PASTEURIZING AND THERMAL PROCESSING HIGH MOISTURE FIELD CORN

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Harris M. Gitlin

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by

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AN ABSTRACT

Submitted to
Michigan State University
in partial fulfillment of the requirements
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Department of Agricultural Engineering

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Approved 

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The F- and z-values of high moisture field corn, based on tests with thermal death time cans, were 0.27 minutes and 17° respectively. In addition, corn was given pasteurizing treatments below 212°F and stored at 40° and 86°F. The treatment and storage temperature influenced the pH that developed in the grain. The results are described and compared with previous work. The significance of Clostridium botulinum is discussed and it is pointed out that the process determined above should not be used until control of this organism is developed. Some suggestions are made where the processes might be used after further investigation.

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SUMMARY

Using thermal death time cans the F - value of the natural flora of shelled field corn in the moisture range of 22% to 32% w.b. was found to be 0.27 minutes with a z-value of 17°F. These values are similar to some previously reported for similar products at higher moisture contents. No determinations were made for the presence of Clostridium botulinum, but the hazard of its presence is emphasized in the literature. The F - value obtained is below the minimum of 2.45 minutes absolutely necessary for anaerobic packaging and room temperature storage. Bacterial counts were made, but not identification, and found to be as high as any reported in the literature for similar products.

Corn was also pasteurized at 170°F, 190°F, and 209°F for periods ranging from 1 to 16 minutes, aseptically canned in hermetically sealed ½ pint jars and stored at 40° and 86°F. Most samples stored at 40°F produced no gas pressure and retained a fresh corn odor and appearance for the first 9 months. All treatments stored at 86°F produced gas pressure and had a pleasant ensiled odor at 9 months. The pH values of the corn stored at 86°F was generally above 4.5.

It is certain that neither the F-value or the pasteurizing and storage process would completely destroy

Clostridium botulinum. Since there is no assurance that *Clostridium botulinum* is destroyed, and that toxin production could not occur, these processes are not recommended. Further investigations are suggested that might take advantage of the processes.

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INTRODUCTION

Shelled field corn (*zea maize* L.) is conventionally stored in one of two ways: as high moisture corn in a silo, or as dry corn in a bin. It is desirable to commence corn harvest as soon as the corn is mature, and to harvest rapidly. Field losses increase steadily with delay in harvesting after maturity.

Ensiling corn is a way of satisfying the need for early, rapid harvest. As a feed, however, shelled corn silage has certain limitations. The product can be fed only on the farm, or nearby. It must be fed at a rate sufficient to prevent surface spoilage. Total feed value losses, expressed as heat energy losses in the ensiling process, may amount to 15% or more of the original feed value (Barnett, 1954). Accidental losses due to molding, surface spoilage, etc. are common.

Drying corn for bin storage also has its problems. The cost of drying may well equal 10% of the value of the crop. Drying as rapidly as it is possible to harvest may cost even more in fixed equipment.

A search for alternate methods of storing high moisture corn was instituted. Three modes of degradation of such a product were immediately apparent: (1) that due to microorganisms, (2) that due to enzymes, (3) and that

due to natural oxidative processes, or other natural chemical processes. The conditions for high moisture corn storage, then, are similar to those encountered in the food industry, so the technology and technique of the food industry were applied to the problem.

Various methods of preserving crops are listed by Hall (1957). Among these methods are: (1) thermal processing, (2) freezing, (3) freeze drying, and dehydration and freeze drying, (4) the use of pH control, along with some other conditions, (5) additives to control osmotic pressure in the product, (6) antifungal and antibacterial agents, added as a gas, liquid, or solid, and (7) ionizing radiations. Rahn (1945) summarized the effects of many of these methods on microorganisms. While some of these methods may be satisfactory for microorganisms they are not also effective for enzyme destruction.

The work reported here is concerned with item (1) thermal processing of field corn.

REVIEW OF LITERATURE

A. Significant Relationships.

Microbiologists, food scientists, and others have developed methematical methods for evaluating the effect of heat on bacterial populations (Ball,1957, and Tischer, 1954). Two parameters are used in describing the effect of heat, z and F. The z is related to Q_{10}. Both F and z are functions of the specific microorganism and substrate. Also, the number of such organisms in the product, and the rate at which these numbers are reduced by the temperature used affect F. These parameters are related as follows:

$$F_{250} = F_T \times 10^{\frac{(T-250)}{z}} \qquad (1)$$

This expression relates the effect of heat treatment at any time and temperature to the equivalent time at 250°F.

$$\text{Log}_{10} \frac{N}{N_o} = -\frac{F_T}{D} \qquad (2)$$

This relates the number of microorganisms remaining after a process to the time of heating at some temperature T. The symbols used throughout this report are: (Ball 1957)

B - The total elapsed time in which heat was applied to a product, minutes. Note that no temperature of the product is implied, although a temperature of the applied heating medium is usually also stated.

D - The time required to reduce the population of a particular microorganism to 10% of its original numbers at some specified temperature, minutes. This value is specific for a microorganism, the temperature of treatment, and the substrate, including pH.

F - The time at 250°F required to obtain 'commercial sterility', minutes. This condition is achieved when no further growth or development of microorganisms will occur under normal conditions of storage and handling. The F-as used in this report refers to the time of treatment at which no swelling was observed in the thermal death time cans, ans swelling was observed at any longer time.

F_{250} The time of heat treatment at 250°F that would have the same lethal value as some other time, F_T, would have at temperature T, minutes.

F_T - The lethal time of a process at some temperature T, minutes.

N - The number of viable microorganisms of a specific type at any time. The number is commonly expressed as number per unit weight or volume.

N_o - The initial number of a specific microorganism that are viable, commonly expressed as number per unit weight or volume.

T - The temperature of a process, degrees F.

$_z$- The slope of the thermal death time curve, expressed as degrees F required to reduce the time of an equivalent process to 10% of its original value.

From equation (2) it will be seen that the number of microorganisms will never reach zero, but may get very small. When N is less than one its value is related to the probability of finding a viable organism in any one container, or unit weight or volume. In commercial processing it is necessary to have this N-value extremely small. In the tests reported here only a relatively small number of containers were used. Based on these results the probability of finding a viable microorganism when a large amount of corn is processed may be higher than desirable.

B. Populations of Microorganisms.

One of the first considerations in establishing a time and temperature for thermal processing of corn would be the numbers, type, and location of microorganisms common in corn. Christensen (1948) cites evidence of almost universal occurrence of fungus mycelium in the outer layers of grains, with some evidence of growth into the endosperm and embryo. The deeper penetration of fungi is resisted by an inner membrane of the corn covering. A list of fungi found internally is included in the article, along with a description of the types of microorganisms found on the corn surface. Mold counts made on corn meal varied from 3500 to

400,000 per gram. Bacterial counts on commercially available corn meal varied from 5,000 to 60,000 per gram, and from 1 to 600,000 per gram in freshly prepared corn meal. When moisture was subminimum for growth the mold count declined with time as much as 90% in two months.

Manns (1921) was interested in fungi causing corn damage and carried inside seeds. His study required a surface disinfection of the grain for study. The point of the kernel contained the greatest infection. The point area included a segment of 1/5 to 1/6 inches back from the point. Most fungi had gained entrance to the kernel only in the cavity under the cap at the point of the kernel, or had penetrated only short distances into the pericarp. Various numbers of corn kernels from many U. S. states were checked in the test. The variation was large, but approximately 50% of the kernels showed internal infection of one or more fungi.

Boruff (1938) made a study of bacterial populations of grains received at a distillery. An example of the data follows:

Date	Number of bacteria per gram	Treatment
1936 October	15,000	Kiln and natural dried
November	35,000	Kiln and natural dried
December	2,400,000	Kiln and natural dried
1937 January	2,000,000	Kiln and natural dried
February	300,000	Only kiln dried corn
March	3,500,000	Only natural dried
April	1,500,000	Only natural dried
May	900,000	Only natural dried
June	200,000	Only natural dried

The range of counts for June (1937) had a maximum count of 800,000 and a minimum of 30,000 with an average of 240,000 counts per gram. The effect of kiln drying on bacterial counts was shown as follows:

Type of drying	Number of samples	Bacteria/gram	Moisture %,w.b.
Natural	20	3,725,000	17.3
Kiln	12	990,000	14.5

The difference in count appears large. In terms of thermal processes required, however, the difference is less than one log cycle and hence the process time would be less than one D-value more for the larger population.

The effect of damaged kernels on bacterial count in corn was also shown by Boruff:

Material	Count/gram	Ratio of count no damage : damage
damaged	18,560,000	
no damage	40,000	1 : 285
damaged	6,464,000	
no damage	45,000	1 : 139

Christensen (1948) made mold counts on 1945 corn received at 5 midwest terminals. The results are summarized as follows:

Grade	Max. moisture content, % w.b.	Number of samples	Range of counts	Average count
1	14.0	9	0-48,000	12,000
2	15.5	17	1450-522,000	96,000
3	17.5	21	0-1,479,000	262,000
4	20.0	16	5,000-1,350,000	390,000
5	23.0	18	25,000-2,270,000	940,000
Sample		39	0-4,375,000	830,000

James (1928) made a study of bacteria prevalent in sweet corn for canning. The initial bacterial count made on ears freshly cut in the field was about 30,000 organisms per kernel when grown on plates at 30°C, and an average of 1 organism per kernel when grown on plates at 55°C. Dilution counts made from corn as it passed through the cannery processes showed the following change:

When sampled	Number at 30°C incubation	Number at 55°C incubation
original	135,000 per kernel	2 per kernel
final	22,500,000 per kernel	2 per kernel

James also reported the following observations and measurements:

(1) A pre-heat of 185°F reduced the 30°C count to 40, but did not affect the 55°C count.

(2) Corn with husks was piled in a bin. In four days the temperature reached 51°C in the corn. At 50°C there were 580,000 viable organisms per kernel in the corn.

(3) Corn remaining in a farmer's wagon overnight in a rain reached a temperature of 55°C and had a 30°C plate count of 10×10^6 per kernel.

(4) Eighteen groups of microorganisms were identified and listed. The largest number were a subtilis type. Thirteen of the 18 groups formed spores readily. Some of these groups were shown to be present in fresh corn, to multiply during storage, and to be unaffected by a pre-heat of 185°F.

(5) Thermal death point determinations were made on all 57 spore forming cultures, using only organisms surviving 240°F pre-treatment for 15 seconds. Suspensions of 10^6 spores per cc. were tested at pH 7.0. The heat treatment and survivor data were listed. In general, 2 to 3 minutes at 250°F were required to get negative responses.

It is evident that there is a wide variation in the numbers and types of microorganisms that may be encountered in corn samples. From equation (2) it can be seen that the time of treatment in a thermal process is some function of the population and type of microorganism, among other factors. The variation in count and types of organisms found on corn led to a search for possible ways to reduce the population to some minimum and more uniform value.

C. Methods of reducing the number of microorganisms.

Thermal processing is a method of reducing the population of microorganisms. The literature search was confined to methods that might lend themselves to a pre-treatment, and rapidly and inexpensively reduce the population count. A physical treatment, common to all food processing, is to wash the produce. Chemical means are limited to those materials that would not be toxic to livestock.

Charlton (1935) made some observations on the germicidal efficiency of chloramine-T and calcium hypochlorite. The germicidal value was found to vary with the time, pH,

temperature, and available chlorine. At a fixed pH and temperature the time for reduction vs. log of remaining population was nearly a straight line. These results would imply that the order of destruction of the population in chlorine is similar to that in heat. As the pH was changed from 6.0 to 7.5 the time for reduction to 1% of the original population varied from 10 to 70 hours. At a fixed pH of 8.7 the time required to reduce the population to 1% of the original was rapidly reduced with increase in temperature. The following table shows the germicidal effectiveness of calcium hypochlorite at 25°C on Bacillus metiens spores.

PPM available chlorine	Reaction pH	Time to destroy 90%
1000	11.3	64 minutes
1000	7.3	less than 20 sec.
100	10.4	70 minutes
20	8.2	5 minutes

It was concluded that pH had the most significant effect on the time required to reduce the spore population. A rise of 18°F decreased killing time by about 82% if initial pH was 6.0 and 71.5% if initial pH was 8.7. Doubling the strength of chlorine, at initial pH and temperature, reduced killing time 40-60%.

LaBree (1960) studied the effect of chlorine on spores of Bacillus coagulans. His primary interest was the effect of temperature on the action of chlorine. Innoculations of 10,000 spores per ml. were treated at three pH values (4.5, 6.8, 7.8) using three concentrations of chlorine (5,10,20 ppm) and four temperatures (15,20,30,60°C). The

data were reported as time to reach 90% reduction in population, or a reduction of one log cycle of the original population. The following data are taken from the work cited:

Temp.°C	Chlorine PPM	Reduction %	Time, in minutes at pH 7.8	6.8	4.5
15	20	90	13.0	9.0	4.0
60	20	90	1.0	0.25	0.30

It was further demonstrated that very little loss of chlorine occurred at 60°C.

It is evident that small concentrations of chlorine at elevated temperatures can reduce bacterial populations in a relatively short time; this is particularly true at the pH of 6.5 to 7.0 expected in water containing shelled corn.

D. The Importance of Clostridium botulinum.

Experience in food processing has resulted in the identification of certain microorganisms whose D-value is high, and whose destruction is particularly important. The destruction of these microorganisms frequently determine the extent of a process.

Clostridium botulinum commonly determines safe processing time for two major reasons: (1) the D-values are relatively high; (2) minute quantities of the toxin of this microorganism are fatal. A literature search was made to obtain some evidence of the effect of this microorganism on livestock, and its distribution in corn.

Bergey's manual (1957) lists four types of

Clostridium botulinum. Two of the four, B and C, are considered pathogens, while two, D and E, are pathogenic on injection only.

Graham (1921) established that cattle are variously susceptable to botulism. Some cattle die, some do poorly or become ill. Clostridium botulinum was definitely located in silage used to feed cattle that had died or were doing poorly. The particular places where the organism was located in the silage were associated with scarcely visible mold or yeast.

