

July 8, 1952

M. G. INGRAM ET AL

2,602,898

ION INTENSITY CONTROL MECHANISM FOR MASS SPECTROMETERS

Filed June 30, 1950

3 Sheets-Sheet 1

Fig. 3

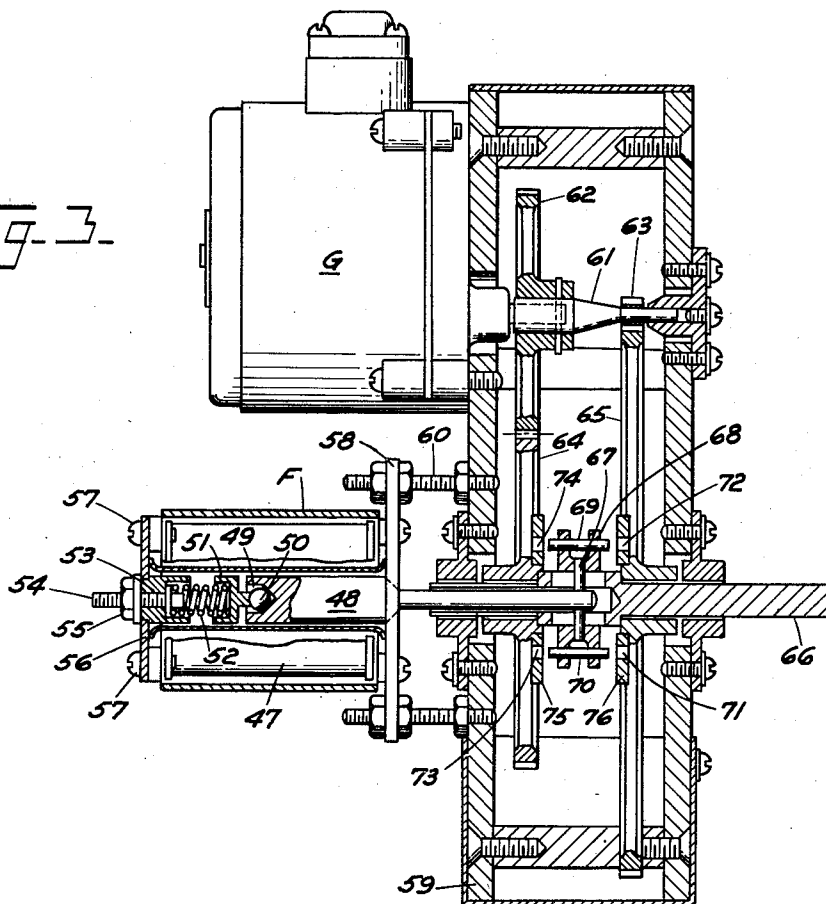
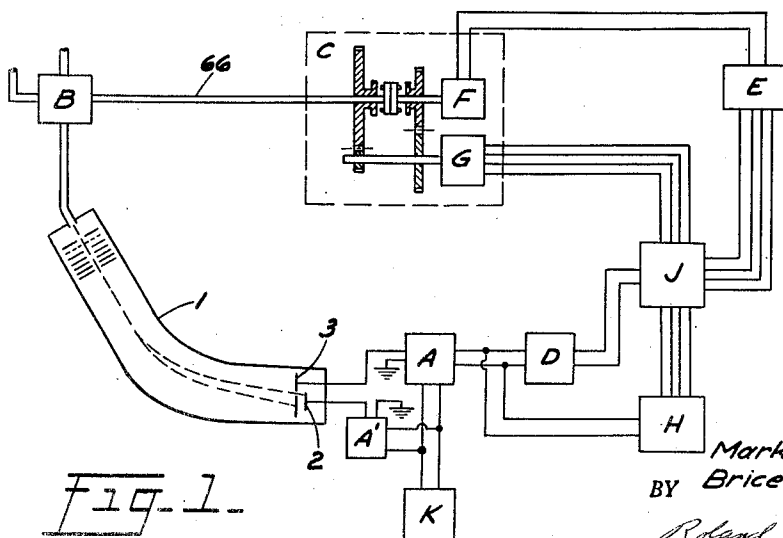


