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Introduction and Review of Literature

THE GENERAL principles of packing dry whole milk in inert gas have been described by Lea, Moran, and Smith *(6)*. If a can is evacuated to "x" millimeters of mercury, and an inert gas such as nitrogen then admitted until the can is again at atmospheric pressure, the percentage of oxygen in the gases in the can may be computed by the following formula:

$$\text{Volume per cent oxygen} = \left(\frac{x}{760} \times 20.9\right) + \text{per cent oxygen in nitrogen}$$

The calculated oxygen percentages after various evacuation pressures, assuming pure nitrogen, are as follows:

Pressure to which evacuated (mm.)	1	5	10	20	30	40	50
Volume per cent oxygen in gases	0.0275	0.1375	0.275	0.550	0.825	1.100	1.375

Theoretically, therefore, the ability of the pump to evacuate the system would appear to be the principal limiting factor in reducing the oxygen content in inert gas-packing. Actually in gas-packing dry whole milk, although the percentage of oxygen in the gases in the can may closely approximate the theoretical immediately after gassing, it increases gradually over a period of several days. This phenomenon was called "desorption" by Lea *et al. (6)*, and this term has become common in the industry, although it probably does not correctly designate what occurs. Lea *et al. (6)* found that "desorption" was essentially complete in five days. The Subsistence Research and Development Laboratory of the United States Army Quartermaster Corps has adopted standards for oxygen percentages in gas-packed dry whole milk based on analyses made seven days after packing *(9)*.

Roller-dried whole milk powders "desorb" very little oxygen, but spray-dried powders "desorb" a considerable although somewhat variable amount. Palmer and Dahle *(8)* have shown that spray-process powder particles, either pressure or centrifugal, enclose bubbles of gas, whereas roller-process powder particles do not. Lea *et al. (6)* computed the volume of the entrapped air per gram of powder from the amount of oxygen "desorbed," but presented no direct evidence of the relation of the amount of "desorbed" oxygen to the volume of the air cells. It seems entirely probable, however, that the "desorption" of oxygen from dry whole milk after evacuating and gas packing is due to the slow diffusion of the oxygen from the entrapped air cells although actual adsorption of the gases on the powder particle as well as solution of the gases in the fat or moisture of the dry milk may be factors.

Hetrick and Tracy *(4)* have established that variations in the manufacturing procedure may account for some differences in the amount of retained oxygen when a standard procedure for gas-packing is used. High spraying pressure and small nozzle orifice produced powder which retained more oxygen than that produced using low pressure and a larger orifice. The amount of retained oxygen decreased with increase in the total solids of the condensed milk sprayed. Dry milk packed from the drier at a temperature of approximately 120° to 125° F. (48.7° to 51.7° C.) retained less oxygen than dry milk cooled to 60° F. (15.5° C.) before packing. Aging the powder for 18 to 24 hours before packing increased the amount of retained oxygen.

Gane *(3)* had previously shown that with centrifugal spray powders the amount of "desorbed" oxygen decreased as the solids content of the milk dried was increased.

Lea *et al. (6)* appear to have established that oxidative deterioration of dry whole milk does not progress to a point sufficient to affect the palatability of the product if the atmosphere in which the powder is stored contains no more than 0.01 ml. of oxygen per gram of powder. They considered that, for a commercial pack, levels of up to 0.05 ml. of oxygen per gram of powder might be accepted as satisfactory, but that lower levels might be desirable. Their data show that even when a gas-tight cabinet, a good vacuum, and pure nitrogen are used, so that the "initial" concentration of oxygen in the free-space gas is less than 0.1 per cent, the particles of spray-dried powder will still retain sufficient oxygen to raise the oxygen content of all the gas in the can to 2 or 3 per cent or even more. Since an oxygen content of 3 per cent in the free-space gas is equivalent, in cans packed with the normal amount of powder, to about 0.03 ml. of oxygen per gram of powder, some other method of gassing must be used if the oxygen content of the canned powder is to be reduced to desirable limits. By regassing after "desorption" is more or less complete, they have shown that the oxygen content may be consistently reduced to 0.01 ml. per gram or less. A similar principle is taken advantage of by Merrell and Schibsted *(7)* in a process involving the holding of the powder under vacuum for 20 hours before the final gassing.

When this work was started, differences were known to exist, for no apparent reason, in the amount of oxygen retained by gas-packed powders from various driers. An explanation for these differences was sought. Data were lacking on the rate of "desorption" or "diffusion" of the oxygen from dry milk. Such data were desired because of their value in establishing desirable time intervals for double gassing or vacuum holding. Whether the use of carbon dioxide would be more advantageous than nitrogen for double gassing was not known. Finally, the uncertainty of the mechanism by which the oxygen is retained by dry whole milk made a study thereof desirable.

Methods

SOURCE OF POWDER

Dry whole milks for use in this study were secured from the following sources:

1. Dry whole milk manufactured in the University of Minnesota experimental spray drier (Coulter, *1)*. This drier utilizes a two-fluid atomizer in which the milk is atomized with compressed air.
2. Dry whole milk from two type 1 pressure spray driers.
3. Dry whole milk from two type 2 pressure spray driers.
4. Dry whole milk from a commercial centrifugal spray drier.

METHOD OF GASSING

The equipment used is shown in figure 1. The empty gassing chamber could be evacuated with the Cenco Hypervac pump to 1 micron or below. When loaded with 12 No. 2 cans of powder, the chamber could be evacuated to 5 mm. in 2.5 minutes or less. The powder in most instances was packed in No. 2 cans in the amount of 325 g. per can. This closely approximates the volume per weight ratio of the standard 5-pound can. Sufficient preliminary work was done with the 5-pound can to indicate that results with the smaller can were comparable. Before evacuation, a 1.5 mm. hole was punched in the sealed can. After evacuating to the pressure and holding for the time desired, nitrogen was admitted to the chamber until a positive pressure of 2 pounds was developed. After 30 seconds the chamber was opened and the holes in the cans soldered. The nitrogen used was tested for purity and found to contain not more than 0.1 per cent oxygen.

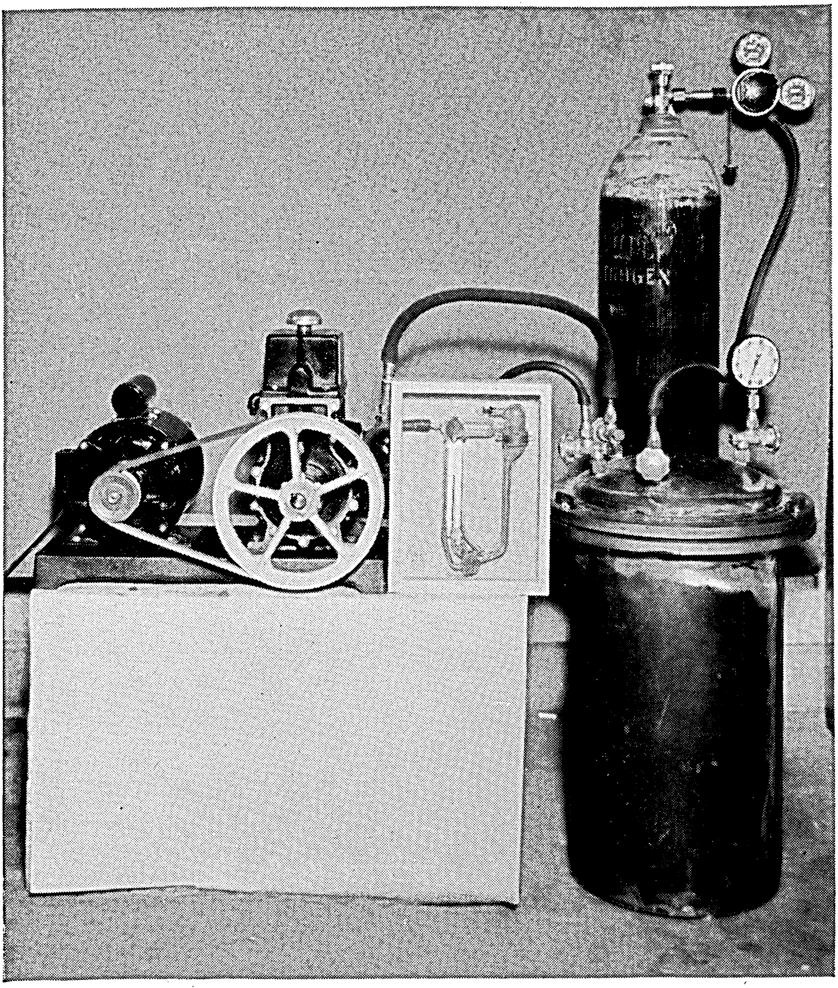


FIG. 1. Equipment used for packing dry whole milk in inert gas

GAS ANALYSIS

Analyses for oxygen in the head-space gas were made with a Fisher Precision Gas Analyzer equipped with a Continental Can Company sampling device. Since in many cases it was desired to express the oxygen content of the gas in the can as ml. (S.T.P.) per 100 g. of powder, a determination of the pressure in the can was necessary. This was done with the arrangement shown in figure 2, in essentially the manner described by Lea *et al.* *(6)*. The tube A was evacuated, stopcock B was closed, the can was punctured, and the resulting pressure was read on the manometer. A sample was then drawn into the burette for analysis. Knowing the volume of the tube A, the volume (S.T.P.) of oxygen per 100 g. of powder was calculated by the following formulae:

$$X = NV_1 \times \frac{P_2}{P_3} \times \frac{273}{298} \times \frac{P_1}{760} \times \frac{100}{W} \qquad (1)$$

$$V_1 = \frac{P_3 \times V_2}{P_1 - P_3} \qquad (2)$$

where:

