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FILAMENT PROTECTION FOR HOT WIRE DETECTORS

Filed July 29, 1966 2 Sheets-Sheet 1

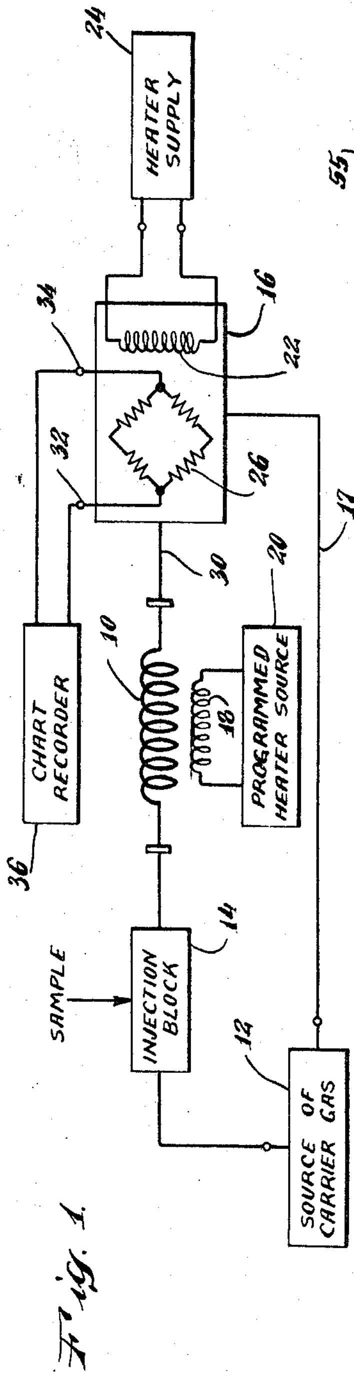

