

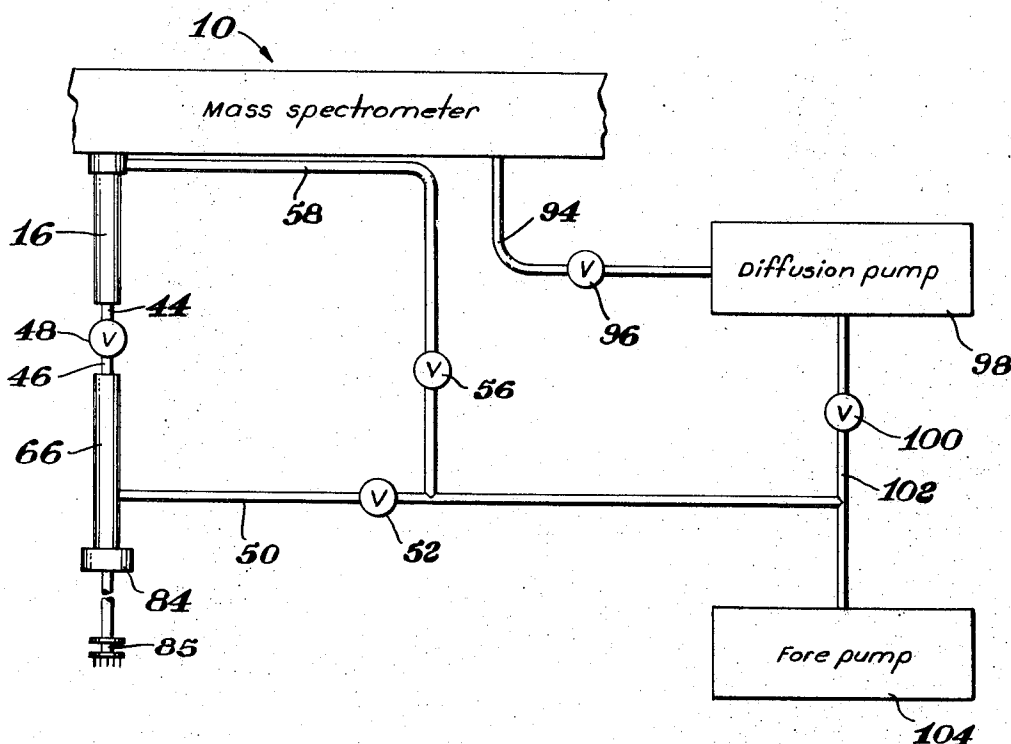
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[56] **References Cited**
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[54] **FURNACE ASSEMBLY FOR THERMAL ANALYSIS USE**
4 Claims, 4 Drawing Figs.

[52] U.S. Cl. 13/31,
250/41.9
[51] Int. Cl. H05b 3/26
[50] Field of Search 13/31, 22,
25; 250/41.9 C, 41.9 S, 41.9 SR

ABSTRACT: This invention relates to a heating furnace assembly which is adapted to be coupled to and become a part of a mass spectrometer adjacent to the ion source (usually) within the instrument. The furnace assembly comprises two furnaces in end-to-end relationship with sealing means between the furnaces and means for transmitting gas from one furnace to another. Each furnace is adapted to be controlled by means of temperature sensing which is adapted to be disposed within the furnace assembly.