X = ml. oxygen (S.T.P.) per 100 g. powder
N = Volume per cent oxygen in free gas in can
V_1 = Volume (ml.) of free space in can
V_2 = Volume (ml.) of tube A
P_1 = Barometric pressure (mm. of Hg.)
P_2 = Pressure (mm. of Hg.) after puncturing gassed can
P_3 = Pressure (mm. of Hg.) after puncturing can at atmospheric pressure
W = Weight (g.) of powder in can

It will be noted that this calculation does not involve the actual pressure in the can before puncturing. Whenever this information was desired it was calculated as follows:

$$\text{Pressure in can (mm. Hg.)} = P_1 \times \frac{P_2}{P_3}$$

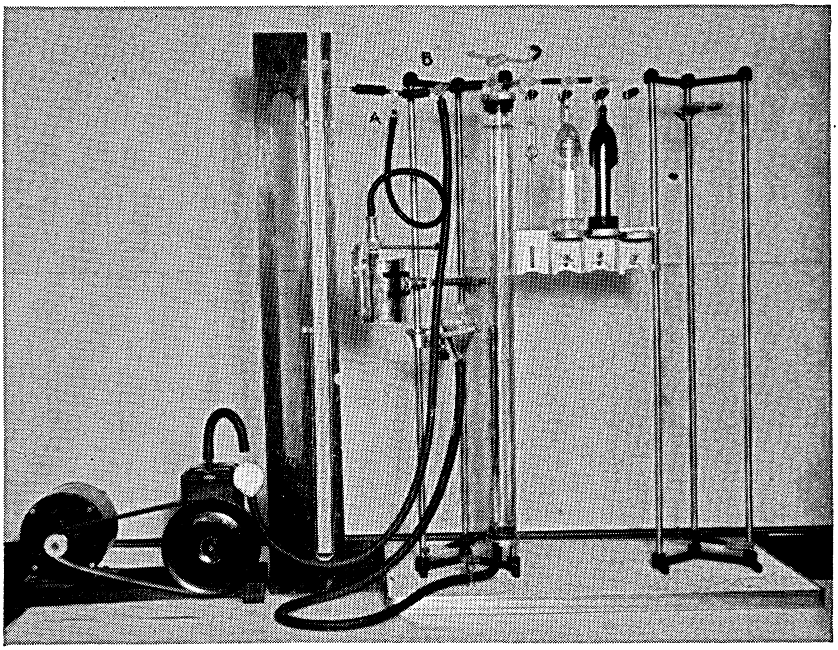


FIG. 2. Equipment for gas analysis

In the few cases in which carbon dioxide was the inert gas used, analyses were made for it as well as for oxygen. It was necessary to dilute the sample with nitrogen in these cases.

As a routine procedure, all cans were analyzed on the seventh day after gassing. It will be shown later (figure 7) that for all practical purposes equilibrium is attained in this length of time.

Experimental Results

RELATION OF VARIATIONS IN THE POWDER PARTICLE TO THE EFFICIENCY OF GAS-PACKING

Influence of the Type of Drying System

The oxygen contents of cans of dry whole milk from a variety of sources as determined seven days after gas-packing are shown in table 1. All powders were held after manufacture for 48 hours or more before gas-packing. Powders from three sources were used in the initial phases of the study. These were sample A, plant 1 (type 1 pressure spray), sample A, plant 3 (type 2 pressure spray), and the regular powder from the University of Minnesota experimental drier. It soon became apparent that there were large differences in the oxygen content of the free-space gas in the cans of the various powders. The cans of powder from the type 1 pressure spray drier consistently had the highest equilibrium oxygen level, and those of the University of Minnesota powder the lowest. The powder from the type 2 pressure spray drier gave appreciably lower oxygen values than that from the type 1 drier. Similar results were secured with other lots of powder from the same sources (samples B, plants 1 and 3), and with powders from different driers of types 1 and 2 (samples A, plants 2 and 4).

Since Hetrick and Tracy *(4)* and Gane *(3)* have pointed out that the degree of precondensing of the milk influences the amount of oxygen "desorbed" from the dry whole milk, this might be assumed to be an explanation for the differences among these powders. Information provided by the manufacturers, however, indicated that the total solids contents of all the milks at the time of spraying were within the range from about 35 to 40 per cent.

In attempting to find an explanation for the wide variations in the amount of retained oxygen, the powders were examined microscopically in mineral oil suspension. Palmer and Dahle *(8)* detected air cells in the powder by mixing the dry milk with a small amount of water on a slide. This method has the obvious

Table 1. Influence of Type of Drying System on Air Entrapment and Gas-Packing Efficiency

Sample	Type of drier	Microscopic measurements			Oxygen content after 7 days*				
						Oxygen as			
						Per cent of gas		ml./100 g. powder	
		No. of particles measured	Particles containing air cells	Cell volume per g.	No. of determinations	Mean	S.D.†	Mean	S.D.†
			Per cent	ml.					
A, Plant 1	1	....	....	....	13	3.85	0.28	....	....
B		481	89.4	0.176	8	3.24	0.56	2.67	0.47
C		448	56.2	0.021	4	2.38	0.15	2.07	0.13
A, Plant 2	1	705	59.3	0.072	4	3.76	0.21	3.08	0.22
B		454	48.9	0.013	2	1.95	....	1.82	....
		....	....	....	.....	2.12	....	1.94	....
A, Plant 3	2	....	....	....	13	2.51	0.30	....	....
B		1,165	38.5	0.040	7	2.30	0.46	2.09	0.28
A, Plant 4	2	1,008	30.2	0.043	4	2.14	0.38	1.80	0.32
127, U. of M.	Regular	1,023	25.0	0.020	4	1.87	0.26	1.78	0.35
138, U. of M.	Air whipped into condensed	1,050	31.3	0.140	2	3.25	....	3.14	....
		....	....	....	.....	3.39	....	3.26	....
A, Plant 5	Centrifugal	....	....	....	4	3.10	0.38	2.40	0.30

* Packed in No. 2 cans. Weight was 0.50 g./ml. for U. of M. powders and 0.54 g./ml. for other powders. Gassed with nitrogen after evacuation to 0.5—5.0 mm. pressure.

† Standard deviation of single determination.

disadvantage that the dry milk soon disperses in the water. The air cells are as readily discernible upon mixing the powder with mineral oil in which the particles are not dispersible.

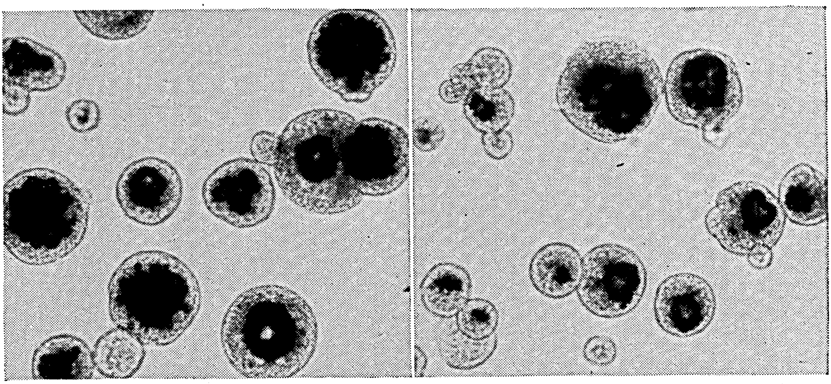
Photomicrographs of a variety of milk powders are shown in figures 3 and 4. The marked differences in the size and number of air cells and thus in the volume of entrapped air in the powders are readily apparent. Each of the authors independently examined a large number of microscopic fields and measured the size of all powder particles and air cells seen. The cell volumes per 100 g. of powder were computed from these data. Since the results were reasonably consistent for any one powder, the mean cell volumes and the percentages of cell-containing particles shown in table 1 are based on the total number of measurements. These values cannot be considered as exact but do reflect the differences among the powders. It is evident from the comparisons shown in table 1 that the calculated cell volumes per gram and the seven-day oxygen analyses are roughly proportional.

