THE DETERMINATION OF THE DEUTERIUM OXIDE CONTENT OF VARIOUS SAMPLES OF WATER

By

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CHAPTER ONE

Statement of the Problem

The purpose of this investigation was to determine the deuterium oxide content of various samples of water by a precise and sensitive method based upon the compressibility of water. These samples were gathered from as many different locations as were deemed necessary.

CHAPTER TWO

Theory

It is a well known fact that a given weight of water will occupy one volume at a certain temperature and pressure but a different volume if either the temperature or the pressure is changed. If the temperature is raised, the volume occupied by the water is made greater. If the pressure is increased, the volume is decreased. This set of conditions form the basis for this thesis.

Since it is a fairly simple thing to assemble a thermostat which will control the temperature to within one one-thousandth of one degree Centigrade and also fairly simple to accurately measure the pressure exerted on the sample, this was done in preference to using any other methods.

By purifying all of the samples to practically the same degree of purity and determining the pressure necessary to keep the float constant at a selected temperature, one is able to calculate the differences in the densities of the various samples of water.

By thus finding these differences in the densities of the samples and assuming that they were due to the varying concentrations of deuterium oxide in the waters, one can calculate the concentration of the deuterium oxide in mol fractions from the equation $\Delta s = 0.1079\ x$,[1] in which Δs is the difference in density and x is the concentration, in mol fractions, of the deuterium oxide.

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[1] Daniels, Matthews and Williams, Experimental Physical Chemistry, Second Edition, McGraw-Hill Book Company, 267

CHAPTER THREE

Apparatus and Procedure

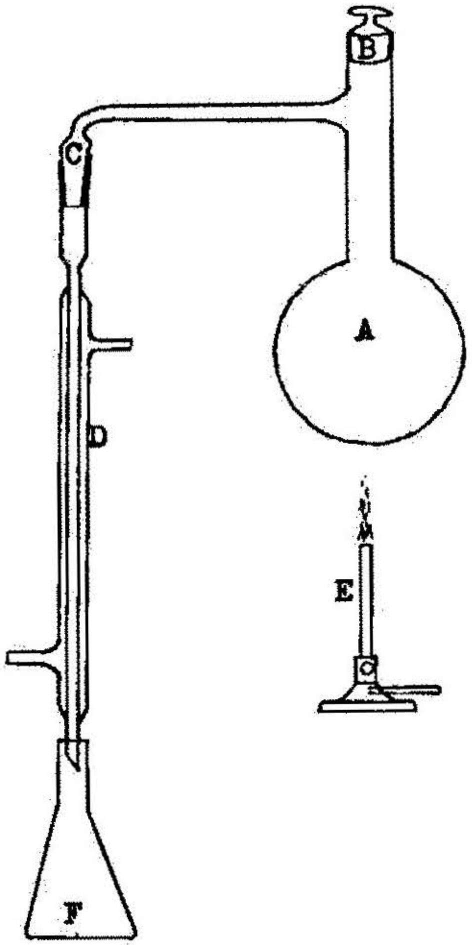
The most important procedure that had to be gone through was that of getting the samples of water purified and in the proper condition for accurate measurements to be taken. For this purification three types of stills were used. All of these stills were constructed entirely of pyrex glass and pyrex ground glass stoppers. Pyrex ground glass joints were used throughout.

The first still (see page 6) was just an ordinary still made by myself. All of the samples were run through this still twice. The first run was just a crude distillation to remove any salts that were dissolved in the water. The second time, some $KHSO_4$ was added as a purifying agent and the water redistilled. From this distillation was collected only the middle portion of the distillate.

The second still (see page 7) was also an ordinary still. $KMnO_4$ was used as a purifying agent in this still. Here, as before, both the first and last portions of the distillate were rejected.

