Dec. 25, 1956 J. R. BENAPFL 2,775,707

HEAT COMPENSATING DEVICE

Filed May 9, 1955 2 Sheets-Sheet 1

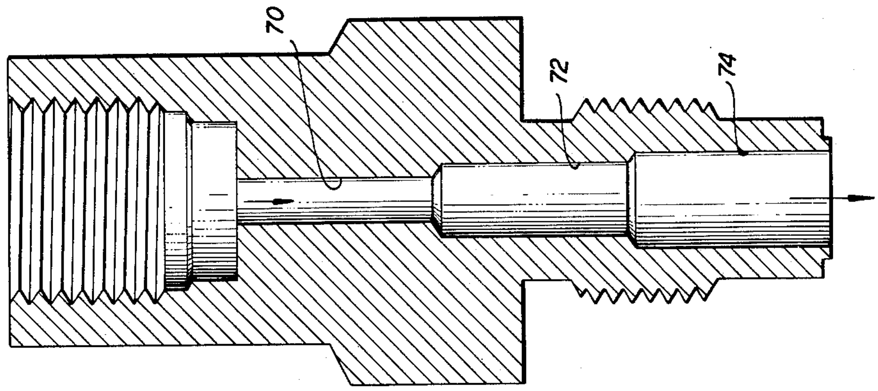

FIG. 4.

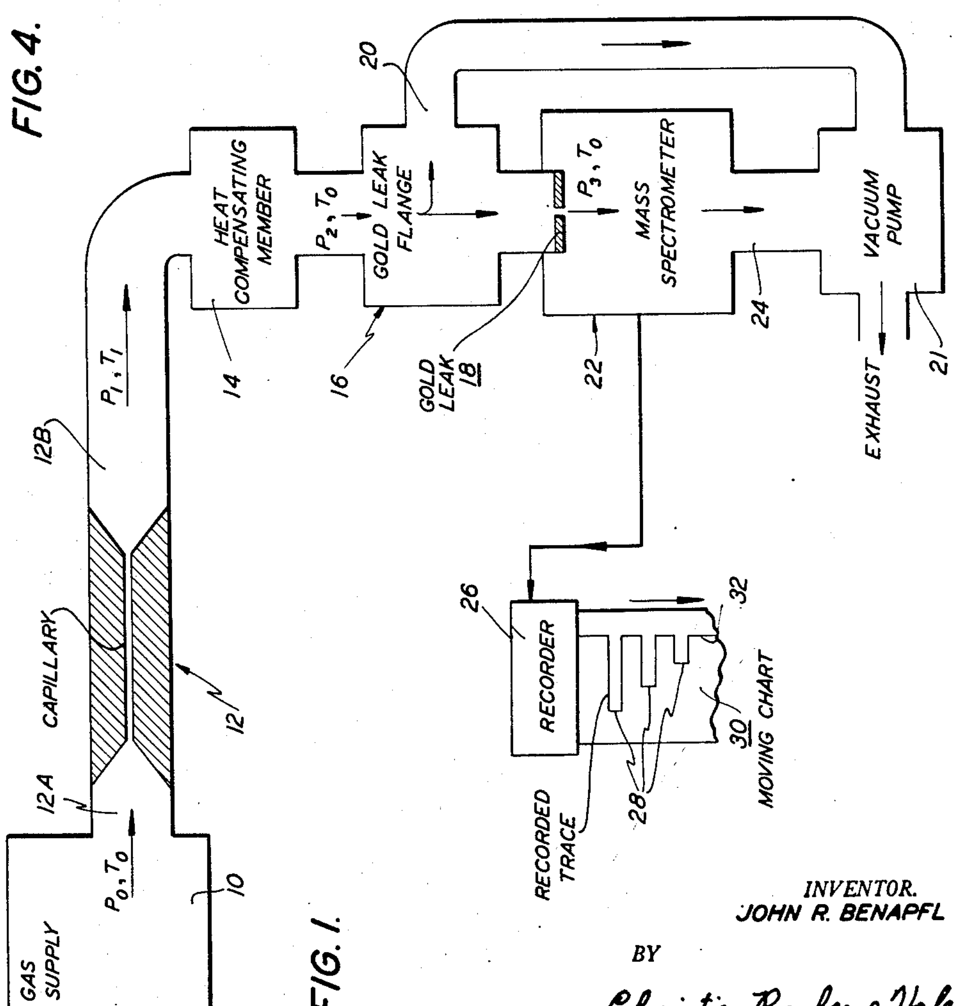

FIG. 1.