A much larger proportion of the powder particles from the type 1 driers (sample B, plant 1 and sample A, plant 2) contained air cells than either those from the type 2 driers (sample B, plant 3 and sample A, plant 4), or from the University of Minnesota drier. Furthermore, although most of the particles from the type 2 driers or the University of Minnesota drier containing air cells had only single cells, many of the particles from the type 1 driers had clusters of cells. The cell volumes per gram of powder were much the largest in the powders from the type 1 driers.

The presence of cell clusters in the powder particles suggested that there might have been foam in the milk at the time of spraying. That this is a reasonable explanation appears to be verified by results secured by whipping air into the condensed milk with a malted milk mixer prior to spray drying in the University of Minnesota drier. As shown in figure 4 (upper right) many of these powder particles contained cell clusters, and from table 1 it may be noted that both the cell volume per gram and the oxygen retained by the gas-packed powder were much greater than for the regular University of Minnesota powder.

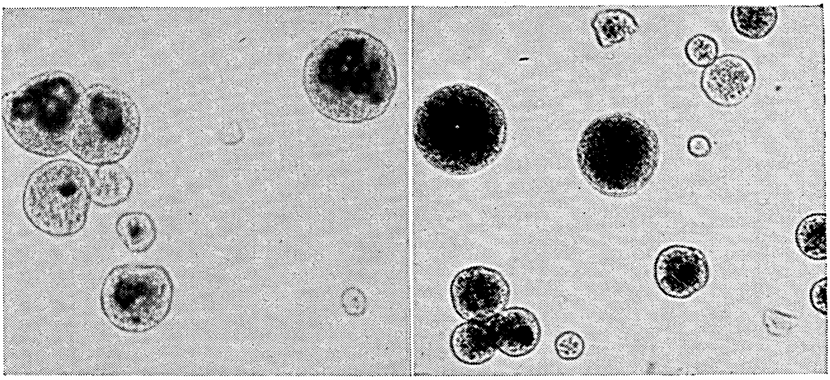
The application of this principle (i.e., removal of foam before spraying) to commercial operation in the type 1 driers was very successful in reducing the amount of entrapped air (sample C, plant 1 and sample B, plant 2) (see figure 3 and table 1). Thus there appears to be no significant difference in the amount of entrapped air in powders from the type 1 and 2 driers if milk equivalent in solids content and in air content is sprayed.

Lea *et al.* *(6)* presented data showing that centrifugal spray



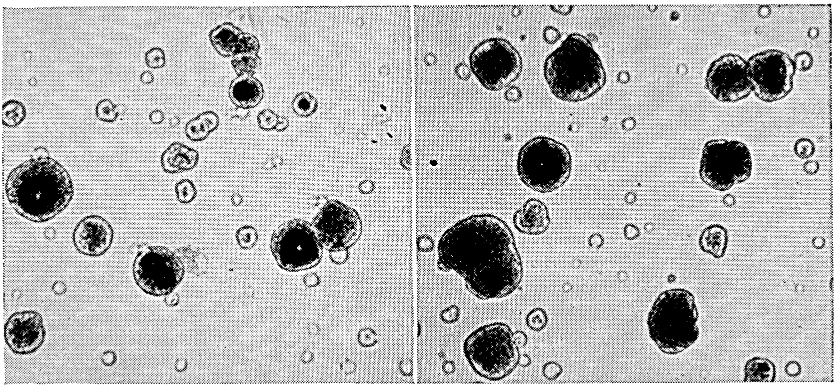
PLANT 1, TYPE 1 DRIER, PRESSURE SPRAY
SAMPLE B

PLANT 2, TYPE 1 DRIER, PRESSURE SPRAY
SAMPLE A



PLANT 1, TYPE 1 DRIER, PRESSURE SPRAY
SAMPLE C, CONDENSED MILK, DEAERATED

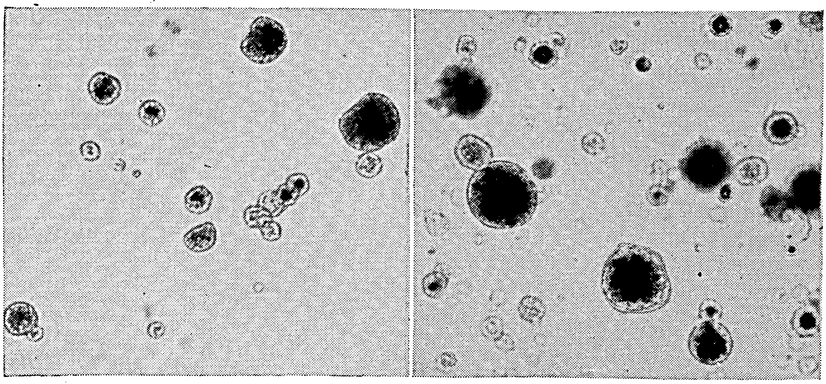
PLANT 2, TYPE 1 DRIER, PRESSURE SPRAY
SAMPLE B, CONDENSED MILK, DEAERATED



PLANT 3, TYPE 2 DRIER, PRESSURE SPRAY
SAMPLE B

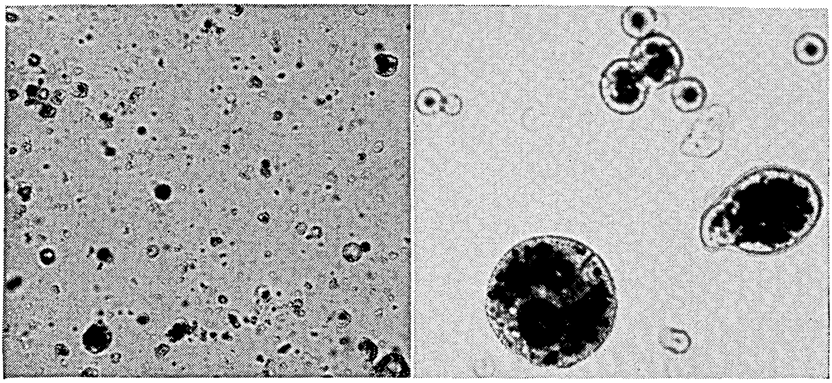
PLANT 4, TYPE 2 DRIER, PRESSURE SPRAY
SAMPLE A

FIG. 3. Photomicrographs of whole milk powder particles



U. OF M. DRIER, TWO-FLUID ATOMIZER
SAMPLE 127

U. OF M. DRIER, TWO-FLUID ATOMIZER
SAMPLE 138, CONDENSED MILK, WHIPPED



U. OF M. DRIER, TWO-FLUID ATOMIZER
SAMPLE 127, GROUND

PLANT 5, CENTRIFUGAL SPRAY
SAMPLE A

FIG. 4. Photomicrographs of whole milk powder particles

powder "desorbed" more oxygen than pressure spray powder. Included in table 1 are data secured with centrifugal spray powder manufactured in one plant. A photomicrograph of a sample of this powder is shown in figure 4. Although there is no certainty that these results are typical for all centrifugal powders, they do indicate that such powders contain considerable quantities of entrapped air and retain an amount of oxygen roughly proportional to this volume.

Effect of Grinding the Powder

The finding that the quantity of retained oxygen is correlated, in a general way, with the number and size of air cells entrapped in the powder, suggested the possibility of improving the efficiency of gas-packing by grinding the powder to disrupt the par-

Table 2. Effect of Grinding Powder on Efficiency of Gas-Packing

	Oxygen content (S.T.P.) after 7 days*	
Sample	Control	Ground†
	ml./100 g.	ml./100 g.
B, Plant 1	2.20	1.06
	2.15	
B, Plant 3	1.94	0.34
	1.76	
127, U. of M.	1.55	0.59
	1.43	
150, U. of M.	1.82	0.65
	1.78	

* All cans evacuated to 0.5 mm. and gassed with nitrogen.

† Ground 4 hours in a ball mill.

ticles. Table 2 affords a comparison of oxygen retention in ground and unground powders. In every case a decided lowering of the equilibrium oxygen level was effected by the grinding treatment. Figure 4 shows the appearance under the microscope of University of Minnesota sample 127 after grinding. It is evident grinding appreciably reduced the relative volume of entrapped air.

These results are rather contrary to the idea that any large portion of the retained oxygen originates in truly adsorbed layers, since it would be expected that the greatly increased surface produced by grinding would enhance adsorption. While grinding itself probably is not a practical means of reducing the oxygen level, it may be concluded from this experiment that any process whereby cell-less or disrupted particles are produced would be advantageous from the standpoint of gas-packing efficiency.

Influence of Age of the Powder

The data in table 3 confirm the observation of Hetrick and Tracy *(4)* that powder packed immediately after drying at 120° to 125° F. (48.7° to 51.7° C.) retained less oxygen than powder aged 18 to 24 hours before packing. This suggests an increase in the oxygen content of the powder on holding or a change in the permeability of the powder particles on cooling or aging.