Fig. 1

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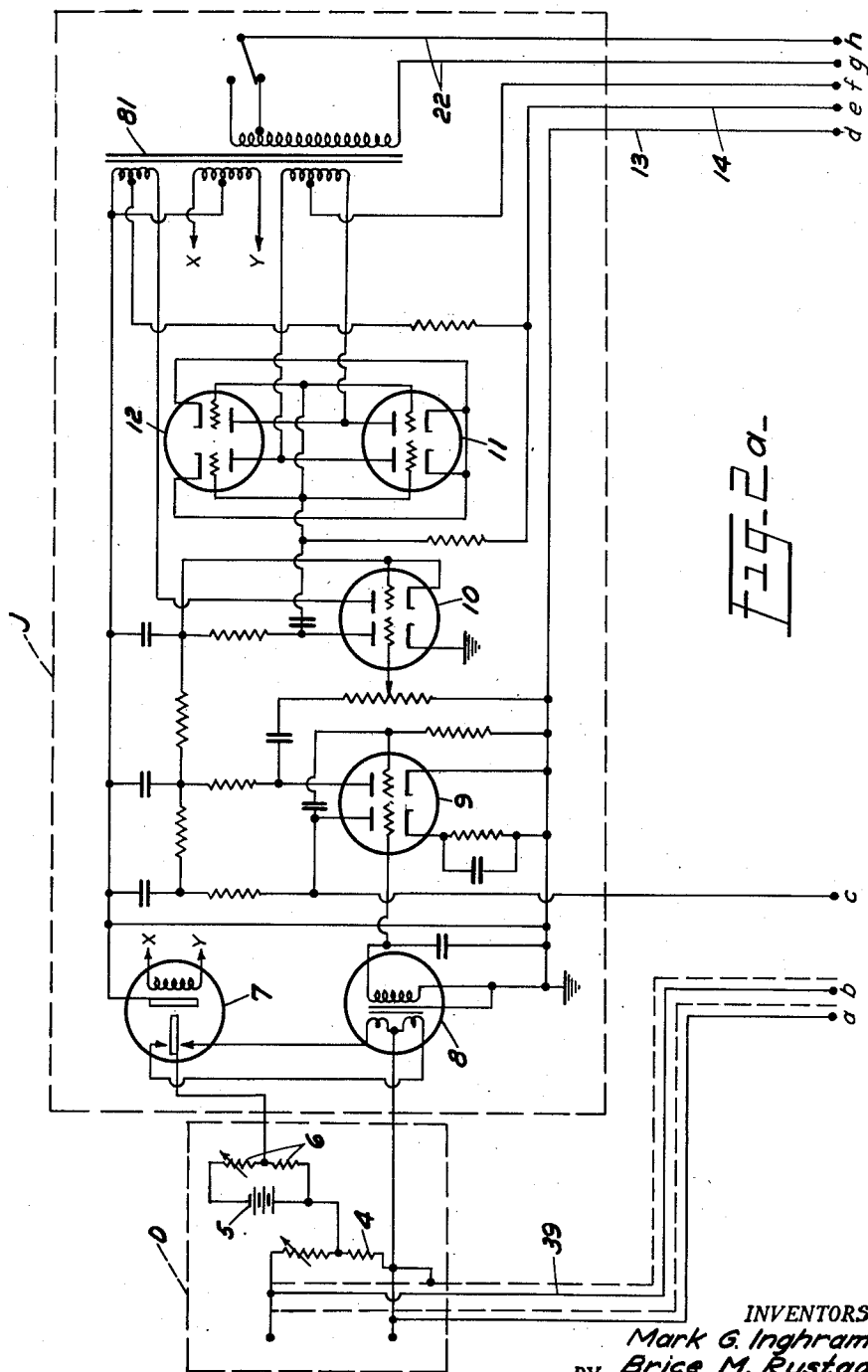


