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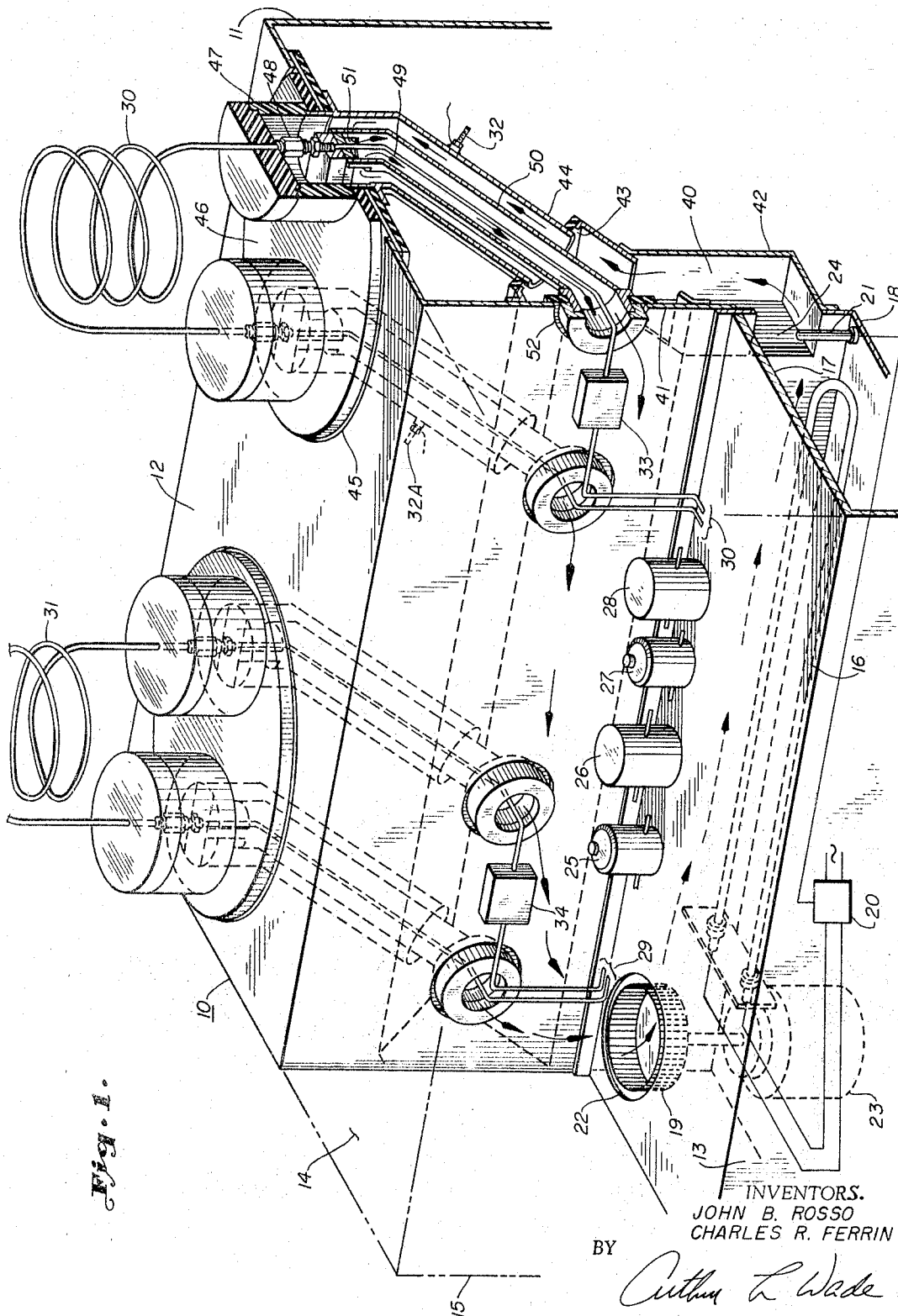
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SYSTEM FOR HEATING CHROMATOGRAPHIC COLUMNS