At least two factors may operate to increase the oxygen content of the powder particle on cooling and aging. As the powder particle cools, the entrapped air must also cool, thus creating a partial vacuum into which additional air may be drawn. Also the air in the particle at the time of drying must be saturated with water vapor. The pressure of the water vapor will be decreased not only by cooling, but by adsorption of the water vapor by the milk powder whether or not cooling occurs. Thus an approximation of the total increase in the oxygen content of the powder on cooling and aging may be arrived at by computation. As a basis

Table 3. Comparative Efficiencies of Gas-Packing Dry Whole Milk Immediately after Drying and after Two Days*

Sample	Fresh-warm powder				Two-day-old powder			
	Time to reach 0.5 mm.	Oxygen after 7 days†		Moisture†	Time to reach 0.5 mm.	Oxygen after 7 days†		Moisture†
	Minutes	Per cent	ml./100 g.	Per cent	Minutes	Per cent	ml./100 g.	Per cent
U. of M.								
128	11.50	1.97	1.80	1.42	5.25	1.57	1.55	1.98
129	14.00	2.06	1.88	2.15	5.25	1.45	1.42	2.61
130	11.75	1.06	0.95	1.60	5.25	1.76	1.73	2.18
132	25.00	1.56	1.17	2.20	9.00	1.73	1.43	2.72
134	12.00	0.46	0.52	3.72	11.00	1.14	1.36	4.42
135	7.50	0.53	0.60	4.57	11.00	1.16	1.40	4.98
136	7.50	1.00	0.86	2.14	11.00	1.93	2.00	2.56
137	10.50	1.58	1.41	1.80	6.00‡	2.18	1.97	1.77
138	6.00	2.34	2.15	1.12	6.00‡	3.32	3.20	1.07
139	15.00	1.80	1.60	2.28	18.00	2.52	2.19	2.60
140	14.25	1.46	1.33	2.67	18.00	1.85	1.72	3.29
149	6.00	1.56	1.48	1.46	5.50	2.30	2.46	1.41
150	9.00	1.48	1.66	3.40	5.50	1.58	1.80	3.40
151	5.00	1.72	1.46	2.64	4.00	1.70	1.93	2.80
153	9.00	0.90	1.35	2.29	9.00	1.26	1.91	2.55

* All evacuated to 0.5 mm. Held in vacuum chamber for a total of 25 minutes from start of evacuation.

† Figures are averages of duplicates.

‡ Three-day-old powder.

for calculation it may be assumed that the powder coming from the drier is at 110° F. (43° C.), and that the air entrapped in the powder particle is saturated with water vapor at a total pressure of 760 mm. The vapor pressure of water at 110° F. is 64.8 mm. Thus the pressure in the powder particle due to air is only 695.2 mm. In being cooled to 68° F. (20° C.) the pressure of the air would be reduced to 643.8 mm. Based on the data of Holm and Greenbank *(5)*, the vapor pressure of the water in spray-dried whole milk powder with a moisture content of 2.5 per cent is about 1.5 mm. Thus after cooling and aging, the total pressure within the air cells resulting from the original air and the vapor pressure of water would be 645.3 mm. If air were drawn into the air cells to increase the pressure to 760 mm., the air content of the powder would be increased $\frac{(760 - 645.3) \times 100}{645.3}$ or 17.8 per cent. This result furnishes at least a partial explanation for the increases reported in table 3.

Studies of the Mechanism of Air Retention by Dry Whole Milk

Knowledge of the mechanism by which gas is retained would greatly facilitate attempts to reduce the oxygen level in milk powder. Results of the following experiments furnish some information on this point.

It is known that the oxygen level in cans of spray-dried egg powder does not increase after evacuation and gassing. The seven-day oxygen analysis of the head-space gas in a sample of egg powder gas-packed by the authors was under 0.1 per cent. A photomicrograph of this egg powder is shown in figure 5. The large number and volume of the air cells in this powder are readily apparent. That egg powder does not "desorb" oxygen cannot be due, therefore, to the lack of entrapped air. The difference between dry whole milk and egg powder in this respect appears to be due to the relative impermeability of the milk powder particle to gases. Troy and Sharp *(11)* and Sharp *(10)* have shown that the lactose in spray-dried milk is in the form of a highly concentrated sirup

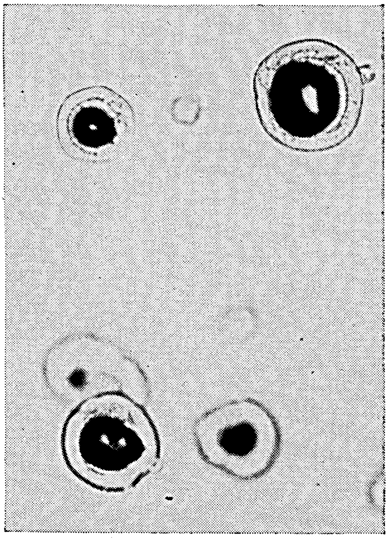


FIG. 5. Photomicrograph of egg powder particles

(glass). This lactose glass undoubtedly constitutes the continuous phase of the milk powder particle and it is reasonable to suppose that it is much less permeable to gases than the proteins of the egg powder particle.

Assuming that the gases within the cells of the milk powder particles are at atmospheric pressure, the cell walls must be able to withstand an internal pressure of about 15 pounds per square inch to retain the gases within the cells during evacuation. To test the hypothesis that it is the relative impermeability of the lactose glass of the powder particle which is responsible for the failure of the bulk of the oxygen to be released immediately when the powder is placed in a high vacuum, eggs were dispersed in a lactose sirup of such concentration that the lactose was present in approximately the same ratio to the egg solids that it bears to the other milk solids in milk, i.e. 5:7.5. The resulting mixture, which had a solids content of about 35 per cent, was spray dried in the University of Minnesota experimental drier. A high proportion of the resulting powder particles contained air cells. This powder was gassed using the standard procedure and after seven days the head-space gas in the cans had an oxygen content of 3.35 per cent. A similar sirup-dried product was prepared using sucrose rather than lactose. The seven-day oxygen content of the head-space gas in cans of this powder was 1.00 per cent. Based on microscopic appearance there was little difference in the volume of entrapped air in the two powders. Presumably, therefore, the difference in the amount of retained oxygen resulted from the more complete removal of the air from the air cells of the sucrose containing powder during evacuation due to the greater permeability of the particles. Lea *et al. (6)* have shown that spray-dried whole milk powder containing 40 per cent of sucrose "desorbed" only about half as much oxygen as unsweetened powder.

The possibility that the permeability of the glass might vary with its moisture content was investigated. A sample of powder was held in a vacuum in a desiccator over phosphorus pentoxide until its moisture content was reduced to 0.20 per cent. Dry air was then admitted and equilibration allowed for 24 hours. The powder was packed in dry cans, evacuated to 0.5 mm., and gassed with nitrogen. After seven days the oxygen content of the free-space gas was 1.86 per cent; in the control powder at a moisture content of 1.75 per cent, it was 2.00 per cent. This result is evidence that the permeability of the powder particle wall is not altered appreciably by dessication.

Lea *et al. (6)* indicate also that "desorption" is slower from skim milk than from whole milk powder. We have noted little

Table 4. Reduction of Oxygen Level by Successive Gassings

Sample	Successive gassings*						
	1	2		3		4	
	Oxygen	Oxygen	Reduction in oxygen	Oxygen	Reduction in oxygen	Oxygen	Reduction in oxygen
	ml./100 g.	ml./100 g.	Per cent	ml./100 g.	Per cent	ml./100 g.	Per cent
B, Plant 1	69.50	11.14	84.0	1.96	82.5	0.64	67.3
B, Plant 2	74.20	7.65	89.7	0.90	88.3	0.42	53.2

* First gassing was with oxygen; the other three with nitrogen. An equilibration period of 7 days was allowed between gassings. All figures are averages of duplicates.

difference in this regard between skim milk and whole milk powders. Spray-dried 35 per cent cream, however, was found to "desorb" very little oxygen. The seven-day oxygen analysis of cans of cream powder evacuated and gassed by the standard procedure was only about 0.3 per cent. This might be because the large amount of fat in the cream particles renders the particles highly permeable to air; however, on microscopic examination relatively few of the particles were found to contain cells.

The failure of the dried cream and the dried eggs to retain appreciable amounts of oxygen, as well as the reduction in the amount of oxygen retained by whole milk powder on grinding, is evidence that adsorption is not a major factor in the retention of oxygen by the particles.

