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Ward

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[54] **SUBJECTING SAMPLES TO ELEVATED TEMPERATURE**

[75] Inventor: **Henry Arthur John Ward,** Essex, England

[73] Assignee: **Electrothermal Engineering Limited,** London, England

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[52] **U.S. Cl.** **432/226,** 432/5, 432/231
[51] **Int. Cl.** **F27b 5/02**
[58] **Field of Search** 432/1, 5, 219, 220, 225, 432/226, 231

[56] **References Cited**

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Primary Examiner—John J. Camby
Attorney, Agent, or Firm—Finnegan, Henderson, Farabow and Garrett

[57] **ABSTRACT**

There is disclosed apparatus for use in subjecting samples to elevated temperature. The apparatus includes first chambers for receiving samples, part of each of the first chambers being within a second chamber which is out of communication with the first chambers and there being a third chamber which communicates by way of entrance openings with the first chambers, each first chamber having an exit opening, there being air in all the first, second and third chambers, an air inlet to the third chamber and means for heating the air. In use of the apparatus, heated air passes from the third chamber by way of the entrance openings into the first chambers, past the samples and out of the first chambers by way of the exit openings and heated air in the second chamber is in contact with the walls of the first chambers and heats them.

8 Claims, 10 Drawing Figures

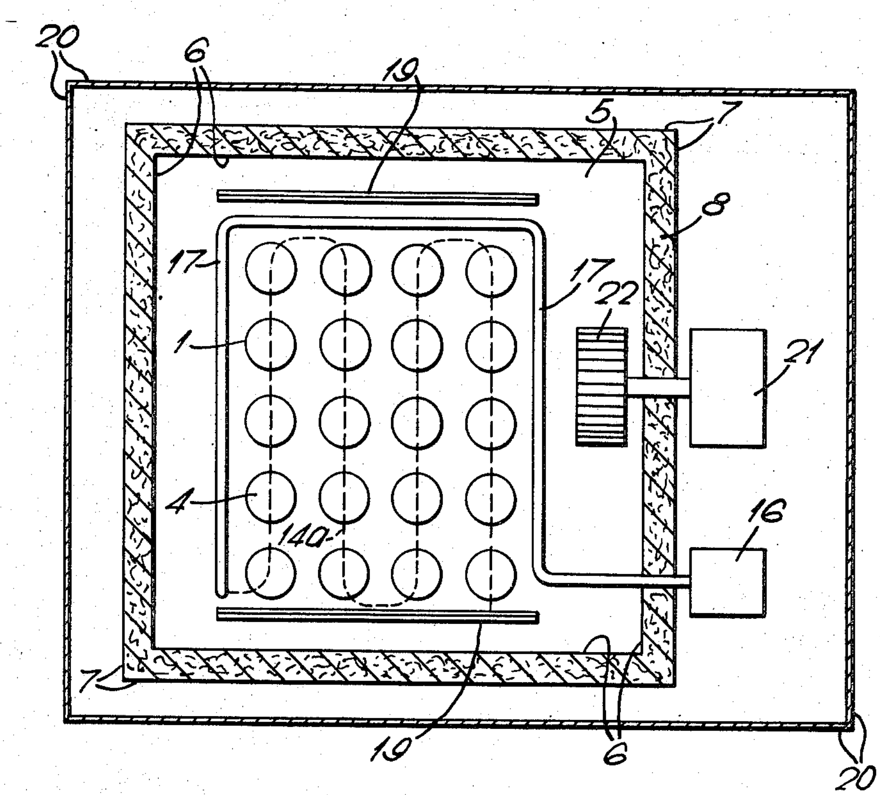

FIG. 1.

