TECHNICAL REPORT
73-56-PR

THE COMPRESSION OF FREEZE-DRIED BEEF TO FORM BARS; PLASTICIZING WITH WATER TRANSFERRED AS A VAPOR

by

Malcolm N Pilsworth Jr

and

Harold J Hoge

June 1973

UNITED STATES ARMY
NATICK LABORATORIES
Natick, Massachusetts 01760

US ARMY MATERIEL COMMAND

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Project Reference:
1J061102A71C-09

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Pioneering Research Laboratory
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FOREWORD

The first formal report to issue from the cooperative program between the Food Laboratory and the Pioneering Research Laboratory was TR-73-12-PR. The present report is the second. It deals with techniques for making compressed bars from freeze-dried beef. The U. S. Army Natick Laboratories have developed a number of satisfactory food bars. These include fruits, vegetables, and some meat products. The results now being presented represent a step toward adding freeze-dried beef to the acceptable list.

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13 ABSTRACT

The plasticizing of freeze-dried beef by water addition and the compression of the beef to form rehydratable bars have been accomplished using two different sets of equipment. Water addition has been accomplished by transfer of water vapor to the meat in an evacuated system. The water added should be about 12% of the weight of the dry meat. It can be transferred in a period of 3 to 5 hours. Fat weakens the bars and when high-fat beef is used it may be desirable to use a binder to give the bars sufficient mechanical strength. A forming pressure of 3000 psi is suitable for some lots of beef. High forming pressures should be avoided since they generally give bars that reconstitute (rehydrate) poorly.

Some useful basic information is given on adsorption and desorption, on the temperatures reached as these processes occur rapidly, and on the vapor pressure and heat of vaporization of the ice and associated volatile constituents of freeze-dried raw beef.

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FOOD COMPRESSION	8		7			
PLASTICIZING	8					
FREEZE DRYING	10					
FREEZE-DRIED FOODS	9		7			
FOOD BARS	4		7			
BEEF	9		7			
DEHYDRATION	10					
WATER CONTENT	10					
MOISTURE CONTENT	10					
REHYDRATION	4					
MILITARY FEEDING	4					
LYOPHILIZATION			6			
COLLOIDING			6			
EVACUATING SYSTEM			10			

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The Compression of Freeze-Dried Beef to Form Bars; Plasticizing with Water Transferred as a Vapor

1. Introduction.

A few freeze-dried foods have made a place for themselves in the general market. A larger number are accepted as specialty foods where their reduced weight is an advantage. For some years the Armed Forces have been interested both in reducing weight by freeze-drying and in reducing bulk by compression, and a considerable body of information on the processes has been built up. Most foods will crumble if compressed in the freeze-dried state; they must be plasticized, normally by the addition of a small amount of water, if the identity of individual pieces is to be retained and especially if the food is to be formed into bars.

Some food bars, mainly fruits, are intended to be eaten in the compressed state. Many bars, however, are intended to be reconstituted by placing them in hot or cold water, where they expand nearly to the volume they occupied before compression and compare favorably with frozen or even fresh food in acceptability. Some bars, known as dual-purpose bars, can either be eaten in the compressed state or can be reconstituted, as desired. Reconstituted bars are unacceptable unless they will absorb a sufficient amount of water in a reasonable time when the food is prepared for eating.

A sustantial amount of work has been done to develop satisfactory dense foods (compressed or molded) in bar, disk, or cube form. The majority of this work has been performed or sponsored by the Natick Laboratories or by the now merged Food and Container Institute of the Armed Forces. Among the most satisfactory food bars developed so far are peas, spinach, green beans, onion, chili con carne, beef hash, beef and vegetables, and chicken and vegetables. The first four of these have been production tested with satisfactory results. The others are now being field evaluated. Other food bars that

show nearly equal promise are blueberries, cherries, meat balls (beef), and pork sausages. All of the primary components of the bars mentioned above were freeze-dried except onion, which was air-dried. Many other foods have been investigated. Representative papers that have been published during the last decade are those of Ishler [1], Durst [2], Lampi [3], Rahman, et al. [4], Pavey [5], Tuomy [6], and Mackenzie and Luyet [7].

None of the bars mentioned above have yet been incorporated into the Military feeding system. However, two somewhat similar bars: Cereal, Premixed, Compressed (MIL-C-3483); and Corn Flake Bar, Survival-type (MIL-C-35074) are a part of the system.

The chief drawback of freeze-dried, compressed foods is their relatively high cost. In the present investigation of beef, the plasticizing water is introduced into the freeze-dried food via the vapor phase, with the beef in a vacuum chamber (this chamber may be the freeze-dryer, if desired). This procedure takes less time than the conventional rehydrating procedure, which is to sprinkle the food with water and let it stand until the water is uniformly distributed.* It therefore offers a possibility of reducing production costs. In addition to studying the process of vapor-phase rehydration itself we have made compressed bars, varying the water content of the bars and the force used in compression, and observing the strength of the bars and their ability to reconstitute.

It is sometimes asked why it is necessary to remove all of the water (ice) from beef by freeze-drying and then restore a part of the moisture as a plasticizer. This problem is discussed in reference [8]. What was reported there is, briefly, that freeze-drying can be stopped when the food contains the appropriate amount of water for plasticizing, but the remaining water will be concentrated in ice cores at the center of each piece. If these ice cores are allowed to melt and the water to redistribute itself in

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* After the first draft of the present paper had been written, some experiments were performed that raised questions regarding this procedure. These experiments are discussed briefly in the final section (Sec. 6) of this report, which is headed: "Addendum: Plasticizing by Spraying."

the pieces, the region of the core will have an air-dried character rather than a freeze-dried character, and may be hard and discolored. As a further difficulty, different pieces dry at different rates, so some pieces in a batch will contain too much water and some too little for optimum plasticizing.

Some information on the basic mechanisms of vapor-phase rehydration has been obtained in the course of the experiments, including a small amount of adsorption-isotherm data, some data on the vapor-pressure of raw beef, and data on the maximum temperatures reached in resorption. Some of these data were taken under unfavorable conditions but they are presented because of the scarcity of such data.

2. Small-Scale Experiments.

The small-scale experiments were performed in 1971 in an all-glass apparatus constructed for freeze-drying studies and described in reference [8]. The apparatus was modified by adding a graduated tube (5 ml) in which water for the rehydration was stored. Figure 1 is a diagram of the modified apparatus. When small specimens were used the amount of water added could be determined from the increase in weight of the specimen and also from the change in level of water in the graduated tube. Changes in weight were determined from spring elongations as measured with a cathetometer. Pressures were measured as appropriate on the mercury or the oil manometer. A few of the later experiments were performed after equipment had been installed to permit automatic recording of weight and pressure. A commercial (Schaevitz) linear variable differential transformer was used to measure spring extensions and a commercial (Datametrics) diaphragm-type electronic manometer was used to measure pressures. Temperatures were measured in the thermostated bath that furnished the liquid circulated to the jacket of the specimen chamber.

Ordinary water contains dissolved air, which can interfere with the flow of water vapor. To get rid of dissolved air, the water in the graduated tube was distilled twice, once before it entered the apparatus shown in Fig. 1 and once again as it was transferred into the tube. One distillation might have been adequate.

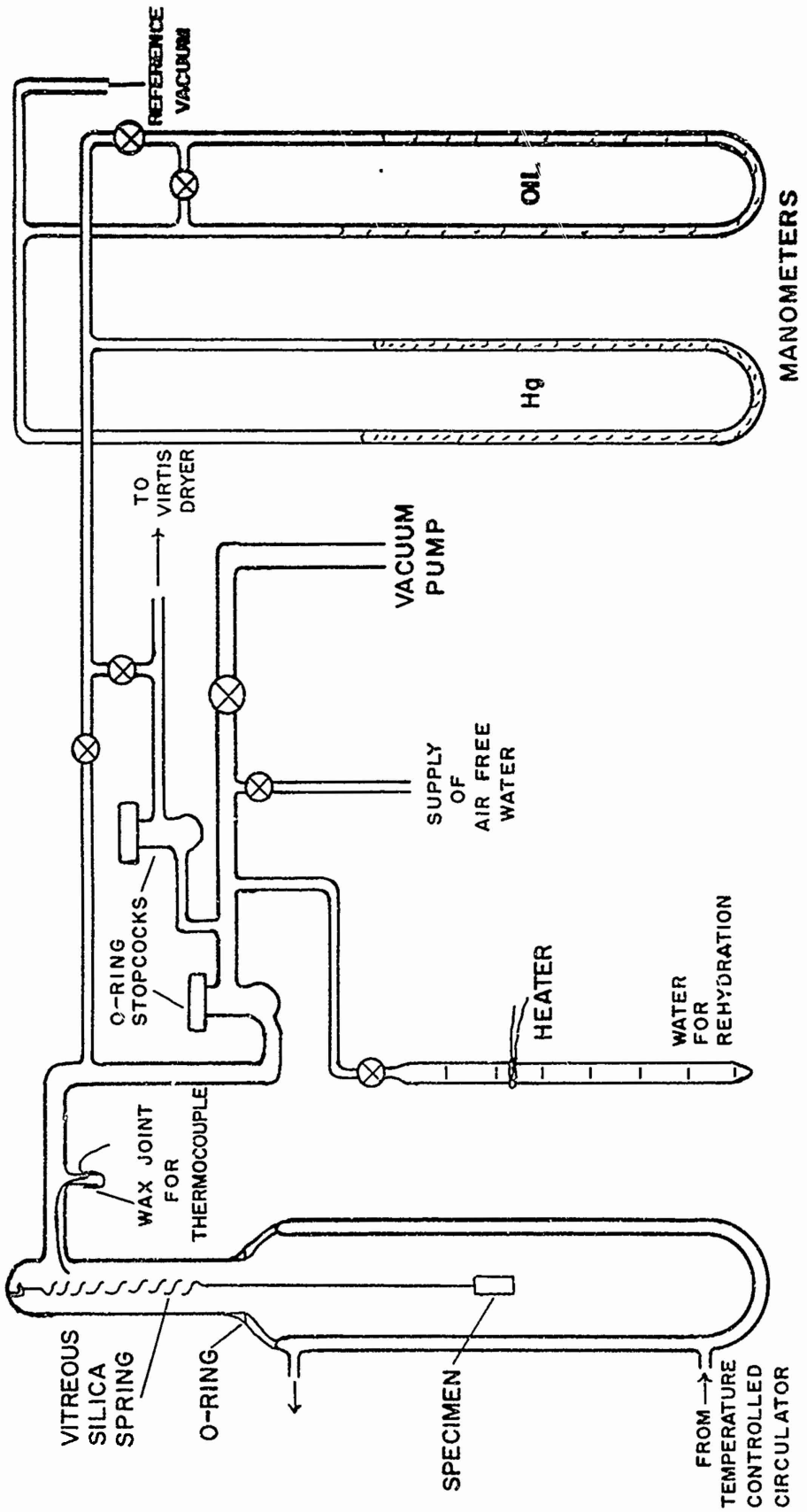

Fig. 1. Apparatus in which the small-scale experiments were performed. The water supply and manometers were also used in the larger-scale experiments.