Fig. 1

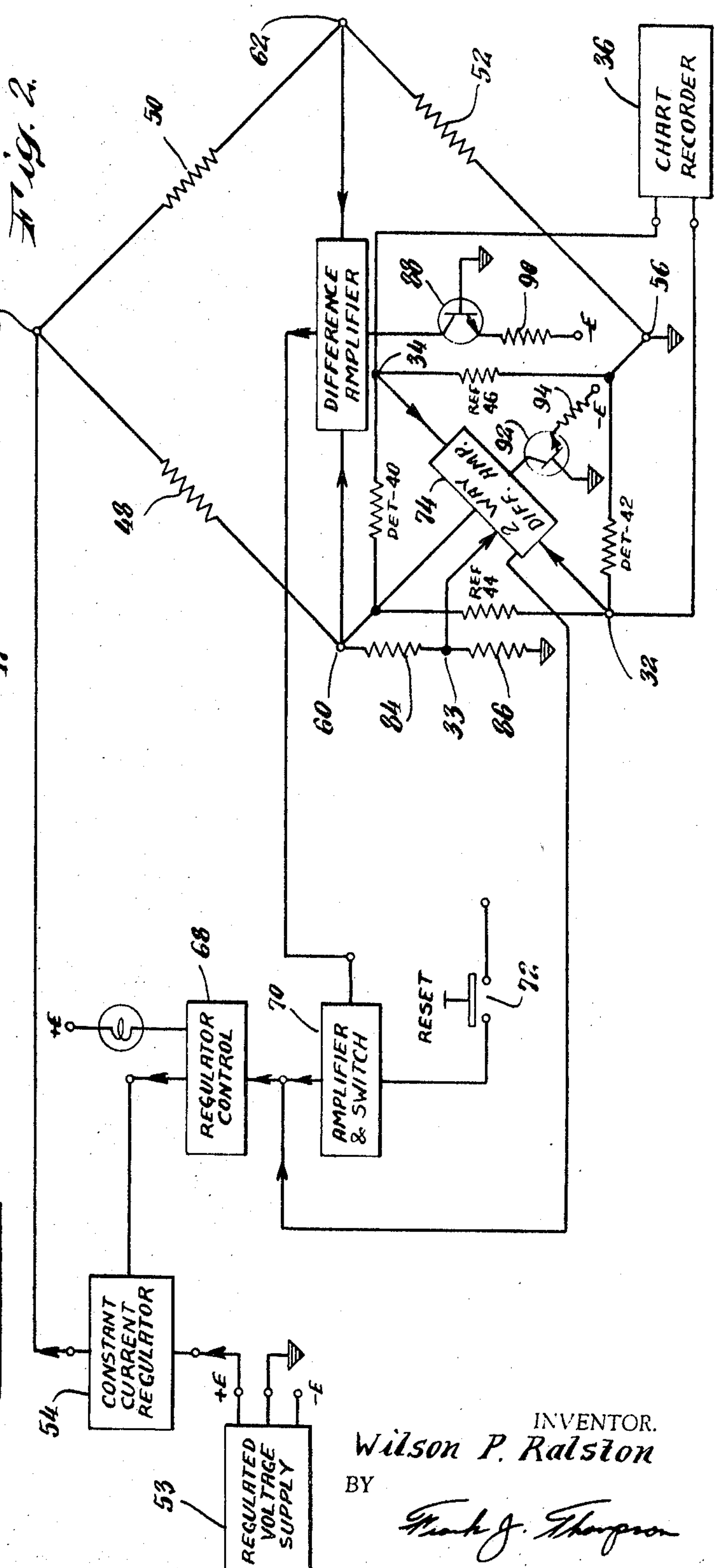

Fig. 2

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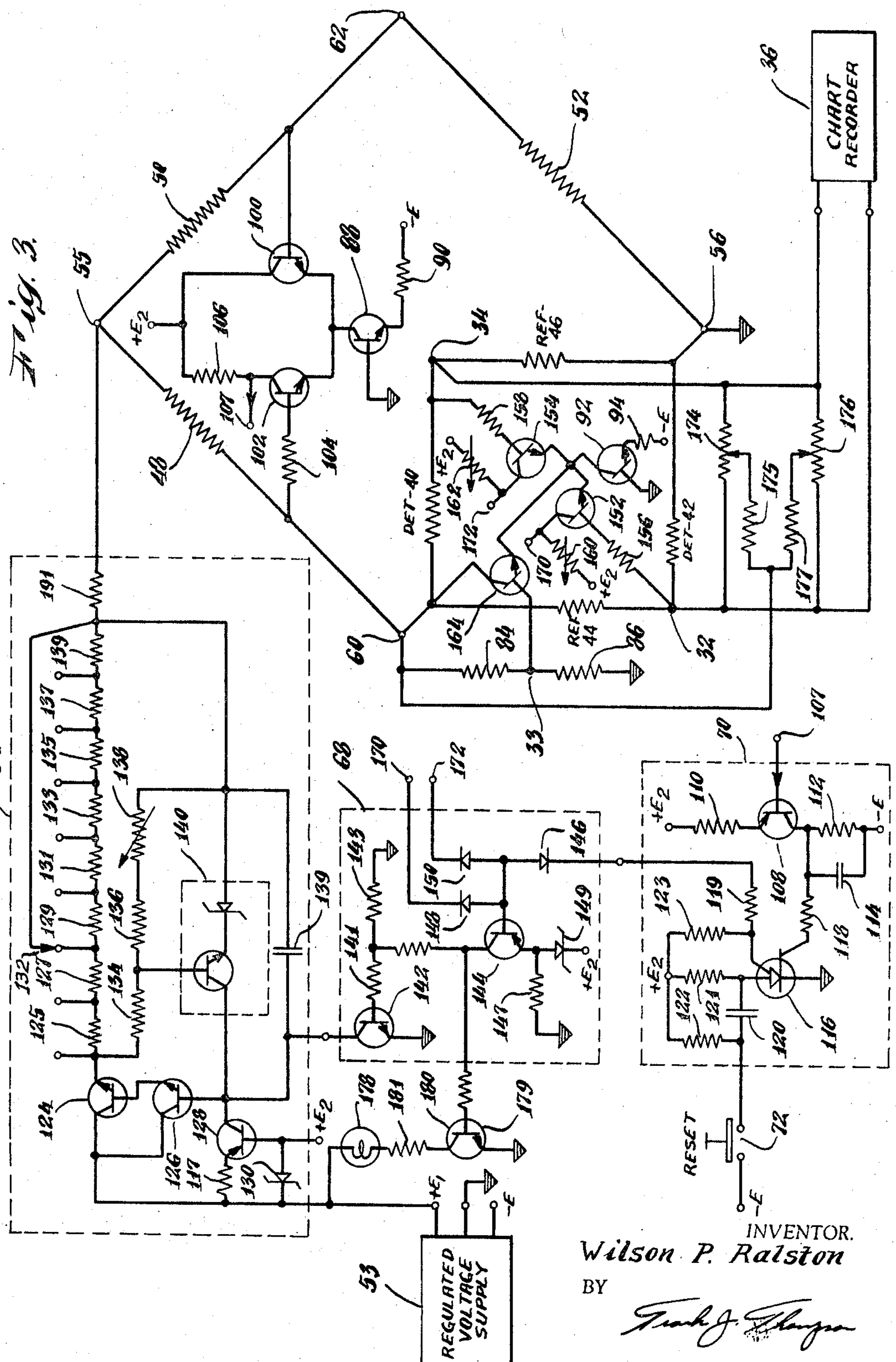

