

Description

The invention relates to an apparatus according to the preamble of claim 1 for heating a length of tubing. An apparatus of that kind is used, for example, in gas chromatography for heating glass capillary gas transfer lines.

The general evolution of gas chromatographs has led to the use of very small glass capillary gas transfer lines and columns. A typical glass capillary tube has an inner diameter of .013 inch (0,330 mm) and .018 inch (0,457 mm) outer diameter. Vaporized chemical samples are carried, usually in a carrier gas, among the various devices in the gas chromatograph and between these devices and other devices located in adjacent detectors or other instruments external to the gas chromatograph through heated transfer lines.

The function of the transfer line heater is to maintain the vaporized chemical sample within defined temperature bounds as it is carried among the various components of the analytical instruments. A particular problem arises when the vaporized chemical samples are allowed to cool in the transfer lines, as this cooling can lead to precipitation of the sample out of the gas and onto the glass tube walls. Once this occurs, the chemical analysis is vitiated and there is the possibility that the glass tube can become plugged or that future chemical analysis can be contaminated. If heated above the temperature bounds, there is the possibility that the chemical sample will chemically react or decompose also vitiating the analysis.

Transfer lines are also used as gas conduits between analytical instruments, for example, between a gas chromatograph and a peripheral detector such as a spectrophotometer. For this use, the heated transfer line should be temperature controlled and powered in one instrument and protrude into the second. Since the small glass capillary tubes are difficult to clean in an instrument and are subject to breakage, the apparatus which heats the capillary tube must also permit its replacement.

In general, heated transfer lines are designed to operate in a temperature range of 150-350° C, concurrent with that of present gas chromatographs. Since many chemical compounds have chemical reactivity or decomposition temperatures near their boiling points it is highly desirable that the temperature range, or tolerance of the transfer lines be small, e.g. within 10° C.

Several instrument oriented constraints on transfer line design have, here-to-for, limited the temperature profile integrity of transfer lines. As a practical matter, it is desirable that a transfer line bridging two separate instruments be securely mounted, controlled and powered from one instrument while simply extending in to the second. The degree of mounting in the second instrument, if at all, may be determined by such influences as the need to isolate the instruments from vibration, or the need for instrument modularity. The external environment surrounding the transfer line as it extends from within one instrument to within another can vary significantly. For example, a transfer line traversing from the inside of a heated gas chromatograph oven, through the oven wall insulation and out to the ambient air gap between instruments experiences a variation in temperatures ranging from perhaps 20° to 350° C.

Prior art transfer lines have been limited to the rather simple approach of inserting the gas capillary tube in a comparatively large body of a highly thermal conductive metal housing which is heated by an enclosed heater, such as a cartridge or band heater. This combination is further surrounded by a thermal insulator of some type, which tends to mitigate changes in the surrounding environment. The larger the metal body and insulation, the more uniform the temperature. Several disadvantages are evident in this approach. First, since the mass of the metal housing is primarily designed to house the heater rather than the small glass tube, such heated transfer devices are particularly power inefficient and subject to substantial heat loss in the surrounding instrument. Analytical detectors such as spectrophotometers are particularly sensitive to temperature gradients due to the precise alignment of their optics and can not afford the heat dissipation from a relatively large transfer line operation at 350° - 400° C, as is required in gas chromatography. Typically, these prior art transfer lines have diameters of 5 cm or more and due to their size, dissipate as much as several hundred watts of power onto their host instruments. These large diameters also inhibit coupling of the transfer line to instrument devices. It is not uncommon for cold junctions to develop at the coupling which are 50°C or more below the desired operating point.

Alternatively, prior art transfer lines have consisted of a weave of highly electrically resistant wire, such as an alloy of 80% nickel and 20% chromium, and fibrous glass or ceramic insulation wound about the glass capillary tube into which the glass capillary may be inserted. Electrical current supplied to the metal wire heats the capillary. Although usually smaller in diameter than the previously described transfer lines, the woven transfer lines lack temperature uniformity when positioned in a temperature environment which changes along the length of the transfer line.

Other prior art transfer lines have consisted of a tubular structure heated at one end, the capillary tube passing through the centre of the tube. The heating element is housed in metal attached to the tube and heat is transferred by conduction down the length of the tube. Although this approach provides for a smaller

size on the nonheated end, it obviously suffers from non-uniform heating of the capillary tube. To maintain a minimum temperature at the cold end the heater must be brought to a higher temperature than required for the chemical analysis.

FR-A-2361035 (HEUTEY METALLURGIE) discloses an infrared furnace for heating small metal blanks within a cylinder. The inside wall of the cylinder is of a refractory steel which carries an electrical current. The supply is fed from the centre of the outer surface of the oven structure through two half cylinders formed from a highly conducting material. The blanks to be heated are passed through the furnace between two water cooled rails.

US-A-1727585 (CARLETON) discloses an apparatus for heating material with an electric current, comprising an electrically conductive heater tube for encircling the material to be heated, the heater tube having first and second heater tube ends, first insulating means substantially covering the heater tube for electrically insulating the heater tube, two first current conducting means having first and second ends electrically connected to the first and second heater tube ends respectively for conducting electrical current through the heater tube, said first current conducting means being arranged in proximity to the heater tube and being electrically insulated from the rest of the heater tube by the first insulating means, and the other ends of said first current conducting means being arranged in proximity and opposite to each other, without touching, at a common point along the heater tube, and two second conducting means attaching to the said other ends, respectively, of said first current conducting means near said common point. The disclosure is corresponding to the preamble of claim 1.