Filed Aug. 24, 1964

2 Sheets-Sheet 1



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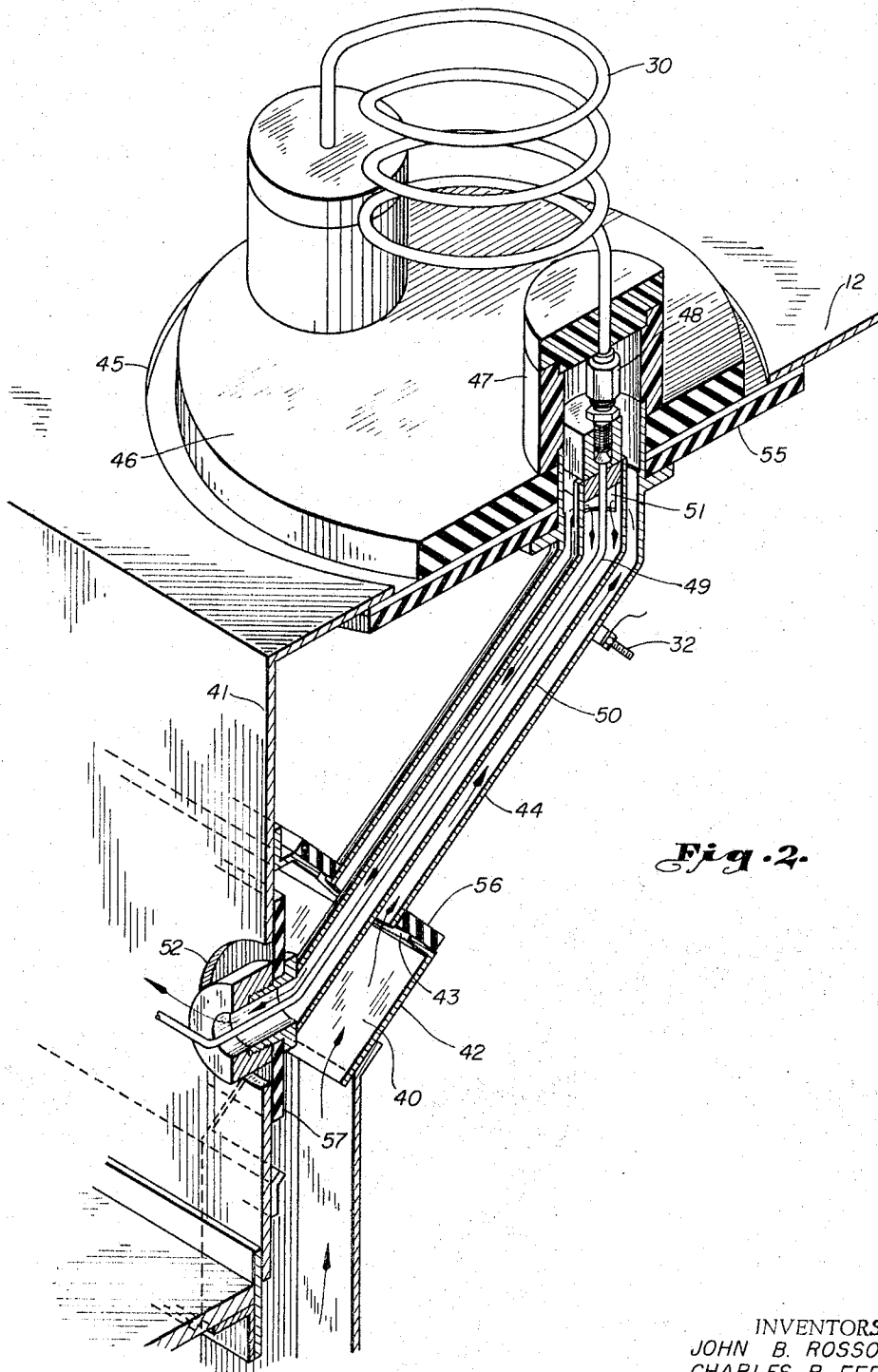
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SYSTEM FOR HEATING CHROMATOGRAPHIC COLUMNS

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4 Claims. (Cl. 219—399)

The present invention relates to heating chromatographic columns. More specifically, the invention relates to maintaining the temperature of the fluids passed through a chromatographic column high enough to enable the column to separate the gases of the sample in the manner desired for analysis.

In the practice of chromatography, it is common to provide an oven for the valving of the entrance system to the column and the detector of the components discharged from the column. The entrance system prepares a sample at oven temperature and passes it to a column which may not be in the oven. It is common to provide a column heated directly by an electric current. This column is mounted outside the oven and must be connected to the valve system to receive the oven-heated sample. At the junction of the two systems there is a problem of keeping the sample temperature stable and under control. The thermal inertia of the small tubes carrying the sample fluids is small and short exposure to a low temperature environment will drop the temperature of sample gases so low that they will not move through the column with well-defined boundaries and at a satisfactory rate. Thermal protection at the point of transfer is required to keep the sample flowing normally from the entrance system to the column and from the column to the detector.

