

CONSTANT TEMPERATURE APPARATUS FOR LABORATORY USE THESIS FOR THE DEGREE OF M. S. C. Derwood Tuttle 1931

Thermostat Title (Wagenvoord & Co.)

Chemistry

## Introduction

In scientific research work one encounters many variables before accurate conclusions can be drawn. One of the most important and most common of these variables is temperature which must be controlled. Therefore, constant temperature apparatus is a necessity in any scientific laboratory. As a result of experimentation a constant temperature apparatus has been constructed which is simple to build and to maintain, and is sufficiently accurate for nearly all scientific work.

The complete assembly of one set of constant temperature apparatus is known as a thermostat. Thermostats may be classified as to the medium which is to be kept at constant temperature, as to the type of thermoregulator, or as to the type of relay used in its construction. A complete classification of themmostats for use in the laboratory within the temperature limits of about 0° to 100°C follows:

# Classification of Thermostats

I <u>Media</u> Thermostats may be classified as to the medium which is to be kept at constant temperature.

These media are usually gases or liquids at room temperatures.

<u>Gases</u>: The only important gas used as a medium to be kept at a constant temperature is air.
 W. M. Clark's book on "The Determination of Hydrogen Ions," gives a very good description of an air thermostat.

2. <u>Liquid</u>: Any liquid that is not too corrosive or does not evaporate too readily may be used as the medium to be kept at constant temperature. The liquid selected depends somewhat upon the temperature at which one wishes to work. Thus the most common ones are water and oil thermostats.

(a) Water

(b) 0il

II <u>Thermoregulator</u>: Thermostats may be classified as to the thermoregulator used in controlling the bath. Thermoregulators may be classified as those which control the gas supply of a gas heating unit or as those which control the electric circuits of an electric heating unit.

1. <u>Thermoregulators for the control of the gas</u> supply to a gas heating unit.

(a) Mercury expansion inside of a glass bulb which controls a by-pass gas orifice.

(b) The expansion of toluene against mercury contained in a glass bulb which in turn controls a by-pass gas orifice.

2. <u>Thermoregulators for the control of an electric</u> <u>current which supplies the electrical heating units</u>. These thermoregulators can be divided into two classes. Those that operate directly in the heating unit circuit and those that operate a relay which in turn opens and closes the heating circuit.

(A) Thermoregulators that make a direct contact in series with the electric heating units.

1. Bimetallic expansion which causes two points to make or break.

2. Mercury expansion inside of a glass bulb which makes and breaks electric contacts.

3. The expansion of toluene against mercury which in turn makes and breaks electric contacts. The containing vessel is made of glass.

4. Differential vapor pressure relay. This relay depends for its actuation upon the difference in coefficient of expansion of air and air saturated with some organic solvent such as ether. The difference in expansion causes mercury to flow from one side of a balanced arrangement to the other, thus operating a make

and break circuit. The tension on a spring fastened on one side of the balanced arrangement determines the temperature at which the points make and break.

(B) Thermoregulators that make and break the exciting circuit of a relay which in turn opens and closes the heating circuit.

1. Bimetallic expansion which causes two points to make and break.

2. Mercury expansion inside of a glass bulb which makes and breaks electric contact.

3. The expansion of toluene against mercury which in turn makes and breaks electric contacts. The containing vessel is made of glass.

4. Mercury expansion inside of a metallic container which makes and breaks electric contacts.

5. The expansion of a gas against mercury in a glass container which causes the circuit to be made or broken.

6. The same as (5), but contained in a metallic vessel. This metal container is often a jacket like container, the inside of which is the constant temperature medium chamber while the inclosed jacket part becomes the thermoregulator expansion chamber.

7. The resistance thermometer can be used as a thermoregulator by using the proper system of photo-electric cells, vacuum tubes, and relays.

8. Many of the above thermoregulators can be equipped with special arrangements for quick changes from one constant temperature to another.

9. In any of the above thermoregulators the space where the circuit is made and broken is often filled with a reducing or inert gas. This eliminates the oxidation of the contact surfaces.

