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Efficiency of Laboratory Steam Deodorization in Quantitative Recovery of Aliphatic Delta-Lactones from Butteroil [1]

Numerous techniques have been employed for isolating aliphatic lactones from milk fat. These include steam deodorization (3, 10); methanol extraction (1); molecular distillation (1, 4); silicic acid column chromatography (2, 5); and thin-layer chromatography (5, 6, 11). Laboratory steam deodorization has been shown to be an effective method in stripping the lactones and methyl ketones from butteroil to improve the flavor stability. Tharp (10) found that deodorization for 3 hr at 125 C and 5.0 ± 0.5 mm Hg was sufficient to generate and completely eliminate the coconut (lactone) flavor from milk fat. In a recent study, Nelson et al. (7) presented preliminary evidence that a statistically significant improvement in flavor stability of butteroil was achieved after 2 hr at 145 ± 5 C and 0.02 mm Hg. Similarly, Patton (8) utilized steam stripping at 125–200 C under pressures of 1–20 mm Hg for 1–3 hr to eliminate the nonoxidative off-flavors during heat treatment and storage of milk fat. The above studies were primarily concerned with the final flavor stability of the resulting butteroil as a criterion for lactone removal. In our laboratory, steam deodorization is being employed for isolation of lactones in conjunction with gas chromatography (GC) for quantitation. This study was undertaken to evaluate the conditions required for maximum recovery of the aliphatic delta-lactones from butteroil.

General Procedure

Fresh pasteurized cream (37–40% fat) was churned in a Waring Blendor at 4 C. The butter granules were warmed and centrifuged at 2,000 rpm (International Centrifuge, Model PR-1) for 30 min to obtain serum-free butteroil. One hundred-gram lots of oil were transferred to 500-ml deodorization flasks and held at 4 C during storage. Oil for these experiments was used within one to three days after churning. The laboratory scale steam deodorization equipment was similar to that reported by Patton and Tharp (9), with the following modifications: A 500-ml deodorization flask was employed, to steam-strip 100-g lots of butteroil. The ethanol dry-ice traps had a capacity of three liters per trap for collection of the steam distillate. An absolute pressure of 0.01–0.5 mm Hg was maintained on the system, measured by a manometer connected to the vacuum line between the second distillate trap and a Welch, high-vacuum Duo-Seal pump. Throughout the entire study the temperature of the butteroil during deodorization was held at 180–195 C. Deodorization time and the amount of steam passed through the butteroil (measured as aqueous distillate) were variable, as reported in the results. Upon completion of the deodorization, the dry-ice traps were allowed to thaw and the distillate transferred to a two-liter Erlenmyer flask. Each dry-ice trap was vigorously washed with 2-125-ml portions of redistilled ethyl ether and added to the aqueous distillate. The aqueous phase was then saturated with salt and extracted with a 1,000-ml separatory funnel. After this initial extraction the aqueous phase was again extracted with 500 ml of ethyl ether. The combined extracts were carefully evaporated on a steam bath under a stream of nitrogen and transferred to 10-ml graduated conical screw-cap tubes. Gas chromatographic analyses were used for quantitation of the lactones present in 1-ml extracts (3). Five microliters were injected on to a diethyleneglycol adipate–acid-treated column and the resulting peak areas converted to parts per million with the aid of calibration curves.

Results and Discussion

During preliminary experiments to quantitatively recover the aliphatic lactones by steam deodorization, it was noted that duration of the deodorization and amount of steam passing through the butteroil influenced the recovery. It was noted also that the δ-C16 and δ-C18 aliphatic lactones were not substantially removed from the butteroil under the steam deodorization conditions employed. These lactones have very little, if any, flavor significance, due to their high boiling points. Results, therefore, are presented for the δ-C8, δ-C10, δ-C12, and δ-C14 aliphatic lactones which are associated with sweet, coconut-like, peach-like, and strawberry-like flavors, respectively.

Figure 1 illustrates data from one of two series of experiments to determine the maxi-

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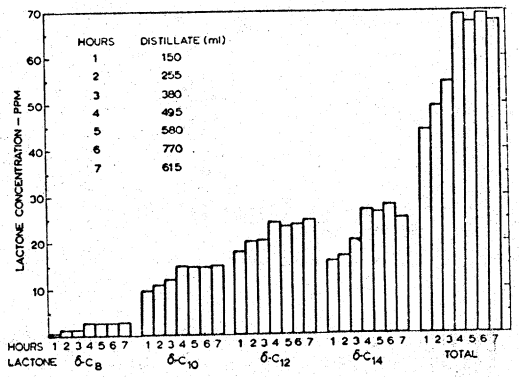

FIG. 1. Effect of steam-deodorization time on lactone recovery from butteroil with constant-steam generation.

deodorization necessary to remove the maximum amount of the δ-C8, δ-C10, δ-C12, and δ-C14 aliphatic lactones. It was evident from these data that the maximum amount of these lactones was recovered in 4 hr of deodorization. The amount of steam generated (measured as aqueous distillate) was at least 100 ml per hour.