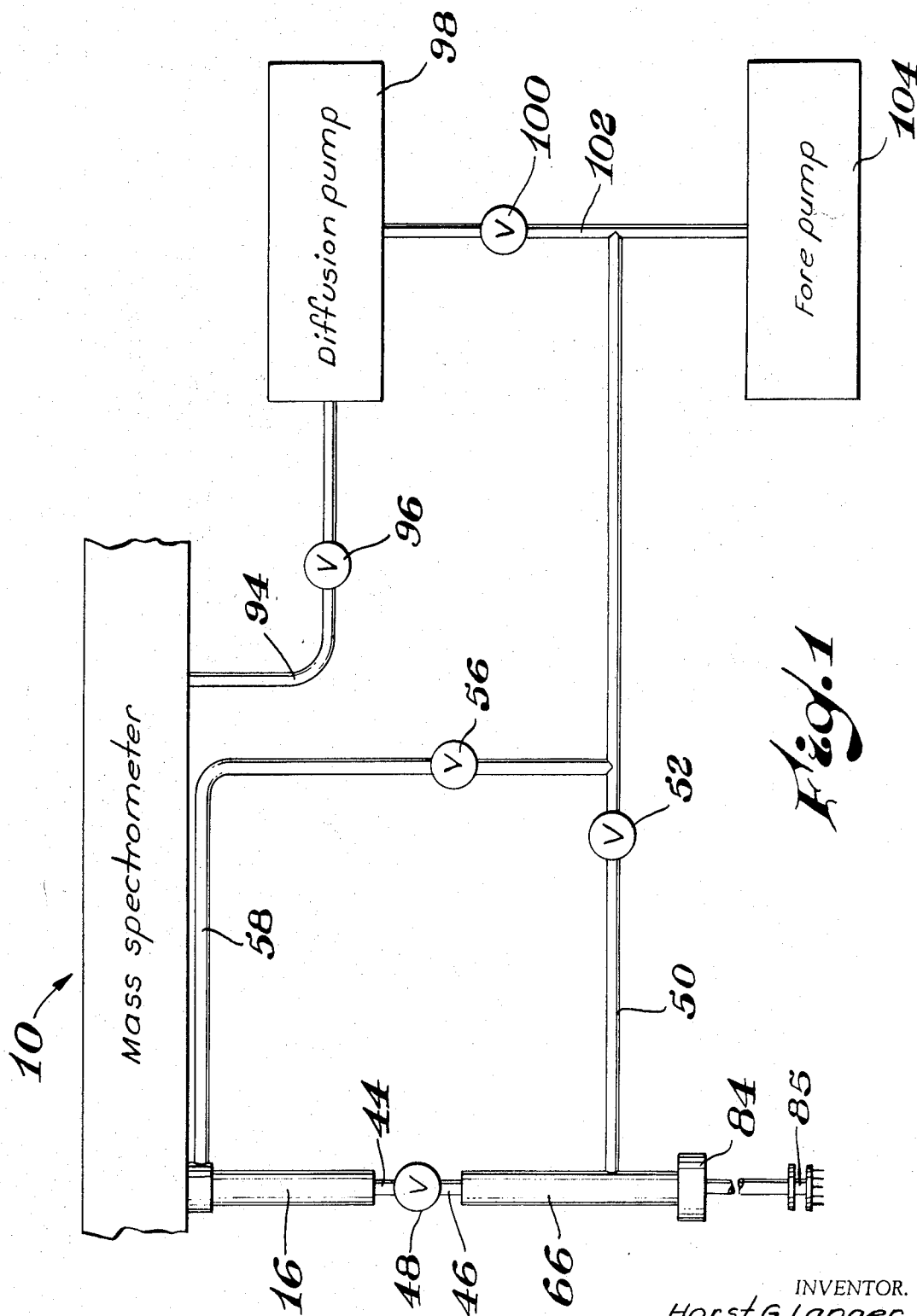


Fig. 1

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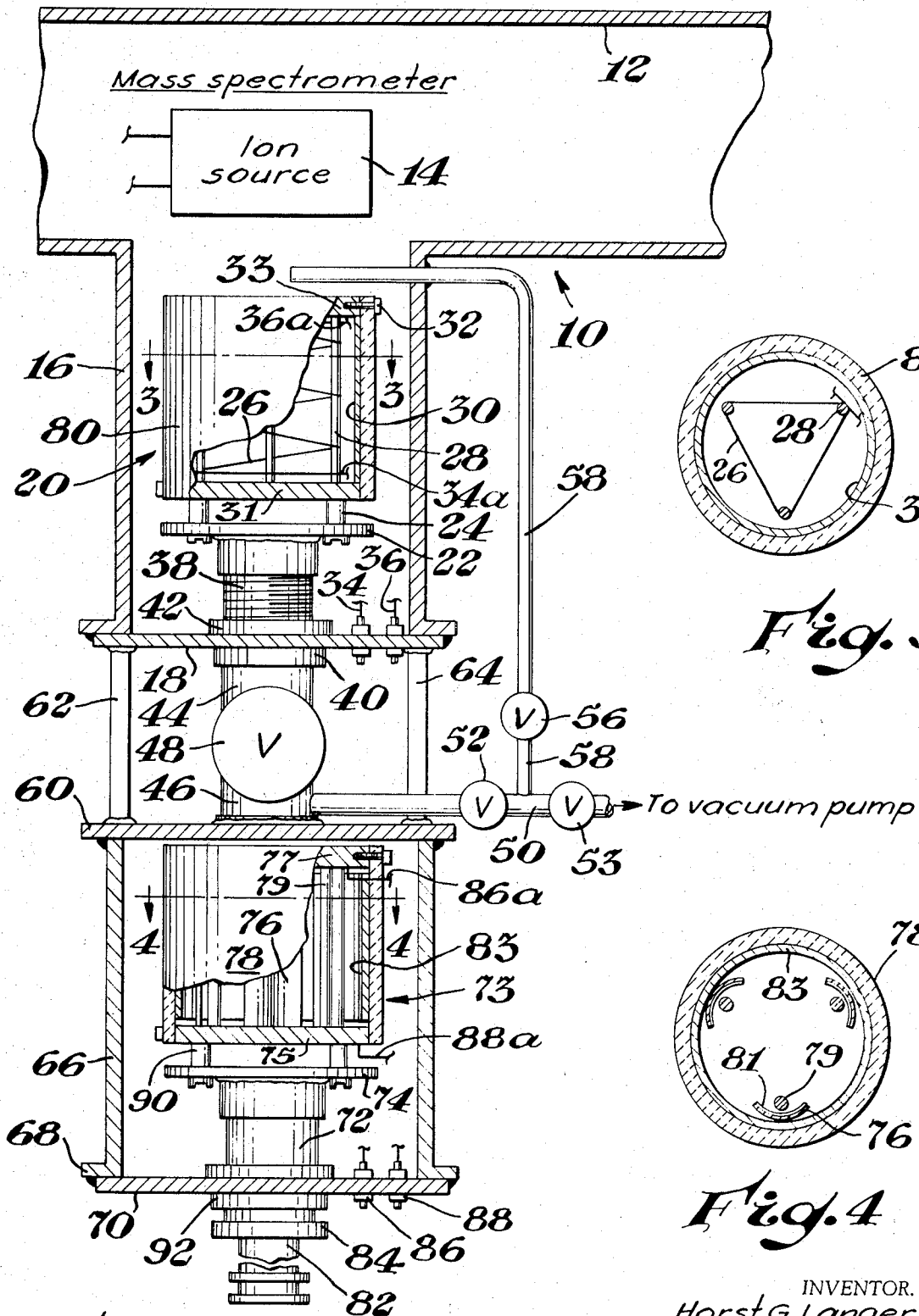


Fig. 2

Fig. 3

Fig. 4

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FURNACE ASSEMBLY FOR THERMAL ANALYSIS USE

BACKGROUND OF THE INVENTION

This invention relates to furnace assemblies adapted to receive a thermal analysis cell and particularly to furnace assemblies which are adapted for use in a mass spectrometer.

Accordingly, a principal object of this invention is to provide an improved thermal analysis cell receiving and heating assembly for use under high vacuum conditions.

Another object of this invention is to provide an improved radiant heating furnace and thermal analysis cell receiving assembly for use under high vacuum conditions in a mass spectrometer.

A further object of this invention is to provide a furnace assembly wherein means are provided to meter gaseous thermal reaction products into mass spectrometer.

Many spectrometers are sometimes equipped with devices which allow the heating of samples within the confinement of the mass spectrometer vacuum or within the ion source, and such devices sometimes also allow the ion source, and such devices sometimes also allow the measurement of sample temperatures during the heating process and are used in the operation known as thermal analysis. A problem arises in the use of such devices, however, because some samples emit a great volume of gases and thus overburden the vacuum system of the mass spectrometer.

