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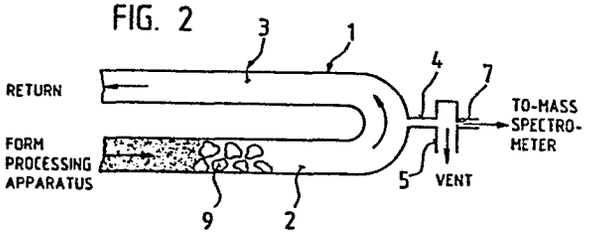
(54) Analytical apparatus.

(57) The instant invention relates to a mass spectometer having an inlet leak and a flow-by sampling arrangement. In accordance with the invention, aerosols and solid impurities are removed from the sample by packing a portion of the flow-by tubing prior to the inlet capillaries or the inlet leak of the mass spectrometer with a packing material coated with a suitable nonvolatile liquid. The packing must have the spaces between the particles thereof small enough to remove fine particles while still permitting adequate flow. With this apparatus the solids are collected by simple filtration through the packing material while the aerosols flowing into the packing are partitioned into the nonvolatile liquid coating the particles and are released at a later time into the sample stream as a vapor or gas.

FIG. 2



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ANALYTICAL APPARATUS

This invention relates to a method and apparatus for use in the analysis of gases and vaporized substances by means of a mass spectrometer. The mass spectrometer is a well known apparatus for on-line analysis and control of processing equipment in the chemical and other process industries. Such an apparatus is described in U. S. Patent 3,648,047. The samples from the processing equipment may be taken from multiple points or locations employing a flow-by-inlet system. Flow within the sample system is established by a pressure head in the process and the unused portion of the sample may be returned to the process or a common sump. These are continuous flow type systems, no batch sampling technique is required. In the flow-by-sample systems where there are multiple sampling points, flow is maintained in all sample lines even though an analysis is taking place from only one line. In the case of multiple sampling, multiple valves, which may be automatically controlled, are employed whereby there may be successive analysis from each flow tube or conduit. In a flow-by-system the sample is obtained through an inlet leak into the mass spectrometer. These inlet leaks are generally a porous barrier type which may be made of suitable materials such as sintered metal.

The flow-by path from each sample point may suitably take the form of a U-shaped tube going from the sample point to the inlet for the mass spectrometer and returning back to the process stream or sump. The successful operation of such system is dependent on the cleanliness of the vaporized liquid or gas entering the analyzer. In many processes aerosols and/or solids are produced which are then present in the sample stream whereby they may plug either the capillary tubing employed to reduce sample pressure or plug the inlet leak to the mass spectrometer itself. If either of these problems occur, the analyzer must be shut down and the problem corrected. In a given conventional apparatus known to the inventor, it was necessary to shut down the apparatus every three to seven days.

Accordingly, it is a purpose of the instant invention to provide a means for preventing the plugging of the capillary and/or inlet leak for the mass spectrometer whereby the time period between shutdowns for cleaning may be increased substantially.

The above purposes of the instant invention are accomplished by packing a portion of the flow-by tubing, prior to the inlet capillaries or the inlet leak of the mass spectrometer with a packing material coated with a suitable

nonvolatile liquid. The packing must have the spaces between the particles thereof small enough to remove fine particles while still permitting adequate flow. With this apparatus the solids are collected by simple filtration through the packing material while the aerosols flowing into the packing are partitioned into the nonvolatile liquid coating the particles and are released at a later time into the sample stream as a vapor or gas.

With reference now more particularly to the drawings wherein corresponding parts in the various figures are represented by the same numbers.

Figure 1 is a diagrammatic representation in section of a coventional flow by mass spectrometer sampling tube of the prior art.

Figure 2 is a diagrammatic representation of the sampling tube of Figure 1 incorporating the features of this invention, and

Figure 3 is a schematic representation of a mass spectrometer and multiple sampling system incorporating the features of this invention.

Referring now more particularly to Figure 1 illustrating a conventional sampling loop or sampling tube, for sampling from a given point in a processing apparatus,

which comprises a tube 1 preferably about 3/32 to 1/2" internal diameter of material which is unaffected by the components normally present in the process streams being analyzed. It must also be physically and chemically stable under the processing conditions, i.e., temperature, pressure, etc. Typical materials would include stainless steel, glass lined metal tubing and polymeric tubing. Tube 1 comprises an inlet arm 2 and an outlet or return arm 3.

A small tube or capillary 4 of suitable material such as for tube 1 leads from the end of the loop or tube 1 to the mass spectrometer. A venting means such as a tube 5 of suitable material as previously described is provided between tube 4 and the mass spectrometer. Tube 5 leads to a roughing vacuum pump (not shown in figure 1). Just beyond tube 5 an inlet leak 7 is provided for leaking a small amount of gas to the mass spectrometer. The inlet leak 7 is generally of a porous material such as sintered glass or sintered metal.