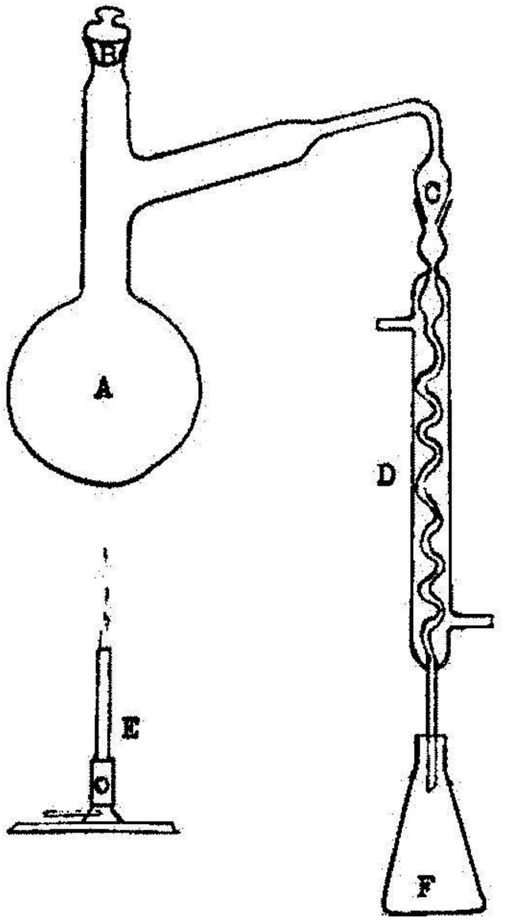
The third still (see page 8) was a very uncommon still. In fact, it does not even resemble a still for the purification of water. However, it is a very efficient piece of apparatus. (As a matter of interest, the conductivity of all of the samples was measured after being run through this still and in all cases was found to be very close to the value 3.8×10^{-6} mhos.) It is far faster than either of the other stills employed and the water, after passing through this still, is purified far more than the other stills could possibly do. This unusual still was the culmination of various original and unoriginal ideas taken from different people and places at odd moments and finally all amalgamated. The water to be purified was placed in flask A and heat was applied. Upon partial condensation of the vapor in trap C, the minute, entrained particles which had been carried over from A in the vapor, were carried down into the trap. The remainder of the vapor, which comprised the greater amount, then passed upwards through the Vigreux column. Through the action of the Vigreux column the vapor was partially fractionated and the higher boiling part condensed and run back into the trap. When this trap became about half full, it was drained by opening the stop-cock in the bottom. This water was then rejected. After passing through the Vigreux column the vapor was split into two parts. The lesser and more volatile part escaped through vent E at the top of the column. This constituted a very small portion of the total vapor. The remainder of the vapor then condensed and was collected in flask G. This was

The First Still



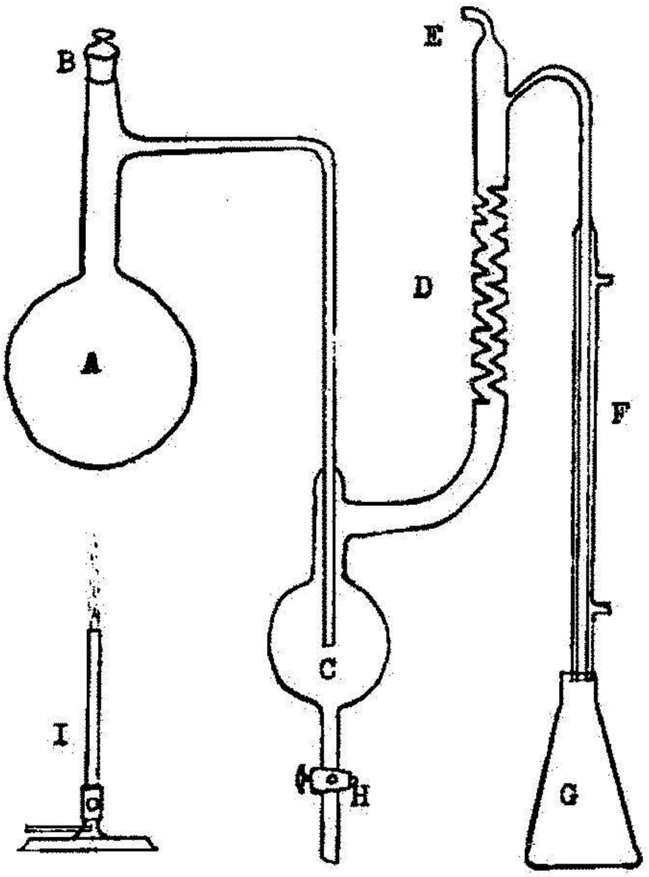
A	2 liter distilling flask
B	Ground glass stopper
C	Ground glass connection
D	Water cooled condenser
E	Bunsen burner
F	Collecting flask

The Second Still



A	2 liter distilling flask
B	Ground glass stopper
C	Ground glass connection
D	Water cooled spiral condenser
E	Bunsen burner
F	Collection flask

The Third Still



A	2 liter distilling flask
B	Ground glass stopper
C	Trap for non - volatile impurities
D	Vigreux fractionating column
E	Open vent for volatile impurities
F	High pressure water cooled condenser
G	Collecting flask
H	Stop cock drain for trap
I	Bunsen burner

the purified water.

The water, after having been distilled in the above manner in order to be as pure as possible, was then boiled under vacuum until the volume that had been taken for that purpose had been decreased by one-third. This step was to remove any dissolved air that might be present and also to make sure that there were no more traces of volatile impurities present. Immediately after this boiling the sample was placed in the sample bulb.

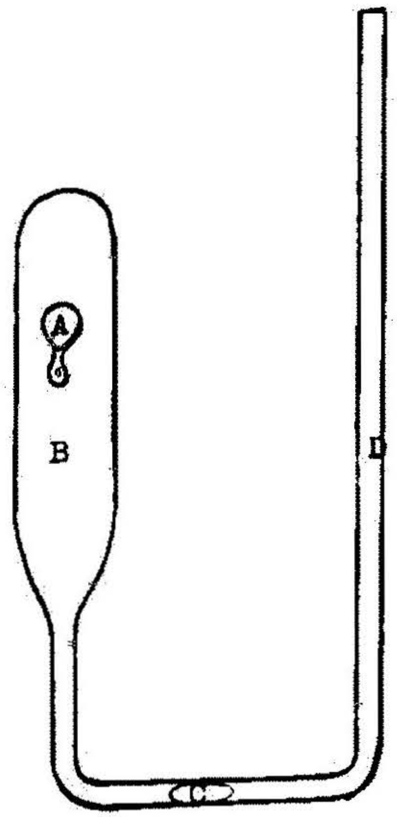
The sample bulb (see page 12) was made from a 16 mm pyrex glass test tube. Inside this tube was placed a pyrex glass float of about 0.05 cc in volume. Then a length of 7 mm pyrex glass tubing was sealed on to the open end of the tube and bent per figure.

