Aug. 23, 1966 R. E. MANNING ETAL 3,267,687

CONSTANT TEMPERATURE DEVICE

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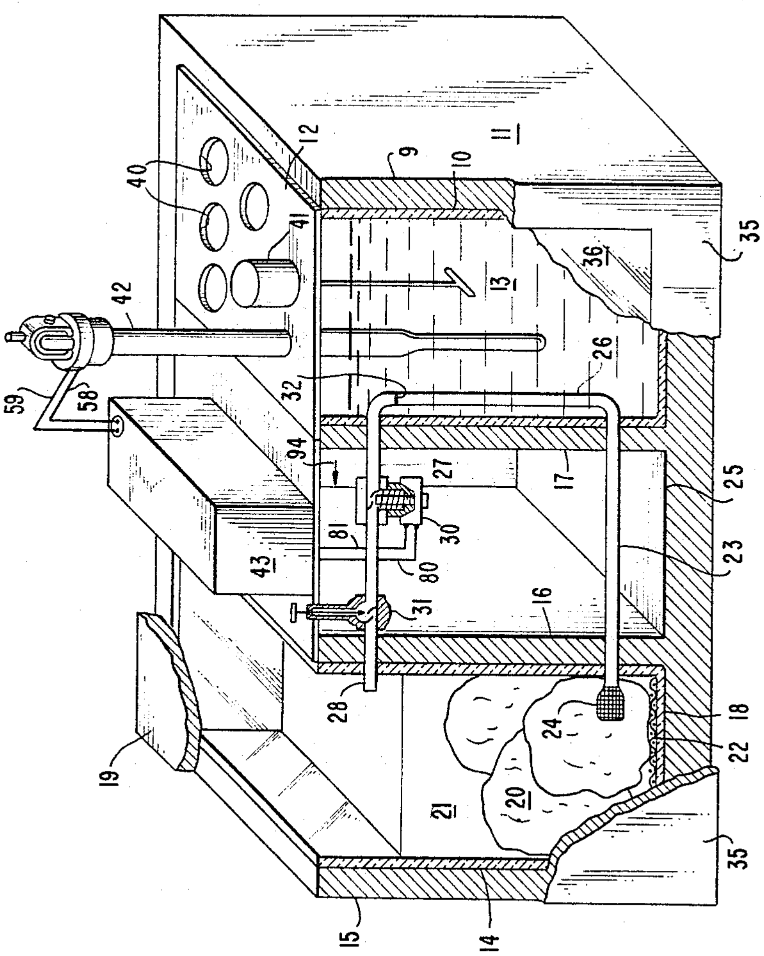
FIG. 1