If adsorption does not contribute to the retention of oxygen, the percentage reduction of oxygen should be constant for each of a series of gassings. If, on the other hand, much oxygen is adsorbed, the percentage reduction would decrease with each successive gassing of a series. An experiment designed to test this point is reported in table 4. The cans were first gassed with oxygen and then three successive times with nitrogen. The percentage reduction of oxygen was constant until the last gassing which yielded a smaller reduction. This result indicates that a small portion, but only a small portion, of the oxygen may be adsorbed.

Solution of gases in the fat cannot be of great importance because neither dried cream nor dried eggs, which contain large amounts of fat, gives off much oxygen after evacuation and gassing. All available evidence, therefore, indicates that "desorption" of oxygen from the dry whole milk is due principally to diffusion of air actually entrapped in cells within the particles.

Sources of Air Cells in Dry Whole Milk

The exact source of the air cells within the powder particles can at present only be conjectured. As has been pointed out, some of the cells may originate from foam in the milk at the time of spraying. Efforts to produce cell-less powder particles by deaerating the milk before spray-drying in the University of Minnesota drier were unsuccessful. The resulting powder retained about the same amount of oxygen after gas-packing as the regular powder, and appeared to contain about the same volume of air cells.

Folger and Kleinschmidt *(2)* have presented high speed photographs of the formation of the powder particles during spray drying. With pressure spray and centrifugal atomizers the milk is dispersed in thin, rapidly rotating sheets which break up and form spherical droplets under surface tension forces. When some materials, as for example tallow, are sprayed into cold air, almost all of the particles are hollow spheres. With two-fluid atomizers such as the one used in the University of Minnesota drier, the milk is torn by the impinging air stream into filaments or threads, from which fragments break off and are quickly reoriented by surface tension forces to form spherical droplets.

We have noted that, regardless of the method of spraying, a large proportion of the particles fail to entrap any air. Why this should be true is not clear, but there is no indication that the air cells are formed in any of the particles by expansion of water vapor in the particle during drying. If air cells were formed by the latter process each powder particle should contain a cell.

STUDY OF METHODS OF GAS-PACKING

Influence of the Extent of Evacuation

It has been pointed out that powders differ in the extent to which oxygen is retained after gassing. The study recorded in figure 6 and table 5 was made to ascertain whether these characteristic differences apply regardless of the extent of evacuation before gassing, and whether evacuation to very low pressures is a practical means of reducing the oxygen level. It can be seen in figure 6 that, in the range 0.5 mm. to 50 mm., the regression of equilibrium oxygen level on pressure follows the slope of the theoretical line for empty cans rather closely. Differences in ordinates at any pressure presumably indicate differences in amounts of occluded oxygen. These differences among the three powders studied are consistent over this range.

It is rather puzzling that the values reported for empty cans should fall no closer to the theoretical. It should be noted that each of these particular empty cans was gassed in the chamber simultaneously with 11 cans of dry whole milk. When empty cans were gassed alone, very much better agreement with theory was obtained. The reason for the discrepancy is not apparent.

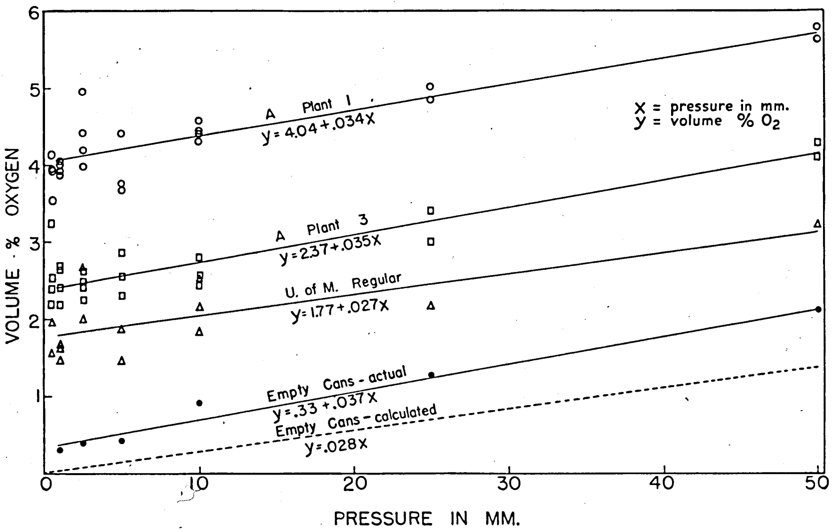


FIG. 6. Influence of extent of evacuation on equilibrium oxygen level in cans of dry whole milk gassed with nitrogen

When evacuating to pressures below 0.5 mm., sufficient periods of time are involved to allow considerable diffusion to occur before the final pressure is reached. For this reason it seemed desirable to make a correction for the effect of time, using coefficients obtained in an experiment to be described later (see figure 12). In table 5 several such corrected values are compared with the uncorrected results. The effect of time is apparent, particularly for powder A, plant 1.

Evacuation to exceedingly low pressures is obviously neither a practical procedure nor, alone, an effective method of reducing the oxygen level. The apparent lowering of the level in some cases merely reflects the amount of diffusion in the time required to reach low pressures. Under practical conditions time and expense would more than offset the slight increment of oxygen removed below a pressure of about 5 mm.

Nitrogen vs. Carbon Dioxide

Results of a comparative study of nitrogen and carbon dioxide as inert gases are recorded in table 6. Since carbon dioxide is absorbed by the powder, the pressure of gas within the can was diminished greatly after seven days. This diminished pressure accounts for the fact that the oxygen percentage was higher in the carbon dioxide-gassed cans than in those gassed with nitrogen. Except for sample B, plant 1, age 1 week, the volume of oxygen per 100 g. of powder did not differ appreciably between the two

Table 5. Influence of Extent and Time of Evacuation on Equilibrium Oxygen Level

Pressure to which evacuated	Time of evacuation	Powder A, Plant 1		Powder A, Plant 3		Regular powder, U. of M.	
		Oxygen after 7 days		Oxygen after 7 days		Oxygen after 7 days	
		Actual	Corrected*	Actual	Corrected*	Actual	Corrected*
mm.	Min.	Per cent	Per cent	Per cent	Per cent	Per cent	Per cent
0.12	75.00	2.26	3.89	2.54	3.33	1.27	1.71
		2.37	4.08	2.45	3.21	1.12	1.51
0.20	75.00	2.40	4.13	2.00	2.62	1.82	2.45
		2.47	4.26	2.29	3.01	....	....
0.25	30.00	2.34	3.35	2.37	2.84	1.45	1.77
		....	....	2.26	2.70	1.45	1.77
	15.00	3.80	4.73	1.97	2.20	1.78	2.00
		3.70	4.60	2.29	2.56	....	....
0.50	6.00	3.95	4.10	2.40	2.44	1.55	1.58
		3.91	4.06	2.19	2.23	....	....
	8.25	3.54	3.91	3.26	3.43	1.97	2.08
		4.15	4.58	2.54	2.67	....	....

* Corrected to 5 minutes under vacuum.

Table 6. Comparison of Nitrogen and Carbon Dioxide for Single Gassing*

Sample	Age	Gassed with nitrogen			Gassed with carbon dioxide			
		Pressure in can	Oxygen		Pressure in can	Oxygen		Carbon dioxide
		mm.	Per cent	ml./100 g.	mm.	Per cent	ml./100 g.	Per cent
A, Plant 1	......	......	4.04	......	......	6.44	......	71.18
	......	......	4.00	......	......	6.64	......	71.10
B	1 week	730	4.17	3.45	490	4.35	2.41	72.89
	......	731	3.91	3.23	......	......	......	......
B	2 months	748	3.32	2.70	490	4.92	2.87	73.36
	......	748	3.36	2.73	......	......	......	......
A, Plant 3	......	......	2.64	......	......	4.38	......	......
	......	......	2.41	......	......	4.24	......	......
B	1 week	747	2.88	2.43	442	3.71	1.85	78.71
	......	747	2.95	2.49	......	......	......	......
B	2 months	771	1.98	1.84	451	4.22	2.40	89.22
	......	773	2.28	2.13	......	......	......	......
110, U. of M.	......	......	1.61	......	......	3.12	......	86.36
	......	......	1.67	......	......	3.45	......	83.28
118, U. of M.	......	730	1.99	1.98	477	2.93	1.91	80.14
	......	730	2.17	2.17	......	......	......	......
127, U. of M.	......	760	2.13	2.12	458	2.43	1.52	60.67
	......	763	2.04	2.04	......	......	......	......

* All evacuated to 1 mm. before gassing. Held 7 days before analysis.

treatments; neither gas may be said to have an advantage over the other in so far as reduction of the oxygen level is concerned. The characteristic differences among the three types of powder are unchanged.

Multiple Gassing

The fact that the air retained in the powder particle after evacuating and gassing slowly diffuses affords an opportunity further to reduce the oxygen level in the canned powder by successive gassings alternated with holding periods for diffusion to occur. Several of the factors involved in such processes have been studied in relation to the various types of powder.