Relative to this prior art, it is the object of the invention to provide an apparatus according to the preamble of claim 1 which has a lower power consumption, smaller heat losses and which has a higher temperature uniformity along the tubing to be heated.

According to the invention there is provided an apparatus for heating material with an electric current, comprising an electrically conductive heater tube for encircling the material to be heated, the heater tube having first and second heater tube ends, first insulating means substantially covering the heater tube for electrically insulating the heater tube, two first current conducting means having first and second ends electrically connected to the first and second heater tube ends respectively for conducting electrical current through the heater tube, said first current conducting means being arranged in proximity to the heater tube and being electrically insulated from the rest of the heater tube by the first insulating means, and the other ends of said first current conducting means being arranged in proximity and opposite to each other, without touching, at a common point along the heater tube, and two second conducting means attaching to the said other ends, respectively, of said first current conducting means near said common point, characterised by second insulating means substantially covering the first and the second current conducting means and the heater tube for thermally insulating the heater tube and the first and the second current conducting means, an outer tube substantially covering the second insulating means, and mounting means connected to the outer tube in a mounting plane which passes through the common point along the heater tube for mechanically mounting the apparatus and providing an exit for the second current conducting means which exit is substantially in said plane.

In summary, the invention embraces an apparatus which permits gases to be transferred from one device to another through a glass capillary tube with minimal changes of temperature. The apparatus consists of a heated transfer line and an optional oven feedthrough. The heated transfer line works by transferring the heat, which is generated when an electrical current is passed through a highly resistive metal tube, to a glass capillary tube, which can be inserted into the metal tube.

The heated transfer line is small in size and low in power dissipation when compared to the prior art. The heater consists of several concentric tubes, each with a special purpose. The heater tube through which current passes is small in diameter and only slightly larger than the glass capillary tube and is designed to efficiently transfer the heat directly to the capillary tube. The heater tube is electrically insulated, except for the ends where power is supplied through two electrical conduits or wires. These conduits form the second layer of the transfer line and also generate heat as a current passes through them. The conduit tubes are specially shaped in order to generate more heat at the ends, without raising temperatures significantly in the middle of the tube. Their length together is approximately equal the length of the heater tube and permits power to be supplied at a central location near the middle of the tube. At this juncture, a thermocouple is mounted which is used to monitor the transfer line temperature. The conduit tubes are covered with a thermal insulator and the entire assembly is enclosed within an outer tube.

The oven feedthrough consists of a short piece of copper pipe mounted to an insulator with a hole in the center through which a heated transfer line, similar to the one described above, may pass. The insulator serves to insulate the copper pipe and heated transfer line from the oven. The inner diameter of the copper

pipe is several times larger than the transfer line which because of its thermal conductivity attempts to keep a constant temperature over its length thereby providing a more gradual change in temperature between the oven and the ambient air for the heated transfer line.

The invention provides considerably more uniform temperature throughout the transfer line than the prior art. The invention dissipates low power, typically 8 watts compared to several hundred watts of the prior art. The invention is small in size, the transfer line heater being 0.25 inches (6,35 mm) in diameter rather than the 2 to 5 inches (50,8 mm to 127 mm) or greater typical of the prior art.

According to claim 2, the heating of the capillary tubing can be accomplished with a simple construction using electrically conductive wires.

In the embodiments according to claims 3 and 6, an even higher temperature uniformity along the tubing to be heated can be achieved.

According to claim 7, heat losses can be further reduced.

The developments according to claims 9 and 12 ensure easy mounting of the apparatus.

Subsequently, embodiments of the invention are explained in detail with reference to the drawings.

Figures 1A, 1B, 1C and 1D illustrate a heated transfer line for capillary tubing according to a first embodiment of the present invention.

Figures 2A, 2B and 2C illustrate a heated transfer line for capillary tubing according to a second embodiment of the present invention.

Figures 3A and 3B illustrate the variations in thickness of the conduit tube walls used to selectively heat the transfer line of Figures 2A-2C.

Figures 4A, 4B and 4C illustrate a capture ring for use with the heated transfer line of Figures 2A-2C.

Figure 5 illustrates the solder lug used to connect an electric conductor to the conduit tubes.

Figure 6 illustrates an assembled view of the heated transfer line of Figures 2A-2C.

Figure 7 is an illustration in cross section of an oven feedthrough.

The heater tube is conveniently selected from hypodermic needle syringe stock, usually hardened stainless steel. The small size of the glass capillary diameter allows the use of small diameter syringe stock with thin walls, both combining to provide smallness of size and an electrical resistance favorable towards its use as a heating element. The small size of the heater tube and its concentric location about the glass capillary results in a small uniform transfer line heater configuration for a glass capillary which is removable. Likewise, with this configuration power consumption is greatly reduced due to the small exposed transfer line surface area required at any given temperature.

In order to supply a current to the heater tube, the heater tube is first electrically insulated over its external surface, then electrical wires, or other electrical conduits, are connected to the uninsulated ends of the heater tube and made to extend back over the length of the insulated heater tube to some centrally located mutual point along its length from which general electrical connection of the heated transfer tube unit can be made externally. A temperature sensor is also coupled to the heater tube, and the leads of the sensor are also brought out at the centrally located mutual point.