In order to fill the sample bulb with the water sample to be used, the following technique was employed (see page 13). The sample was placed in a 200 cc distilling flask, the side arm of which was connected to a suction pump and the bulb of which was connected per figure. At the same time, a little heat was gently applied to the bulb and the suction was turned on. Upon the withdrawal of the heat from the bulb the water slowly began to creep up the tube into the bulb. The speed with which the bulb filled was accelerated by applying the heat to the water in the flask and also by slowly turning off the suction. Extreme care had to be taken during this filling so that the sample bulb would not break. After the bulb was filled, about 3 cc of pure mercury was placed in the tube to prevent contamination and also to keep oxygen from dissolving in the water sample.

In order to empty the sample bulb a slightly different technique was employed. (see page 14) The full bulb was connected to a 125 cc distilling flask per figure and the side arm of the flask led to a suction pump. The suction was then applied, heat gently applied to the sample bulb, and the water gently boiled out of the bulb.

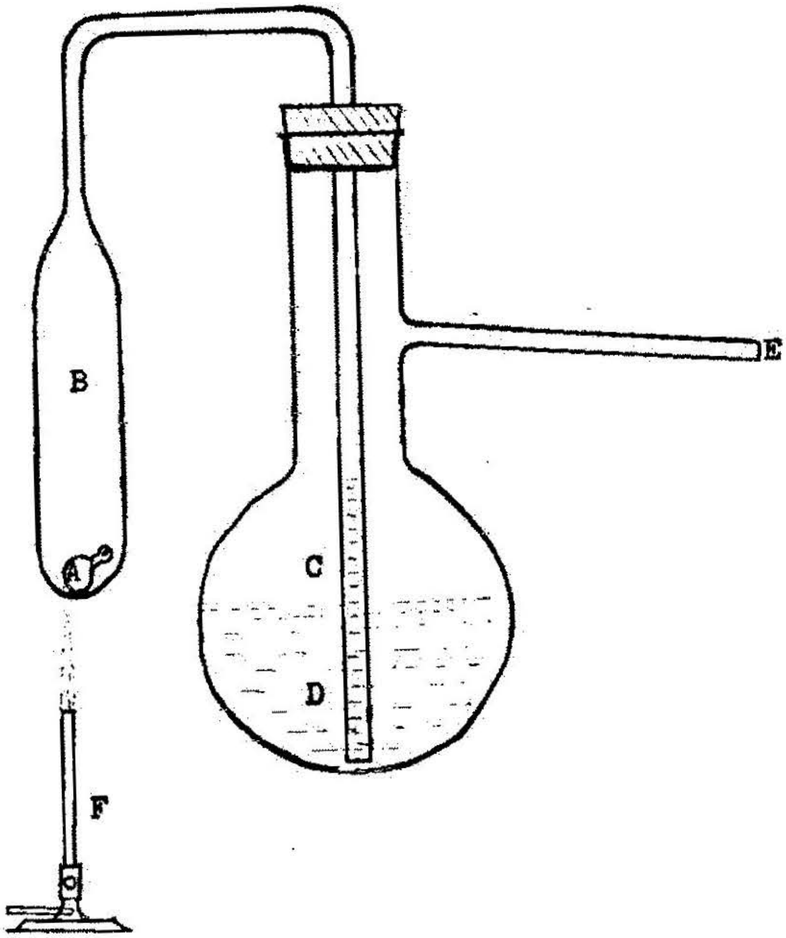
This sample bulb was then placed in a thermostat (see page 15) consisting of a one-gallon, wide mouth, unsilvered pyrex glass vacuum bottle made by the Thermos Bottle Company. This thermostat was filled with once distilled water. The reason for using distilled water was that the sides of the thermostat would not become encrusted with a layer of salt deposit as a result of evaporation and so make the taking of accurate readings difficult if not impossible. In this thermostat was a thermoregulator made by myself from 10 feet of 10 mm pyrex glass tubing. This thermoregulator was bent in the form of a horseshoe (from top view) and extended from the top to the bottom of the thermostat. The reason for this peculiar shape was that the thermoregulator would be almost completely surrounding the water sample and thus render more accurate temperature control. The thermoregulator was filled with Toluene and a mercury contact was made in the straight tube where the connections were placed. One of these connections was a platimum wire sealed into the tube where it was always in contact with the mercury. The other contact was a makeshift affair that was very successful. It was composed of an inch of platimum wire soldered to an old thermoregulator top. Copper wires were then soldered to both of these contacts and thence hooked up properly. This thermoregulator was very good for the purpose, but when it was connected to a relay, it was found that the make and break of the platinum-mercury contact caused a spark to pass, thus oxidizing the mercury and making necessary a constant changing of the regulator. To eliminate this source of error a condenser was made from two aluminum plates immersed in a concentrated solution of pure Castile soap. The condenser was then completely covered by placing it in a 250 cc beaker and pouring hot paraffin over it and allowing it to cool. Thus equipped, the thermoregulator maintained the temperature in the thermostat at a constant value of plus or minus less than one one-thousandth of one degree Centigrade.