In a second series of experiments, the time of steam deodorization was held constant at 5 hr and variable amounts of steam were allowed to strip the butteroil by adjusting the outlet of the steam-generator flask. It was evident that the rate of steam passed through the oil influenced the amount of lactone recovered (Figure 2). To obtain the maximum amount

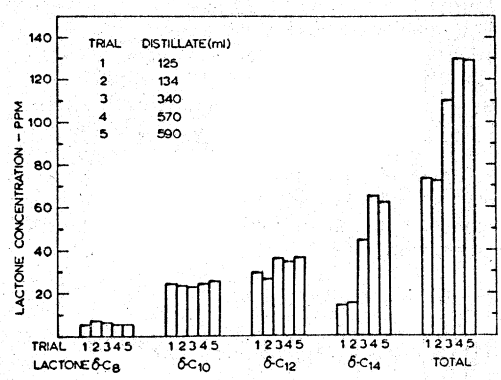

FIG. 2. Effect of steam-generation rate on lactone recovery from butteroil with constant-steam-deodorization time.

of δ-C12 and δ-C14, it was necessary to strip the butteroil with a minimum of 100 ml steam per hour.

With the above parameters established, radioactive 1-C^{14} lactones were added to 100-g aliquots of butteroil which were then steam-deodorized. The amount of label present in the distillate was that assayed following the extraction technique outlined in the general procedure; therefore, the per cent recovery was a measure of efficiency for the total system prior to GC. The data (Table 1) indicate yields

TABLE 1

Recovery of added 1-C^{14} lactones from 5-hr steam deodorization of butteroil[a]

1-C^{14} lactone	Distillate	Label added	Label recovered in distillate		Label found in connecting tube[b]		Label remaining in butteroil	
	(ml)	*(cpm)*	*(cpm)*	*(%)*	*(cpm)*	*(%)*	*(cpm)*	*(%)*
δ-C8	650	328,200	338,000	103.0	891	0.3	2,235	0.7
δ-C12	665	387,968	362,320	93.4	1,300	0.3	3,822	1.0
δ-C14	740	431,692	395,400	91.6	13,232	3.0	9,445	2.2
Mixture[c]	725	792,061	710,720	89.7	7,834	1.0	6,616	0.8

[a] One hundred-g aliquots isolated from blended cream.
[b] Refers to tube connecting the deodorization flask with the first dry-ice trap.
[c] Pooled aliquots of δ-C8, δ-C12, and δ-C14 1-C^{14} lactones.

TABLE 2

Comparison of lactone recovery from butteroil employing steam deodorization and silicic acid column chromatography[a]

	Concentration—ppm								
	Trial 1			Trial 2			Trial 3		
Lactone	SD	SACC	Difference	SD	SACC	Difference	SD	SACC	Difference
δ-C8	2.8	.....[b]	−2.8	2.3	.....	−2.3	6.2	.....	−6.2
δ-C10	15.3	9.6	−5.7	13.7	12.4	−1.3	24.0	15.0	−9.0
δ-C12	23.7	23.2	+0.5	21.9	25.4	+2.5	40.0	32.4	−7.6
δ-C14	34.0	38.0	+4.0	22.3	29.6	+7.3	44.0	54.4	+10.4
δ-C16	14.0	28.0	+17.8	3.3	27.6	+24.3	11.0	41.4	+30.4

[a] Reference, Dimick et al. (2).
[b] Not measurable on GC.

of 90% or more for each lactone and for the pooled mixture. A decrease in per cent recovery was noted with increasing lactone carbon-chain length. The decrease from 103 to 92% is offset by the increasing amount of label remaining in the butteroil and connecting tube from the deodorization flask to the first dry-ice trap. Recovery of the radioactive label in the distillate was a measure of free lactones and would not reflect the efficiency of conversion from the hydroxyacid-ester to the lactone.

Silicic acid column chromatography (SACC) has been used to isolate (2) and quantitate (5) the lactones in milk fat. Various combinations and orders of solvents have been employed to elute the lactone-rich fraction which, when followed by saponification and acidification or methylation, yields the lactones for GC analysis. This technique, reported previously from this laboratory (2), was compared with the steam-deodorization method for recovery of the aliphatic lactones. A comparison between the two methods, using three different butteroil samples, is presented in Table 2. As expected, the recovery of the δ-C16 lactone in the SACC method was greater than that recovered by steam deodorization. Interestingly, however, more δ-C8 and δ-C10 lactones were recovered when the samples were steam-deodorized. Similar losses, utilizing SACC methods for isolation, were noted by Jurriens and Oele (5).

Results of this study show that 5 hr of deodorization with at least 100 ml/hour of steam, measured as aqueous distillate, passed through the butteroil yield the maximum amount of δ-C8, δ-C10, δ-C12, and δ-C14 obtainable with the equipment employed.

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