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Result:	TESTING COMPLETED	Report Date: 05-JAN-2011
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Customer Name:	BPI	
Tested To:	ASTM Standard D5511	
Description:	Plastic Bottles	
Test Type:	Biodegradability Testing	
Job Number:	J-00091962	
Project Number:	9091399	
Project Manager:	Cheryl Navarro	

Thank you for having your product tested by NSF International.

Please contact your Project Manager if you have any questions or concerns pertaining to this report.

Report Authorization: _____

Ata Ciechanowski, P.E. Assistant Director- Engineering Laboratory

Objective:

The objective of this test is to evaluate plastic bottles as per the requirements of ASTM D5511, “*Standard Test Method for Determining Anaerobic Biodegradation of Plastic Materials Under High-Solids Anaerobic-Digestion Conditions*”. The plastic bottles were supplied by BPI.

Sample Description:

Testing was performed on the AQUAMANTRA ENSO plastic bottles. The photograph in Figure 3 shows the plastic bottle samples cut up in to small pieces of 2x2 cm. The AQUAMANTRA ENSO bottle is PET mixed with an organic additive.



Figure 1: Sample of Plastic Bottle



Figure 2: Sample of the Biodegradable AQUAMANTRA ENSO Plastic Bottles



Figure 3: Sample of Plastic Bottles Cut into 2x2 cm for Degradation Experiment.



Test Protocol:

Determining the degree of anaerobic biodegradation of the plastic bottles was performed as per section 9.1 of ASTM D5511. The anaerobic digested sewage sludge mixed with household waste was obtained from the Portage Lake Water and Sewage Authority in Houghton, Michigan. A sample of the anaerobic digested sewage sludge was analyzed for pH, percent dry solids, and volatile solids, as well as, the amount of CO₂ and CH₄ evolution during the testing. Table 1 lists the results of this initial testing.

Table 1: Sewage Sludge Properties

Property	Requirement	Actual
pH	7.5 to 8.5	7.8
Kjeldahl nitrogen	0.5 and 2g/kg wet weigh	7800 mg/L
Dry Solids at 105 degrees C	20%	16.7%
Volatile Solids at 550 degrees C	N/A	60% of dry solid

Each vessel was charged with approximately 1000 grams of anaerobic digested sewage sludge mixed with household waste that contained roughly 25 grams of dry inoculums. The following table shows that each vessel was charged with approximately 25 g of the plastic bottle samples.

Table 2: Experimental Charge

	Vessel #1	Vessel #2	Vessel #3
Anaerobic Digested Sewage Sludge (g)	1003.53	1000.28	1004.32
Cut Pieces of Plastic Bottles(g)	25.04	25.04	25.06

The charged vessels were kept at 52°C and maintained under diffused light. Cellulose was used as a positive control, polyethylene was used as a negative control, and anaerobic digested sewage sludge was used as a blank. The cellulose, polyethylene (PE), blank, and the test sample were tested in triplicate. The cellulose used in this testing is a high purity cellulose powder of 20 micron size. It was purchased from Sigma Aldrich and has a catalog number S3504. The polyethylene used in this testing is a natural grade from Ineos Inc. with a product # K44-15-122.



A Micro-Oxymax respirometer from Columbus Instruments was used to monitor levels of carbon dioxide and methane volumes by the trapping device of each compost vessel. This information was used to calculate the amount of carbon dioxide and methane produced during the testing period. Based on this data, the cumulative amount of carbon dioxide and methane evolved from each vessel was calculated. This information was compared to the amount of CO₂ and CH₄ evolved from blank specimens to determine percent degradation. For detailed calculations, refer to ASTM D5511 Section 12.

Test Results:

The following graph provides a comparison of degradation of the plastic bottle samples with the positive control - cellulose and the negative control - polyethylene.