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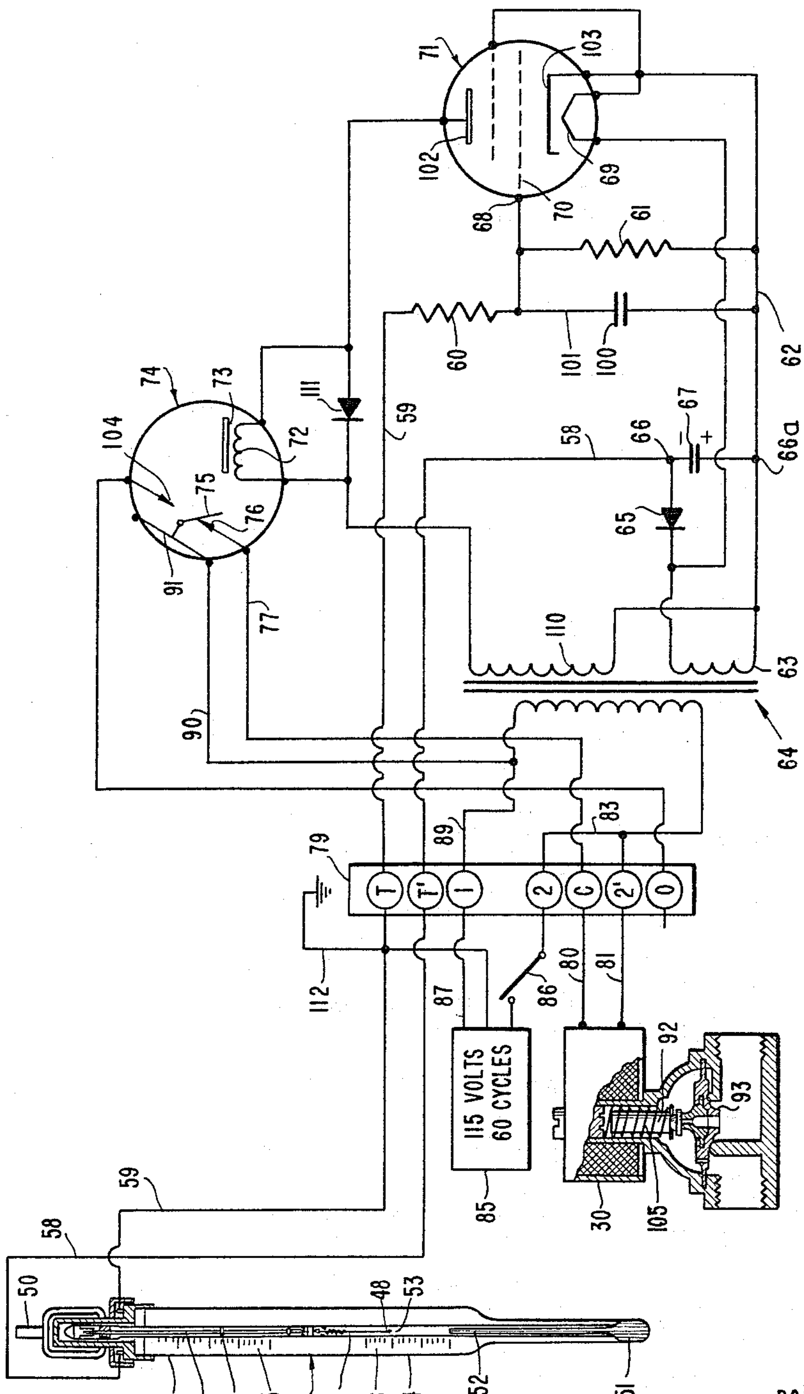

FIG. 2

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CONSTANT TEMPERATURE DEVICE

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This invention relates to a constant, low temperature bath device for conducting experiments, such as viscosity measurements, at a low temperature which is closely controlled within narrow limits.

A related bath or bath device is described in copending application Ser. No. 261,174, filed February 26, 1963, now Patent 3,214,937, issued November 2, 1965. Like that device, the present bath is of value for testing lubricants, fuels, hydraulic fluids, and the like at low temperatures. For example, military installations in arctic climates have generated much interest in the viscosity behavior of lubricants and other fluids at reduced temperatures, and as a natural consequence, considerable attention has been shown to instruments and apparatus for measuring viscosity at these temperatures. Conventional refrigeration equipment can, of course, be used to hold a test material at low temperature, but it tends to be quite bulky and to require valuable space; and furthermore, such equipment is expensive, complicated to install and operate, and apt to require expert maintenance.

According to the invention, there is provided a cooling device which is of desirable simplicity and sound operation, like the bath of said prior application; additionally it affords unique advantages which enhance its utility. More particularly, it is characterized by an improved cooling system which enables the cooling rate to be closely controlled without reliance on an added heater; this means that only necessary heat is removed, avoids overcooling, and affords economies in the use of coolant. Furthermore, space is conserved, and the compactness of the device is increased. Compactness is also favored by the disposition of the coolant reservoir and the bath container, these being arranged to have their fluid levels at more or less the same heights, so that accessibility as well as compactness is improved. Other advantages will become apparent from the ensuing description.