Fig. 3.

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FILAMENT PROTECTION FOR HOT WIRE DETECTORS

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ABSTRACT OF THE DISCLOSURE

A detector circuit arrangement includes a detector filament and a reference filament arranged in a balanced bridge circuit and means for causing a heating current to flow in the filaments. Circuit means are provided for sensing variations in the equivalent resistance (R_e) of the bridge circuit and for automatically reducing heating current to the bridge circuit when the resistance (R_e) exceeds a preselected limiting value (R_L). Circuit means are also provided for sensing resistance unbalance of detector and reference filaments and for automatically varying the amplitude of heating current flowing in the filaments in a manner for limiting the resistance ratio of the filaments. Through this arrangement a limit on the resistance of each filament is established thus inhibiting detector filament burnout.

This invention relates to detector arrangements employed with chromatographic apparatus. The invention relates more particularly to a thermal conductivity type of gas chromatographic detector.

Thermal conductivity detectors utilized with gas chromatographic apparatus generally include one or a plurality of detector wire filaments and one or a plurality of reference wire filaments mounted in a heated detector block and arranged in a balanced bridge circuit. Circuit means cause a heating current to flow in the filaments. A carrier gas flows from a source in the apparatus and is passed directly over the reference filaments while an output, including the carrier gas and separated components, from a separating column in the apparatus is passed over the detector filaments. In the absence of separated components, the flow of carrier gases establish a predetermined thermal conductivity between the filaments and a wall of the detection block. Upon separation by the column of a component of a sample being analyzed, the component is carried over the detector filaments and decreases thermal conductivity between the detector filaments and the detector wall. The decrease in thermal conductivity increases the temperature and thus the resistance of the detector filaments by an amount proportional to the concentration and inversely proportional to the thermal conductivity of the separated component. The bridge circuit thereby becomes unbalanced and an indication as to the quantity of a separated component is provided.

The sensitivity of the bridge circuit is greater when the average heating current flowing in the filaments is relatively large. However, a relatively large current can result in overheating and cause filament burnout. This is true even though the filaments are carefully wound and the resistance deviations between the filaments is held to practical manufacturing limits.