Fig. 2a-

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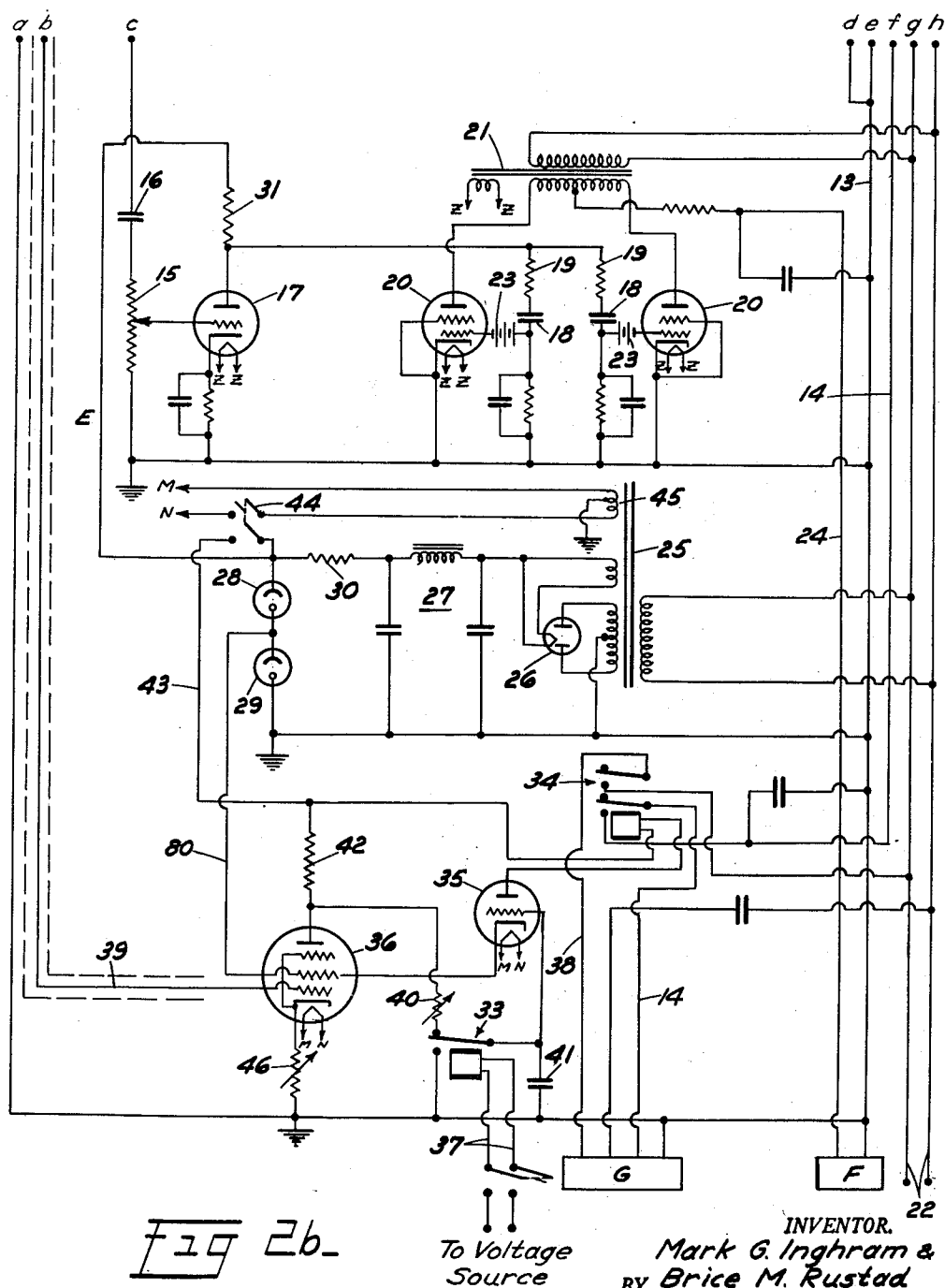
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UNITED STATES PATENT OFFICE

2,602,898

ION INTENSITY CONTROL MECHANISM FOR
MASS SPECTROMETERS

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12 Claims. (Cl. 250—41.9)

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Our invention relates to mass spectrometers and more particularly to an arrangement for automatically controlling the ion intensity thereof to facilitate continuous and rapid analysis of gases.

