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HEAT TRANSFER INTO OPEN METAL THERMORESISTOMETER CUPS[a]

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During the initial testing of the thermoresistometer developed *(6)* in this laboratory for the study of the thermal resistance of bacterial spores it was observed that samples of 0.01 ml. magnitude did not spread themselves uniformly across the bottom of the cup. Instead, a droplet with a depth of more than four times that of a uniform layer was formed either in the center or at the edge of the cup. When larger samples that would cover the bottom of the cup were used, a meniscus was formed at the edge of the cup giving a much greater depth at this point than would be obtained if the sample were spread uniformly across the cup. It was also observed that when sample volumes as large as 0.10 ml. were used the results were not always the same as with smaller samples. Since at high temperatures the time intervals for a series of tests might be in the order of 0.010, 0.015, 0.020, 0.025, 0.030, 0.035 and 0.040 min., a small lag correction factor resulting from increased sample depth could produce erroneous results.

Sognefest and Benjamin *(8)* studied the heating lags in thermal death-time cans and tubes to arrive at accurate correction factors. Stumbo *(9)* calculated the heating rates of samples in his thermoresistometer cup and concluded that since the food samples were within 0.3°F. of the steam temperature for at least 9 seconds (0.15 min.) of a 10 second (0.167 min.) process and for a greater percentage of the time for all longer processes, no correction was necessary. The present study was made to determine the accuracy of exposures as short as 0.005 min. The four phases of the problem presented here are (1) the effect of a lag correction factor on the resulting thermal death-time curve (2) heating characteristics of samples in thermoresistometer cups (3) measurement of the heating rates of samples in thermoresistometer cups, and (4) effect of sample depth on spore destruction time.

The sample cups are 0.443 in. in diameter, 0.333 in. deep, and were drawn from 0.25 lb. tinplate (0.008 in. thick). The depth of sample in the cup unless otherwise stated refers to the depth if the sample were spread uniformly across the bottom of the cup. The initial temperature of the sample is taken at 100.0°F. below the exposure temperature. Lag correction factors are calculated to 0.1°F. below the exposure temperature ($g = 0.1$°F.).

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THE EFFECT OF A LAG CORRECTION FACTOR ON THE THERMAL DEATH-TIME CURVE

A thermal death-time curve that is a straight line on semilogarithmic paper can be described by the equation

$$\log \frac{t}{F} = \frac{250 - T}{z} \tag{1}$$

or

$$t = (F)\ 10^{\frac{(250 - T)}{(\ z\ )}} \tag{2}$$

When data are available for time-temperature relationships that sterilize only a portion of the replicate samples exposed, the method described by Stumbo, Murphy and Cochran *(10)* can be used. The time of heating (U) in minutes at exposure temperature is used in the formula

$$D = \frac{U}{\log a - \log b} \tag{3}$$

to obtain a value "D," which is the time required to reduce the number of organisms by 90%. When a thermal death-time curve is constructed by plotting "D" values as a function of temperature, the resulting curve will be a thermal death-time curve representing the time for 90% (1D) desruction of the organisms. This thermal death-time curve can be described by the equation

$$\log \frac{D}{D_{250}} = \frac{250 - T}{z} \tag{4}$$

Failure to apply a lag correction factor (t_c) may alter a straight thermal death-time curve so that it will no longer be a straight line on semilogarithmic paper. If the lag correction factor is the cause of the digression from the true thermal death-time curve, the digression will take place only when the lag correction factor is significant in comparison to the heating time.

If the heating lag is greater than the cooling lag, the lag correction factor (t_c) will be subtracted from the exposure time (B_b) to give the time of heating (U) or

$$B_b - t_c = U \tag{5}$$

When the cooling lag is greater than the heating lag, the heating time (U) will be the sum of the lag correction factor and exposure time.

When uncorrected values are used; the equation of the thermal death-time curve for 1D is

$$D_f = \frac{B_b}{\log a - \log b} \tag{6}$$

Rearranging equation (5)

$$B_b = U + t_c$$

and substituting in equation (6)

$$D_f = \frac{U + t_c}{\log a - \log b}$$

$$D_f = \frac{U}{\log a - \log b} + \frac{t_c}{\log a - \log b} \tag{7}$$

The error is found by subtracting equation (3) from equation (7)