A first embodiment of the invention is shown in Figures 1A, 1B, 1C and 1D. A heater tube 100 made of fully hardened stainless steel or a high resistance nickel/chromium alloy tubing having a .031 inch (0,787 mm) outer diameter and a .020 inch (0,508 mm) inner diameter is inserted into an alumina tube 110 having an outer diameter of .062 inch (1,575 mm) and inner diameter of .032 inch (0,813 mm) as shown in Figure 1A. The heater tube 100 extends out beyond the alumina tube 110 .09 inch (2,286 mm) on each end. Copper wires 120 and 123 of 28 gauge size (AWG 28) are wrapped about and silver soldered to the exposed ends 125 and 127 of the heater tube 100 and each wire 120 and 123 is brought to an intermediately located ferrule position 130 as shown in Figure 1B. A thermocouple 140 is cemented with a high temperature ceramic cement on the outer diameter of the alumina tube 110 at the ferrule position 130. Lengths of a center drilled ceramic fibre tube insulation 150 and 155 having an outer diameter of .210 inch (5,334 mm) and inner diameter of .062 inch (1,575 mm) are slid over and abutted to the ceramic tube 110 and over the 28 gauge (AWG 28) copper wires 120 and 123. Yet another tube 160, a stainless steel sheath having outer diameter .250 inch (6,35 mm) and inner diameter .210 inch (5,334 mm), as shown in Figure 1A is slid over the assembly of Figure 1B. The thermocouple 140 and copper wires 120 and 123 are pulled out through the insulation 150 and 155 and through holes 170 drilled radially in the tube 160 at 120 degrees radial spacing. A stainless steel ferrule 180 shown in Figure 1A is slid over the tube 160 and positioned with the wires 120 and 123 and the leads of thermocouple 140 entering 120 degrees spaced grooves 185 in the ferrule 180. The leads are ceramic cemented in place and exit through .032 inch (0,813 mm) inner diameter, .062 inch (1,575 mm) outer diameter alumina tube 190 via three parallel holes 192, 193 and 194. The ferrule 180 is silver soldered to the tube 160. Alumina end caps 200 and 205 are cemented with a high temperature alumina cement to each end 207 and 209 of the tube 160. Onto end cap 205 is cemented a

stainless steel fitting 210 having a thread. The fitting 210 and end caps 200 and 205 each has a central hole passing therethrough so that a glass capillary tube (not shown) can enter through either end 212 or 214 of the thus formed heated transfer line 215 shown fully assembled in Figure 1C and pass out the other end. Holes 220 located radially about the flange 230 of the ferrule 180 as shown in Figure 1D allowed the mounting of the heated transfer line 215 to a flat surface.

In operation the transfer line 215 of Figure 1C is powered by passing current through the copper wires 120 and 123 which thus allows current to flow uniformly through the entire length of heater tube 100. The power dissipated in the heater tube 100 causes its rapid heating. The surrounding ceramic fibre insulation 150 and 155 restricts heat loss while the ceramic tube 110 and ceramic end caps 200 and 205 isolate the heater tube 100 electrically from the tube 160 exterior and the copper wires 120 and 123.

The above described heated transfer line has a temperature capability in excess of 400° C. However, the metal ferrule 180 may serve as an undesired heat sink creating a localized lower temperature in the heater tube 100 at the ferrule location 130. In addition, the construction is comparatively labor intensive.

A second embodiment shown in Figures 2A, 2B, and 2C is now disclosed to overcome these difficulties while maintaining both the function and size of the first embodiment. A fully hardened stainless steel or high resistance nickel/chromium alloy tube 250 having a .031 inch (0,787 mm) outer diameter, .023 inch (0,584 mm) inner diameter and 5.676 inch (144,17 mm) length is wrapped with a .062 inch (1,575 mm) wide by .001 inch (0,025 mm) thick polyimide insulation tape 260 about the heater tube 250 periphery and along its length to within about .25 inch (6,35 mm) of the end of tube 250 as shown in Figure 2A. A .001 to .0015 inch (0,0254 mm to 0,038 mm) thick silicone based adhesive on one side of the insulation tape 260 allows adhesion between the tube 250 and tape 260. The adhesive is only needed during assembly, and actually evaporates during use.

A thermocouple 270 having .010 inch (0,254 mm) diameter leads 271 and 272 is positioned on the tape 260 on tube 250 approximately .5 inch (12,7 mm) from one end of tube 250. The thermocouple 270 and both of its leads 271 and 272 are held in place over opposing sides of the insulated heater tube by sliding the capture ring 280 over the thermocouple 270, and covering the open end of the capture ring 280 with the Vespel washer 281 thereby trapping the thermocouple against the insulated heater tube and between the washer 281 and the capture ring 280. Top, left side and right side views of capture ring 280 are shown in Figures 4A, 4B, and 4C respectively and thermocouple leads 271 and 272 will exit through slots 285 in capture ring 280. The function of capture ring 280, in addition to positioning and holding thermocouple 270 directly against the tape 260, is to position the leads 271 and 272 of the thermocouple 270 and to electrically isolate them from each other, and to thermally insulate the heater tube 250 from its surroundings.