The present device comprises generally a bath section for holding bath fluid to be cooled and maintained at a constant low temperature, a reservoir section for holding fluid coolant, and a central section intermediate the other two. Temperature control means, suitably comprising electrical switching circuits, are associated with the control section. The bath and reservoir sections, as also the central section, have the same floor level, and as noted, the first two sections have substantially the same fluid levels. Temperature-sensitive means extend into the bath fluid and serve to actuate said control means.

Fluid coolant passes between the reservoir and bath sections, suitable conduit or transfer means being provided for passing coolant from one section to the other, preferably in a lateral direction, and back again, also preferably laterally. In the bath section most of the coolant flow is preferably in an up-and-down direction, although it may be in other directions also, including a spiral, zigzag, or other circuitous route. Valve means are provided in the coolant transfer means which are responsive to the temperature control means for controlling flow of coolant.

When the temperature-sensitive means in the bath fluid signals a high bath temperature, the electrical switching circuits or means are actuated to operate the valve means, thereby to open the latter to permit coolant flow therethrough; and when the bath is sufficiently cooled, the temperature-responsive means signal to inactuate the switching circuits and the valve means close to shut off

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coolant flow. Overcooling of the bath does not take place, as will be described.

While a number of coolants are suitable, commercial Dry Ice, or frozen carbon dioxide, is admirable, and the invention may be described in detail in connection with this material. The Dry Ice is placed in chunks in the reservoir together with a small amount of a solvent therefor. Cooled solution, comprising solvent together with some dissolved Dry Ice, flow from the reservoir into the transfer means, which extends through the bath, where the solution cools the bath fluid by indirect heat exchange. Heat abstracted from the bath boils out dissolved carbon dioxide from the solution, and the gas and some liquid flow on through the conduit means and back to the Dry Ice reservoir. In turn, additional cold solution flows from the reservoir to the bath via the conduit means between them and in this way the temperature of the bath may be brought down to a desired low temperature, at which point the flow of gas and liquid through the conduit means is reduced, this in turn reduces the flow of cold solution from the reservoir into the conduit means, and a fine control temperature-regulating system is actuated to maintain the temperature.

The invention may be better understood by referring to the accompanying drawings, which are diagrammatic, and in which:

FIG. 1 is a perspective view of the constant temperature device, with portions broken away; and

FIG. 2 is a circuit diagram of a switching system.

Referring to FIG. 1, the constant temperature device comprises a bath container **10** supported in a cabinet **11** which encloses the container on all sides. The container, which is in the form of an open top vessel of glass or stainless steel or other suitable material, is disposed on one side of the cabinet and is covered by a lid **12** which provides a working area for placing test equipment in the container. The latter is nearly filled with bath liquid **13** comprising the liquid which is to be cooled and maintained at constant temperature. On the other side of cabinet **11** is a reservoir **14** comprising a thick-walled tank for holding coolant. The tank is insulated on all sides by walls of insulating material, as at **15** and **16**, as is container **10** by walls **9** and **17**. The floor **18** and lid **19** are similarly protected by or constructed of insulating material.

The coolant in reservoir **14** comprises chunks or blocks **20** of Dry Ice and are immersed in a Dry Ice solvent **21** such as methanol or acetone. The blocks rest on a screen partly indicated in section at **22** which is disposed a small distance above floor **18**, say ¼ to 1 inch, preferably ½ to ¾ inch, and thus solvent has free access to the blocks. However, the screen is dispensable. The level of solvent in the tank may be sufficient to cover the blocks, as shown, or may only partially cover them.