In a prior arrangement, detector filaments such as thermistors which exhibit a negative-temperature coefficient were employed. The resistance of the filament decreased upon overheating and filament power dissipation was thereby limited. However, filament materials having negative resistance-temperature coefficients are undesirably limited in their upper operating temperature. Since it is generally desirable to operate the detector block at a temperature equal to or greater than the temperature of the separating column, thermistor filaments find limited use. A "hot wire" form of thermal conductivity detector capable of operating at the desired elevated temperature and having filaments exhibiting positive resistance-temperature coefficients has been utilized. However, as previously employed these detectors required the operator to monitor and control the current applied to the filaments in order to prevent burnout. Generally, the operator controlled the detector current flow in a manner for operating with a substantial safety margin. This, however, undesirably reduced the operating sensitivity of the detector.

Accordingly, it is an object of the present invention to provide an improved arrangement for inhibiting filament burnout in a thermal conductivity detector.

Another object of the invention is to provide an arrangement for inhibiting filament burnout in a thermal conductivity detector of the type employing filaments having positive resistance-temperature coefficients.

A further object of the invention is to provide for the operation of a "hot wire" detector at maximum detector sensitivity while substantially reducing the possibility of filament burnout.

Still another object of the invention is to provide an improved arrangement for inhibiting filament burnout in a "hot wire" thermal conductivity detector and which eliminates the need for current monitoring and control by the operator.

In accordance with a feature of the present invention, a detector circuit arrangement includes a detector filament and a reference filament arranged in a balanced bridge circuit and means for causing a heating current to flow in the filaments. Circuit means are provided for sensing variations in the equivalent resistance (R_e) of the bridge circuit and for automatically reducing heating current to the bridge circuit when the resistance (R_e) exceeds a preselected limiting value (R_L). In accordance with another feature of the invention, circuit means are provided for sensing resistance unbalance of detector and reference filaments and for automatically varying the amplitude of heating current flowing in the filaments in a manner for limiting the resistance ratio of the filaments. Through this arrangement a limit on the resistance of each filament is established thus inhibiting detector filament burnout.

These and other objects and features of the present invention will be apparent with reference to the following specifications and drawings wherein:

FIGURE 1 is a diagram illustrating the general arrangement of a gas chromatographic apparatus employing a thermal conductivity detector;

FIGURE 2 is a diagram, partly in block and partly in schematic form, of a thermal conductivity detector and burnout inhibiting circuit arrangement constructed in accordance with features of the present invention; and

FIGURE 3 is a detailed schematic diagram of the circuit arrangement of FIGURE 2.

Referring now to FIGURE 1, a gas chromatographic apparatus with which the present invention is advantageously employed will be briefly described. The apparatus is shown to include a separating column **10**, a source of carrier gas **12**, and a means comprising an injector block **14** for injecting a sample having components which are to be separated into a carrier gas stream. The sample is carried through the column **10** by the carrier gas and flows to a detector means **16** for detecting the quantity of the separated components. The carrier gas also flows directly to the detector means **16** through suitable piping **17**. Means including a heater **18** and a source of heater power **20** are provided for causing programmed heating of the column **10** in a conventional manner.

The detector means indicated generally as **16** comprises a thermal conductivity detector having a filament mounting block, not specifically shown. The block is heated by heater coil **22** which is energized by a heater supply **24** for causing the block to operate at a desired temperature, generally higher than the highest temperature of the programmed column **10** and on the order of 400° C. Reference and detector filaments are mounted in the block of the detector **16** and are indicated generally by the bridge circuit **26**. In an alternative arrangement, single hot wire reference and detector filaments are arranged in a bridge circuit along with resistors of fixed value. The resistors do not perform temperature sensing functions. Output terminals **32** and **34** of the bridge are coupled to a chart recorder **36** or other desired recording means for recording in a desired manner the relative magnitudes of the detected peaks. As indicated previously, the carrier gas is passed directly over reference filaments of the detector **16** while the carrier gas and separated component of a sample are coupled to the block **16** via a tubulation **30** and passed over the detector filaments.