The small-scale experiments began with some measurements of adsorption and desorption, with rough measurements of the temperatures reached. We then measured some vapor pressures and calculated heats of vaporization from them. Finally, freeze-dried beef in sufficient quantity to make bars was rehydrated and compressed.

Adsorption and desorption. Specimen 44 (raw beef, 1/2 x 1/2 x 1 inch) was first freeze dried, then rehydrated in place in the evacuated chamber, with the chamber walls held at 25 C. A fixed pressure of water vapor was maintained in the chamber by connecting it to a thermostated reservoir containing water at some temperature below 25 C. Conditions were maintained until adsorption was substantially complete. After the necessary measurements had been made, the adsorbed water was pumped off and the experiment was repeated, with the specimen chamber still at 25 C but with a different pressure of water vapor, obtained by changing the temperature of the thermostated reservoir. In this way the data in Table 1 were obtained. The third point is an attempt to repeat the second one, after the water previously adsorbed had been pumped off. At the fourth setting equilibrium was not reached; the water supply was cut off after 50 minutes of adsorption, when the water content was about 12%. The pressure in the chamber fell rapidly as adsorption continued up to about 14%, giving very convincing evidence of the strong affinity of freeze-dried beef for water vapor.

Table 1. Adsorption of water vapor by freeze-dried raw beef at 25 C, where P_{sat} = 23.756 torr.

Reservoir Temperature C	P/P_{sat}	g H_2O per 100 g dry beef
13.0	0.473	8.5
21.9	.830	19.1
21.9	.830	18.4
24.1	.948	(interrupted)

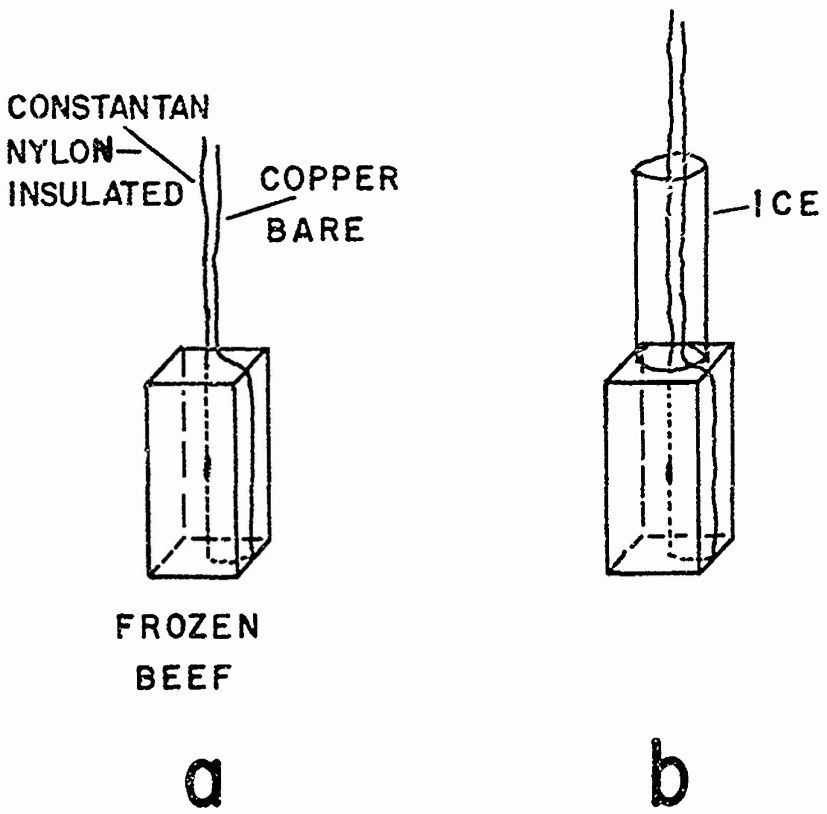

Fig. 2. Method of installing thermocouples at center of beef specimens. (a) normal installation, (b) with cylinder of ice to help bring thermocouple to temperature of specimen.

Temperatures reached. Attempts were made to measure temperatures within specimens when freeze-drying, sorption, and desorption were in progress. The results are subject to error because the specimens were small and the immersion of the thermocouple was insufficient to bring it to the temperature of the specimen if the temperature gradient along the thermocouple integrated to more than a few degrees. However, the results are useful because they give lower limits on the high temperatures and upper limits on the low temperatures that were reached within the specimens. The experiments were made by using two similar specimens of raw beef (1/2 x 1/2 x 1 inch) together in the drying chamber. One specimen was hung from the spring in the usual way and the other was suspended by means of the thermocouple that it contained. The complication of having to make mass measurements on a specimen containing a thermocouple was thus avoided, at the expense of having to assume that both specimens underwent the same changes in temperature and mass. Figure 2a shows how the thermocouple was installed. A hole was drilled entirely through the specimen along its long axis, an AWG 36 bare copper thermocouple wire was pushed thru the hole and soldered to the insultated constantan wire. The junction was pulled to the center of the specimen and the copper leg was bent as shown. Tests showing the conditions under which the thermocouple errors became significant will be described later.

A series of measurements were made with specimen 46 containing a thermocouple and specimen 47 hung on the spring. Due to experimental difficulties, no temperature data were obtained during the initial freeze-drying, but temperatures were measured during subsequent adsorptions and desorptions of water vapor. Figure 3 shows the temperature of specimen 46 and the weight of specimen 47 during a rapid adsorption. The vapor supply was a 1-liter flask containing water at 24.5 C, which gives a vapor pressure of 23.1 torr. With the dry specimens in the chamber at 25C, vapor was admitted at time zero. The heat of adsorption very rapidly raised the temperature so that the thermocouple indication very rapidly rose from 25 to 50 C. Heat loss then

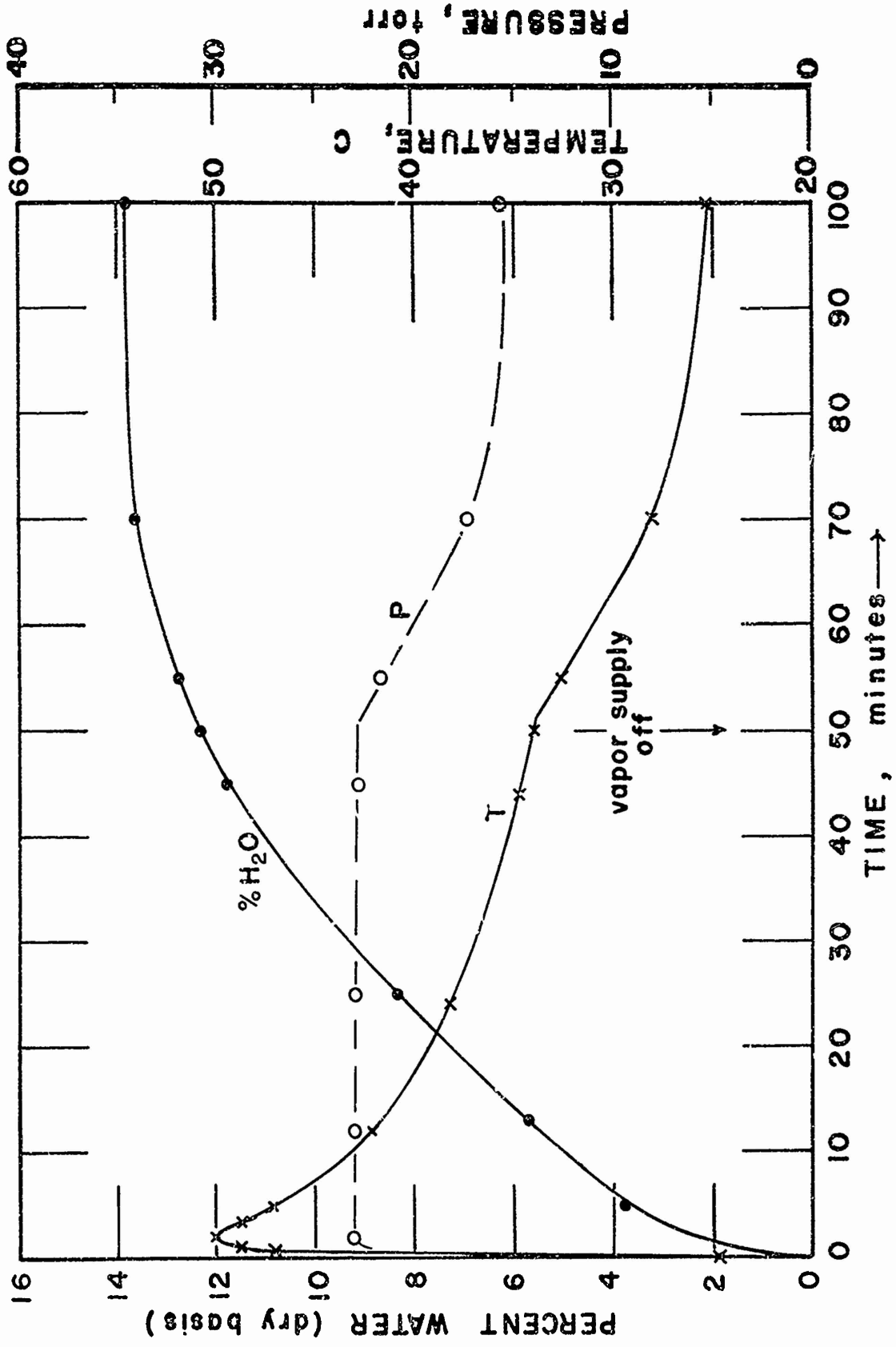

Fig. 3. Adsorption of water by freeze-dried specimens: P, pressure of water vapor; T, temperature indicated by thermocouple at center of specimen 46; %H_2O, grams water per 100 grams dry weight taken up by specimen 47.

exceeded production and the temperature began to fall. After 50 minutes the temperature had fallen to 34 C and the specimen on the spring had adsorbed 0.140 g of water vapor (12.3% of its dry weight). At this time the vapor supply was cut off and the temperature began to fall more rapidly. Adsorption continued at a lower rate and the pressure of water vapor in the chamber fell.

As mentioned earlier, the thermocouple immersion was insufficient when large temperature gradients existed in the legs of the couple. On the basis of later experience it is estimated that the actual temperature inside the specimen was about 53 C when the thermocouple indicated 50 C.