In the control part of cabinet **11** there is disposed a compartment **25** between the two side compartments for the disposition or support of control means comprising a conduit member or coolant flow tube **23**, an electric control valve **30** (shown partly in section), a hand throttle valve **31**, and, disposed above the compartment, although it may be located in any suitable position, near or remote, an electrical switching system **43**. The coolant flow tube **23** extends horizontally into the reservoir **14** a short distance where its inlet end, which is disposed well below the level of coolant **21**, is provided with a screen **24**; and opposite such end the tube extends horizontally into the container **10**, where it turns and extends vertically, as at **26**, then turns again to a horizontal disposition and reenters compartment **25**, note section **27**, and finally extends again into reservoir **14** by means of outlet **28** disposed above the level of coolant **21**. Outlet **28** may also be located below the coolant level, but preferably is above; in any case it is above the inlet 24.

Lid 12 is provided with several openings, such as at 40, for receiving conventional instruments like viscometers. Another opening is occupied by a motor-operated stirrer 41, and another by a conventional thermoregulator 42 shown with leads 58 and 59 connected thereto which extend into the switching system 43. Another pair of leads 80 and 81 are shown connecting the coil of the solenoid valve 30 to the system 43.

A side panel 35 is adapted to cover the side of the device nearest the observer, and in the bath compartment 10 this panel suitably has a window 36 to enable the operator to watch the progress of tests. It is helpful to provide a light to aid visibility and a small electric heater to prevent frosting and condensation on the window; these are not shown for sake of a simplified showing. Also not shown are a pump and connections for removing bath fluid from container 10 and circulating the same to equipment external to the container.

The operation of the device may be described briefly as follows.

Coolant 21 in reservoir 14, comprising, for example, a methanol-carbon dioxide solution, will flow into tube 23 through inlet screen 24, the valves 30 and 31 being open for this purpose, and will reach a level indicated at 32 in section 26 of the tube, this level corresponding to the level in reservoir 14. The bath liquid 13, for example methanol, transfers heat to the cold solution in tube 26, causing carbon dioxide to boil out of solution, and this gas will push or entrain some methanol through section 27 of the tube in the direction of arrow 94, the mixture passing forcefully through the open valves 30 and 31 and into reservoir 14. It may exit from tube 27 with such force as to strike the opposite wall of the reservoir. Because of the pressure differential created, additional saturated solution from the reservoir enters inlet 24 of the tube. Thus, if valves 30 and 31 remain open, circulation is continuous and the bath liquid 13 will cool.

When the bath cools to a desired control temperature, the thermoregulator 42 operates through switch system 43 to close the solenoid valve 30, this action being described below. Carbon dioxide continues to boil out of the liquid in tube 26 but now the pressure in this tube above the liquid builds up rapidly, forcing the liquid back through inlet 24 into the reservoir. Hence, the cooling ceases, and the bath begins to warm from heat transfer from the surroundings. By virtue of the rapid action of the cooling cessation step, undercooling (sometimes designated overcooling) is avoided and fine control of the bath temperature is possible. It will be noted that cessation of cooling is self-regulated, i.e., a self-ceasing cooling action is involved.

When the temperature moves slightly above the control point, the thermoregulator again signals valve 30 to open, the pressure in tube 26 is reduced, cold solution again fills tube 26, and cooling commences again. In this manner the bath can be held to within slight variations above and below a desired temperature.

Throttle valve 31 can be adjusted such that the rate of cooling more nearly approximates the rate of heat gain from the surroundings. This tends to reduce the degree of cycling of the temperature. The motor-stirrer 441 tends to establish a uniform temperature throughout the bath. Carbon dioxide gas is vented from reservoir 14 by means not shown.

From the foregoing operation, it may be apparent that flow tube 23 comprises the inlet portion, the tube 27 the outlet portion, and the tube 26 the heat transfer portion of the coolant transfer means. Portion 26 thus comprises coolant flow transfer means as well as heat transfer means, and is constructed of suitable heat conducting material.

The desired low temperature of the bath will depend in part on the solvent, but with this in mind, a wide range of temperatures is available, extending for example from just below room temperature down to about −100 degrees Fahrenheit. The bath liquid 13, of course, is one that is liquid and stable at the chosen temperature, and may include wax-free hydrocarbons, acetone, methanol, etc. If desired, the bath liquid may be the same as the carbon dioxide solvent, described below.