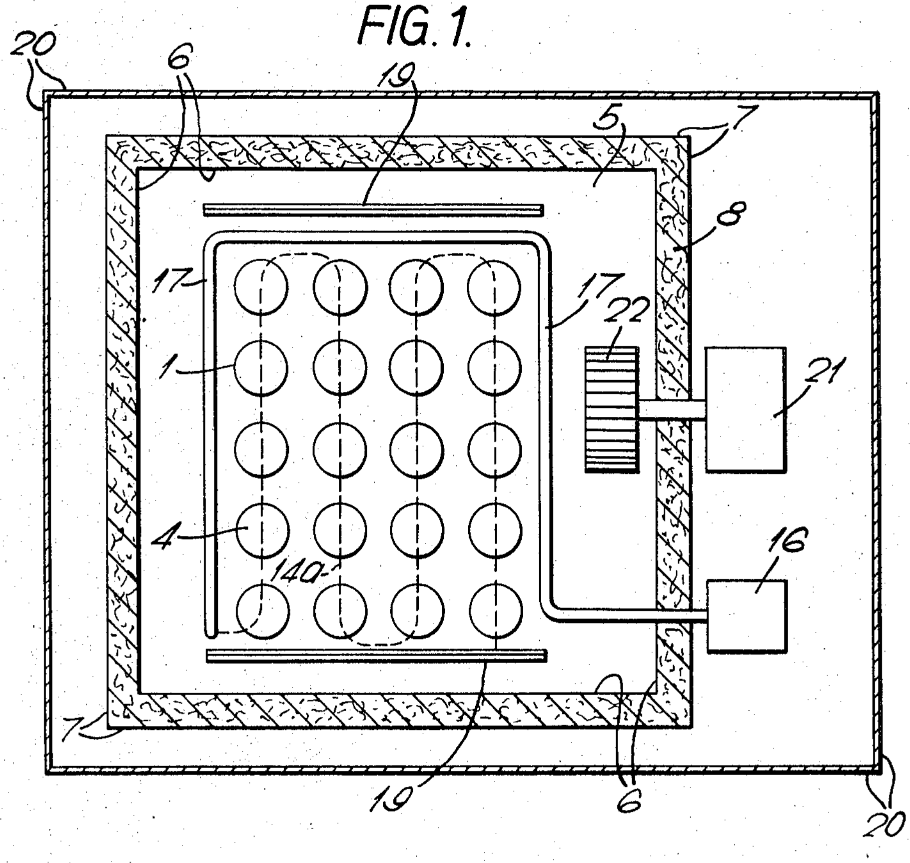
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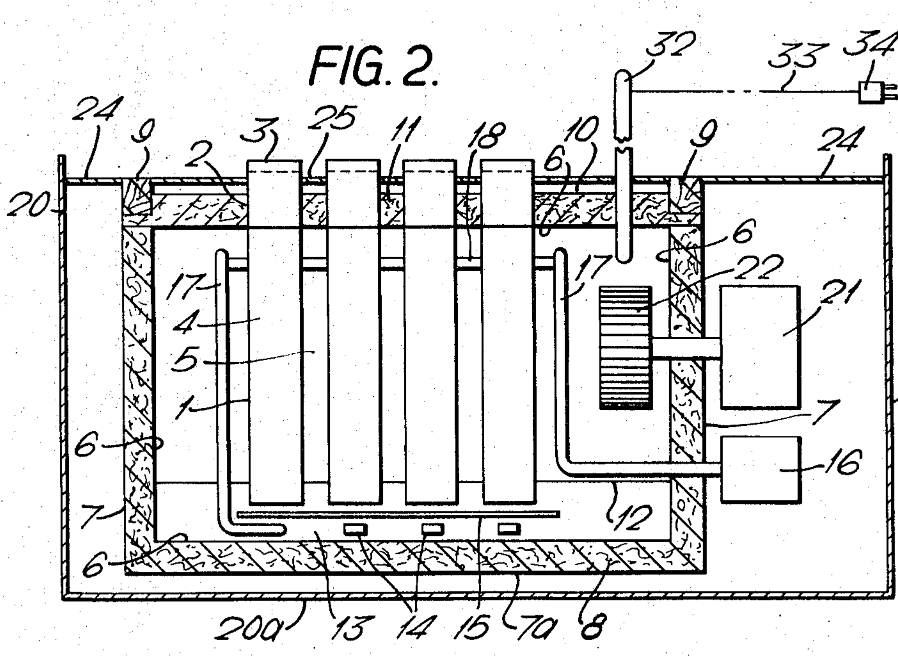
FIG. 2.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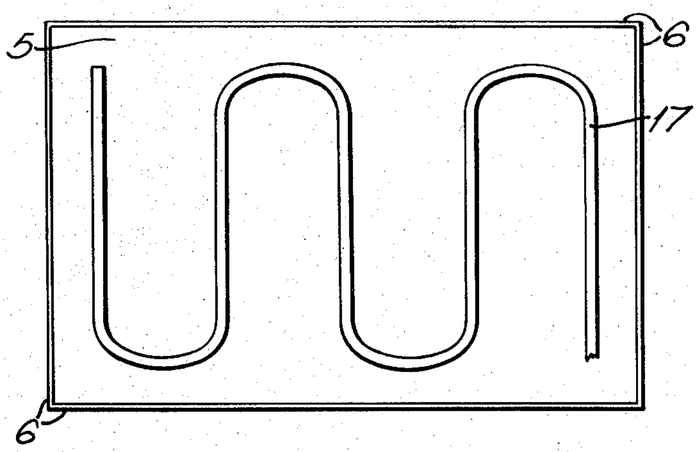


