DIRECT RECOVERY OF OFF-FLAVORS FROM FOAM-DRIED WHOLE MILK

F. E. KURTZ

Dairy Products Laboratory, Eastern Utilization Research and Development Division, ARS USDA, Washington, D. C.

ABSTRACT

Off-flavor compounds were obtained by a short-path migration at 1 μ pressure to a liquid nitrogen-cooled condenser, from dried milk warmed to 40 C or less for 3 hr. Milks reconstituted from stale and oxidized powders, both before and after treatment, and market milk, both with and without the condensed volatiles, were evaluated by a five-man taste panel. The volatile flavors were oxidized, stale, unclean, astringent, and foreign. As with the author's earlier findings with regard to added ketones, the procedure is made more effective by first freeing the fat in the powder. Compounds recovered from a slightly stale dried milk gave a stale flavor to control milk, but their removal did not improve the residual powder. Compounds recovered from a strongly oxidized dried milk gave an oxidized flavor to control milk and their removal definitely improved the flavor of the residual powder. From four experiments with high free-fat oxidized dried milk, the average scores were: powder control—27.2; powder residue (powder control less volatiles)—30.7; milk control—37.8; milk plus volatiles—28.8.

Ketones incorporated into dried whole milk can be recovered in substantial quantities directly from the milk powder by a procedure which requires only moderate heating in a vacuum (1). Volatiles recovered in this way are associated with only a small volume of water and both the residual powder and the volatiles can be utilized, without further treatment, for taste panel evaluations. These characteristics make the recovery procedure appear attractive for collecting material for the study of flavor compounds in dried milk. If the volatilities of the flavor compounds are similar to those of the ketones studied previously, their recoveries would be equally successful. If their volatilities were pronouncedly less, however, as from complexing with nonvolatile constituents in the dried milk, their recovery by this procedure might well be unsuccessful. This study, accordingly, has been made to determine the applicability of the direct recovery method to the recovery of off-flavor compounds from dried whole milk.

MATERIALS AND METHODS

Foam-dried whole milks were prepared by a standard procedure (2). Each lot was nitrogen-packed, 60-65 g per can, in sealed cans. Separate cans of powder, but taken from the same lot, were used in each experiment concerned with the recovery of stale flavor. Oxidized powder was prepared by mixing the contents of a number of cans and holding the powder at room temperature in a glass-stoppered bottle until it had reached the desired stage of oxidation. This took about 7 wk. The oxidized powder was stored at 0 C; all material used in recovering oxidized flavor was taken from this same batch of powder.

Free fat was determined by the method of Tamsma et al. (3), modified as previously described (1).

EXPERIMENTAL PROCEDURE

The apparatus and procedure used in the recovery of off-flavor volatiles were those previously employed in the recovery of added ketones (1). In all instances, the volatiles from 35.0 g of dried milk were collected on the cold finger. In one experiment, the powder was held at 25 C for 3 hr. Otherwise, the powder was warmed to 40 C for 3 hr. With the one exception, Table 2, in which the volatiles were collected from an unaltered powder, the dried milk was hydrated within the collection system before collecting the volatiles.

At the conclusion of a collection, the system was brought to atmospheric pressure with nitrogen gas. The condensed volatiles were taken up in 200 ml of fresh milk, as previously described (1). Free fat was determined on 5 g of the residual powder, and the remaining 30 g of

Received for publication October 31, 1964.

TABLE 1

Recovery of off-flavors from a stale powder [a]

Sample	40 C				25 C			
	Average score	Median score	Oxidized flavor [b]	Stale flavor [b]	Average score	Median score	Oxidized flavor [b]	Stale flavor [b]
Powder control	34.8	34	– – – – –	S S S S –	33.4	33	D – – – –	D D S S –
Powder residue	34.0	34	– – – – –	D S S S –	33.2	33	S – – – –	D D S S –
Milk control	37.2	37	– – – – –	– – – – –	37.2	37	– – – – –	– – – – –
Milk + volatiles	33.6	33	– – – – –	D D S S –	34.2	35	S – – – –	D S S S –

[a] Fat freed by hydration.
[b] P: pronounced flavor; D: definite flavor; S: slight flavor; –: no flavor.

powder was reconstituted with 200 ml of water. The control powder was taken from the same batch as the powder from which volatiles were collected. The four samples—powder control, powder residue (powder control less volatiles), milk control, and milk plus condensed volatiles—were held overnight at 40 F. After warming to about 80 F, the samples were presented to a five-man panel of experienced judges for flavor evaluation and scoring.

RESULTS AND DISCUSSION

At the time of these experiments, the most important flavor problem connected with dried whole milk was the development of oxidized flavor during storage. Stale flavor was also an important problem, and has become increasingly so. The latter not only is found in some freshly prepared dried whole milks but frequently develops during storage, so that an oxidized flavor may be overlaid on a stale flavor. The experiments reported here concern the recovery of these two flavors, with the emphasis on oxidized flavor.

all judgments shown in Table 2 were made by the same five judges. The judgments in the first and second recoveries in Table 3 were made by the same five judges, whereas the judgments concerning the third recovery in this table were made by a four-man panel—these four having been part of the preceding panels.