A primary object of the present invention is to transfer a gaseous sample into, and from, a chromatographic column with thermal stability.

Another object is to maintain control of the temperature of a sample for a chromatograph as the sample is passed into, and out of, the column.

The present invention contemplates a heating system for an oven of a chromatograph and a duct directing air circulated through the oven up to the ends of a column not heated in the oven. The heated air is directed over the tubing connection between the oven-heated unit and the fixture coupling the column to the tubing, so there will be no uncontrolled variations of the gaseous sample. The air directed over the connection is then routed back to the oven and distributed over the units heated in the oven. The element providing heat for the air is regulated to establish a desired oven temperature and temperature for the sample as it passes into the column which will enable the column to operate at a desired speed and not permit condensation of components in the sample and evaporated substrate to occur.

Other objects, advantages and features of the present invention will become readily apparent from the following detailed description of the invention with a specific reference to the accompanying drawings in which:

FIG. 1 is an isometric representation of a complete, multi-column chromatograph, sectioned to illustrate the structure in which the present invention is embodied; and

FIG. 2 is an enlargement of the sectioned duct structure of one column to show essential elements of the embodying structure in greater detail.

Referring to FIG. 1, there is disclosed a partially sectioned isometric view of the portion of a complete chromatograph system in which the present invention is embodied. A cabinet 10 has a rear wall 11, a top 12 and front 13. The columns are mounted on the top 12 and the entrance systems and detectors for the columns are mounted in an oven enclosure 14.

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Access to the oven 14 is obtained by swinging lid 15 upward to expose the oven in the front, top volume within the cabinet. Lid 15 is hinged in a conventional manner; the lid is only indicated by broken lines and no hinges are shown as they would needlessly clutter the drawing and perhaps obscure the invention.

Oven temperature control

The oven is heated by a resistance element 16. The element 16 is in hair-pin form and extended horizontally below a sub-floor 17 of the oven. This sub-floor supports much of the entrance and receiving units of the columns which must be heated in the oven. Between sub-floor 17 and lower floor 18 the heating element is positioned to heat air circulated in the oven. Floors 17 and 18, with vertical panels of the cabinet, form a passage. Air is forced down this passage, by blower 19, and over element 16 to pick up enough heat to bring the temperature of the oven up to a desired level.

The controls for supplying electrical power to element 16 are represented as within box 20. A line supply of electrical power is indicated and a temperature sensing element 21 is mounted on floor 18 to sense the temperature of the air blown over element 16. Temperature sensing element 21 is connected to box 20 to illustrate control over the power supplied to element 16. These controls can be regarded as conventional arrangements with which to select a predetermined temperature for the oven and automatically maintain this temperature.

Blower

Blower 19 is a conventional form of air mover mounted at the left end of the heater element passage. Air is drawn down through a hole 22 in sub-floor 17 for heating. An electric motor 23 is indicated by dash lines as mounted below lower floor 18 from which position it can rotate blower 19.

No controls for the motor 23 are illustrated. In most cases, the speed of actuation of blower 19 is fixed, blowing the oven air over element 16 at a constant rate. The adjustments of oven temperature are normally made by regulation of the power delivered to element 16 under control of element 21.

The air drawn down through hole 22 at the left end of the passage is flowed over element 16 to exit through hole 24. The air passed through hole 24 is then placed in a manifold passage at the rear of the oven, passed up to flow over couplings at the ends of the columns and back down to the oven along the tubes connecting the columns with their entrance systems and detectors in the oven. This circulation of air through the oven, and up to the ends of the columns, will keep the essential elements of the chromatograph heated hot enough to operate as desired, considering the type of column and samples moved into and through the column.

Basic units heated in the oven

Oven enclosure 14, with its lid 15, is intended to form a desired temperature environment for the valves in which the samples to be analyzed are prepared and routed to the column. Two columns are shown in FIG. 1 to represent a plurality of columns in one chromatograph. Actually an instrument may have only one column, or more than two, depending upon the needs for analysis.

There may be a need for several valve units to be connected to each column. More than one column may be connected to each other through a valve system. The "plumbing" variations between columns and valves are many and varied.