The two specimens containing adsorbed water were next redried by evacuation. The results are shown in Fig. 4. As before, the chamber was held at 25 C. The water content of the specimen on the spring was 0.146 g (12.8% of its dry weight) and the water content of the other specimen is assumed to be the same. Since water vapor is now being desorbed rather than adsorbed, the temperature and the water content show trends that are the opposite of those in Fig. 3. The thermocouple indication reached a minimum of 3.5 C at t = 3 minutes. Making allowance as above for the error of the thermocouple, the actual temperature of the specimen at the minimum was estimated to be 1 C. The water content of the weighed specimen was reduced from 12.8% to 4.7% in 1 hour of pumping.

Another pair of specimens, Nos. 48 (with thermocouple) and 49 (hung on spring) were prepared and freeze-dried in order to see what temperatures were reached in the initial freeze-drying. As soon as the initial transients died out, the thermocouple indicated a temperature of -13.8 C. This value rose almost linearly with time, reaching -8.2 C after 2 hours of drying. About all this tells us is that the values were at least as low as the thermocouple indicated. When temperatures are calculated from sublimation pressures under conditions similar to those of this experiment, the early specimen temperature is in the neighborhood of -22C.

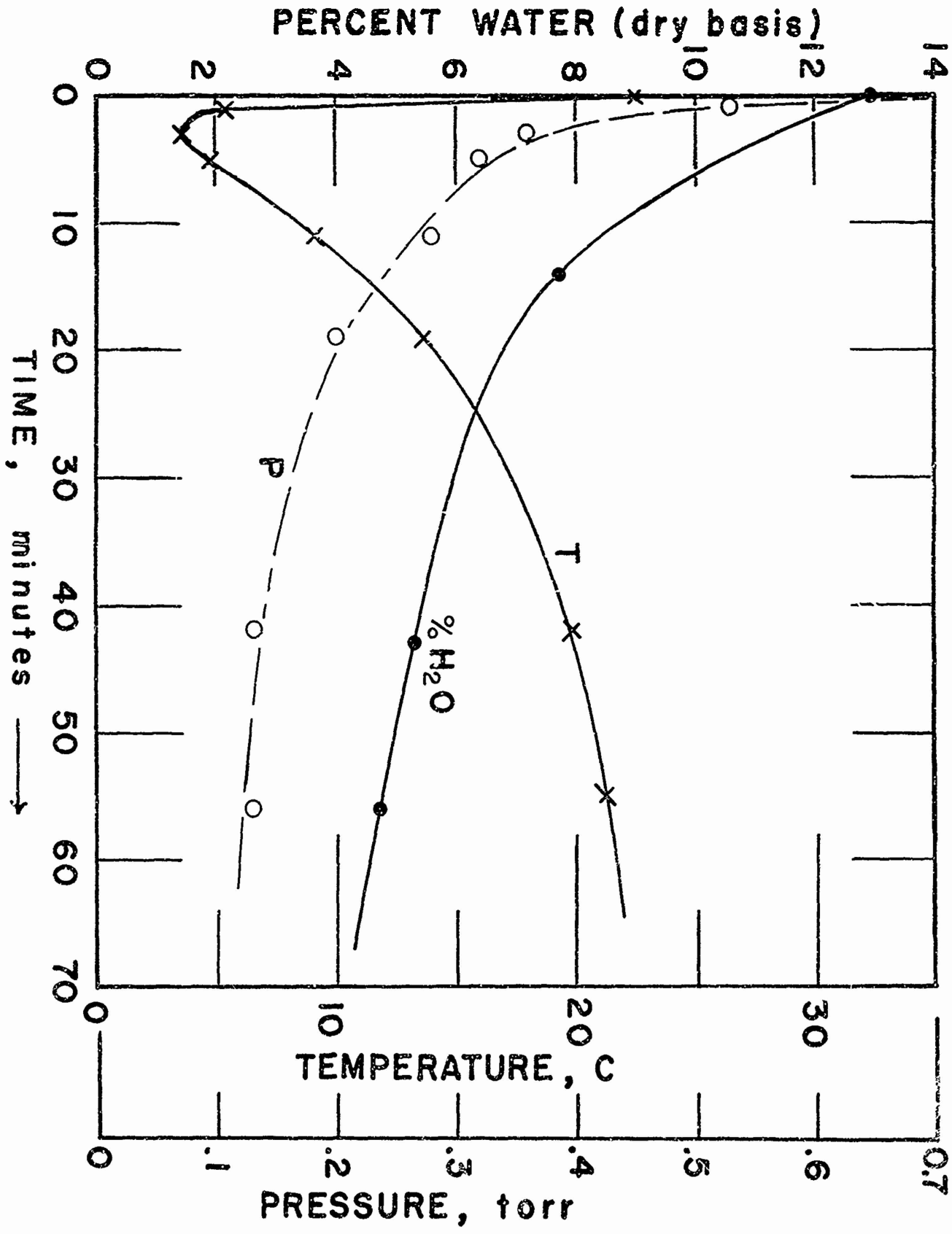

Fig. 4. Desorption following the adsorption shown in Fig. 3: P, pressure of water vapor; T, temperature indicated by thermocouple at center of specimen 46; $\%H_2O$, grams water per 100 grams dry weight, for specimen 47.

An attempt was made to determine the thermocouple error experimentally. A couple was mounted in specimen 54 in the usual way and then a cylinder of ice was built up at the top of the specimen, as shown in Fig. 2b. The purpose of the cylinder of ice was to bring the thermocouple close to the temperature of the specimen before it actually entered the specimen. The ice was built up by using a paper form, cooling the specimen and form in a dry-ice-cooled region, and dropping water slowly into the form When the form was full it was peeled off and then the specimen and the ice were freeze-dried in the usual way, but without continuous weighing. The results were analyzed by comparing them with the data obtained when specimens 48 and 49 were freeze dried. The results indicated that the thermocouple protected by the ice cylinder did indeed indicate temperatures lower than the unprotected couple. The differences ranged from 1.3 to 4 C. Our belief is that neither couple was actually correct, because the copper leg of the protected couple probably picked up heat after it left the ice to pass over the outer surface of the specimen and enter it at the bottom. During the freeze-dryings the chamber walls were at 65 C so the difference between the wall and ice temperatures was about 85 C.

Vapor pressures. Measurements of the vapor pressures of pure ice and of frozen raw beef were made in the apparatus shown in Fig. 1. The measurements on ice permitted us to check the accuracy of our methods. It was found that satisfactory measurements could be made but that precautions were necessary and the apparatus was somewhat clumsy to manipulate when temperature change was desired. To permit good measurements to be made, the walls of the specimen chamber should be at a slightly higher temperature than the specimen. Otherwise water vapor can condense on the walls of the chamber and we will measure the vapor pressure of this water (or ice) rather than the vapor pressure of the specimen. All vacuum lines connecting to the chamber must likewise be above the specimen temperature. However, if the chamber walls were too hot, the thermocouple that indicated the specimen temperature could give too high a reading. A wall temperature of 0 to 1 C above the specimen temperature was optimum, but some good data were obtained outside this range.

In order to cool the specimen rapidly it was necessary to pump on it, producing a slight freeze-drying and a lowering of the temperature by sublimation. The liquid used for thermostating the chamber walls had to be cooled to a suitable temperature for each measurement, of course. Care was taken not to freeze-dry the beef specimen any more than necessary when cooling it. The vapor pressure of frozen beef undoubtedly changes slightly between, say, the 5% freeze-dried and the 95% freeze-dried states. The change may be too small to detect or it may be significant. The present measurements were made on a specimen that was only slightly freeze-dried. Ice, being a pure substance, should not change in vapor pressure as it is freeze-dried.

After learning how to use the apparatus we made 4 good measurements on the vapor pressure of ice, using an ice specimen frozen with a thermocouple at its center, more or less as shown in Fig. 2a. These measurements covered the range -11.00 to -1.79 C and agreed with accepted values within 0.05 torr or better. A thermocouple was then installed in specimen 55 (frozen raw beef, 1/2 x 1/2 x 1 inch) and vapor-pressure measurements were made on it. The results are given in Table 2 and plotted in

Table 2

Vapor Pressure of Frozen Raw Beef

Temp. C	Pressure torr
-22.0	0.58
-20.5	.64
-19.2	.79
-18.2	.87
-17.2	.96
-16.1	1.07
-15.1	1.17
-13.5	1.35
-11.9	1.57
-10.2	1.81
- 8.0	2.22
- 6.0	2.64
- 5.1	2.89

Fig. 5. The pumping and the manipulating of the temperature of the chamber walls were performed rather quickly so that long waits for equilibrium were avoided. With this procedure the errors associated with the build-up of pressure from any gases released by the beef would be minimized. It seems likely that gases such as CO_2, N_2, and perhaps O_2 may be released, but the quantities may be too small to be of consequence. The vapor-pressure curve of frozen beef in Fig. 5 may be compared with the curve for pure ice, which is plotted (from handbook data) for comparison. At -20C the vapor pressure of beef is about 7% below that of ice; at -5C the difference is about 4%.

Because of the possibility of changes in concentrations, frozen beef probably does not have a true vapor pressure in the strict sense. Vapor pressure is an equilibrium property, and in a system as complicated as beef and the vapor above it, it is improbable that equilibrium is ever reached. Diffusion within both phases will carry the system toward equilibrium but the process will be slow. Hence the observed "vapor pressures", tho useful, will depend to some extent on the conditions of measurement.

Heats of sublimation. Heats of sublimation can be calculated from vapor pressures, and since they are of direct interest in freeze-drying, we have calculated them from both our ice-data and our frozen-beef data. Again the results obtained for ice were used as a check on accuracy. For pressures at which the volume of the condensed phase is negligible in comparison with that of the vapor phase, the thermodynamic relation for the calculation is

$$\frac{d(\ln P)}{d(1/T)} = -\frac{\ell}{R} \qquad (1)$$

where ℓ is the latent heat of sublimation and R is the gas constant for the vapor in question. On the basis of this equation we plot the logarithm of the vapor pressure versus the reciprocal of the Kelvin temperature and should obtain a straight line of slope $-\ell/R$. The slope is measured and ℓ calculated from it.