For a heating element a 25 Watt, General Electric, Sample Bulb



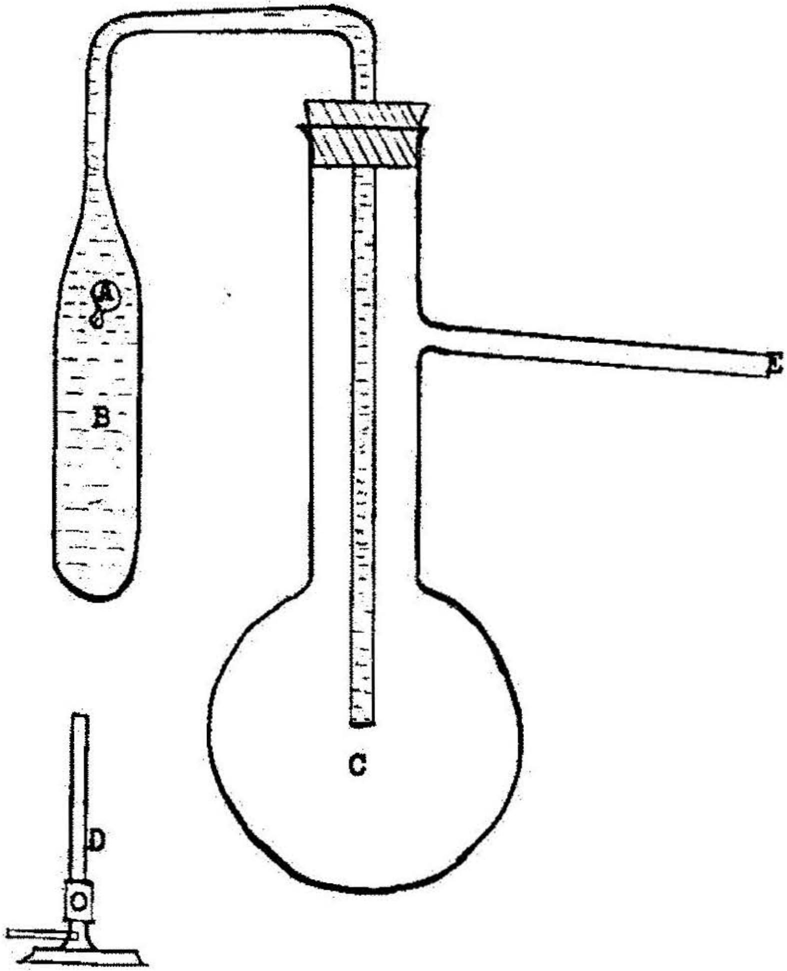
A	Pyrex glass float
B	Sample bulb for water
C	Mercury seal
D	Tube leading to pressure column

Apparatus for Filling the Sample Bulb



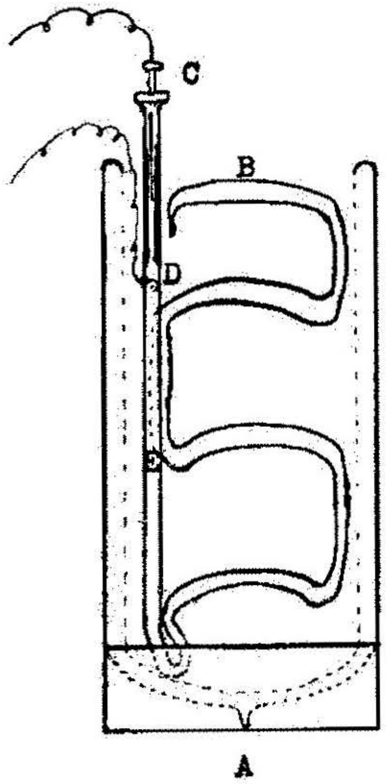
A	Pyrex glass float
B	Sample bulb for water
C	200 cc distilling flask
D	Water sample to be used
E	Side arm to vacuum pump
F	Bunsen burner

Apparatus for Emptying the Sample Bulb



A	Pyrex glass float
B	Sample bulb with water
C	125 cc distilling flask
D	Bunsen burner
E	Side arm to vacuum pump

Thermostat



A	Thermostat filled with water
B	Toluene filled part of thermoregulater
C	Upper Platinum - Mercury contact
D	Lower Platinum - Mercury contact
E	Mercury filled part of thermoregulator

