

# FOOD SAFETY LAB / MILK QUALITY IMPROVEMENT PROGRAM



Standard Operating Procedure

Title: Gerber Method for Butterfat Measurement of Ice Cream

SOP #: 7.34 Version: 02 Revision Date: 2023-09-15 Effective Date: 2023-08-31

Author: Samuel Reichler Approved by: Nicole Martin

### <u>Gerber Method for Butterfat Measurement of Ice</u> <u>Cream</u>



Effective 2023-09-15

Version 02

Revised 2023-09-15

#### **TABLE OF CONTENTS**

1.	Introduction	3
2.	Materials	6
3.	Procedure	7
4.	Troubleshooting	10
5.	References	10
6.	Method Review and Changes	11



Effective 2023-09-15

Version 02

Revised 2023-09-15

#### SECTION 1 INTRODUCTION

#### 1.1 Purpose

The purpose of this procedure is to measure the percentage of butterfat in an ice cream sample. This will be done using the Gerber Fat Method, one of the simplest and fastest methods for butterfat determination in dairy products.

#### 1.2 Scope

This SOP is to be used by qualified and trained individuals who need to determine the butterfat content of ice cream.

#### 1.3 Definitions

**Gerber Fat Method:** The basic principle of the Gerber Fat Method is that concentrated sulfuric acid and isoamyl alcohol are mixed with a dairy product to produce an exothermic reaction that disintegrates the emulsion structure in milk to release free fat. The free fat is collected in the graduated portion of the Gerber bottle, which is calibrated to express the fat content of the product on a percentage fat by mass.

**Gerber Bottle:** Also called a butyrometer. This is a specialized piece of glassware that is used both for the chemical reaction and to directly read the butterfat percentage. It is fitted with a lock stopper to seal it securely.

### 1.4 Safety

The reagents used for the Gerber Method are very dangerous. Required laboratory PPE must be worn, including safety glasses, a lab coat, nitrile gloves, and closed-toe shoes. In addition, legs should be fully covered, and we have disposable pants available for this purpose. Full face shields are also available and can be worn if desired.



Effective 2023-09-15

Version 02

Revised 2023-09-15

Sulfuric acid can cause severe skin burns and eye damage and can produce toxic fumes that are harmful to the respiratory tract. Lab coats, safety glasses, and nitrile gloves should be worn for the duration of the experiment, and as much work as possible should be performed inside of the fume hood. If skin or eye exposure takes place, flush with plenty of water for at least 15 minutes and seek medical attention. The SDS for the sulfuric acid used in this protocol can be found here:

https://d163axztg8am2h.cloudfront.net/static/doc/d0/8a/0eb146ebf6bb650ec307 0d9f8250.pdf

Isoamyl alcohol may be harmful if swallowed, inhaled, or exposed to skin. It can cause serious eye damage. Lab coats, safety glasses, and nitrile gloves should be worn for the duration of the experiment, and as much work as possible should be performed inside of the fume hood. If skin or eye exposure takes place, flush with plenty of water for at least 15 minutes and seek medical attention. The SDS for isoamyl alcohol can be found here:

 $\frac{https://d163axztg8am2h.cloudfront.net/static/doc/c0/48/c0142ef7c52f9a7d3b16}{d20f4bf4.pdf}$ 

The lock stoppers may leak or fail if the rubber is cracked or damaged. Inspect the lock stoppers when they are fully extended with the key before insertion into the Gerber bottles. If the rubber is cracked or flaking off, discard the stopper. The lock stoppers may fail to create a secure seal if there is moisture or residual ice cream in the neck of the Gerber bottle, or if the lock stopper is not completely dry. Always keep the stoppered end of the Gerber bottle pointed away from you, and do as much work as possible either inside of the fume hood or over the sink. If you are exposed to the contents of a Gerber bottle, follow the steps outlined in the previous paragraphs for sulfuric acid and isoamyl alcohol exposure.

