Supplementary Guidance Documents

Purpose of Supplementary Guidance Documents (SG)

Detailed procedures have been developed that are used in the various test methods that are a part of the Guidelines for Assessing the Flushability of Disposable Nonwoven Products.

Since these procedures are common to multiple test methods, they have been described once in the Supplementary Guidance Documents, which are referenced within the test methods. Five supplementary guidance documents are collated in this single supplement for use in conjunction with the Test Methods to carry out a flushability assessment for disposable nonwoven wipes.

The Supplemental Guidance Documents needed for a given test are referenced in Section 3 of the Test Methods. In addition, the Test Methods, for which such guidance may be needed, are referenced in Section 2 of each Supplemental Guidance Document.

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1.0 Introduction

This Supplementary Guidance Document includes recipes for preparing two types of simulated fecal material (SFM), either of which can be used for the test at the discretion of the sponsor or study director. However, only one type of SFM should be used within a study.

2.0 Test Methods

This Supplementary Guidance Document is relevant for the following test methods:

- FG501.R1(18): Toilet and Drain-line Clearance Test
- FG503.R1(18): Household Pump Test

3.0 Safe Practices

These procedures involve handling boiling water as well as the use of electrical and mechanical equipment. Precautions should be taken to avoid and protect from accidental exposure to hot liquids. Moreover, individuals should avoid any exposure to moving parts and minimize risks of electrical shock. Each laboratory is responsible for developing and implementing its own safe practices for this procedure including use of appropriate personal protective equipment and lock-out procedures for mechanical and electrical equipment.

4.0 Recipe 1: Dog Food Based SFM

4.1 Ingredients for 36 pieces each with a mass of 50 - 55 g

- 59 oz. (1673 g) of Gravy Train® with Beef Chunks dog food or equivalent
- 3/4 oz. (21 g) gelatin
- 350 mL of water

4.2 Equipment

- Large mixing bowl;
- Electric mixer (e.g. Kitchenaid® Stand Mixer);
- Can opener;
- Suitable container in which to heat water;
- Device for heating water;
- 500 mL measuring cup;
- Mixing spoon or fork;
- 9" (23 cm) x 13" (33 cm) x 2" (5 cm) aluminum cake pan;
- Refrigerator;
- Cutting templates;
- Palette knife or similar;
- Spatula.
4.3 Procedure

1. Place dog food into a mixing bowl.
2. Stir for two minutes at lowest speed setting.
3. Add gelatin to 175 mL of cold water and stir until it is clear (approximately two to three minutes).
4. Bring 175 mL of water to a boil.
5. Add this boiling water to the gelatin mixture and stir for one minute.
6. Pour this mixture into the dog food and mix for two minutes at the lowest speed setting.
7. Pour this mixture into cake pan and refrigerate overnight.
8. Using a template as a guide, cut the solidified mixture into 36 pieces with a nominal size of 3" (7.6 cm) x 1.1" (2.8 cm).

4.4 Storage

- The SFM pieces should be used within two days.
- For extended life, SFM can be stored in the refrigerator until use.
- Stored SFM should be discarded when it becomes less solid or otherwise loses its integrity.

5.0 Recipe 2: Feclone® Based SFM

5.1 Ingredients for one batch

- 2.9 L of tap water;
- 1 bag of Feclone® BFPS-7
  (SiliClone Studios, 230 Jug Hollow Road, P.O. Box 86, Valley Forge, PA 19481);
- 20 mL of a dishwashing detergent (e.g. Dawn) solution - 1.3% (v/v) in tap water.

5.2 Equipment:

- Large mixer (e.g., Hobart™ mixer) with a bowl sufficiently large to mix at least a gallon of material safely;
- Plastic 50 mL centrifuge tubes with screw caps (approximately 100) with holes poked in their conical end;
- A sausage making device (e.g. Hobart 4812 Grinder/Stuffer, see below) for filling the 50 mL centrifuge tubes (recommended);
- Large spatula;
- Suitable container in which to heat water;
- Device for heating water.

