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ARTICLE

Determination of Shelf Life for Butter and Cheese Products in Actual and Accelerated Conditions

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Abstract

The aim of this study was to estimate the shelf life of butter and cheese products, with shelf life being a guide used to determine the storage period of food before deterioration. Butter and cheese samples stored at 10°C and 15°C had a shelf life of 221 d, while those stored at 25°C and 35°C had a shelf life of 109 d. Quality changes, including total cell count, coliform counts, *Listeria monocytogenes* counts, acid value, moisture content, pH, acidity and overall sensory evaluation, were monitored. In order to pass the overall sensory evaluation, a quality score of 5 points on a 9-point scale was required. For other quality criteria, legal quality limits were established based on the "Process Criteria and Ingredient Standard of Livestock Products" by the Animal, Plant and Fisheries Quarantine and Inspection Agency (Republic of Korea). The non-legal quality limit was estimated by regression analysis between non-quality criteria (y) and overall sensory evaluation (x). The shelf life was estimated based on the number of days that the product passed the quality limit of the quality criteria. The shelf life of samples stored at 10°C, 15°C, 25°C and 35°C was 21.94, 17.18, 6.10 and 0.58 mon, respectively, for butter and 10.81, 9.47, 4.64 and 0.20 mon, respectively, for cheese.

Key words: shelf life, butter, cheese, quality limit, accelerated conditions, guideline

Introduction

The domestic food hygiene research in Korea has advanced in recent years, but food poisoning is still present due to clustering of dietary life and growth of the food service industry (Park *et al*., 2001; Kwun and Lee, 2007; Groth, 2014). Due to this issue, food safety and sanitation methods are very important factor for consumers in purchasing food and beverages (Lee, 2001), and the growing awareness of food safety among consumers has led to increased food product safety. The safety among consumers has led to the increase in food product safety. According to Park and Min (1995), nearly 69.2% of consumers check food labeling at the time of purchase. The food label provides information regarding the product name, raw material, expiration date, preservation method, nutritional ingredients, and other details (Bernues *et al*., 2003), and contains important information that aids the consumer in ascertaining food safety (Lee and Kim, 2001). According to a survey on food safety awareness conducted by the Korean National Council of Consumer Organizations in 2009, 25.4% of consumers considered the expiration date the most important aspect of food labeling when purchasing food products; this aspect scored higher than price, raw material, origin, and food additives. A recent study by Kim and Lee (2010) also suggests that consumers consider the expiration date as the most important aspect on the labeling, above all other details (Sikorski *et al*., 1989). Based on these reports, it is clear that the expiration date is a crucial factor for purchase decisions, clearly demonstrated by the fact that consumers tend to buy products that have relatively long expiration dates, though there are no issues with the consumption of a product close to its expiration date (Evert-Arriagada *et al*., 2014). Products that are past their expiration dates are taken off the shelves and discarded. According to the Korean Consumer Agency, more than 65 million tons of food are disposed of each year due to surpassed expiration dates, resulting in high resource waste. Section 2 of the Livestock Labeling Standards, by the Animal and Plant Quarantine Agency,

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defines expiration dates as the period between production and purchase of the product during which the product can be legally sold. The expiration date is also referred to as the "Sell-by Date", where the product can be offered to the consumer (Codex, General Standard for the Labeling of Prepackaged Food). The expiration date also discloses the longest period of time the product can be sold to a consumer, as determined by the manufacturer and based on processing standards and compositional specifications (Labuza and Fu, 1993). Originally, the government recommended a certain expiration date for each type of product, until the release of expiration dates on July 1, 2002. As companies are now allowed to set the expiration dates for individual products, a growing number of companies have determined expiration dates of products without scientific evidence, while other companies copied the expiration dates of competitors. Therefore, a guideline is needed in setting expiration dates. The Korea Food and Drug Administration have proposed the development of scientifically determined guidelines for expiration dates, as well as guidelines for the storage of dairy products and other livestock products. The Product Liability Law (PL) was passed shortly after the release of expiration dates, causing the manufacturers to be responsible for mishaps due to incorrect expiration dates, and satisfying consumers by providing products with longer expiration dates and better quality control. However, small businesses, which constitute up to 70% of livestock production in Korea, may lack the necessary funding and/or advancements to conduct proper experiments. Also they are alleged to set expiration dates without scientific evidence (Hwang and Park, 2007). Issues occur when small businesses determine the expiration date of their products based solely on competing products from larger companies without considering the manufacturing process or the packaging of the product. Accordingly, the Korea Food & Drug Administration has provided a scientifically sound process of determining food product expiration dates via the "Guideline for Establishment of Shelf-life of Foods". For livestock products, however, specific regulations have not been set for expiration dates or experimental manuals, resulting in difficulties. Butter and cheese are more likely to be imported and exported than other dairy foods, such as whole milk and yogurt. When butter and cheese are circulated, the risk of deterioration increases due to long distances and time (Krause *et al.,* 2008). Although there have been several studies focused on the shelf life of whole milk (Chung *et al*., 2002; Lim, 2003; Choi, 2004), studies on other dairy products, such as butter and cheese, for determining shelf life are lacking. Therefore, we used legal and non-legal (not illegal) methods in this study to conduct quality tests of butter and cheese stored at 10°C, 15°C, 25°C, and 35°C, respectively, to identify a scientifically valid expiration date.

