United States Patent

[11] **3,560,627**

[72] Inventor **Horst G. Langer**
Wayland, Mass.

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[73] Assignee **The Dow Chemical Company**
Midland, Mich.
a corporation of Delaware

[54] **FURNACE ASSEMBLY FOR THERMAL ANALYSIS USE**
6 Claims, 3 Drawing Figs.

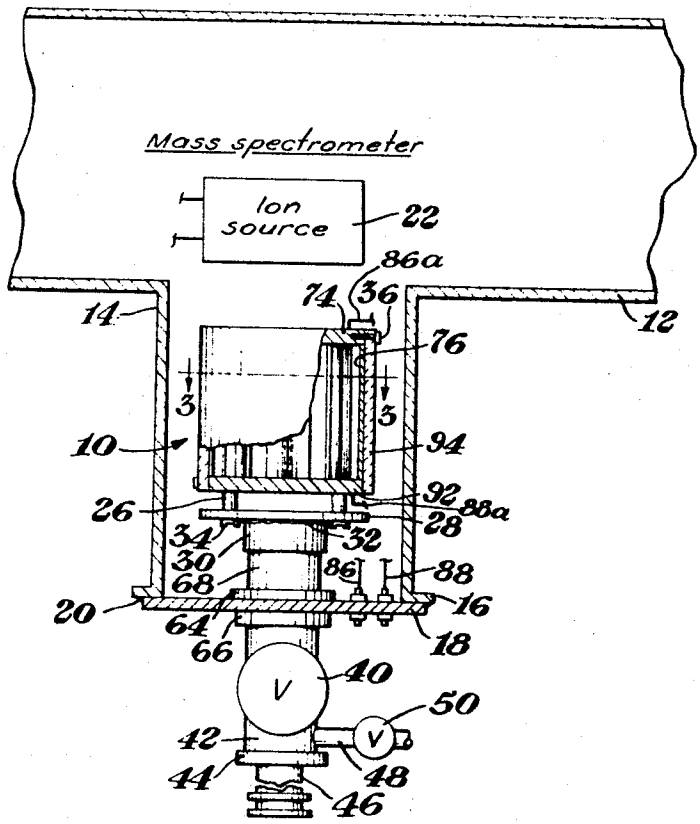
[52] **U.S. Cl.** **13/31,** 250/41.9
[51] **Int. Cl.** **H05b 3/00**
[50] **Field of Search** ..... 13/31, 20, 25; 250/41.9ISR, C

[56] **References Cited**
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Primary Examiner—Bernard A. Gilheany
Assistant Examiner—Roy N. Envall, Jr.
Attorneys—Griswold and Burdick and Earl D. Ayers

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 is a radiant heating device utilizing glow bar-type heating rods, with or without an adjacent reflective surface and is adapted to be controlled by means of temperature sensing means disposed within a separate thermal analysis cell which is adapted to be disposed within the furnace.