III <u>Relays</u> Thermostats may be classified according to the type or system of relays used in its construction. The first form would be thermostats using a single relay through which the sensitive thermoregulator works to turn the heating current off and on. The second type would be a thermostat using a very low current low voltage relay responding to the thermoregulator and this relay actuating the current in a second relay of higher current higher voltage type of relay to turn the heating circuit off and on.

1. Single stage relays:

(a) Alternating current relays. These relays have not been used very successfully in accurate low temperature control. They are used mostly in large air thermostats and both large and small electric furnace installations. There are many many designs of these relays.

(b) Direct current relays.

(1.) Simple low voltage low current direct electromagnetic type of relay. Example: A telegraph relay of

50 ohms resistance or better. This relay is suitable for small thermostats, that is, one in which the current required is not more than 125 watts at 110 volts. Ordinarily 125 watts is more than enough for laboratory thermostats.

(2) Polarized low voltage low current type. Example: A polarized telegraph relay of 50 ohms resistance or better has been used on a bath 11 x 11 x 16 inches continuously for four or five months at a time without any adjustments being necessary. Ordinary current carrying capacity on the points is 125 watts at 110 volts. Silver contact points increase this value.

(c) Mercury in an atmosphere of hydrogen is often arranged so that a make or break in an electric circuit can be produced between two pools of mercury by a tipping action produced by either A. C. or D. C. current.

2. <u>Multiple stage relays</u>: The following list is self explanatory.

(a) A sensitive low voltage D. C. relay operating either a higher voltage A. C. or D. C. relay.

(b) Vacuum tube amplifiers operating either an A. C. or D. C. relay.

(c) Photo-electric cells. This is used in conjunction with the resistance thermometer of (II part B thermoregulator 7). The photo-electric cell is excited by the light from the resistance thermometer's galvanometer mirror.

This impluse is amplified by vacuum tubes to such a point that the out put current can operate a relay.

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(d) Thyratron tubes can be used as a relay on A. C. current. The Grid Glow tubes can be used as a relay on either A. C. or D. C. currents.

# Apparatus

The following list of apparatus is necessary in order to set up a simple, reliable, and accurate thermostat.

1. A vessel of some sort in which to build the thermostat.

2. A good agitator and a source of power with which to drive it.

3. A good thermoregulator.

4. An ordinary polarized relay of high sensitivity and 50 ohms resistance or better. Two dry-cells are required to operate the relay.

5. Such auxiliary laboratory equipment as is necessary to mount the various pieces of apparatus.

The tanks used for the thermostats have been of two kinds. The first were large glass jars  $ll \ge ll \ge l6$  inches. The other form consisted of a metal tank of the required size jacketed with a wooden box about three quarters of an inch in thickness. The agitators used were of two types. The first was a disc of heavy sheet copper or aluminum with a hole in the center for the driving shaft and 4 or 6 radii cuts in it which were turned up in propeller fashion. The second was merely a small propeller of wood or metal. Both types of agitator blades were driven by a special shaft which ran through a bicycle front hub and was driven by a washing machine motor. A wooden *L* on which the motor was mounted carried an arm which held the bicycle hub. A belt from the motor to a large wooden pulley on the top of the agitator shaft reduced the speed of rotation and brought the power to the shaft.

In general two types of thermoregulators were used. The best one for long continuous use was a commercial product of the glass container mercury, hydrogen filled type. The chief objection to this regulator is that it is difficult to set at a definite temperature. However, once set it stays fixed. The other type was one made in the laboratory. It was a glass mercury expansion type. A large thin walled pyrex test tube (75 to 100 cc.) was used as the base in constructing this regulator. A pyrex stop-cock was sealed on to the top of the test tube and a glass tube, containing a capillary in the middle of it, was sealed to the side of the test tube near the top. This tube was bent up parallel to the stop-cock tube in such a manner that the top of the

capillary would be on a level with the stop-cock. A platinum or tungsten wire sealed through the side of the test tube made one contact with the mercury and the other contact was made through the top of the side tube with a nut and screw arrangement fastened to its top with a cement. Nichrom wire soldered to the screw of this arrangement makes a very fine contact with the mercury in the capillary tube. <sup>Th</sup>e mercury and the capillary tube should at all times be very clean in order to work at its best. One degree centigrade rise should cause at least 1 cm. rise in the capillary of the side tube.