Heretofore, the mass spectrometer has been utilized as a continuous gas analyser. It has been the practice to continuously sample a gaseous mixture in a system. A sample is customarily bled from the system and continuously fed to the source of a mass spectrometer where it is subjected to electron bombardment. As a result of this bombardment, ions of the components of the mixture are formed in proportion to the relative concentrations of such components in the sample. The ions are then separated according to their masses, and the respective ion currents are measured. From the relative magnitudes of these currents the quantitative composition of the mixture can be determined.

In general, the intensity of the ion beam does not stay constant, but varies with changing spectrometer source conditions and with the pressure of the sample gas. When this intensity variation is rapid as compared with the time required for analysis, a great amount of care must be exercised by the mass spectrometer operator in both readjusting the ion intensity, and in collecting a sufficiently large quantity of data, in such a manner as to statistically minimize the errors caused by the variation in intensity.

Applicants with a knowledge of these problems in the prior art have for an object of their invention the provision of a mass spectrometer for continuously sampling and analysing gases by automatically controlling the ion intensity to overcome the necessity for time consuming manual adjustments and operations.

Applicants have as another object of their invention the provision of a mass spectrometer for continuous analysis, arranged to control the ion intensity of the ion beam through regulation of the flow of gas into the mass spectrometer by making such flow responsive to the ion intensity of the beam to be controlled.

Applicants have as another object of their invention the provision of a mass spectrometer having a valve for regulating the flow of gases to be analysed, and an anti-hunt arrangement for limiting the action of the valve to insure uniform flow of gases upon adjustment.

Applicants have as a further object of their invention the provision of a mass spectrometer for continuous analysis of gases with an arrangement for automatically preventing changes in the

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flow of the gases to be analysed while the background readings are being taken.

Applicants have as a still further object of their invention the provision of a mass spectrometer for the continuous analysis of gases with an arrangement for automatically preventing changes in the flow of gases to be analysed when the sample is being pumped out of the spectrometer.

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 our improved system for controlling the ion intensity of a mass spectrometer. Fig. 2a is a schematic of the circuits of the intensity selector and the motor controller of our improved system. Fig. 2b is a schematic of the circuits of the range speed control, power supply and automatic shut-off control of our improved system. Fig. 3 is an elevation, partly in section, of the anti-hunt drive and gear shift mechanism for our improved system.

The control method of the present invention is based on an automatically controlled by-pass valve which is located in the sample lead to the spectrometer tube. The automatic intensity control unit is shown in the block diagram in Fig. 1. The spectrometer tube 1, shown in that figure, is of conventional design such as the type used in the analysis of uranium hexafluoride, separating the ions into $U^{235}F_5^+$ and $U^{238}F_5^+$. The collector 2 collects the $U^{235}F_5^+$ ions and the slit plate 3 collects the $U^{238}F_5^+$ ions. The ion currents are preferably fed into separate highly stable degenerative feed-back amplifiers A, A', that is, the currents from the plate 3 of the mass spectrometer 1 are fed into amplifier A while the currents from plate 2 thereof are fed into amplifier A'. Their respective outputs are then fed to conventional recording instruments at K. The amplifiers in question are of the D. C. type conventionally employed to amplify the output of a mass spectrometer, and may take the general form of that disclosed in the prior co-pending application of Brenholdt, Ser. No. 21,022, filed April 14, 1948, now Patent No. 2,541,198. However, as an alternative, the ion currents from the mass spectrometer 1 may be fed into a dual feed-back amplifier instead of the single amplifiers shown in Fig. 1. The dual amplifier may be of conventional design where the ion currents pass through separate grid resistors to ground thereby setting up two separate voltages which are dependent on the ion currents. These

voltages are independently amplified and then are transmitted to suitable voltage recording instruments and from the values of these output voltages, the analysis of the gas is indicated. This amplifier is designed with as much negative feed-back as possible, in order to make the amplifier extremely stable and as far as possible independent of any fluctuating conditions which might occur in the electronic elements within the feed-back amplifier.