Merely to represent the basic requirement of a valving system, and means for detecting the separated components of samples, a single valve unit and a detector are shown

associated with each column. It is to be understood that the specific connections between these units and the columns depend upon requirements not a part of the present disclosure.

Specifically, valve 25 and detector 26 form a set of units associated with one column and valve 27 and detector 28 form a set of units associated with the second column. Each unit is mounted in oven 14 on sub-floor 17. In this position the units are maintained at oven temperature. From this position the valves are actuated by manual, and possibly automatic, controls from outside the oven. No attempt has been made to illustrate controls to the valves which may be extended to the valves from outside the oven. Such disclosure would needlessly obscure the invention.

Finally, the interconnections between the tubes connected to the columns and the valves and detectors have not been shown. All that has been shown are short piping connections out of the valve and detector housings and the ends of tubes at 29 and 30 extending into the oven enclosure 14, the tubes coming from the columns.

Column heating

Chromatographic columns 30 and 31 are mounted on top 12. In this location the columns can be isolated from the oven heat, readily changed and serviced as required. There are many ways to heat these columns. One way is to form the column sheath of electroconductive material and apply electrical power to heat the column as a resistance element.

An electrical system which is suitable for resistance heating such columns is disclosed in patent application S.N. 389,013 filed August 12, 1964, by Owen D. Tibbetts et al. and S.N. 389,072 filed August 12, 1964, by Owen D. Tibbetts. This system is not disclosed in this application, but power leads 32 and 32A are indicated as connected to the electroconductive duct-work in electrical connection with the column 30. This power is electrically isolated from the oven-heated valves and detectors by terminal stations 33, 34. For the present, it is sufficient to understand that the columns are heated independently of the oven 14. The columns can be heated and cooled more quickly in this manner. However, this advantage is penalized by the problem of keeping the samples passed to, and from, the column thermally stable and controlled.

Thermal control and stability of chromatographic samples

Should the temperature of the gaseous sample for analysis by the chromatograph drop below a predetermined temperature, the chromatogram or chart drawn by the detectors of the instrument may not represent the components of the sample. Errors in the record, from this one cause are a result of:

- (1) Condensation of one or more components of the sample before entrance to the column, and/or
- (2) Condensation of the substrate and/or component of the sample after leaving the column.

Consider the entrance system first; as the temperature of the sample is allowed to lower, the vapor pressure of one of the components of the sample reaches the pressure of the system. Condensation of this component begins to take place. Carrier gas continues to pass over the condensate. Evaporation of the components reoccurs. The condensed and reevaporated component follows that portion of the component into the column which did not condense. This results in elution from the substrate over a prolonged period of time. The chromatogram then rises to a peak, representative of this component, but the trailing edge of the peak returns to the base line slowly.

The prolonged retention of one component in the column, because of the loss of thermal control at the entrance to the column, has two undesirable results. First, it is difficult to compute such an area for the basic

analysis. Second, the elution of another component may begin within this trailing area. The overlap of component areas on the chromatogram results in a loss of information on both components.

Next, consider the exit end of the column. The column discharges the components of the sample in slugs. The substrate is continually evaporated from the column by the heat. This evaporated substrate causes no difficulty in obtaining a chromatogram. The detector responds to the substrate but this only means a shift of the base line of the chromatogram.

However, if substrate material condenses down stream from the column, reabsorption of the components occurs. Because the substrate material will condense in uneven thickness on the inside of the tubing, the elution time of the dissolved components will vary as a function of the thickness of the condensed substrate. This has the effect of improper separation of the components and information may be lost, or peaks masked by one another. Also, with this temperature dropped, out of control, the condensing substrate material may eventually close the tube conducting the output of the column and positively terminate all detection by the column.

The present invention is directed to retaining control of the temperature of the sample between the oven-heated units of the system and the column. Further, this thermal stability and control is achieved without thermal interaction between the heating system for the column and the heating system for the oven. The oven environment is, in effect, extended up to the column for the sample components while the temperature of the column is independently controlled. All of these results are obtained with a simple arrangement of structure which is readily assembled, easily serviced and inspected and readily replaced when defective.

Manifold-duct—conduit system

Having established the need for controlling heat to the sample as it is conveyed between the oven-heated units and the column, the specific structure disclosed to provide this heat is considered. The heating of the air circulated in the oven has been discussed. The blower-driven heated air is forced through opening 24 and into a manifold passage 40 extending horizontally along the rear of the oven.