In FIGURE 2, the detector bridge circuit including the detector and reference filaments is illustrated in greater detail in a circuit arrangement adapted for inhibiting burnout of the filaments. The detector bridge circuit comprises the detector filaments **40** and **42** forming opposite arms of a balanced bridge circuit and the reference filaments **44** and **46** also forming opposite arms of the bridge circuit. In accordance with a feature of the present invention, circuit means are provided for sensing an increase in the equivalent resistance (R_e) of this bridge circuit, and for reducing the current to the bridge circuit when (R_e) exceeds a pre-established limiting value (R_L). In an arrangement for performing this function, the detector bridge circuit itself is coupled as one arm of another bridge circuit, referred to hereinafter as the limiting bridge circuit. The limiting bridge includes the impedance arms comprising resistances **48**, **50** and **52**. A filament heating current is derived from a voltage source **53** and is applied to the limiting bridge via a constant current regulator **54** and the terminals **55** and **56**. The impedance values of resistances **48**, **50** and **52** establish an initially first unbalanced limiting bridge condition when (R_e) is less than (R_L), a balanced limiting bridge condition when (R_e) equals (R_L), and a second unbalanced limiting bridge condition when (R_e) exceeds (R_L). The first unbalanced condition, for example, establishes output sensing terminal **62** relatively positive with respect to terminal **60** while terminal **62** is relatively negative with respect to terminal **60** when the second unbalance condition occurs. The relationships between the bridge impedances for a balanced condition are expressed by:

$$(R_e)=(R_L)=\frac{R_{48}\times R_{52}}{R_{50}} \qquad (1)$$

A differential amplifier **58** is coupled between the output sensing terminals **60** and **62** of the limiting bridge and is arranged to provide an output voltage indication when (R_e) exceeds the pre-established limiting value (R_L).

The detector and reference filaments are carefully wound during manufacture and closely matched within reasonable manufacturing tolerances. Their cold resistance is generally on the order of 30 ohms. The flow of filament heating current causes the filament resistance to increase and the power dissipated by these filaments increases accordingly. An increase in resistance beyond some safe value increases the likelihood of burnout. A safe equivalent resistance (R_e) equal to (R_L) for the detector bridge can thus be determined. For example, and in a typical case, the filaments may exhibit hot resistances providing a safe equivalent resistance (R_e) up to 80 ohms at a temperature of 550° C. The equivalent limiting resistance (R_e) of the detector bridge is thus established and the resistances **48**, **50** and **52** can be selected to satisfy the Equation 1 and to satisfy other circuit requirements.

When a limiting condition is attained, the limiting bridge becomes balanced, and the balance is sensed by the differential amplifier **58**. Power to the detector bridge circuit is automtaically reduced as (R_e) exceeds (R_L). As shown in FIGURE 2, and as previously indicated, power is derived from the supply **53** and is applied to the detector bridge circuit via the constant current regulator circuit **54**. This regulator operates in a conventional current regulating mode when (R_e) is less than (R_L). However, as (R_e) exceeds (R_L), the regulator current amplitude is controlled by a control circuit **68**. The differential amplifier **58** provides an output voltage indication which is applied to an amplifier and switch circuit **70**. In a particular arrangement, the amplifier and switch circuit cause the regulator control **68** to operate on the constant current regulator **54** and interrupt current to the bridge circuit. The amplifier and switch circuit **70** operates as a manually resettable bi-stable device for controlling the regulator. A reset button **72** is provided for reapplying power to the bridge circuit after power has been interrupted.

Although the equivalent resistance (R_e) of the detector bridge circuit is sensed to determine the relationship of its magnitude to a preselected value of resistance (R_L), it would be possible for the individual filaments to exhibit a combination of excessively high and low resistance values while the equivalent resistance (R_e) remains less than the limiting value (R_L). In accordance with another feature of the invention, circuit means are provided for sensing the magnitude of detector bridge unbalance in excess of the magnitude of unbalance for a pre-established sample concentration and for varying the current flow to the detector bridge circuit in a manner for limiting the excessive unbalance. This circuit means includes a differential amplifier **74** and means comprising resistances **84** and **86**, the impedances of which are selected for establishing a limit for filament unbalance. When the ratio of detector filament resistance to reference filament resistance or, conversely, reference filament resistance to detector filament resistance exceeds the ratio R_{86}/R_{84} a limiting condition is established, i.e.,

$$\frac{R_{\text{det.}}}{R_{\text{ref.}}}\geq\frac{R_{86}}{R_{84}};\ \frac{R_{\text{ref.}}}{R_{\text{det.}}}\geq\frac{R_{86}}{R_{84}} \qquad (2)$$

