The Association of Postconsumer Plastic Recyclers

PET Oven Bake Test

PET-R-01

The following protocol is designed to provide guidance on detecting and quantifying contaminants, which discolor during oven baking of PET flake. PVC flakes discolor under these conditions.

This protocol does not purport to address all of the safety issues, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.

Equipment/Supplies List

- PET flakes about 1 cm (3/8 inch) nominal
  - Either cleaned by other methods or dirty and to be cleaned by this procedure
  - Be sure to collect a representative sample
- Circulating or forced air high temperature oven equipped with a temperature readout device (220°C capability). Should be able to hold temperature to +/- 5°C.
- Baking pans, typically 23 x 33 x 5 cm (9 x 13 x 2 inches), sized to fit into the oven.
- Balance:
  - One suitable for weighing 500 grams +/- 0.001 grams.
- Tweezers
- Hot plate capable of heating to 190^0C
- Beaker or metal pail capable of holding 10 liters of very hot water
- Thermally insulated gloves, potholders, etc.
- Bench-top drill press fitted with paint stirrer or a laboratory blender (needed for dirty flake only)
- One 20 liter (five-gallon) metal pail (needed for dirty flake only)
- Kitchen strainer sized to fit onto 20 liter pail, capable of holding over 0.5 kg (1

pound) of flake (needed for dirty flake only)

- Triton® X-100 nonionic surfactant
- Caustic (granular NaOH)

Be sure to read all material safety data sheets.

Developed from a procedure developed for NAPCOR, National Association for PET Container Resources Used with permission from NAPCOR. NAPCOR is not responsible for the details of this procedure.

Preparation of PET Sample Flake

Be sure to use appropriate laboratory safety procedures / Gloves, safety glasses, etc.

NOTE: This test has been sized for the analysis of 500 gram quantities of flake. If larger quantities are used, the length of the heating time in the oven may have to be increased.

Procedure for cleaned and washed flake only:

Step A1.

1. Select about 500 grams (1 pound) of flake for the test and obtain an accurate weight to +/- 0.001 grams.
2. Spread the flake out onto the baking pan.
3. Proceed to Step B1 below.

Procedure for cleaning dirty ground flake:

Step A2.

1. Select about 0.5 kg (nominally, about 1 pound) of dirty flake for the sample to be tested.
2. Obtain an accurate weight of the sample to +/- 0.001 grams.
3. Prepare "wash" solution in the beaker consisting of 3,300 ml water, ~0.3% by weight (10.0 grams or 9.5 m1*)* of Triton® X-l00 and 1.0% by weight (33 grams) caustic (NaOH).

Note: Triton X-100 must be dissolved in cold water prior to the addition of caustic!

BE CARFUL TO AVOID SKIN OR EYE CONTACT WITH THE CAUSTIC OR WASH SOLUTION

4. Heat wash water to 87°C (190°F) +/-3°C. Place beaker of hot solution on sturdy surface adjacent to the stirring mechanism.

   BE CAREFUL TO AVOID BURNS OR DIRECT EXPOSURE TO THE WATER. THE TEMPERATURE IS SUFFICIENT TO CREATE THIRD DEGREE BURNS TO SKIN.

5. Place the dirty flake into wash solution in the 10 liter beaker.
6. Cover the beaker with a lid. A long paint stirrer can be fitter through a hole placed in the center of the lid.
7. Position the beaker under the drill press or laboratory stirrer. Attach stirrer and vigorously stir the mixture for 15 minutes.

   BE CAREFUL NOT TO SPLASH LIQUID OR EXPOSE SKIN TO VERY HOT LIQUID

8. Remove from stirrer and add cold water to bring the level up to within several centimeters of the top of the pail.
9. Gently stir to break up foam and to help separate trapped “floatables” from the PET flake.
10. Wait five (5) minutes to allow floatables to rise and the PET to sink while gently stirring the floatables to allow any trapped PET or PVC to sink.
11. Place a strainer over a 20 liter pail.
12. Carefully pour out most of the water, causing the floatables to be trapped in the strainer during the transfer. Do not allow PET flake to escape from the initial 10 liter beaker. Dispose of the diluted wash water properly.
13. Add more cold water to the PET flake. Again bring the water level up to within several centimeters of the top of the pail.
14. Vigorously hand stir the flake/water mixture to allow further separation of trapped floatables.
15. Allow the mix to resettle. Gently stir the floatables to allow any trapped PET or PVC particles to sink.