Electric current is supplied to the heated transfer line through lead wires 290 and 295. Typically, wires 290 and 295 are 22 gauge single strand silver plated copper wires insulated with Teflon. Electrical lead wires 290 and 295 are connected to the two respective solder lugs 310 and 315 by crimping and silver soldering the respective joints. Because the power required to heat the transfer line is quite small, a very small increase in power or the addition of a relatively small heat loss can drastically affect the local temperature of the transfer line. Therefore, the goal of the connector design is to provide a good electrical connection which is thermally invisible to the heater and conduit tubes. Since the connector would normally conduct heat away from the heater tube, this may be accomplished by passing a current through the connector which must heat the connector such that its temperature and thermal conductivity combine to provide an apparent thermal resistance which matches that of the insulation which surrounds the heater and conduit tube. Figure 5 illustrates a solder lug whose dimensions meet the above conditions by use of a precisely dimensioned ribbon 500 between the connecting points. The two solder lugs 310 and 315 are then attached to the ends of their respective conduit tubes 300 and 305. Silver soldering is chosen to restrict high temperature corrosion at the electrical crimp. Conduit tubes 300 and 305 are typically stainless steel tubes having an outer diameter of .072 inch (1,829 mm) and inner diameter .040 inch (1,016 mm). Once the solder lugs 310 and 315 are attached, conduit tubes 300 and 305 are slid over heater tube 250 oriented with the solder lugs at 120 degrees as shown in Figure 6. With this orientation, the unattached ends 330 and 335 of conduit tubes 300 and 305 are silver soldered to the exposed ends 337 and 339 of heater tube 250. The insulation tape 260 serves to electrically insulate heater tube 250 from conduit tubes 300 and 305 such that current will be allowed to travel down the conduit tubes 300 and 305 to the silver solder joints 330 and 335, and through the heater tube 250 and back out. Figure 6 illustrates the partially assembled transfer line where the solder lugs 310 and 315 and thermocouple mounting hardware meet.

Short and long outer tubes 340 and 345, respectively are inserted into upper and lower ferrules 350 and 355, respectively as shown in Figure 2A and staked into place. Staking occurs by flaring out the exposed end of each tube into the respective ferrules to prevent removal. Ferrules 350 and 355 are constructed from

polyimide for purposes of temperature resistance and to further restrict the flow of heat out of the assembled transfer line heater 240 shown in Figure 2B. Teflon flex tubing 360 is inserted over each lead 271 and 272 of thermocouple 270 and crimped in place with crimp 370. During assembly, crimp 370 fits into an enclosed cavity 380 in ferrule 350 such that the flex tubing 360 is not easily pulled out of the assembled transfer line heater 240. Each ferrule 350 and 355 with tube 340 and 345 is slid over their respective ends 335 and 330 of the previous assembly, as shown in Figure 2A. Each wire 290 and 295 and the leads 271 and 272 of thermocouple 270 are positioned respectively in grooves 390, 395, and 397 in upper ferrule 350. Ceramic fibre insulating tubes 400 are slid over the ends 335 and 330 of the above assembly in the annulus formed between the electrical conduits 305 and 300 and the outer sheaths 340 and 345. The insulation tubes 400 are pushed down against capture ring 280 and fit snugly adjacent to each other. The outer most insulating tube 400 is trimmed to allow the total proper length of insulation. Ferrules 350 and 355 are then riveted together with eyelets 410. The center holes of eyelets 410 serve as holes for mounting of transfer line heater 240. Polyimide insulators 420 and 425 are slid and positioned over the ends 335 and 330 of conduits 300 and 305. Insulators 420 and 425 serve to electrically insulate the conduits 300 and 305 from the outer tubes 345 and 340. End cap 440 and fitting 450 are made of stainless steel and are dipped in liquid nitrogen then quickly tapped into their respective ends 460 and 465 of the outer tubes 340 and 345. The expansion fit forms an interfering fit when the end cap 440 and fitting 450 are warmed to room temperature in the outer tubes 340 and 345.

A uniform tube generates uniform temperature rise along its length when an electric current is passed through the tube. A temperature profile of an operating uniform tube reveals however an uneven temperature profile due to uneven heat transfer to the environment. This problem is particularly severe toward the ends of the tube. As a major improvement of the present heated transfer line, its uniform temperature is accomplished by varying the wall thickness of the conduit tubes along the length of the transfer line heater. At any given position along said length the total power per segment is equal to the power losses of the heater tube and the power losses of the surrounding conduit tube. Since the current I is identical for each segment the power input per segment, q , is:

$$q = I^2(R_{ht} + R_{ct}) \quad (1)$$

Where R_{ht} and R_{ct} are the electrical resistances of the heater and conduit tubes respectively. The power loss from the heater tube to the environment varies along the length of the tube, for example, the end losses are greater than losses at the center of the transfer line. Therefore in order to keep a uniform temperature, it is desirable to add additional power to those segments with greater losses. Referring to equation 1, this can be accomplished by locally increasing the electrical resistance in those segments with greater losses. The resistance in a given segment may be increased by adjusting the wall thickness of any of the heating tubes. Since in the preferred embodiment the heater tube has assumed a minimal practical size for the overall resistance of the transfer line, a wall thickness adjustment is most easily made by altering the outer diameter of the conduit tubes.