An exemplary value for the ratio R_{86}/R_{84} is 1.06 which provides limiting for concentrations greater than 10% for an air sample in a helium carrier gas. The differential amplifier **74** then functions to reduce the current flow to the detector bridge by an amount sufficient to reduce filament heating and reduce the detector filament-reference filament resistance ratio to within the limiting range. Output voltages of the differential amplifier **74** are coupled to the regulator control **68** for effecting the desired reduction in heating current.

In FIGURE 3, a particular circuit arrangement of the filament burnout inhibiting means of FIGURE 3 is illustrated. Elements in FIGURE 3 which perform similar functions as elements in FIGURE 2 bear similar reference numerals. The limiting bridge differential amplifier **58** is shown to comprise NPN transistors **100** and **102** having emitter electrodes connected to a collector electrode of a transistor **88** operating as a constant current source. Operating voltages are established for maintaining transistor **102** in a collector current cutoff state when (R_e) is less than (R_L). An increase in the equivalent resistance (R_e) of the detector bridge circuit to and in excess of the value (R_L) causes a voltage at terminal **60** to increase in a positive sense and results in a corresponding flow of collector current in the transistor **102**. The varying voltage at collector electrode of transistor **102** is applied via an output terminal **107** to the amplifier and switch stage **70**. This stage, described in greater detail hereinafter, is indicated in FIGURE 3 by the circuit within the dotted rectangle **70**.

The detector bridge differential amplifier **74** employs a common reference transistor, **164**. One differential amplifier including transistors **152** and **164** senses the voltage between output terminals **32** and **33** while the other differential amplifier including transistors **154** and **164** sense the voltage between terminals **34** and **33**. When the voltage at either of terminals **32** or **34** becomes more positive than the voltage at terminal **33**, the collector current of the differential amplifier increases and a limiting action is initiated. Voltages occurring at output terminals **170** and **172** of these amplifiers are applied to the regulator control circuit **68** and the detector bridge current is reduced by an amount sufficient for maintaining the pre-established filament resistance ratio.

Considering the operation of the detector bridge differential amplifier in greater detail, as terminal **32** becomes more positive than terminal **33**, then transistors **152** and **164** function together as a differential amplifier and collector current flows in transistor **152**. Collector voltage is applied via output terminal **170** and isolating diode **148** to the base of transistor **144** of the regulator control circuit. Similarly, as terminal **34** becomes more positive than terminal **33**, the transistors **154** and **164** function as a differential amplifier and an increase in the collector current of transistor **154** results. The collector output voltage is applied via terminal **172** and isolating diode **150** to the regulator control circuit **68**. The magnitudes of collector voltage variations of transistors **152** and **154** correspond to the degree of bridge unbalance. These voltages cause the regulator control **68** to reduce the amplitude of heating current flowing by an amount sufficient for maintaining the pre-established filament resistance ratio. In order that the differential amplifiers **58** and **74** have high common mode rejection and operate independently of the quiescent bridge current, the constant current regulators are provided. A first regulator for difference amplifier **58** comprises the ground base transistor **88** which is returned to a relatively negative voltage —E through a resistor **90**. A second regulator for difference amplifier **74** comprises a grounded base transistor **92** which is also returned to a relatively negative voltage by a resistor **94**. Adjustable impedances in the circuit are provided for bridge balancing and transistor compensation. A detector bridge balancing means comprises the fine and course adjusting potentiometers **174** and **176** respectively. The adjustable resistances **160** and **162** are provided for compensating for β variations of the transistors.