This thermoregulator operates an ordinary polarised relay of 50 chms resistance which was designed for this kind of work and therefore contains the necessary condensers. It has two sets of contact points of silver. If a polarized telegraph relay is used it is necessary to use small condensers across the contact points and the thermoregulator. These condensers can be easily made by any one. All that is necessary is two sheets of tin foil about 2.5" x 12" rolled tightly between three sheets of writing paper. Wires to make contact with the two foils must be included. The whole thing is coated with paraffin to keep out moisture. The polarized telegraph relay is the cheapest and is the best because its contact points are mounted directly on the relay's armature and the frame of

the relay while those built for this purpose have two sets of contacts, two are mounted on the relay armature through a piece of spring metal and the other two to the frame of the relay. The experience of this laboratory has shown us that two sets of relay points will not wear uniformily and when once out of adjustment the relay requires a general overhaul which is time consuming; while in the telegraph relay most all of the adjustments can be made with thumb screws. If two sets of points are required, use two polarized telegraph relays in parallel. Other types of sensitive relays work very well on small baths which do not draw in excess of 100 watts at 110 volts heating capacity through the relay.

> The Method of Procedure Used In Assembling Constant Temperature Apparatus

The following procedure was adopted as the result of setting up ten constant temperature baths and running them continuously for three months or more. One bath was taken apart and set up many times and as a result of this experiment this procedure was found to be necessary in order to insure reliable and accurate working thermostats.

1. Select the vessel to contain the constant temperature medium.

2. Select the constant temperature medium and place it in the vessel.

3. Assemble the agitating apparatus. The agitator should be placed in the center of the bath and near the bottom. Drive or set the blades of the agitator in such a direction that it will push the constant temperature medium to the bottom of the vessel where it will obtain a higher velocity and spread out from the center to the sides of the vessel and up the sides and down in the center of the bath. Agitation should be as vigorous as possible and not whip air into the media if it is a liquid.

4. After the relay has been properly adjusted mount it in a suitable place. The relay should be mounted in such a manner as not to be getting too much vibration or jars. This is not so important with polarized relays. The adjustment of relays depends upon the type of relay and therefore must be left to the ingenuity of the worker. Polarized relays should always be operated with the adjustable pole faces as close to the armature as possible and still have positive action. If this is not watched closely the magnet will lose its charge. If for any reason the pole faces have to be opened widely a magnetic

keeper must be used across the end of the magnet.

5. Set the thermoregulator for the desired temperature.

6. After the thermoregulator has been set for the desired temperature mount it (temporary) in the bath. It is necessary that the thermoregulator be as free as possible from vibrations.

7. The determination of the capacity of the heating units is a most important factor in setting up a thermostat. If room temperature humidity, and circulation are constant factors it is obvious that the bath will have a definite cooling rate over any definite temperature range. Theoretically, the ideal conditions within a thermostat would be such that the capacity of the heating units would cause the rate of temperature rise to be equal to the rate of temperature lowering. When the above conditions are fulfilled the heating units will be heating the same length of time as it takes for the cooling in order to maintain a constant temperature. This condition can be determined experimentally as follows:

(a) Put in a heating unit of any size and start the thermostat. It is best to have the heating unit for these measurements much too large.

(b) Make sure that each high and low temperature readings (using a Beckmann thermometer) are the same for several complete cycles of heating and cooling.

(c) With two stop watches take readings of the time off and the time on for the heating units. Ordinary light bulbs make good heating units.

(d) From this data and the wattage being consumed by the heater the proper wattage can be computed.

Examples:

The following examples were readings and computations made on the same bath in under as near as possible the same conditions.