DOUBLE GASSING

Influence of time interval between gassings—The fundamental factor influencing the efficiency of double gassing is the time interval allowed between the gassings. The basis for establishing a satisfactory time interval is, of course, the rate at which the retained oxygen diffuses from the powder after gassing. Results of a study of the rate of equilibration in two samples of powder are shown in figure 7. As already mentioned, these data justify the practice of analyzing after seven days, since equilibrium is established before that time.

Next, an attempt was made to formulate a general relation between oxygen diffusion and time. The rate of simple diffusion or flow of a gas through an orifice from one chamber (I), which originally contains it, to another (II), which originally does not contain it, may be expressed mathematically as follows:

$$\frac{dx}{dt} = k\left(\frac{a-x}{v_I}\right) - k\frac{x}{v_{II}} \qquad (3)$$

where:

$\frac{dx}{dt}$ = rate of increase of gas content in (II)
a = moles of gas in (I) initially
x = moles of gas in (II) at time t
$a - x$ = moles of gas in (I) at time t
v_I = volume of (I)
v_{II} = volume of (II)
k = a constant depending on the characteristics of the orifice and of the gas

Thus at any instant, the rate at which the gas concentration is increasing in (II) is proportional to the difference in pressure between (I) and (II) at that instant.

In applying such an equation to diffusion of oxygen from the particles of milk powder to the free-space, one other factor must

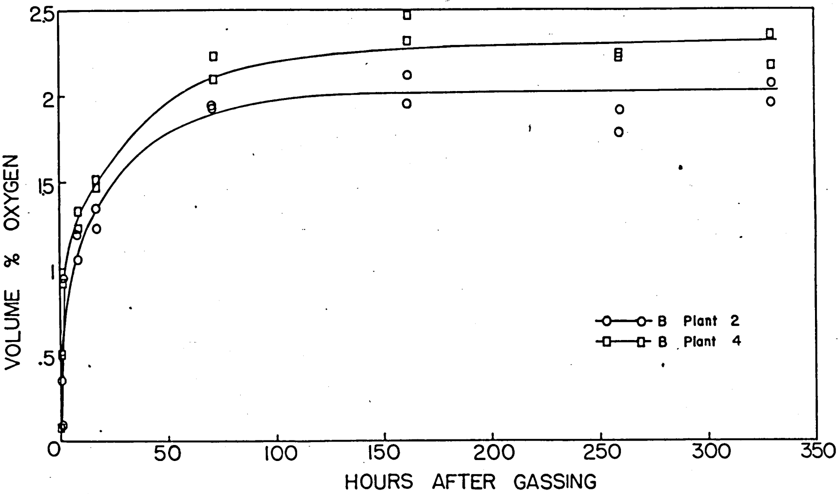


FIG. 7. The rate of equilibration of oxygen after gassing

be considered. This is the fact that the oxygen is not originally in a single chamber, but is distributed among a great many air cells of varying size in particles of varying wall thickness. Therefore, each particle may be considered as a separate chamber to which the above equation applies. The value of k (the fractional diffusion rate) would be expected to be inversely proportional to the thickness of the particle wall and hence to differ greatly among particles. The "over-all" value of k would be a resultant of values for the individual particles. Now, since various particles approach equilibrium at different rates, the value of k will not be constant over the entire period, but will decrease as the various particles become equilibrated. Since the cell wall thickness undoubtedly varies in a continuous fashion among particles, it may be assumed that k decreases uniformly as the process proceeds. In other words, k is inversely proportional to time or $k = \frac{f}{t}$, where f is a constant. It might be added parenthetically that retention of any of the oxygen by true adsorption would also be expected to cause a decrease in the fractional diffusion rate with time, because the more tightly adsorbed gas would be released more slowly. Equation (3) now may be rewritten as follows:

$$\frac{dx}{dt} = \frac{f}{v_I t}(a - x) - \frac{f}{v_{II} t}x \qquad (4)$$

or if f_1 and f_2 be defined as $\frac{f}{v_I}$ and $\frac{f}{v_{II}}$ it may be simplified to

$$\frac{dx}{dt} = \frac{f_1}{t}(a - x) - \frac{f_2}{t}x \qquad (5)$$

or
$$\frac{dx}{dt}=\frac{1}{t}(f_1+f_2)\left(\frac{f_1a}{f_1+f_2}-x\right) \tag{6}$$

or
$$\frac{dx}{\left(\frac{f_1a}{f_1+f_2}-x\right)}=\frac{dt}{t}(f_1+f_2) \tag{7}$$

Integrating and setting $x=0$, when $t=0$

$$\log\left(\frac{\frac{f_1a}{f_1+f_2}-0}{\frac{f_1a}{f_1+f_2}-x}\right)+A=\log t\,(f_1+f_2)+B \tag{8}$$

where A and B are constants of integration.

Simplifying and setting $C=A-B$

$$\log\left(\frac{a}{a-\left(\frac{f_1+f_2}{f_1}\right)x}\right)+C=\log t\,(f_1+f_2) \tag{9}$$

Now when equilibrium is reached, $\frac{dx}{dt}=0$, and if the equilibrium value of x be designated $x_{eq.}$, then equation (5) may be rearranged:

$$f_1(a-x_{eq.})=f_2x_{eq.} \tag{10}$$

or
$$a=x_{eq.}\left(\frac{f_1+f_2}{f_1}\right) \tag{11}$$

or
$$a=x_{eq.}\left(\frac{\frac{k}{v_I}+\frac{k}{v_{II}}}{\frac{k}{v_I}}\right) \tag{12}$$

or
$$a=x_{eq.}\left(1+\frac{v_I}{v_{II}}\right) \tag{13}$$

and substituting back in equation (9), the relation obtained is:

$$f\left(\frac{1}{v_I}+\frac{1}{v_{II}}\right)\log t=\log\left(\frac{x_{eq.}}{x_{eq.}-x}\right)+C \tag{14}$$

In other words a straight line relation should exist between log t and $\log\left(\frac{x_{eq.}}{x_{eq.}-x}\right)$. When the data of figure 7 are replotted on a logarithmic scale, a straight line is obtained as is shown in figure 8. In this figure log $(x_{eq.}-x)$ is plotted against log t, so that the slope of the line is negative which is entirely in accord with what a rearrangement of equation (14) would indicate.

Attention was next turned to the practical implications of the rate of equilibration for double gassing. As might be expected from figure 7, and as actually found in the study recorded in figure 9, there is a progressive decline in equilibrium oxygen level as the time between the gassings is increased. When double gassing is employed, the equilibrium oxygen level in the free-

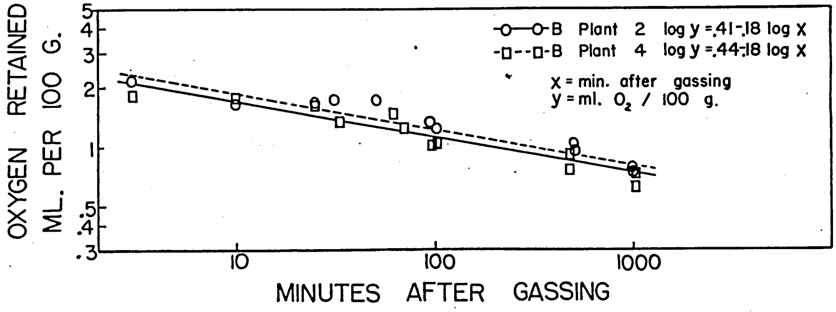


FIG. 8. Relation of retained oxygen to time after gassing (logarithmic scale)

space gas after the second gassing is a measure of the oxygen concentration that was in the air cells immediately before the second gassing. Consequently one would expect a negative log-log linear relation between equilibrium oxygen percentage and time between gassings. Figure 10 shows that such a relationship actually does exist, thus verifying the relation established in the study of rate of equilibration. A second study of the same point, although not shown in figures 9 and 10, corroborated the data presented therein.