$$D_f - D = \frac{t_c}{\log a - \log b} \tag{8}$$

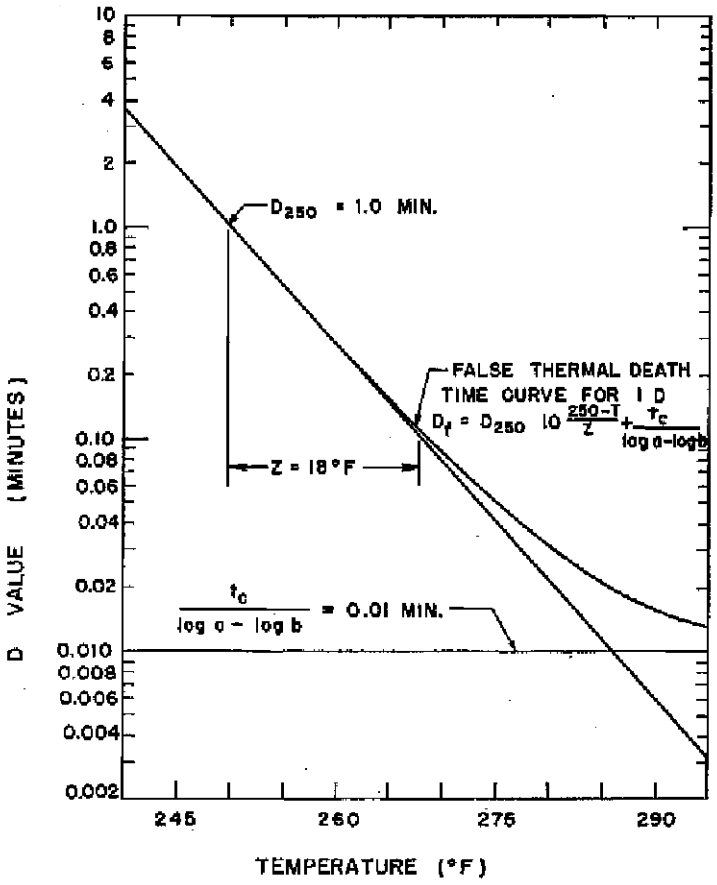

Figure 1. Relationship of true thermal death time curve for 1D, false thermal death time curve for 1D and the D curve error factor $\frac{t_c}{\log a - \log b}$ with a true D_{250} of 1.0 min., z of 18°F. for a 0.10 ml. sample in a thermoresistometer cup where t_c is 0.05 min. and (log a − log b) is 5.0.

In Figure 1 the relationship of D, D_f, and t_c (log a − log b) is illustrated graphically.

When the sample under study fails to reach 0.1°F. below the exposure temperature (g = 0.1°F.), the data obtained are not truly representative of the thermal resistance at that temperature and perhaps should be discarded. In the thermoresistometer cup, where the inoculum is assumed to heat as an infinite slab and when the thermal death-time curve has a z (z is defined as the F.° required for the thermal death-time curve when plotted on semilogarithmic paper to traverse one log cycle.) of 18°F. the limiting ratio of B_0/t_c is 2.25. When B_0/t_c is less than 2.25, the point of slowest heating in the sample will not reach 0.1°F. below the exposure temperature.

Heating Characteristics of Samples in Thermoresistometer Cups

The sample of material in a thermoresistometer cup is geometrically a finite cylinder. The height of the cylinder corresponds to the depth of the sample, and the diameter is the inside diameter of the cup. When the sample is introduced into the steam chamber, heat flows from the steam into the sample. The ratio of sample diameter to sample thickness is large enough to permit the sample to be treated as an infinite slab without

introducing appreciable error. It is assumed that the sample is of such consistency that it heats by conduction.

Undoubtedly there is some air surrounding the cup when it is drawn into the steam chamber. The effect of this air has been neglected since the quantity of air is small and the steam molecules can diffuse through the air layer and condense directly on the cup where less than 0.002 g. of steam is required to heat the cup and 0.01 ml. sample.

For purposes of calculation, it will be assumed that heat will flow into the sample from the top and the bottom only. If we consider the top surface first, the sample is at a lower temperature than the surrounding steam; therefore, heat will flow from the steam into the sample. As heat is removed from the steam, some vapor will condense; this condensation will take place on the top of the sample, since initially the sample has a lower vapor pressure than the surrounding steam. This condensation will aid in heat transfer, since there will be mass transfer as well as heat conduction. For practical purposes, it can be concluded that the top surface of the sample will approach the steam temperature the instant the sample is carried into the steam chamber.