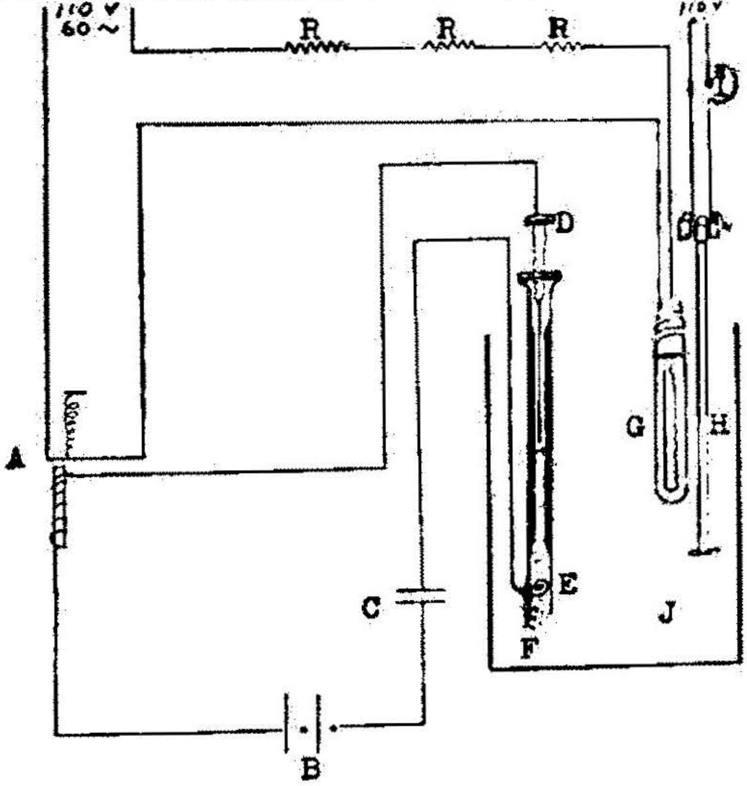
tubular, show case light globe was used. To regulate the heat given off by this globe a bank of lamps arranged in a series and ranging from 10 to 100 Watts in power was used. (see page 17)

An ordinary electrically driven stirrer with rheostat control was used to keep the water in the thermostat in constant agitation.

For the relay that was most satisfactory a CENCO super sensitive dry cell relay made by the Central Scientific Company was used. As its name implies it is operated on dry cells, two of the cells being used in this instance.

In order to exert pressure on the sample in the bulb an open air manometer half filled with mercury was made. Whenever pressure was to be exerted upon the sample, an oxygen tank was connected to the manometer and the pressure from it used to raise the mercury. This was one of the reasons leading to the placing of the mercury seal in the bend of the tube leading to the sample of water. The seal kept the oxygen from dissolving in the sample and thus leading to erroneous results. All of the connections in the part of the system were of pyrex glass and black rubber pressure tubing. All rubber-glass joints were wired together. The maximum pressure that could be attained

Electrical Diagram of Set Up



A	CENCO super sensitive relay
B	2 dry cells
C	Castile soap condenser
D	Upper Platinum - Mercury contact
E	Lower Platinum - Mercury contact
F	Mercury filled part of thermoregulator
G	Electric light globe heater
H	Electrically driven stirrer
I	Reostat
J	Thermostat
R	Light globe resistances

was 2,700 mm of mercury over atmospheric pressure.

In order to read the approximate temperature in the thermostat, an alcohol filled thermometer of range -50°C to -50°C was used.

In order to read the exact temperature, a Beckmann thermometer set at approximately 22.5° Centigrade was used. It was read with a magnifying eyepiece to facilitate the accurate reading of the temperature.

In order to determine the rest point of the float in the sample bulb a cathetometer was used.

The procedure for getting readings was as follows: The sample of water was placed in the sample bulb. The sample bulb was placed in the thermostat and connected to the manometer. The sample bulb was allowed to remain in the thermostat for eight hours before any attempt to obtain a reading was made. This was in order to make sure that the sample and the thermostat were at exactly the same temperature and also to make sure that the temperature of the sample was the same throughout. After thus making sure that the temperature was known exactly, pressure was applied until the float in the sample bulb rose to the top of the bulb. Then this pressure was slowly released until the float began to slowly sink towards the bottom of the bulb. From this point on the observations were made with the cathetometer. It is easy enough to observe the float when it rises or falls rapidly, but when it is in the middle of the bulb and is arriving somewhere near to the point at which it will neither rise nor sink in the water, it becomes very difficult to observe any movement at all during a period of less than ten minutes. If, at this point, the cathetometer is focused on the float, its slightest movement becomes very evident. When the float was at last brought to such a position, by means of regulating the pressure, that it neither rose nor sank, a reading was taken. The cathetometer was left focused upon the float for at least an hour, and if at the end of that time the float was still at the same point, the reading was then recorded. It was especially easy fo focus the cathetometer on the float and leave it for an hour and then come back to see if the float were still in the same position, because the cathetometer that was used had two horizontal cross hairs, one of which was movable. Therefore, when a reading was to be taken, the cross hairs were focused upon the tail end of the float and thus one was enabled to instantly detect a rise or fall of approximately 1 mm. Thus being able to stablilize the float with such a degree of accuracy, being able to control the temperature of the thermostat to within one one-thousandth of one degree Centigrade, and being able to read the pressure exerted upon the sample by the mercury column and by the atmosphere to within 1 mm, the results obtained should most certainly be reliable.