Tanner (1944) postulated that the development of Clostridium botulinum in acid foods may be due to pockets of alkaline areas caused by molds, as in silage.

Clostridium botulinum was identified as the cause of 'duck sickness' by Shaw (1936). Thousands of wild ducks were dying in their usual swamp feeding grounds along their flyways. Later, Quortrup (1941) identified potential toxin (Cl. botulinum) producing areas in the western North American duck marshes. The areas were identified by having practically 0% oxygen in the water. If decaying vegetation was present in the water then Clostridium botulinum toxin could be present. It was noted that aerobic bacteria quickly destroyed pre-formed toxin. Later Quortrup (1943) described some ecological relations between Pseudomonas aeruginosa and Clostridium botulinum type C. The oxygen consuming and alkali producing capacity of Ps. aeruginosa were observed. A definite symbiotic relationship with Clostridium botulinum was demonstrated using broths and swamp weed substrates.

Escherichia coli did not support Clostridium botulinum even though the oxygen content of the water was reduced. This observation was attributed to a reduction in the pH that was also caused by E. coli.

An intensive survey of the distribution of Clostridium botulinum in all states of the United States and Canada was reported by Meyer (1922). In California there was evidence of the microorganism on fruits and vegetables, in manured and cultivated soils, in virgin soils, and deep in the earth uncovered by rock slides. The organism was also found in pastures, in hay, and in sewage. Clostridium botulinum was found in every state in the United States, including Hawaii and Alaska. There was more frequent evidence of the organism in the western states than in the central states. Examples where Clostridium botulinum was found include: corn roots and leaves in Minnesota, silage from Michigan State University, and cornstalks in Louisiana and Mississippi. Michigan was listed as having a low spore index.

In the total survey Clostridium botulinum was found in 7.5% of the cultures made from corn husks, leaves and stalks, compared to 20% of the cultures made from silage.

Considering the distribution and lethal character of Clostridium botulinum (Dack, 1943) a literature search was made for further information about the conditions required for toxin production. It was reasoned that the production of toxin could be prohibited under storage conditions adequate for livestock feed where it could not be

done for human food.

Studies of the metabolism of Clostridium botulinum in various media were made by Wagner, et al (1925). They found evidence that the production of gas by this organism depended upon crabohydrate utilization. Type B did not produce toxin in specific preparations using glucose, although this was not true for type A. Some interest was shown in the effect of the presence of air on the microorganism. It appeared that the composition of the medium determined the degree of anaerobiosis necessary for the growth of an obligate anaerobe.

Lewis, et al, (1947) searched for media and environmental conditions for producing highly toxic cultures of Clostridium botulinum, type A. The optimum media consisted of corn steep liquor, glucose and powdered milk. The importance of pH was noted in this work. Production of toxin was reduced as pH dropped below neutral. It was also noted that those cultures which were shaken did not produce as much toxin as the undisturbed. The reduced production was attributed to air incorporated in the media by shaking.

A schematic of events leading to final toxicity of culture filtrates of Clostridium botulinum was presented by Bonventre (1960).

The effect of pH on growth of Clostridium botulinum was studied by Townsend, et al, (1954). No growth was observed below pH 4.7. In corn steep liquor there was growth at pH 4.98 but none at 4.77, and there was no toxin produced

at the latter pH. They also found that toxin would be present at a pH lower than that at which gas was formed.

Sognefest, et al, (1948) determined the effect of pH on thermal process requirements of canned foods. Thermal death time cans were used in their tests and sweet corn was included in the foods tested. Selections of their data are included here:

pH	White corn		Yellow corn	
	F (min)	z (°F)	F (min)	z (°F)
5.0	1.80	18	0.45	14
5.5	3.70	17	3.30	17
6.0 - 6.3	5.6	20	7.90	20
6.5 - 6.8	4.40	17	9.0	21

It should be noted that these data are not entirely clear on the exact conditions of the tests, including innoculation numbers.

Spores of Clostridium botulinum were germinated by Wynne and Foster (1948) in atmospheres of natural gas and in air. There was a greater delay in germination of the spores in air than in the natural gas, but germination took place in both environments. The lag period was described as:

$$L = \frac{C}{\text{Log } I}$$

Where: L - is the lag period of the spores germinated in air, hours.

C - is a constant from 95 to 101.

I - is the number of spores per ml. in the innoculum.

There is considerable evidence in literature to support the following observations:

1. Clostridium botulinum is toxic to livestock.
2. It is almost universally distributed.
3. Corn provides a good substrate for toxin production.
4. Toxin can be produced in the presence of some air.
5. Toxin production can be eliminated at low pH values.

Thermal processing will destroy Clostridium botulinum, and considerable study has been made on this subject for about forty years.

Esty and Meyer (1922) reported determinations of the heat resistance of Clostridium botulinum. The following values were determined under optimum growth conditions:

Time to destroy all growth in a fixed number of spores, minutes.	Temperature, °C.
4	120
33	110
330	100

Another part of this work involved counting the spores remaining after various times of treatment at 100°C. Although the concept of D-value had not been proposed at this time, the logarithmic order of death was noted, as well as the relation between time and temperature of treatment, z. Esty, et al, also reported that as much as 100 days of incubation at 35°C may be required before evidence of growth of the remaining spores will appear. One sample germinated 378 days after treatment when stored at 36-37°C.

Esty pointed out the influence of concentration of microorganisms upon time required for destruction and

found variations with the strain selected. It was further observed that a reduction in pH reduced the time required to destroy the organism. For example, there was very little difference in the time required to destroy spores in ripe olives at pH 7.93, corn at pH 6.35, and spinach at pH 5.05. In food juices with pH below 4.5, however, there was marked increase in destruction rate. According to the work of Townsend et al (1954) there may be some question whether it is a change in destruction rate or inhibition of growth. The interaction of substrate and pH might have been inferred from the work of Esty and Meyer.

Tanner and McCrea (1923) reported that it took ten minutes at 120°C and pH 6.8-7.4 to destroy Clostridium botulinum; however, no count of the numbers was made.

Further studies of the thermal death time of Clostridium botulinum spores by Dickson, et al, (1925) included a test using 37,000 tubes of spores and covered a period of 28 to 39 months. The problem of skips and delayed germination was reported. Once case of germination of heat treated cultures was delayed 37 months.

Townsend, et al, (1938) made extensive tests to determine the heat resistance of Clostridium botulinum. One object of the work was to locate some less toxic anaerobe that had similar thermal characteristics. Two types of Clostridium botulinum spores were used, type 62A and 213B, along with a putrefaction anaerboe labeled PA 3679. Some of the data are included here because of their bearing on the

work reported.

Product	Organism	Population X 10^6	Temperature of treatment 105°C Survive min.	105°C Destroy min.	110°C Survive min.	110°C Destroy min.
Raw yellow bantam corn	62A	75	40.0	45.0	4.0	5.0
	213B	25	75.0	80.0	4.0	5.0

As a composite of both types of Clostridium botulinum the following values were reported:

Substrate	F, Min.	z, °F.
PO_4 buffer	1.9	17.6
Asparagus	0.39	15.0
Spinach	0.70	15.5
Peas	0.87	13.6
Whole milk	0.55	14.5

Retort temperatures for these tests included 100, 105, 110, 115, and 120°C. Different numbers of organisms were involved in the above data.

Examples of variations using the same number of organisms but different substrates are shown in the following data:

Type organism	Population	Substrate	F Min.	z, °F.
62A	10 X 10^6	Asparagus	0.19	14.3
		Spinach	0.33	15.1
	20 X 10^6	Peas	0.30	13.5
		Spinach	1.05	18.0
	75 X 10^6	PO_4 buffer	0.40	16.5
		raw corn	1.14	18.5
		canned corn	1.79	20.8

Type organism	Population	Substrate	F Min.	z °F.
213B	12×10^6	Peas	0.38	13.4
		Asparagus	0.27	14.2
	2×10^8	Spinach	0.68	15.5
		Peas	2.15	18.2
	25×10^6	Canned corn	1.38	17.2
		Raw corn	0.90	14.8
		PO_4 Buffer	2.53	20.4
		PO_4 Buffer	0.30	13.8

Comparisons of composite F-and z-values for all runs and all suspensions are included in the selected values below:

Substrate	62A		213B	
	F Min.	z °F.	F Min.	z °F.
PO_4 buffer	1.70	16.4	2.00	18.0
Asparagus	0.39	15.0	0.09	15.0
Peas	0.30	13.4	1.40	15.6
Spinach	0.45	14.7	0.50	14.3

Values of F for Clostridium botulinum in corn products were reported by Tanner (1944), and include the selection below:

Product	pH	F value or heating time in minutes					
		194°F.	203°F.	212°F.	221°F.	230°F.	239°F.
Hominy	6.95	600	495	345	120	35	10
Corn	6.45	555	465	255	105	30	15

The following information is taken from a study reported by Reed, et al (1951) on organisms of significance in processing:

Population	Time to destroy 99.999% of Type A *Clostridium botulinum* spores in corn at 212°F, minutes
2,500,000	78
1,750,000	37.2
125,000	20.7
100,000	9.4

In the work an F-value for *Clostridium botulinum* in fresh corn was established as 1.12 minutes with a z of 17.7°F. Considerable variation in D-values was experienced, primarily due to erratic nature of recovery of viable spores near the end of the treatment. This irregularity reduced any utility of D-values in determining thermal death time curves. The erratic behavior was attributed to particular resistances or weaknesses in cultures or organisms used.

Dack (1943) pointed out that some cultures of *Clostridium botulinum* produced gas and some did not; and that cattle are susceptable to these toxins. The four toxins listed were all destroyed at 80°C for ½ to 6 minutes, at 72°C for 2 to 18 minutes, and at 65°C for 10 to 85 minutes.

The persistence of spores of *Clostridium botulinum* was demonstrated by Evans (1960). Spores of *Clostridium boutulinum*, NCA 3679, and four other bacteria were stored for 40 months at 30°C in buffer solutions at pH of 5.0, 6.0, and 8.0. A pre-heat treatment of 85°C for 15 minutes or 100°C for 10 minutes was given a portion of the spores of each bacteria. In the case of *Clostridium botulinum* and NCA 3679, the viability of the spores was practically

unchanged over the storage period. Their capacity to survive was not appreciably influenced by the pre-heat treatment. Spores of the other four bacteria varied greatly in their viability over the storage period.

It is evident that considerable variation in F-and z-values may be expected in dealing with microorganisms. In the case of Clostridium botulinum it is reasonable that only maximum values determined could be accepted because of the hazard involved.

E. Control of enzymes.

Enzymes as well as microorganisms must be controlled to preserve a food or feed product. Sizer (1943) noted that inactivation of the majority of enzymes is marked at 50 - 60°C. Sumner and Somers (1947) indicate that nearly all enzymes are irreversibly destroyed by heating to 80°C. According to Blanck (1955) it is customary to pasteurize all juices and acid food at temperatures not less than 190°F for 30 to 90 seconds to definitely eliminate enzymes as a source of deterioration.

Processes at temperatures of 250°F and below have generally destroyed enzymes in the time designed for microorganisms. Recently, high temperature-short time processing has resulted in difficulty with enzyme inactivation in the product due to the high z-value of enzymes.

Guyer and Holmquist (1954) were interested in the regeneration of the enzyme peroxidase. Peroxidase was

chosen because it is easily identified and difficult to destroy by heat. They concluded that the curve of destruction of peroxidase was flatter than that of most bacteria. F-values for peroxidase destruction were obtained, but the concept of z-values for enzymes was not presented.

Farkas, et al (1956) determined that peroxidase in stock pea filtrate had an F-value of 6 minutes and a z-value of 48°F. Further work by Zoueil and Esselen (1959) included a study of the mechanism and rate of regeneration of peroxidase in green beans and turnips. The following table shows F-and z-values determined as sufficient to destroy peroxidase activity (a) immediately after treatment and (b) after storage up to 12 weeks.

Product	Immediately after treatment		After storage	
	250°F. Min.	z, °F.	250°F. Min	z, °F.
Green beans	0.48	41.0	3.0	47.0
Turnips	1.8	23.0	11.3	46.0

The concept of D-value as the time to destroy 90% of the peroxidase activity is shown in this work.

F. Chemical preservation.

Food and feed have been preserved by chemical additives under conditions where Clostridium botulinum is not a hazard. Salt concentrations up to 10% have failed to prevent survival and growth of Clostridium botulinum according to Wyant and Normington (1920). The conclusion

of this study was that pH is the best method of control.

Seven compounds were tested on sixteen common food spoilage fungi by Klis,et al, (1959). Myprozine and rimocidin were found to be effective against all fungi at 10 ppm. Sorbic acid at 500 ppm was also effective on fungi in this test, but York and Vaughn (1955) had found that sorbic acid at 1.0% did not destroy or inhibit growth of Clostridium botulinum.

Gases are also used to control microorganisms. Lloyd and Thompson (1956) present a detailed account of the use of ethylene oxide in various combinations as a sterilant. Foods and surgical materials were sterilized with the gas. They found that the gas may be considered a bactericide, fungicide, viricide, and possibly a sporicide, but it could not be called an inhibiting agent.

FIGURE 1

Type of jars and cans used in the tests.

1. Thermal death time can cover, and inverted can showing 'dimple' in the bottom.

2. Thermal death time can with corn and water ready to close.

3. A closed thermal death time can.

4. A thermal death time can that has swelled, showing arrangement of Ames dial gage for measuring thickness.

5. A half-pint jar of corn used in the pasteurizing tests, showing the two-piece lid.

FIGURE 2

Special equipment used in the pasteurizing tests.