The amplifier of the switch circuit arrangement **70** comprises a PNP transistor **108** arranged as a common emitter circuit for inverting the signal polarity. The collector resistor **112** is returned to a source of potential —E and is by-passed by a capacitor **114**. Collector current flow in transistor **102** indicating a limiting condition causes collector current flow in transistor **108**. When a voltage developed across resistor **112** reaches the value of E, the gate terminal of a silicon controlled switch **116** is driven positive and the switch is triggered on. A negative going voltage occurs across resistor **123** and is applied to the regulator control **68** through an isolating diode **141**. The regulator control circuit **68** responds in a manner for controlling current flow to the bridge. The silicon control switch **116** is a bi-stable device and is required to be reset. A switch **72** is provided which, through capacitor **120**, momentarily clamps the anode of the SCS **116** to a potential —E. This arrangement turns off current flow in the SCS **116**. The SCS **116** then remains in an off condition until retriggered. It will be retriggered automatically if the bridge resistance remains above the limiting resistance ($R_e > R_L$); i.e., if the filament fault condition has not been corrected.

The constant current regulator **54** of FIGURE **2** is indicated in FIGURE 3 by the circuit arrangement within the dotted rectangle. This regulator is conventional and includes the series regulating transistors **124** and **126**. A transistor **128** supplies a current to the base circuit of the transistor **126**. Zener diode **130** establishes a fixed bias voltage for the base of **128** and in addition is used to drop $+E_1$ to a lower $+E_2$ for use in other circuits. Variations in the amplitude of output heating current will cause variations in the voltage across resistor **134** of the regulator circuit. A control amplifier indicated generally as **140** and including a transistor and reference voltage Zener diode responds to this variation and alters the magnitude of base current being applied to the base of transistor **126** by the transistor **128** in order to accomplish the current regulating function. The regulator will maintain a nominal constant heating current output which is initially determined by an operator by adjusting the tap **132**. The tap is adjusted to include a desired number of resistances from the group including resistors **125, 127, 129, 131, 133, 135, 137** and **139** which are connected in series in the emitter circuit of transistor **124**.

The regulator control circuit **68** of FIGURE 2 shown within the dotted rectangle of FIGURE 3 operates, responsive to a voltage from the amplifier and switch circuit **70** or the outputs of the detector bridge differential amplifier **74**, to control the magnitude of heating current. A transistor **142** of this regulator circuit is coupled to the base of transistor **126** of the regulator circuit, **54**. This transistor operates to vary the base current to the regulator transistor **126**, in accordance with a control voltage applied to its base from transistor amplifier **144**. A limiting condition indicator lamp **178** is also provided and is energized by transistor **144** when the regulator control circuit **68** causes an interruption or reduction of the current to the detector bridge circuit.

In one form of the circuit of FIGURE 3, the following components values, which are not construed to limit the invention in any aspect, were found to provide satisfactory operation.

Transistors

88, 92, 100, 102, 126, 142, 152, 154, 164, 180—type 2N697
108, 128, 144—type 2N3250
116—type 3N58 silicon controlled switch
124—type 2N1485
140—General Electric reference amplifier, type RAIA

Diodes

130, 149—Zener IN3828A, 6.2 v.
146, 148, 500—IN4004

Voltages

$+E_1=+45$ v.; $+E_2=+38$ v.; $-E=-6.2$ v.

Resistors

R48—22Ω, 10 w., 1%	R134—1.2K
R50—2.2K, 1%	R135—10Ω, 1%
R52—8.0K, 1%	R136—3.3K
R84—6K, ½%	R137—16Ω, 1%
R86—6.4K, ½%	R138—20K adjustable
R90—5.6K	R139—20Ω, 1%
R94—3.3K	R141—10K
R104—4.7K	R143—1.2K
R106—18K	R145—4.7K
R110—8.2K	R147—8.2K
R112—10K	R156—4.7K
R117—2.7K	R158—4.7K
R118—10K	R160—20K
R119—15K	R162—20K
R121—15K	R174—10K adjustable
R122—1 Meg.	R175—390K
R123—15K	R176—2K adjustable
R125—1.8Ω, 1%	R177—1.2K
R127—2.0Ω, 1%	R179—4.7K
R129—2.2Ω, 1%	R181—470Ω, 2w
R131—6.2Ω, 1%	R191—22Ω
R133—7.5Ω, 1%	

Capacitors

114—0.47 μf.; **120**—.22 μf.; **139**—.22 μf.