Accurate control over the bath test temperature is possible, the variation being less than plus or minus 0.04 degree F. at bath temperatures of 0 to −65 degrees F., and in many cases the variation being no more than plus or minus 0.01 degree F. For bath temperatures between room and 0 degree F. the variation may be less than 0.02 degree F.

The carbon dioxide solvent may suitably be chosen from low molecular weight compounds including alcohols, ketones, ethers, esters, aldehydes, organic acids, etc. Low molecular weight compounds are desirable because, in general, their viscosity is low and they flow easily at the low temperatures involved. Illustrative specific solvents are methanol, ethanol, propanol, isopropanol, n-butanol, isobutanol, acetone, methyl ethyl ketone, methyl acetate, ethyl acetate, propyl acetate, butyl acetate, methyl butyrate, propyl butyrate, acetaldehyde, propionaldehyde. Other compounds are the various pentanes of hexanes, and methylene chloride.

The lateral position of the Dry Ice tank 14 relative to the bath 10 is a convenient one for replenishing Dry Ice and increases the compactness of the device. It will be understood that the tank may have other positions relatively to the bath and that forced flow of the Dry Ice solution may be employed. Also, the tube or member 23 may be spaced at varying heights above the floor of compartment 25, resulting in varying lengths of the cooling section 26 in the bath. Tube 23 may have any suitable cross-sectional shape. It will be appreciated that the rate of heat transfer increases with increasing surface area, all other things being the same. Any suitable material may be used to construct the tube 23, but preferably the material is a metal like stainless steel, brass, aluminum, copper, nickel, zinc, iron, titanium, molybdenum etc.

Turning to FIG. 2 the thermoregulator 42 is a conventional contact thermometer comprising two sections, an upper section 43 for setting the device to the desired temperature, and a lower section 44 for reading the actual temperature. Each section has its own temperature scale, indicated at 45 and 46, both being the same. In upper section 43 there is disposed an adjustable rotatable screw-threaded electrode 47 having a long thin wire 48 attached to its lower end which extends into lower section 44. A marker 49 is attached to electrode 47 and is visible in upper section 43; it indicates the temperature on the upper scale 45 at the same time that the lower end of the wire 48 indicates the same temperature on the lower scale 46. Electrode 47 is rotated by turning knob 50 at the top of the device. The lower end 51 of the device comprises a bulb of mercury (preferably mercury-thallium) and is disposable in the bath liquid 13. When the liquid warms, the mercury expands and the mercury column 52 reaches the set temperature, thereby making contact between the mercury and the end 53 of the wire 48, and this closes a circuit. Thus, the device 42 acts as a switch.

The electric circuits which are energized by the thermoregulator, and which control the operation of the solenoid valve 30, may be described briefly.

Consider that the bath liquid 13 has become heated to a temperature above the desired level, and that as a result the mercury column 52 expands, making contact at 53 with the lower end of the wire 48. This closes a circuit in the thermoregulator, which then operates the switching system generally designated as 43, note FIG. 1, to which the leads 58 and 59 are connected. In turn, a circuit is established, designated 58–59, note FIG. 2, involving lead 59, resistance 60 (47,000 ohms), resistance

61 (1 megohm), lead 62, secondary coil 63 (6.3 volts) of isolation transformer 64, diode 65, and lead 58. At point 66 of capacitor 67 (5 mfd.), the potential is —10 volts, or any suitable value in the range of —4 to —20 volts. This follows from the fact that secondary coil 63 of transformer 64 supplies alternating current which is rectified by diode 65 so that capacitor 67 is charged at point 66 by a negative potential, suitably about —10 volts relative to the point 66*a*. The coil 63 also supplies current to the heater 69 of thyratron 71 (2D21). It is the function of circuit 58–59 to transfer the potential at 66 to the point 68 through the current limiting resistor 60. The latter limits the current through thermoregulator 42 to values preferably below 1 ma.