1. The screened cage, holding over $2\frac{1}{2}$ pints of corn.

2. The handle of the cage showing the valve operating rods.

3. The location of the two valves that assured dumping the desired quantity in each jar.

4. The sheet steel palette holding five-$\frac{1}{2}$-pint jars.

5. The electromagnetic wand, with magnet resting on a jar lid; note location of switch at handle on other end.

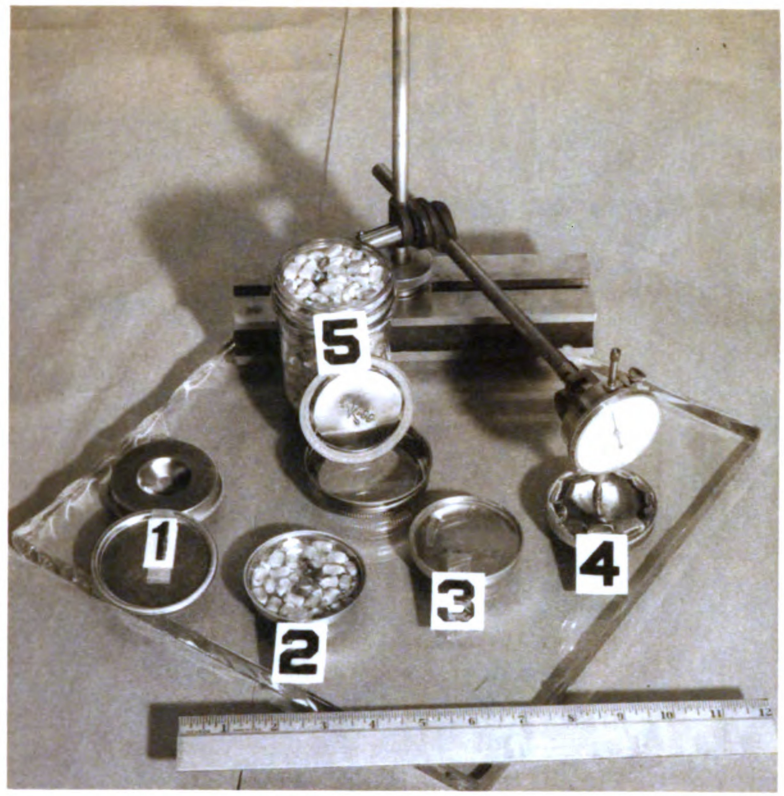

Fig. 1. Type of jars and cans used in the tests.

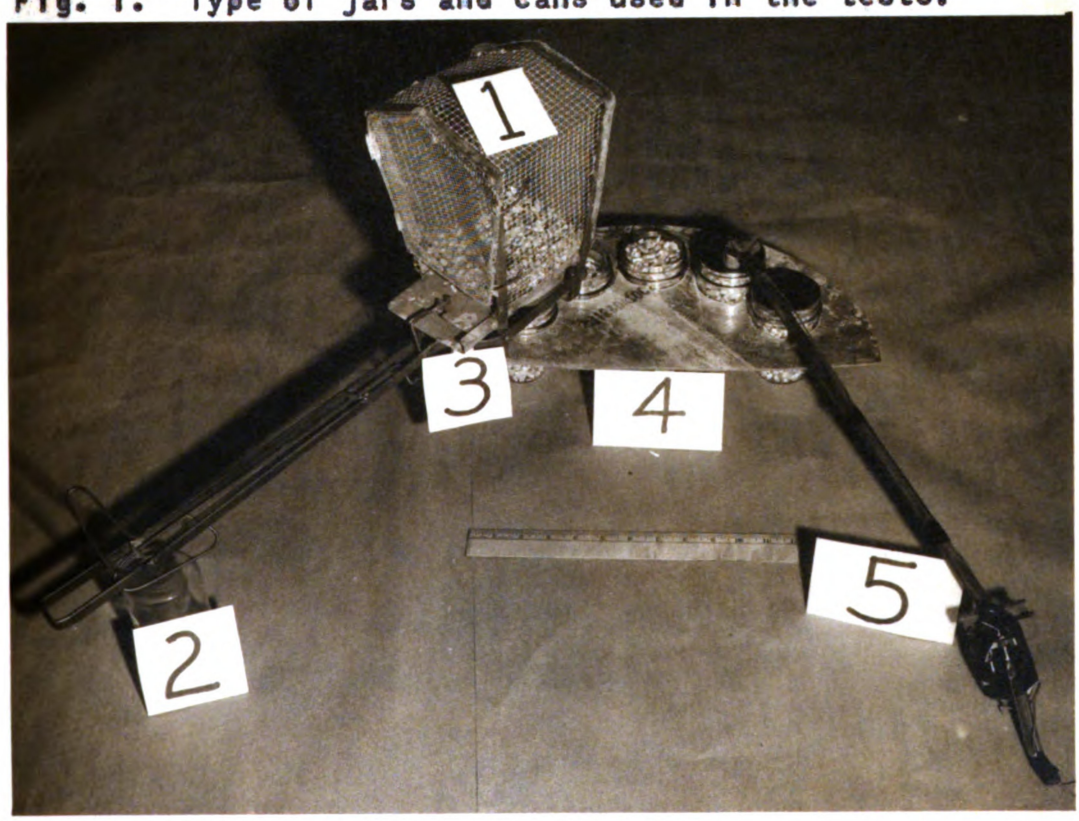

Fig. 2. Special equipment used in the pasteurizing jars.

FIGURE 3

The retort and extension used in pasteurizing corn.

1. The lid of the retort extension showing glass window.
2. The handle of the screened cage extending from the slot in the retort extension.
3. The valve controlling live steam access to the retort extension.
4. The upper portion of the retort.

FIGURE 4

The miniature retorts used to process the TDT cans

1. Miniature retort with packing gland for admission of thermocouple wires.
2. To the right is a petcock, in the retort cover, used to vent during processing.
3. The 'quick-on' steam supply valve.
4. Valve to drain, also slightly opened during processing.
5. The 'quick-on' cooling water supply valve.
6. Above is the 'quick-on' retort top drain valve used to outlet water during cooling.
7. Retort cover clamp.

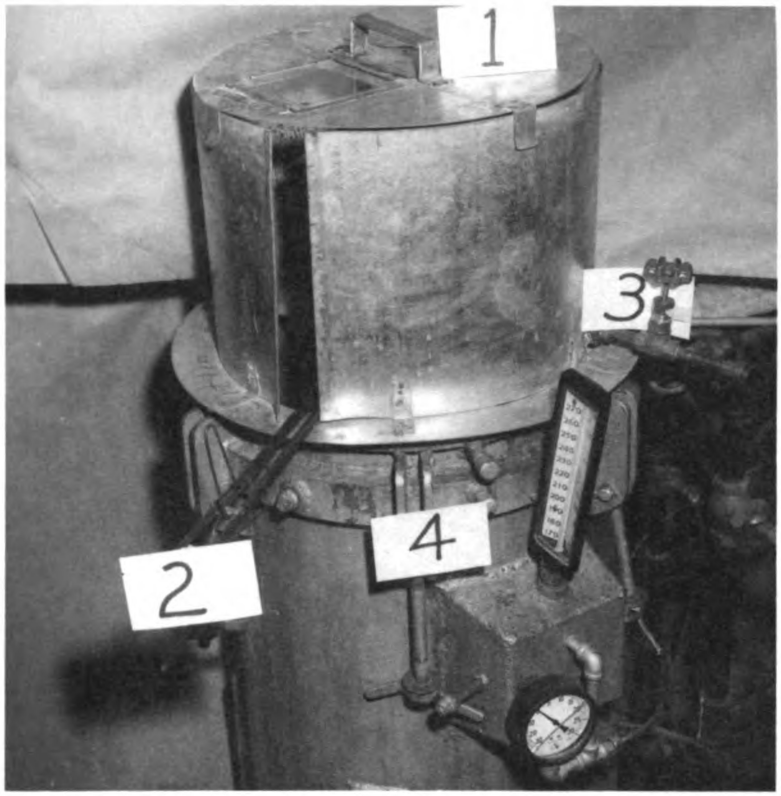

Fig. 3. The retort and extension used in pasteruizing corn.

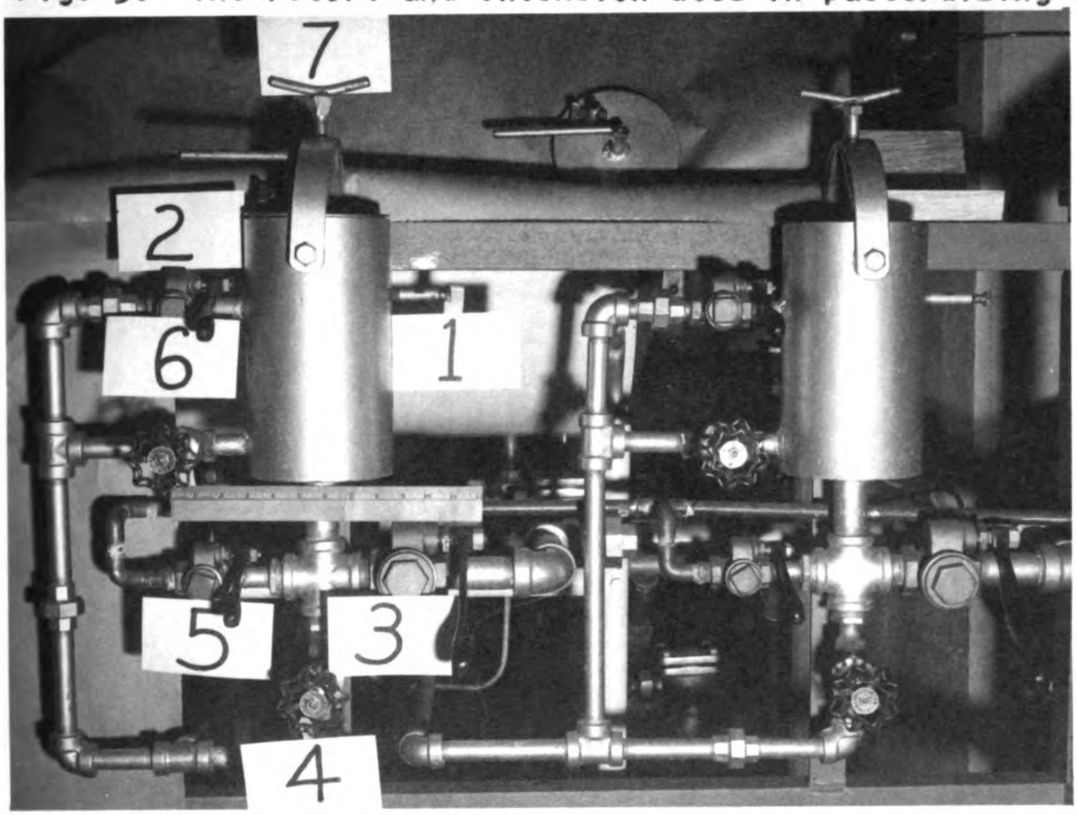

Fig. 4. The miniature retorts used to process the TDT cans.

EXPERIMENTAL PROCEDURE AND RESULTS

The experimental work was largely exploratory in nature, each phase being planned after results of the previous phase were known. The requirements for heat processing occupied most of the experimental work, and will be described in the order performed. A close approximation of an F_{250} and z-value for high moisture field corn was desired.

The majority of tests were made with thermal death time (TDT) cans (Sognefest, et al, 1944) using miniature retorts. A detailed description of each test is included in Appendix II. After each heat process the cans were incubated at 86°F. Distinct swelling of the cans was taken as evidence that some microorganisms survived the process, and this was recorded as (X). No swelling of the cans was interpreted as satisfactory destruction of the microorganisms, and was recorded as a (-). 'Distinct swelling' was interpreted as 0.030 inches or more, while 'no swelling' was considered as less than 0.010 inches. Any swelling between 0.010 and 0.030 was recorded as (?). Sensory examination of swelling in this latter range gave no evidence of any microbiological activity.

In most tests 8 grams of corn and $9\frac{1}{2}$ ml. of tap water were placed in each TDT can. Initial corn moisture was determined for each test and recorded as percent, wet

basis. In general, the moisture content varied between 22 to 32%.

Several times during the series of tests, and at each temperature used, a TDT can was equipped with a thermocouple and the temperature history recorded. These data were used to determine the equivalent time at any temperature, using the method described in Appendix I. The equipment used is shown in Figures 1 to 4. The combined results of all TDT can tests are shown on Table 1. The results of the pasteurized tests are shown on Table 2.

A brief description of each test by number is given to assist in an understanding of the Tables 1 and 2; the details are given in Appendix II.

TEST NO. 1:

Whole kernel corn of the 1959 crop was immersed in water at the temperatures and times shown in Table 2. The corn was placed in half-pint jars for storage. Ten jars were filled at each temperature and time, five were stored at 40°F and five at 86°F. After 9 months of storage no mold was found in the 130 jars treated, including the control jars that had no process. Very little change in color was observable at this time.

After 9 months storage one jar from each storage temperature was opened from the five that had been pasteurized 8 minutes at 190°F. The corn from 40° storage had a good fresh corn odor, and a pH of approximately 6.0. The

corn stored at 86° had a good ensilage odor and a pH of 4.5. Lithmus paper was used to determine pH at this time.

TEST NO. 2:

Early indications from preliminary tests suggested that an F of 0.1 minutes, or slightly greater, may be adequate if a z of 18° is assumed. This test was designed to get a wider range of values for estimating both F and z.

Whole kernel corn of the 1959 crop and water was processed in TDT cans. In all the temperatures above 200°F the times shown in the Table I are calculated time at retort temperature based on a z-value of 17°F. Originally a z of 18° was assumed, but the times were changed when further data was available. At temperatures below 200° the times are actual times of immersion in water.

The results of this test indicated that an F of 0.16 minutes and a z of 18° were the approximate values to use in planning future tests.

In all tests to this date the corn, which had been stored at 0°F, and held for about 2 hours at room temperature before the test started. Thereafter, all corn under test was held for 18 hours at room temperature before test.

TEST NO. 3:

This test was made to check the approximate F-and z-values assumed from previous tests before planning more comprehensive tests. The influence of a longer holding

period could be observed by a brief test. The results indicated that F may be slightly higher than supposed from Test No. 3, and that z was certainly not higher than 18°F.