Thus, an arrangement has been described which advantageously inhibits burnout of positive resistance-temperature coefficient filaments in a thermal conductivity detector and which eliminates the need for an operator to monitor current flow and to make corresponding adjustments therein.

While I have illustrated and described particular embodiments of my invention, it will be understood that various modifications may be made therein without departing from the spirit of the invention and the scope of the appended claims.

I claim:

1. In a thermal conductivity detector,
a detector bridge circuit arrangement including a detector filament and a reference filament each having a positive resistance-temperature coefficient,
circuit means coupled to said bridge circuit for causing a current to flow in said filaments,
said detector bridge having an equivalent resistance (R_e) which increases as the resistance of a filament increases,
circuit means coupled to said bridge circuit for sensing when the equivalent resistance (R_e) exceeds a pre-established limiting value and for causing a reduction in the amplitude of filament current flow when the equivalent resistance (R_e) exceeds the pre-established limiting value, and
circuit means coupled to said bridge circuit for sensing when a ratio of filament resistances exceeds a pre-established value and for causing a reduction in the amplitude of filament current flow when the ratio of filament resistances exceeds the pre-established value.

2. In a thermal conductivity detector,
a detector bridge circuit arrangement including a detector filament and a reference filament each having a positive resistance-temperature coefficient,
circuit means for applying a heating current to said filaments,
said bridge circuit having an equivalent resistance (R_e) which increases as the resistance of a filament increases, and
circuit means coupled to said bridge circuit for sensing when the equivalent resistance (R_e) exceeds a pre-established limiting value and for reducing the amplitude of current applied to the detector bridge circuit when the equivalent resistance (R_e) exceeds the preselected value.

3. In a thermal conductivity detector,
a balanced detector bridge circuit including a first resistive detector filament and a first resistive reference filament each having a positive temperature coefficient and arranged as arms of the bridge circuit,
said detector bridge circuit having an equivalent resistance (R_e),
impedance means arranged as an initially unbalanced limiting bridge circuit and including said detector bridge circuit as an arm thereof,
said limiting bridge having sensing terminals providing voltages therebetween having relative amplitudes indicative of unbalance in a first sense when (R_e) is less than a preestablished limiting value (R_L) and unbalance in an opposite sense when (R_e) is greater than (R_L),
means for applying a heating current to said filaments, and
circuit means coupled between said sensing terminals and arranged for reducing filament heating current when said limiting bridge becomes unbalanced in the second sense.

4. In a thermal conductivity detector,
a detector bridge circuit including a detector filament and a reference filament each having a positive resistance-temperature coefficient,
circuit means coupled to said bridge circuit for causing a heating current to flow in said filaments, and
circuit means coupled to said bridge circuit for sensing when a ratio of detector and reference filament resistances exceeds a pre-established value and for controlling the heating current in a manner for limiting the ratio of detector and reference filament resistances to a pre-established value.

5. In a thermal conductivity detector,
first and second resistive detector filaments and first and second resistive reference filaments arranged as opposite arms of a balanced detector bridge circuit,
said bridge circuit having an equivalent resistance (R_e),
circuit means for applying a filament heating current to said filaments,
circuit means including a differential amplifier coupled between arms of said detector bridge and arranged for detecting an unbalance of said detector bridge circuit and varying the heating current in a manner for limiting a ratio of detector to reference filament resistances to a pre-established value,
circuit means including first, second and third resistances and said detector bridge circuit arranged as a limiting bridge circuit, and
circuit means including a differential amplifier coupled between arms of said limiting bridge circuit for detecting when (R_e) exceeds a pre-established value (R_L) and for interrupting filament heating current.

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