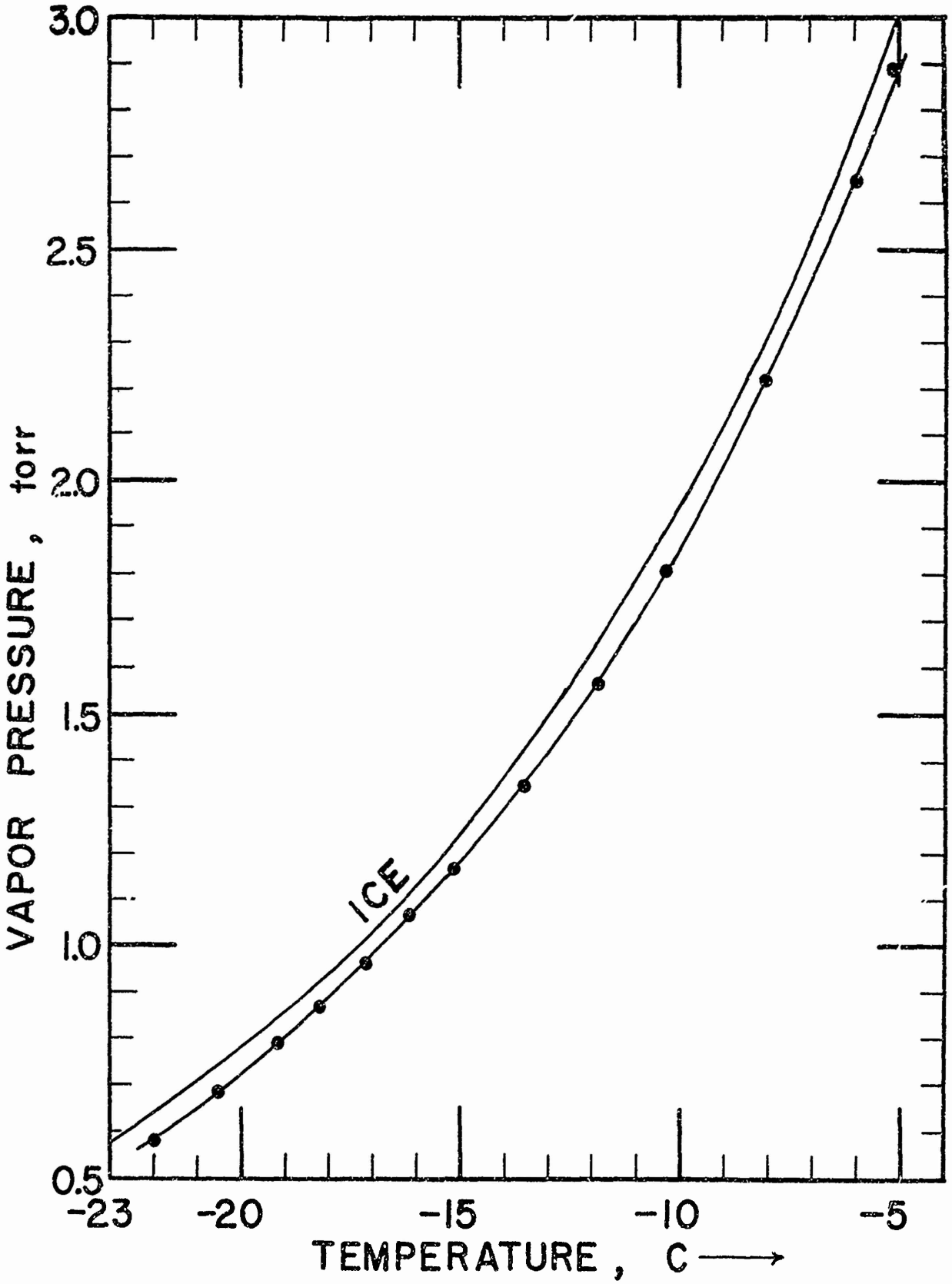

Fig. 5. Vapor pressure of frozen raw beef, with the accepted curve for pure ice shown for comparison.

When our four good values for ice were plotted as described above, the measured slope gave ℓ = 2833 j/g, which is only 2 per mille below the accepted value of 2838. A similar graph was plotted for beef, using all of the data in Table 2, and yielded ℓ = 2907 j/g. Our frozen beef therefore has a heat of sublimation only 2.4% higher than that of pure ice. The only data available for comparison appear to be those of Hill and Sunderland [9], who found the vapor pressure of frozen beef to be 20% below that of ice and the heat of vaporization to be 22% higher than that of ice. In view of the substantial discrepancy the true values must remain uncertain until further work is done.

Vapor-phase rehydration. The most important of the series of small-scale experiments was an attempt to plasticize freeze-dried beef for compression by vapor-phase rehydration. Two experiments were performed, each on a separate lot (~35g) of freeze-dried beef. The first lot was raw, the second, cooked. Both lots were prepared and freeze-dried by the Food Laboratory. They were in the form of small pieces (ca 3/4 x 3/4 x 1/2 inch). In the first experiment 25 pieces (36.6 g) of the raw lot were strung on wires and suspended in the drying chamber of the apparatus shown in Fig. 1, which was thermostated at 25 C. The system was evacuated to remove moisture and other gases that might have accumulated on the specimen surface. Then the vacuum line was closed and the line to the water supply in the graduated tube was opened. Transfer of vapor from the tube to the beef was continued, heating the supply in the tube to speed up the process, until 4.3 ml (11.7% of the dry weight of the beef) had been transferred. The process required about 2 hours. Figure 6 shows the amount of water transferred as a function of the time. The plasticized (partially rehydrated) sample was given to Food Laboratory personnel who compressed it into bars. Lieutenant Richard O. Shuler reported to us that good bars were made from the sample, about equal in strength to a control group rehydrated conventionally by sprinkling and allowing to stand for 2 days.

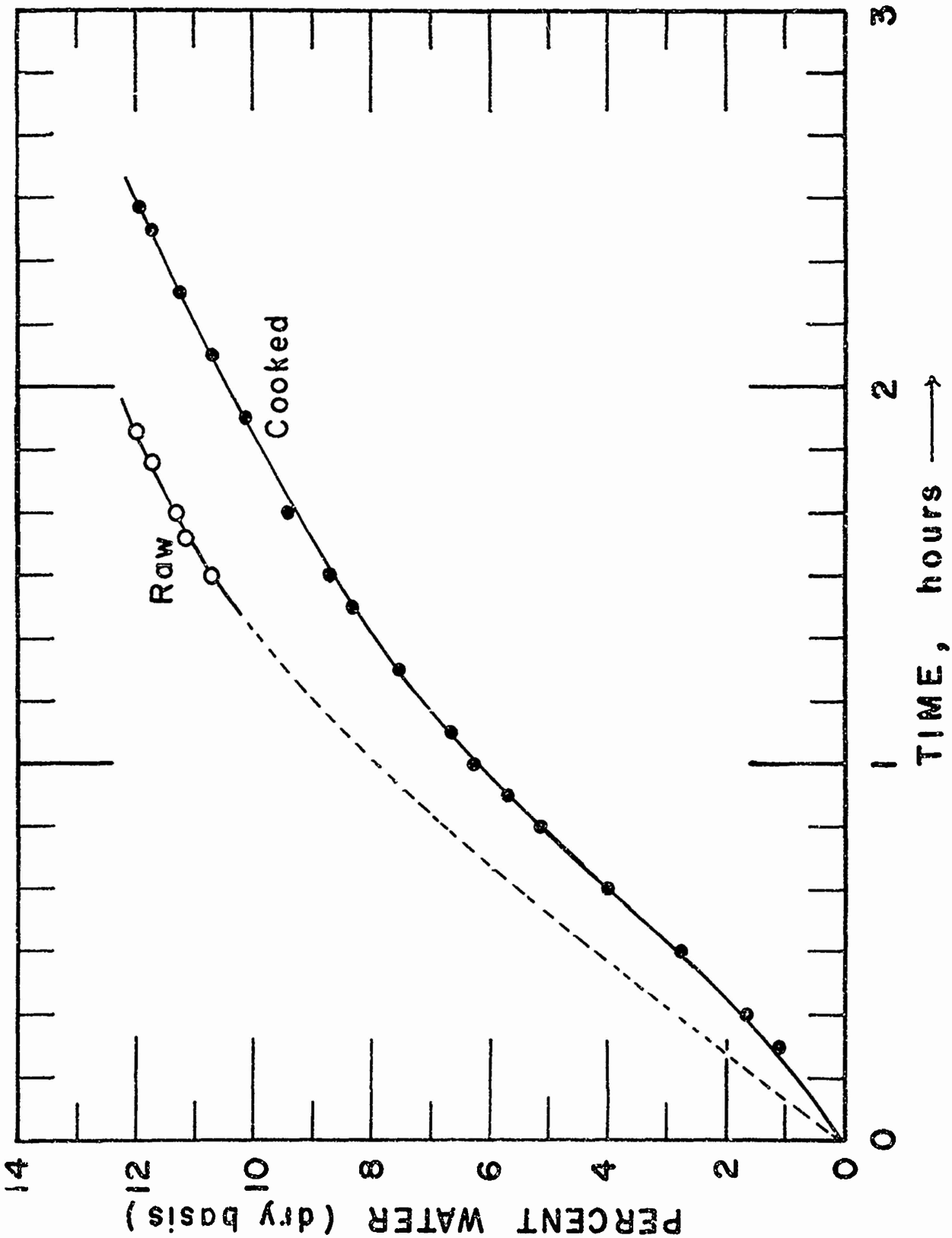

Fig. 6. Vapor-phase plasticizing (rehydrating) of raw and cooked freeze-dried beef in the small-scale apparatus.

When the raw-beef bars were rehydrated ready for cooking by placing them in water at 88C (190F), they did not reconstitute well. This behavior has been observed in raw beef before. A second lot of beef was then prepared. This lot was cooked before freezing and freeze-drying. A sample consisting of 22 pieces (41.0 g) of this lot was plasticized by adding 4.89 ml (11.9% of the dry weight) of water, using the same procedures as for the raw beef. Bars were again made by FL; good mechanical properties were again found and in addition the cooked meat was found to reconstitute satisfactorily in water at 88C (190F). The plasticizing by vapor transfer required about 2.5 hours. Cooked beef therefore appears to require longer to take up a given water content than raw beef. However, not all of the conditions were the same in the two experiments, so this finding should be confirmed. Results for the cooked beef are also shown in Fig. 6.

The completely dry beef has a strong affinity for water; it adsorbs water rapidly at first and then more and more slowly. As the surface layers of the beef become covered, the affinity weakens and the heat of condensation falls, approaching the heat of sublimation of pure water. The limiting factor in the rate of adsorption is undoubtedly the temperature rise of the sample. This rise can be substantial ($\sim$25 C) as shown earlier. The vapor transfer could be speeded up if there were an improved way to get rid of the heat that is released as the vapor condenses. We had no way to speed up the heat transfer other than to raise the pressure of water vapor to the highest safe level and thus raise the temperature of the sample as high as possible. To control this pressure an electric heater that could be slipped over the water-supply-tube was built. This heater was kept at the level of the meniscus and the power supplied to it was controlled to keep the pressure of water vapor in the system as high as practical while avoiding condensation at any place except on the beef.

