ABSTRACT
A sample of instantized nonfat dry milk which had been stored in well sealed tins in a Los Angeles home basement for 20 yr was evaluated. The powder was light yellow in color and had a stale odor. Flavor evaluation indicated that it was unsuitable for drinking and most cooking uses. Reconstituted nonfat dry milk containing as little as 5% of the old milk mixed with 95% fresh was characterized as stale, cooked, oxidized, and "old casein." Moisture content was 6.9%, approximately double that of the original powder. Vitamin analyses indicated losses ranging from 61% for thiamine to 2% for niacin. Microbiological testing showed bacterial content similar to fresh nonfat dry milk. Although available lysine content was decreased by 30%, protein quality as measured by protein efficiency ratio was still high, 2.69 versus 2.5 for casein. Additional feeding trials with rats indicated that the milk powder was health and growth promoting.

INTRODUCTION
Nonfat dry milk (NDM) lends itself well to storage because of its low moisture and fat. In a test with instantized NDM stored for 2 yr in its retail package of cardboard with a foil overwrap, Hodson and Miller (9) showed no loss of protein efficiency nor of available lysine in the milk. Moisture in their sample increased during the 2 yr of storage from 3.4% to 5.8%.

Henry et al. (8) evaluated the physical, chemical, and flavor changes, as well as changes in the biological values of the protein of NDM samples which had been stored up to 5 yr. Their protocol involved noninstantized powders with either low (2.8%), medium (5.0%), or high (7.3%) moisture contents stored at three temperatures in both air packed and nitrogen packed cans. Storability was most dependent on the moisture content; the low moisture powder was not altered appreciably in any way even after 2 yr regardless of temperature or type of pack. On the other hand, the high moisture milk rapidly deteriorated in all properties in a matter of days or weeks. Storage temperature (20, 28.5, 37 C) was the second most important factor, followed by packing atmosphere (air or N₂). Microbiological assays for lysine (with an acid hydrolysate of the protein) showed a 23% loss after just 95 days at 37 C in air pack. When an enzyme hydrolysate of the milk was used, the loss was 50% after 176 days in air pack at 28.5 C. However, enzyme hydrolysis yielded lower results than acid hydrolysis.

Commercial instantized NDM contains 3 to 4% moisture, low enough to ensure satisfactory flavor for at least 1 yr of storage by distributors and consumers. After about a year, palatability of commercial NDM begins to decrease when held under normal warehouse or home shelf conditions. However, tests in our laboratory have shown no decrease in protein efficiency for 1-yr-old NDM purchased from a retail grocery store.

Carnation Company recently obtained a sample of its Instant Nonfat Dry Milk which had been stored since March 1957. This study was undertaken to evaluate the milk's sensory and nutritive qualities after 20 yr of storage.

MATERIALS AND METHODS
A sample of Instant Nonfat Dry Milk was obtained in September 1977 from a private Los Angeles citizen who had stored the product in his basement since March of 1957. The basement was a small subterranean room with air vents to the outside atmosphere. No temperature or humidity controllers were present although the house radiator was located there. Maximum temperature in the basement during the summer was estimated at 27 C.

The NDM had been packaged by Carnation Research Laboratory.
Company in double-sealed tin containers with tight-fitting friction lids and lined screw caps. The NDM was specially packaged for the Mormon Church to distribute to its members. Dry milk processing in 1957 was similar to the method used today except that then it was not fortified with vitamins A and D.

Flavor evaluation of the reconstituted 20-yr-old NDM mixed in various proportions with fresh NDM was conducted by a six-member panel of scientists trained in scoring techniques for NDM. Scoring was based on the American Dairy Science Association’s scoring method for NDM as published by Nelson and Trout (10). Flavor characteristics and intensities of the reconstituted product were determined by a five-member panel which had been trained in flavor profile analysis.

Microbiological testing included standard plate and coliform counts by procedures of the American Public Health Association (6) and staphylococcus and streptococcus counts following AOAC methodology (5). Proximate analysis, vitamin analyses, and protein reducing values were determined by AOAC methods (2). Available lysine was determined by the method of Carpenter (4).

Protein efficiency ratio (PER) by rat trial was conducted on the dry milk powder with the AOAC procedure (2). Standard PER rations were prepared to contain 10% protein provided by 28.25% NDM in the test ration and 10.65% casein in the control. Also, 16% lactose was added to the control to approximate the test ration.

Following the 4-wk trial, half of the animals from each group were sacrificed with chloroform and examined by gross necropsy techniques. The data were analyzed by analysis of variance (ANOVA) and Scheffe’s test.

The remaining five rats from the test group were placed immediately on a ration consisting of the 20-year-old NDM plus 8% corn oil and four mineral salts, FeS4, MgSO4, MnSO4, and CuSO4. The animals were fed and weighed weekly for 4 wk at which time they were sacrificed with chloroform and examined by gross necropsy.

RESULTS AND DISCUSSION

Mild browning of the milk had occurred as evidenced by 1) the change from its natural white color to a butter-yellow shade, 2) the elevated protein reducing value (Table 1), and 3) the decrease in available lysine (Table 1).

The powder had an odor resembling old casein, similar to old casein glues. The odor and taste were not repulsive, although the powder would have to be considered unsuitable for drinking and most cooking.

