S. Pat. No. 3,617,136, the inert gas should be one which is inert to pararosaniline which is reactive with a sulfide ion for the development of color. A number of inert gases can be used in the practice of this invention and include nitrogen, helium, argon, air, so long as it is free from even trace amounts of the pollutant gas, as well as numerous others.

As used herein, the term "pollutant gas" is intended to refer to and include any gas to which the analytical system of an instrument to be calibrated is sensitive and which is capable of diffusing through a gas in permeable material at a slow, controlled rate. Such gases and gas permeable materials are themselves well known to the art, and include SO_2 , NO_2 , H_2S , hydrocarbons such as butane, etc.

Referring now to the drawings for a more detailed description of a preferred embodiment of this invention, there is shown in FIG. 1 a permeator system which includes a permeator body 10 having inlet means 12 to

of gas flow through the analytical instrument is greater than the output of the mixture of the inert gas and the pollutant has from the permeator of this invention. For example, the calimeter described in my earlier U.S. Pat. No. 3,617,136, is usually connected to a source of vacuum to draw a gas to be subjected to analysis through the colorimeter. Wherein the flow rate of the gas steam to be analyzed through such colorimeter is greater than the output of the permeator, the vacuum applied to the colorimeter simply serves to exhaust from the permeator of this invention through branch 88 not only the mixture of the inert gas and the pollutant gas but also air of the light ambient gas which is drawn through the filter 86 into the T-fitting 84.

In the preferred apparatus of this invention, the permeator body 10 is formed of a heat conductive material, and preferably a material such as aluminum, steel or the like. When the material forming the permeator body is not completely inert to the pollutant gas, it is frequently desirable to provide the interior face 90 with a coating 92 which is inert to the pollutant gas to prevent or substantially minimize reaction of the pollutant gas, which diffuses from the capsule 64 into the central opening 62, with the material forming the permeator body 10. The coating 92, as illustrated in FIG. 3 of the drawing, can simply be a plastic film, such as Teflon or the like.

In the operation of the apparatus of this invention, an inert gas is supplied to the inlet means 12 by the tank 26, and the inert gas flows through the needle 44 and the small diameter needle 46 into the opening 42. Depending somewhat upon the procedure employed, there is some expansion and hence cooling of the inert gas as it emerges from the needle 46, although such expansion is not necessary in the practice of this invention. The inert gas supplied to the opening 42 through the needle 46 is then passed through the opening 54 into the continuous groove 50 whereby the inert gas in continuously passed through the passage defined by the groove 50 over the length of the barrel 40 to establish or at least approach thermal equilibrium with the barrel 40. The inert gas emerges from the passage defined by the groove at the base of the barrel 40 and is caused to flow into the space 60 and upwardly through the central opening 62 where the inert gas is admixed with the pollutant gas which diffuses from the capsule 64 into the central opening 62. The pollutant gas which has diffused through the capsule 64 is thus swept upwardly through the central opening 62 and through the outlet means 14 for passage to the instrument to be calibrated. As will be appreciated by those skilled in the art, since thermal equilibrium is established between the inert gas supplied to the passage defined by the continuous groove and the barrel 40 of the permeator body, the capsule 64, which is displaced in the central opening 62 of the barrel 40, is maintained at the equilibrium temperature thereby established. Consequently, the pollutant gas diffuses through the walls forming the container 64 at a constant rate determined by the equilibrium temperature established.

As is illustrated in FIG. 1, the heating elements 18 are positioned to supply heat to the inert gas flowing through the continuous groove 50 to thereby enable the temperature of the inert gas and consequently the equilibrium temperature of the permeator body 10 to be adjusted at the desired level. As is shown in FIG. 1, the heating elements 18 are positioned adjacent to and

surrounding the sleeve 34 although it will be understood by those skilled in the art that the heating means may be integrated with the sleeve 34 or positioned elsewhere so as to supply heat to the inert gas flowing through the passage defined by the continuous groove 50. In this way, the temperature to which the inert gas is heated and hence the equilibrium temperature of the system, can be selected without regard to the ambient temperature.