The Gerber bottles may break during use due to age-related stress on the glass, visible or invisible flaws in the glass, or failure to properly balance the centrifuge. Lab coats, safety glasses, and nitrile gloves should be worn for the duration of the experiment, and as much work as possible should be performed inside of the fume hood to contain a potential failure, which may occur violently. If you are exposed to the contents of a broken Gerber bottle, follow the steps outlined in the previous paragraphs for sulfuric acid and isoamyl alcohol exposure. If you have been injured by broken glass, seek immediate medical attention. The lab's chemical spill and broken glass cleanup kit can be found at the end of bench 8.



Effective 2023-09-15

Version 02

Revised 2023-09-15

Find a qualified lab member to assist you with the cleanup. If the bottle breaks inside of the centrifuge, the contents must be cleaned up as quickly as possible to avoid corrosion and damage to the centrifuge.

#### To reduce the risk of bottle breaks:

- Carefully inspect the Gerber bottles before each use to ensure they are free from cracks or chips.
- Always centrifuge the bottles with the stopper down and ensure that the bottles are fully inserted to the bottom of the centrifuge tubes.
- The centrifuge must always be balanced with bottles of the same weight. Do
  not balance the centrifuge with an empty bottle or with a bottle filled with
  only water.
- If the centrifuge vibrates or makes an unusual noise while running, stop the centrifuge immediately.
- If a break occurs inside of the centrifuge, the liquid and broken glass must be disposed of as chemical waste seek assistance from a qualified lab member to ensure proper disposal. Be sure that all broken glass is removed from the centrifuge tube, as this could cause the next bottle to sit at an off angle and greatly increase the chance of another break occurring.



Effective 2023-09-15

Version 02

Revised 2023-09-15

#### **SECTION 2 MATERIALS**

#### **Reagents:**

- Isoamyl alcohol (Weber Catalog No. 1087-00)
- Deionized water (dH<sub>2</sub>O)
- Sulfuric acid, diluted (Weber Catalog No. 1083-00)

#### **Supplies and Glassware:**

- 2 oz dairy vials
- Plastic sampling spatulas
- 25 mL disposable serological pipettes
- Disposable transfer pipettes with the ends cut off
- Gerber bottle support rack
- 20% ice cream Gerber bottles (Weber Catalog No. 1015-01)
- Bottle top dispenser for sulfuric acid
- 300 mL Erlenmeyer flask or similar vessel
- P1000 micropipette and pipette tips for isoamyl alcohol
- Lock stoppers and hand-held key. The stoppers must be completely dry at the time of use.
- Kimwipes
- Alconox detergent

#### **Equipment:**

- Balance with 0.01 g sensitivity
- Pipette controller
- Chemical fume hood
- Gerber Micro II Centrifuge
- Water bath capable of maintaining a temperature of 60-63°C



Effective 2023-09-15

Version 02

Revised 2023-09-15

#### **SECTION 3 PROCEDURES**

#### **Preparation:**

- 1. Set the water bath to 63°C and fill to a level where only the bulbs of the inverted Gerber bottles will be exposed to the air.
- 2. Transfer each ice cream sample from its original container to a 2 oz dairy vial using a sampling spatula. Allow to fully melt before use.