> Room temperature 73.5°F. Thermostat -- about 25° C. Heating current 240 watts Mean of several "time on" 1.087 min. 11 Ħ "time off" 6.06 min. .12° C. Temp. interval  $240 \times 1.087 = 43$  watts 6.07

Heating current 120 watts Mean of several "time on" 1.975 min. " " " " "time off" 7.885 min. Temp. interval .11°C 120 x <u>1.975</u> = 30 watts 7.885

Heating current 80 watts Mean of several "time on" .483 min. " " " " time off" 1.2446 min Temp. interval .005° C. 80 x .483 = 31 watts 1.2446

Heating current 40 watts Mean of several "time on" .7615 min. " " " "time off" .777 min. Temp. interval .0012° C. 40 x .7615 = 39 watts

<u>Note</u>\* - The smallest division on the scale of the wattmeter used was equal to 40 watts. This will account for most of the variation in results.

These results indicate that a 40 watt heater should be used. However a 50 or possibly a 75 watt heater is necessary in order to be sure that the heater will take care of peak loads due to varing conditions in the room. <u>The important thing to be remembered is that it is</u> <u>necessary to have enough heat for all conditions of the</u> room, but never too much heating capacity.

8. Install the proper heating unit.

9. Move the thermoregulator mounting from the free from vibration mounting to one where there is vibration.

Usually there is sufficient vibration in the motor or agitator supports for this purpose. So all that is necessary to do is to clamp the thermoregulator to the motor or agitator support. The purpose of vibrating the thermoregulator is to decrease the length of period for heating and cooling. Vibration does this without disturbing the ratio of heating and cooling.

#### Results

Out of many thermostats set up in 11 x 11 x 16 inch glass jars not one varied more than .03°C over rather long periods of time. Over short periods of time, one or two days, the best ones did not vary over .005°C. Three of these baths maintained a temperature so constant that over periods of 4 hrs. no change of temperature could be noticed on a good Beckmann thermometer through a sighting arrangement to eliminate parallax and a good reading glass. All thermostats had a tendency to drift. This is most notionable with newly set up apparatus. As a result of the experiments on the above thermostats the following thermostat was designed and built. The vessel in which the thermostat was built is as follows. A box 24" x 24" and 16" deep (outside dimensions) was built of galvanized iron.

This was jacketed with a 24" x 24" and 20" deep (inside measurements) box made out of 3/4" lumber. A wood strip 4" wide was fastened with screws across the middle of the top. On each side of this strip was fitted a cover. This center piece served as a support on which to mount the apparatus. The agitator was cut out of heavy sheet copper as described above and mounted in the center of this strip through a bicycle hub as its bearing. A washing machine motor provided the power through a leather belt. A cooler plate of sheet copper 4  $1/2^n \ge 10^n$  and  $1/4^n$  thick was mounted just above the agitator and to one side of the center of this strip. The commercial glass container mercury hydrogen filled thermoregulator was mounted on the opposite side of the agitator. Oil was used as the constant temperature medium. Agitating the oil in this thermostat without any other form of heating or cooling would cause the temperature to rise. Thus a cooler is necessary in this bath. One should notice that this arrangement of heater and cooler will distribute the oil which comes in contact with them all through the bath as quickly as possible. This thermostat has maintained a temperature constant to within .013°C over a period of six months of continuous operation. At the end of this time the thermoregulator failed to work properly. At two different times it

was necessary to stop the bath for a half day or so for minor repairs. The second thermoregulator has been in the bath for a little over two months. The temperature was constant to within about .011°C. A variation of .007°C., for a short time, was observed for which the cause is unknown. Except for this the temperature control was within .005°C. These changes in temperature are slow and can only be obtained by making many readings, since one cannot as a rule see these changes by watching, a Beckmann thermometer as described above.

## Conclusion

By following the above outline any student of physical chemistry can build an inexpensive thermostat that will maintain a constant temperature to within .01°C., and with some additional care an apparatus may be built which will maintain a temperature which is even more constant, possibly within the limits + 0.002°C.

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