In considering the steam metal cup interface on the bottom, some of the same conditions hold true as for the top. However, in this case, any condensate that forms will remain to increase the resistance to heat flow. The coefficient of heat transfer through the steam-cup interface where condensate will slowly accumulate can be estimated within limits *(4)* to be of the magnitude of 2000 to 2600 BTU $ft.^{-2}$ $hr.^{-1}$ $°F.^{-1}$ for filmwise condensation.

Since the resistance of the steam-cup interface is due primarily to the condensate film, it can be concluded that when the cup is dry the resistance to heat flow will be near zero and will reach a maximum when the condensate layer reaches its greatest depth. On the basis of this reasoning it can be assumed that the initial resistance of the outside surface of the bottom of the cup approaches zero; therefore, the bottom of the cup will approach the exposure temperature the instant the cup is surrounded with steam. The effect of the metal cup upon the rate of heating will be greatest when the sample is smallest.

To determine the effect of the metal cup on the rate of heating of a thin sample, the method of Boelter and Tribus *(2)* was employed. Their numerical method makes possible the calculation of the rate of heat flow through 2 bodies of different thickness and with different thermal diffusivities. The problem was set up in work sheet form and then solved numerically. The solution of the problem gave an f_h (f_h is defined as the number of minutes required for the straight line portion of the heating curve plotted on semilogarithmic paper to traverse one log cycle.) value of 0.00036 min. with a j of 1.27 for a 0.01 ml. sample in a thermoresistometer cup. The combined effect of increased f_h value and j would increase the lag correction (t_c) by less than 6%. The result is significant in pointing out that with very thin samples the effect of the cup cannot be disregarded. When the possible variations in sample depth are considered, the over-all effect of the cup in this case is negligible.

It can be concluded at this point that for sample volumes of 0.01 ml. and greater, the error will be small if we consider that the top surface of the sample and the bottom surface of the sample approach the exposure temperature the instant the samples are drawn into the exposure chamber. With these initial conditions, the equation for the f_h value of an infinite slab was found to be

$$f_h = 0.933 \frac{S^2}{a} \tag{9}$$

with j = 1.273. These values are given by Olson and Jackson *(5)*. The assumptions that are made in deriving this equation are: that the time intervals are large enough that all terms except the first of the originally derived equation are small enough to be neglected; that the initial temperature distribution through the sample is uniform; and that the surface temperatures are instantly brought up to the final temperature at time zero. Examination of equation (9) shows that the f_h value is directly proportional to the square of the sample half thickness (S).

The f_h value as given by equation (9) is a function of thermal diffusivity and sample depth. Thermal diffusivity is a property of the material being heated and is a function of the temperature; Jackson *(3)* used the value 0.016 $in.^2$ $min.^{-1}$ for food

products that were heated to 240°F. For food products heated to 212°F. the value of 0.015 in.^{2} min.$^{-1}$ is reasonable. The lag correction factor (t_c) that must be evaluated is the difference between the total heating time B_b and the lethality (U) at the exposure or retort temperature. The lag correction factor can be shown in equation form as:

$$t_c = B_b - U \tag{10}$$

Applying the methods of Ball *(1)* where

$$B_b = f_h \ (\log jI - \log g) \tag{11}$$

and making the assumptions that the slope of the thermal death-time curve of the organism under study is 18°F. and that the lag correction factor will be calculated only to a temperature of 0.1°F. below the exposure temperature ($g = 0.1$°F.) the value of the parameter $\frac{fh}{U}$ is 0.58 or

$$U = 1.725\, f_h. \tag{12}$$

Combining equations (10), (11), and (12)

$$t_c = f_h \ (\log jI - \log 0.1) - 1.725\, f_h$$

$$t_c = f_h \ (\log jI - 0.725). \tag{13}$$

If, for the general case, the initial temperature is assumed to be 100°F. below the exposure temperature and a value of j of 1.378 is used; the expression for the lag correction factor becomes

$$t_c = 1.379\, f_h. \tag{14}$$

An expression for the f_h of an infinite slab of finite thickness (2S) has been presented as a function of thickness and thermal diffusivity (a) in equation (9). This value can now be substituted in equation (14).

$$t_c = 1.379\ (0.933\, S^2/a)$$

$$t_c = 1.285\, S^2/a \tag{15}$$

Substituting the value for the thermal diffusivity (a) of food products in this temperature range (0.016 in.^{2} min.$^{-1}$); the expression for the lag correction factor (t_c) of a food sample heating by conduction as an infinite slab of finite thickness is given by

$$t_c = 80.4\, S^2 \tag{16}$$

Measurement of Heating Rates

When the lag correction factor for a thermoresistometer cup is calculated by equation (16), certain errors are present that arise from approximations necessary for solving the problem. An experiment was set up to determine whether the actual heating rates differ markedly from those calculated from theoretical considerations. In the initial tests sample depths of ¼, ⅜ and ½ in. were used. The cups had diameter to depth ratios of 8 and were considered to heat as infinite slabs. The cups were made by soldering a tinplate bottom to sections of 2-, 3-, and 4-in. pipe, respectively ¼, ⅜ and ½ in. long. A thermocouple junction was made from No. 30 gauge wire and was located at the midpoint of the cup.