### Examples of Equipment
- Hobart Mixer (image left)
- Hobart 4812 Grinder/Stuffer (image right)

#### 5.3 Procedure

1. Bring the 2.9 L of tap water to a boil.
2. Slowly pour the boiling water into the mixing bowl of the food mixer.
3. Add one bag (approximately 900 g) of Feclone™ BFPS-7 dry concentrate or equivalent.
4. Add 20 mL of 1.3% (v/v) dishwashing detergent solution.
5. Mix on low speed for one minute.
6. Stop the mixer and scrape the sides of the bowl to mix in all of the dry powder.
7. Increase the mixer speed to medium and continue mixing an additional two minutes or until fully mixed.
8. Using a spatula, transfer the mixture into 50 mL centrifuge tubes.
9. Alternatively, transfer the material to a pan, and cut the SFM into small squares.
10. Place these small squares in the chute of the sausage filling device while extruding the SFM into 50 mL centrifuge tubes.
11. Replace the screw caps on the end of the tubes and store them in a refrigerator (4°C).

#### 5.4 Storage

- The SFM can be stored in the refrigerator for up to three days.
- If the storage time will be longer, store in the freezer (-10°C) to prevent fungal growth.

#### 5.5 Preparation for Use

- Bring the the SFM to room temperature and remove it from the tube immediately before use.

#### 5.6 Cleaning Procedure

- After using the SFM, the tubes should be washed in a soapy solution (or a glassware cleaner) and scrubbed with a brush to remove all remaining SFM.
- Soak the tubes in a dilute household chlorine bleach solution (250 mL of household chlorine bleach diluted in 16 L of tap water) for at least 1 h, rinse with tap water and air dry before refilling.
## 6.0 Version Control

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1.0 Introduction

This Supplementary Guidance Document describes two approaches for pre-rinsing test samples to remove lotions or other additives from products before using them in a test. The first method, which is recommended, involves flushing the samples down a toilet and through a drain-line using tap water. This approach simulates the rinsing process that occurs when a material is flushed on its way to a wastewater conveyance system. When a toilet and drain-line are not available, an alternative method can be used that involves swirling samples in a container of tap water. This method is specifically designed for disposable nonwoven wipes; use with other products must be assessed on a product-by-product basis.

2.0 Test Methods

This Supplementary Guidance Document is relevant for the following test methods:

- FG502.R1(18): Slosh Box Disintegration Test
- FG503.R1(18): Household Pump Test
- FG504.R1(18): Settling Test
- FG505.R1(18): Aerobic (A) Biodisintegration/(B) Biodegradation Tests
- FG506.R1(18): Anaerobic (A) Biodisintegration/(B) Biodegradation Tests

3.0 Test Product Selection

- When conducting a test to support a flushable claim, the products used for testing must be the same as those offered in the intended market.
- Obtain a sufficient number of samples to conduct the intended tests.
- If there is a need to determine the average dry weight for the test material, at least ten more samples will be needed, and when samples exhibit high variability in their weight, more than ten may be needed.
- Test samples should be randomly obtained from different sections of one or more packages to ensure that they are broadly representative. This is particularly important for test materials such as wipes, which occur in a roll or stack.

4.0 Toilet and Drain-line Method

4.1 Equipment

- Toilet and drain-line with catch basket before the drain (refer to FG501.R1(18) for set up).

4.2 Procedure

- Prior to adding any materials to the toilet bowl or initiating a flush, ensure that the toilet has stopped running and the water in the bowl is at a normal level.
When adding a test sample (e.g. nonwoven wipe) place it in the center of the toilet bowl and allow sufficient time for it to become fully saturated with water (typically ten seconds) before adding another sample or flushing the toilet.

- For guidance on the number of samples to add to each flush, one can refer to FG501.R1(18). However, as a default for nonwoven wipes, no more than two wipes should be flushed at one time.
- Retrieve the samples before they enter the basket or as soon as practically possible to prevent any disintegration by water flowing out of the pipe.
- When necessary, use additional flushes without test samples to move materials out of the drain-line for collection.

### 5.0 Alternative Method

#### 5.1 Equipment

- Containers with a capacity of approximately 20 L (e.g. 5 gallon plastic buckets).

#### 5.2 Procedure

- Fill the containers with tap water.
- Submerge the test samples in the water and swirl them for approximately 30 secs or longer if necessary to remove any perceptible lotion or additives.
- To maintain the ratio of water to test product existing in the toilet and drain-line above, no more than six samples should be placed together at one time in a single container with 20 L of tap water.

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1.0 Introduction

This Supplementary Guidance Document describes the procedures for determining dry weights of products that are used for various tests and product residues recovered at the end of disintegration tests. This method is specifically designed for disposable nonwoven wipes; use with other products must be assessed on a product-by-product basis.