FIG. 3.

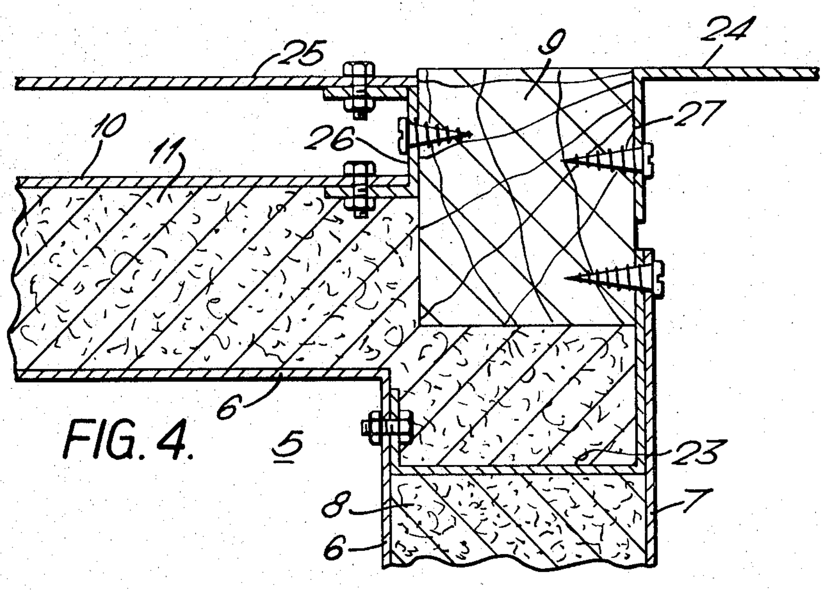


FIG. 4.

FIG. 7(a)

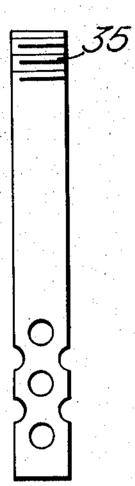


FIG. 7(b)

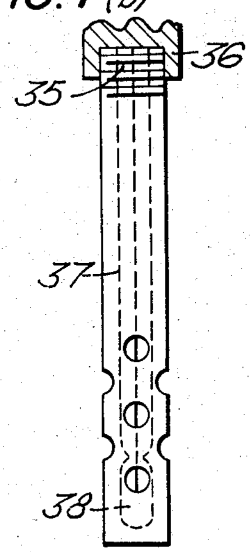


FIG. 7(c)