Materials and Methods

Storage conditions and materials

Milk product samples were chosen from dairy product companies in Korea, from brands consumed the most by the Korean population as assessed by market research. Cheese is typically pasteurized for 5 min at 80°C; a soft-process cheese consumed by many consumers in Korea was used. Butter is typically pasteurized at 85°C. Butter (unsalted) is pasteurized for 15 s at 85°C. Samples were transferred immediately to the laboratory under refrigerated conditions, and the initial sample quality analyzed. Butter and cheese were both stored at 10°C, 15°C, 25°C, and 35°C respectively. The experiment was performed at a circulating temperature of 10°C and 15°C, while the expedited experiment was performed at 25°C and 35°C. The storage temperature of 10°C is the maximum limit in a refrigerated environment; 15°C was used to simulate storage conditions in refrigerated shelves in distributing markets, considering that most dairy products have a surface temperature greater than 10°C (Korea Consumer Agency, 2007). To simulate the scenario of the consumer leaving the product at room temperature, we used a set temperature of 25°C. Butter and cheese samples used for experimentation were kept for 221 d at 10°C and 15°C. Samples that were stored at 25°C and 35°C were kept for 109 d.

Physicochemical analyses

To assess changes occurring in the samples over time, acidity, acid value, pH, and moisture content were measured. Lipid oxidation negatively affects product flavor and taste. However, as it plays a crucial part in determining the shelf life of a product, the acid value, acidity, and pH, which are indicators of rancidness, must be analyzed for butter and cheese samples (Panseri *et al*., 2011). Acidity was measured by adding 10 mL of distilled water to 10 mL of the sample along with 0.5 mL of phenolphthalein, and the solution was titrated with 0.1 N NaOH until the reddish color persisted for more than 30 s. The acid value was measured by carefully measuring out 5-10 g of the sample and adding to an erlenmeyer flask, while also adding 100 mL of ethanol-ether mixture (1:2) and titrating the solution with a 0.1 N neutral KOH-ethanol solution until the reddish color lasted for more than 30 s. The moisture content was measured by carefully weighing out 3-5 g of the sample and placing it in a dry oven set at 98-100°C for 3-5 h. The sample was then placed in a desiccator, where it was cooled for approximately 30 min, and then weighed. pH was measured using a pH meter. All of the above experiments were conducted based on the livestock processing and compositional standards as defined by the Animal and Plant Quarantine Agency (2012).

Microbiological analysis

To observe the microbiological activity over time, the number of total bacteria, coliform, and growth of *Listeria monocytogenes* (*L. monocytogenes*) was monitored. The number of bacteria was counted by plating the solution on Plate Count Agar (Difco, USA) and incubating at 37°C for approximately 48 h. Plates with 25-250 colonies were counted and multiplied by the dilution factor to determine the actual colony count. Coliform count was performed by homogenizing the undiluted solution and then diluting by decimal dilution. The resulting in *Lis. monocytogenes* dilutions were plated on PALCAM agar plates with supplement (Difco, USA) and incubated at 35°C for 24 h. Colonies with a dark brownish or blackish ring were counted and multiplied by the dilution factor to obtain the total number of colonies. All microbiological tests were carried out according to the livestock processing and composition standards (Animal and Plant Quarantine Agency, 2012).