The control circuit may be made to keep constant either of the ion currents or the sum total of both ion currents. In the example being described, it was decided to keep the $U^{238}F_5^+$ ion beam constant. To do this the output voltage indicating the presence of $U^{238}F_5^+$ was fed into the intensity selector, D of Fig. 1. The intensity selector is set at a value that produces the value of voltage which bucks out the desired value of $U^{238}F_5^+$ ion beam. Thus, if the $U^{238}F_5^+$ ion beam is greater than desired the output of the circuit, being the algebraic difference between the output of the dual feed-back amplifier and the intensity selector, would indicate a voltage of a specific polarity. If the ion beam is smaller than is desired the output of this circuit would be of opposite polarity. If the ion beam is at the desired intensity the output of this circuit is a null voltage.

The output voltage from selector D is fed into a motor controller J of a conventional type, such as the Brown motor controller, manufactured by the Brown Instrument Company of Philadelphia. The D. C. output from the intensity selector D is first converted to an alternating current of a particular frequency, and then amplified in the motor controller J and finally applied to one winding of an appropriate motor G. The control motor G may be a conventional two phase induction motor, sometimes called a split phase induction motor. One phase can be continuously excited from a 110 volt A. C. line while the other phase is excited from the audio frequency amplifier of the motor controller unit J. The direction of rotation of such a motor depends upon the time phase relation between the fixed phase voltage and control phase voltage. The motor G is geared to the throttle by-pass valve B in Fig. 1. The connection to the windings of the motor G are chosen so that if the intensity of the ions impinging upon the ion collector is greater than the desired intensity, as determined by the adjustment of selector D, the motor rotates in a direction which opens the by-pass valve, and accordingly greater portions of the sample are shunted away from the mass spectrometer I. When the ion intensity falls below that determined by the setting of the selector D, the motor reverses direction, thereby permitting a greater flow of the sample to pass into the spectrometer. This process comprises the regulating action of the automatic ion intensity control unit.

Due to time lags between the change of sample flow at the by-pass valve B and the corresponding change in ion intensity, large oscillations can develop in the system. That is, if for some reason the ion beam intensity decreased, the control motor would turn the by-pass valve in a manner to increase the flow of gas into the spectrometer tube. The valve would continue to open until such time as the ion beam would be at the desired level of intensity, but because of the time delay in the circuit, by this time the valve would be open to the point where an excess amount of gas would be in the tube, and as a result the ion beam

would continue to increase to the point where the valve would start to turn in the opposite direction, and if the time delay were sufficiently large, large oscillations would result rendering the system useless. In order to overcome this tendency, a range speed control circuit E and an anti-hunt system, shown more in detail in Fig. 3, were provided.

This system utilizes a range-speed control electronic circuit E and a gear shift solenoid F. The range speed control circuit E excites the gear shift solenoid F whenever the ion intensity is not within a given range; thus, when the ion beam intensity is very nearly at the desired value the gear shift solenoid is not excited and the by-pass valve B is rotated at about 10 R. P. M. Whenever the ion beam intensity changes by more than a certain specified value, voltage is applied to the solenoid F, which shifts gears and changes the speed of rotation of the by-pass valve B to 120 R. P. M. Therefore, for large changes of ion beam intensity the speed of the valve adjustment will be large enough to bring the system rapidly to within the range of the desired beam intensity, and then the valve speed will decrease to an extremely slow speed, thereby preventing any possible oscillations from occurring.

To provide additional simplicity of operation two other modifications were incorporated into the electronic circuits by means of the automatic shut-off circuit H of Fig. 1. This circuit accomplishes two purposes: (a) Prevents the by-pass valve from turning to the end of its stroke when the sample is being pumped out of the mass spectrometer tube. (b) Prevents the valve from being turned when the "peaks-in-out" switch is thrown.