This effect can be expressed mathematically in simple form by noting the electrical circuit analogy to the field of thermodynamics. The heater tube temperature T_o is related to the ambient temperature T_a , the power input per segment q and the thermal resistance between T_o and T_a of R_{th} by the equation:

$$\begin{aligned} T_o &= qR_{th} + T_a \\ T_o &= I^2(R_{ht} + R_{ct})R_{th} + T_a \end{aligned} \quad (2)$$

At the transfer line midpoint the thermal resistance R_{th} consists primarily of a sum of series resistances for a thermal dissipation path which extends radially out from the heater tube. Moving toward the ends, a parallel resistance enters into R_{th} due to axial losses (assuming the ends are cooler). This effectively lowers R_{th} , consequently lowering T_o for a constant electric current I . As R_{th} varies, the electrical resistance of the

conduit tube, R_{ct} , can be varied to compensate by altering preferably the outer radius of the conduit tube at the affected section. For a tube the electrical resistance can be calculated as:

$$R_t = \frac{\rho L}{\pi (d_o^2 - d_i^2) / 4} \quad (3)$$

where ρ is the resistivity of the tube material at the operating temperature T_o , L is the evaluated tube length, π is 3.14..., and d_o and d_i are the outer and inner tube diameters, respectively.

Thermal resistance can be calculated in simple configurations but may be more complex when all parameters are not known. The temperature distribution along a transfer line can easily be measured, however, by inserting a .010 inch (0,254 mm) diameter thermocouple probe into the end of a glass capillary and traversing the assembly down the length of the heater tube when operating.

Assuming the following values:

R_{ht} : $d_o = .031"$ (0,787 mm) $d_i = .023"$ (0,584 mm), $\rho = 3.8386e-5$ ohm-inch

R_{ct} : $d_o = .063"$ (1,60 mm) $d_i = .038"$ (0,965 mm), $\rho = 3.8386e-5$ ohm-inch

$T_a = 25^\circ \text{C}$ and $T_o = 300^\circ \text{C}$

and, due to practical grinding restrictions, a minimum outer diameter $d_o = .050"$ (1,27 mm), then applying the above formulas 2 and 3 it may be shown that a temperature rise of 45°C is possible. In the preferred embodiment an even larger tube was used to obtain an even higher temperature rise.

This analysis works well as a first order approximation, although an exact solution is not achieved since an adjusted segment is affected by adjoining unadjusted segments and due to changes in R_{th} which are temperature dependant.

Figure 3A, which is a picture of conduit tube 300, shows the various reductions in diameter for a particular application of the preferred embodiment. At point 803, the tube diameter is unchanged. The tube diameter is ground down .009" (0,229 mm) in sections 802 and 804, and further ground to .053" (1,346 mm) in section 800, and still further ground to an outer diameter of .050" (1,27 mm) at point 801. Figure 3B shows the similar grinding on conduit tube 305. The tube diameter is unground at 810, it is ground down .009" (0,227 mm) at 811 or 812 and still further ground to .050" (1,27 mm) at section 813.

The second embodiment of the transfer line heater 240 as shown in Figures 2B and 2C achieves uniform heating to 400°C with less than 10 watts of dissipated power with a variation of less than 10°C .

Apparant variations in the external temperature environment of the transfer line when mounted in various gas chromatograph oven configurations has also led to a need to improve the method of connecting the transfer line to the oven while minimizing gas chromatograph thermomechanical differences in transfer line performance. An additional feedthrough device for this purpose is shown in Figure 7. The feedthrough consists primarily of a copper tube 903 whose internal diameter is substantially larger than the transfer line heater's outer diameter. The copper tube mounted in the gas chromatograph oven wall insulation 901 strives to be at constant temperature along its length. The copper tube is purposefully oversized to allow sufficient thermal grounding of the tube to the ambient to reduce the variations in temperature of the tube along its length, and also, to allow for variations in oven wall thickness among the various gas chromatograph instruments.

To reduce the thermal sink effect of the copper tube on the oven itself, the copper tube is not allowed to protude through to the oven interior, but instead extends into a refractory ceramic fibre block 902 containing a centrally located hole to allow the axial passage of the heated transfer line 904 into the oven. The block also serves to isolate the heated transfer line from exposure to the high temperature oven and to prevent convection flow out of the oven and over the transfer line. Use of the oven feedthrough permits a heated transfer line with less than a plus or minus 10°C variation when used with a gas chromatograph.

Claims

1. An apparatus for heating material with an electric current, comprising an electrically conductive heater tube (100;250) for encircling the material to be heated, the heater tube having first (125;337) and second (127;339) heater tube ends, first insulating means (110;260) substantially covering the heater tube for electrically insulating the heater tube, two first (120;123;300;305) current conducting means having first and second ends (330;335) electrically connected to the first (125;337) and second (127;339) heater tube ends respectively for conducting electrical current through the heater tube, said first current conducting means being arranged in proximity to the heater tube and being electrically



conduit tube, Rct, can be varied to compensate by altering preferably the outer radius of the conduit tube at the affected section. For a tube the electrical resistance can be calculated as:

$$R_t = \frac{pL}{\pi(d_o^2 - d_i^2)/4} \quad (3)$$

where p is the resistivity of the tube material at the operating temperature To, L is the evaluated tube length, pi is 3.14..., and do and di are the outer and inner tube diameters, respectively.

Thermal resistance can be calculated in simple configurations but may be more complex when all parameters are not known. The temperature distribution along a transfer line can easily be measured, however, by inserting a .010 inch (0,254 mm) diameter thermocouple probe into the end of a glass capillary and traversing the assembly down the length of the heater tube when operating.