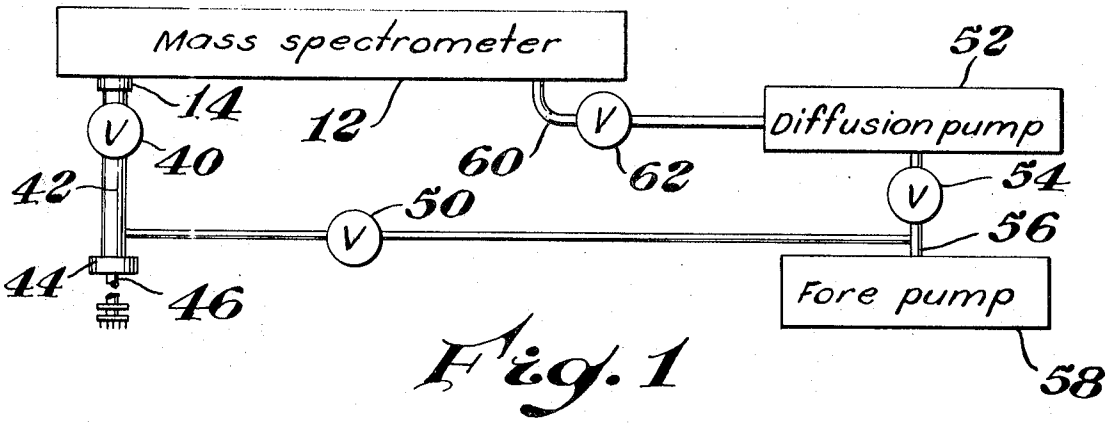


Fig. 1

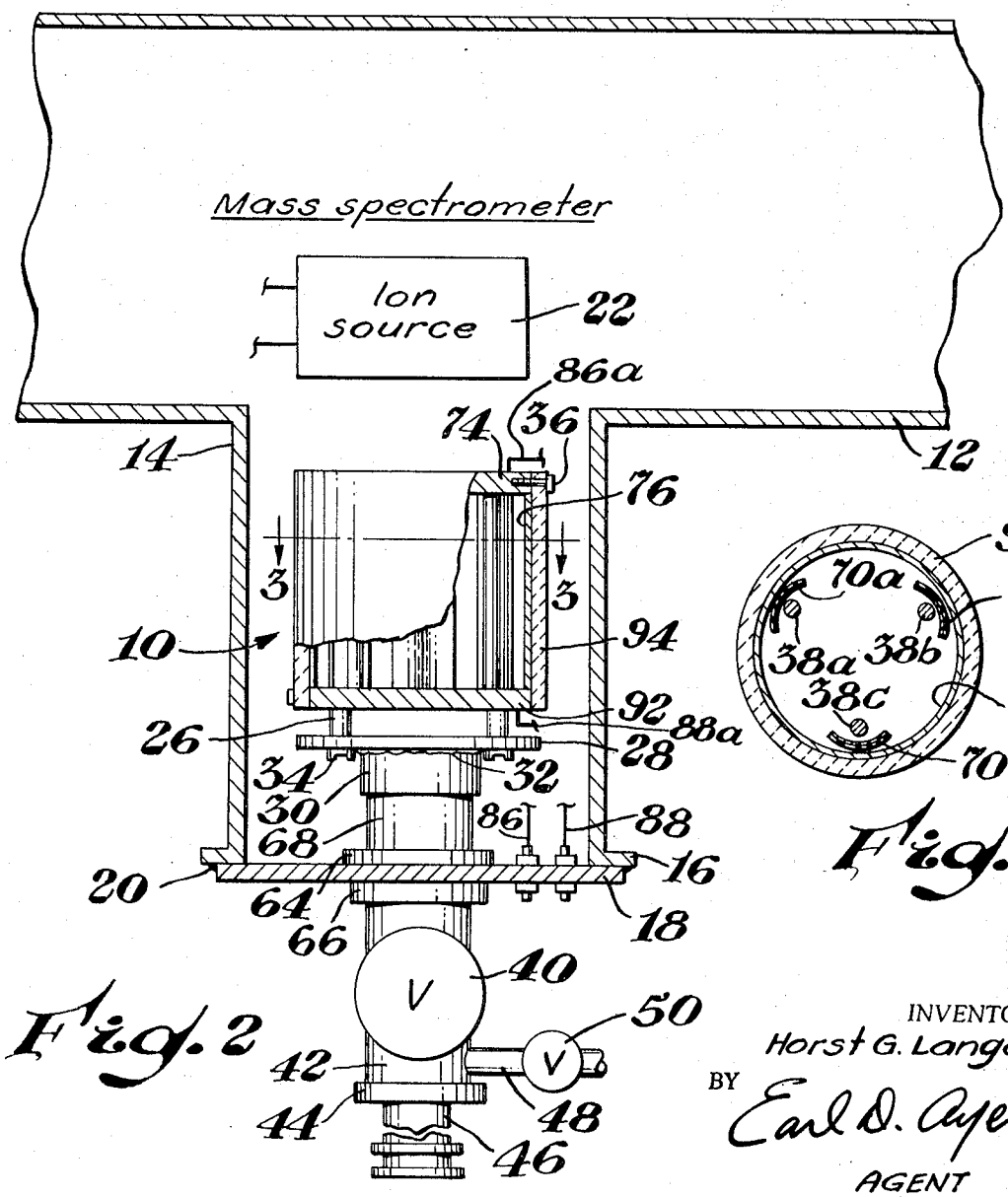


Fig. 2

Fig. 3

INVENTOR.
Horst G. Langer
BY Earl D. Ayers
AGENT

FURNACE ASSEMBLY FOR THERMAL ANALYSIS USE

BACKGROUND OF THE INVENTION

This invention relates to radiant-heated furnaces adapted to receive a thermal analysis cell and particularly to furnace and cell insertion assemblies which are adapted for use under high vacuum conditions.