Point 68 is at the control grid 70 of the thyratron 71, and the latter will not conduct when the control grid is at a potential of —10 volts, and therefore no current will flow in coil 72 of solenoid 73 in the relay 74, and consequently movable switch 75 will be in contact with point 76. A circuit, designated 75–91, and involving switch 75 and point 76 should not be considered. It comprises switch 75, point 76, lead 77, contact C of terminal strip 79, lead 80, the coil of solenoid valve 30, lead 81, contact 2′ of terminal strip 79, lead 83, contact 2 of strip 79, power source 85 (115 volts, 60 cycles) when switch 86 is closed, lead 87, contact 1 of the strip, leads 89, 90, and 91, and back to switch 75. Establishment of the foregoing circuit, by closing switch 86, energizes the coil in solenoid valve 30, and the plunger 92 is thereby raised to open the valve 93 and thus permit coolant to flow in pipe 26 in the direction of arrow 94, note FIG. 1. The flow of coolant acts to cool the bath 13 so that its temperature decreases.

Cooling of the bath, in time, results in opening circuit 58–59 at the point 53 owing to contraction of the mercury, note FIG. 2. The negative voltage of —10 volts is thus no longer imposed at point 68; in turn the capacitor 100 (0.1 mfd.) begins to discharge through the side 101 and resistor 61, and the voltage at point 68 is raised to a valve of about —2 volts, or even higher, going to 0 volt. When point 68 is at —2 volts, thyratron 71 will conduct current when its plate 102 is positive with respect to cathode 103. This current energizes coil 72 of solenoid 73, and as a result switch 75 is drawn away from point 76, thus opening circuit 75–91 at the latter point. (Switch 75, during the foregoing action, will make contact at point 104, but this is of no concern to the invention.)

As a result of the opening of circuit 75–91, solenoid 30 is no longer energized, and therefore plunger 92 is moved by action of spring 105 to close the valve 93 so that coolant flow through pipe 26 is stopped. At this point of the operation, bath 13 begins to warm up.

In connection with the plate 102 of the thyratron, this is periodically made positive by the alternating voltage supplied by secondary coil 110 (117 volts). In other words, the alternating voltage is positive on every other half cycle but negative on each following half cycle. The thyratron does not conduct when the plate is at a negative potential. In order to keep coil 72 of solenoid 73 energized during the time the plate 102 is negative, reliance is placed on the inductive effect of this coil, which acts to keep current flowing through it, and through the diode 111, during the half cycle when the plate is negative.

The terminal strip 79 represents a convenient means of connecting together the thermoregulator, power supply, solenoid valve, and, to the right of the strip, the relay circuits. The thermoregulator is connected in at the contacts T and T′; the power supply is connected at contacts 1 and 2; and the solenoid valve is connected at contacts C, 2′, and O. The pair of contacts at C and 2′, or at 2′ and O, is used depending upon whether the solenoid valve is to be normally open or normally closed. As shown, the power supply is grounded through line 112, to which line 59 is also connected.

The invention may be illustrated by the following examples.

Example 1

A cooling device was constructed subtantially in the manner illustrated in FIG. 1, with 1-gallon plastic foam-insulated vessels serving as bath container and Dry Ice reservoir. A 7-inch long heat exchange tube (corresponding to 26 in FIG. 1) was fabricated from ¼-inch O.D. copper tubing, as were the conduits corresponding to 23 and 27 in FIG. 1. A solenoid valve was placed in the upper return conduit and flow regulation was accomplished by inserting a small rubber stopper containing a small orifice in the discharge end of the upper conduit. This entire assembly, consisting of the two vessels connected by ¼-inch O.D. copper tubing and the solenoid valve, was placed in an insulated tank with inside dimensions of 17½″ L x 8½″ W x 11″ D and outside dimensions of 19″ L x 10″ W x 12″ D. A small screen was placed over the lower inlet tube in the Dry Ice reservoir. A thermoregulator was placed in the bath container and, through an electronic relay, operated the solenoid valve. The regulator, relay, and valve were exactly as shown in FIG. 2. The bath container was filled with methanol, and Dry Ice was placed in the other vessel. Methanol was poured over the Dry Ice. The thermoregulator was set for a temperature considerably below that of the methanol in the bath container so that the solenoid valve remained open. Within two minutes after addition of methanol to the Dry Ice, cold methanol-Dry Ice solution began circulating through the conduit and the bath liquid began to cool. Some typical cooling rate data obtained for this system are:

