Acknowledgment

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References


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

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 non-oxidative 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 Blender 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 150-195 °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 Erlenmeyer 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 diethylene glycol 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-
Effect of steam-deodorization time on lactone recovery from butteroil with constant-steam generation. 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 of lactones, it was necessary to strip the butteroil with a minimum of 100 ml steam per hour.

With the above parameters established, radioactive 1-C\textsuperscript{14} lactones were added to 100-g aliquots of butteroil. The amount of label present in the distillate was that assayed following the distillation 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 of 8-C\textsubscript{8}, 6-C\textsubscript{12}, 8-C\textsubscript{14}, and 10-C\textsubscript{14} lactones from constant-steam deodorization.

![Figure 2](image2.png)

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

**Table 1**

<table>
<thead>
<tr>
<th>Lactone</th>
<th>Distillate (ml)</th>
<th>Label added (cpm)</th>
<th>Label recovered in distillate (cpm)</th>
<th>Label found in connecting tube (cpm)</th>
<th>Label remaining in butteroil (cpm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>8-C\textsubscript{8}</td>
<td>650</td>
<td>328,200</td>
<td>338,000</td>
<td>103.0</td>
<td>891</td>
</tr>
<tr>
<td>6-C\textsubscript{12}</td>
<td>665</td>
<td>387,968</td>
<td>362,320</td>
<td>93.4</td>
<td>1,300</td>
</tr>
<tr>
<td>8-C\textsubscript{14}</td>
<td>740</td>
<td>431,692</td>
<td>395,400</td>
<td>91.6</td>
<td>13,232</td>
</tr>
<tr>
<td>Mixture *</td>
<td>725</td>
<td>792,061</td>
<td>710,720</td>
<td>89.7</td>
<td>7,834</td>
</tr>
</tbody>
</table>

\* One hundred-g aliquots isolated from blended cream.
\* Refers to tube connecting the deodorization flask with the first dry-ice trap.
\* Pooled aliquots of 8-C\textsubscript{8}, 6-C\textsubscript{12}, and 8-C\textsubscript{14} 1-C\textsuperscript{14} lactones.
Comparison of lactone recovery from butteroil employing steam deodorization and silicic acid column chromatography

<table>
<thead>
<tr>
<th>Lactone</th>
<th>SD</th>
<th>SACC</th>
<th>Difference</th>
</tr>
</thead>
<tbody>
<tr>
<td>δ-C8</td>
<td>2.8</td>
<td>......</td>
<td>-2.8</td>
</tr>
<tr>
<td>δ-C10</td>
<td>15.3</td>
<td>9.6</td>
<td>-5.7</td>
</tr>
<tr>
<td>δ-C12</td>
<td>23.7</td>
<td>23.2</td>
<td>+0.5</td>
</tr>
<tr>
<td>δ-C14</td>
<td>34.0</td>
<td>38.0</td>
<td>+4.0</td>
</tr>
<tr>
<td>δ-C16</td>
<td>14.0</td>
<td>28.0</td>
<td>+17.8</td>
</tr>
</tbody>
</table>

Trial 1 | Trial 2 | Trial 3
---|---|---
| SD | SACC | Difference |
| SD | SACC | Difference |
| SD | SACC | Difference |

- Reference, Dimick et al. (2).
- Not measurable on GC.

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.

Acknowledgments
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References