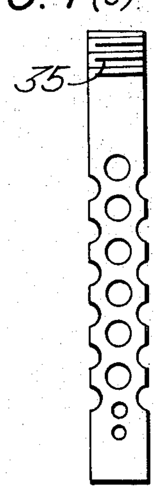
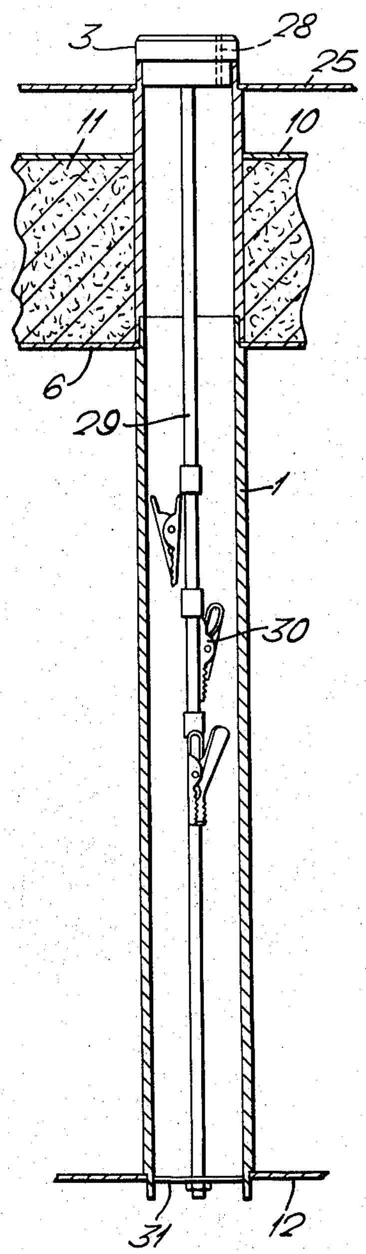


FIG. 5.



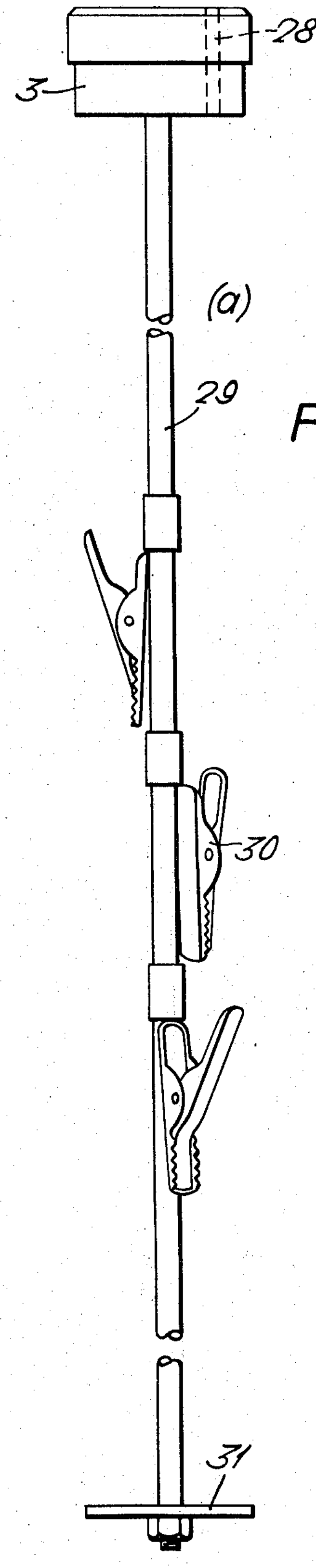


FIG. 6. (a)

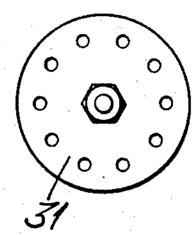


FIG. 6(b)

SUBJECTING SAMPLES TO ELEVATED TEMPERATURE

BACKGROUND OF THE INVENTION

This invention relates to subjecting samples to elevated temperature, for example for carrying out an accelerated aging test on a sample.

One form of accelerated aging test of, for example, a sample of rubber or plastics material consists in subjecting a test sample to controlled deterioration by air at elevated temperature and at atmospheric pressure, after which one or more physical properties of the sample is or are measured and compared with a corresponding property or properties of a similar sample which has not been so heated.

For the heating of test samples, two types of ovens including chambers for receiving samples have been proposed. One is an oven incorporating a heated oil bath for heating the chambers and the other an oven incorporating a heated block of solid material which receives the chambers for heating them. The former usually has the disadvantages of being too limited in temperature and cumbersome, and the latter usually has the disadvantages of being cumbersome and suffering from inherent temperature gradient problems.

THE INVENTION

The object of the invention is to provide apparatus for use in subjecting a sample to elevated temperature including a chamber for receiving such a sample, in which heated air is used for heating the chamber.