#### **Procedure:**

- 1. Place the 20% ice cream Gerber bottles into the bottle support rack. Ensure that the rack is balanced, it will tip over if all of the bottles are on one side. Label each bottle on the white patch using a Sharpie.
- 2. Inside of the fume hood, measure 10 mL of dilute sulfuric acid into each bottle using the bottle top dispenser. Ensure the acid completely fills the bulb at the bottom of the bottle if bubbles are present, flick the bottle to dislodge them.
- 3. Place each Gerber bottle inside of the Erlenmeyer flask and place the flask on the balance. Tare the balance to 0.
- 4. Using the cut off transfer pipette, aspirate the fully melted ice cream sample while avoiding any fruit pieces, wipe off the exterior of the pipette using a Kimwipe, and then carefully dispense the melted ice cream into the Gerber bottle, avoiding getting ice cream on the neck of the bottle. Dispense the melted ice cream dropwise at first, until the acid is fully covered, to prevent a violent reaction with the acid from occurring. Continue adding melted ice cream to the bottle in this manner until 5±0.05 g has been added.
- 5. Remove the bottle from the balance and return to the support rack. Using the 25 mL serological pipette and pipette controller, add 5 mL of dH<sub>2</sub>O to each bottle, using the water stream to rinse any ice cream clinging to the neck of the bottle downwards.
- 6. Using the micropipette and tips, add 1 mL of isoamyl alcohol to each bottle.
- 7. Using a folded Kimwipe, carefully wipe the inside of the neck of each bottle to remove residual ice cream and moisture. The neck of the bottle should appear completely clean and dry.
- 8. Insert the key into the hole on the base of the lock stopper (stopper must be completely dry) and press until the stopper is fully extended. Carefully inspect the stopper for cracks or damage, and discard it if any are present. Discoloration does not necessarily indicate damage. Insert the stopper as far as it will go into the neck of the bottle, and then remove the key to seal the bottle. The base of the stopper should be perpendicular to the length of the bottle if it is tilted, this



Effective 2023-09-15

Version 02

Revised 2023-09-15

- may indicate a poor seal and will also create difficulties when the bottle is submerged in the water bath later.
- 9. Turn on the Gerber centrifuge and run it empty for 5 minutes to preheat it to 55°C.
- 10. Inside of the fume hood, with the stoppered end of the bottle facing upward and away from your face, grasp the test bottle at the graduated column and shake it vigorously until the curd is completely digested.
- 11. Once the curd is no longer visible, continue shaking vigorously for an additional 60 seconds. Do not allow the bulb of the bottle to empty while shaking.
- 12. Holding the bottle at the stopper and neck, invert it at least 4 times to mix the acid remaining in the bulb with the contents. The bottle will be quite hot wear insulated gloves if necessary.
- 13. Before the contents of the bottles have cooled below 60°C, place the bottles stopper side down in the preheated Gerber centrifuge, ensuring that they are pushed fully down into the centrifuge tubes and that the centrifuge is correctly balanced. Close the centrifuge cover and centrifuge the bottles for 5 minutes.
- 14. Remove the bottles from the centrifuge and, without inverting, place them stopper side down in the 63°C water bath, leaving only the bulbs exposed out of the water.
- 15. After at least 5 minutes in the water bath, remove the bottles one at a time from the water bath for measurement. Working over the sink and without inverting the bottle, insert the key into the hole in the lock stopper. Push on the key to raise the bottom of the fat column upward until the fat-liquid interface meets the 0% graduation on the column. This may require some force. Avoid placing pressure on the graduated neck of the bottle.
- 16. While maintaining the bottom of the fat column at the 0% graduation, read the value at the bottom of the meniscus at the top of the fat column to the nearest 0.2% graduation. This is the percentage of butterfat in the sample. Proper fat columns contain no light or dark particles or zones. They are pale to strong yellow in color. Repeat the test from the beginning if the fat column is imperfect. Place the bottles back in the water bath after reading until all bottles have been read.
- 17. Without inverting the bottles, place them back in the centrifuge as described above and centrifuge for 5 minutes.
- 18. Return the bottles to the water bath for at least 5 minutes, and then reread the fat column as in steps 15 and 16. If the second reading differs by more than 0.2% from the first, the initial shaking was inadequate, and the test must be repeated



Effective 2023-09-15

Version 02

Revised 2023-09-15

from the beginning. When the second reading is less than 0.2% above the first, report the result of the first reading.

19. Return the bottles to the water bath after reading to keep the butterfat melted and allow the bottles to be more easily cleaned.