CHAPTER FOUR

Materials

The various samples of water used were obtained from the following sources:

Bad Water Pools in Death Valley
Salt Pools in Death Valley
The American Potash and Chemical Corporation
The Great Western Electro-Chemical Company
San Francisco Bay

When water evaporates over a long period of time, the concentration of deuterium oxide is increased. That is, the light water evaporates more readily than the heavy water. Therefore one would expect greater concentration of deuterium oxide in water subject to much evaporation than in water not so subjected.

Bad Water Pools in Death Valley; California:

This water was obtained in April, 1936. Owing to the extreme rate of evaporation one would expect that this water would be quite heavy as a result of the concentration of the deuterium oxide. However, there is evidently a fresh water supply leading into these pools because they are not very concentrated in dissolved salts as is the water from the Salt Pools.

Salt Pools in Death Valley, California:

This water was obtained in April, 1936. Here one would definitely expect to find the water heavier than that in Stockton because of the very rapid rate of evaporation. The large concentration of dissolved salts in the water indicates that more evaporation has taken place than in the Bad Water.

The American Potash and Chemical Corporation at Trona, California:

This water was obtained in April, 1935, from one of the Company's old boilers. Since they very rarely drain their boilers, this water is the residue left from the long distillation of large quantities of water. For this reason one would expect to find it heavier than the Stockton water.

The Great Western Electro-Chemical Company at Pittsburg, California:

There were two samples of water from this place. The first sample came from one of the electrolytic cells in which hydrogen and chlorine are constantly being liberated. Since this sample came from an old electrolytic cell, one would expect to find a heavier water than Stockton water. This concern uses the chlorine that they generate but has no use for the hydrogen. Therefore they burn it in air. The second sample is some of this synthetic water that they have thus made. One would expect to find it quite a bit lighter than the cell liquor.

San Francisco Bay near the San Mateo Bridge:

This water was obtained in April, 1935. One would expect it to be a little bit heavier than the Stockton water but not very much.

Stockton Water from College of the Pacific, Stockton, California:

This water comes from wells at a depth of 310 feet. One would not expect this water to be very heavy for there is very little, if any, evaporation taking place.

CHAPTER FIVE

Calculations

The different concentrations of deuterium oxide in the various samples were merely relative, for this method does not enable one to find the absolute density of a water sample but enables one to find the density of any sample of water relative to a standard sample. In this thesis the synthetic water from the Great Western Electro-Chemical Company was used as the standard. The reason for this is that it was found to be the lightest water of all that was used, or in other words, had the least amount of deuterium oxide present. The concentration of deuterium oxide is determined by calculation from the relative densities of the water samples measured by the method previously described (Chapter Three).

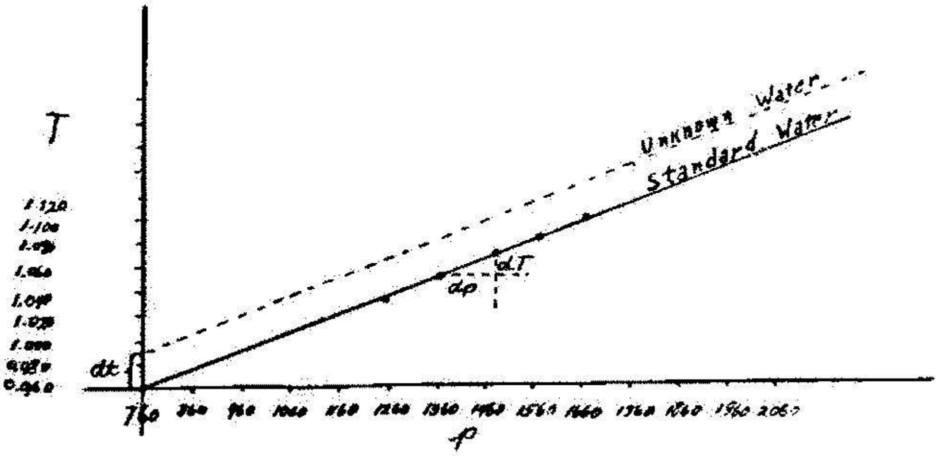
It is easy to convert a change in temperature into a change in density by direct substitution in a formula that will be derived later on in this paper. However, it is a slightly different matter to have to convert a change in pressure into a change in density. A pure sample of water was placed in the sample bulb and the temperature and pressure necessary to keep the float at one point in the middle of the bulb were recorded. Then the temperature was raised 0.02 degrees Centigrade. The pressure had to be increased also in order to keep the float in the same position. A series of readings were obtained (see Table I) and graphed (see below). This graph represents the change in pressure necessary to keep the density of the water the same as it was originally before the temperature was changed.