According to the invention there is provided apparatus for use in subjecting a sample to elevated temperature, including:

a. first wall means, defining a first chamber for receiving a sample and having an entrance opening and an exit opening,

b. second wall means, defining (*i*) a second chamber within which part of said first wall means is disposed and which is out of communication with said first chamber and (*ii*) a third chamber which communicates by way of said entrance opening with said first chamber, there being air in all three chambers,

c. an air inlet to said third chamber and

d. means for heating the air, whereby heated air can pass, in use of the apparatus, from said third chamber by way of said entrance opening into said first chamber, past the sample and out of said first chamber by way of said exit opening and heated air in said second chamber is in contact with said first wall means and heats it.

The testing could be an artificial aging test.

There could be at least one heater in said second chamber for heating the air in that chamber and/or at least one heater in said third chamber for heating the air in that chamber, but it is preferred to have at least one heater in said second chamber for heating the air in that chamber and an air inlet duct to said third chamber running through said second chamber for preheating the air supplied to said third chamber by heat exchange with the air in said second chamber (the air supplied to said third chamber not being further heated whilst in said third chamber). There may be a plurality of such first wall means, each defining a respective first chamber for receiving a sample and having a respective entrance opening and a respective exit opening, in which case they (the first chambers) are all spaced

apart so that their entrance openings, by way of which they communicate with said third chamber, can receive from that chamber air at substantially equal temperature. This may be achieved by having the above-mentioned air inlet duct extend across said third chamber, for example in serpentine form, and formed with holes of varying size distributed along it so that air can escape into said third chamber in a substantially uniform manner. It helps to provide a grid, for example of wire, between these holes and said entrance openings.

An example in accordance with the invention is described below with reference to the accompanying drawings, in which:

FIG. **1** shows a diagrammatic sectional plan view of apparatus for use in testing samples at elevated temperature,

FIG. **2** shows a sectional side view of the apparatus,

FIG. **3** shows a detail of part of the apparatus,

FIG. **4** shows a detail of part of what is shown in FIG. **2,**

FIG. **5** shows in greater detail means forming one of the first chambers and its associated parts, including sample carrying means,

FIG. **6***a* shows the sample carrying means per se and FIG. **6***b* an end view of part of the sample carrying means, and

FIGS. **7***a* and **7***c* show different devices that can be used with a thermometer in the apparatus, FIG. **7***b* showing the manner of using one of them.

In the example, twenty thick-walled vertical aluminum tubes **1** of constant circular cross-section are used to hold samples of rubber or plastics material to be subjected to an accelerated aging test. Each tube is at least 30 cm. long and is dimensioned in accordance with British Standard Specification No. 903 Part 19 (1956). At the top of each tube is a tube extension **2** made of polytetrafluoroethylene and to that is attached a top cap **3** of polytetrafluoroethylene from which, in all cases except one, a conventional sample carrier depends, this being capable of carrying samples of material spaced apart along the tube and out of contact with the wall of the tube. Each tube provides a chamber **4** and most of each of them is within a chamber **5** formed by an oblong aluminum tank having walls **6,** this being within another tank made of mild steel and having side walls **7** and a bottom wall **7***a*, there being glass wool or other heat-insulating material **8** of two inches thickness between the two tanks. The tank having walls **6** and the tank having side walls **7** and bottom wall **7***a* are secured to a square wooden frame **9** in a manner to be described below, a mild steel intermediate plate **10** being provided with glass wool or other heat-insulating material **11** of two inches thickness between it and the uppermost wall **6.** The tank having walls **6** has a partition **12** extending across it so that the tank forms another chamber **13.** The chambers **4** do not communicate with the chamber **5,** but their lower ends open into the chamber **13.** In this chamber there is a serpentine pipe **14** and between this and the lower ends of the chambers **4** there is a steel wire mesh **15.** Cold air is drawn in from outside the apparatus and supplied by means of a diaphragm pump **16** and it flows through a tube in the chamber **5** in which it is heated, this tube having two serpentine portions **17** as shown in FIG. **3** (which for simplicity shows no other details of the apparatus other than the tank having walls 6) at opposite sides of the chamber and a straight portion 18 interconnecting them. Then the heated air flows from the tube in the chamber 5 into the pipe 14 in the chamber 13. This pipe is closed at its downstream end and has holes of varying size distributed along it so that it distributes the heated air substantially uniformly over the chamber 13, 14*a* in FIG. 1 showing path of the pipe 14. The heated air flows through the wire mesh 15, which increases the uniformity of its distribution, into the lower ends of the chambers 4. It flows upwardly over the samples and out through small vent holes (see FIGS. 5 and 6*a*) in the top caps 3 to atmosphere.