TEST NO. 4:

This test was made to avoid the possible errors in temperature determinations and their significance in the very small F-values assumed. In this test hot water was used and the times were large enough so that come-up time was not significant. This would also be a check on z-values assumed. It became evident that z, F, or both would have to be altered.

TEST NO. 5:

Variations in the number of microorganisms present in a container will change the F required. The 8 grams of corn in each TDT can included about 25 corn kernels. It was possible that extreme variations in the microbiological population occurred in any set of replicates. The object of this test was to create a more uniform distribution of surface microorganisms throughout the entire corn sample.

Sufficient corn was placed in a jar for the entire test. The jar was then filled with the amount of water needed for the test and the total shaken thoroughly for 5 minutes. The water was then drained off and placed in another jar. The two components, corn and water, were then added in measured amounts in the TDT cans. This procedure

evidence that toxin production is inhibited. In the present state of knowledge this latter process cannot be recommended.

It was found that corn could be readily dried after each of these processes, if desired. Corn taken from the TDT cans some weeks after a sterilizing process was readily dried and had a good appearance and odor. This was true also for pasteurized corn stored at 40°F. However, the pasteurized corn stored at 86°F retained an odor, after drying, that was unpleasant.

There are several harvesting and storage systems wherein the results of these tests may be utilized. The process time is short, far shorter than the time required to dry corn at 20 to 30% moisture. It may be preferable to process wet corn as fast as the harvest can proceed, rather than dry it. The product can then be fed in its moist state when desired, but should not get into commercial channels. If normal storage was desired the corn could be dried later at a slower and more efficient rate without fear of spoilage in the meantime.

According to the 1954 Census of Agriculture, over 60% of the corn produced in the U.S.A. did not get into commercial channels.

The corn could be pasteurized and maintained at 40°F, or lower. This type of process could be one that might economically precede a slower, more efficient drying operation; in the meantime harvest could continue at a rapid rate since pasteurizing would not take much time.

was presumed to create greater uniformity in the distribution of the microbiological population in each can. The test indicated no advantage for the attempts at uniformity, and indicated that F may be somewhat above 0.16 minutes if z was 18°.

TEST NO. 6:

The 1960 corn crop was being harvested and it was desired to compare the fresh crop with the frozen 1959 corn that had been previously used. The sample of 1960 corn in this test had been kept at 40°F for several days. The procedure used was the same as that of the previous test except that the water treatment was not repeated. The test showed that the F of 1960 corn was about the same as that of the 1959 corn.

TEST NO. 7:

Bacterial counts, but not identification, were made on the 1959 and 1960 corn samples at the Department of Microbiology and Public Health with the following results:

a. After 18 hours at room temperature, non-heat shock count:

   1959 corn 65×10^6 organisms per gram

   1960 corn 13×10^6 organisms per gram

b. From the same originally frozen sample, after 18 hours at room temperature:

   non-heat shock count (repeat) 1959 corn
   34×10^6 per gram

   heat shocked (10 min. at 100°C) 1959 corn
   16×10^6 per gram

In the magnitude of 10^6 counts the plate counts reported here may vary as much as one log cycle without much significance.

Test No. 7 was made from corn samples in the same storage bag, and processed at 232°F after 18 hours at room temperature. The results indicated little difference in F between 1959 and 1960 corn.

TEST NO. 8:

The test was conducted primarily to see if a repeat of test No. 7 could be made. The data shows that a higher F-value would have to be used. The data of this test are used as the critical point in establishing the line for the thermal death time curve in Figure 5.

From Figure 5 a z-value of 17°F and F- of 0.27 minutes was established. Figures 6, 7, and 8 show the heating curves for water and corn in the TDT cans at retort temperatures of 214°, 232°, and 250°F respectively. Figures 9, 10, and 11 are the lethal rate curves from which actual t-values were determined for the 214°, 232°, and 250°, respectively. The basis for plotting and reading these curves is explained in Appendix I.

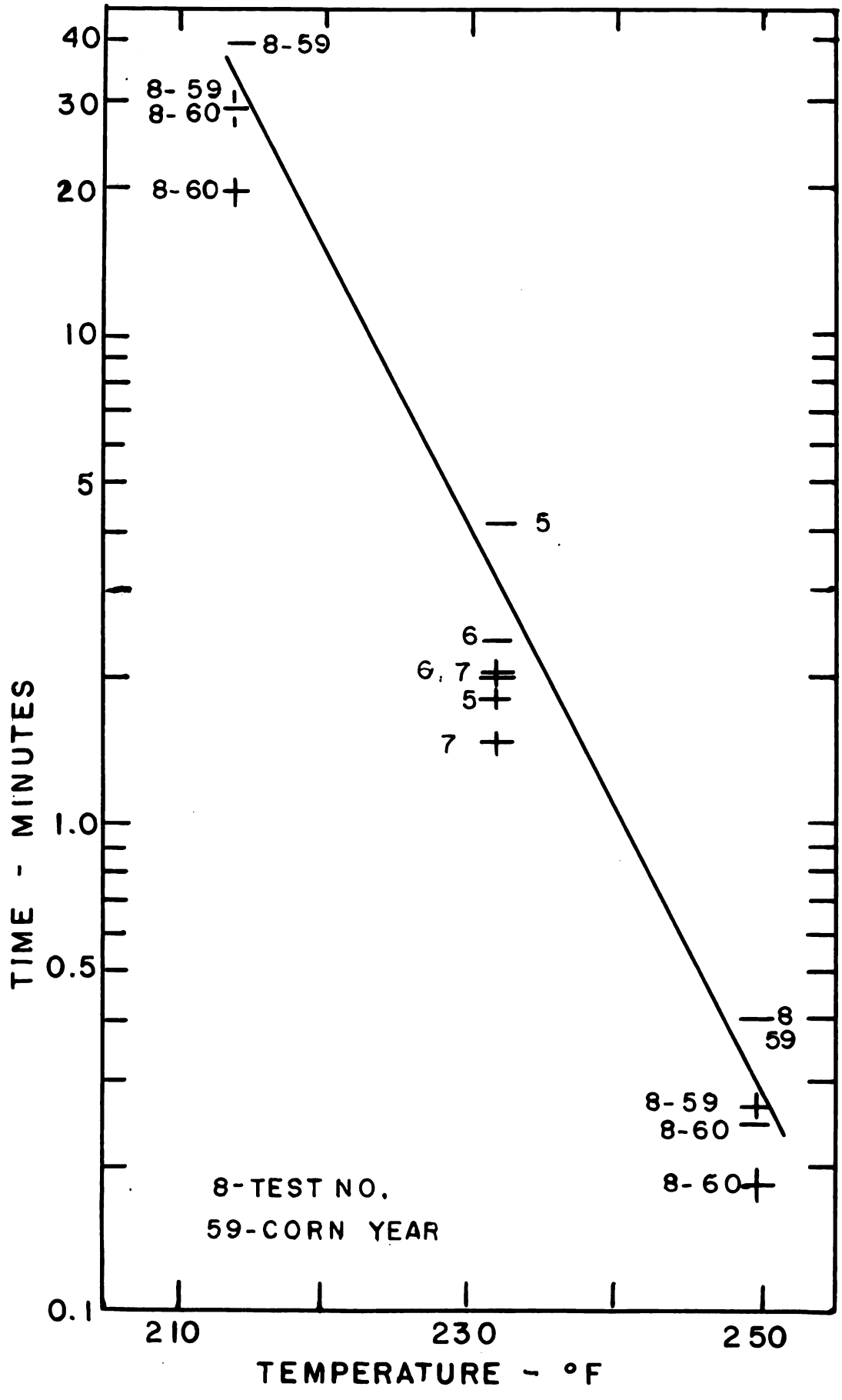

FIGURE 5 Thermal death time curve for field corn

DISCUSSION OF RESULTS

In view of the logarithmic order of death of microorganisms it is clear that variations in the populations in a can will vary the time for destruction of this population. In the usual manner of performing TDT tests a sterile medium is innoculated with a known number of knowm microorganisms. By contrast, the corn used in this test had a heterogeneous population of unknown numbers. Each type of microorganism had a population of its own, with and F-and z-value. The last organisms to be destroyed were not necessarily of the type with the highest F or the highest number, but the one with the highest combination of N_o, F, and z. It was noted that the colonies counted in the spore count all seemed to be one type of organism. It is possible that the F-and z-values shown here are due to some one organism, and to the one in the greatest numbers.

In the literature cited previously there were cases where the F-and z-values for Clostridium botulinum were similar to the F- and z-values for corn found in the tests reported here. There were also cases of much higher F-values determined for Clostridium botulinum. Townsend, et al, (1938) place a minimum F- of 2.45 minutes as absolutely necessary for anaerobic packaging and room temperature storage of products with a pH near 7.0. An F- of 4 to

5 minutes is more commonly accepted in order to be safe. This latter value should certainly be considered minimum for processing field corn.

There are microorganisms that would require a higher F-value and could be encountered in corn. Specifically, the flat-sour organism with high heat resistance such as NCA 1518. Vetter, et al, (1957) injected steam at 268°F into sweet corn directly. The corn was in No. 2 cans (307 X 409). The cans had been innoculated with 5×10^6 spores of NCA No. 1518 per can. An F_{250} of 16.4 minutes was insufficient to stop acid production while 26.1 minutes stopped all acid production. The temperature inside a corn kernel was also measured. The kernel interior reached retort temperature in about 45 seconds, while surface measurements throughout the can (4 places) all took 25 seconds to reach this temperature. The lag between surface and kernel interior temperatures is comparable to the results reported here (see Figure 8).

The tests described in this report demonstrate that high moisture field corn may be preserved by thermal processing. Two procedures for processing are demonstrated, sterilizing and pasteurizing. Both of the processes assume hermetic sealing. Pasteurizing assumes aseptic packaging. Sterilizing results in a product that will keep at room temperatures while pasteurizing will not prevent reduction in pH even at 40°F, in 2 years. The latter case it is certain that Clostridium botulinum is not destroyed, and no

Some form of pasteurizing, possibly along with innoculation with a desired organism, could be used to enhance the quality of normally ensiled shelled corn.

A mechanism for pasteurizing is not difficult to visualize. A temperature near the boiling point of water and a holding time of 2 to 4 minutes is sufficient.

The full process, 250°F for 5 minutes, followed by aseptic storage would be more difficult. The following are some of the general considerations that might apply to this problem:

1. The heat energy requirements for processing would be considerably less than those required for drying.
2. The time for processing would be far less than that for drying.
3. Some techniques of food processing may apply to feed:
    a. Wash the product before processing.
    b. Chlorinate at high temperatures before processing. It may be that feeds can be chlorinated at higher rates than foods.
    c. Inject steam directly into the corn for rapid processing.
    d. Package aseptically in large packages of 1000 pounds or more. Pallet boxes lined with plastic might be used and the packages would require no special storage structure.

It is recognized that the presence of Clostridium botulinum toxin is the major hazard in any of these processes. In this connection there is one small note in the literature that might bear further investigation. Quortrup and Holt (1941) noted that aerobic bacteria quickly preformed toxin of Clostridium botulinum. Is it possible that in the handling of animal food some use may be made of this fact, and the problem of Clostridium botulinum eliminated?

It was found that the corn used in this test would not germinate after storage at 0°F before processing, which might be expected in high moisture corn. For that reason the effect of processing on the germinative ability of corn is not known. Full thermal processing would surely destroy the germ but some of the pasteurizing processes may not.

An inspection of the pattern of spoilage in TDT cans gave rise to the following possiblity:

> The range of processes included treatments sufficient to destroy vegetative cells but not sufficient to activate spores. Longer processing activated the spores but did not destroy them.

This would explain a reduction in can swelling, followed by an increase in percent of swells before reducing to no swells. To check this hypothesis a number of TDT cans were selected because they seemed to fall in the category of insufficient heat to activate the spores. These selected cans were treated at 212°F for 10 minutes and checked for swelling. None of the cans showed any swelling after this test, indicating that the hypothesis was

probably incorrect. The history of the cans selected for this test is given in Appendix II.

CONCLUSIONS

1. Shelled field corn in the moisture range of 22% to 32% may be preserved by thermal processing sufficent to destroy Clostridium botulinum, followed by aseptic packaging and hermetic sealing. The $F_{\neg}$ and z-values determined in this work are below the minimum recommended for Clostridium botulinum and hence cannot be used until some other means of controlling the organism is developed.

2. A pasteurizing process given high moisture field corn before hermetic sealing will result in a higher pH value of the final product, compared to no process. In general, lower temperatures of storage also result in higher pH values. Since pH values are generally above 4.5, there is a hazard of the presence of Clostridium botulinum in the product. Pasteurizing may provide a desirable control over the ensiling of shelled corn, but further work will be required to establish the method.