Accordingly, a principal object of this invention is to provide an improved thermal analysis 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.

Even though mass 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 even if such devices sometimes also allow the measurement of sample temperatures during the heating process, these devices do not allow the operation known as thermal analysis. In such high vacuum operations it is essential that the sample be heated at a linear predetermined rate of heating, that the sample temperature is known and indicated at all times, and for differential thermal analysis operations the sample temperature is continuously compared with that of an inert material in the same cell. In general, this requires that three thermocouples located in the thermal analysis cell should be precisely at the same temperature at all times unless a chemical reaction occurs in the sample. Thus, it is also of extreme importance that equal heat transfer is guaranteed from the heat source furnace to the thermal analysis cell, that no temperature gradient exists in the cell itself, that fast heat transfer is provided from the cell to the sample and that each thermocouple remains electrically insulated.

In addition, to make a cell useful it must be possible to load a sample into the cell and introduce the cell with the sample into a mass spectrometer without shutting down the operation of a mass spectrometer or other evacuated systems.

In accordance with this invention, there is provided a furnace which is generally cylindrical in overall configuration and is supported by thermally insulating means attached to a valved tubular thermal analysis cell input and sealing assembly.

The furnace has a symmetrical array of glow bar-type heating elements surrounding an open central axial part into which a differential thermal analysis cell is inserted. Heat reflective elements may be disposed adjacent to the glow bar elements to direct heat towards the above-mentioned cell.

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;

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; and

FIG. **3** is a sectional view taken along the line **3.3** of FIG. **2**.

Referring to the drawing, and particularly to FIG. **1**, there is shown a mass spectrometer **12** having a tubular member **14** extending perpendicularly therefrom. An additional tubular element **42** having a compression coupling **44** at its outer end and having a ball-type vacuum sealing valve **40** adjacent to the member **14** is coupled to the outer end of the member **14**. A cell-probe assembly **46** (See FIG. **2**) is shown partly inserted into the entry and sealing assembly. A diffusion pump **52** is coupled by means of valve **54** and tube **56** to a fore pump **58** and by means of a tube **60** and valve **62** to the mass spectrometer **12**. The tubular element **42** is coupled through tube **48** and valve **50** to the tube **56** between the valve **54** and the fore pump **58**.

Referring now to FIG. **2** and FIG. **3**, as well as to FIG. **1**, there is shown the mass spectrometer tube **12** having a tubular member **14** extending transversely therefrom adjacent to the ion source **22** of the mass spectrometer. The member **14** has an outwardly extending flange **16** at its outer end. A metal plate **18** on which is supported the furnace and cell probe entry and sealing assembly **10** is sealed, as by the fillet **20**, for example, to the flange **16**. A threaded tube **68** extends transversely through the plate **18** generally in coaxial relationship with the member **14**. A tubular element **42** containing a so-called vacuum ball valve **40** is coupled to the tube **68** by means of coupling **66**. A seal-nut element **64** seals the tube **68** and element **42** (and coupling **66**) against the plate **18** through which the tube **68** passes.

A coupling **30** having a flange element **28** welded thereto as at **32** is threadedly coupled to the end of the tube **68** which lies within the tubular member **14**. A metal annular plate **92**, usually copper or brass and of substantially, larger diameter than the diameter of the coupling **30** is coupled to the top of the flange element **28** by means of screws **34** and insulating spacer elements **26**.

An annular-supporting sleeve **94** made of electrically insulating material and is supported from the plate **92** to which it is secured by screws.

An annular plate **74**, usually made of metal and at least approximately of the same outer dimension as the diameter of the annular plate **92**, is disposed parallel to the plate **92** and is secured to the supporting sleeve **94** by screws **36**.

