Sept. 9, 1952 W. W. WALTON ET AL 2,610,300

FLOW CONTROL

Filed Aug. 7, 1951

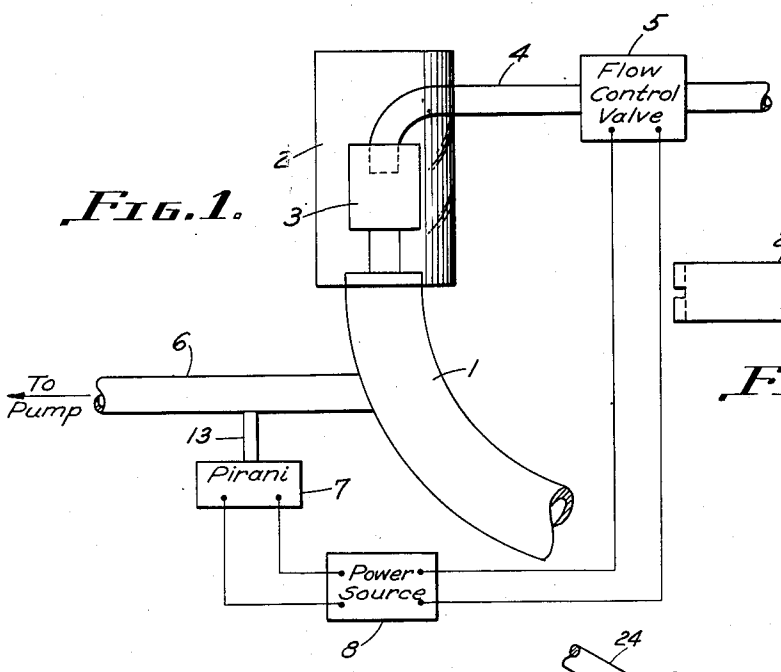



FIG. 1.

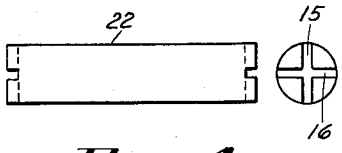



FIG. 4.

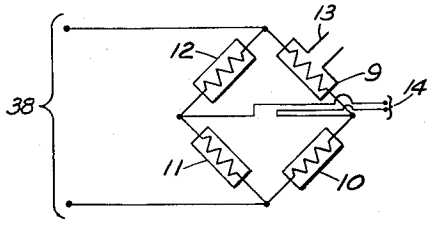



FIG. 2.

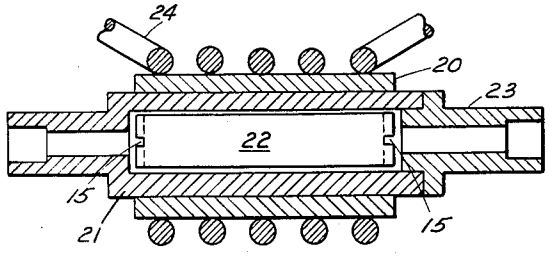



FIG. 3.

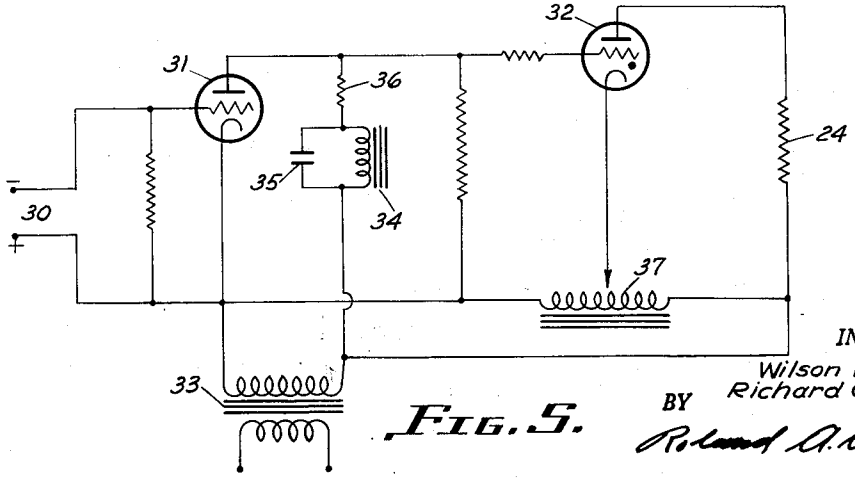



FIG. 5.