Assuming the following values:

Rht: do = .031" (0,787 mm) di = .023" (0,584 mm), p = 3.8386e-5 ohm-inch

Rct: do = .063" (1,60 mm) di = .038" (0,965 mm), p = 3.8386e-5 ohm-inch

Ta = 25° C and To = 300° C

and, due to practical grinding restrictions, a minimum outer diameter do = .050" (1,27 mm), then applying the above formulas 2 and 3 it may be shown that a temperature rise of 45° C is possible. In the preferred embodiment an even larger tube was used to obtain an even higher temperature rise.

This analysis works well as a first order approximation, although an exact solution is not achieved since an adjusted segment is affected by adjoining unadjusted segments and due to changes in Rth which are temperature dependant.

Figure 3A, which is a picture of conduit tube 300, shows the various reductions in diameter for a particular application of the preferred embodiment. At point 803, the tube diameter is unchanged. The tube diameter is ground down .009" (0,229 mm) in sections 802 and 804, and further ground to .053" (1,346 mm) in section 800, and still further ground to an outer diameter of .050" (1,27 mm) at point 801. Figure 3B shows the similar grinding on conduit tube 305. The tube diameter is unground at 810, it is ground down .009" (0,227 mm) at 811 or 812 and still further ground to .050" (1,27 mm) at section 813.

The second embodiment of the transfer line heater 240 as shown in Figures 2B and 2C achieves uniform heating to 400° C with less than 10 watts of dissipated power with a variation of less than 10° C.

Apparant variations in the external temperature environment of the transfer line when mounted in various gas chromatograph oven configurations has also led to a need to improve the method of connecting the transfer line to the oven while minimizing gas chromatograph thermomechanical differences in transfer line performance. An additional feedthrough device for this purpose is shown in Figure 7. The feedthrough consists primarily of a copper tube 903 whose internal diameter is substantially larger than the transfer line heater's outer diameter. The copper tube mounted in the gas chromatograph oven wall insulation 901 strives to be at constant temperature along its length. The copper tube is purposefully oversized to allow sufficient thermal grounding of the tube to the ambient to reduce the variations in temperature of the tube along its length, and also, to allow for variations in oven wall thickness among the various gas chromatograph instruments.

To reduce the thermal sink effect of the copper tube on the oven itself, the copper tube is not allowed to protude through to the oven interior, but instead extends into a refractory ceramic fibre block 902 containing a centrally located hole to allow the axial passage of the heated transfer line 904 into the oven. The block also serves to isolate the heated transfer line from exposure to the high temperature oven and to prevent convection flow out of the oven and over the transfer line. Use of the oven feedthrough permits a heated transfer line with less than a plus or minus 10 C variation when used with a gas chromatograph.

Claims

1. An apparatus for heating material with an electric current, comprising an electrically conductive heater tube (100;250) for encircling the material to be heated, the heater tube having first (125;337) and second (127;339) heater tube ends, first insulating means (110;260) substantially covering the heater tube for electrically insulating the heater tube, two first (120,123;300,305) current conducting means having first and second ends (330,335) electrically connected to the first (125;337) and second (127;339) heater tube ends respectively for conducting electrical current through the heater tube, said first current conducting means being arranged in proximity to the heater tube and being electrically



insulated from the rest of the heater tube by the first insulating means, and the other ends of said first current conducting means being arranged in proximity and opposite to each other, without touching, at a common point along the heater tube, and two second conducting means (120,123;310,315) attaching to the said other ends, respectively, of said first current conducting means near said common point, characterised by second insulating means (150,155;500) substantially covering the first and the second current conducting means and the heater tube for thermally insulating the heater tube and the first and the second current conducting means, an outer tube (160;340,345) substantially covering the second insulating means (150,155;400), and mounting means (180,350;355) connected to the outer tube in a mounting plane which passes through the common point along the heater tube (100;250) for mechanically mounting the apparatus and providing an exit for the second current conducting means which exit is substantially in said plane.

2. An apparatus as in claim 1, wherein the first current conducting means comprise wires (120,123), the wires also serving as second current conducting means near the common point.
3. An apparatus as in claim 1, wherein the first current conducting means comprise: first (300) and second (305) conduit tubes having lengths substantially equal to the distance between the first (337) and the second (339) heater tube ends respectively and the common point along the heater tube (250), and first (290) and second (295) connection wires connected to the first and second conduit tubes near the common point along the heater tube.
4. An apparatus as in claim 3, wherein the outer tube is composed of first (340) and second (345) outer tube pieces having first and second tube ends with the opposite ends of the outer tube pieces coming into close proximity at the common point along the heater tube.
5. An apparatus as in claim 3 or 4, wherein the first (300) and the second (305) conduit tubes are composed substantially of a stainless steel.
6. An apparatus as in any of the claims 3 through 5, wherein the first (300) and second (305) conduit tubes are composed of a tube with one or more predetermined variations in tube wall thickness.
7. An apparatus as in any of the claims 3 through 6, wherein the thermal resistances of both second conducting means (310,315) are approximately equal to the thermal resistance of the second insulating means (400).
8. An apparatus as in any of the preceding claims, characterized by a temperature sensor (140;270) thermally connected to the heater tube (100;250) and having leads (271,272) exiting the apparatus substantially in the mounting plane.
9. An apparatus as in any of the preceding claims, characterized in that the mounting plane is substantially perpendicular to the central axis of the heater tube (100;250).
10. An apparatus as in any of the preceding claims, characterized in that the heater tube (100;250) is composed substantially of a high resistance nickel/chromium alloy.
11. An apparatus as in any of the preceding claims, characterized in that the heater tube (100;250) is composed substantially of stainless steel.
12. An apparatus as in any of the preceding claims, characterized by first (210;440) and second (200;450) tube fittings connected to the first and second outer tube ends, respectively.