Sensory tests

To determine changes in the quality of each sample, a sensory test was performed using sight (color, luster, appearance, molding, and yeast formation), taste (flavor and sourness), and smell (smell and odor). The sensory analysis was performed by 10 individuals trained in sensory testing and scored based on a 9-point scale, where 9 was "liked extremely," 8 was "liked very much," 7 was "liked moderately," 6 was "liked slightly," 5 was "neither liked nor disliked," 4 was "disliked slightly," 3 was "disliked moderately," 2 was "disliked very much," and 1 was "disliked extremely" (Ministry of Food and Drug Safety, 2011). The examiners were instructed to give the product a score of 5 points when the product had reached its quality limit (Kim *et al*., 1993, Pérez Elortondo *et al*., 2007).

Calculation of the appropriate shelf life of butter and cheese

Each experiment was classified as legal and non-legal. For legal indicators, the samples were considered to have passed quality when the samples were above legal specifications. For non-legal indicators, sensory evaluation and linear regression analysis were used to evaluate the standard value. However, for *L. monocytogenes*, the Guidelines for Assessing the Microbiological Safety of Ready-to-Eat Foods Place on the Market (2009) was referenced to calculate the standard values. Since the shelf life of butter and cheese is greater than 6 mon, a streamlined experiment was conducted as stated in the expiration date selection experiment guidelines, which defines a streamlined process as an experiment conducted in a harsher environment than that of its original environment because its expiration date is longer than three months. The rate constant (K) was calculated using physiochemical, microbiological, and sensory experimental data. The rate constant (K) was derived through a zero-order reaction, which displayed a constant degradation rate regardless of the quality attributes, and a first-order reaction, which showed exponential change in the degradation rate based on the quality attributes. After using the above equations, the reaction rate by temperature and linear regression model of $\ln K_1$ with 1/T was calculated and chosen as the most reliable order of reaction. The Arrhenius equation was used to select the expiration date according to $\ln K = \ln A - (Ea/R) \times 1/T$ equation for each indicator, where K is the specific reaction rate, Ea is the activation energy in kJ/mol, R is the gas constant (1.987 cal/mol K), and T is the absolute temperature (K = 273 + °C).

Results and Discussion

Changes in microbiological quality

No coliforms or bacteria were detected, indicating that the product is of acceptable quality based on its composition, according to the livestock processing and component standards. Additionally, the non-legal indictor, *L. monocytogenes*, was not detected. Although bacteria and *L. monocytogenes* were found intermittently in cheese samples after long storage periods, no trends and/or patterns were observed. Coliforms were not detected in cheese samples throughout the storage period (data not shown), while *L. monocytogenes* was detected intermittently, but did not show a consistent trend (data not shown). *Escherichia coli* O157:H7 and *L. monocytogenes* were inoculated into the butter samples, giving initial bacterial counts of 10^5 CFU/g, and after storage for 21 d at 4°C and 21°C, all samples except sweet whipped salted butter had a bacterial cell count less than 10^1 CFU/g (Sarah *et al*., 2003). These results indicate that butter and cheese do not support bacterial growth. A previous study on the effect of long-term food storage by animals (Lee and Park, 1998) found no bacterial pollutants. This corresponds with the results obtained in this study, indicating that butter and cheese, which have more fat than other products, do not support bacterial growth.

Changes in physicochemical quality

As a result of physiochemical changes observed in butter samples stored at 10°C, 15°C, 25°C, and 35°C, the initial acidity, a legal quality indicator, was 0.51 mg KOH/g on d 0, which was much lower than the quality limit of 2.80 mg KOH/g. After 221 d of storage at 10°C, the acidity was 0.67 and at 15°C, was 0.73. When stored for 109 d, the acidity was 0.71 at 25°C and 1.39 at 35°C; these results show a tendency towards increased acidity from the initial value, and imply that any increase in the storage temperature rapidly increases the acidity. pH, a non-legal quality indicator, was 6.96 in the initial butter sample; after storage for 221 d at 10°C, it was reduced to 6.33, and at 15°C was 6.31. Furthermore, sample pH after storage for 109 d at 25°C was 5.12 and 4.70 at 35°C, indicating that long-term storage of butter in cold temperatures decreases the pH, while storage at high temperatures results in a greater decrease in pH. Acid value, another non-legal quality indicator, was 0.01 in the initial butter sample. After storage for 221 d at 10°C, the acid value was 0.14, and 0.23 at 15°C. After storage for 109 d, the acid value of the sample stored at 25°C was 0.16, and 0.84 in the sample stored at 35°C.