INVENTORS
Wilson W. Walton
Richard C. Bowers
BY
Roland A. Anderson

Patented Sept. 9, 1952 2,610,300

# UNITED STATES PATENT OFFICE

2,610,300

## FLOW CONTROL

Wilson W. Walton, Oak Ridge, Tenn., and Richard C. Bowers, Berkeley, Calif., assignors to the United States of America as represented by the United States Atomic Energy Commission

Application August 7, 1951, Serial No. 240,738

13 Claims. (Cl. 250—41.9)

1

Our invention relates to mass spectrometers, and more particularly to devices for controlling the flow of gases to a mass spectrometer or other appropriate equipment.

The mass spectrometer is an apparatus for sorting ions according to their specific mass. Gases to be examined are fed into the apparatus and are ionized by ionizing particles, preferably by bombardment with electrons. The ions thus formed are ejected in the form of a beam to the main body of the tube by means of accelerating potentials. Here they are ordinarily subjected to the influence of a magnetic field causing them to travel in arcuate paths whose radii are determined by the masses and charges of the respective ions. By focusing the desired ion beam on a collector at one extremity of the tube, the ions are caused to impinge successively on the collector and are discharged. The current thus obtained is an index of the ions in the selected beam.

In the prior art it has been the practice to provide an ion source inclosed in a glass envelope, and to inclose the collector in a separate glass envelope, so that they are spaced apart from each other but joined together through the extremities of an arcuate piece of flattened copper tubing which defines the main body of the tube through which an ion beam travels and in which it is subjected to a magnetic or electric field. The glass envelope of the source and the collector respectively are sealed to the copper tubing.

Operational efficiency and reliability of operation are factors which are under constant observation and study with a view to improvement. An element of operational efficiency and stability is the charge consumption rate and this is dependent among other things, upon the vapor flow to the mass spectrometer. This in turn depends upon the constriction through which the gas must pass in order to reach the spectrometer.

In the prior art vapor flow has been controlled by a manually operated valve in accordance with instrument readings, but this requires the undivided attention of the operator who must make frequent adjustments in response to changing conditions in the operation of the spectrometer. Under such conditions it is difficult to maintain uniform pressures within the tube and to insure proper flow or optimum operation.

Applicants with a knowledge of these problems in the prior art have for an object of their invention the provision of a system for automatically controlling the vapor flow to a mass spectrometer or other appropriate equipment.

Applicants have as another object of their invention the provision of a heat controlled valve for regulating the flow of vapor to a mass spectrometer or other appropriate equipment.

Applicants have as a further object of their invention the provision of an arrangement for regulating the flow of a gas to a mass spectrometer tube, simply and without moving parts.

2

Applicants have as a still further object of their invention the provision of a flow control which may be manufactured with precision and in quantity, and any number of which can be made to give closely reproducible results.

Applicants have as a still further object of their invention the provision of a system responsive to pressure within a spectrometer tube for automatically controlling the flow of vapor thereto in order to maintain substantially uniform pressure therein.

Other objects and advantages of our invention will appear from the following specification and accompanying drawings, and the novel features thereof will be particularly pointed out in the annexed claims.

In the drawings, Fig. 1 is a schematic of a portion of one form of mass spectrometer incorporating our improved flow control therein. Fig. 2 is a schematic of a conventional Pirani gauge for controlling the flow through our improved valve arrangement. Fig. 3 is a cross section of our improved flow control valve. Fig. 4 is a detail of the piston of our improved flow control valve. Fig. 5 is a schematic of the electronic control circuit for our improved control valve.