Revendications

1. Appareil pour chauffer un matériau avec un courant électrique, comportant un tube chauffant électriquement conducteur (100;250) pour entourer le matériau à chauffer, le tube chauffant présentant une première (15;337) et une seconde (127;339) extrémités de tube chauffant, des premiers moyens isolants (110;260) recouvrant substantiellement le tube chauffant pour isoler électriquement le tube chauffant, deux premiers moyens (120,123;300,305), conducteurs du courant, présentant une première et une seconde extrémités (330,335) électriquement reliées à la première (15;337) et à la seconde (127;339) extrémités du tube chauffant, respectivement, pour conduire le courant électrique à travers le tube chauffant, lesdits premiers moyens conducteurs du courant étant disposés à proximité du tube chauffant et étant électriquement isolés d'avec le reste du tube chauffant par les premiers moyens isolants, et les autres extrémités desdits premiers moyens conducteurs du courant étant disposées à proximité et en face l'une de l'autre, sans se toucher, en un point commun situé le long du tube chauffant, et deux seconds moyens conducteurs (120,123;310,315) étant attachés auxdites autres extrémités, respectivement, desdits premiers moyens conducteurs du courant, près dudit point commun, appareil caractérisé par des seconds moyens isolants (150,155;500) recouvrant substantiellement les premiers et les seconds moyens conducteurs du courant ainsi que le tube chauffant pour isoler thermiquement le tube chauffant et les premiers et les seconds moyens conducteurs du courant, par un tube extérieur (160;340,345) recouvrant substantiellement les seconds moyens isolants (150,155;400) et par des moyens de montage (180,350;355) reliés au tube extérieur dans un plan de montage qui passe par le point commun situé le long du tube chauffant (100;250) pour permettre de monter mécaniquement l'appareil et pour fournir une sortie pour les seconds moyens conducteurs du courant, sortie qui se trouve substantiellement dans ledit plan.
2. Appareil selon la revendication 1, dans lequel les premiers moyens conducteurs du courant sont constitués de fils conducteurs (120,123), les fils conducteurs servant également de seconds moyens conducteurs du courant près du point commun.
3. Appareil selon la revendication 1, dans lequel les premiers moyens conducteurs du courant sont constitués : d'un premier (300) et d'un second (305) tubes conducteurs de longueurs substantiellement égales à la distance entre la première (337) et la seconde (339) extrémités du tube chauffant, respectivement, et le point commun situé le long du tube chauffant (250) ainsi que d'un premier (290) et d'un second (295) fils conducteurs de liaison reliés au premier et au second tubes conducteurs près du point commun situé le long du tube chauffant.
4. Appareil selon la revendication 3, dans lequel le tube extérieur est constitué d'un premier (340) et d'un second (345) éléments de tube extérieur présentant une première et une seconde extrémités de tube, les extrémités opposées des éléments de tube extérieur venant très près l'une de l'autre au point commun situé le long du tube chauffant.
5. Appareil selon la revendication 3 ou 4, dans lequel le premier (300) et le second (305) tubes conducteurs sont substantiellement composés d'acier inoxydable.
6. Appareil selon l'une quelconque des revendications 3 à 5, dans lequel le premier (300) et le second (305) tubes conducteurs sont constitués d'un tube présentant une ou plusieurs variations prédéterminées d'épaisseur de paroi du tube.
7. Appareil selon l'une quelconque des revendications 3 à 6, dans lequel les résistances thermiques des deux seconds moyens conducteurs (310,315) sont approximativement égales à la résistance thermique des seconds moyens isolants (400).
8. Appareil selon l'une quelconque des revendications précédentes caractérisé par un détecteur de température (140;270) thermiquement relié au tube chauffant (100;250) et présentant des conducteurs (271,272) qui sortent de l'appareil substantiellement dans le plan de montage.
9. Appareil selon l'une quelconque des revendications précédentes caractérisé par le fait que le plan de montage est substantiellement perpendiculaire à l'axe central du tube chauffant (100;250).



10. Appareil selon l'une quelconque des revendications précédentes caractérisé par le fait que le tube chauffant (100;250) est substantiellement composé d'un alliage nickel/chrome à haute résistance.
11. Appareil selon l'une quelconque des revendications précédentes, caractérisé par le fait que le tube chauffant (100;250) est substantiellement composé d'acier inoxydable.
12. Appareil selon l'une quelconque des revendications précédentes, caractérisé par un premier (210;440) et un second (200;450) raccords tubulaires respectivement reliés à la première et à la seconde extrémités extérieures du tube.