16. Pour off the majority of the water through the strainer, again removing the floatables in the process
17. Transfer the trapped floatables to paper towels or newspaper and allow to air dry.
18. Accurately weigh the floatables to +/- 0.001 grams
19. Clean the strainer.
20. Isolate the PET flake from the last remaining water in the initial pail by pouring the mix through the cleaned strainer. Transfer all of the PET flake into the strainer. Do not lose any of the sample.
21. Place the PET, and potentially PVC, flake in the strainer over a sink for several minutes to allow any water to drain. Tilt the strainer to assist in removal of trapped water.
22. Allow the flake sample to air dry, or hasten drying in a low temperature (<50°C or <122°F), drying oven. Flakes must be free of moisture to proceed. Touching flake with clean tissue and examining tissue for water pickup can assess surface moisture.
23. Weigh accurately about 500 grams of sample flake, measured to +/- 0.001 grams, and place into the baking pan. Spread the flake evenly in the pan.
24. Proceed to Step B1.

Oven Test Procedure

Be sure to use appropriate laboratory safety procedures / Gloves, safety glasses, etc.

Step B1. Baking Procedure

1. Adjust the oven temperature to 208°C +/- 5°C and allow the oven to equilibrate to that temperature.
2. Place the pan of flake into the oven quickly.
3. Allow oven temperature to restabilize to the baking temperature before starting the timing.
4. After 90 +/-5 minutes of baking, remove the hot pan from the oven and place the pan on a suitable surface to cool
5. Spread the cooled, baked flake out onto a clean, white work surface.
6. Using a pair of tweezers and good lighting, sort through the flake and remove any black, burnt pieces of plastic.
7. Typically, burnt PVC pieces can be easily isolated. One the other hand, burnt,

melted polyethylene or PET with blacked glue residue usually have a different physical appearance than does PVC and usually cannot be separated as easily from the attached PET flake as can PVC.

Step B1A. Alternate Baking Procedure

1. Baking at 232°C (450°F) for 30 minutes has been found to be satisfactory for darkening PVC flakes. The operator should be aware, though, that some PVC may degrade at the higher temperature and the calculated parts per million may be lower than actually present.

   The higher baking temperature with shorter exposure time is useful in monitoring the reclaiming process. The lower baking temperature with longer exposure time is useful in qualifying sales product.

Step B2. Optional PVC Verification

1. Heat a copper wire in a propane torch flame until it glows red.
2. Touch the hot wire to the blackened, suspect particle and melt a small amount of the plastic onto the wire.
3. Reheat the wire tip with the plastic attached in the flame of the torch.
4. If the material is PVC, a bright green flame will be visible as the wire heats up and the plastic burns off. If there is no green flame, the particle is not PVC.

PVC ppm Calculations and Reporting

1. Measure the weight of the PET flake sample obtained for testing.

   Example: 498.951 grams starting flake sample

2. Obtain the true weight of the PET sample by subtracting the weight of any dried floatables from the starting weight of the flake sample

   Example: 498.951 grams flake sample – 103.023 grams floatables =
   395.928 grams PET flake

3. Calculate the weight percent of floatables in the sample by dividing the weight of the floatables by the starting weight of the sample flake and multiplying by 100.

   Example: (103.023 grams/498.951 grams) x 100 = 20.6% floatables

4. Calculate the PVC contamination level in parts per million (ppm) by dividing the weight of the PVC found in grams by the weight of the PET flake in grams, then multiplying the result by 1,000,000

   Example: (0.125 grams PVC/395.928 grams PET) x 1,000,000 = 316 ppm

5. Report the following:
   a. Floatables, percent of flake, (for dirty flake only)
   b. PVC, parts per million, ppm

Test Comments

- Paper residues, polyethylene, and glue residues on the PET flake will all complicate the test. It is preferable to run this test on a flake sample that is as clean as possible. The presence of these contaminants will usually require running the confirmatory copper wire flame test for PVC.
- While placing wet flake into the high temperature oven can be done, the moisture present will tend to keep the flake cooler for a longer period of time. The use of surface-moisture dry flake is important to obtaining correct results. Longer heating times, over 90 minutes, may be necessary to compensate for baking wet flake.
- If the heating time is too short, the PVC may not char completely and the discoloration may stop at the yellow/orange stage. Insufficient heating may affect the test results since the yellow/orange colors are also typically produced when glue residue are heated in this same oven test. It is imperative, therefore, that the PVC be heated long enough and at a high enough temperature to totally blacken the PVC flakes. PVC flakes buried in the middle of the mound of PET may not degrade as quickly as flake on the bottom of the pan or as quickly as

that which lies in the exterior regions of the pile of PET flake.

- A small pan of known PVC flake should be placed in the oven along with the pan of test material and observed for charring so as to be sure the time and temperature of exposure has been sufficient to discolor PVC.

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The guidance contained in this document does not guarantee acceptance or rejection of tested materials. APR encourages users of this protocol to discuss results with various knowledgeable parties using and specifying post consumer PET material to determine usefulness of subject test materials. Any guarantees or warranties are expressly disclaimed, including without limitation any implied warranties of MERCHANTABILITY or FITNESS FOR A PARTICULAR PURPOSE.