Referring now to Figs. 2a and 2b which constitute one circuit diagram and show certain of the circuits used in our improved system, 4 designates a voltage divider or potentiometer of the intensity selector D, which is connected across the output of a conventional amplifier A, referred to above, that amplifies the $U^{238}F_5^+$ ion intensity. A center tap of the voltage divider 4 is connected to a source of bucking voltage including battery or other E. M. F. source 5 across which is bridged a second potentiometer 6. The output of this intensity selector then feeds into a vibrating voltage converter 7 of the controller J, preferably of the standard Brown motor controller type, where it is converted into A. C. potential of a selected frequency, such as 60 cycles. The converter is energized from a secondary winding of power transformer 81 and the output is passed through an input transformer 8 and is amplified by conventional resistance coupled audio frequency power amplifier 9, 10, 11 and 12 fed by the conventional power transformer 81 from line 22. The output of this amplifier of the motor controller J is then applied through leads 13 and 14 to one phase of the motor G. Plates of tubes 9, 10 are supplied by a separate secondary of transformer 81 from that employed to supply the plates of tubes 11, 12. The filaments of the tubes may be supplied from a third secondary in a conventional manner.

The range of slow speed operation is limited by the setting of the potentiometer 15 of the range speed control E shown in Fig. 2b. Since potentiometer 15 is coupled through condenser 16 to the output of the first stage 9 of the amplifier of the motor controller J, it is apparent that the voltage across the potentiometer is that of the output of the first stage 9 of the amplifier. A portion

of this voltage, depending upon the setting of the potentiometer 15, is fed directly to the control grid of a triode 17 and is amplified by it. Energizing potential for triode 17 is supplied by a power pack of conventional form through load resistor 31. The power pack includes power transformer 25 connected to power line 22, double wave rectifier 26, filter 27, VR tubes 28, 29, and resistor 30.

The output of triode 17 is impressed through resistors 19, 19 and condensers 18, 18 upon the control grids of thyratrons 20, 20, which are normally biased to cut-off by E. M. F. sources 23, 23. The plates of the thyratrons 20, 20, are supplied with potential from transformer 21 in the conventional way with a filament winding thereon also serving at Z, Z to supply heating current to the filaments of tubes 17, and 20, 20. The transformer 21 receives its power from the power line 22. When the ion beam intensity increases, the voltage output of the triode 17 increases, and at a predetermined magnitude, fires the thyratrons 20, 20 which act as electronic switches applying line voltage from the output of transformer 21 to the gear shift solenoid F through lines 13, 24, for operating it in the manner heretofore described.

The peaks-in-out switch 32 may be employed to deflect the ion beam of the spectrometer away from the collector plates 2, 3 by an electrostatic field in a conventional manner (not shown) when the background readings are to be taken. Deactivation of the motor G, during this period, can be accomplished by relays 33, 34, a triode 35 and a pentode 36. The current for energizing the relay 34 is that which flows through the output circuit of triode 35. When switch 32 is closed, external voltage is applied through line 37 to the winding of relay 33 to energize it, causing the armature to move to front contact position, completing a circuit from ground through the front contact and armature to the control grid of tube 35. This operates, in view of the positive potential on the cathode, to reduce the conductivity or render the tube non-conductive, deenergizing the winding of relay 34 in the output circuit thereof, and permitting its armatures to fall to back contact position. This opens the power circuit through lines 14, 38 to the motor G, and serves to deactivate the motor. When the switch 32 is again opened, permitting the beam of the mass spectrometer to return to the collector plates 2, 3, the winding of relay 33 is de-energized and the armature returns to back contact position. However, by virtue of the time lag introduced by the large resistor 40, preferably of one megohm, and the condenser 41, preferably of 1 mfd, tube 35 does not immediately commence to conduct and relay 34 remains de-energized. This is due to the action of the resistor-capacitor combination which prevents the grid 35 from changing potential immediately, and delays its return to normal conduction. This in turn delays the operation of motor G and prevents the by-pass valve B of Fig. 1 from changing its setting until the collector plates of the mass spectrometer are fully charged and the output of the feed-back amplifier A has returned to normal. Further control is realized by connecting the control grid of the tube 36 through line 39 to the output of amplifier A for the application of potential therefrom to the tube, so that when the potential of this grid is raised to a predetermined value by an increase in the output of amplifier A, the plate current of tube 36 increases, thereby causing a

large voltage drop across the load resistor 42, of preferably 150,000 ohms in the plate circuit of the tube. Since the control grid of tube 35 is coupled thereto, this action will have the effect of reducing the conduction of tube 35 to a point at or near cut-off. This drop in flow of current across tube 35 will serve to de-energize the winding of relay 34 and permit the armatures thereof to fall to back contact position, and open the power circuit to the motor G. The stopping of motor G thus prevents the by-pass valve B from being turned to the end of its stroke when gas pressure is reduced to a low value.