2.0 Test Methods

This Supplementary Guidance Document is relevant for the following test methods:

- FG501.R1(18): Toilet and Drain-line Clearance Test
- FG502.R1(18): Slosh Box Disintegration Test
- FG505.R1(18): Aerobic (A) Biodisintegration/(B) Biodegradation Tests
- FG506.R1(18): Anaerobic (A) Biodisintegration/(B) Biodegradation Tests

3.0 Test Sample Selection

- When conducting a test to support a flushable claim, the products used for testing must be the same as those offered in the intended market.
- To determine the average dry weight for the test material, at least ten samples will be needed, and when samples exhibit high variability in their weight, more may be needed.
- Test samples should be randomly obtained from different sections of one or more packages to ensure that they are broadly representative. This is particularly important for test products such as wipes, which occur in a roll or stack.

4.0 Drying and Weighing of Samples and Sample Residues

4.1 Equipment

- Oven capable of maintaining a constant temperature between 40° and 100°C;
- Weighing dishes;
- Forceps;
- Desiccator;
- Analytical Balance (reads to 4 decimal places in grams).

4.2 Procedure

- Test samples with lotions or additives should be pre-rinsed using the procedures described in Supplementary Guidance Document SG001.R1(18) prior to determining their dry weight.
- Set the oven to a temperature appropriate for the chemical and physical properties of the sample in order to fully evaporate moisture in the material without causing
thermal degradation. This is typically done at 60°C, but could be done as low as 40°C.
• Place product samples to be analyzed in an oven safe weighing dish or on a piece of foil.
• In the case of difficult to handle sample residues, it may be appropriate to place the samples into pre-weighed (tared) aluminum weigh pans.
• Dry the samples overnight in the oven. To achieve a steady-state weight, samples are typically dried for a minimum of twelve hours.
• Transfer the samples from the oven to a desiccator and allow to them to cool.
• Weigh the samples and record the sample weights.
• Return the samples to the oven for approximately 30 minutes and once again allow them to cool in a desiccator and determine their weight.
• Repeat this process as necessary until they reach constant weights.
• Calculate and record the average and standard deviation for the dried weights of the samples.

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1.0 Introduction

This Supplementary Guidance Document describes procedures regarding the collection, transport and storage of untreated wastewater, activated sludge and anaerobic digester sludge. In addition, it describes procedures for characterizing samples of these environmental matrices and preparing them for use in the various tests in which they may be used.

2.0 Test Methods

This Supplementary Guidance Document is relevant for the following test methods:

- FG502.R1(18): Slosh Box Disintegration Test
- FG504.R1(18): Settling Test
- FG505.R1(18): Aerobic (A) Biodisintegration/(B) Biodegradation Tests
- FG506.R1(18): Anaerobic (A) Biodisintegration/(B) Biodegradation Tests

3.0 Safe Practices

These procedures involve untreated wastewater, activated sludge and anaerobic digester sludge. Individuals performing this test should take measures to prevent direct and indirect exposure to these environmental matrices, which could contain microorganisms and chemicals with unknown etiological and toxicological properties. Each laboratory is responsible for developing and implementing its own safe practices for this test including use of appropriate personal protective equipment.

**WARNING:** Digester sludge produces flammable gases that present fire and explosion risks. Gas generated from digester sludge placed in a sealed container can produce pressure sufficient to pose an explosion hazard.

4.0 Collection, Transport and Storage of Environmental Samples

4.1 Equipment

- 10 L Nalgene plastic bottles or equivalent with screw-on unvented caps or other suitable containers for transporting and storing wastewater samples;
- 20 L plastic buckets with lids that seal or other suitable containers for transporting sludge samples;
- A dipper on a pole (recommended) or other sampling device. A dipper can be fabricated or purchased. Suppliers include [www.envexp.com](http://www.envexp.com), [www.conbar.com](http://www.conbar.com) and [www.belart.com](http://www.belart.com).
4.2 Untreated Wastewater

- Collect wastewater from a municipal treatment plant that receives wastewater from predominantly residential sources and has low levels of industrial inputs.
- DO NOT collect wastewater if there has been heavy rainfall in the past 24 hours to ensure adequate solids content.
- Obtain wastewater at a point in the facility that is located up-flow from any screening or grinding processes.
- Use a dipper, pump or other method that minimizes personal contact with wastewater when acquiring the samples.
- Place samples into 10 L Nalgene plastic bottles or other suitable containers, which are sealed and transported to the laboratory.
- While it is recommended that it be used immediately, wastewater can be stored in open containers (e.g. plastic buckets) for up to 48 hours in a cool, well-ventilated area away from direct sunlight. The samples and the area should have appropriate labeling and signage regarding any potential biohazard risks.