As a result of physiochemical changes was observed in butter samples which was stored at 10°C, 15°C, 25°C, and 35°C, pH of the base sample was 5.91, and after storage for 221 d at 10°C, was 5.66, and 5.59 at 15°C. After 109 d, the sample stored at 25°C had a pH of 5.62 while the sample stored at 35°C had a pH of 5.49. The results show an inverse relationship between pH and storage time/temperature; pH falls as the storage time increases and a higher storage temperature results in a faster decrease in the pH value. The initial cheese sample acidity was 0.50. After 221 d of storage, the acidity of the sample stored at 10°C was 0.80, whereas at 15°C, it was 1.00. After 109 d, the acidity of the sample stored at 25°C was 0.94 and 1.13 at 35°C, indicating that increased storage time increases the acidity and that higher temperatures cause a rapid increase in acidity. The initial cheese sample moisture content, a non-legal indicator, was 50.23. After 221 d of storage, the moisture content of the sample stored at 10°C was 46.65 and 46.53 when the sample was stored at 15°C. After 109 d, the moisture content of the sample stored at 25°C was 47.29 and 46.55 at 35°C. These results indicate that increased storage time correlated with lower moisture content.

Changes in sensory evaluation

The sensory evaluation of butter samples, a legal indicator for evaluation, was performed using a 9-point scale (Ministry of Food and Drug Safety, MFDS. 2013), with d 0 scored as 9 points and the sensory quality limit set at 5 points. Butter samples were analyzed after storage at 10°C, 15°C, 25°C, and 35°C. The butter sample stored at 10°C scored 9 points for 67 d whereas the sample stored at 15°C scored 9 points for only 63 d. After the mentioned dates, the samples stored at 10°C and 15°C were above the quality limit of 5 points until 221 d, while the sample stored at 25°C dropped to 5 points in 1 d. Also, the sample stored at 35°C reached the sensory quality limit after 1 d. Sensory evaluation of cheese samples was analyzed after storage at 10°C, 15°C, 25°C, and 35°C. The cheese sample stored at 10°C scored 9 points for 88 d, while those stored at 15°C, 25°C, and 35°C scored 9 points up to 81, 53, and 46 d, respectively. The quality of these samples dropped after the above dates, with scores lower than 5 points.

Calculated actual shelf life

Linear regression analysis was performed based on the storage time. Calculated quality indicator standard values were used in the linear regression equation to determine the quality limit date. The linear regression equation for butter was shown in Table 1. After storage of butter and cheese samples at 10°C, 15°C, 25°C, and 35°C, the acid value, appearance, acidity, and pH were evaluated for the butter samples, while the sensory evaluation, moisture content, and acidity were evaluated for the cheese samples. The data were then used to calculate a linear regression graph based on the storage time. Counts of coliforms, total bacteria, and *L. monocytogenes*, which were never found, were excluded from the quality limit decision process. The expiration date was typically decided via determining a proper quality indicator and measuring the point at which the product loses its value by sensory evaluation. In this study, compositional specifications of the "Animal and Plant Quarantine Agency, Process Criteria and Ingredient Standard of Livestock Products" were used as quality indicators to determine the quality limit when the product reached the legal specification, and the results of the quality limit date based on the legal specifi-

Table 1. Shelf life of butter and cheese samples stored at 10°C, 15°C, and 25°C by legal quality limit

	Quality criteria	Temperature (°C)	Quality limit[2)]	Estimated shelf life (mon)
Butter	Acid value	10	2.80	107.80
		15	2.80	93.53
		25	2.80	50.57
	Sensory overall evaluation[1)]	10	5	21.94
		15	5	17.38
		25	5	7.14
Cheese	Sensory overall evaluation	10	5	23.98
		15	5	17.88
		25	5	6.51

[1)]Penal number=10; 1=very different from control, 5=different from control (quality limit of overall sensory evaluation), 9=same as control.
[2)]Legal quality limit: "Process Criteria and Ingredient Standard of Livestock Products" by Animal, Plant and Fisheries Quarantine and Inspection Agency.