#### **Cleanup:**

- 1. Prepare a 1% detergent solution in a dish tub by mixing Alconox with lukewarm tap water at a rate of 10 g/L. Place a wire basket in the dish tub.
- 2. Remove the bottles from the water bath and place them stopper side up into the bottle support rack.
- 3. Remove the lock stoppers from the bottles using the key and place them in the wire basket in the detergent solution.
- 4. Agitate the basket to remove the fat and acid reaction mixture from the stoppers.
- 5. Rinse the stoppers thoroughly in 3 changes of cold water to remove the detergent.
- 6. Shake the basket to remove as much excess water as possible. Let the stoppers air dry on a paper towel or underpad.
- 7. Drain the contents of the Gerber bottles into a properly labeled chemical waste bottle.
- 8. Submerge the drained Gerber bottles in the detergent solution, allowing them to fill completely. Remove the full bottles from the detergent solution and dump the contents down the drain, **not back into the detergent solution**.
- 9. Repeat step 8 once more, for a total of 2 rinses in detergent.
- 10. Fill the bottles with lukewarm water and invert them to drain. Repeat once more for a total of 2 rinses with water.
- 11. Place the bottles upside down in a wire basket to air dry. Bottles can be placed in the drying oven to accelerate drying.
- 12. If the fat has hardened in the bottles, make the detergent solution using hot water rather than lukewarm, and let the bottles sit for 15 minutes while filled with the detergent solution, drain, then proceed from step 8.



Effective 2023-09-15

Version 02

Revised 2023-09-15

#### SECTION 4 TROUBLESHOOTING

If melted ice cream is foaming into the neck of the Gerber bottle after the water is added, preventing the secure insertion of the lock stopper, the following corrective actions can be attempted:

- 1. The melted sample can be warmed for a short time in a water bath set to 40°C to accelerate the release of air from the sample.
- 2. Samples can be left overnight at a temperature ≤4°C to allow for air in the sample to be released.

The original sources (citations below) can be referenced for additional information that may be missing from this SOP.

Larry Tozzi, the Gerber expert at the Division of Milk Control & Dairy Services, NYS Department of Agriculture & Markets, is very experienced in the technique and expressed his willingness to provide advice should we need it. His email address is <a href="mailto:Lawrence.Tozzi@agriculture.ny.gov">Lawrence.Tozzi@agriculture.ny.gov</a>.

Weber Scientific seems to know a lot about the Gerber method, so you could contact them. Dave Barbano emphatically doesn't use the Gerber method, but you could probably still ask him about it because he's basically a Dairy God.

#### SECTION 5 REFERENCES

- Standard Methods for the Examination of Dairy Products, 17<sup>th</sup> ed., §15.085 Gerber Fat Method
- Weber Scientific Memoranda accessed from the Documents tab at <a href="https://www.weberscientific.com/8-gerber-milk-test-bottle">https://www.weberscientific.com/8-gerber-milk-test-bottle</a>
  - The Gerber Test for Fresh Milk
  - o The Gerber Method for Sweetened Products and Frozen Desserts
  - Washing Gerber Equipment
  - Gerber Butyrometers Breaking Troubleshooting Checklist
- Samuel Reichler's notes from a training on the Gerber method for ice cream given by Deanna Simons, retired QA manager of Cornell Dairy, on June 2, 2023.
- Samuel Reichler's notes from a training on the Gerber method for ice cream given by Dr. Nicole Martin.



Effective 2023-09-15

Version 02

Revised 2023-09-15

- A 2023-09-15 email to Nicole Martin from Larry Tozzi, the Gerber expert at the Division of Milk Control & Dairy Services, NYS Department of Agriculture & Markets
- Protocol titled "Determination of Butterfat Content of Ice Cream Mix by Gerber Method," written and last updated in November 2008 by former MQIP technician Pat Wood.
- Gerber Centrifuge Micro II Instruction Manual, 2004
- Email



Effective 2023-09-15

Version 02

Revised 2023-09-15

### SECTION 6 METHOD VERSION & CHANGES

VERSION	DATE	EDITOR	COMMENTS
Version 1	2023-08-31	sjr267	Original SOP
Version 2	2023-09-15	Sjr267	Addition of troubleshooting steps for foamy ice cream and a contact at Ag & Markets experienced in the Gerber method.
Version 3			