3. Larger-Scale Experiments.

The small-scale experiments were completed in the spring of 1971. They showed that beef could be plasticized for compression by exposing the dried material to water vapor. They gave indication that a satisfactory compressed product could be made but did not

give much information on how much water should be added or what forming pressures should be used. The Food Laboratory undertook the next stage of the investigation and obtained some data which, however, indicated the need for more accurate control of the process variables. After discussions it was decided that the Pioneering Research Laboratory would reassume responsibility and attempt to carry out the next stage of the investigation. This second phase of the work was begun in PRL in the summer of 1972.

Apparatus. The small commercial freeze-dryer shown in Figure 7 was used in the work. This dryer, loaned to PRL by FL, was a Virtis Company Model 10-109. The drying chamber is cylindrical, with a heavy lucite door at the front and three shelves for food. The total shelf area is about 2 sq. ft. Liquid (ethylene glycol and water) from a thermostated reservoir is circulated to the shelves thru a tube that has coils fastened to the under side of each shelf. Normally the circulating liquid was used to heat the shelves, but when rehydrations were performed it was desirable to cool the shelves, and the circulating liquid was cooled rather than heated. For the present experiments the part of the apparatus containing the drying chamber and the temperature-control equipment (top) was separated from the refrigerating equipment and vacuum pump (bottom) as shown in the photo.

The main purpose of separating the two parts of the apparatus was to permit a heavy rubber hose (1 inch id x 2 inches od) to be introduced, which would serve as part of a reliable, leak-proof valve. Two strips of wood and a large C-clamp formed the rest of the valve. When the valve was closed by squeezing the hose there was never any indication of leaking. Before this valve was installed, a ball valve consisting of a bored rotatable stainless steel ball clamped between teflon hemispheres was used. This ball valve usually worked satisfactorily but occasionally leaked.

Plasticizing of the freeze-dried beef by partial rehydration was performed in the same chamber where the meat was originally dried. Rehydration required a water supply and pressure-measuring devices not present in the commercial equipment. The drying chamber was therefore connected to the small-scale equipment shown in Fig. 1 and the graduated water-storage tube in Fig. 1 was replaced by a larger (25 ml) tube. Plasticizing with the

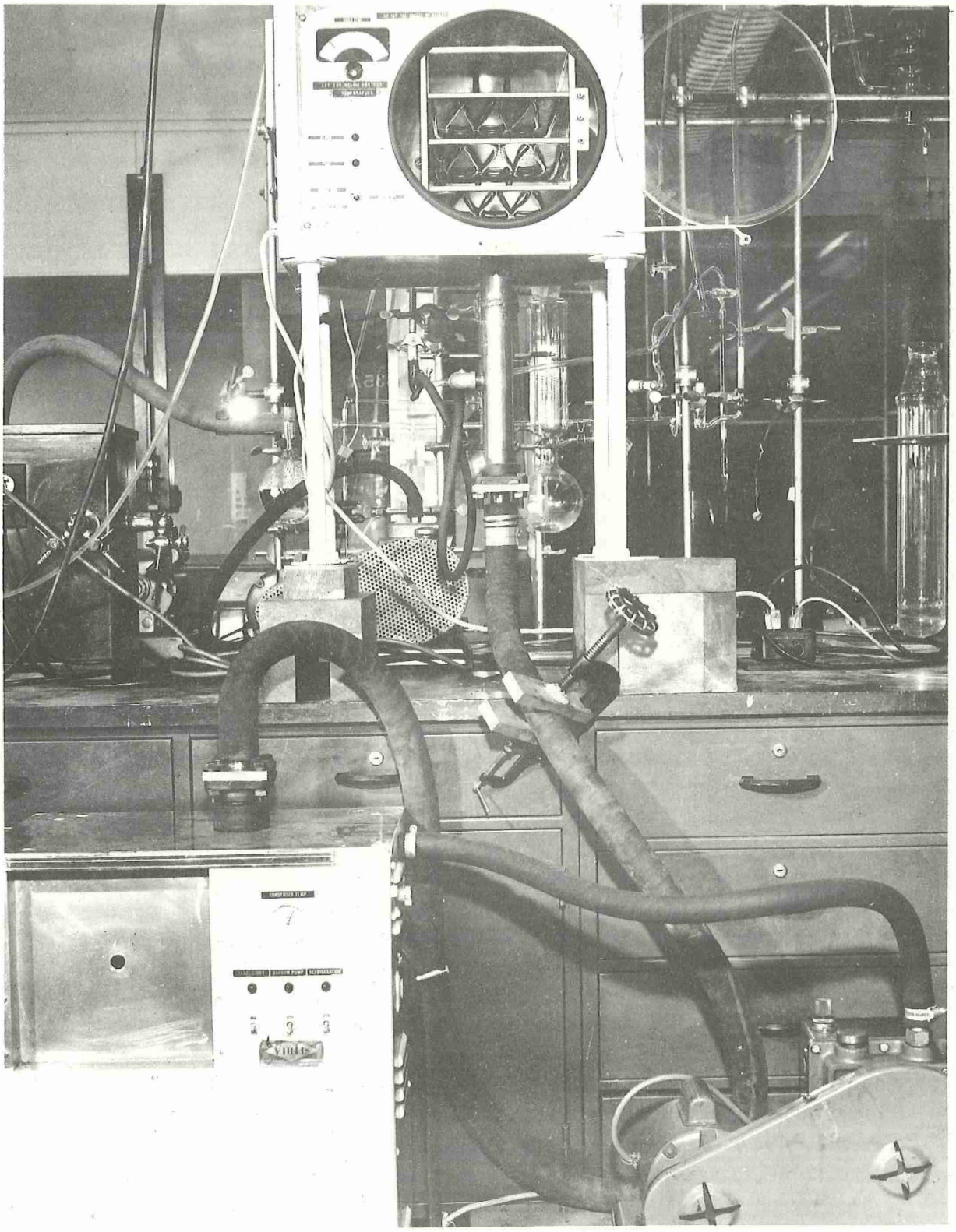

Fig. 7. Small commercial freeze-dryer used in the larger-scale experiments. A heavy rubber-hose vacuum-line joins the upper and lower parts and contains a C-clamp valve.

material still in the drying chamber was considered desirable because it would be advantageous in industrial practice. If a food can be plasticized in a relatively short time before it is removed from the dryer, one step in the handling of the product is made unnecessary.

In preparing the apparatus for use, much effort was expended (a) to get the drying chamber and shelves clean and (b) to get rid of all leaks. Dirt and leaks can be more troublesome in rehydration than in freeze-drying. In freeze-drying, any non-condensible gases (mainly air) that leak into the chamber or are given off by the food or by dirt in the apparatus are carried to the condenser by the flowing water vapor and are removed from the condenser by the vacuum pump. In rehydration, the vapor flows toward the food and condenses on it. Any air in the space surrounding the food will be carried along and will build up inside the pores. The effect is cumulative and after a time there will be a barrier of air that the water vapor must diffuse thru before it can be adsorbed by the food. When the process becomes diffusion-limited, vapor-phase rehydration is too slow to be attractive.

Our apparatus had one leak in the drying chamber, in the glass-to-metal seal where electrical circuits can be brought into the chamber. This leak was sealed by painting it with glyptal lacquer, using vacuum to suck the lacquer into the hole. A new rubber door-gasket was installed, probably unnecessarily. The greatest difficulty was experienced from dirt, probably food residues. Examination showed that the dirt covered the walls of the chamber and the tops and bottoms of the trays. It also seemed to have penetrated into cracks in the putty or potting compound that was used to improve the thermal bond between the temperature-control coils and the bottoms of the shelves. The dirt in the chamber appeared to adsorb both water vapor and air. When the chamber was open to the atmosphere, adsorption occurred, and when the chamber was subsequently evacuated the previously adsorbed material was released. The time required to pump down was therefore lengthened. The adsorption and desorption is undesirable for two reasons: it interferes with the measurement of how much water has gone into the meat during plasticizing, and it will interfere with vapor-phase water transfer if noncondensible gases are desorbed along with water vapor. Almost all of the putty

bonding the coils to the shelves was removed so that the shelves could be thoroughly washed. This reduced the adsorption and desorption of the apparatus walls and the remaining dirt to the level of a slight nuisance.

The equipment used to form the compressed bars is shown in Fig. 8. The press was one normally used for pressing pellets for infrared spectroscopy. The gage was calibrated in terms of pressure and also in terms of total force exerted by the ram. The force-scale extended up to 50,000 lbs. The mold consisted of a sleeve, a base, and a plunger, all separable. All bars had dimensions, 1x3xh inches, where h, the thickness, was determined by how much material was placed in the mold. Two bars of cooked beef made in the press are shown in Fig. 8. One is a good (firm) bar and the other was so weak that it broke apart when handled.

Material. Three lots of cooked beef, designated B, C, and D, were investigated. Lots B and C were U. S. Choice; lot D was U. S. Good. The purchasing and all of the preparation of the material up to the point of freeze-drying were performed by the Food Laboratory. Each lot was prepared from a semi-membranosus beef muscle. The muscle was excised and trimmed of almost all visible fat, after which it was cooked under 6 psi gage pressure. At this pressure saturated steam has a temperature of 230 F. Cooking was continued until the internal temperature of the muscle reached 170 F, which required about 1 1/2 hours. The cooked beef was cooled at 40 F for about 1 hour and then machine-sliced into 1/4-inch slices. The slices were then diced by hand into rough squares. The size of the squares was not carefully controlled but was roughly 5/8 x 5/8 x 1/4 inches with a spread of ±20% in the large dimensions and a spread of ±10% in the smaller dimension. During the slicing all pieces of artery and connective tissue having a diameter greater than about 1/8 inch were removed. The trimmed and selected pieces were canned under nitrogen (lots B and C) or sealed in plastic bags under nitrogen (lot D) and immediately frozen and stored at -10 F.