An array of electrical glow bar-heating elements **38***a*, **38***b*, and **38***c* are disposed between the annular plates **74**, **92** and are disposed so that the cell **46** may pass within the array without touching the support rods. Elongated heat reflective elements **70***a*, **70***b*, and **70***c* are disposed behind the glow bar elements **38***a*, **38***b*, and **38***c*, respectively.

Wire leads **86***a*, **88***a* from the elements **74**, **72** are coupled to insulated feed through lines **86**, **88**, respectively, and thence to a suitable electrical energization source (not shown).

In operation, with the mass spectrometer pumped down by the diffusion pump **52** and with valves **40** and **54** closed, the cell-probe **46** is inserted in the tubular element **42** between the closed valve **40** and the "opened" compression fitting **44**. The compression fitting is then tightened around the tube of the probe **46** and, after a sufficient reduction of pressure by means of the fore pump **58**, the valve **50** is closed and ball valve **40** and valve **62** are opened. The cell-probe **46** is then slowly pushed through and past the compression fitting **44**, past the valve **40** and into the bore in the furnace body inside of the glow bar array **38***a*, **38***b*, **38***c*. The glow bar elements are energized at a controlled rate from a controller energization source (not shown) in coordination with the readings from control thermocouples or other temperature sensing means in the sample cell-probe assembly **46**, as is known to those skilled in the art of differential thermal analysis.

Because of the location of the furnace adjacent to the ion source **22**, the material vaporized on heating of the sample material carried in the cell-probe **46** is emitted into the ion source area of the mass spectrometer, where the vaporized material is ionized and analysis of the sample by mass spectrometric means occurs simultaneously with the differential thermal analysis of the sample.

A cell-probe assembly which is adapted for use with this invention is disclosed and claimed in Horst G. Langer et al. copending application Ser. No. 742,869, entitled, "Differential Thermal Analysis Cell Assembly," filed Jul. 5, 1968 now abandoned.

The reflective elements **70***a*, **70***b*, **70***c* may be made of an electrically nonconductive material having a heat reflective coating facing the glow bar elements or may be made of metal in event they are electrically insulated from the elements **74**,**92**.

The glow bar elements may be Nernst glow bars or rods of silicon carbide, for example.

The sleeve **94** may also, if desired, be provided with an internal heat reflecting surface or may be eliminated if the glow bar elements and/or the reflective elements **70***a*, **70***b*, **70***c* provide sufficient structural support for the heating assembly.

I claim:

1. A furnace and sample insertion assembly for use under high vacuum conditions, comprising first tubular means, a first disc having a central bore which is at least as large as the inner diameter of said first tubular means, said first disc being disposed perpendicularly with respect to said first tubular means and secured thereto an array of rodlike heating elements electrically coupled and disposed perpendicularly with respect to said first disc and secured adjacent to the outer periphery of said first disc, a second disc, said second disc being disposed above said first disc and electrically and mechanically coupled to said heating elements, a second tubular element, said second tubular element being axially aligned with a flanged mounting plate, said flanged plate extending transversely from said tubular element means intermediate the ends thereof, said flanged plate being hermetically sealed to said tubular element means, said tubular means having a valve therein on the side of said flanged plate remote from said rodlike heating elements, said tubular element having an inner diameter which is slightly larger than the diameter of a thermal analysis cell assembly adapted to be inserted therethrough and into the space within said rodlike heating elements, means for sealing the tubing means to said cell assembly, and lead means for energizing said heater winding.

2. An assembly in accordance with claim **1**, wherein said means for sealing the tubing means to said cell assembly comprises a compression seal.

3. An assembly in accordance with claim **1**, wherein a heat reflective surface is disposed adjacent to each of said rodlike heating elements and each presents a concave surface to its rodlike heating element.

4. An assembly in accordance with claim **1**, wherein a sleeve extends between said first and second discs.

5. An assembly in accordance with claim **1**, wherein said flanged plate is adapted to be coupled to a mass spectrometer.

6. An assembly in accordance with claim **4**, wherein said sleeve has a heat reflective inner surface.