Table 2. Shelf life of butter and cheese samples stored at 10°C, 15°C, and 25°C by non-legal quality limit

	Quality criteria	Temperature (°C)	Quality limit[1)]	Regression equation	Correlation coefficient	Estimated shelf life (mon)
Butter	pH	10	3.55	y = -0.0037× + 6.9999	0.8529	31.10
		15	3.94	y = -0.0043× + 7.0717	0.8425	24.25
		25	4.62	y = -0.012× + 7.235	0.6279	7.28
	Acidity	10	0.41	y = 0.0005× + 0.0474	0.5671	24.23
		15	0.43	y = 0.001× + 0.0295	0.9030	13.19
		25	0.23	y = 0.0011× + 0.0285	0.6070	6.10
Cheese	pH	10	4.35	y = -0.0018× + 5.9693	0.6578	29.98
		15	4.98	y = -0.0019× + 5.9585	0.8681	17.18
		25	5.43	y = -0.0029× + 5.9241	0.9205	5.71
	Moisture content	10	44.82	y = -0.0165× + 50.167	0.9802	10.81
		15	45.42	y = -0.0163× + 50.078	0.9873	9.47
		25	45.16	y = -0.0299× + 50.078	0.9308	5.48
	Acidity	10	1.04	y = 0.0007× + 0.6358	0.4596	19.21
		15	1.16	y = 0.0018× + 0.6263	0.8655	9.83
		25	1.03	y = 0.0027× + 0.6528	0.6081	4.64

[1)]Quality limit: Estimated by regression analysis between non-legal quality criteria (y) and overall sensory evaluation (x) during the storage period.

cations are presented in Table 1. In case of butter, acid value and sensory evaluation, which are both legal specifications, showed the following results: expiration date of the sample stored at 10°C was 107.80 mon (based on acid value) and 21.94 mon (based on sensory evaluation); at 15°C, it was 93.53 mon (based on acid value) and 17.38 mon (based on sensory evaluation); and at 25°C, it was 50.57 mon (based on acid value) and 7.14 mon (based on sensory evaluation). For quality indicators that do not have legal specifications, a linear digression equation was calculated based on quality indicators and the 9-point scale. The expiration date was determined by inserting the quality limit of 5 points into the linear digression equation (Table 2). In case of butter, measurements of acidity and pH, which are non-legal quality indicators, showed that the expiration dates of butter samples were 31.10 mon (based on pH) and 24.23 (based on acidity) when stored at 10°C; 24.25 and 13.19 mon when stored at 15°C; 7.28 and 6.10 mon when stored at 25°C. The quality limit was determined using the indicator that gave the shortest quality limit date. Therefore, when using sensory evaluation as the indicator, the quality limit date for samples stored at 10°C was 21.94 mon, which was the shortest limit when compared to results obtained using other indicators. The quality limit date for samples stored at 15°C using acidity as the indicator was 13.19 mon, which was the shortest result. Similarly, the quality limit date for samples stored at 25°C, with acidity as the indicator, was the lowest at 6.10 mon. The quality limit of cheese was also determined in a similar manner. In case of cheese, sensory evaluation, which are only legal specifications, showed the following results: expiration date of the sample stored at 10°C was 23.98 mon, at 15°C, it was 17.88 mon; and at 25°C, it was 6.51 mon. For quality indicators that do not have legal specifications, a linear digression equation was calculated based on quality indicators and the 9-point scale. In case of non-legal biomarker, for samples stored in 10°C, 15°C and 25°C, the quality limit based on the acidity, pH, and moisture which are non-legal quality indicators. It showed that the expiration dates of cheese sample were 29.98 (based on pH), 10.81 (based on moisture contents) and 19.21 mon (based on acidity) when stored at 10°C; 17.18, 9.47 and 9.83 mon when stored at 15°C; 5.71 (based on pH), 5.48 (based on moisture contents), and 4.64 (based on acidity) when stored at 25°C. The quality limit was determined using the indicator that gave the shortest quality limit date. As a result, using moisture contents as the indicator, the quality limit date for samples stored at 10°C was 10.81 mon, which was the shortest limit when compared to results obtained using other indicators. The quality limit date for samples stored at 15°C using moisture contents as the indicator was 9.47 mon, which was the shortest result. Similarly, the quality limit date for samples stored at 25°C, with acidity as the indicator, was the lowest at 4.64 mon.