A 5% bentonite solution, prepared according to the method of Townsend *et al. (11)*, was used as a sample to ensure conduction heating. The cups were heated in steam in a manner similar to the thermoresistometer cups. The steam in these tests had to be at atmospheric pressure to facilitate the rapid introduction of the samples into the steam. An autoclave with the door partly blocked to make a steam reservoir was used as the heating chamber (Figure 2). Temperature control was not a problem since the reservoir of steam was maintained at atmospheric pressure. A thermocouple was located at the center of the sample, and the temperature was measured by a recording potentiometer. Since the results from these tests could not be applied directly to the sample in a thermoresistometer cup, an effort was made to reduce the sample depth to approach more nearly that of a sample in a thermoresistometer cup.

A special steam chamber and cup holding apparatus was constructed (Figure 3) to expose a thin sample in a small (¾ × 1¾ in.) rectangular cup to steam at atmospheric pressure. Filter paper squares the same size as the cup were placed in the cup, a 36-gauge thermocouple was placed between the layers of filter paper so as to be located near the center of the sample. It was not necessary, as pointed out by Olson and Jackson *(5)*, to locate the thermocouple at the exact center of the sample since the slope of the heating curve is a constant for a given sample depth and product. Instru-

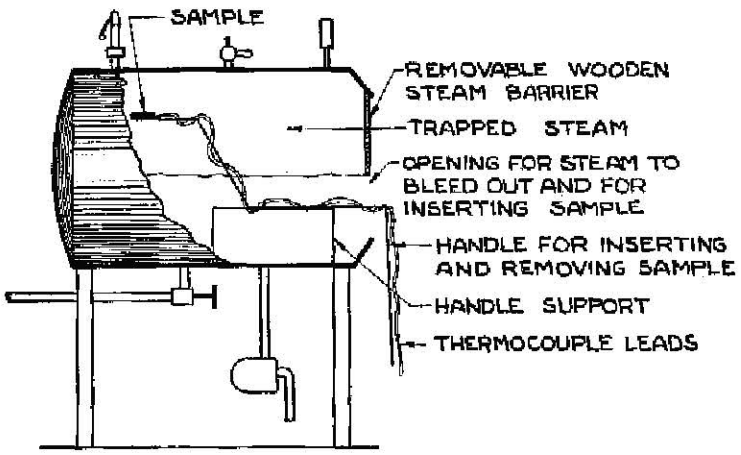

Figure 2. Autoclave with door partially blocked providing steam heating conditions at atmospheric pressure.

mentation was set up to measure accurately the time required for the temperature of the sample to go from 140°F. to 207°F.

The procedure followed in making a test was to pipette a volume of sample into the cup to give the desired depth. Both cup and sample were maintained below 80°F. during the loading period. To expose the sample the circular cup-carrier was turned, carrying the cup and sample into the steam chamber. The time required for the sample to heat from 140°F. to 207°F. was automatically recorded; the exposure temperature was also measured. The f_h value was determined by constructing the portion of the heating curve from 140°F. to 207°F. on semilogarithmic paper according to the method of Olsen and Jackson and then determining the f_h value. It was assumed that this portion of the heating curve was a straight line since the samples in the ¼, ⅜, and ½ in. cups (described above) when heated from 80°F. to 212°F. produced heating curves