4.3 Activated Sludge

- Collect activated sludge from a municipal treatment plant that receives wastewater from predominantly residential sources and has low levels of industrial inputs.
- Obtain the activated sludge directly from the aeration basin.
- DO NOT collect activated sludge if there has been a moderate to heavy amount of rain in the past 48 hours to ensure adequate solids content.
- Use a dipper, pump or other approach that minimizes personal contact with activated sludge when acquiring the material.
- Place the material in 20 L buckets or other suitable containers. Do not fill the containers to more than 75% of their capacity. Seal the containers and transport them to the laboratory.
- While it is recommended that it be used immediately, activated sludge can be stored in open containers (e.g. plastic buckets) with aeration for up to 48 hours in a cool, well-ventilated area away from direct sunlight. The containers of sludge and the area should have appropriate labeling and signage regarding any potential biohazard risks.

4.4 Anaerobic Digester Sludge

- Collect anaerobic digester sludge from a municipal treatment plant that receives wastewater from predominantly residential sources and has low levels of industrial inputs.
- Obtain the anaerobic sludge from a second stage digester if available or where the wastewater treatment operator advises that the sludge is stabilized but still active.
- Before sampling, purge all sludge from any pipes or valves to ensure that the material is being sourced from the actual digester and is fresh.
- Place the sampled sludge in 20 L buckets or other suitable containers. Do not fill the containers to more than 75% of their capacity.
• Seal the containers and transport them to the laboratory. *Note: Never keep anaerobic sludge in sealed containers for a long period to prevent a build-up of pressure. Depending upon travel time, it may be necessary to use a vented container or periodically release any pressure.*

• While it is recommended that it be used immediately, anaerobic digester sludge can be stored for up to 48 hours, preferably at 35°C, in a well-ventilated area away from direct sunlight. Any storage container should be sealed and vented in such a way that it limits intrusion of air but prevents any build-up of pressure. The containers of material and the area should have appropriate labeling and signage regarding any potential biohazard risks.

**5.0 Characterization and Preparation of Environmental Samples for Testing**

**5.1 Equipment**

• 1 mm wire mesh sieve; USA Standard testing sieve #18 (1 mm mesh size): 20 cm in diameter; or equivalent ISO 3310-1 and BS410-1;
• pH Meter;
• Portable Suspended Solids (TSS) Analyzer (e.g. Insite IG Model 3150);
• Equipment for determining Total Suspended Solids (TSS) using Method No. 2540D in Standard Methods for the Examination of Water and Wastewater. Refer to the method, which can be found at [http://www.standardmethods.org](http://www.standardmethods.org);
• Equipment for determining Total Solids (TS) using Method No. 2540B in Standard Methods for the Examination of Water and Wastewater. Refer to the method, which can be found at [http://www.standardmethods.org](http://www.standardmethods.org);
• Thermometer or other temperature measuring device.

**5.2 Untreated Wastewater**

• Prior to use, sieve untreated wastewater through a 2 mm wire mesh sieve to remove any large solids.
• Measure and record the pH of the wastewater. If the pH is between pH 6 and pH 9, the wastewater can be used. Otherwise, new wastewater should be collected.
• Stir the wastewater to evenly distribute the solids and determine the level of total suspended solids (TSS) using a TSS analyzer or the procedures described in Method 2540D in Standard Methods. If the TSS level between 75 and 400 mg/L, the wastewater can be used. Otherwise, new wastewater should be collected.
• Allow the temperature of the wastewater to equilibrate with the temperature in the laboratory before using it in a test.

**5.3 Activated Sludge**

• Prior to use, sieve the activated sludge through a 1 mm wire mesh sieve to remove any large solids.
• Measure and record the pH of the activated sludge. If the pH is between pH 6 and pH 9, it can be used. Otherwise, new activated sludge should be collected.
• Stir the activated sludge to evenly distribute the solids and determine the level of total suspended solids (TSS) using a TSS analyzer or the procedures described in Method 2540D in Standard Methods.

• If the solids level does not fall between 2000 and 4500 mg/L, adjust the level using one of the following approaches;
  o To increase the solids level, allow the sludge to settle and decant sufficient supernatant that once re-suspended the solids level will be in the designated range.
  o To decrease the level of solids, dilute the sludge with supernatant or untreated wastewater.

• After adjusting the solids level, it is recommended that the TSS level be re-determined on the actual sludge used in the test.

• Allow the temperature of the sludge to equilibrate with the temperature in the laboratory before using it in a test.