Calculation of accelerative experimental shelf life

The sample stored at 35°C was tested using an accelerated model to find the quality limit date (Table 3). According to zero-order and first-order reactions, the product with the largest R^2 was chosen and used to determine the quality limit by the equation $\ln K = -(Ea/R)\times 1/T + \ln A$. For butter samples, the quality limit was based on the first-order acid value, zero-order appearance, zero-order pH, and first-order acidity. In case of butter, the zero-order acid value showed a quality limit of 9.92 mon, while zero-order sensory estimated 1.83 mon. Also, the zero-order pH estimated a quality limit of 0.58 mon, and first order acidity estimated a quality limit of 2.75. In case of cheese, the zero-order sensory value showed a quality limit of 3.49 mon, while first-order estimated 4.22 mon. Also, the zero-order moisture content estimated a quality limit of 4.28 mon, and zero order acidity estimated a quality limit of 0.20 mon.

Accordingly, the shortest quality limit date (by acidity) was chosen as the quality limit. After considering factors that may cause quality changes, such as characteristics, distribution process, and the risk involved (Kim *et al.*, 1993), a safety factor under one was chosen as an expiration date multiplication factor. As a result, Table 4 shows the shelf-life, estimated as the earliest date among all date of quality limit when each quality criteria were reached to quality limit. Shelf-life of butter was estimated after 21.94 mon at 10°C, 17.18 mon at 15°C, 6.10 mon at 25°C, and 0.58 mon at 35°C; that of cheese was estimated after

Table 3. Shelf life of butter and cheese samples stored at 35°C using an accelerated experiment

Quality criteria			Order of reaction	K[3)]	A_0[4)]-A_t[5)]	Estimated shelf life (mon)
Butter	Legal quality limit[1)]	Acid value	Zero order	0.00956	2.85	9.92
			First order	-6.38620	0.54	10.75
		Sensory overall evaluation[3)]	Zero order	0.00541	0.30	1.83
			First order	-3.44790	3.43	3.59
	Non-legal quality limit[2)]	pH	Zero order	0.13224	2.29	0.58
			First order	-3.23640	1.70	1.44
		Acidity	Zero order	0.04105	4.00	3.25
			First order	-4.94250	0.59	2.75
Cheese	Legal quality limit	Sensory overall evaluation	Zero order	0.03816	4.00	3.49
			First order	-5.30120	0.59	3.93
	Non-legal quality limit	pH	Zero order	0.00381	0.50	4.35
			First order	-7.27410	0.09	4.22
		Moisture content	Zero order	0.03706	4.76	4.28
			First order	-7.20000	0.10	4.45
		Acidity	Zero order	0.12185	0.72	0.20
			First order	-3.34640	0.89	0.84

[1)]Legal quality limit: "Process Criteria and Ingredient Standard of Livestock Products" by Animal, Plant and Fisheries Quarantine and Inspection Agency.

[2)]Non-legal quality limit: Estimated by regression analysis between quality criteria (y) and overall sensory evaluation (x) during the storage period.

[3)]K: rate constant.

[4)]Initial date of index quality attribute.

[5)] date of index quality attribute as t time passes.

Table 4. Estimated shelf-life of butter and cheese at 10°C, 15 °C, 25°C, and 35°C

Temperature (°C)	Shelf life (months)	
	Butter	Cheese
10	21.94	10.81
15	17.18	9.47
25	6.10	4.64
35	0.58	0.20

10.81 mon at 10°C, 9.47 mon at 15°C, 4.64 mon at 25°C, and 0.20 mon at 35°C. Finally, shelf-life can established to applied safety factor (<1), as shelf life can change due to factors or other quality standard criteria. Therefore, although it can not be applied to all butter and cheese products, there has been no report that butter and cheese has previously been researched on establishment in shelf life. Taken together, these results suggest that serve a basis data in establishment shelf life on butter and cheese.

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