In accordance with this invention, there is provided an enclosed furnace assembly comprising two furnaces disposed in end-to-end relationship and which are cylindrical in overall configuration and is coupled to a valved tubular thermal analysis cell input and sealing assembly.

The furnaces each have internal structure defining, in effect, an axial concentric bore with, usually, a reflective inner surface extending therethrough which is concentrically aligned with the cell input and sealing assembly. Means are provided to introduce part or all of the thermal reaction or decomposition products from a thermal analysis cell in that furnace to a point near the gas outlet end of the second or inner furnace.

The assembly is inserted into a mass spectrometer adjacent to the ion source.

The invention, as well as additional objects and advantages thereof will best be understood when the following detailed description is read in connection with the accompanying drawing, in which:

FIG. 1 is a diagrammatical view showing apparatus in accordance with this invention coupled to a mass spectrometer, and

FIG. 2 is a side elevational view, partly in section, of a furnace and cell probe insertion and sealing assembly in accordance with this invention;

FIG. 3 is a sectional view taken along the line 3—3 of FIG. 2, and

FIG. 4 is a side elevational view taken along the line 4—4 of FIG. 2.

Referring to the drawing, and particularly to FIG. 1, there is shown a mass spectrometer, indicated generally by the numeral 10, having a tubular section 16 extending therefrom sections 44, 46 of reduced diameter, in end-to-end relationship, are joined together by valve 48 which is usually a ball-type vacuum sealing valve.

One end of the section 44 is joined to the outer end of the section 16. The other end of the section 46 is coupled to one end of a tubular section 66 which is usually similar in diameter to the section 16. A compression type coupling 84 is at the outer end of the section 66, and is adapted to receive an elongated thermal analysis cell assembly 85 therethrough and seal about said cell.

A vacuum line 50 having valves 52, 53 therein is coupled at one end to the section 66 and to line 102 which is coupled to a fore pump 104 and through a valve 100 to a diffusion pump 98. The end part of section 16 is coupled to the line 50

between valves 52 and 53 through the line 58 and valve 56. The mass spectrometer 10 is coupled to the diffusion pump 98 through line 94 and valve 96.

Referring now to FIG. 2, 3, and 4, there is shown mass spectrometer apparatus, indicated generally by the numeral 10, comprising a flight tube 12, an ion source 14, and a perpendicularly disposed tubular member 16 having a flange at its outer end coupled to the flight tube 12 and facing the ion source 14. The flanged end of the member 16 is closed by a plate 18 which is sealed to the flanged end.

A tubular section 44 is coupled to the plate 18 by coupling 40 and seal nut element 42.

A ball valve 48 is coupled to the one end of a tubular section 46. The other end of tubular section 46 is coupled and sealed to a transversely disposed plate 60.

Support rods 62, 64 also help mechanically retain the plates 18, 60 in fixed position with respect to each other.

A flanged tubular member 66 similar to the member 16, is sealed to the plate 60 in axial alignment with the member 16 and tubular elements 44, 46.

A plate 70 covers the end of the member 66 having the flange 68 and is sealed to the flange.

A furnace element 72 having a compression seal fitting 84 at one end extends through the plate 70 and is sealed thereto by coupling means 92. An annular plate 74 having an inner diameter at least as large as the inner diameter of the element 72 is secured to the upper end of the element 72.

A furnace member, indicated generally by the numeral 73 is supported from the plate 74 by means of spacer elements 90.

The furnace 73 comprises spaced apart upper and lower annular plates 75, 77, an electrically insulating cover 78 which surrounds the space between the plates 75, 77, and heating bar and heat-reflecting elements 79, 76 respectively disposed in axial alignment between the plates 75, 77.

The heating elements 79 are energized through electrical leads 86a, 88a, which are coupled to the metal plates 77, 75 respectively. Feed through coupler 86, 88 permit connecting the leads 86a, 88a, to an external power source. The heat reflectors 76 are made of an electrically insulating material which has a heat-reflecting coating 81 thereon. A heat-reflective coating 83 may also be provided on the internal surface of the cover 78.