Referring to the drawings in detail, 1 designates the main body of the mass spectrometer through which the ion beam travels and which is preferably made of copper tubing. To the end of body 1 is joined a glass envelope of any appropriate configuration, shown symbolically at 2. Disposed within the envelope 2 and mounted on the tube 1 is an ion source 3. The electrical leads (not shown) which feed the ion source and its various elements come into the envelope 2 on either side thereof through appropriate reentrant meshes or the like. Sample line 4 may enter the side of the envelope in the usual manner and may terminate within the source adjacent to its upper extremity. Flow of gas through line 4 is controlled by valve 5 which is disposed therein between the gas source and the spectrometer.

Projecting from the wall of tube 1 is a vacuum outlet extension 6 which leads to an appropriate vacuum pump (not shown). A Pirani gauge 7 is provided for communication with the extension 6 leading to the vacuum pump, and is connected to control the operation of the electronic control circuit which feeds the heater element of the flow control valve 5 so that gas flow to the spectrometer may be such as to maintain a substantially uniform pressure within the tube 1.

The Pirani tube makes a very convenient pressure responsive means for the flow control element, since it provides a continuous flow of information. This gauge measures pressure indirectly and the operation of this hot wire gauge is based on the principle that the heat conductivity of a gas at low pressure, e. g. below 1000

microns, is proportional to its pressure. The gauge measures the resistance of a length of wire. The wire is inclosed in a tube attached to the vacuum system so that the wire is exposed to the same vacuum as is in the system. The wire is heated by an electric current flowing through it. Within a certain range, the temperature of the wire, and thus its resistance depends upon the pressure of the gases surrounding the wire. When the pressure is decreased, relatively less heat escapes from the wire, its temperature rises with consequent increases in resistance, and less current flows therethrough.

A simplified arrangement of the Pirani tube is illustrated in Fig. 2. It is desirable that the gauge be very sensitive to pressure. In order to be responsive to various slight changes in the resistance of the wire in the Pirani tube for slight changes in pressure, the length of wire is made one of the four resistances of a sensitive Wheatstone bridge. The gauge unit which is attached to the vacuum system, preferably contains all four resistances **9**, **10**, **11** and **12**. Resistances **10**, **11** and **12** are preferably sealed in tubes. Wire resistance **9** is in a tube connected directly to the vacuum system, as indicated at **13** in Fig. 1. Resistances **10**, **11** and **12** are at substantially constant pressure and uniform temperature. The resistance of element **9** changes in response to changes in pressure. Slight changes of resistance unbalance the Wheatstone bridge and are reflected in voltage changes at the output **14**. Ordinarily they are recorded by a meter in the usual vacuum systems. However, they serve to control the power feed to the flow control valve **5** in this arrangement. Power is fed to the system at the juncture of resistances **9**, **12** and **10**, **11** from the usual D. C. power source through leads **38**.

The flow control valve disclosed in detail in Fig. 3, and shown schematically in Fig. 1, comprises a variable orifice, the port area of which is controlled by temperature. A sleeve **20** of a substantially non-expansible material, such as Invar, surrounds an inner sleeve **21**, of some material which is chemically inert to the gases to be metered. Within the sleeve **21** is placed a piston or cylinder **22** which is substantially solid and is of a material having a higher coefficient of expansion than the sleeve **20**. Application required operation with a pressure drop of about one pound per square inch across the control and a flow of about .003 standard cc. per minute. The dimensions of the control determine the flow range and pressure drop, and they must be accurately determined, probably by trial and error to satisfy a given set of conditions. It was estimated that a piston length of 2 inches and a diameter of .497 inch, working in a cylinder bored to .500 inch would satisfy the conditions stated. Increasing the clearance between cylinder bore and piston will increase the flow or decrease the pressure drop, while increasing the length will have the reverse effect.

As shown in Fig. 3, the cylinder **22** has cross slots **15**, **16** milled or otherwise formed in each end to permit passage of the gas in the event the cylinder becomes lodged against one end of the sleeve **21** or against a perforated plug **23** which is installed in the other end of sleeve **21** to hold the cylinder in place. The plug **23** may be silver soldered or otherwise sealed to the sleeve **21**.