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FIG. 2.

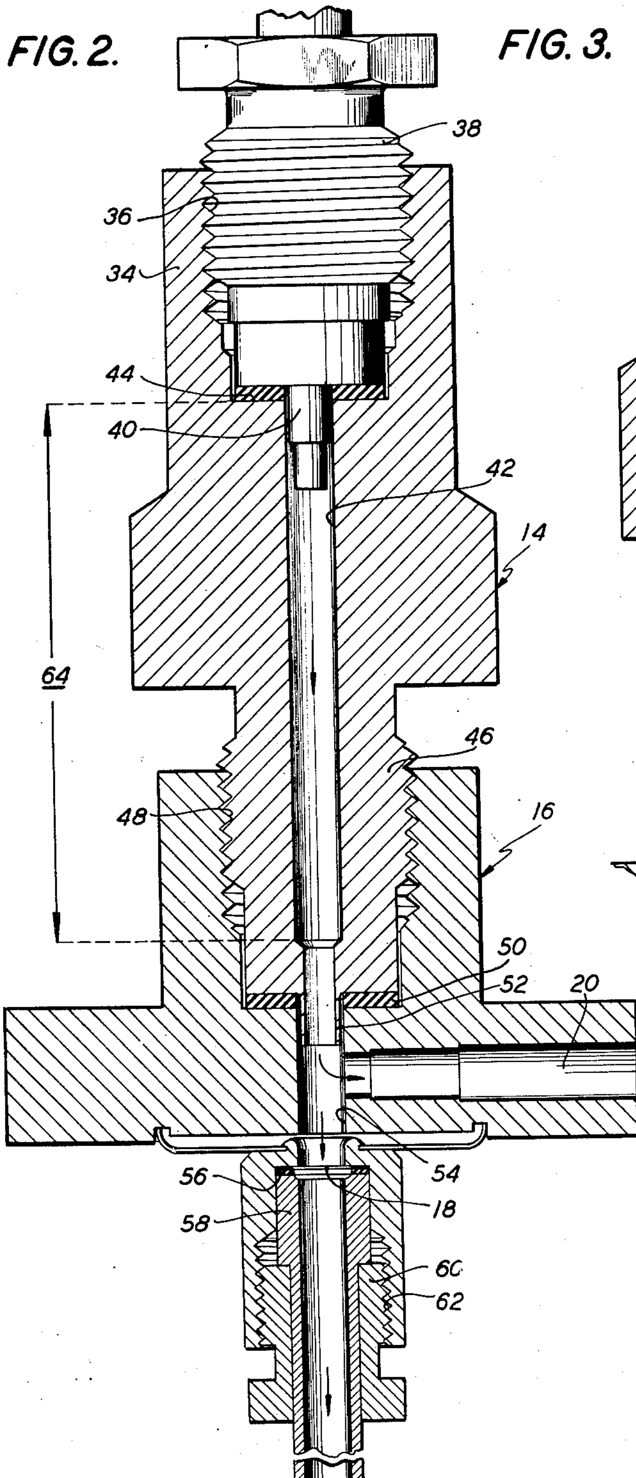
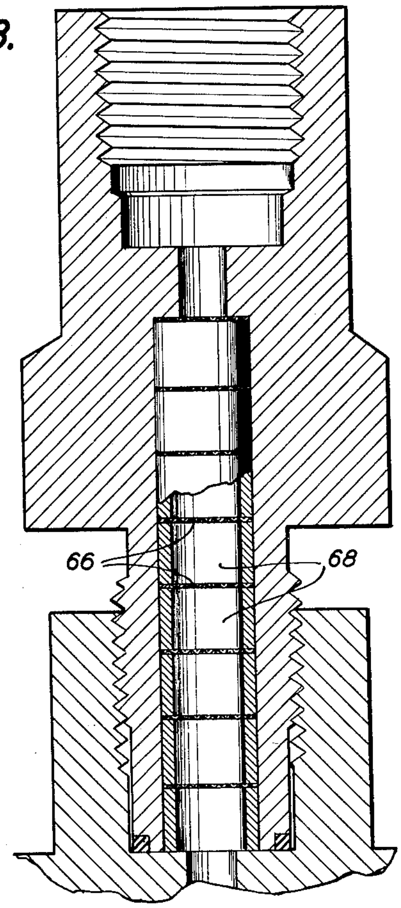