In accordance with the prior art, as shown in figure 1, the gas samples passing through the tube 1 often contain aerosols and solids which flow through the tube and back to the process or sump and also through tube 4 to the inlet leak 7 whereby they have the effect of plugging the inlet leak 7.

In accordance with the instant invention, as shown in figure 2, particulate material coated with a nonvolatile liquid 9 is packed into the inlet arm 2 of sample loop or tube 1. Any packing or liquid support material could be used as the particulate material which is physically and chemically stable when exposed to the process stream. However, it must absorb the liquid in the range specified so as not to form droplets or pools of liquid. In a preferred embodiment the coated particulate material 9 consists of diatomaceous earth with a particle mesh size of from about 40 to 180 coated with a nonvolatile liquid such as a silicone-type liquid. The percentage by weight of the silicone liquid based on the total weight of the particulate material is about 3 to 15 percent. The packing must be air-sized to remove the fine particles having a mesh size greater than 180 which might also plug the sample capillary 4 or the inlet leak 7. Other suitable particulate materials include molecular sieves, charcoal, glass fibers, and open cell polymeric foams. Other suitable coating materials include any substance that is liquid at the operating temperature of the device and having a low vapor pressure such as polyethers, hydrocarbons and derivatives thereof.

The coated particulate material 9 is secured within the arm 2 of tube 1 by means of small wads of inert glass wool or other similar inert packing material (not

shown). The packing of the particulate material 9 into the arm 2 of tube 1 is done in such a way as not to form fine particles. This is accomplished by adding the packing to the tubing while gently tapping on the tubing with a 1/4" diameter wooden stick or pencil. This yields a packing of coated particulate material having uniform density and minimum fine particles. By employing the coated particulate material in the inlet arm 2 of tube 1 the aerosols and solids are removed. While the solids could be collected by simple filtration with conventional filters, aerosols would pass unchanged through filters. When employing coated particulate material 9, the aerosols pass into the packed material 9 and are partitioned into the non-volatile liquid coating the particles and are released at a later time into the sample stream as a vapor or gas.

A single tube or loop 1 may be connected to a mass spectrometer or a plurality of sample loops or tubes 1 may be connected through a suitable valving arrangement to a single mass spectrometer. For example, with reference more particularly to Figure 3, four such sample loops or tubes 1 are illustrated. In accordance with this embodiment of the invention, the tube or capillary 4 from each sample loop or tube 1 is connected through a valve 11 of any conventional design and another tube 13 to a tube or header 15. A suitable tube, capillary, etc., 17 is connected to the

header at one end and is provided with an inlet leak 7 therein beyond which it is connected to the mass spectrometer 19. A vacuum generally from about 25 to 100 TORR is maintained in the sample inlet system by provision of a roughing pump 21 connected to the vent tube 5. An ion pump 23 maintains a vacuum within the mass spectrometer apparatus of about 10^{-5} to 10^{-7} Torr. All of such apparatus, i.e., the mass spectrometer 19, ion pump 23, roughing pump 21, vent tube 5 and the arrangement therefor, are well known to those skilled in the art and are described in more detail in the manufacturers' literature. The valves 11 may be controlled to open in sequence thus providing samples from each source in turn, i.e., from each sample loop or tube 1 for a short period of time after which the one valve is closed and the next valve is opened, only one valve being open at a time generally speaking. This sequencing operation can be accomplished by any conventional control apparatus (not shown). In most cases the discharge from the roughing pump is returned to the process apparatus at or near the point where the sample is removed.

The embodiments of the invention in which an exclusive privilege or property is claimed are defined as follows:

1. A process analyzer apparatus comprising a sample conduit having an inlet connected to the sample point on the process apparatus and an outlet returning the sample to said process apparatus, a mass spectrometer, second conduit means connecting said sample conduit to said mass spectrometer, inlet leak means provided in said second conduit between said sample conduit and said mass spectrometer, a vent provided in said second conduit means substantially adjacent said inlet leak means and between said inlet leak means and said sample conduit means, packing means provided in said sample conduit means between said inlet and said second conduit means comprising particulate material of about 40 to 180 mesh coated with nonvolatile liquid.

2. The apparatus of claim 1 wherein the amount of said nonvolatile liquid is from about 3.0 to 20.0 percent by weight of the particulate material.

3. The apparatus of claim 2 wherein the pressure in the inlet to the mass spectrometer is maintained at about 25 to 100 TORR by means of a roughing pump pulling a vacuum through said vent and the pressure within the mass spec-

trometer is maintained at about 10^{-5} to 10^{-7} TORR by an ion pump.

4. The apparatus of claim 2 wherein the packing material is diatomaceous earth and said nonvolatile liquid coating material is a silicone.