One batch of the stored material, ready for freeze-drying, was reserved for fat and water analyses. These analyses were made by FL according to methods 24.005 and 24.003 respectively, of the Association of Official Analytical Chemists [10]. Results

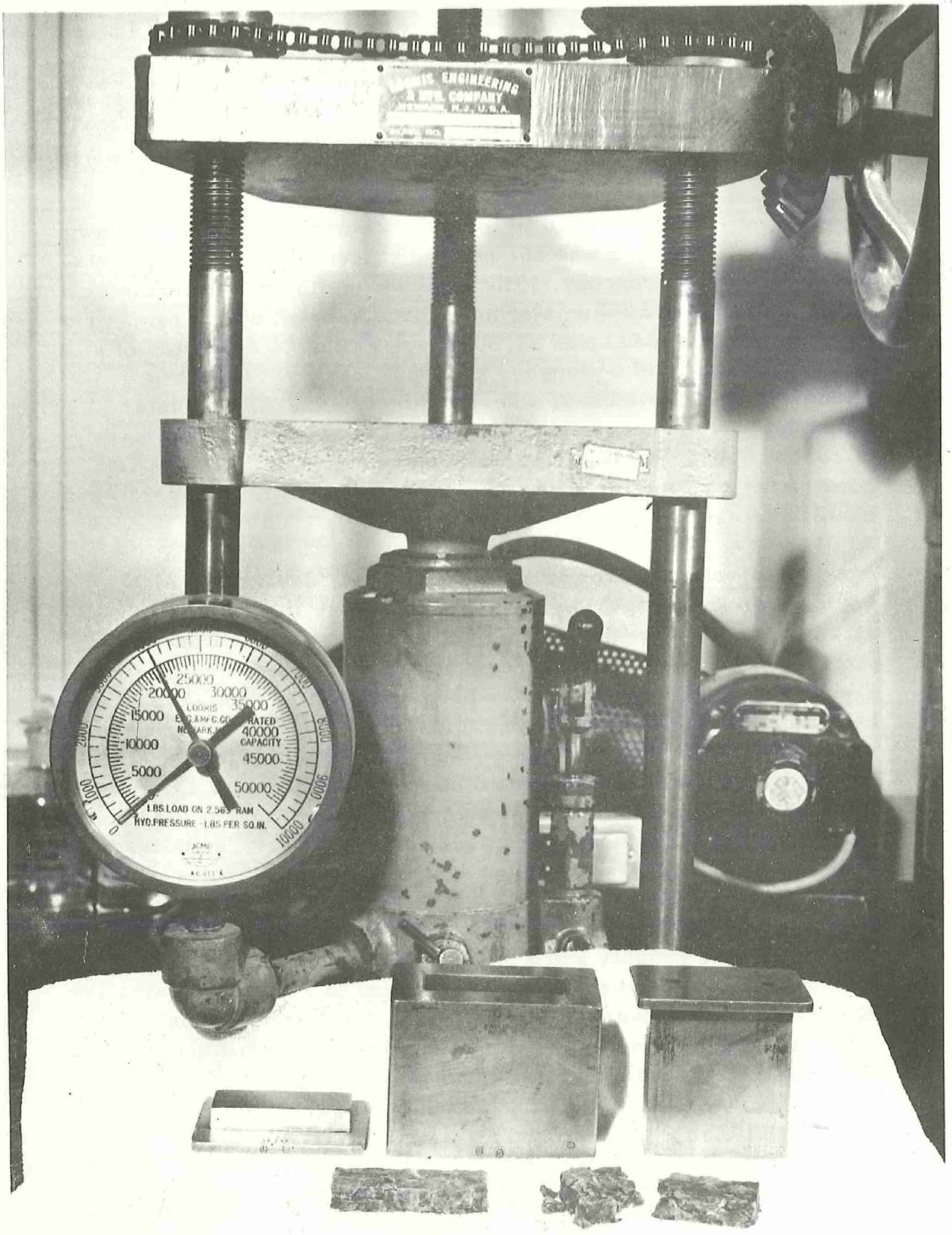

Fig. 8. Two compressed bars of freeze-dried beef (one strong, one weak) and the press and mold used for forming them.

are given in Table 3. Each lot of beef was well mixed after dicing

Table 3. Water and fat content of each lot and batch of beef

Lot			Batch	
No.	% Fat* AOAC Method 24.005	% Water* AOAC Method 24.003	No.	% Water* before f-d
B	2.43	60.33	B-1	60.7
			B-2	59.5
			B-3	60.1
			B-4	60.0
			B-5	60.1
			B-6	59.9
			B-7	60.0
C	7.88	56.16	C-1	57.4
			C-2	57.1
			C-4	56.4
D	5.63	57.49	D-1	57.7
			D-2	57.8
			D-3	57.7
			D-4	57.6
			D-5	57.5
			D-6	57.1

* Total - weight basis

and before dividing into batches, so that all batches from the same lot would be similar.

Procedure. In a typical run, the refrigerating unit of the dryer is turned on 1 hour or more before drying is to begin. A can of frozen beef is removed from the refrigerated room, weighed, placed on dry ice, opened, and the contents (typically 300 g) spread on the 3 shelves of the dryer. The shelves are at room temperature at this point. The vacuum pump is turned on and drying is begun immediately. As soon as the pressure falls to operating level the circulating liquid is heated to operating temperature. The temperature oscillations of the liquid and the shelves are rather large: the peak temperature is 66 C (150 F) and the minimum is 57 C (135 F). Freeze drying was usually started in the morning and continued over night, but usually the drying was well along at the end of the working day and the shelf heat was turned off then, so that the beef would not be exposed to the drying temperature over night.

The progress of freeze-drying can be monitored by observing the pressure in the drying chamber. However, the relation between pressure and the fraction of water (including ice) removed depends on many parameters, such as the size and nature of the drying equipment, the kind of food being dried, and the wall temperature. When all such factors remain constant there is a unique relation between chamber pressure and fraction of water removed. In our experiments with the Virtis dryer, using a load of 300 g of frozen, cooked beef, the initial chamber pressure after drying had gotten well started was about 1.1 torr. This pressure steadily fell to a limiting value of about 0.05 torr in a period of 8 to 10 hours. However, freeze-drying was complete from the practical standpoint in a much shorter time. On two occasions freeze-drying was interrupted long enough for a batch to be removed and weighed, after which drying was completed. Batch D-4, measured after 4 hours of drying, contained water equal to 1.2% of its original weight. Batch D-6, measured after 5.5 hours of drying, contained only 0.14%. A drying time of 5 hours is adequate when the piece size is 5/8 x 5/8 x 1/4 inch. When freeze-drying is to be followed immediately by plasticizing it would be permissible to stop after only 4 hours.

After freeze-drying is completed, the beef is removed from the dryer, weighed, and returned to the dryer. The vacuum pump and refrigerator are run for 30 minutes or more to produce a good vacuum. Then the exhaust line is closed and the line from the water supply to the drying chamber is opened. Water vapor begins to adsorb rapidly on the meat. As soon as the rate slackens the heater is slipped over the graduated tube containing the water supply and the power is raised until the pressure of water vapor in the chamber reaches about 24 torr. The maximum permissible value of this pressure is determined by the temperature of the connecting lines and chamber walls. As mentioned earlier it is undesirable to have water condense anywhere except on the meat itself. If condensation elsewhere does occur it does not ruin the experiment, but the water must be carefully driven off of the places where it is not wanted. Otherwise the amount of plasticizing water put into the meat will be unknown.

The rehydration process normally took from 4 to 5 hours. Figure 9 shows the mass of water adsorbed versus time for two typical lots (B-4 and C-4). The progress of rehydration was followed by observing the fall of the water level in the graduated tube, making an allowance for the vapor that fills the chamber. This is significant. For example at a pressure of 22 torr and a temperature of 25 C, the drying chamber would contain about 0.56 g of water in the vapor phase. This is based on a volume of 26.5 liters (0.936 ft^3) calculated from rough measurements. An additional allowance of 0.75 g is made for adsorption within the apparatus but not on the meat. This adsorption was somewhat erratic but was verified many times by comparing the increase in weight of the meat with the water transferred from the supply. The location of this adsorbed water was not definitely determined. Some of it would adsorb on metal walls, but the bulk of it must have gone elsewhere. The rubber gasket and the plastic door are better adsorbers than metals but even so the disappearance of water was puzzling.

After rehydration the beef is removed from the dryer and weighed again. This weight and the dry weight give the accurate value for the amount of water introduced. The material is divided into portions of about 30 g, and a bar is pressed from each portion.

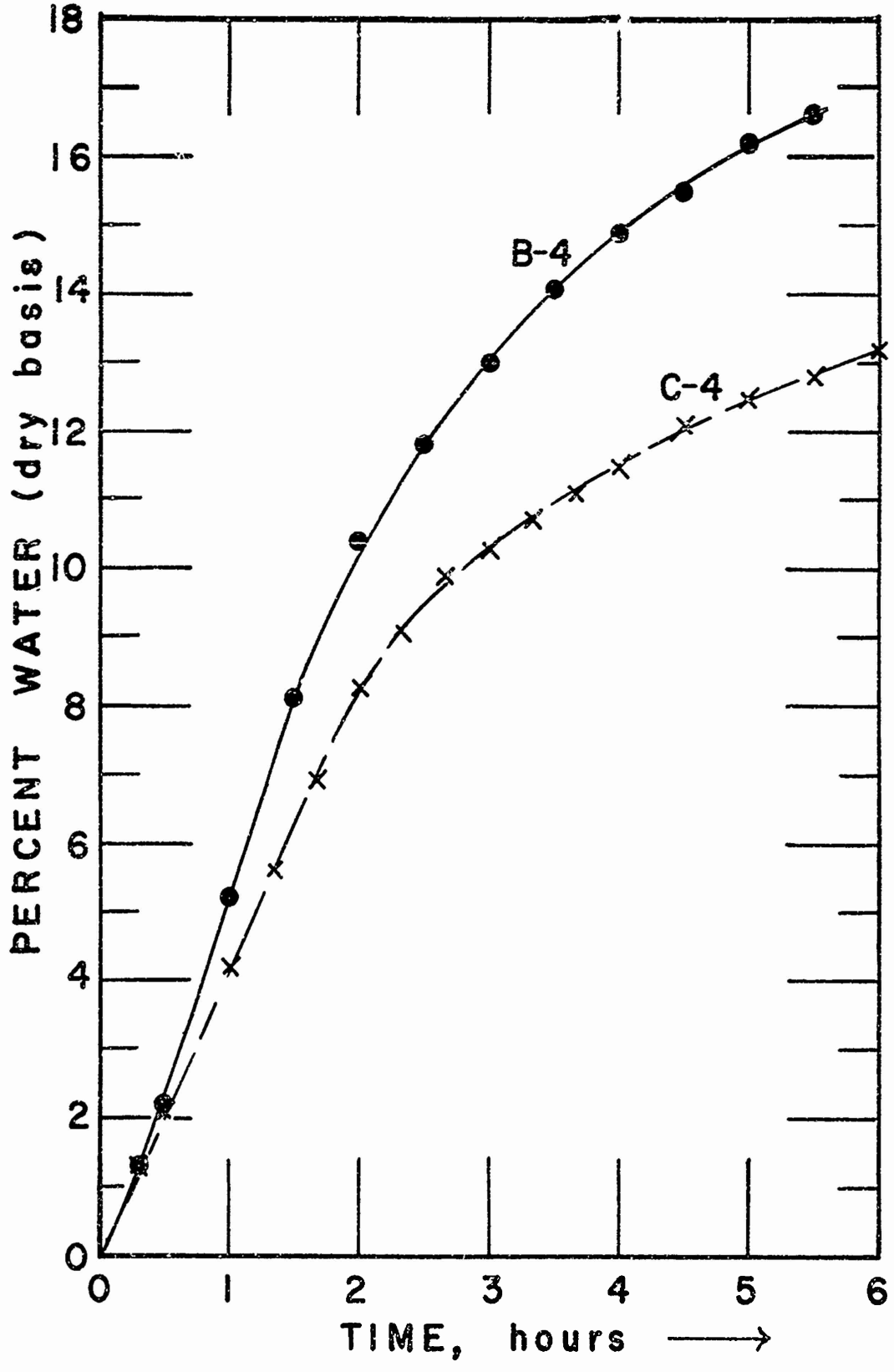

Fig. 9. Vapor-phase plasticizing (rehydrating) of cooked freeze-dried beef in the larger-scale apparatus.