Patentansprüche

1. Vorrichtung zum Erhitzen von Stoffen mit elektrischem Strom, die aufweist:
eine elektrisch leitende, den zu erhitzenden Stoff umgebende Heizröhre (100; 250) mit einem ersten (125; 337) und einem zweiten (127; 339) Heizröhrenende; einen ersten Isolator (110; 260), der die Heizröhre zu deren elektrischer Isolation im wesentlichen abdeckt; zwei erste (120, 123; 300, 305) stromdurchlässige Vorrichtungen mit ersten und zweiten, jeweils mit dem ersten (125; 337) bzw. dem zweiten (127; 339) Heizröhrenende elektrisch verbundene Ende (330, 335), um elektrischen Strom durch die Heizröhre zu leiten, wobei die ersten stromdurchlässigen Vorrichtungen in der Nähe der Heizröhre angeordnet und vom Rest der Heizröhre durch den ersten Isolator elektrisch isoliert sind, und die anderen Enden der ersten stromdurchlässigen Vorrichtungen einander benachbart und gegenüberliegend, jedoch berührungslos, an einem gemeinsamen Anschlußpunkt der Heizröhre angeordnet sind; und
zwei zweite stromdurchlässige Vorrichtungen (120, 123; 310, 315), die jeweils mit den beiden anderen Enden der ersten stromdurchlässigen Vorrichtungen in der Nähe des gemeinsamen Anschlußpunktes verbunden sind,
gekennzeichnet, durch
einen zweiten Isolator (150, 155; 400), der die ersten und zweiten stromdurchlässigen Vorrichtungen und die Heizröhre im wesentlichen abdeckt, um die Heizröhre und die ersten und zweiten stromdurchlässigen Vorrichtungen thermisch zu isolieren,
eine äußere Röhre (180; 340, 345) die den zweiten Isolator (150, 155; 400) im wesentlichen umgibt, und eine Befestigungsvorrichtung (180, 350; 355), die mit der äußeren Röhre in einer Befestigungsebene verbunden ist, welche durch den gemeinsamen Anschlußpunkt entlang der Heizröhre (100; 250) verläuft, um die Vorrichtung mechanisch zu befestigen und einen Ausgang für die zweiten stromdurchlässigen Vorrichtungen zu schaffen, der im wesentlichen in dieser Ebene liegt.
2. Vorrichtung nach Anspruch 1, bei der die ersten stromdurchlässigen Vorrichtungen Drähte (120, 123) aufweisen, die auch als zweite stromdurchlässige Vorrichtungen in der Nähe des gemeinsamen Anschlußpunktes dienen.
3. Vorrichtung nach Anspruch 1, bei der die ersten stromdurchlässigen Vorrichtungen aufweisen:
ein erstes (300) und ein zweites (305) Leitungsrohr, deren Längen im wesentlichen gleich den entsprechenden Strecken zwischen dem ersten (137) bzw. dem zweiten (339) Heizröhrenende und dem gemeinsamen Anschlußpunkt an der Heizröhre (250) sind, und erste (290) und zweite (295) Verbindungsdrähte, die an dem ersten und dem zweiten Leitungsrohr in der Nähe des gemeinsamen Anschlußpunktes längs der Heizröhre angeschlossen sind.
4. Vorrichtung nach Anspruch 3, bei der die äußere Röhre aus einem ersten (340) und einem zweiten (345) äußeren Röhrenabschnitt mit ersten und zweiten Röhrenenden zusammengesetzt ist, wobei sich die entgegengesetzten Enden der äußeren Röhrenabschnitte in unmittelbarer Nähe des gemeinsamen Anschlußpunktes an der Heizröhre befinden.
5. Vorrichtung nach Anspruch 3 oder 4, bei der das erste (300) und das zweite (305) Leitungsrohr im wesentlichen aus rostfreiem Stahl bestehen.



6. Vorrichtung nach einem der Ansprüche 3 bis 5, bei der das erste (300) und das zweite (305) Leitungsrohr aus einem Rohr mit einer oder mehreren vorgegebenen Abweichungen in der Rohrwandstärke bestehen.
- 5 7. Vorrichtung nach einem der Ansprüche 3 bis 6, bei der die thermischen Widerstände beider zweiter stromdurchlässiger Vorrichtungen (310, 315) ungefähr gleich dem thermischen Widerstand des zweiten Isolators (400) sind.
- 10 8. Vorrichtung nach einem der vorangehenden Ansprüche, **gekennzeichnet**, durch einen Temperatursensor (140; 240), der thermisch mit der Heizröhre (100; 250) verbunden ist und Anschlußleitungen (271, 272) aufweist, welche die Vorrichtung im wesentlichen in der Befestigungsebene anregen.
- 15 9. Vorrichtung nach einem der vorangehenden Ansprüche, dadurch **gekennzeichnet**, daß die Befestigungsebene im wesentlichen senkrecht zur Mittelachse der Heizröhre (100; 250) verläuft.
- 20 10. Vorrichtung nach einem der vorangehenden Ansprüche, dadurch **gekennzeichnet**, daß die Heizröhre (100; 250) im wesentlichen aus einer hochohmigen Nickel/Chrom-Legierung besteht.
11. Vorrichtung nach einem der vorangehenden Ansprüche, dadurch **gekennzeichnet**, daß die Heizröhre (100; 250) im wesentlichen aus rostfreiem Stahl besteht.
- 25 12. Vorrichtung nach einem der vorangehenden Ansprüche, **gekennzeichnet**, durch erste (210; 400) und zweite (200; 450) Paßstücke, die mit dem ersten bzw. dem zweiten äußeren Röhrenende verbunden sind.

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