FIG. 3.



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HEAT COMPENSATING DEVICE

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Application May 9, 1955, Serial No. 506,845

8 Claims. (Cl. 250—41.9)

This invention relates to apparatus for introducing a sample of a gas or gaseous mixture into an enclosure which is being constantly evacuated by a vacuum pump, and it has particular reference to means for compensating for heat loss suffered by the gas passing through said apparatus. The invention is especially useful in conjunction with apparatus for introducing a gas sample into a mass spectrometer.

For analyzing gases or mixtures of gases, mass spectrometry involves ionization of a sample of the gas, as by bombardment with an electron beam, segregation of the resultant ions in accordance with their mass-to-charge ratio, and selective measurement of the discharge of ions of a given mass-to-charge ratio. The magnitude of the current developed by discharge of the ions of a given mass-to-charge ratio provides a basis for calculating the partial pressure of those molecules in the sample from which these particular ions were derived. Where a sample is to be analyzed for more than one component, as in the analysis of a gas mixture, the practice is to scan the mass spectrum by successively discharging ions of differing mass-to-charge ratio. The spectrum is scanned by varying one or more of the operational variables which determines the paths of travel of the ions.

Since the mass spectrometer of necessity must operate at a relatively low pressure, means must be provided to reduce the pressure of the gas sample to a low value before entering the instrument. This may be accomplished by allowing a sample from a supply of gas to pass through a capillary tube and then through a gold leak. The gold leak serves to control the rate of entry of the gas into the mass spectrometer, and usually takes the form of a gold foil, having a small opening through which the gas must pass.

Gas entering a capillary tube at a relatively high pressure and falling through a pressure drop to a relatively low pressure naturally undergoes an expansion. If this is effectively an isothermal expansion, the ratios of the partial pressures of the gases in the expanded sample remain the same as they were in the gas mixture supply, and the intensities of the signals produced at a recorder of the instrument are in the same ratio as the partial pressures of the gases in the gas mixture supply.

In present mass spectrometers, gas inlet systems have been arranged to include a capillary tube through which the gas expands, with the downstream end of the capillary connected to a gold leak flange having a gold leak and a by-pass outlet. The expanded gas sample enters into the gold leak flange and most of it is exhausted out the by-pass outlet. However, a small part of the gas enters the mass spectrometer through the gold leak, undergoing another expansion by this process.

Heretofore the supposition has been maintained that gas flow through a specified length and diameter capillary has in effect approximated an isothermal expansion process. Under this condition it would be expected that the downstream pressure would be linear with the upstream pressure and independent of gas viscosity. However, if the expansion of the gas through the capillary follows in effect an adibatic process rather than an isothermal, the downstream pressure will be dependent upon the ratio of the specific heat of the gas at constant pressure to that at constant volume, and so will vary with the type of gas used.

In analyzing gas mixtures, I have observed that the ratios of the partial pressures of the gas mixture constituents are changed by expansion of the gas through a capillary, and the intensity of the signals produced at the recorder of the mass spectrometer are not quite in the same ratio as the partial pressures of the gases in the gas mixture supply from which the sample was taken. The result is an erroneous analysis of the gas mixture.

Mass spectrometers are sensitive instruments and, in analyzing gases, one has to be on the guard against aspirator type pumping in the gold leak flange. This may be caused by the gas jetting past the by-pass outlet at supersonc velocities, and would tend to produce variations in the continuous gas flow through the gold leak resulting in variations in the gas analysis results.