Table 1. Experimental results after 9 months storage

Test No.	Description	Corn Moisture, % w.b.	Temp., °F.	B Min.	F_{250} Min.	Condition
1.	Pasteurized in ½-pint jars and stored at 86°F	22.6	170	2	-	X X X X X
				4		X X X X X
				8	-	X X X X X
				16	-	X X X X X
			190	1	-	X X X X X
				2	-	X X X X X
				4	-	X X X X X
				8	-	X X X X X
			210	1	-	X X X X X
				2	-	X X X X X
				4	-	X X X X X
				8	-	X X X X X
	Stored at 40°F		170	2	-	X X - - X
				4	-	- - - - -
				8	-	- - - - -
				16	-	- - - - -
			190	1	-	- - - X X
				2	-	- - - X -
				4	-	- - - - -
				8	-	- - - - -
			210	1	-	- - - - -
				2	-	- - - - -
				4	-	- - - - -
				8	-	- - - - -

X can swelled more than 0.030 inches
? can swelled from 0.010 to 0.030 inches
- can swelled less than 0.010 inches

Table 1. Experimental results after 9 months storage - cont'd

Test No.	Description	Corn Moisture, % w.b.	Temp., °F.	B Min.	F_{250} Min.	Condition
2.	Whole corn in water	32.0	250	0.16	-	- - - - -
				0.32	-	- - - - -
				0.48	-	- - - - -
			232	0.2	-	X X - - -
				0.3	-	X - ? ? -
				0.5	-	X X X X X
				1.5	0.15	- ? - - ?
			215	1.1	-	X X X X X
				1.6	-	X X X X X
				2.2	-	X X ? X X
				3.2	-	X ? ? ? X
				4.8	-	X X X X X
			197	5.0	-	X X X X X
				10.0	-	X X X ? X
				15.0	-	X ? X ? X
				22.0	-	X X X X X
				32.0	-	X ? X X X
			180	16.0	-	X X X X X
				32.0	-	X X X X X
				48.0	-	X X X X X
3.	Whole corn in water. Corn thawed 18 hours 1959 crop	32.0	250	0.05	-	X X X X X
				0.12	-	- ? X - -
				0.17	-	X X ? - -
				0.17	-	- ? - - ?
			232	1.0	-	- - X X -
				1.25	-	- X X ? -
			214	6.0	-	X X X X X
				8.0	-	X - X X X
				10.0	-	X X X X X
			197	60	-	? X ? X X
				80	-	X X X X X
				100	-	X - X - X

Table I. Experimental results after 9 months storage - cont'd

Test No.	Description	Corn Moisture, % w.b.	Temp., °F.	B Min.	F_{250} Min.	Condition
4.	Same as 3. F based on z of 18°.		196	100	-	X X X X X
				125	-	X X X X X
				150	-	X X X X X
				175	0.175	X X X - X
			209	19	-	X X X - X
				23.8	-	- X X X X
				28.6	-	X - - - -
				33.3	0.175	- - X - X
5.	Whole corn in water. 1959 crop. P-Normal process W-Washed to equalize organisms.	23.8	232	1.0	-	PX X X X X
						WX X X X X
				2.0	.018	PX X X X X
						WX X X X X
				4.0	.16	P- - X - -
						W- ? ? - -
				6.0	.36	P- - - - -
						W? - - - -
				8.0	.56	P- - - - -
						W- - - - -
				16.0	1.36	P- - - - -
						W- - - - -
				20.0	1.76	P- - - - -
						W- - - - -
				30.0	2.76	P- - - - -
						W- - - - -

Table I. Experimental results after 9 months storage - Cont'd

Test No.	Description	Corn Moisture, % w.b.	Temp., °F.	B Min.	F_{250} Mfn.	Condition
6.	Whole crop in water 1960 crop.	30.0	232	2.5	0.055	---X-X-XX-
				3.0	0.085	---------X
				3.5	0.129	--X--?--?-
				4.0	0.171	--X-----?-
				4.5	0.205	----------
				5.0	0.249	----------
				5.5	0.293	----------
				6.0	0.337	----------
7.	1959 corn	24.9	232	0.5	-	not run
				1.0	0.002	XXXXXXXXXX
				1.5	0.009	-----XXXXX
				2.0	0.027	X?X---X-XX
				2.5	0.055	------X-XX
				3.0	0.085	-X--X---XX
				3.5	0.129	-X-X-X---X
				4.0	0.171	----------
				4.5	0.205	----------
				5.0	0.249	----------
	1960 corn			0.5	-	XXXXXXXXXX
				1.0	0.002	XXXXXXXXXX
				1.5	0.009	----------
				2.0	0.027	----------
				2.5	0.055	----------
				3.0	0.085	-X--------
				3.5	0.129	------?---
				4.0	0.171	----------
				4.5	0.205	----------
				5.0	0.249	----------

Table 1. Experimental results after 9 months storage - Cont'd

Test No.	Description	Corn Moisture % w.b.	Temp., °F.	B Min.	F_{250} Min.	Condition
8	1959 corn	25.4	250	Secs.		
				45	0.015	XXXX-XXXXX
				55	0.035	-XXXXXXXXX
				65	0.082	??---?-??X
				78	0.176	??X?-XX-??
				85	0.240	----X-X-XX
				99	0.40	-------?--
				110	0.55	----------
				120	0.68	----------
	1960 corn	26.5	250	Secs.		
				45	0.015	X?XXXXXXXX
				55	0.035	XXXX?XXXX-
				65	0.082	----------
				78	0.176	--X-------
				85	0.240	----------
				99	0.40	-----?----
				110	0.55	----------
				120	0.68	----------
	1959 corn	25.4	214	Min.		
				7	0.05	X?X???X-X?
				12	0.10	??--X--??-
				17	0.15	----------
				27	0.25	-X--?-----
				37	0.35	----------
				47	0.45	----------
	1960 corn	26.5	214	7	0.05	--X-----X-
				12	0.10	?---------
				17	0.15	--------X-
				27	0.25	----------
				37	0.35	----------
				47	0.45	----------

Table 2. Evaluation of pasteurized corn tests after two years of storage.

Test no. & treatment	40°F storage Odor & appearance	lid	pH	pH	86°F storage lid	Odor & appearance
A	Sweet	N	5.1			
B 170°F	Slightly sweet	N	5.6			
F 2 Min.				4.2	N	Sweet,kernels dark
G				4.8	N	Sweet,kernels dark
A	Slightly butyric	N	5.2			
B 170°F	Slightly sweet	N	5.3			
F 4 Min.				4.7	V	Slightly,sweet,dark
G				4.6	N	Slightly,sweet,dark
A	Slightly sweet	P	5.1			
B 170°F	Very slightly sweet	P	5.4			
F 8Min.				4.6	P	Sweet,kernels dark
G				4.6	P	Sweet,kernels dark
A	Sweet	N	5.5			
B 170°F	Slightly sweet	N	5.2			
F 16 Min.				5.0	P	Sweet,kernels dark
G				4.6	P	Ensile,kernels dark
A	Sweet	N	5.3			
B 190°F	Neutral	V	5.6			
F 1 Min.				5.1	N	Sweet & ensile,dark
G				4.9	N	Ensile,sl.butyric
A	Slight acid	V	5.9			
B 190°F	Slight acid	V	5.6			
F 2 Min.				4.8	V	Sl.acid & fish,dark
G				5.2	N	Sl.ensile,some dk.
A	Ensiled	V	5.9			
B 190°F	Good ensiled odor	V	5.9			
F 4 Min.				4.7	P	Ensiled
G				4.8	P	Ensiled

N - No net pressure in jar
P - Pressure in jar
V - Vacuum in jar

Ensile - common odor of ensilage
Acid - usually acetic
Sweet - Similar to Butane or Ethanol

Table 2. Evaluation of pasteurized corn tests after two years of storage - Cont'd

Test no. & treatment	40°F storage Odor & appearance	lid	pH	pH	lid	86°F storage Odor & appearance
A	Slight corn odor	P	5.4			
C 190°F	Corn odor	V	5.8			
F 8 Min.				5.0	P	Ensiled
H				4.9	P	Ensiled
A	No odor	V	5.8			
B 210°F	Slight corn odor	V	6.0			
F 1 Min.				5.0	P	Slight acid
G				4.6	P	Acid,sl.butyric
A	Slight corn odor	V	5.9			
B 210°F	Good corn odor	V	5.4			
F 2 Min.				5.3	N	Top moldy,(opened)
G				6.1	V	Cooked corn odor
C	Slight corn odor	N	5.1			
D 210°F	Slight acid	?	5.9			
F 4 Min.				5.1	P	Strong corn &ensile
G				5.0	N	Strong corn &ensile
B	No odor	V	6.2			
C 210°F	No odor	V	6.1			
F 8 Min.				4.8	P	Cooked corn
G				5.3	P	Cooked corn
A	Good ensile sl. dk.	P	5.0			
B Control	Good ensile sl. dk.	P	4.9			
I				4.3	V	Strong acid, dark
J				4.3	N	Acid,all kernels dark

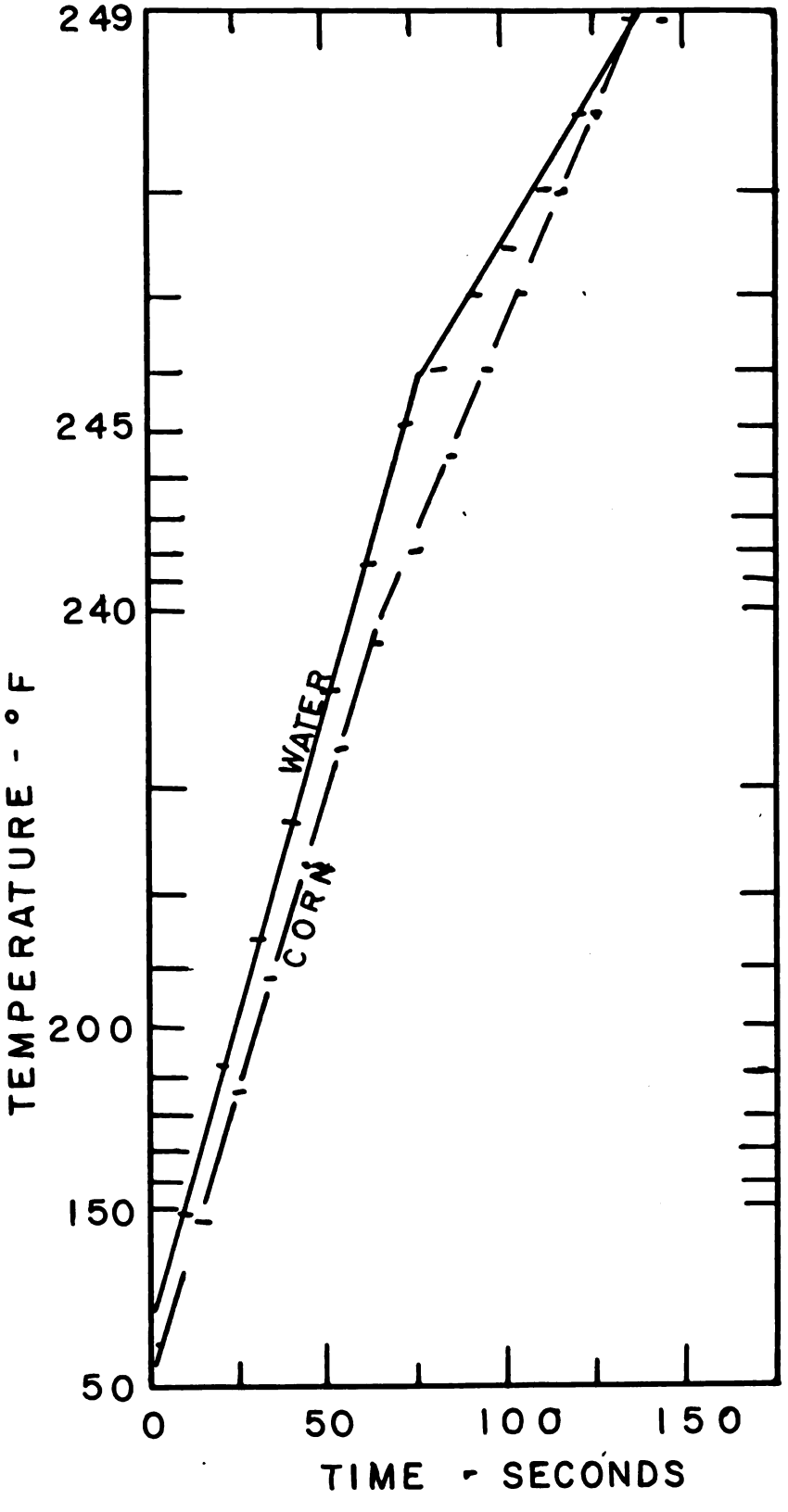

FIGURE 6 Heating curve for thermal death time cans. Retort temperature - 250°F

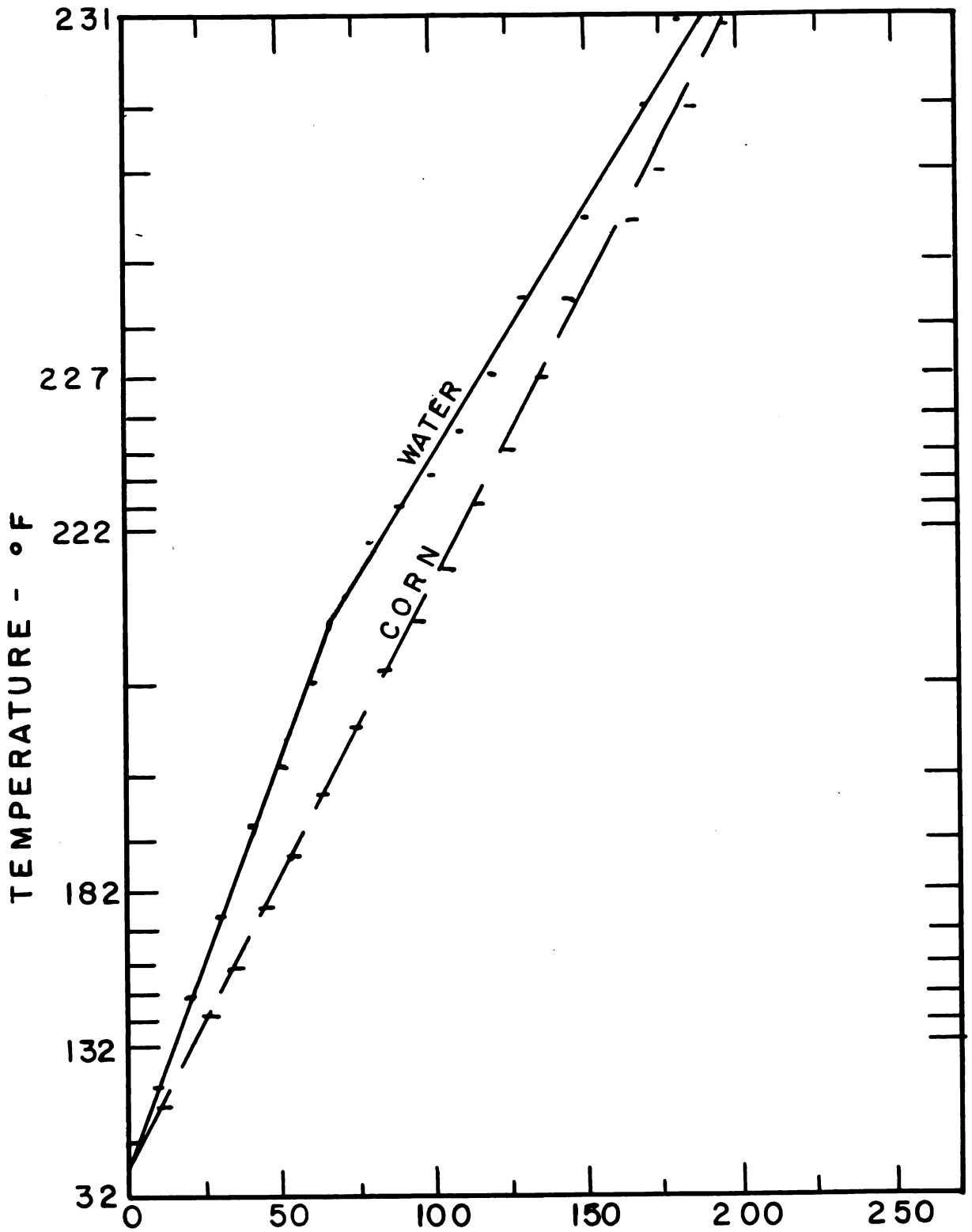

FIGURE 7 Heating curve for thermal death time cans. Retort temperature 232°F.