In addition to the principal off-flavors, oxidized and stale, the following criticisms were attributable, in certain instances, to the condensed volatiles: astringent, foreign, and unclean. The judges, in making their quality estimates, used a scoring system which ranges from 40, for a perfect milk, to 0. However, in only two out of a total of 136 flavor judgments involved in Tables 1-3 was the principal criticism based on a defect other than stale or oxidized flavor. The scoring range for a milk with stale flavor was 36-28, and for oxidized flavor 36-24. In two instances, both concerning the same judge, the sample of milk containing condensed volatiles was described as having an objectionable foreign flavor, and the respective scores were 20 and 15. Such scoring, as well

TABLE 2

Relationship between recovery of off-flavors and free fat of an oxidized milk powder

Sample	11% Free fat				86% Free fat			
	Average score	Median score	Oxidized flavor [a]	Stale flavor [a]	Average score	Median score	Oxidized flavor [a]	Stale flavor [a]
Powder control	29.4	30	D D D D –	P – – – –	28.0	28	P P P D –	P – – – –
Powder residue	29.2	29	P P D D –	D – – – –	31.6	32	D D S – –	D D D D –
Milk control	37.6	38	– – – – –	– – – – –	37.6	37	– – – – –	– – – – –
Milk + volatiles	37.0	37	– – – – –	S – – – –	32.0	32	D S – – –	D D D – –

[a] P: pronounced flavor; D: definite flavor; S: slight flavor; –: no flavor.

An attempt was made to utilize the same judges throughout the experiments, but this was not entirely possible. However, while each of the experiments was judged at a different session, the comparisons shown on an individual table were generally made by the same judges. Specifically, all judgments shown in Table 1 were made by the same five judges. Likewise, as any unusually high or low scoring, has a tendency to unbalance the judgment of the majority of a panel. For this reason, the median score, as well as the more commonly used average score, has been included in the tables. As was mentioned, one of the panels had only four judges. Their judgments of each sample were arranged in the order of their

TABLE 3

Recovery of off-flavors from an oxidized milk powder[a]

Sample	Average score	First recovery[b] Median score	First recovery[b] Oxidized flavor[c]	First recovery[b] Stale flavor[c]	Average score	Second recovery[b] Median score	Second recovery[b] Oxidized flavor[c]	Second recovery[b] Stale flavor[c]	Average score	Third recovery[b] Median score	Third recovery[b] Oxidized flavor[c]	Third recovery[b] Stale flavor[c]
Powder control	28.0	28	P P P D S	D — — — —	26.6	27	P P P P P	D — — — —	26.3	26.5	P P P —	P D — —
Powder residue	31.2	31	P D D S —	D D — — —	30.6	30	P D S — —	D D S — —	29.3	30	P D D —	D D — —
Milk control	37.4	38	— — — — —	— — — — —	37.8	38	— — — — —	— — — — —	37.8	37.5	— — — —	— — — —
Milk + volatiles	28.0	28	P P D — —	P S — — —	26.8	28	P S — — —	P P S — —	26.5	26	P P P —	D — — —

[a] Fat freed by hydration.
[b] Second recovery made two days, and third recovery, three months after initial recovery. Powder stored at 0 C between experiments.
[c] P: pronounced flavor; D: definite flavor; S: slight flavor; —: no flavor.

values. If the second and third values were identical, this common value was recorded as the median value. Otherwise, a calculated value equidistant between the second and third values was arbitrarily taken as a median.

The data in Table 1 show that stale flavor can be recovered directly from a stale dried milk. Results from heating at 25 C are similar to those from heating at 40 C. The differences in flavor scores appear to be consistent with the earlier finding that the recovery of tridecanone at 25 C was about 80% of that at 40 C. These data, as well as some of the data in the other tables, give an impression that the off-flavors may have been intensified by the recovery process. This question is being studied further. At this time, a definite answer cannot be given because of insufficient information on the effects of dilution and interaction of the flavors involved.

The data in Table 2 show that oxidized, as well as stale flavor, was recovered directly from the dried milk. As was found with added ketones, for most complete recovery of off-flavors it is necessary to first effect the structural alterations associated with freeing the fat. The dried milk used in obtaining these data had been stored nine months before being oxidized by exposure to air. Although its flavor had not been evaluated at the time of manufacture, it is likely that a stale flavor, as well as an oxidized flavor, had developed by the time of the experiments recorded in this table.

The data in Table 3 substantiate the conclusion that can be drawn from the high free-fat section of Table 2; namely, that oxidized flavor can be recovered directly from an oxidized powder. The fact that the flavor of the residual powder was improved definitely establishes that off-flavor compounds were actually removed from the powder; they could not have been created—entirely, at least—by the recovery process. The data of Table 3, along with those of Table 2, indicate the variability in results that might be expected from a combination of experimental variation and flavor judgments made at separate sessions.

REFERENCES

(1) KURTZ, F. E. 1965. Direct Recovery of Added Ketones from Foam-dried Whole Milk. J. Dairy Sci., 48: 269.

(2) SINNAMON, H. I., ACETO, N. C., ESKEW, R. K., AND SCHOPPET, E. F. 1957. Dry Whole Milk. I. A New Physical Form. J. Dairy Sci., 40: 1036.

(3) TAMSMA, A., EDMONDSON, L. F., AND VETTEL, H. E. 1959. Free Fat in Foam-Dried Whole Milk. J. Dairy Sci., 42: 240.