The present invention solves the isothermal expansion problem by providing means for restoring the correct ratios of the partial pressures of the gases in the expanded sample before entering the mass spectrometer, and it also reduces the probability of aspirator type pumping by reducing the translational velocity of the gas. The invention comprises a heat compensating member having an inside surface with which the gas makes turbulent contact for regaining the heat necessary to bring its temperature to substantially its upstream condition, thus restoring the linear relationship between the upstream and downstream pressure and making the ratio of these pressures substantially independent of the nature of the gas.

In a preferred embodiment, the heat compensating member is adapted to be connected between the downstream end of the capillary tube and the gold leak flange, the member maintaining itself approximately at the temperature of the gas mixture supply by natural heat exchange with the atmosphere of its surroundings. The inside surfaces of the member may be formed in a variety of ways, such as by providing a center bore through the member, the principle being to provide sufficient inside surface area for sufficient heat transfer to the gas under these conditions.

It is notable that the heat compensating member may be provided with suitable adaptors on each end so that it may be conveniently attached in the gas inlet line of a mass spectrometer without requiring any modification of any parts of the mass spectrometer system. Also, such addition of this heat compensating member suffices to restore the linear relationship between upstream and downstream gas pressure which might otherwise have to be accomplished through a re-design of the whole gas inlet system of the mass spectrometer, and is therefore a very useful and economical thing.

The invention is more clearly understood with reference to the drawings, in which:

Fig. 1 is a schematic drawing showing how the invention might be applied to the gas inlet system of a mass spectrometer;

Fig. 2 is a drawing of an embodiment of the invention shown in section and illustrates the mechanical connections between the invention and other components of the gas inlet system of the mass spectrometer;

Fig. 3 is a drawing of an alternate embodiment of the invention shown in section; and

Fig. 4 is a drawing of a further alternate embodiment of the invention shown in section.

With reference to Fig. 1, a gas supply 10 supplies a gas mixture at a total pressure P_0 and temperature T_0 to the upstream end 12A of a capillary tube 12. The gas mixture expands through the capillary tube and exits at the downstream end 12B of the capillary tube at a substantially reduced total pressure P_1 and a slightly reduced temperature T_1. Thereupon, the gas enters into the heat compensating member 14 of the invention which maintains itself approximately at a temperature of T_0 by natural heat exchange with the atmosphere of its surroundings. For example, both the gas supply 10 and the heat compensating member 14 may be at room temperature, and the heat compensating member 14 tends to restore the gas of reduced pressure to room temperature.

By making turbulent contact with inner surfaces of the heat compensating member, the gas mixture regains sufficient heat to restore its temperature back up to the supply temperature T_0 and assumes a pressure P_2 which is slightly greater than the exit pressure from the capillary tube P_1. This causes the gas expansion between the gas supply and the point where it exits the heat compensating member to approximate in effect an isothermal expansion and results in the ratio of the pressures P_0 to P_2 being substantially independent of the nature of the gas used.

The heat compensating member has its downstream end connected to a gold leak flange 16 having a gold leak 18 and a by-pass outlet 20. The gas exits from the heat compensating member and enters into the gold leak flange, most of the gas leaving the gold leak flange through the by-pass outlet which leads to a vacuum pump 21. A portion of the gas, however, flows through the gold leak 18 into a mass spectrometer 22 which has an exit 24 leading to the vacuum pump for constantly evacuating the mass spectrometer. In passing through the gold leak there is a further expansion in the gas mixture. The expansion through the gold leak is observed to approximate an isothermal process. The reason for this is not certain but it has been proposed that there is viscous flow through the capillary and molecular flow through the gold leak. Thus the pressure and temperature of the gaseous mixture inside the mass spectrometer may be termed approximately as P_3 and T_0, respectively.