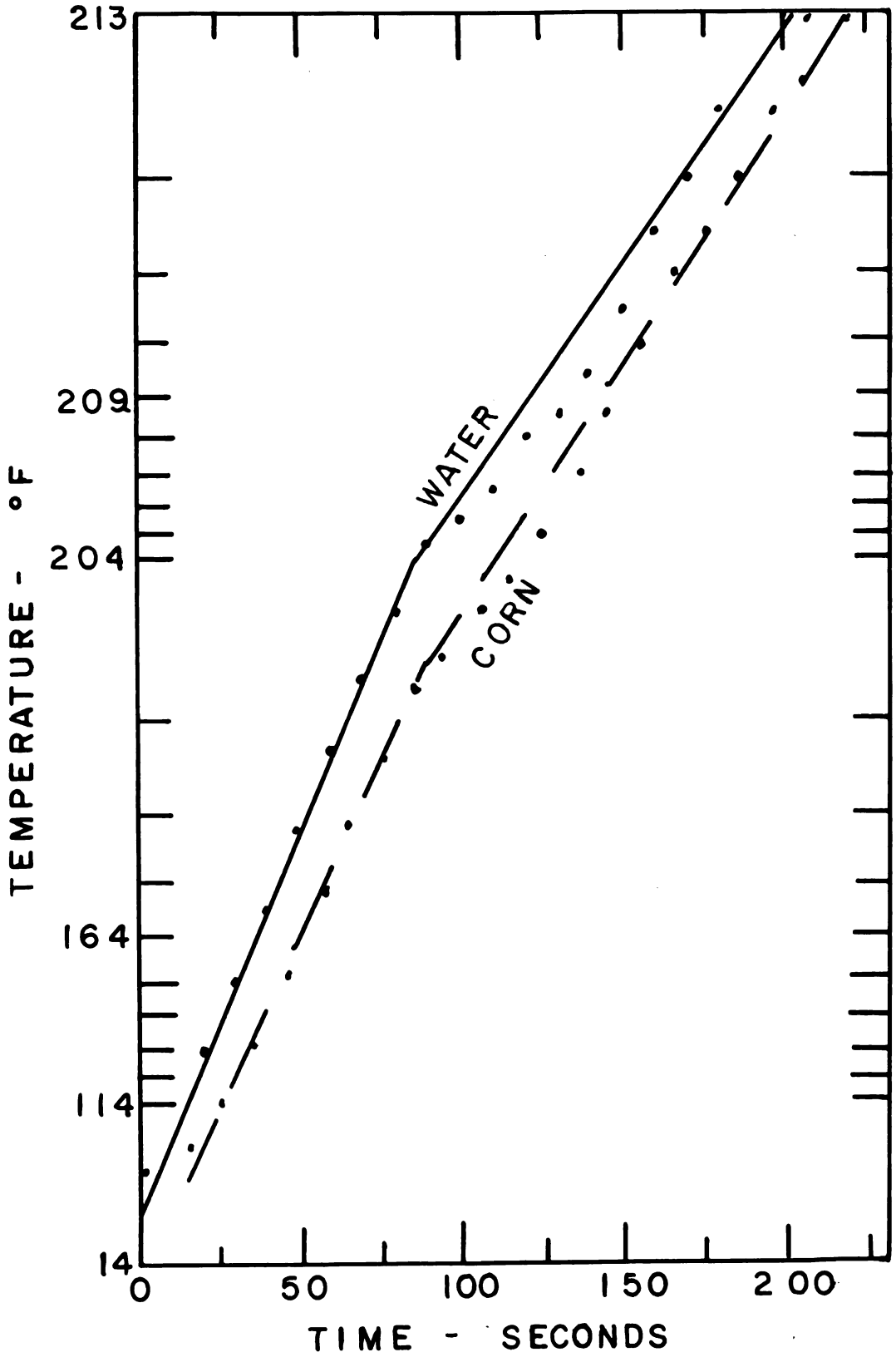

FIGURE 8 Heating curve for thermal death time cans. Retort temperature 214°F.

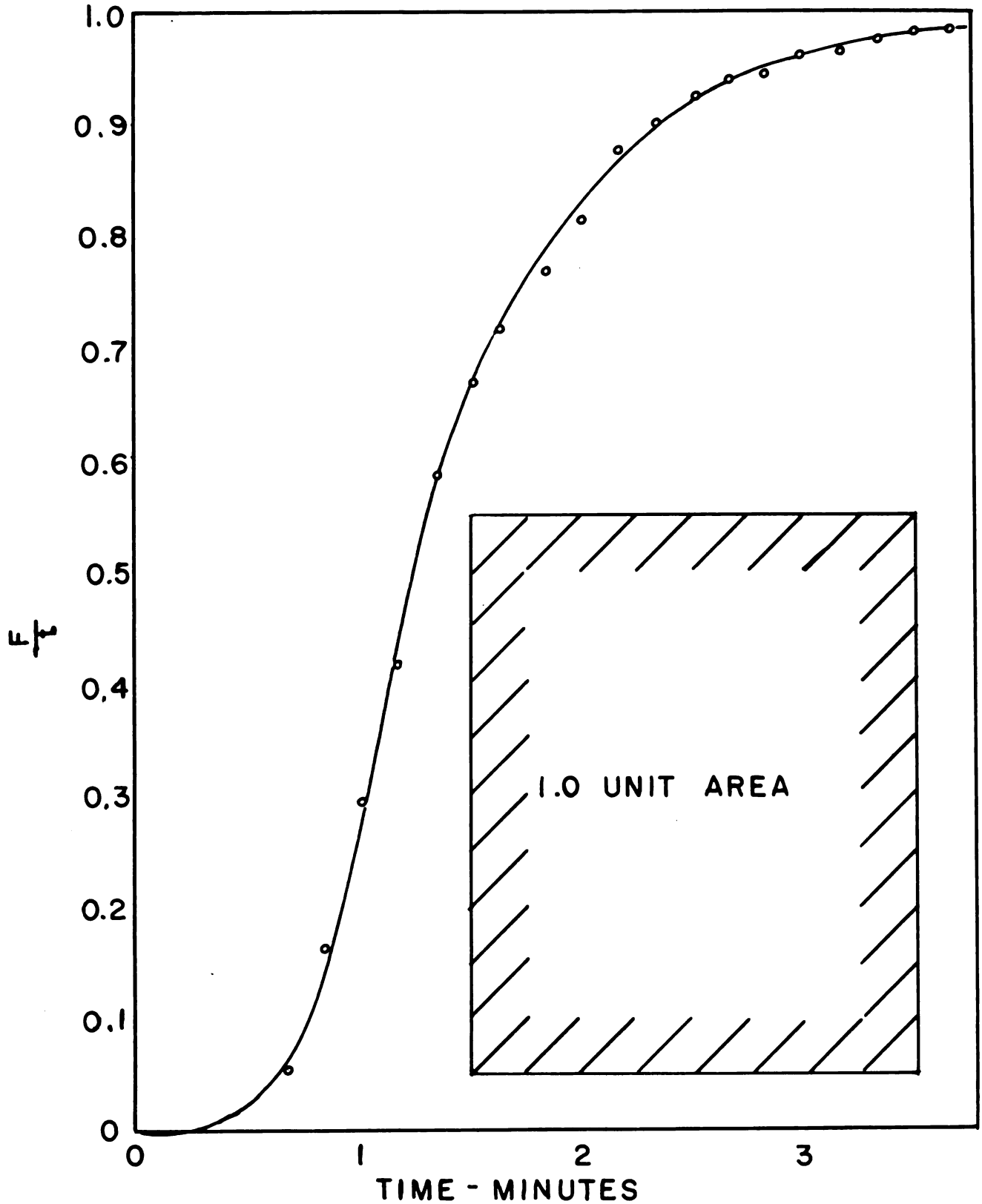

FIGURE 9 Lethal rate curve for 250°F retort temperature. Unit area is 1 minute at 250°F.

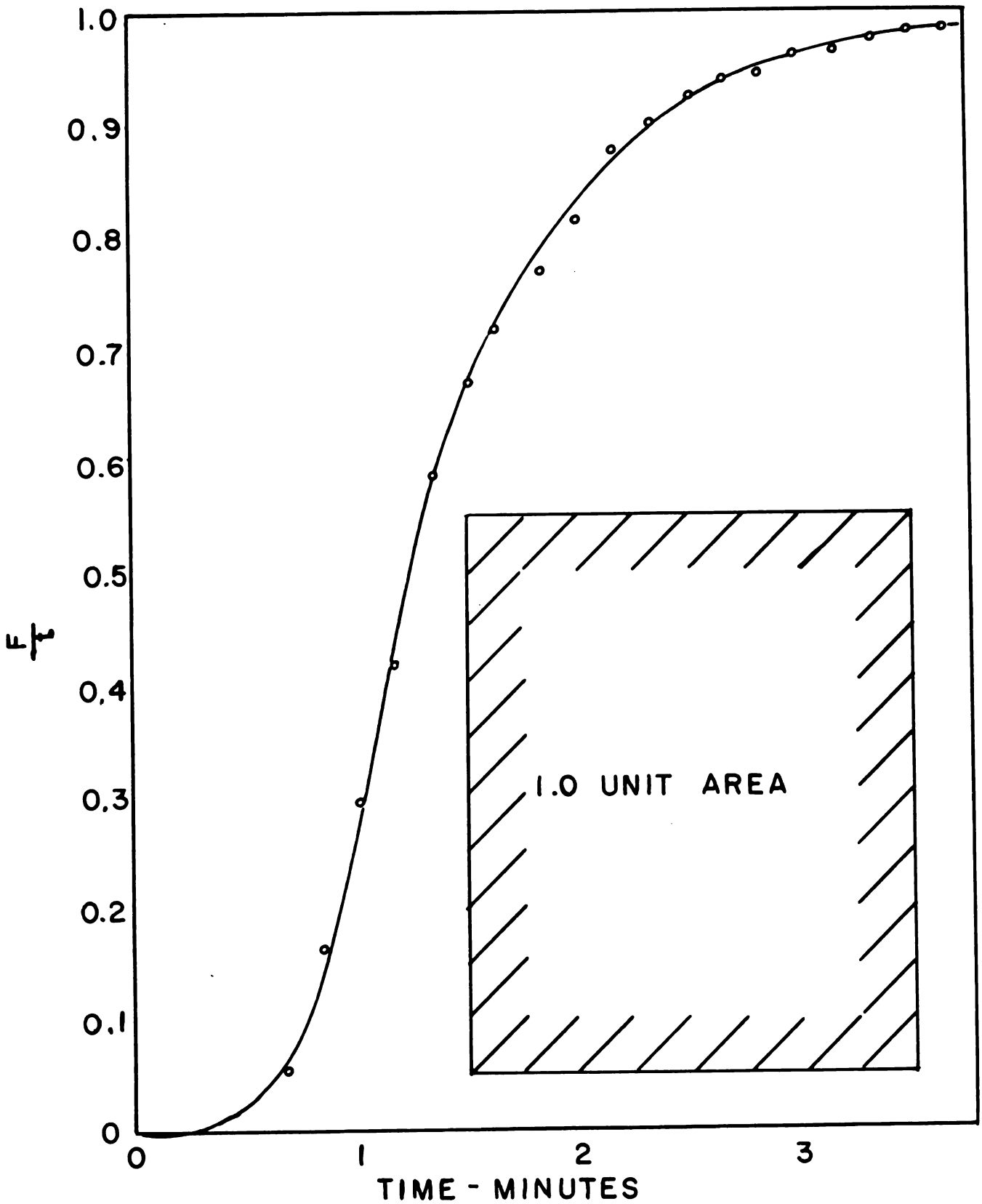

FIGURE 9 Lethal rate curve for 250°F retort temperature. Unit area is 1 minute at 250°F.