In operation the clearance between the cylinder **22** and the sleeve **21** is regulated by the temperature of the whole assembly. A convenient means for maintaining the assembly at an elevated temperature is a heating coil **24**, wrapped thereabout and may be in the form of a standard Calrod heater. As the temperature of the assembly is raised, the cylinder **22** expands more than the sleeve **21**, because the latter is restricted by the relatively non-expansible sleeve **20** which is preferably shrunk fit on the sleeve **21**.

Since the sleeve **21** serves the sole purpose of protecting the sleeve **20** from the action of the gases which may pass through the orifice, it is obvious that the function of the sleeve **21** may be dispensed with in many cases, for example, a simpler and just as effective arrangement is to make the sleeve **21** out of the relatively non-expansible material of sleeve **20** and to omit sleeve **20** entirely.

A number of alternative arrangements in the choice of materials and dimensions of the parts will be apparent. For example, it may be desirable to provide a flow control which will cut off the flow on failure of the heating element **24**, and will pass the gas only on being heated. This arrangement might also be of general application. Such a device is obtained when the materials of the cylinder **22** and the sleeve **20** are interchanged. It is obvious that if the cylinder **22** is made of Invar and the sleeves **21** and **22** of copper, the gas flow increases with temperature rather than decreases as in the modification first described. In either case, the Invar element may be protected from the action of the gases being metered by a suitable coating of a resistant metal. The coating may take the form of a machined sleeve, as shown in the drawing, or it may be a layer electroplated or otherwise formed on the element to be protected. Another useful variation of the invention lies in the use of different plastics for the material from which the cylinder or sleeves are made. Many plastics and ceramics are available which have sufficiently high softening points to be dimensionally stable when formed in stress free masses.

The variable orifice described above is particularly suited for automatic regulation of the gas flow to a mass spectrometer tube such as that shown in Fig. 1. An appropriate power circuit is shown in Fig. 5 wherein the terminals **30** are connected to the signals of a Pirani gauge through leads **14** of the bridge. The signals, suitably amplified through the tube **31**, activate a gas filled tube **32** such as a thyratron. The thyratron **32** is connected so as to regulate the current supplied to the resistance or heating coil **24**. The source of potential to the anode of the tube **31** may be any standard type of power supply, although a simple arrangement is shown wherein the output of a transformer **33** is rectified by the tube **31** and filtered sufficiently for the purpose described by the filter circuit comprising inductance **34**, capacitance **35**, and resistance **36**. The thyratron **32** is supplied with alternating current, which may be from the transformer **33**, or a separate transformer may be used in accordance with well known practices in the art. As shown, the potential applied to the grid of the tube **32** is adjusted by means of a variable autotransformer **37**, commonly known as a "variac" to provide preset control, for the signal voltage supplied to the tube **32** will control the current through the resistance **24**.

In operation, as the Pirani bridge, **9**, **10**, **11**, **12** becomes unbalanced due to pressure conditions, the potential changes and current flows through the circuit including leads **14** and the resistance in the input of tube **31**. The signals impressed

upon the grid of tube 31 are amplified and impressed upon the input of thyratron 32 where they act to trigger the tube and cause current to flow in the anode circuit thereof including the load resistance 24. The thyratron 32 is so biased as to permit the grid to gain control after firing in response to signals. Pirani unbalance signals could be more easily amplified to a higher level by exciting the Pirani bridge with 60 cycle alternating voltage and using a transformer coupled high gain amplifier.

From the foregoing it will be apparent that gas flowing through feed line 4 enters the mass spectrometer source 3 and is ionized in the usual manner. The ions are pulled out from the source into the tube 1 by the usual accelerating potentials and in tube 1 they are acted upon by a magnetic field to cause them to travel in arcuate paths whose radii correspond to their respective masses. The pressure in tube 1 controls the flow of current through the Pirani gauge 7 and in turn this current controls the operation of the power source or electronic control circuit 8 for changing the current flow through the control valve 5, thereby changing the restriction of the orifice therein and the flow of gas through line 4 to the spectrometer.