For the purpose of defining the manifold 40, the oven 14 is defined as having a rear wall 41. The lower portion of this wall supports the rear edge of sub-floor 17 and lower floor 18. Between these floors, through rear wall 41, hole 24 is formed. The manifold passage 40 has wall 41 as its front side and plate 42 attached to wall 41 with a shape to form the manifold passage 40 along the back wall 41.

It is to be understood that the specific problems of manufacture may make it evidently advantageous to form plate 42 of several different sections which can then be joined to form the manifold passage. This problem is one of design and manufacture. What the invention provides is the manifold passage, however formed, shaped and arranged to receive the heated air of the oven and distribute the air to delivery ducts extending to the ends of the columns.

Both FIG. 1 and FIG. 2 should be studied for a complete and detailed disclosure of the structure in which essential elements of the invention are embodied. FIG. 2 makes the smaller details of the heat-conveying structure clear while FIG. 1 orients this structure with the other structure of the chromatograph.

Each column end has a heat-conveying duct extending to it from manifold 40. An aperture 43 is shown in plate 42 and the lower end of duct 44 is mounted in this aperture. The upper end of the duct 44 is shaped and extended up through top 12 of the chromatograph cabinet. All duct holes are formed in the above manner in manifold 40 and top 12. Thus the heated air of the oven is

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distributed to the ends of the columns by ducts of which duct 45 is representative.

More specifically, the upper ends of the ducts extend through holes in platform members mounted in holes in the cabinet top 12. Focusing the description on duct 44, aperture 45 is seen, in FIG. 1, to accommodate the mounting of platform 46. This platform structure could be given many specific forms, however, its basic purpose is to mount the column 30 and support the upper end of duct 44 so it will supply the oven air to the end of the column. While providing this structural support, insulating members are placed to isolate the power supplied the column from the cabinet and oven-heated units. These structural arrangements need not be described in greater detail to disclose the invention.

The upper end of duct 44 is covered by a housing 47 which fits down around the duct end and mounts on the platform 46. The end of column 30 extends down through the top of this housing and the housing basically functions to deflect the upcoming heated air from the duct 44 downward toward the oven.

The end of the column terminates in a coupling 48 through which the column is communicated with tube 49. Tube 49 is the all-important link between the column and the oven-heated units. It is this tube which must be provided with a thermally stable environment to present the detrimental effect which go to make up the problem solved by the present invention. Tube 49 is shown extending from the coupling 48, down to terminal station 33; from there, the connections to the oven-heated units are deliberately not illustrated in limiting detail.

The terminal 48 is necessarily a relatively large mass of metal, compared to the mass of the column it connects to tube 49. This large mass of metal could quickly drop the temperature of a sample passing through the coupling; therefore, it is vital that the heated oven air out of duct 44 be utilized to efficiently maintain the temperature of this structure at a level which will prevent the condensation of the problem.

The coupling 48 is located centrally within the housing 47. Moreover, the coupling is located centrally within the upper end of duct 44. The hot air is forced upward, around the coupling, on all sides, transferring heat to the coupling evenly and constantly.

After downward deflection of the heated air within the housing, a passage is provided for the heated air along, and around, the tube 49. Specifically, a conduit 50 is attached at its upper end to the coupling structure 48 and provided with holes 51 to return the air to the oven. The tube 49 is extended along conduit 50 and is thereby exposed to the heated air as it travels along the tube, within the conduit, back to the oven.

The invention is not limited to a specific relation between duct 44, conduit 50 and tube 49. In this preferred embodiment, they are concentrically arranged. The heated air then surrounds both conduit 50 and tube 49 as it travels up to the end of the conduit. Further, the heated air is returned to the oven along the tube 49. The sample within tube 49, and the coupling 48 within housing 47, are thus continually surrounded by an environment of heated oven air whose temperature is controlled. The level of temperature is maintained high enough to prevent the problem arising.

Only a few details remain. The lower end of conduit 50 is mounted through hole 52 in wall 41 of the oven. Both the tube 49 and air are thus returned to the units 25, 28 mounted in oven 14.

The housing 47 is arranged to expose the end of column 30 to as little of the oven-heated air as possible. Therefore, the heat of the oven air is not transferred to the column to disrupt the independent heating program of the column.