T = temperature on Beckmann thermometer.

dT = change in temperature.

dt = difference in temperature at which different samples were measured.

dp = change in pressure.



For all of the water samples the same type of graph was obtained. Since they were all straight lines of equal slope, the equation,

$$T = kp + b \qquad \text{(Type equation of a straight line)},$$

can be applied. From the experimental data k and b can be obtained. The slope of the line $(\frac{dT}{dp})$ is, of course, k. If the coordinates are so chosen that b = 0 for the standard water sample, then for any other sample

$$b = dt.$$

In the standard sample:

$$T = kp + 0$$

While in the unknown samples:

$$T = kp + dt$$

Differentiating with respect to T:

$$1 = k \cdot \frac{dp}{dT} + \frac{dt}{dT}$$

$$dT = k \cdot dp + dt$$

$$dt = dT - k \cdot dp$$

Then, the increase in volume (dV) of one cc of water when the temperature is increased dt degrees must be found. To find this, the cubical expansion equation for liquids[1] was employed. This equation and the corresponding values of α, β and γ for water are:

$$V = V_o\ (1 + \alpha t + \beta t^2 + \gamma t^3)$$

$$\alpha = -0.0643 \times 10^{-3}$$

$$\beta = +8.505 \times 10^{-6}$$

$$\gamma = +6.790 \times 10^{-8}$$

For water the equation then becomes:

$$V = V_o(1 - .0000643t + .000008505t^2 + .0000000679t^3)$$

Differentiating the general equation with respect to t:

$$dV = V_o(\alpha + 2\beta t + 3\gamma t^2)\ dt$$

---

[1] Handbook of Chemistry and Physics, 20th edition, pp. 1201, 1202, Chemical Rubber Company, 1935

If $V_o = 1$

$$dV = (\alpha + 2\beta t + 3\gamma t^2)\ dt$$

but

$$dt = dT - k \cdot dp$$

Substituting

$$dV = (\alpha + 2\beta t + 3\gamma t^2) \cdot (dT - k \cdot dp)$$

For $23.5^{\circ}C$ the equation then becomes, for water

$$dV = \left[(-.0000643) + (2)(.000008505)(23.5) + (3)(.0000000679)(23.5)^2\right] \cdot (dT - k \cdot dp)$$

Combining

$$dV = .000447928325\ (dT - k \cdot dp)$$

Since k is $\frac{dT}{dp}$, it was calculated from Table I, which was obtained by taking a pure sample of water and varying both the temperature and pressure and obtaining readings.

TABLE I

Trial	T	p	dT	dp	$\frac{dT}{dp}$ or k	Mean $\frac{dT}{dp}$ or k
1	1.040	1262.3				.000196295
			.020	92	.00021744	
2	1.060	1354.3				
			.020	112	.00017848	
3	1.080	1466.3				
			.020	108	.00018518	
4	1.100	1574.3				
			.020	98	.00020408	
5	1.120	1672.3				

Substituting for the k

$$dV = .000447928\ (dT - .000196295\ dp)$$

After thus obtaining the value for dV, the difference in the densities ($d\rho$) of each of the unknown water samples and the standard water sample can easily be calculated.

Let ρ_o be the density of the standard water sample. Since the results are only comparative with respect to this standard, the mass (M_o), the volume (V_o) and the density(ρ_o) can all be set equal to 1.

$$\rho_o = \frac{Mo}{V_o} = 1$$

Let ρ_1 be the density of the unknown water sample and M_1 and V_1 the mass and volume.

$$\rho_1 = \frac{M_1}{V_1}$$

Where V_1 is the volume of one gram of the unknown water sample

$$\rho_1 = \frac{1}{V1}$$

Because of taking as a standard the lightest water sample

$$dV = V_o - V_1$$

$$V_1 = V_o - dV$$

$$\rho_1 - \rho_o = d\rho$$

$$d\rho = \frac{1}{V_1} - 1$$

$$d\rho = \frac{1}{V_o - dV} - 1$$

$$d\rho = \frac{1 - V_o + dV}{V_o - dV}$$

But $V_o = 1$

$$d\rho = \frac{1 - 1 + dV}{1 - dV}$$

$$d\rho = \frac{dV}{1-dV}$$

But, if dV is very small

$$d\rho = dV$$

Therefore, for water at 23.5°C

$$d\rho = dV = .000447928(dT - .000196295\ dp)$$

After having gotten this far, it is a simple matter to convert the change in density ($d\rho$) into mol fractions of deuterium oxide (X) present in the sample from the equation:

$$d\rho = 0.1079\ X$$

$$X = \frac{d\rho}{0.1079}$$

Substituting:

$$X = \frac{.000447928(dT - .000196295\ dp)}{0.1079}$$

or

$$X = .004151421\ (\ dT - .000196295\ dp\ )$$

CHAPTER SIX

Experimental Results

The following data were obtained with the thermoregulator set at a fixed temperature and the pressure varied. As will be noted, the temperatures are not exactly the same in all of the tables, but this was due to a slight changing in the height of the mercury column in the regulator due to settling and vibrations. However, regardless of the reading on the Beckman thermometer, the temperature of the thermostat was always constant within less than one one-thousandth of one degree Centigrade.