It is interesting to note that the diffusion of retained oxygen occurred at different rates in the several powders studied and that the differences among powders, when single gassed, are not

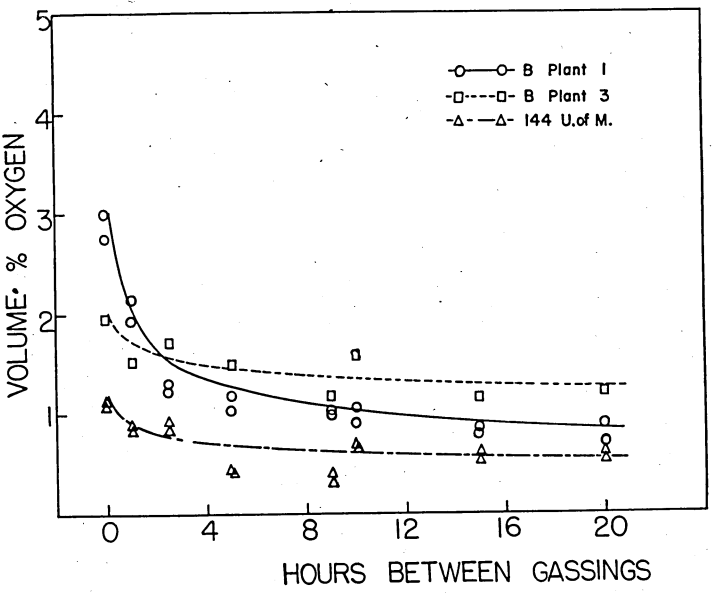


FIG. 9. Relation of equilibrium oxygen level to time between double gassings

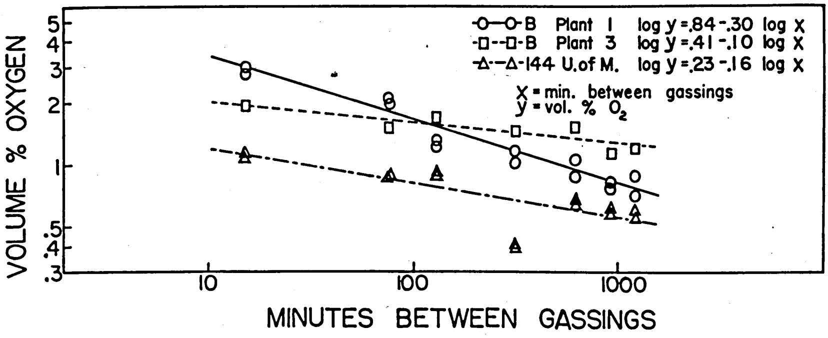


FIG. 10. Relation of equilibrium oxygen level to time between double gassings (logarithmic scale)

preserved upon double gassing. This finding would appear to indicate differences in the rate of change of k with time, owing perhaps to differences in the degree of homogeneity of powder particle size and in the thickness of the cell walls.

These results indicate that there is a definite advantage in holding for 24 hours and longer between gassings. Most of this advantage is attained in 20 to 24 hours and such a practice easily could be meshed into plant routine.

Influence of extent of evacuation—In agreement with calculation, it was shown in figure 6 that, for single gassing, evacuating to 1 mm. reduces the equilibrium oxygen content to a level only about 0.25 volume per cent lower than evacuating to 10 mm. Since 10 mm. more nearly approximates the extent of evacuation attained commercially, the reduction in oxygen level of three samples by a double gassing treatment involving two evacuations to 10 mm. was compared to that attainable by a similar treatment in which the evacuations were carried to 1 mm. The results of this study are presented in table 7. As expected, the actual differences between the 10 mm. and 1 mm. series after single evacuations were of the same order of magnitude as the calculated difference of $0.2750 - 0.0275 = 0.2475$. (See page 3.)

Assuming no diffusion during or between the gassings, an immediate second gassing would be expected further to reduce the oxygen level by only $0.0275 - \left(\frac{1}{760} \times 0.0275\right) = 0.0275$ per cent in the 1 mm. series and by $0.275 - \left(\frac{10}{760} \times 0.275\right) = 0.261$ per cent in the 10 mm. series. The reductions actually produced by immediate second gassing were 0.52, —0.04, and 0.01 per cent in the 1 mm. series and 1.14, 0.26, and 0.39 per cent in the 10 mm. series. The greater discrepancy between actual and calculated reductions in the case of sample B, plant 1 undoubtedly is due to considerable diffusion of gas from that powder during the double gassing process. The more rapid diffusion of gas from that powder has already been noted in figures 9 and 10. After the immediate second gassing, the oxygen level in the 10 mm. series should be $\left(\frac{10}{760} \times 0.275\right) - \left(\frac{1}{760} \times 0.0275\right) = 0.0036$ per cent higher than that in the 1 mm. series. Actually it was 0.16, 0.18, and 0.03 per cent higher in the latter, but this probably is an experimental error.

Naturally a holding period of 24 hours between gassings improved the efficiency of the process in both series. Equation (4) would lead one to expect a slower over-all rate of diffusion during the holding period in the cans of the 10 mm. series, wherein more oxygen was left in the free-space gas after the first gassing than in those of the 1 mm. series. Unfortunately data from which the magnitude of the decrease in rate of diffusion might be calculated are not at hand. However, the actual amounts diffused can be calculated. Thus for A, plant 1, 1 mm.

$$(3.96 - x) + \frac{1}{760} x = 0.95$$

$$x = 3.01\%\ O_2 \text{ diffused into free-space}$$

and for the same sample, 10 mm.

$$(3.96 - x) + \frac{10}{760} (x + 0.46) = 1.80$$

$$x = 2.20\%\ O_2 \text{ diffused into free-space}$$

Similar figures for the other samples are given in table 7. The smaller amount of diffusion in the 10 mm. series verifies the prediction of equation (4).

Comparison of various combinations of nitrogen and carbon dioxide—A comparison was made of the efficiency of nitrogen-nitrogen, carbon dioxide-nitrogen, and nitrogen-carbon dioxide treatments in double gassing three samples. The results, recorded in table 8, indicate that these three treatments are about equally effective in reducing the equilibrium oxygen level (ml./100 g.). It is interesting to note that the carbon dioxide taken up by powder gassed with it is released upon subsequent gassing with nitrogen, thus raising the pressure within the can above atmospheric.

REPEATED GASSING

The effect of five successive gassings at 24-hour intervals on the equilibrium oxygen level is shown in table 9. As expected, the oxygen level is lowered with each successive treatment.

It appears that the oxygen is less readily removed from sam-

Table 7. Comparison of Oxygen Levels after Evacuations to Pressures of 1 mm. and 10 mm.*

Sample	Single gassing			Double gassing—immediate			Double gassing—24-hour interval			Oxygen diffused in 24 hours	
	1 mm.	10 mm.	Diff.	1 mm.	10 mm.	Diff.	1 mm.	10 mm.	Diff.	1 mm.	10 mm.
						Per cent					
A, Plant 1	4.04	4.30	....	3.67	3.09	....	0.89	1.39	....	....	....
	4.00	4.43	....	3.21	3.47	....	1.01	2.04	....	....	....
	3.90	4.57	....	....	....	....	....	1.96	....	....	....
	3.98	4.40	....	....	....	....	....	....	....	....	....
Mean	3.96	4.42	0.46	3.44	3.28	—0.16	0.95	1.80	0.85	3.01	2.20
A, Plant 3	2.64	2.44	....	2.66	2.47	....	1.02	1.60	....	....	....
	2.41	2.80	....	2.39	2.21	....	1.04	1.66	....	....	....
	2.19	2.56	....	....	....	....	....	1.73	....	....	....
	2.68	....	....	....	....	....	....	1.80	....	....	....
Mean	2.48	2.60	0.12	2.52	2.34	—0.18	1.03	1.70	0.67	1.45	0.79
108, U. of M.	1.61	1.83	....	1.63	1.60	....	0.75	1.05	....	....	....
	1.67	2.15	....	....	....	....	....	1.20	....	....	....
Mean	1.64	1.99	0.35	1.63	1.60	—0.03	0.75	1.13	0.38	0.89	0.52
Calculated difference	....	....	0.25	....	....	+0.0036	....	....	....	....	....

* All analyses made 7 days after final gassing.

Table 8. Comparison of the Efficiency of Various Combinations of Nitrogen and Carbon Dioxide in Double Gassing Treatments*

Sample	Double gassing with nitrogen			Carbon dioxide followed by nitrogen				Nitrogen followed by carbon dioxide			
	Can pressure	Oxygen		Can pressure	Oxygen		Carbon dioxide	Can pressure	Oxygen		Carbon dioxide
	mm.	Per cent	ml./100 g.	mm.	Per cent	ml./100 g.	Per cent	mm.	Per cent	ml./100 g.	Per cent
B, Plant 1	751	1.00	0.87	798	0.84	0.74	20.50	498	1.83	1.00	73.51
	753	1.02	0.85	804	0.71	0.63	19.09	....	....	....	....
	....	....	....	728	1.43	1.15	18.93	503	1.37	0.77	72.66
	....	....	....	772	1.17	1.00	18.62	....	....	....	....
B, Plant 3	764	1.40	1.31	865	0.78	0.79	17.00	451	1.90	0.99	82.70
	....	....	....	869	0.70	0.71	17.52	....	....	....	....
	731	0.98	0.80	842	1.24	1.16	16.44	427	1.85	0.88	84.39
	730	1.02	0.82	....	....	....	....	....	....	....	....
127, U. of M.	762	0.49	0.52	853	0.39	0.45	18.76	450	1.48	0.91	88.97
	755	0.51	0.54	858	0.34	0.39	18.77	....	....	....	....
118, U. of M.	723	0.59	0.57	830	0.43	0.48	18.30	463	0.70	0.44	86.28
	723	0.75	0.73	830	0.55	0.61	17.75	....	....	....	....