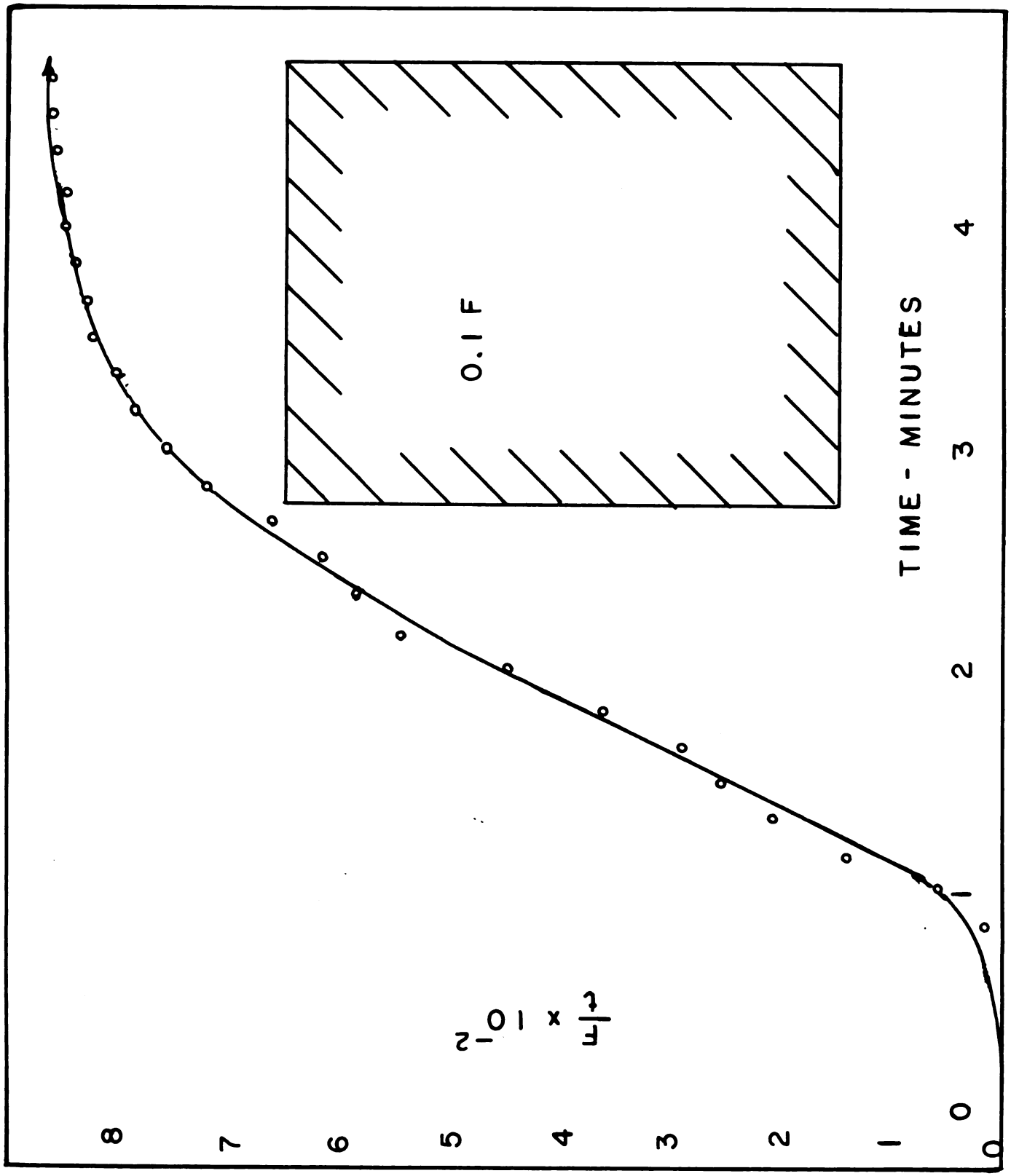

FIGURE 10 Lethal rate curve for retort temperature of 232°F. Unit area is 0.1 minutes at 232°F.

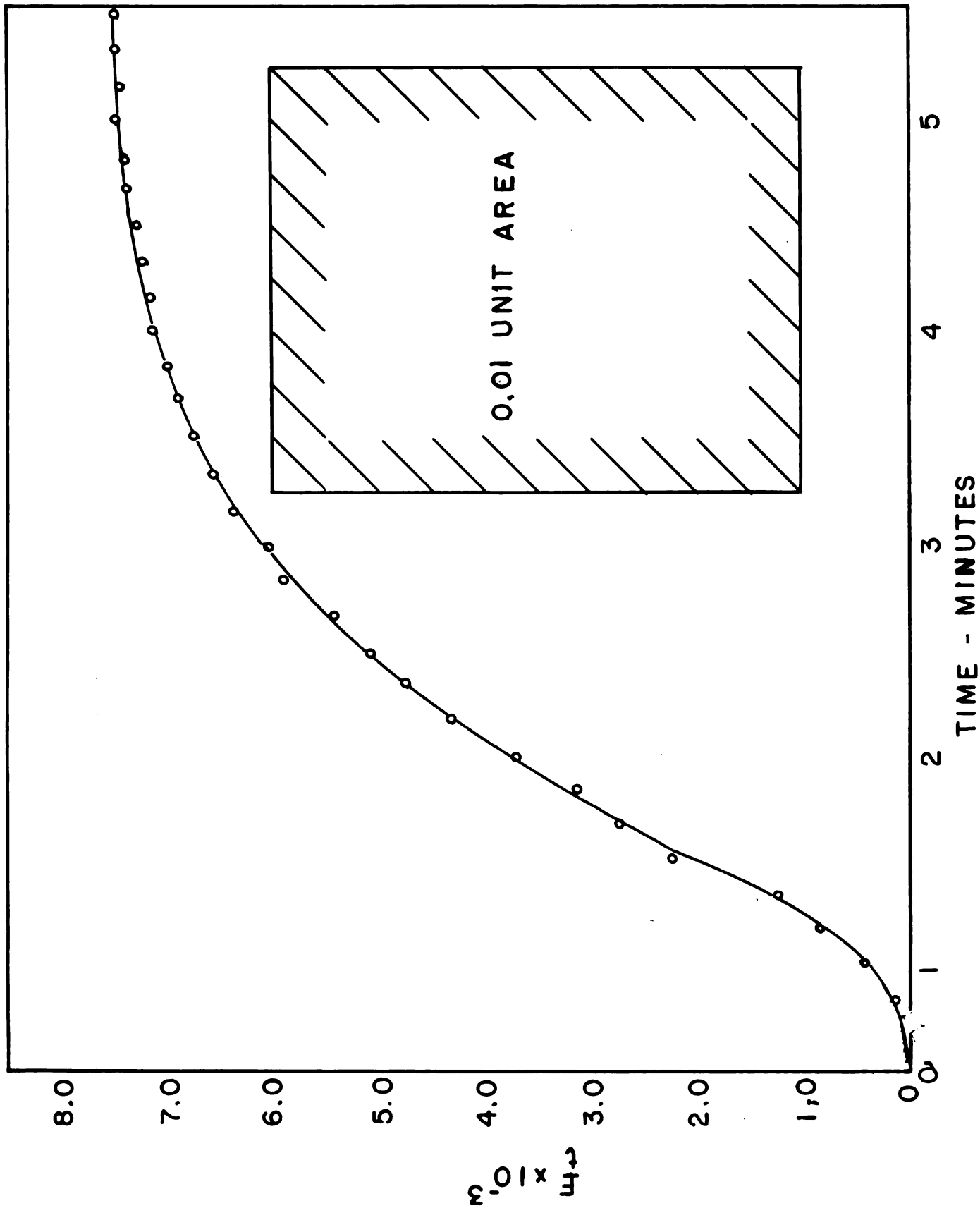

FIGURE 11 Lethal rate curve for retort temperature of 214°F. Unit area is 0.01 minutes at 214°F.

SUGGESTIONS FOR FUTURE WORK

1. A measure of feeding value should be made along with any future work in thermal processing.

2. The value of pasteurizing corn before ensiling could be investigated as a possible aid to ensiling the product. This investigation could include: (1) the possibility of innoculation of the corn after ensiling to enhance proper biological action; (2) the process of adding an acid to create surface pH desired; (3) the possible aerobic destruction of the toxin of Clostridium botulinum if conditions would permit its formation.

3. A study of mechanizing thermal processing of corn, including aseptic packaging, if the process itself shows promise.

4. An investigation of processing could be made, including heat and particle irradiation.

5. A study of the thermal properties of corn is needed.

6. Investigations are being made of chemical means of preserving corn, and may provide a method of storing high moisture corn.

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APPENDIX I

Method of calculations

For determination of lethality at different temperatures reference is made to Ball (1957).

A. When processed in hot water:

Most of the process times were large so that come-up time was not considered significant since cooling time was also not counted. Thus the B-value was also the F_T value reported. The total immersion time was used in the equation:

$$F_{250}, \text{ Equivalent time at } 250°F = {}^{F}{}_{T} \cdot 10^{\frac{T-250}{z}} \quad (3)$$

Where T is the temperature of the water and z was orginally 18°F but later corrected for 17°F.

B. When processed by steam:

A trial run was made at the desired steam temperature using a TDT can containing a thermocouple connected to a recording potentiometer. A table of equivalent times at 250°F was made for temperatures above 200°F and for times of 5 second duration, which was the cycling time of the temperature recorder. From each recorded temperature the equivalent time at 250° was

determined, using equation (3) above. A z-value is required for this purpose. In the original work a z of 18° was assumed. When a z of 17° was finally established the values used were corrected. In this manner it was possible to determine the actual retort time required to give the interior of a TDT can any desired process based on time at 250°F.

C. For lethal rate values:

The heating curves, Figures 6, 7, and 8 were first determined. The method of getting these data is described in Appendix II. Tables were made up from the heating curves listing the temperatures at each 10 seconds of time. A calibration of the recording potentiometer indicated that approximately $\frac{1}{2}$ degree should be added to all temperatures below 225 degrees F and 1 degree to all temperatures above this value. These corrected temperatures were used in the final plotting of the heating curves.

Referring to equation (3), let 'Equivalent time at 250°' be equal to F. Then $\frac{F}{t}$ is the ratio:

$$\frac{F_{250}}{F_T} = \frac{\text{Equivalent time at } 250°}{\text{Time at temperature T}} = 10^{\frac{T - 250}{z}}$$

The value of $\frac{F}{t}$ was calculated from the heating curves for each 10 seconds. These data are shown on Tables

3, 4, and 5. The ratio $\frac{F}{t}$ was plotted on the ordinate of rectangular coordinates against t on the abscissa, where t= ^{F}T. This was done in Figures 9, 10, and 11. The area under these curves at any time is : $\frac{F}{t}$ ^{X} t = F, or equivalent time at 250°F. The unit areas blocked out in each of the above figures represents the labeled number of minutes at 250° equivalent. A planimeter can be used to measure the area under the curve for any heating time t, and the equivalent number of minutes at 250° immediately determined. The F-values shown in Table I, and plotted in the thermal death time curve, Figure 5, were determined in this manner.

It should be noted that the F used in the explanation above is a simplification (in print) of F_{250} as defined in this report. Also, t is used for ^{F}T for the same reason. This simplification was made to conform with the explanation by Ball (1957).

APPENDIX II

TEST NO. 1. 1959 corn at 22.6% moisture w.b.

This test involved processing corn at temperatures below 212°F by direct immersion into hot water. The bulk corn, after immersion, was aseptically loaded into half-pint jars and hermetically sealed. The process times were short enough so that this process may be called pasteurizing.

A cage of 1/8 inch hardware cloth was made with a volume slightly more than 5 half-pint jars. (Figure 2, No.1). The bottom of the cage was fitted with a funnel to fit over the jars, along with two sliding valves. (Figure 2, No.3). The valves permitted selection of approximately one half pint from the bulk in the cage and trapping it between the two valves. This selected quantity was then dropped into a jar. The valves were operated from rods extending along the handle of the screened cage. (Figure 2, No. 2).

The entire loaded cage was immersed in the water in a retort that was maintained at the desired temperature. Since the retort contained over 20 gallons of water there was no significant change in the temperature of the water when the charge was submerged. The cage was vigorously moved around during the immersion period.

To approach aseptic conditions during filling of the jars a sheet steel extension of the retort was made,

extending vertically up from the retort, and fitted with a removable lid. (See Figure 3). A slot in the wall of this extension permitted the passage of the handle of the screened cage. A sheet steel palette was made to fit inside the extension, covering less than half of the surface area of the retort. This palette held the five jars.

The removable lid of the extension permitted introduction of the palette and screened cage into the retort extension, with the handle of the cage fitting out through the slot. A glass in the lid, along with a light above the glass, provided visibility through the slot into the extension for filling the jars. A steam line and valve permitted the introduction of live steam into the extension during processing.

The procedure began by autoclaving 5 jars and lids at 250°F for 15 minutes. The lids were placed askew on the jars during this process. When processed, the lids were placed over the jars by means of electromagnetic wand, touching only the top of the lids. The jars were then located in the palette and allplaced in the retort extension.

The required amount of corn was weighed into the screened cage and immersed in the water of the retort for the desired time. While immersed, the cage was continuously stirred, and the steam was turned on in the extension. At the proper time the cage was pulled out of the water, the steam turned off in the extension, and loading of the jars accomplished. The loading operation took about 1 minute.

The electromagnetic wand was used to remove and replace the jar lids. All manipulation of the wand and the cage was accomplished from the handles of each projecting through the slot of the extension.

When all 5 jars were loaded and covered the entire paletter with the jars was quickly removed from the extension and placed in water at 60°F. The jars remained in the water for at least one hour.

The times and temperatures used are listed in the main body of this report. A repeat of this entire operation was made using thermocouples to determine corn surface temperatures. It was found that regardless of the treatment temperature, the corn temperatures were in the area of 150°F by the time the jars were all filled. After being placed in the cold water, cooling in the center of the jars proceeded at about 3 to 5 degrees per minute, at least down to 100°F. However, the outside kernels in the jar received the least process and cooled quite rapidly.

TEST NO. 2, 1959 corn, 26.2% moisture.

Ten grams of corn and 10 cc. water were used per can. Miniature retorts were used in temperatures above 212°F (Figure 4) and a hot water retort in temperatures below 212°F.

A thermocouple in a TDT can with water only was used to determine corn surface temperatures. The thermocouple was first checked in distilled ice water, using a Brown

Electronik recording potentiometer (A.E. 668). The times shown in Table I are based on equivalent time at the temperature shown, with correction for a 17°F as noted previously.

In all cases where the miniature retorts were used the temperature of the steam was determined from a mercury thermometer in the steam supply tank. The well of the thermometer was vented slightly. The automatic control was adjusted to maintain this steam pressure (related to the temperature desired) and permitted variations of $\frac{1}{2}$°F or less.