5. The apparatus of claim 4 wherein said inlet leak is of sintered glass.

6. The apparatus of claim 4 wherein said inlet leak is of sintered metal.

7. The apparatus of claim 5 wherein the pressure in said mass spectrometer inlet is maintained at about 25 to 100 TORR by means of a roughing pump connected to said vent and the pressure within the mass spectrometer is maintained at about 10^{-5} to 10^{-7} TORR by an ion pump.

8. The apparatus of claim 6 wherein the pressure in said mass spectrometer inlet is maintained at about 25 to 100 TORR by means of a roughing pump connected to said vent and the pressure within the mass spectrometer is maintained at about 10^{-5} to 10^{-7} TORR by an ion pump.

9. The apparatus of claim 7 wherein multiple sample tubes are provided for sampling various points of a processing apparatus, valves are provided in said second

conduit means between said sample tubes and said mass spectrometer, said valves being connected to a header means with a single conduit from said header means to said vent, inlet leak and mass spectrometer, and means for sequentially opening and closing said valves.

10. The apparatus of claim 8 wherein multiple sample tubes are provided for sampling various points of a processing apparatus, valves are provided in said second conduit means between said sample tubes and said mass spectrometer, said valves being connected to a header means with a single conduit from said header means to said vent, inlet leak and mass spectrometer, and means for sequentially opening and closing said valves.

11. A process for analyzing a stream comprising a gas, vaporized liquid, and mixtures thereof containing aerosol impurities by passing said stream along a predetermined path through a conglomeration of irregular solid surfaces in close proximity to one another which surfaces are coated with a nonvolatile liquid, thereby removing said aerosols, and continuing to pass a major portion of said stream from said conglomeration of solid surfaces in a predetermined path back to the processing apparatus while passing a small second portion of said stream in a predetermined path to a porous surface, venting off a major portion

of said second portion of said stream prior to said porous surface and leaking a minor portion of said second portion into a mass spectometer through said porous surface.

12. The process of claim 11 wherein said vented portion is returned to said processing apparatus.

13. The process of claim 12 wherein the pressure at the inlet of said mass spectrometer is about 25 and 100 TORR the pressure within said mass spectrometer is about 10^{-5} to 10^{-7}.

Drawings

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FIG. 1

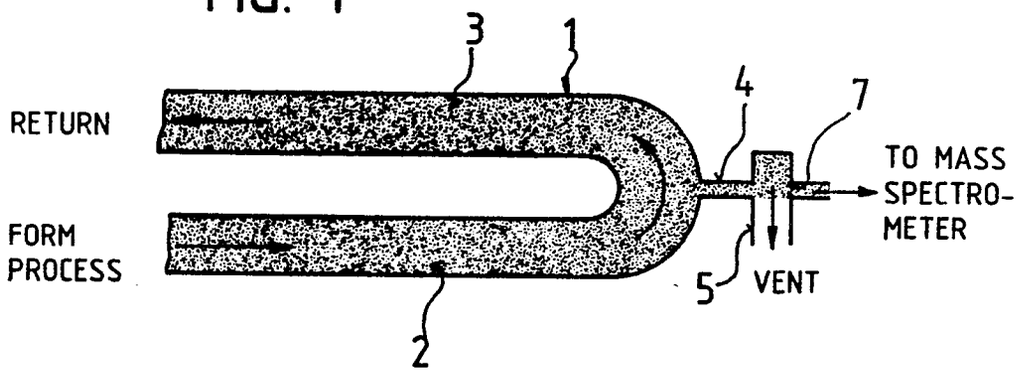

FIG. 2

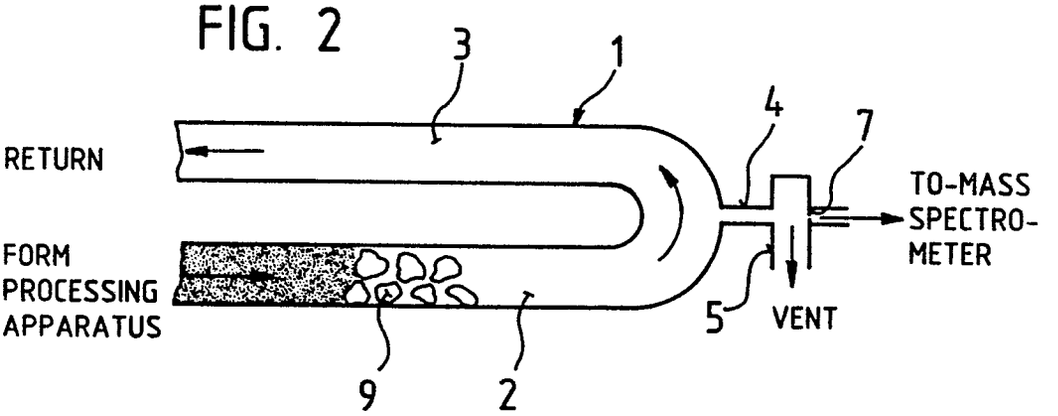

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FIG. 3