A thermal analysis cell 82 is shown inserted through the compression seal element 84 and into the tubular element 72.

A tubular element 38 within the member 16 is coupled to the seal element 42 and carries secured at its upper end a perpendicularly disposed annular plate 22 whose inner diameter is at least as large as the inner diameter of the tubular element 38.

A furnace assembly, indicated generally by the numeral 20, is supported from the annular plate 22 by spacer elements 24.

The furnace assembly 20 comprises upper and lower annular elements 31, 33 cover 80 which encloses the space between the elements 31, 33 and is secured to them (as by screw 32), insulating support rods 29 coupled to elements 31, 33 and a spirally wound heater winding 26 which is disposed around the rods 29. Leads 34a, 36a from the winding 26 are brought through 18 by feed through elements 34, 36 respectively.

The cover 80 may have a heat reflective coating 30 on its inner surface.

A vacuum line 50 having valves 52, 53 therein is coupled to the tubular element 46 near the top of the furnace assembly 73.

A line 58 having a valve 56 therein is coupled to the line 50 between valves 52, 53 and extends through the part of the tubular member 16 which is adjacent to the flight tube 12. The end of the line 58 terminates adjacent to the central part of the upper end of the furnace assembly 20.

The furnace assembly 20 may be, for example, the type disclosed and claimed in Horst G. Langer's copending application Ser. No. 840,363, for "Furnace Assembly for Differential Thermal Analysis Use," filed July 9, 1969.

The furnace assembly 73 may be, for example, the type disclosed in Horst G. Langer's copending application Ser. No. 848,232 filed Aug. 7, 1969 entitled "Furnace Assembly for Thermal Analysis Use."

In operation, with the valves 48 and 56 closed, a thermal analysis cell in position in the furnace assembly 73 and sealed by compression seal 84, valves 52 and 53 are opened and the vacuum pump(s) started. When the interior of tubular member 66 and tubular element 46 are suitably evacuated, the furnace is energized, valve 56 is opened and valve 53 is closed, permitting gas to flow through tube 58 to a location near the ion source 14.

In event a substantial amount of gas is released from the sample material in cell 82, valve 53 is opened and valve 56 is partially closed to restrict gas flow through the line 58.

The amount of opening and degree of closing of valves 53, 56 respectively, will depend on operating conditions encountered and will be adjusted as needed by those skilled in the mass spectroscopy art.

After the large amounts of gas have been given off by the sample material, the valve 48 is opened and the cell 82 is advanced through the valve 48 into the furnace assembly 20.

Furnace assembly 20, adapted for use under more or less vacuum conditions, is energized by a wire wound heater 26, whereas the furnace assembly 73 is heated, for example, by so called glowbar-type elements 79 which operate well under both vacuum and nonvacuum conditions.

The heating winding 26, if not already energized to preheat the furnace area, is energized and valves 52, 53, and 56 are closed because the relatively minor amounts of gas emitted by

the sample material may now enter the mass spectrometer without unduly taxing its vacuum system.

What is claimed is:

1. A furnace and sample insertion assembly for use under high vacuum conditions, comprising a first furnace and a second furnace, said furnaces being disposed in spaced apart end to end relationship in axial alignment with each other, each of said furnaces having heating means surrounding a central open part which is adapted to receive a generally cylindrical cell structure carrying material which emits gas when heated, valved tubular means sealed between and axially aligned with said furnaces, valved tubular input means coupled to and axially aligned with said first furnace, said first furnace and said second furnace each being enclosed in a gastight enclosure, said valved tubular means passing through each enclosure and said valved tubular input means passing through said enclosure of said first furnace, means for withdrawing gas from said enclosure of said first furnace and means for introducing at least a part of said withdrawn gas to said enclosure for said second furnace.

2. An assembly in accordance with claim 1, wherein said means for withdrawing gas comprises valved tubular means adapted to be coupled to a vacuum system.

3. An assembly in accordance with claim 1, wherein said means for introducing gas comprises tubular means coupled to said means for withdrawing gas.

4. An assembly in accordance with claim 1, wherein said furnaces have dissimilar heating means.

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