* All evacuations were to 1 mm.; 24 hours were allowed between gassings. Analyses were made 7 days after the final gassing.

Table 9. Effect of Repeated Nitrogen Gassings at 24-Hour Intervals on the Oxygen Level after Seven Days*

Sample	Number of gassings				
	1	2	3	4	5
	ml. oxygen (S.T.P.)/100 g. powder				
B, Plant 1	2.70	0.85	0.43	0.29	0.07
	2.73	0.87	0.37	0.29	0.00
B, Plant 3	1.84	1.31	0.69	0.43	0.28
	2.13	....	0.62	0.42	0.26
125, U. of M.	2.12	0.52	0.17	0.24	0.08
	2.04	0.54	0.28	0.17	0.08
	....	....	....	....	0.00
	....	....	....	....	0.16
148, U. of M.	1.74	....	....	....	0.23
	1.80	....	....	....	....

* All evacuations were to 1 mm.

ple B, plant 3 than from the other powders. Interestingly enough, this observation corroborates the results of double gassing and vacuum holding studies wherein this particular powder was found to release its oxygen more slowly than others.

Holding the Dry Whole Milk under Vacuum

Another general method for permitting diffusion of oxygen from the powder particle to occur before final gassing is to hold the powder continuously under a vacuum.

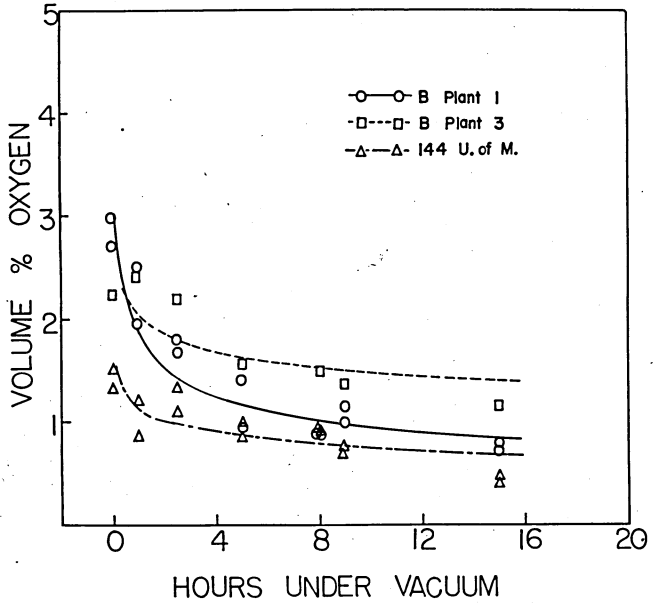


FIG. 11. Relation of equilibrium oxygen level to time held under vacuum before gassing

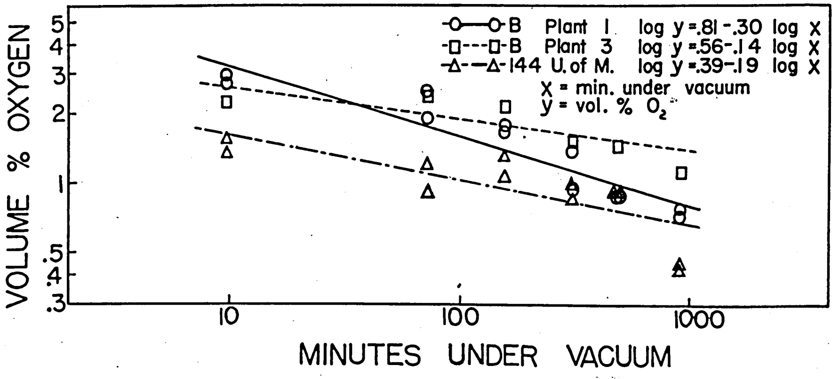


FIG. 12. Relation of equilibrium oxygen level to time held under vacuum before gassing (logarithmic scale)

Figures 11 and 12 show the progressive decline in equilibrium oxygen level obtained by holding the dry milk under a vacuum for various intervals prior to gassing. As might be expected, holding under a vacuum has approximately the same effectiveness as double evacuation and gassing at comparable time intervals. There is the same inverse log-log linear relation between equilibrium oxygen content and time allowed for diffusion before final gassing, and the same differences in the rate of diffusion among the various powders. This general relation was verified in another study involving three samples.

The regression coefficients given in figure 12 were used in making the corrections for time under vacuum shown in table 5.

As a practical procedure, holding the powder in bulk under a vacuum to permit diffusion before canning, final evacuation, and gassing might be preferable to holding the canned powder under a vacuum. That this procedure would be equally as satisfactory from the standpoint of the oxygen content of the canned powder is shown by the data in tables 10 and 11. If the bulk powder is canned, evacuated, and gassed promptly after the vacuum on the bulk powder is broken, it seems to be relatively

Table 10. Reduction of Oxygen Level by Holding Bulk Powder under Vacuum before Canning as Compared to Regular Gassing Treatments

Oxygen content after 7 days*							
Single gassing		Double gassing—24-hour interval		Under vacuum 16 hours—released with N_2†		Under vacuum 24 hours—released with air†	
Per cent	ml./100 g.	Per cent	ml./100 g.	Per cent	ml./100 g.	Per cent	ml./100 g.
1.79	1.74	0.61	0.59	0.75	0.70	0.59	0.55
1.70	1.66	0.55	0.53	0.68	0.64	0.48	0.45

* Sample was U. of M. 120. All evacuations were to 0.5 mm.

† After release of vacuum the powder was canned immediately, evacuated to 0.5 mm., and gassed with nitrogen.

Table 11. Reduction of Both Oxygen and Moisture by Holding Dry Whole Milk in Vacuum over Desiccant* before Canning and Gassing

Sample	Control			Held in vacuum desiccator 16 to 24 hours		
	Oxygen†		Moisture	Oxygen†		Moisture
	Per cent	ml./100 g.	Per cent	Per cent	ml./100 g.	Per cent
U. of M.						
145	1.92	2.07	3.28	0.88	0.94	2.04
	1.60	1.74	3.26	0.88	0.89	1.98
146	2.39	2.57	2.44	0.73	0.76	1.18
	2.06	2.21	2.44	0.68	0.71	1.16
147	1.32	1.43	4.64	0.75	0.81	3.40
	1.79	1.93	4.70	0.72	0.78	3.40
148	1.93	1.74	1.96	0.89	0.76	0.98
	2.09	1.80	1.94	1.05	0.90	1.00
154	1.43	1.55	3.30	0.95	1.00	1.80
	1.68	1.84	3.28	0.58	0.61	1.80
155	1.74	1.90	3.26	1.00	1.07	2.00
	1.72	1.88	3.26	0.74	0.79	2.00

* Alumina was the desiccant used in these experiments.
† Analyses 7 days after gassing.

immaterial whether the vacuum is broken with nitrogen or with air.

Furthermore, it is shown in table 11 that a substantial reduction in the moisture content of the powder can be effected by use of a desiccant in the vacuum chamber. This technique may offer possibilities should reduction of the moisture content ever be required in the future.

Summary

Direct evidence is presented that the increase in the oxygen content of the head-space gas in cans of dry whole milk after single evacuation and gassing is roughly proportional to the total volume of entrapped air in the powder particles. Actual adsorption on the surfaces of the powder particles represents at most a minor portion of the retained oxygen. The air is retained in the powder under evacuation because the walls of the air cells are a highly concentrated lactose sirup (glass) which is relatively impermeable to gases. The presence of foam in the milk sprayed may increase greatly the amount of air entrapped in the powder particles, although powder from milk deaerated before spraying does not contain less air than powder from normal milk.

When the amount of oxygen surrounding the powder particle is reduced by evacuation and inert gas-packing or by holding continuously under a vacuum, the oxygen diffuses from the air cells until equilibrium is attained. The diffusion occurs at such rates, characteristic for each powder, that the logarithm of concentration is inversely proportional to the logarithm of time. The final oxygen content of gas-packed powder may be reduced by regassing after a time interval or by holding the powder continuously under a vacuum. This reduction, which is proportional to the rate of diffusion, is of sufficient magnitude in 20 hours to offer a very effective method of lowering the oxygen level. Furthermore, the vacuum-holding treatment can be utilized to lower the moisture content of the powder. In single gassing the equilibrium oxygen level is reduced by only about 0.0275 per cent per mm. decrease in pressure. This is the theoretical reduction due solely to evacuation of the gases from the free-space of the can. Since the rate of diffusion of the oxygen is dependent upon its pressure in the can, evacuation to low pressures in double gassing or in vacuum holding is more effective than in single gassing.

Nitrogen and carbon dioxide are equivalent as inert gases for packing dry whole milk in so far as oxygen levels are concerned.

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