5.4 Anaerobic Digester Sludge

• When manipulating anaerobic digester sludge, measures must be taken to ensure that contact between the sludge and oxygen (air) is limited as much as practically possible. Suggested approaches include: a) performing procedures in a glove box containing an oxygen free atmosphere, b) maintaining a blanket of flowing nitrogen on the surface of sludge, when it is open to the air, c) purging containers with flowing nitrogen prior to and after adding digester sludge to displace any air before closing them, and d) rather than pouring, using a peristaltic pump to transfer sludge from one container to another.

• Prior to use, sieve the digester sludge through a 1 mm wire mesh sieve to remove any large solids.

• Measure and record the pH of the digester sludge. If the pH is between pH 6 and pH 9, it can be used. Otherwise, new digester sludge should be collected.

• Stir the sludge to evenly distribute the solids and determine the level of total solids (TS) using the procedures described in Method 2540B in Standard Methods.

• If the solids level does not fall between 8,000 and 10,000 mg/L, adjust the level using one of the following approaches;
  o To increase the solids level, allow the sludge to settle and decant sufficient supernatant that once re-suspended the solids level will be in the designated range.
  o To decrease the level of solids, dilute the sludge with supernatant or untreated wastewater.

• After adjusting the solids level, it is recommended that the TS level be re-determined on the actual sludge used in the test.
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1.0 Introduction

This Supplementary Guidance Document describes the sieving, rinsing and recovery of sample residues from the various disintegration tests. Once samples are transferred to a sieve in these tests, these procedures are used to rinse small materials through the sieve and recover the residues for gravimetric analysis. This method is specifically designed for disposable nonwoven wipes; use with other products must be assessed on a product-by-product basis.

2.0 Test Methods

This Supplementary Guidance Document is relevant for the following test methods:

- FG502.R1(18): Slosh Box Disintegration Test
- FG505.R1(18): Aerobic (A) Biodisintegration/(B) Biodegradation Tests
- FG506.R1(18): Anaerobic (A) Biodisintegration/(B) Biodegradation Tests

3.0 Equipment

- Peerless shower head Model 76114WH with hose assembly (pictured at right) attached to a faucet (tap) with a regulator adjusted to deliver 4 L/min;
- 4 L beaker (recommended);
- Stopwatch or other timing device;
- Forceps;
- Drying pans.

4.0 Procedures

1. Turn on the faucet and adjust the regulator to a flow rate of 4 L/min. The flow rate can be determined by measuring the volume delivered to a suitable container with graduations after a specified time period. For example, it should take exactly 60 seconds to deliver 4 L of water to the 4 L mark on a beaker. Once the flow is adjusted, this measurement should be repeated at least three times and should vary less than 5%.

2. Locate the sieve directly over the sink drain and ensure the sieve face is raised off the sink surface to allow the shower to freely drain through the sieve. Alternatively, a sieve spacer can be used to raise the sieve off the sink surface per the image below. Ensure that the sieve is placed such that potential back-splashing is minimized.

3. When transferring the contents from a disintegration test to the sieve, pour the contents of the test vessels slowly while distributing them over the complete surface of the sieve.
4. With the handheld showerhead spray nozzle held approximately 10-15 cm (4-6 in.) above the top surface, gently rinse smaller materials through the sieve. Constantly move the spray over the entire surface without concentrating the spray on any specific areas. Do not force the passage of any material through the sieve.

5. After two minutes of rinsing, quantitatively recover all the retained materials from both sides of the sieve using forceps or by backwashing the material into a smaller sieve and then using forceps. (Note: If any bio-solids are retained on the residual product see Section 5.0.)

6. Transfer these materials into labeled drying pans or tared aluminum weigh pans to determine their dry weight (refer to Supplementary Guidance Document SG002.R1(18)).

![Sieve with spacer.](image)

**5.0 Procedure for Separating Sludge Solids from Sample Residue**

In order to get an accurate weight when processing the anaerobic and aerobic biodisintegration samples, it is important to remove as much sludge as possible during the rinsing and the sieving steps. However, it can be difficult to separate sludge solids from the bits of sample residue. When this situation arises use the following procedure:

- With the forceps, hold the residue collected from the sieve below the surface of tap water contained in a small container (<250 mL) and gently swirl the residue to float off any sludge solids.
- Alternatively, this residue can be placed in a small volume of water, and forceps are used to separate pieces of the sample (e.g. fibers) from the sludge.
- After both these processes, great care must be taken to ensure that all sample residues are quantitatively recovered for dry weight analysis.
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