Time, p.m.:	Temp., deg. F.
3:35	44
3:26	56
3:35	44
3:43	32
Time, a.m.	
9:51	63
10:01	53
10:05	47
10:17	30
10:43	2

Additional cooling rate data were obtained by measuring the temperature drop over a short time interval with a stop watch. The rate at —38 degrees F. was approximately 0.5 degrees F./min., and at —65 degrees F. it was 0.15 degrees F./min. By placing in the discharge line corresponding to line 27 of FIG. 1 a small stopper having an orifice of about ⅓₂ inch diameter, bath temperature could be controlled to plus-or-minus 0.02 degree F. at 0 degree F., —38 degrees F., and —65 degrees F. The stopper functions as the hand throttle valve 31 of FIG. 1 but does not, of course, have the flexibility of the latter.

Example 2

A bath identical to the construction shown in FIGS. 1 and 2 (except for the window in the bath compartment and the remote location of relay 43) was constructed. This unit had outside dimensions of 16″ L x 8″ W x 10½″ D, and the bath container and Dry Ice reservoir each measured 5¾″ L x 4¾″ W x 8½″ D. The heat exchange tube 26 was approximately 7″ long and was fabricated from ⁵⁄₁₆″ O.D. stainless steel tubing. The unit operated at 0 degree F. and at —65 degrees F. with a control better than plus-or-minus 0.02 degree F. The rate of cooling of the unit was of the order given in Example 1.

It may be seen that the invention makes available a bath of improved compactness and accessibility of parts. Control over the rate of cooling is provided without necessity for the presence of a heater, thus simplifying the control operation and conserving coolant. Undercooling is avoided, resulting in shortening of the time necessary to attain temperature equilibrium. The bath further incorporates the feature of self-regulation of the cooling step, whereby cooling is self ceasing through the action of carbon dioxide gas in quickly building up back pressure in tube section 26.

It is noteworthy that, with bath compactness achieved, resulting in decreased heat capacity in the bath, very good temperature control was attained.

Other noteworthy features include the wide variation that is possible in the coolant flow rate, i.e., a large flow rate is available for rapid precooling of the bath and a very small flow rate for precise temperature control. Furthermore, all of the action is carried out without a mechanical or other pump.

Liquid carbon dioxide may be used instead of Dry Ice. Additionally, liquid helium, liquid oxygen, liquid nitrogen, liquid argon, and in fact any other normally gaseous material which can be liquefied at a suitable low temperature, including the "Freons" (a group of halogenated hydrocarbons containing one or more fluorine atoms), may be used. Liquid carbon dioxide under pressure is suitable. These fluid colants may be used with or without a solvent.

Other temperature sensing devices may be substituted for the thermoregulator, such as a resistance thermometer, thermistor, gas thermometer, bi-metallic strip, etc., with appropriate circuitry, for controlling the constant temperature bath.

The invention is applicable to other systems besides a constant temperature bath. For example, if bath compartment 11 contains, say, air instead of liquid, then the device may constitute an environmental chamber suitable for maintaining sensitive substances like tissue specimens, bacterial cultures, etc. at a closely controlled low temperature. Frozen foods may similarly be maintained at a constant low temperature. Additionally, the size of the device may be increased as desired, even to that of a walk-in chamber. In these applications, the lid of course would be replaced by a non-apertured air-tight panel or wall. Or the container 11, having any suitable type of fluid therein, may be used for other purposes, including application in a freeze drying system and the like.