In the chamber 5, at opposite sides of it, there are two panel-type electric heaters 19, consisting of a wire mesh heater sandwiched between two sheets of asbestos, which are heated to incandescence but heat the air, which is trapped, in the chamber 5. There is heat exchange between this air and the air in the tube having the parts 17 and 18 and the air in the chamber 13 has in consequence substantially the same temperature as that in the chamber 5.

There is a mild steel casing having side walls 20 and a bottom wall 20*a* in which is disposed the tank having side walls 7 and bottom wall 7*a*, and spaced from it by at least one inch, this containing, outside the latter tank, the diaphragm pump 16 and an electric motor for driving it, and an electric motor 21 which drives a fan 22 inside the chamber 5 and which distributes substantially uniformly the air in the chamber so that it is all substantially at the same temperature. Heat sinks of aluminum are fixed to the stator of the motor 21 and the motor which drives the diaphragm pump 16, to keep the bearings cool and prevent seizing up.

Attached to the side walls 20 of the outer mild steel casing is a mild steel panel 24 having an opening which carries a bracket 27 around its inside edge which is screwed by wood screws to the wooden frame 9 (see FIG. 4). The inner tank having walls 6 is carried by the wooden frame 9 by means of stainless steel L-shaped brackets 23, the latter being fixed by wood screws to the frame 9 and bolted via flanges to the tank, the wood screws also fixing the walls 7 to the frame 9. The brackets 23 provide a long heat path between the walls 6 and the frame 9. On the inner side of the frame 9 a square bracket 26 of U-shaped cross-section is fixed by wood screws, and bolted to the lower flange of the bracket 26 is the intermediate plate 10 and bolted to the upper flange is a Duralumin facia panel 25 through which the tops of the extensions 2 extend. There is a one inch air gap between the facia panel 25 and the intermediate plate 10.

Referring to FIGS. 5, 6*a* and 6*b*, each top cap 3 (having a vent hole 28) carries a stainless steel rod 29 to which are fixed up to five clips 30 (three only being shown) for carrying respective samples to be heated, and at the opposite end of the rod 29 is fixed an apertured aluminum catcher plate 31 (shown in end view in FIG. 6*b*) for preventing samples falling from the clips into the chamber 13. The tubes 1 should not be made of or contain copper or copper alloy, except perhaps if they are clad with appropriate material, for example by being chromium or cadmium plated. Sealing rings (not shown) of a silastomer surround the tubes 1 where they pass through the partition 12.

The temperature in the chamber 5 is monitored by a variable-setting mercury-type contact thermometer 32 which includes a tungsten electrode which can be adjusted in position so that contact between it and the mercury occurs when the air temperature in the chamber is 70°, 100°, 125°, 150°, 175°, 200° or 250°C. When contact is made a signal supplied by way of a cable 33 and a jack plug 34, which engages in a socket not shown, ensures that no current can be supplied to the heaters 19 and when contact is broken it is arranged that current can be supplied to them, so that the temperature in the chamber 5 is maintained substantially at the value set at the contact thermometer. When the jack plug is not in its socket, current cannot be supplied to the heaters. The contact thermometer 32, cable 33 and jack plug 34 can be removed for transit of the apparatus.

The chamber 4 which does not contain a sample carrier instead contains the lower end of a thermometer which projects through the top cap 3 of that chamber.

In use of the apparatus, samples are held by the clips 30, a flowmeter in the apparatus measures the rate of supply of air to the tube having the parts 17 and 18 and a valve controlled by the flowmeter ensures that the rate of supply of air per hour is equal to the number of chambers 4 times the volume of each chamber times a factor not less than three and not more than ten. The samples are subjected to a constant elevated temperature and substantially atmospheric pressure for an appropriate time and then one or more of their physical properties is or are measured and compared with a corresponding property or corresponding properties of similar samples which have not been heated.

Another electric motor drives a fan, which cools the space, which contains this motor and fan, between the tank having the side walls 7 and the casing having side walls 20, which is formed with louvres for the inlet and outlet of cooling air.