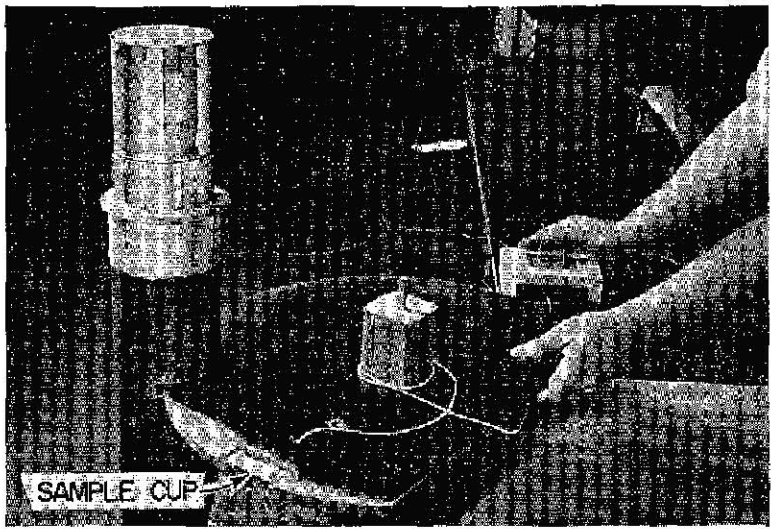

Figure 3. Apparatus for exposing cup with thin samples of media to live steam at atmospheric pressure.

whose straight line portion extended from below 100°F. to 211°F. By this method f_h values were obtained for 3 different sample volumes; the sample depths of these tests were 0.041 in., 0.070 in. and 0.100 in.

The results of the 6 tests are listed below:

Sample depth (inches)	f_h (minutes)
0.041	0.0358
0.070	0.0729
0.100	0.1565
0.250	0.072
0.375	2.15
0.500	3.85

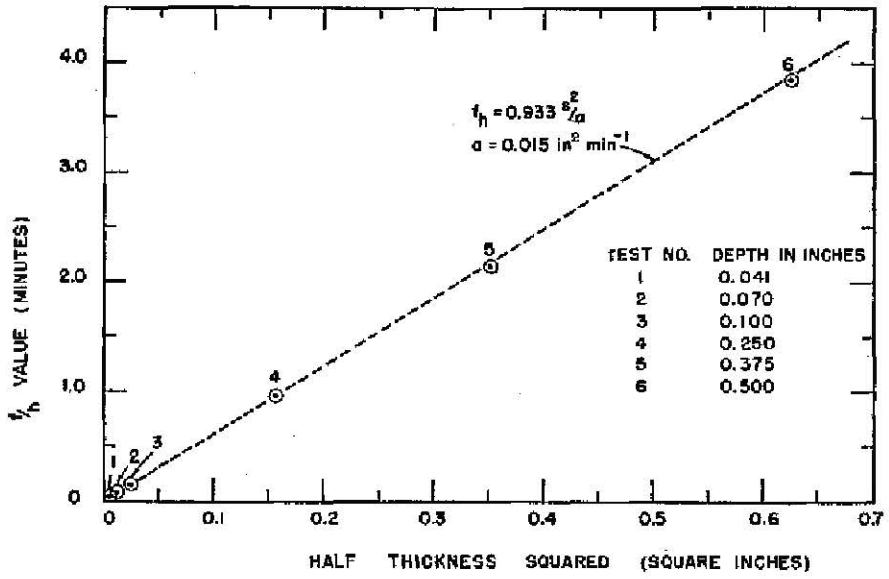

Figure 4. Measured f_h values of media in cups similar to thermoresistometer cups compared to calculated f_h values.

In Figure 4 the results are illustrated graphically. The f_h values as determined experimentally were for all practical purposes the same as those calculated by using equation (9). The thinnest sample we were able to test was 0.041 in. thick and corresponded to an 0.08 ml. sample in a thermoresistometer cup.

Effect of Sample Depth on Spore Destruction Time

The effect of sample depth on spore destruction time was measured to obtain lag correction factor data under actual operating conditions. At 280°F., 24 samples were exposed to a series of time intervals using 0.01 ml. per cup, and repeated using 0.05 and 0.10 ml. per cup. The sample volumes of 0.01, 0.05 and 0.10 ml. gave sample depths of 0.005 in., 0.025 in. and 0.050 in., respectively. Spores of PA 3679 were suspended in neutral phosphate buffer; dilutions were made to give 10,000 spores per cup in each test. The bacteriological procedure was the same as that used by Pflug and Esselen (7). The whole test outlined above was repeated 4 times; the results of one test are presented in Table 1.