In the following tables the temperatures are as read on a Beckman thermometer which was set at about 22.5° Centigrade. The pressures are in millimeters of mercury.

TABLE II

Synthetic Water (Great Western Electro Chemical Co.)

Sample	T	p	Mean T	Mean p
1	1.042	1384.0	1.042	1381.75
2	1.042	1379.0		
3	1.042	1384.0		
4	1.042	1380.0		

TABLE III

Cell Liquor (Great Western Electro Chemical Co.)

Sample	T	p	Mean T	Mean p	dT	dp
1	1.042	1139.0	1.0425	1135.25	+.0005	-246.5
2	1.044	1136.0				
3	1.042	1132.0				
4	1.042	1134.0				

TABLE IV

Stockton Water

Sample	T	p	Mean T	Mean p	dT	dp
1	1.042	1262.3	1.040	1260.3	-.002	-121.45
2	1.039	1247.3				
3	1.038	1264.8				
4	1.041	1266.8				

TABLE V

Bad Water (Death Valley)

Sample	T	p	Mean T	Mean p	dT	dp
1	1.042	1296.5	1.040	1285.625	-.002	-96.125
2	1.038	1276.5				
3	1.040	1285.0				
4	1.040	1284.5				

TABLE VI

Salt Pools (Death Valley)

Sample	T	p	Mean T	Mean p	dT	dp
1	1.042	1126.5	1.04175	1121.0	-.00025	-260.75
2	1.042	1121.5				
3	1.041	1119.5				
4	1.042	1114.5				

TABLE VII

Boiler Blow Down (Trona)

Sample	T	p	Mean T	Mean p	dT	dp
1	1.042	1154.5	1.041	1148.75	-.001	-233.0
2	1.042	1146.5				
3	1.040	1145.0				
4	1.040	1149.0				

TABLE VIII

Mother Liquor No. 2 (Trona)

Sample	T	p	Mean T	Mean p	dT	dp
1	1.040	1167.5	1.040	1171.0	-.002	-210.75
2	1.040	1169.5				
3	1.040	1174.5				
4	1.040	1172.5				

TABLE IX

Sea Water (San Francisco Bay)

Sample	T	p	Mean T	Mean p	dT	dp
1	1.040	1234.5	1.040	1235.0	-.002	-146.75
2	1.040	1235.5				

TABLE X

Mol Fractions of D_2O

Sample	dT	dp	X
Synthetic Water	0	0	0
Cell Liquor	+.0005	-246.5	2.0295×10^{-4}
Stockton	-.002	-121.45	9.0670×10^{-5}
Bad Water	-.002	- 96.125	7.0030×10^{-5}
Salt Pools	-.00025	-260.75	2.1145×10^{-4}
Boiler Blow Down	-.001	-233.0	1.8572×10^{-4}
Mother Liquor No. 2	-.002	-210.75	1.6345×10^{-4}
Sea Water	-.002	-146.75	1.1129×10^{-4}

TABLE XI

Mol Fractions of D_2O in Increasing Order

Sample	
Synthetic Water	0.000
Bad Water	7.0030×10^{-5}
Stockton Water	9.0670×10^{-5}
Sea Water	1.1129×10^{-4}
Mother Liquor No. 2	1.6345×10^{-4}
Boiler Blow Down	1.8572×10^{-4}
Cell Liquor	2.0295×10^{-4}
Salt Pools	2.1145×10^{-4}

In all of the calculations figures beyond the sixth decimal place were discarded, because the readings were not accurate beyond that place. The temperature readings were only accurate to four decimal places. The pressure readings were only accurate to three decimal places.

CHAPTER SEVEN

Discussion of the Results and Conclusion

The results agree favorably with the theoretical results one would expect to obtain as discussed in Chapter Four with the exception of the Bad Water sample. One would think that the Bad Water sample would be almost as heavy as the water from the Salt Pools, but such is not the case. The peculiar location of this source of water in Death Valley has unquestionably something to do with these peculiar results. There was a very small amount of dissolved material in the Bad Water samples, but there were approximately 370 grams of dissolved material per liter in the samples from the Salt Pools. This tends to show that there is either a lack of evaporation or else a good, steady supply of fresh running water somewhere underground.

E. S. Gilfillian, Jr.[1] has published an article on the isotopic composition of sea water in which he uses a method similar to that used in this thesis. It does not seem that his results can be as accurate as those obtained by this method, for in the thermostat that was used in his method the temperature control was by means of a water-ice equi-

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[1] The Journal of the American Chemical Society, 1934, Volume 56, Page 406

librium. In my opinion, this would not be constant to within one one-thousandth of one degree Centigrade, and I do not believe that good results can be obtained unless this temperature control is effected. Also, I do not believe that the method of employing test tubes open to the air as a container for the water sample is accurate. In this thesis the water samples were sealed from the air as much as was possible, and a mercury seal was placed in the bulb to keep the sample pure.

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