In connection with the above circuit, it will be understood that power for energizing the plates of tubes 35, 36 is supplied through line 43 and switch 44 from the conventional power supply, referred to above. Appropriate heating current for the filaments for these tubes is supplied from a secondary winding 45 on transformer 25 through switch 44 as indicated at MN. Bias for tube 36 is regulated by inserting an adjustable cathode resistor 46 in the cathode circuit of the tube between the cathode and ground. Potential for the screen grid of pentode 36 and for raising the potential of the cathode of tube 35 to an appropriate biasing is supplied through a line 80 from the juncture of VR tubes 28, 29 of the power pack.

The valve which controls the input flow of gas by shunting part of it away from the mass spectrometer I may be similar to that set forth in the co-pending application of Herod, et al., Ser. No. 651,284, or it may take any other suitable form for the purpose.

Now referring to Fig. 3 showing one form of gear shift and drive mechanism used in our improved system for preventing hunting, F represents generally the solenoid for shifting the gears. It includes an outer coil 47 with an axial bore therethrough. Positioned in the bore is an armature or plunger 48 which is slidable axially or longitudinally of the bore. It also serves as a shaft for rotation, having a recess 49 in one end for the reception of a ball 50 therein. A cupped shaped member 51 is joined through a shank or extension to the ball 50 and serves to urge the ball against the plunger through the action of coil spring 52, having one end thereof seated in the cupped shaped member. The opposite end of the spring is seated in an adjustable cupped shaped retainer 53, having an adjusting screw 54 passing therethrough and secured to or engaging the end of spring 52. The adjusting screw 54 is maintained in adjusted position by a lock nut 55. The retainer 53 is, in turn, held in place by cross member 56 which receives it and which is joined to the solenoid case by screws 57, 57, or other appropriate means. The solenoid means F is secured to bracket 58 which is in turn mounted on frame 59 by adjusting studs or screws 60. Also mounted on the frame 58 is the driving motor G having a shaft 61 upon which is mounted driving gears or pinions 62 and 63 in spaced relation. These gears are permanently meshed with gears 64 and 65 which are rotatably mounted on plunger 48 and shaft 66. Shaft 66 is connected to and serves to drive by-pass valve B to closed position. One end of shaft 66 is enlarged and receives the reduced end of plunger 48 which is journaled in a conventional bearing in the frame and is connected to shaft 66 through key 67. Mounted on shaft 66 and plunger 48 at their juncture is a collar 68 which serves as one part of a positive duplex clutch. The collar or clutch ele-

ment is also secured in place on the shaft 66 and the plunger 48 by the ends of key 67 which pass through openings therein. The collar or clutch element 68 also carries pins or keys 69, 70 positioned in spaced relation about the circumference of the collar and which are adapted to seat in apertures 74, 73 or 71, 72 in mating clutch elements 75 or 76 carried by gears 64 and 65, respectively, depending upon the position of the plunger 48. If the relay F is energized so that the plunger is in the inner position and the collar 68 is interlocked with gear 64, it will be understood that the shaft 66 will rotate at relatively high speeds, due to the lower ratios of gears 62, 64. However, when solenoid F is de-energized, the spring 52 acting through ball 50 will force the plunger 48 outwardly or longitudinally to the right, disengaging collar 68 from clutch element 75 of gear 64 and bringing it into engagement with the clutch element 76 carried by gear 65. As pins 69, 70 seat in openings 72, 71 of the clutch element 76, the collar or clutch element 68 becomes interlocked with the clutch element 76 of gear 65. In this relation, the shaft 66 is locked to and rotates with gear 65, which, due to the relatively high ratios between gears 63 and 65, rotates at relatively lower speeds than when locked to rotate with gears 62 and 64.