The mass spectrometer analyzes the gas mixture, and as molecules of different mass-to-charge ratio are brought into focus in the mass spectrometer, a proportional electrical signal is sent to a recorder 26 which records a particular peak of the plurality of peaks shown at 28 on a moving charge 30. The various peaks recorded on the moving chart by the recorder as measured from a base line 32 will bear the same relation to each other as do the corresponding partial pressures of the gases in the gas mixture of the gas supply.

With reference to Fig. 2, the heat compensating member has an adaptor portion 34 at its upstream end in which there is a threaded recess 36 for receiving a threaded plug adaptor 38 that is rotatably fitted on the downstream end of the capillary tube 12. The plug adaptor for the capillary tube has a protruding portion 40 which depends from the downstream end of the plug adaptor and extends into a center bore 42 of the heat compensating member for a short distance. A gas-tight fit between the capillary plug adaptor and the heat compensating member is accomplished by means of an annular gasket 44. The center bore 42 of the heat compensating member has a cross-sectional area considerably larger than the cross-sectional area of the capillary opening in the capillary tube, and its surfaces are those which supply the heat to the gas mixture.

The heat compensating member has an exteriorly threaded portion 46 at its downstream end which is adapted to be received in a threaded recess portion 48 of the gold leak flange 16. A gas-tight fit is provided by an annular gasket 50, and when screwed into position a protruding portion 52, which protrudes from the downstream extremity of the heat compensating member, extends for a short distance into a center bore 54 of the gold leak flange. The by-pass outlet 20 of the gold leak flange is disposed perpendicular to the center bore of the flange, opening into the center bore of the flange and, in the other direction, extending to the vacuum pumping system. Downstream from the by-pass outlet the gold leak 18 is supported across the center bore 54 by an annular gasket 56, an extension tube 58 and a threaded plug adaptor 60, the adaptor 60 screwing into a threaded recess 62 at the downstream end of the gold leak flange.

By way of example, the heat compensating member of Fig. 2 is found to naturally maintain itself at room temperature (the temperature of the gas mixture supply) while imparting sufficient heat to the gas under the following conditions. The member is made from ⅝" stock hexagonal steel; the length of its center bore 42, as shown by the dimension 64, is 1.043 inches; and the bore has a diameter of 0.0938 inch. The gas is supplied to the upstream end of the capillary tube at one-atmosphere pressure and expands through the capillary to a pressure of about 1 millimeter of mercury, and thereafter expands through the gold leak to a pressure of about 0.0001 millimeter of mercury.

It should be noted that, if the gas mixture supply is not at room temperature, a portion of the gas mixture of the supply can be passed around the heat compensating member by means of a conventional jacket to insure that the heat compensating member maintains itself at the temperature of the supply gas mixture by natural heat exchange with an atmosphere of the gas mixture itself. Thus, no external heating or heat control means are required for normal operation.

With reference to Fig. 3, the heat compensating member may be provided with a plurality of fine mesh wire screens 66 spaced along the bore by a plurality of annular spacers 68. The screens facilitate thermal transfer to the gas, further reduce the translational velocity of the gas, and also serve as a filter to keep foreign particles from plugging the gold leak. Commercially available screens of stainless steel with a 2-mil mesh are suitable.

With reference to Fig. 4, the heat compensating member may be provided with a plurality of center bores 70, 72, 74, the bores becoming larger in the downstream direction. This arrangement also facilitates heat transfer to the gas.

I claim:

1. Apparatus for introducing a sample of gas from a gas supply into an enclosure which is being constantly evacuated by a vacuum pump comprising capillary means connected to the gas supply with the gas expanding through the capillary means and thereby lowering its temperature and pressure, and heat compensating means connected between the capillary means and the evacuated enclosure and having at least one surface with which the gas makes turbulent contact for regaining the heat necessary to bring its temperature substantially back up to that of the gas supply before it enters the evacuated enclosure.

2. Apparatus for introducing a sample of gas from a gas supply into a mass spectrometer comprising capillary means connected to the gas supply with the gas expanding through the capillary means and thereby lowering its temperature and pressure, and a heat compensating member connected between the capillary means and the mass spectrometer and having inside surfaces with which the gas makes turbulent contact for regaining the heat necessary to bring its temperature substantially back up to that of the gas supply before it enters into the mass spectrometer.