Having thus described our invention, we claim:

1. A vapor flow control system of the character described comprising a mass spectrometer, a pressure responsive gauge within the spectrometer responsive to pressure conditions therein for producing signals, a power supply responsive to signals from said gauge for operation, and a flow control valve actuated by said power for regulating the flow of vapor to said spectrometer.

2. A vapor flow control system of the character described comprising a chamber, means responsive to vapor changes in the chamber for producing electrical signals, a power source operated by said signals, and vapor flow control means responsive to heat energy supplied by said power source for regulating the flow of vapors to said chamber.

3. A vapor flow control system of the character described comprising a mass spectrometer, means responsive to changes in vapor content in said spectrometer for producing electrical signals, and vapor flow control means responsive to heat energy supplied by said power source for regulating the flow of vapors to said spectrometer.

4. A vapor flow control system of the character described comprising a chamber, means responsive to vapor content in communication with said chamber for producing signals, a source of heat energy responsive to said signals and a flow valve actuated by the heat energy from said source for regulating vapor flow to said chamber.

5. A vapor flow control system of the character described comprising a chamber, means responsive to vapor content in said chamber for producing electrical signals, a power supply controlled by said signals, and a flow valve closing in response to heat energy from said power source for regulating the flow of vapors to said chamber.

6. A vapor flow control system of the character described comprising a chamber, means responsive to pressure changes in the chamber for producing signals, a flow control valve responsive to changes in temperature for regulating the flow of vapors to said chamber, and a power source controlled by signals from said pressure responsive means for supplying power to said valve for controlling its temperature and operation.

7. A vapor flow control system of the character described comprising a chamber, a pressure gauge responsive to pressure changes in said chamber for changing its resistance and producing electrical signals, a power supply coupled to said gauge and controlled by signals therefrom, and a flow control valve operated by power supplied from said power supply for regulating the flow of vapors to said chamber.

8. A vapor flow control system of the character described comprising a chamber, an electrical bridge, means for altering the balance of the bridge to produce electrical signals, a power circuit fed by the bridge and responsive to signals therefrom for providing electrical energy, and a valve operated by an electrical heating element and fed by said power circuit for regulating the flow of vapors to said chamber.

9. A vapor flow control system of the character described comprising a chamber, a bridge network, means responsive to changes in vapor content of said chamber for altering the balance of the network to produce signals, means for amplifying said signals, means coupled to and controlled by the operation of said amplifying means for providing electrical power, means fed by said last means for converting electrical power into heat, and a valve operated in response to heat energy from said converting means for regulating the flow of vapors to said chamber.

10. A fluid flow control valve of the character described comprising an outer casing, an inner member smaller than the casing and having a different coefficient of heat expansion than said casing disposed therein, and means for applying heat to said casing and said member for altering relative sizes to regulate the flow of fluid through the casing.

11. A flow control valve of the character described comprising a casing having a bore therein for the passage of fluids, an expansible element having a different coefficient of heat expansion than said casing disposed in said bore, and means for applying heat to said casing and said element to alter the relative sizes of the bore and expansible element to regulate the flow of fluid through the valve.

12. A flow control valve of the character described comprising a sleeve having a longitudinal bore, a plug smaller than said bore and having a different coefficient of heat expansion than said sleeve positioned in said bore, and means for applying heat to said sleeve and said plug to alter their relative sizes and regulate the flow of fluid through the valve.

13. A flow control valve of the character described comprising a sleeve having a bore for the passage of fluid, a plug smaller than said bore and having a different coefficient of heat expansion than said sleeve positioned in said bore, and an electrical resistance element disposed about the sleeve for applying heat to said sleeve and said plug for altering their relative sizes and regulating the flow of fluid through the bore.

WILSON W. WALTON.
RICHARD C. BOWERS.

REFERENCES CITED

The following references are of record in the file of this patent:

UNITED STATES PATENTS

| Number | Name | Date |
|---|---|---|
| 2,463,318 | Schneider et al. | Mar. 1, 1949 |
| 2,564,175 | Roper et al. | Aug. 14, 1951 |