Reconstituted NDM samples containing 30%, 20%, and 10% concentrations of the 20-yr-old milk received unacceptable scores of

<table>
<thead>
<tr>
<th>Nutrient</th>
<th>Fresh NDMa (per 100 g)</th>
<th>20-yr-old NDM (per 100 g)</th>
<th>Difference from fresh (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Moisture, g</td>
<td>4.3</td>
<td>6.9</td>
<td>+60%</td>
</tr>
<tr>
<td>Nitrogen, g</td>
<td>5.49</td>
<td>5.25</td>
<td>...</td>
</tr>
<tr>
<td>Fat, g</td>
<td>.7</td>
<td>.5</td>
<td>...</td>
</tr>
<tr>
<td>Carbohydrate, g</td>
<td>52.2</td>
<td>51.4</td>
<td>...</td>
</tr>
<tr>
<td>Ash, g</td>
<td>7.9</td>
<td>7.7</td>
<td>...</td>
</tr>
<tr>
<td>Thiamine, mg</td>
<td>.413</td>
<td>.161</td>
<td>-61%</td>
</tr>
<tr>
<td>Riboflavin, mg</td>
<td>1.428</td>
<td>.716</td>
<td>-50%</td>
</tr>
<tr>
<td>Vitamin B6, mg</td>
<td>.345</td>
<td>.310</td>
<td>-10%</td>
</tr>
<tr>
<td>Niacin, mg</td>
<td>.891</td>
<td>.879</td>
<td>-2%</td>
</tr>
<tr>
<td>Vitamin B12, µ</td>
<td>3.99</td>
<td>3.22</td>
<td>-20%</td>
</tr>
<tr>
<td>Available lysine, g</td>
<td>2.496</td>
<td>1.747</td>
<td>-30%</td>
</tr>
<tr>
<td>Protein reducing value, mg/g</td>
<td>250 to 500b</td>
<td>829</td>
<td>+66 to 232%</td>
</tr>
</tbody>
</table>

aValues taken from (1).
bValues obtained from analyses of fresh Carnation Instant Nonfat Dry milk.
30, 30, and 31, respectively, based on the ADSA scoring system. Scores of 33.5 or less indicate unacceptable product while a 40 is excellent; fresh NDM usually scores between 36 and 38. The off-flavor characteristics of high intensity noted by the panel at these three dilutions were primarily stale, scorched, cooked, and “old casein.”

Reconstituted samples containing 5%, 3%, 1%, and 0% of the 20-yr-old milk blended with fresh NDM received acceptable ratings of 34, 35, 35, and 37. These samples were rated less intense in the characters of stale and cooked. The old casein and oxidized notes were not detectable in the 3%, 1%, and control samples.

The flavor profile of the 20-yr old reconstituted sample indicated that the major undesirable aroma characteristics were “old casein/glue” at strong intensity, and “stale/dusty” at moderate to strong. Sensory evaluation revealed that “casein/glue” and “stale/dusty” were present at strong intensities, and a “burnt/cooked note” was moderate to strong.

Microbiological results did not differ significantly from those of fresh NDM. Standard plate count was 1800/g while coliform, staphylococcus, and streptococcus counts were less than 10/g. These findings were expected because the water activity of the NDM was too low for bacterial growth even with the increase in moisture to 6.9% (3). Therefore, bacteria would be expected to slowly die over time.

Results of the proximate and vitamin assays are in Table 1. Powdered milk values from USDA Handbook 8-1 (1) are included for comparison. Moisture content of the 20-yr-old sample was 6.9%. The original moisture content was not known, but it would have been approximately 3 to 4% according to the Carnation Company product manager for powdered milk. Thus, moisture increased about 3% during storage.

The nitrogen, fat, carbohydrate, and ash contents would not be expected to change during storage. Differences from handbook values as shown in Table 1 would be within the ranges expected for commercial NDM.

Vitamins decreased to varying degree with thiamine showing the greatest loss (61%). Riboflavin decreased 50% whereas only a 2% loss occurred for niacin.

The protein reducing value was 829 mg/100 g versus a range for fresh NDM of 250 to 500
mg/100 g. Thus, the reducing capacity approximately doubled during 20 yr of storage.

The protein efficiency ratio for the 20-yr-old NDM was 2.69 versus 2.5 for casein. The PER data are in Table 2. The ANOVA indicated a significant difference between treatments (P<.05). Scheffe’s test also showed significance between the two PER values (P<.1). For comparison, fresh NDM has a PER value of approximately 2.8.

Studies (7, 8) reported that the decrease in PER of stored NDM was accompanied by a loss of available lysine. High moisture NDM (7.3% moisture) had a loss of approximately 40% of the available lysine upon storage. Although our results indicated only a 30% loss, one still would expect the protein quality to be affected significantly. However, the PER trial indicated only a minor loss of quality, which was similar to the results of Hodson and Miller (9) for 2 yr of storage. Thus, the animals were able to utilize the lysine even though the chemical assay indicated that 30% of the e-amino groups was unavailable. Post mortem examinations showed normal, healthy tissues in all animals.

The feeding trial with five rats consuming a ration of the 20-yr-old NDM plus 8% corn oil revealed that the milk sample was still safe and nutritious. The food efficiency ratio was .316 (146.2 g weight gain divided by 462.2 g dry matter consumption). Only one animal exhibited diarrhea, which is a common side effect of high milk diets. Necropsy examination showed, for the most part, healthy tissues in all rats. One animal had a slightly mottled kidney, and an enlarged spleen was seen in another. However, these were isolated observations.

Thus, nutritionally, the milk was preserved well. Protein quality was reduced only slightly, and most vitamins were still reasonably high; only thiamine and riboflavin were appreciably low. The most serious nutritional deterioration was probably the 50% riboflavin loss as milk is a significant dietary source of this vitamin.

Presumably the milk was preserved because of its low exposure to oxygen, humidity, and high temperature. The tin containers in which the NDM was stored prevented contact with the atmosphere more effectively than would occur with conventional packaging. Thus, instant milk stored for long periods under favorable conditions still can be used for nutritional purposes even though it might have low flavor acceptability.

ACKNOWLEDGMENT

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REFERENCES