Usually a gradually increasing series of pressures is used. A batch (initially about 300 g) normally would make 4 bars. Typically one bar might be made at 2000 psi, another at 3000, and so on. However, the choice of pressures and the intervals between them varied widely. After compression the bars were allowed to stand for several minutes and then examined for strength by flexing them slightly and by noting whether any pieces had fallen off. A short waiting period before examination was desirable because some of the bars were stronger just after they were pressed than they were later. Oil or fat was squeezed out of the bars when they were formed and some of it was reabsorbed after the pressure was released. We surmised that the fat weakened the bars.

The bars were weighed after forming and were then returned to the freeze-dryer where they were again dried. In the second drying the rate of water loss is slower than in the original freeze drying, but of course there is much less water to be removed. The second drying was continued for a minimum of 10 hours and often was finished over night. The pressure gradually falls as drying takes place, but it is lower than in the original freeze-drying. There was no way to measure the weight of the bars in the Virtis dryer during the final drying, but a single bar was small enough to be hung from the spring in the small-scale apparatus. Two bars were dried in that apparatus, with the results shown in Fig. 10. The bars were similar except that D-3-3 was formed at 4500 psi and D-3-4 at 6000 psi. The bar subjected to the higher pressure dried a little more slowly than the other but the difference would not be very important in practice. The shape of the drying curves is different from those obtained in the initial freeze-drying. This is shown by the final portion of the freeze-drying curve of specimen 69, which is plotted in Fig. 10 for comparison. This specimen was a slab having dimensions not greatly different from those of bars D-3-3 and D-3-4.

After the second drying the bars (or their constituents in those cases where the bars fell apart) were removed from the dryer and weighed again. Finally they were reconstituted by immersing each bar in a separate beaker of water at 88 C (190 F).

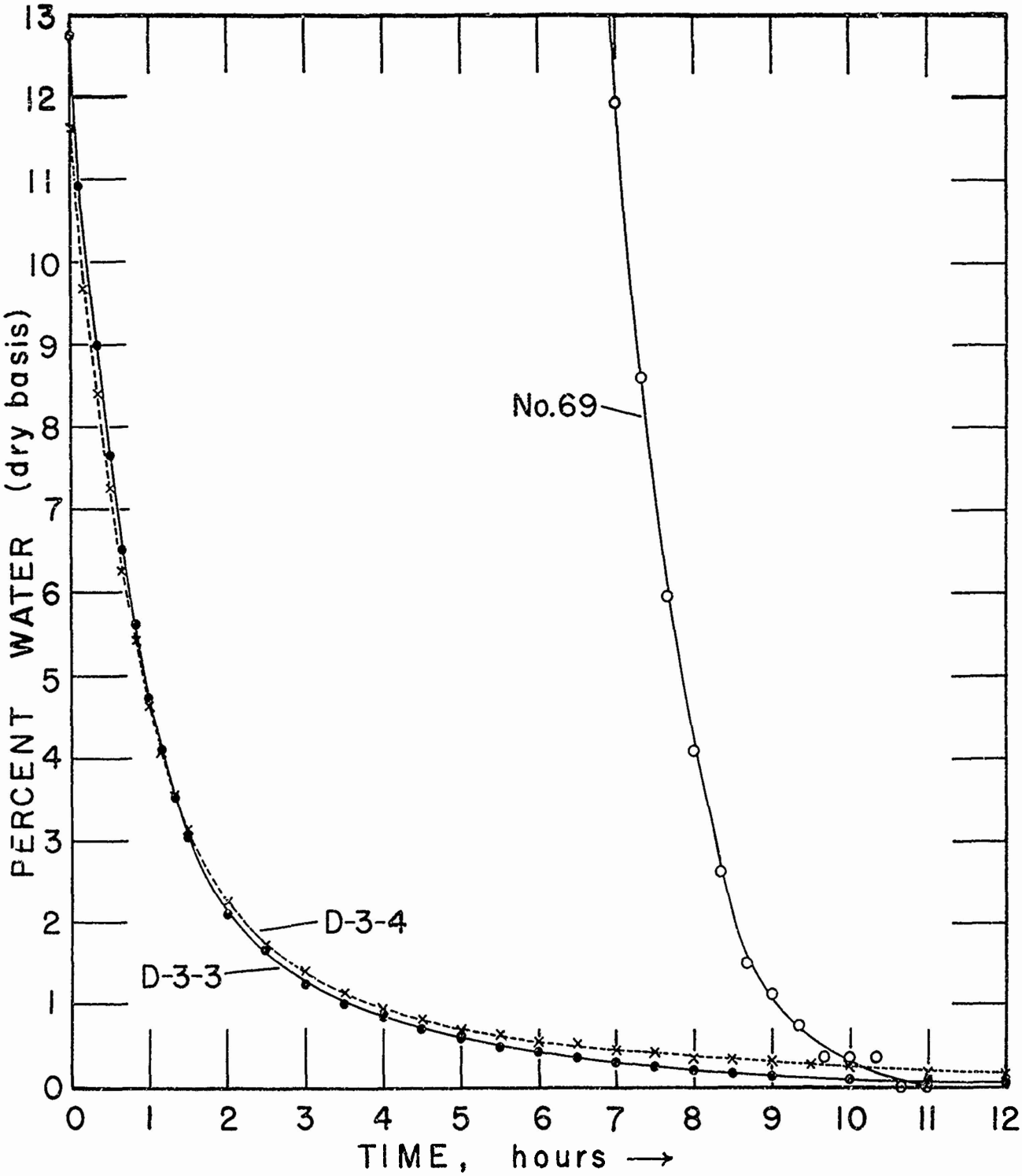

Fig. 10. Vacuum drying of compressed bars D-3-3 and D-3-4 to remove the plasticizing water. Wall temperature, 65C. For comparison, the final stage of the initial freeze-drying of specimen 69 is shown.

The water was not stirred. After 10 minutes the beef was removed from each beaker, drained for about one minute on a paper towel, and then weighed for the final time. The reconstituted beef was not evaluated by a taste panel. Some of it was eaten by the authors and others and was judged to be satisfactory although perhaps not outstanding.

Results. In all, 16 batches of beef have been freeze-dried and rehydrated as described above. All came from lots B, C, and D of cooked beef. A total of 52 bars were formed, and 40 of these have been redried and reconstituted. Table 4 gives data on each bar. The ability to reconstitute well when placed in hot water is the most important of the properties listed. Good reconstituion requires adequate rehydration plus adequate and uniform swelling of the compressed meat so that it will be almost as acceptable as food as it was just after cooking. In the present study the degree of rehydration was our principal criterion, supplemented by visual examination. The water content of each bar after reconstitution is given in Table 4, and the ratio of this water content to the original content (just before freeze-drying) is given in the column headed r. The original content is that of the batch from which the bar was made, as given in Table 3. A rehydration ratio in the range 0.80 to 1 is desirable. Some foods under certain treatments can have $r > 1$ and this can be as much of a drawback as underrehydration.

The strength of the bars is given in Table 4 on a strong-fair-weak scale (S, F, W), with + and - signs used to qualify the rating in some cases. The ability to form strong bars suitable for packing and handling is a convenience but not an essential. Weak bars could undoubtedly be made stronger by adding some palatable material that would serve as a binder.

Table 4. Properties of freeze-dried, compressed, cooked-beef bars, as influenced by the amount of plasticizing water and the forming pressure.

Bar No.	%H_2O ** as Plasticized	Forming Pressure psi	Strength	%H_2O * Reconstituted	Rehydration Ratio r
B-1-1	12.6	1800	F	57.7	0.95
2	"	1800	F	-	-
3	"	2360	S	53.7	.88
4	"	2360	S	-	-
B-2-1	10.0	2360	W	-	-
2	"	2360	W	56.0	.94
3	"	2830	F-	-	-
4	"	2830	F-	55.7	.94
B-3-1	13.7	1800	S	50.6	.84
2	"	1800	S	-	-
3	"	1200	F+	-	-
4	"	1200	F+	48.8	.81
B-4-1	16.6	1200	S	44.5	.77
2	"	1200	S	-	-
3	"	800	F+	46.3	.77
4	"	800	F+	-	-
B-5-1	11.2	3865	S-	57.8	.96
2	"	1800	W+	57.4	.96
3	"	1200	W+	57.8	.96
4	"	2828	F+	55.9	.93
B-6-1	12.0	2360	F	50.5	.84
2	"	3000	S-	49.4	.82
3	"	3530	S	49.6	.83
4	"	4000	S+	50.0	.84
B-7-1	10.2	3000	W	57.9	.96
2	"	4000	F	52.7	.88
3	"	5000	S	46.3	.77
4	"	7500	S+	40.0	.67

* Total - weight basis

** Dry basis

Table 4. Continued

C-1-1	12.3	2000	W	44.2	.77
2	"	3000	S-	46.1	.80
3	"	4000	S-	45.5	.79
4	"	2000/1 min.	F-	42.6	.74
C-2-1	12.5	3000	F	45.6	.80
2	"	3000	F	48.6	.85
3	"	4000	W	45.6	.80
4	"	4000	F	47.5	.83
C-4-1	13.2	2000	W-	45.7	.81
2	"	3000	W+	47.3	.84
3	"	4000	F-	49.7	.88
4	"	6000	S-	50.3	.89
D-1-1	13.4	2333	W	52.9	.92
2	"	3000	F+	54.7	.95
3	"	4000	S-	51.7	.90
4	"	5000	S+	53.7	.93
D-2-1	12.2	1500	W-	53.5	.93
2	"	2000	F-	55.7	.96
3	"	3000	F	52.7	.91
4	"	4000	S-	55.0	.95
D-3-1	12.5	3000	F	-	-
2	"	4500	S	-	-
3	"	4500	S	-	-
4	"	6000	S+	-	-

The relation between level of rehydration and forming pressure for Lot B is shown in Fig. 11, where the abscissa is water content and the ordinate is forming pressure. This lot was investigated more fully than either Lot C or Lot D. Two curves are drawn in the figure. One, which we will call the strength line, divides the diagram into a region in which mechanically strong bars were obtained and another region in which weak bars were obtained. The location of the line was found by plotting a point for each bar in the lot, labeling it with its strength symbol, and drawing the line so that substantially all the strong bars fell above it and all the fair and weak bars below it.