The retorts were vented for 10 seconds after the steam was first turned on, and then a very slow but continuous venting held.

TEST NO. 3, 1959 corn, 32% moisture.

An instrumented TDT can was used, after checking the thermocouple at 0°F. Calculations again were based on z of 18°, and later corrected.

The corn in this test had been at room temperature for 18 hours prior to the test. In previous tests the corn was allowed to thaw at room temperature for about 3 hours. Ten grams each of corn and water, were used per can.

TEST NO. 4, 1949 corn, 32% moisture.

Corn remaining from the package used for test No. 3 above was placed at 0°F after the test. Five days later the same corn was thawed for $3\frac{1}{2}$ hours at room temperature and

used in this test.

Eight grams of corn and 12 grams of water per can were used in this test. An extra 5 replicates at 196°F for 100 minutes was made using 10 grams of corn and 10 cc. of water. All cans swelled, but the time for swelling was greater in the case of the 10 grams of water.

TEST NO. 5, 1959 corn, 23.8% moisture.

Eight grams of corn and 12 grams of water were placed in each TDT can. Ten cans, including 5 replicates of each of two conditions were processed at one time. Treatment labeled P (in Table I) was treated normally; treatment labeled W was shaken in water as described under the Procedure. The cans were processed in one stack, 10 high, alternating replicates. It should be noted here that the method of stacking in the retort and the method or reporting are related. In Table I the first can reported (reading from left to right) is the can that was on top. The last can was on the bottom. This is the only test (no.5) where alternate stacking occurred.

TEST NO. 6, 1960 corn kept at 40°F for 5 days, 30% moisture.

Eight grams of corn and 10 grams of water were used in each can. An instrumented can was used following the test to determine the F value of the treatment in the method previously described. In all cases the 10 replicates were stacked in the retort as described above.

TEST NO. 7, 1959 corn at 24.9% moisture and 1960 corn at 25.4% moisture.

Eight grams of corn and 10 cc. of water were used in each can. The ten replicates each of 1959 and 1960 corn were placed in two parallel stacks in the retort at the same time. Again, replicates ran from A to J in top to bottom order. The F values were based on a subsequent run with an instrumented TDT can.

Both 1959 and 1960 corn had been kept at 0°F. A period of 18 hours at room temperature was allowed before processing.

As an additional factor in this test, 1959 and 1960 corn samples sufficient for 10 replicates each were soaked in water with 200 ppm chlorine at approximately 70°F. The water still gave indication of approximately 200 ppm chlorine after the 3 minutes allowed for soaking. The corn was then washed twice before filling the TDT cans. Water from the second washing showed 25 ppm chlorine residual. This ppm was determined by indicating paper. The initial 200 ppm was based on 2 cc of Roman Cleanser to 500 cc water. Process time for the chlorine treated corn was at least as great as that for the untreated corn in this single test.

The results of the chlorine treated corn are not shown in the results in Table I, primarily because the F-value of these replicates was uniformly greater than the normally treated. Since these results are distinctly contrary to the literature they either represent an error or

the entire procedure should be more carefully investigated. The investigation obviously required is not within the scope of the work at this time.

TEST NO. 8, 1959 corn at 25.4% moisture and 1960 corn at 26.5% moisture.

Test No. 7 provided data sufficient to assume an F-value in a range that indicated a z-value when related to previous data. This test was conducted to see if the results could be repeated, and to use points (250° and 214°) such that z-value would be verified in the same test that an F was established.

The procedure in this test was the same as that for No. 7, except that no further work was done with chlorine solutions. From Table I it will be seen that the results indicated a higher F than that of previous tests and a slightly different z (17°). This was the last test made.

Method of determining temperatures in TDT cans.

The methods described in the individual tests above involved the use of a single thermocouple located in the water in the can to determine the surface temperature history of the corn. The method described here was used to get both surface and corn interior temperatures. The heating curves, Figures 6, 7, and 8 are based on the method described below.

Two copper-Constantan thermocouples, 30 gauge wire, lacquer coated and fiber-glass covered were used in a TDT

can. The entrance to the can was made in a 1/16 inch hole at the bottom bend of the can. The fiber-glas insulation was stripped off the wires from the point of entrance to the can on to the thermocouple itself. The entrance was sealed with Epocast 10F epoxy resin, about 2 cc. The can was filled with 6 grams of corn and sufficient water to reach the same level as the water in the normally filled cans.

One of the two thermocouples was passed through a short piece of rubber tube, so that the contact area was miantained in the water. The other thermocouple was inserted in the center of a corn kernel and sealed with DuPont Duco cement. The leads of this couple were arranged to lie above the water in the can, while the leads to the couple in the water passed through the water. The stripped portion of the wires in the can were painted with resin to reduce the possibility of shorts.

A twelve couple recording potentiometer (A.E. 2114) was used with the thermocouples. The leads were alternated so that all odd numbers recorded water temperatures and even numbers recorded kernel interior temperatures.

The instrumented can was first checked at 32°F (distilled ice water) with a Leeds Northrup potentiometer (A.E. 2036X). Later the recording potentiometer was checked against the Leeds and Northrup potentiometer. Corrections were made to the data to account for the difference noted. For example, below 225°F, $\frac{1}{2}$° was added to each temperature noted, while above 225°F a 1° increase was given in each

temperature noted.

Description of the corn used in the tests:

The corn used in these tests, 1959 and 1960, came from experimental hybrid plots of Michigan State University Agronomy Department. The plots were harvested with a field picker-sheller. The corn used was actually a mixture of an unknown number of hybrids. A field mixed sample of about 2 bushels was packaged in on-quart polyethylene bags and sealed. The bagging operation took place at room temperature and lasted about 2 hours. Following bagging the corn was stored in a 0°F freezer. An exception was made in test No. 7 where fresh corn from 1960 was kept at 40°F for 5 days before being used for a test.

In the discussion it was noted that some TDT cans were later selected for further heat processing. A description of these cans and their process history is given below:

Test No.	Original treatment °F	Original time of treatment	Replicate
2	250	10 sec.	D
2	250	10 sec.	E
2	232	12 sec.	D
2	232	18 sec.	D
2	232	1 min.	D
2	232	1 min.	E
2	215	3.2 min.	C

Test No.	Original treatment °F	Original time of treatment		Replicate
2	197	15 min.		A
2	197	15 min.		B
3	250	4.8 sec.		D
3	232	48 sec.		A
3	232	48 sec.		B
3	214	8 min.		C
3	197	60 min.		C
3	197	100 min.		D
4	209	19 min.		D
6	232	2.5 min.		A
6	232	2.5 min.		B
7	232	1.5 min.		I
7	232	1.5 min.		J
7	232	1.5 min.		C
7	232	1.5 min.		D
7	232	2.0 min.		A
7	232	2.0 min.	1959 & 1960	D
7	232	2.0 min.		E
8	250	65 sec.	1959 & 1960	A
8	250	65 sec.	1959 & 1960	B

TABLE 3

Data for lethal rate curve - 250°F

Time Sec.	Time Min.	Temp. °F	250 - T	$\frac{250 - T}{z}$	$\frac{t}{F}$	$\frac{F}{t}$
0	0.0	70.	171.5	10.1	-	-
10	0.167	149.5	100.5	5.9	-	-
20	0.333	194.5	55.5	3.27	1863.	0.000538
30	0.50	215.	35.0	2.06	115.	0.0087
40	0.667	228.	22.0	1.295	19.72	0.0508
50	0.833	236.5	13.5	0.795	6.237	0.161
60	1.00	241	9.0	0.529	3.381	0.296
70	1.167	243.5	6.5	0.382	2.41	0.415
80	1.333	246	4.0	0.235	1.718	0.582
90	1.50	247	3.0	0.1765	1.502	0.667
100	1.667	247.5	2.5	0.147	1.403	0.714
110	1.833	248	2.0	0.1175	1.311	0.763
120	2.00	248.5	1.5	0.0882	1.227	0.815
130	2.167	249	1.0	0.0588	1.145	0.875
140	2.333	249.2	0.8	0.0471	1.115	0.900
150	2.50	249.4	0.6	0.0353	1.085	0.921
160	2.667	249.5	0.5	0.0294	1.070	0.935
170	2.833	249.6	0.4	0.0235	1.056	0.943
180	3.00	249.7	0.3	0.0177	1.042	0.960
190	3.167	249.75	0.25	0.0147	1.035	0.966
200	3.333	249.82	0.18	0.0106	1.025	0.975
210	3.50	249.87	0.13	0.0077	1.018	0.982
220	3.667	249.9	0.10	0.0059	1.014	0.987

TABLE 4

Data for lethal rate curve - 232°F

Time Sec.	Time Min.	Temp. °F	250 - T	$\frac{250 - T}{z}$	$\frac{t}{F}$	$\frac{F}{t}$
40	0.667	194	56	3.30	1996.	0.000502
50	0.833	203	47	2.76	575.5	0.00174
60	1.00	212	38	2.23	169.9	0.00588
70	1.167	218.5	31.5	1.85	70.80	0.0141
80	1.333	221.5	28.5	1.68	47.87	0.0208
90	1.50	223.	27.	1.59	38.91	0.0257
100	1.667	224.	26.	1.53	33.89	0.0294
110	1.833	225.5	24.5	1.44	27.55	0.0363
120	2.00	227.	23.	1.35	22.39	0.0446
130	2.167	228.5	21.5	1.26	18.20	0.0549
140	2.333	229.	21.	1.23	16.99	0.0588
150	2.50	229.5	20.5	1.21	16.22	0.0617
160	2.667	230.	20.	1.18	15.14	0.0662
170	2.833	230.5	19.5	1.15	14.13	0.0709
180	3.00	231.	19.	1.12	13.19	0.0758
190	3.167	231.2	18.8	1.105	12.74	0.0787
200	3.333	231.35	18.65	1.095	12.45	0.0803
210	3.50	231.5	18.5	1.087	12.22	0.0820
220	3.667	231.6	18.4	1.083	12.11	0.0825
230	3.833	231.67	18.33	1.077	11.94	0.0840
240	4.00	231.74	18.26	1.073	11.83	0.0845
250	4.167	231.79	18.21	1.071	11.78	0.0849
260	4.333	231.83	18.17	1.068	11.70	0.0855
270	4.50	231.87	18.13	1.066	11.64	0.0859
280	4.667	231.90	18.10	1.064	11.59	0.0863
290	4.833	231.92	18.08	1.063	11.53	0.0867
300	5.00	231.935	18.065	1.062	11.51	0.0869

TABLE 4

Data for lethal rate curve - [illegible]

Time Sec.	Time Min.	Temp. °F	250 - T	[illegible]
40	0.667	194	56	[illegible]
50	0.833	203	47	[illegible]
60	1.00	212	38	[illegible]
70	1.167	218.5	31.5	[illegible]
80	1.333	221.5	28.5	[illegible]
90	1.50	223.	27.	[illegible]
100	1.667	224.	26.	[illegible]
110	1.833	225.5	24.5	[illegible]
120	2.00	227.	23.	[illegible]
130	2.167	228.5	21.5	[illegible]
140	2.333	229.	21.	[illegible]
150	2.50	229.5	20.5	[illegible]
160	2.667	230.	20.	[illegible]
170	2.833	230.5	19.5	[illegible]
180	3.00	231.	19.	[illegible]
190	3.167	231.2	18.8	
200	3.333	231.35	18.65	
210	3.50	231.5	18.5	
220	3.667	231.6	18.4	
230	3.833	231.67	18.33	
240	4.00	231.74	18.26	
250	4.167	231.79	18.21	
260	4.333	231.83	18.17	
270	4.50	231.87	18.13	
280	4.667	231.90	18.10	
290	4.833	231.92	18.08	
300	5.00	231.935	18.06	

TABLE 5

Data for lethal rate curve - 214°F.

Time Sec.	Time Min.	Temp. °F.	250 - T	$\frac{250 - T}{z}$	$\frac{t}{F}$	$\frac{F}{t}$
30	0.50	154.5	95.5	5.62	416900	0.0024
40	0.667	170.	80.0	4.71	51200	0.0195
50	0.833	182.5	67.5	3.97	9333	0.1072
60	1.00	191.5	58.5	3.44	2754	0.3630
70	1.167	197.5	52.5	3.09	1230	0.8130
80	1.333	201.5	48.5	2.85	780	1.282
90	1.50	205.	45.0	2.64	436.5	2.290
100	1.667	206.5	43.5	2.56	363.1	2.754
110	1.833	207.5	42.5	2.50	316.2	3.163
120	2.00	208.8	41.2	2.43	269.2	3.714
130	2.167	209.8	40.2	2.365	231.7	4.323
140	2.333	210.5	39.5	2.33	213.8	4.689
150	2.50	211	39.0	2.295	197.2	5.071
160	2.667	211.5	38.5	2.265	184.1	5.422
170	2.833	212.0	38.0	2.23	169.8	5.893
180	3.00	212.35	37.65	2.215	164.1	6.093
190	3.167	212.6	37.40	2.195	156.7	6.382
200	3.333	212.85	37.15	2.180	152.4	6.561
210	3.50	213.05	36.95	2.170	147.9	6.780
220	3.667	213.25	36.75	2.160	144.5	6.920
230	3.833	213.35	36.65	2.150	141.3	7.077
240	4.00	213.47	36.53	2.145	139.6	7.173
250	4.167	213.57	36.43	2.143	139.0	7.194
260	4.333	213.64	36.36	2.138	137.4	7.267
270	4.50	213.70	36.30	2.135	136.5	7.325
280	4.667	213.75	36.25	2.132	135.5	7.379
290	4.833	213.80	36.20	2.130	134.9	7.412
300	5.00	213.83	36.17	2.127	134.0	7.463
310	5.167	213.86	36.14	2.126	133.7	7.479
320	5.333	213.88	36.12	2.125	133.4	7.496
330	5.50	213.9	36.10	2.123	132.7	7.558