The invention provides a structure which dynamically controls and stabilizes the heating of the sample of a

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chromatograph in transit between a column and oven-heated unit of the chromatograph. The oven environment, in effect, is extended as needed to protect the sample from thermal variations. In providing this extended environment, the heating of the units within the oven is made more uniform; the return system is actually an oven distribution of the heated air within the oven 14.

FIG. 2 illustrates how the top of housing 47 is brought down close to the top of coupling 48 to avoid the oven heat being applied to column 30. The resistance heating of column 30 is carried out separately from the oven heating and it is best to keep them isolated. Therefore, the housing 47 reduces the possibility of an overlap in the heating at the ends of the column.

The electrical connections through which electrical power is applied to the column 30 have been previously mentioned. Connection 32 is shown in this FIG. 2. This electrical heating is carried out by the circuit being completed with the conductive connections through duct 44, conduit 50 and coupling 48 to column 30. This assembly, as a unit, is electrically insulated from the cabinet by insulators 55, 56 and 57. The greatest resistance in the unit is column 30, therefore, column 30 heats when power is applied between the two ducts.

The control of the power and measurement of the temperature reached is not a part of the present disclosure. The invention here is embodied in the structure disclosed.

From the foregoing it will be seen that this invention is one well adapted to attain all of the ends and objects hereinabove set forth, together with other advantages which are obvious and which are inherent to the apparatus.

It will be understood that certain features and sub-combinations are of utility and may be employed without reference to other features and subcombinations. This is contemplated by and is within the scope of the claims. As many possible embodiments may be made of the invention without departing from the scope thereof, it is to be understood that all matter herein set forth or shown in the accompanying drawings is to be interpreted as illustrative and not in a limiting sense.

The present invention having been described, what is claimed is:

1. Apparatus for heating a chromatographic column, including,

- an oven enclosure,
- an electric heating element within the oven,
- a blower mounted within the oven in a position to move air over the heating element,
- a manifold conduit connected to the oven arranged to receive the air heated by the element,
- a duct connected to the manifold for receiving at least part of the heated air of the manifold conduit,
- a tube coupling mounted at the end of the duct and receiving one end of a chromatographic column,
- a housing over the coupling and duct end to receive the heated air and pass it over the coupling,
- a conduit connected to the housing and extending from the coupling to receive the heated air from the duct and connected to the oven to return the air to the oven,

and a tube for a sample to be analyzed by the column connected to the column with the coupling and extending within and along the length of the conduit to the oven in contact with the air returned to the oven.

2. Heating system for a chromatograph, including,

- an oven,
- a resistance heating element mounted in the oven and supplied electric power to develop a desired temperature in the oven,
- a resistance-heated chromatographic column mounted outside the oven and thermally insulated from the oven heat,
- a tube adapted to be connected to an entrance system

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in the oven and one end of the chromatographic column for transport of a gas sample to be analyzed between the entrance system and column,
 a delivery duct extending from the oven to the end of the chromatographic column for transport of air of the oven heated by the resistance heating element,
 a return conduit extending from the end of the chromatographic column to the oven for the return of air from the duct to the oven around the tube positioned within the conduit,
 and a blower positioned in the oven to circulate air over the heating element and through the delivery duct and return conduit to the oven,
 whereby the sample to be analyzed from the entrance system is maintained at a temperature which will cause desired separation and a desired rate of movement of the separated components through the column.

3. Heating system for a chromatograph, including,
 a cabinet in which and on which the structure of the chromatograph is mounted,
 an oven enclosure within the cabinet,
 a sub-floor in the oven,
 a second sub-floor below the first sub-floor forming a passageway between the two sub-floors,
 a blower mounted beneath a hole in the upper sub-floor to draw air from above the upper sub-floor and force the air along the passage between the two sub-floors,
 a heating element mounted in the passage and controlled to heat the air forced along the passage to maintain the air heated to a predetermined temperature,

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a resistance-heated chromatographic column mounted on the cabinet,
 a tube adapted to connect units heated in the oven and the column mounted on the cabinet,
 a conduit about the tube for the return of heated air to the oven while heating the tube,
 and a duct connected to the passage for receiving heated air from the passage and conducting it to the connection between the tube and the column and connected to the conduit to return the air to the oven by way of the conduit.

4. The system of claim 3 including,
 a deflector housing about the tube and over the end of the duct to isolate the column from the heat of the air and divert the air from the duct and into the conduit.

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