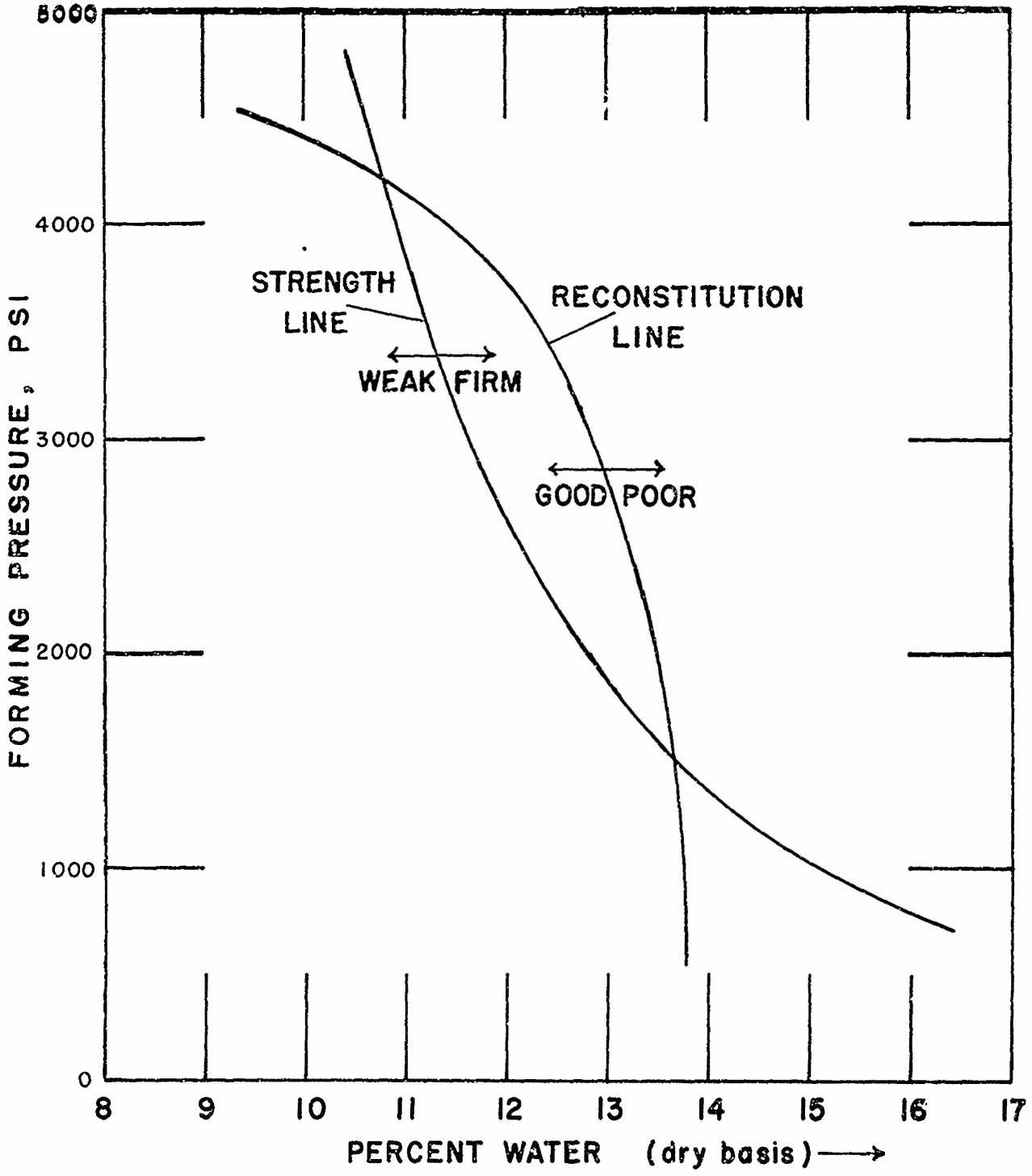

Fig. 11. Graph showing, for lot B (cooked, freeze-dried beef), the conditions under which good bars were formed. Good strength was obtained only above the strength line and good reconstitution only below the reconstitution line.

Of course the division is not actually sharp and it is best to stay some distance away from the line (above and to the right) to be sure that strong, firms bars are obtained.

The second line drawn in Fig. 11 we will call the reconstitution line. Below and to the left of this line the reconstitution, as measured by water uptake, is good. On the other side of the line reconstitution is poor. As with the strength line, the demarcation is not sharp and it is better to stay some distance below the line in order to obtain good reconstitution.

It will be seen that only a limited region between the two lines, centered at about 11.5% rehydration and 3500 psi, can give strong bars and good reconstitution. If this were the whole story, the problem of making satisfactory bars could be considered solved. However, when measurements similar to those just described were made on beef lots C and D it was found that the problem is more complicated. Besides water content and forming pressure, fat content is important in determining whether good bars can be made or not. It is also possible that other, as yet undetermined, factors have an influence, but fat content appears to be next after water content and forming pressure in importance.

Lot B had an unusually low fat content, 2.43%. Lot C, which had the relatively high fat content of 7.88%, made no really strong bars under any of the conditions tried. Lot D (5.63% fat content) was intermediate. The results obtained on these 3 lots of beef indicate that a diagram such as Fig. 11 may be drawn for any of them, but if the fat content is too high the strength line will be displaced upward relative to the reconstitution line so that the curves do not cross. When this is the case it is impossible to make reconstitutable bars that are also strong. Note that high fat content is normally found in prime and choice beef, and lower fat content in good and standard grades; the lower the quality the better the chance of making satisfactory bars. Since top quality is generally preferred in a relatively high-cost specialty food such as freeze-dried compressed beef it may be desirable to use a binder when bars are required.

4. Miscellaneous Results.

A few experiments were made in which ethanol was used as the plasticizing agent. It was found that the affinity of the freeze-dried beef for ethanol was less than for water and that vapor-phase transfer of the alcohol to beef was slower. In fact we did not get above 5.3% (dry basis) and got very weak bars. By sprinkling ethanol on the material, a content of somewhat over 15% was achieved and better bars were made, but certainly no better than those made with water. The experiments were performed on beef Lot C, which had the high fat content of 7.88%. This lot did not make good bars with either water or ethanol, but for this lot at least it was judged that ethanol was somewhat inferior to water as a plasticizer.

An experiment was made in which a specimen of pure ice was freeze-dried in the apparatus of Fig. 1. In reference [11] a theory of freeze-drying is developed and tested by applying it to experimental results. It was thot that the application of the same theory to the freeze-drying of pure ice would confirm the validity of this theory. When pure ice is freeze-dried the entire specimen shrinks gradually to zero, whereas when a food is freeze-dried there is only a small shrinkage. An ice specimen weighing 4.823 grams was prepared and dried, and the rate of weight loss computed by our usual method [11]. The results were plotted and compared with those for a sphere of frozen beef (96S) that had an initial weight of 5.017 g. The drying time of the pure ice was 8.1 hours and that of specimen 96S was 7.4 hours. The curves of weight loss per hour ($\dot{m}$) were roughly parallel. The overall value of $\dot{m}$ for the pure ice was 0.60 g/hr; for the frozen beef, 0.48. The ice specimen was not spherical. Estimating it to be a prolate spheroid with a/b = 4/3, its surface area could be determined from its observed weight. The emissivity of ice was taken to be 0.96 and the thermal conductivity of water vapor (k_v) was calculated from the experimental results. Reasonable values of k_v were obtained, rising from 0.193 mW/cm. K to 0.217 and then falling to 0.202 during the main part of the experiment. This result is taken as confirmation of the theory of reference [11].

5. Discussion.

Freeze-dried beef can be plasticized for compression by partial rehydration in which water is transferred to the beef in the form of vapor. When the rehydration was performed in a small all-glass apparatus it took 2.5 hours to rehydrate to the desired level of 11 or 12% moisture. When rehydration was performed in the small commercial dryer the time required was 4 to 5 hours. The longer time as compared with the all-glass apparatus was probably due to greater build-up of non-condensibles by desorption from the chamber and shelves and by release from the meat. It might be possible to speed up the transfer by a quick pump-out of both vapor and non-condensibles about half way thru the rehydration. This was not tried because the pumping would remove an unknown amount of water and we would not be able to stop at a known water content.

On the basis of both the small-scale and the larger-scale experiments, we believe that plasticizing can be carried out without removing the beef from the freeze-dryer, provided the dryer contains no significant leaks and is clean. A tight valve must be available to isolate the drying chamber from the condenser and the vacuum pump. Also, a water supply system must be connected to the chamber; vapor is formed in this system and flows from it into the chamber. The rehydration could be carried out in 4 or 5 hours, and probably in a shorter time (2 or 3 hours) after the technique was perfected.

6. Addendum: Plasticizing by Spraying.

While the manuscript of the present report was being prepared, the authors decided to make a test of plasticizing of freeze-dried beef by spraying. Cooked, diced, freeze-dried beef was sprayed with a fine spray immediately after removal from the freeze-dryer. It was spread in a thin layer on a sheet of aluminum foil for the spraying. After estimated portions of the water had been sprayed on, the batch was weighed and stirred; this process was repeated until water equal to 11.5 to 12% of the dry weight had been added. The plasticized beef was then put into a jar and covered. After various time intervals suitable amounts of the beef were removed and immediately pressed into bars using a pressure of 5000 psi and a dwell time of 20 sec. Times of 15 minutes, 30 minutes, and 1 hour after moistening and before pressing were tried. All gave satisfactory bars, judged equal in strength and other qualities to bars made after a 2 or 3 day waiting period or to bars made after vapor-phase rehydration.

The sprayed material reconstituted just as well as (or better than) bars plasticized in other ways.

No confirmed explanation of the results can be given at this time. In comparing the procedures that yielded good bars after spraying with those that did not, one possible difference is the time allowed to elapse after freeze-drying and before spraying. It is possible that if the freeze-dried material stands for many hours before it is sprayed, it must also stand for many hours after spraying before it is compressed. However, this hypothesis should not be accepted without confirmation. What we have found is that if certain procedures are followed both of these waiting periods can be essentially eliminated.

It should be pointed out that the results mentioned in this addendum make vapor-phase plasticizing much less important than it would otherwise be. In applications, spraying should be tried first. Then if, in some situations, spraying is unsatisfactory, vapor-phase rehydration is available as an alternative.

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