The device of the invention is sometimes termed a "bath," or a "constant temperature bath," or a "constant temperature environmental device."

It will be understood that the invention is capable of obvious variations without departing from its scope.

In the light of the foregoing description, the following is claimed.

1. A constant temperature device comprising a bath section for holding bath liquid, a reservoir section for holding coolant solution, and a central section intermediate said first two sections, said coolant solution being maintained out of direct contact with the bath liquid, switching means associated with said central section, said bath and said reservoir sections having the same floor level and substantialy the same liquid levels, temperature-sensitive means in said bath liquid, means for transferring coolant laterally from the reservoir section to the bath section for indirect heat exchange therewith and means for transferring the coolant laterally back to the reservoir section, said transfer means having a valve disposed therein responsive to said switching means for controlling coolant flow, said switching means being actuatable by said temperature-sensitive means to operate said valve, said valve being operative to open to permit coolant flow therethrough when the temperature-sensitive means signals a high bath liquid temperature and to close to shut off coolant flow when the temperature-sensitive means signals a low bath liquid temperature.

2. A constant temperature device comprising a bath section for holding bath fluid, a reservoir for holding fluid coolant, and a central section intermediate said first

two sections, electrical switching means supported by said central section, said bath and reservoir sections having the same floor level and substantially the same fluid levels, temperature-sensitive means in said bath section, means for transferring coolant laterally from the reservoir section to the bath section and back again, said transfer means comprising a lower inlet conduit and an upper outlet conduit both connected by an upwardly directed conduit, said upper conduit having electrically operable valve means disposed therein for controlling coolant flow and being located in said central section, said switching means being actuatable by said temperature-sensitive means to operate said valve means, and said upwardly directed conduit being disposed in said bath fluid.

3. A constant low temperature device comprising a container having a bath fluid which is to be maintained at a constant temperature below room temperature, a conduit of heat-conducting material extending into the container in heat exchange relation with said fluid, a reservoir of a low boiling carbon dioxide solvent adjacent the conduit and laterally spaced from the container, ends of the conduit extending into said reservoir, a mass of solid carbon dioxide resting in the solvent and dissolving therein to form a cold solution, said solution being adapted to flow from the reservoir into the conduit and thereby cool the bath fluid, the heat of the bath fluid acting to boil out dissolved carbon dioxide from the solution, said conduit conducting the resulting carbon dioxide gas to said reservoir, said conduit in turn being refillable by cold solution from the reservoir, means in the conduit to reduce the flow in the conduit, said means being responsive to the temperature of said bath fluid, and a second means in the conduit manually operative to reduce the flow in the conduit.

4. The device of claim 3 wherein one of said conduit ends is disposed beneath the level of solution in said reservoir and the other of said ends is above said level.

5. A constant temperature device comprising a bath section for holding bath fluid, a reservoir section for holding coolant, temperature-sensitive means in the bath section, coolant conduit means extending from the reservoir section into and through the bath section and back to the reservoir section and comprising (1) an inlet portion to receive coolant from the reservoir, (2) a heat transfer portion disposed generally vertically in the bath section, and (3) an outlet portion for returning coolant to the reservoir section, and flow control means in said outlet portion responsive to said temperature-sensitive means for controlling the flow of coolant therein, said coolant comprising Dry Ice and a solvent therefor, the solvent dissolving the Dry Ice to form a cold saturated solution which functions as said coolant, said solution flowing from said reservoir section into said conduit means and acting to cool said bath fluid by indirect heat exchange through said heat transfer portion, said solution thereby becoming sufficiently warmed to boil out carbon dioxide gas, said heat transfer portion and outlet portion conducting said gas to the reservoir section and thereby providing for the flow of additional solution into said inlet portion from said reservoir section, and said flow control means, in response to said temperature-sensitive means when the bath fluid is cooled, acting to close off the flow of gas in said outlet portion.

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