Control of the heating by the heaters 19 is effected by altering, by turning a heat control knob, the ratio between "on" and "off" periods of a substantially sinusoidal current which is supplied to the heaters when the contact thermometer allows it, switching on and off being effected when the voltage passes through zero. The heat control knob and the flowmeter are situated on a control panel accessible at the outside of the apparatus and on this panel there are also a lamp for indicating whether or not current is being supplied to the heaters and another lamp for indicating whether or not the mains are switched on, switching on being effected by a master switch on the control panel which, when open, disconnects all the electrical apparatus from the mains.

In addition, there is a safety thermostat which will switch off the supply to the heaters in the event of the temperature in the chamber 5 rising to, say, 10°C. above that set on the contact thermometer, due to a failure.

The contact thermometer 32 has its stem, projecting below the scale and into the chamber 5 and including a mercury filled bulb, surrounded by one or the other of two so-called "calibration tubes", one for use when the temperature within the tubes 1 is to be 125°C. or lower and the other when this temperature is to be above 125°C. The tubes are made of chromium-plated brass and have a wall thickness of 1/16 inch. The first is shown in FIGS. 7*a* and 7*b* and has a screw-thread 35 at its upper end which is screwed into the contact thermometer housing **36** (see FIG. 7*b*). At its lower end the tube has two diametrically opposite sets each of three holes and two diametrically opposite sets each of two holes, all the holes being ⅜ inch in diameter and passing right through the wall of the tube. The stem, made of glass, of the thermometer is shown at **37** and the mercury bulb at **38**. The second tube differs from the first in that it has, as shown in FIG. 7*c*, two diametrically opposite rows each of eight holes and two diametrically opposite rows each of six holes, all the holes passing right through the wall of the tube and all being ⅜ inch in diameter except the lowest two of the eight-holed rows, which are ¼ inch in diameter.

In each case the tube is shown from one side; its appearance from the opposite side is the same. The "calibration tubes" ensure that when the temperature of the chamber **5** is rising, the reading of the contact thermometer lags the actual temperature in the chamber **5**. The arrangements of holes in the tubes are chosen to ensure substantial equality between the temperature within the tubes **1** and the temperature in the chamber **5** and to ensure that this temperature is about equal to that set on the contact thermometer.

I claim:

1. Apparatus for use in subjecting a sample to elevated temperature, including:

a. first wall means, defining a first chamber for receiving a sample and having an entrance opening and an exit opening,

b. second wall means, defining (i) a second chamber within which part of said first wall means is disposed and which does not communicate with said first chamber and (ii) a third chamber which communicates by way of said entrance opening with said first chamber, there being air in all three chambers,

c. an air inlet to said third chamber and

d. means for heating the air, whereby heated air can pass, in use of the apparatus, from said third chamber by way of said entrance opening into said first chamber, past the sample and out of said first chamber by way of said exit opening and heated air in said second chamber is in contact with said first wall means and heats it.

2. Apparatus according to claim **1**, including:

a. at least one heater in said second chamber for heating the air in that chamber and

b. an air inlet duct to said third chamber running through said second chamber for preheating the air supplied to said third chamber by heat exchange with the air in said second chamber.

3. Apparatus according to claim **1**, including a plurality of such first wall means, each defining a respective first chamber for receiving a sample and having a respective entrance opening and a respective exit opening, the first chambers being spaced apart so that their entrance openings, by way of which they communicate with said third chamber, can receive from that chamber air at substantially equal temperature.

4. Apparatus according to claim **2**, including a plurality of such first wall means, each defining a respective first chamber for receiving a sample and having a respective entrance opening and a respective exit opening, the first chambers being spaced apart so that their entrance openings, by way of which they communicate with said third chamber, can receive from that chamber air at substantially equal temperature.

5. Apparatus according to claim **4**, wherein said air inlet duct extends across said third chamber and has formed in it holes of varying size distributed along it so that air can escape into said third chamber in a substantially uniform manner.

6. Apparatus according to claim **5**, wherein said air inlet duct is of serpentine form.

7. Apparatus according to claim **5**, including a grid between said holes and the said entrance openings.

8. Apparatus according to claim **1**, wherein the first chamber has in it carrying means for carrying a sample.

* * * * *