3. In apparatus for introducing a sample of gas from a gas supply into a mass spectrometer, including a capillary having an upstream end and a downstream end and means for supplying the gas to the upstream end of the capillary with the gas expanding through the capillary and thereby lowering its temperature and pressure, the improvement which comprises a heat compensating member connected between the downstream end of the

capillary and the mass spectrometer and having inside surfaces with which the expanded gas makes turbulent contact for regaining the heat necessary to bring its temperature substantially back up to that of the gas supply before it enters into the mass spectrometer and thereby causing the ratio of the upstream gas pressure to the downstream gas pressure to be substantially independent of the nature of the gas.

4. Apparatus according to claim 3 wherein the heat compensating member maintains itself approximately at the temperature of the gas supply by natural heat exchange with the atmosphere of its surroundings.

5. In apparatus for introducing a sample of gas from a gas supply into a mass spectrometer, including a capillary having an upstream end and a downstream end and means for supplying the gas to the upstream end of the capillary with the gas expanding through the capillary and thereby lowering its temperature and pressure, the improvement which comprises a heat compensating member connected between the downstream end of the capillary and the mass spectrometer, the heat compensating member having a center bore of considerably greater cross-sectional area than that of the capillary, the center bore providing an inside surface with which the expanded gas makes turbulent contact for regaining the heat necessary to bring its temperature substantially back up to that of the gas supply before it enters into the mass spectrometer and thereby causing the ratio of the upstream gas pressure to the downstream gas pressure to be substantially independent of the nature of the gas.

6. In apparatus for introducing a sample of gas from a gas supply into a mass spectrometer, including a capillary having an upstream end and a downstream end and means for supplying the gas to the upstream end of the capillary with the gas expanding through the capillary and thereby lowering its temperature and pressure, the improvement which comprises a heat compensating member connected between the downstream end of the capillary and the mass spectrometer, the heat compensating member having a center bore of considerably greater cross-sectional area than that of the capillary and a plurality of fine mesh screens spaced along the center bore, the screens and the bore providing surfaces inside the heat compensating member with which the expanded gas makes turbulent contact for regaining the heat necessary to bring its temperature substantially back up to that of the gas supply before it enters into the mass spectrometer and thereby causing the ratio of the upstream gas pressure to the downstream gas pressure to be substantially independent of the nature of the gas.

7. In apparatus for introducing a sample of gas from a gas supply into a mass spectrometer, including a capillary having an upstream end and a downstream end and means for supplying the gas to the upstream end of the capillary with the gas expanding through the capillary and thereby lowering its temperature and pressure, the improvement which comprises a heat compensating member connected between the downstream end of the capillary and the mass spectrometer, the heat compensating member having a plurality of coaxial center bores, the cross-sectional area of the opening formed by the bores becoming progressively larger in the downstream direction, and the bores providing surfaces inside the heat compensating member with which the expanded gas makes turbulent contact for regaining the heat necessary to bring its temperature substantially back up to that of the gas supply before it enters into the mass spectrometer and thereby causing the ratio of the upstream gas pressure to the downstream gas pressure to be substantially independent of the nature of the gas.

8. In apparatus for introducing a sample of gas from a gaseous mixture supply into a mass spectrometer which is being constantly evacuated by a vacuum pump, including a capillary having an upstream end and a downstream end and means for supplying the gaseous mixture to the upstream end of the capillary tube at supply temperature and pressure with the gas mixture expanding through the capillary and thereby lowering its temperature and pressure, the improvement which comprises a heat compensating member connected between the downstream end of the capillary and the mass spectrometer, the heat compensating member naturally maintaining itself approximately at supply temperature by natural heat exchange with the atmosphere of its surroundings and having inside surfaces with which the gaseous mixture makes turbulent contact and regains the heat necessary to bring its temperature substantially back up to supply temperature, thereby causing the ratios of the partial pressure of each gas in the gaseous mixture supply to the corresponding partial pressure of each gas in the expanded gaseous mixture below the downstream end of the capillary tube to be approximately equal.

No references cited.