The effect of the physical size of the sample was considered in evalauting the variation in results. It was found that a 0.01 ml. droplet would remain in the cup if the cup were inverted. Sometimes the 0.05 ml. sample would remain in the cup when

TABLE 1

Time (minutes)	0.01 ml. per cup		0.05 ml. per cup	0.10 ml. per cup
	Number of positive tubes out of 24 rep.	Range of probability limits at 5% level[1]	Number of positive tubes out of 24 rep.	Number of positive tubes out of 24 rep.
0.060	24	21-24	24	24
0.080	24	21-24	24	24
0.100	24	21-24	24	24
0.120	21	16-23	24[2]	24[2]
0.140	19	14-22	24[2]	19
0.160	23	19-24	18[3]	13[3]
0.180	17	12-21	12	13
0.200	16	11-20	10[3]	9[3]
0.221	11	6-16	6	14
0.241	15	10-20	5[3]	10
0.261	1	0-5	3	7
0.281	2	0-6	4	0
0.301	0	0-3	1	2
0.321	0	0-3	1	2
0.341	0	0-3	1	1
0.359			1	3
0.380			0	0
0.400			0	0
0.420			0	0
0.440			0	0
0.459			0	0
0.478				0
0.500				0

[1] Pflug and Esselen (7).
[2] Exceeds the higher limit of 5% probability level.
[3] Below the lower limit of 5% probability level.

inverted but often part of the sample would fall out. A part of the 0.10 ml. sample would always fall out on inverting the cup. An experiment was set up to determine the effect of the rapid deceleration of the thermoresistometer on the sample in the cups. It was found that with the 0.10 ml. sample some of the sample was thrown out regularly. With the 0.01 and 0.05 ml. samples, no loss could be detected.

RESULTS AND DISCUSSION

Thermal resistance studies at high temperatures and correspondingly shorter heating times are subject to greater potential errors than studies at lower temperatures. Data obtained by any thermal resistance method at the highest temperature possible for the method will be subject to the greatest errors. The point on the thermal death-time curve representing this data will be at the end of the curve. If a heating lag is present and the data are not corrected for this error, a straight line thermal death-time curve will no longer be a straight line (Figure 1). A cooling lag will bend the thermal death-time curve in the opposite direction as a heating lag.

A study of the heat transfer characteristics of a sample in a thermoresistometer cup has led to the conclusions that: (a) Only with a very thin sample will the lag correction factor be negligible for heating times of 0.005 min. (b) When the sample is an infinite slab of finite thickness (2S) the lag correction factor (t_c) will be a function of the square of the thickness. (c) The theoretical lag correction factor (t_c) for an infinite slab of finite

thickness (2S) measured to g = 0.1°F. with z = 18°F. and a = 0.016 in.^{2} min.$^{-1}$ is,

$$t_c = 80.4\,S^2 \qquad (16)$$

The theoretical heating rates of open metal cups were compared to the measured heating rate for 6 different depths, the thinnest of which corresponds to 0.08 ml. in a thermoresistometer cup. Good agreement was found between the calculated and measured values (Figure 4).

Results from the test measuring the effect of sample depth on spore destruction times fail to point out any significant difference between the 0.10, 0.05, and 0.01 ml. per cup samples. Since the calculated lag correction factor of the 0.10 sample is one hundred times that of the 0.01 ml. sample, the results can only be interpreted to mean that the lag correction factor of the 0.10 ml. samples tested was not significant in relation to the other variables present. The important conclusion that can be drawn is that if the lag of the 0.10 ml. sample is not significant the lag of an 0.01 ml. sample must approach zero.

The effect of the meniscus and of slight tilting of the cup was considered as possibly increasing the lag correction factor in the larger samples. In the 0.01 ml. sample with one filter paper disc the level of the sample is near the top surface of the paper disc. Since the sample is held by the capillary attraction of the paper fibers, the sample will be of uniform thickness regardless of whether the cup is level or tilted. With the 0.05 and 0.10 samples the liquid surface is above the paper disc and a meniscus is formed. When the cup is slightly tilted the depth of the sample near the wall is increased, which should increase the lag correction factor. These phenomena may be responsible for the increased number of stragglers observed with the 0.05 and 0.10 ml. samples. An attempt was made to relate tilted cups with positive tubes, but no significant results were obtained.

SUMMARY

A study of the heating rates and lag correction factors of samples in thermoresistometer cups has been made. An equation was developed for the lag correction factor of a sample in a thermoresistometer cup as a function of sample depth. The heating rates of samples in cups that were larger but had heating characteristics similar to samples in thermoresistometer cups were determined and found in agreement with calculated values. It was impossible to measure the heating rate of a sample thinner than the equivalent of an 0.08 ml. sample in the thermoresistometer cup. A study was made on spore destruction times when the same number of spores were used as an 0.01, 0.05 and 0.10 ml. sample per cup. This study, which was repeated four times, gave added proof that the lag correction factor of an 0.01 ml. sample in a thermoresistometer cup is insignificant and can be neglected.

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