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

I claim:

1. A permeator assembly to supply a source of a pollutant gas admixed with an inert gas comprising a permeator body including a barrel portion having an elongate groove about the periphery thereof, a casing adapted to receive the barrel portion and having an inner face, with the inner face and the casing groove defining a passage having a high ratio of length to cross sectional area, means to supply an inert gas to said passage communicating with said passage, a centrally disposed opening extending through the body, said opening communicating with said passage opposite the means to supply an inert gas and adapted to receive a pollutant gas permeable container in said opening to diffuse pollutant gas into said opening for admixture with inert gas supplied through said passage to said opening whereby the inert gas supplied to said passage reaches thermal equilibrium with the body and is then passed into said opening to form a mixture of pollutant gas and the inert gas, means to remove the mixture from said opening and means to heat the inert gas supplied to said passage.

2. An assembly as defined by claim 1 wherein the means to supply an inert gas to said passage includes a small diameter orifice.

3. An assembly as defined by claim 1 wherein said passage is a helical passage having a length sufficient to permit the inert gas supplied thereto to reach thermal equilibrium with the permeator body.

4. An assembly as defined by claim 3 wherein the casing includes a sleeve portion adapted to receive the barrel portion of the permeator body and temperature insulating means surrounding the sleeve portion.

5. An assembly as defined by claim 4 wherein the heating means are positioned adjacent to the sleeve portion.

6. An assembly as defined by claim 1 wherein the central opening contains a thin film of a material to prevent reaction of the pollutant gas with the material forming the permeator body.

7. An assembly as defined by claim 5 wherein the barrel portion and the casing define a chamber opposite the means to supply gas to said passage whereby the central opening communicates with said passage through said chamber.

8. An assembly as defined by claim 1 wherein the means to remove said mixture from the central opening includes means to measure the flow rate of said mixture therethrough.

9. An assembly as defined by claim 1 which includes means for measuring the temperature of the permeator body.

10. An assembly as defined by claim 1 wherein the means to heat the inert gas is adjusted to control the temperature of the permeator body.

11. An assembly as defined in claim 1 wherein the means to remove said mixture from the central opening includes filter means adapted to admit ambient gas to the stream of said mixture removed from the means to remove said mixture from the central opening.

12. A permeator comprising a body including a barrel portion, an elongate groove about the barrel portion to define a passage having a high ratio of length to cross sectional area, means to supply an inert gas to said passage communicating with said passage, a centrally disposed opening extending through the body, said opening communicating with said passage opposite the means to supply an inert gas and adapted to receive a pollutant gas permeable container in said opening to diffuse pollutant gas into said opening for admixture with inert gas supplied through said passage to said opening whereby the inert gas supplied to said passage reaches thermal equilibrium with the body and is then passed into said opening to form a mixture of pollutant gas and the inert gas, and means to remove the mixture from said opening.

13. A permeator as defined in claim 12 wherein the means to supply an inert gas to said passage includes a

small diameter orifice.

14. A permeator as defined in claim 12 wherein the permeator body includes a flange portion and a barrel portion, with the passage defined by the periphery of the barrel portion.

15. A permeator as defined in claim 13 which includes a continuous groove about the periphery of the barrel portion.

16. A permeator as defined in claim 16 wherein the groove is a helical groove extending over substantially the entire length of the barrel portion.

17. A permeator as defined by claim 12 wherein the central opening contains a thin film of a material to prevent reaction of the pollutant gas with the material forming the permeator body.

18. A permeator as defined by claim 12 wherein the means to remove said mixture from the central opening includes means to measure the flow rate of said mixture therethrough.

19. A permeator as defined by claim 12 wherein the means to remove said mixture from the central opening includes a filter means adapted to admit ambient gas to the stream of said mixture removed from the means to remove said mixture from the central opening.

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