Having thus described our invention, we claim:

1. A control system for an ion source comprising a mass spectrometer having an ion source and collector, an inlet for feeding vapors to said source, and means responsive to the flow of ion current to the collector of said spectrometer for regulating vapor flow to said source.

2. A control system for an ion source comprising a mass spectrometer having an ion source and collector, an inlet line for feeding sample gases to said source, a valve in said inlet line, and means responsive to ion current at the collector for actuating the valve to control the flow of gases through said line.

3. A control system for an ion source comprising a mass spectrometer having an ion source and a collector, an inlet line for feeding a gaseous mixture to said source, a valve interposed in said inlet line for regulating the flow of said gaseous mixture to the source, means for actuating said valve to open and closed position, and means responsive to ion current at the collector for controlling the operations of the actuating means.

4. A control system for an ion source comprising a mass spectrometer having an ion source and a collector, an inlet line for feeding sample gases to said ion source, a valve interposed in the inlet line for controlling the flow of gases to the source, a motor for actuating the valve to open and closed position, and means responsive to ion current at the collector for controlling the operation of the motor.

5. A control system for an ion source comprising a mass spectrometer having an ion source and a collector, an inlet for feeding gaseous mixtures to the source, and means responsive to large changes in ion current for rapidly adjusting the flow of said mixtures to said source and to small changes in ion current for slowly adjusting the flow of said mixtures.

6. A control system for an ion source comprising a mass spectrometer having an ion source and a collector, an inlet line for feeding a gaseous mixture to said source, a valve in said inlet line,

and means responsive to large changes of ion current at said collector for rapidly adjusting said valve and to small changes of ion current at the collector for slowly adjusting said valve to provide close regulation of the flow of said mixture.

7. A control system for an ion source comprising a mass spectrometer having an ion source and a collector, an inlet line for feeding a gaseous mixture to said source, a valve in said inlet line for regulating the flow of said mixture in said line, means for actuating said valve to open and closed position, and means responsive to large changes of ion current at said collector for controlling said actuating means to rapidly actuate said valve and to small changes of ion current at said collector for causing said actuating means to slowly actuate said valve.

8. A control system for an ion source comprising a mass spectrometer having an ion source and a collector, an inlet line for feeding sample gases to said ion source, a valve interposed in the inlet line for controlling the flow of gases to the source, a motor for actuating the valve to open and closed position, and means responsive to large changes of ion current at the collector for controlling the operation of the motor to rapidly actuate the valve and to small changes of ion current for controlling the operation of the motor to slowly actuate said valve.

9. A control system for an ion source comprising a mass spectrometer having an ion source and a collector, an inlet for feeding vapors to said source, means responsive to the flow of ion current to the collector of said spectrometer for regulating vapor flow to said source, and means responsive to cessation of ion current for suspending operation of said regulating means.

10. A control system for an ion source comprising a mass spectrometer having an ion source and a collector, an inlet line for feeding a gaseous mixture to said source, a valve in said inlet line, means responsive to ion current at the collector for actuating the valve to control the flow of gases through said line, and means responsive to the cessation of ion current flow for preventing movement of said valve.

11. A control system for an ion source comprising a mass spectrometer having an ion source and a collector, an inlet line for feeding a gaseous mixture to the source, a valve interposed in said inlet line for regulating the flow of said gaseous mixture to the source, means for actuating said valve to open and closed position, means responsive to ion current at the collector for controlling the operation of the actuating means, and means responsive to cessation of the flow of ion current for rendering said actuating means inoperative.

12. A control system for an ion source comprising a mass spectrometer having an ion source and a collector, an inlet line for feeding sample gases to said ion source, a valve interposed in the inlet line for controlling the flow of gases to the source, a motor for actuating the valve to open and closed position, means responsive to ion current at the collector for controlling the operation of the motor, and means responsive to the cessation of ion current flow for rendering said motor inoperative.

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No references cited.