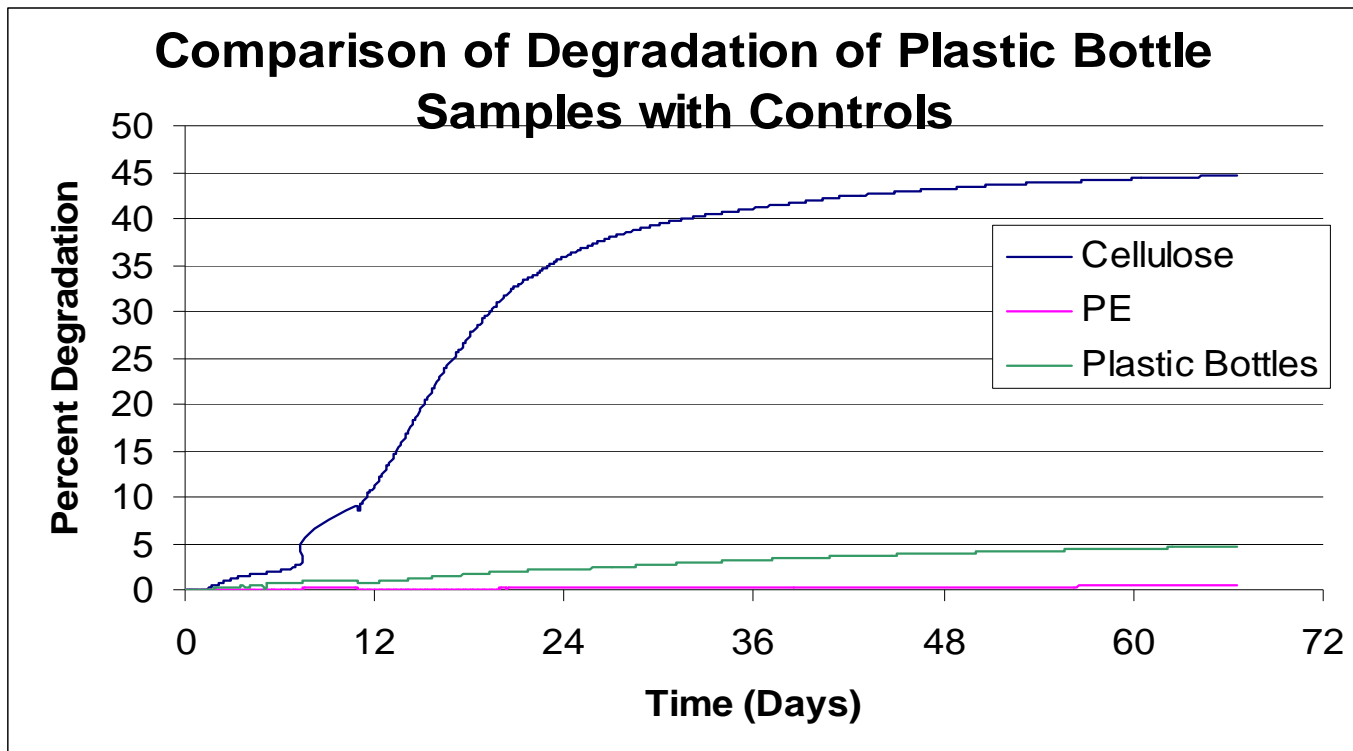


Figure 4: Progress of Degradation of Cellulose and PE Measured Under Similar Conditions as Plastic Bottles as a Function of Time; Reference ASTM D5511.



The following table shows the percent degradation of cellulose at 15 days of testing. The average degradation of cellulose at 15 days was 22%.

Table 3: Degradation of Cellulose at 15 Days of Testing

Sample	Percent Degradation of Cellulose
Average	22.34

Discussion: D5511 requires that the percent of cellulose be greater or equal to 70% after 15 days. However the percent degradation was only 22% after 15 days. This is due to the fact that the laboratory had to use a different type of inoculum as stated in D5511 section 9 since there are no municipalities in the US operating an anaerobic digester functioning with a pretreated household waste as a sole substrate. However, the inoculum used still met all the properties as listed in section 9 and degradation still occurred. Due to the reasons listed above, it is the opinion of the laboratory that the data can still be used to assess the relative degradation of the bottle material. After 60 days of testing, the positive control reached 44.3 % degradation while the bottle material only reached 4.5%, which is 10% relative to the positive control. In addition, samples of the exposed bottles after 60 days of incubation were compared to unexposed samples and showed no signs of visual degradation – figure 5 and 6. Experience from other test performed under aerobic and anaerobic conditions has shown that visual change occurs if sufficient level of biodegradation is obtained.

After 60 days, a plateau was observed for the overall level of biodegradation and therefore the test was stopped. The following table shows the percent degradation of cellulose, polyethylene, and the plastic bottle samples over 60 days of testing. The percent degradation was calculated by the measured cumulative carbon dioxide and methane production from each sample after subtracting carbon dioxide evolution and methane evolution from the blank samples at the end of 60 days of testing. Calculations were made based on total organic carbon of 66.03% for each vessel of plastic bottles, 44.4% for cellulose and 85.7% for polyethylene.



Table 3: Carbon Dioxide and Methane Evolution at 60 days

	Plastic Bottles Total CO ₂ grams	Positive control (cellulose) Total CO ₂ grams	Negative control (PE) Total CO ₂ grams	Plastic Bottles Total CH ₄ grams	Positive control (cellulose) Total CH ₄ grams	Negative control (PE) Total CH ₄ grams	% Degraded Plastic Bottles Samples	% Degraded of Positive Control Cellulose	% Degraded of Negative Control PE
Sample 1	3152.1	13582.6	3102.6	1622.9	3505.7	1113.6	5.57	38.86	-1.56
Sample 2	5134.1	3540.9	3702.5	2048.7	1370.9	1828.3	3.36	N/A	1.70
Sample 3	4310.5	14359.1	4049.2	1668.8	4837.7	1509.2	N/A	49.76	1.03
Average							4.47	44.31	0.39

Conclusion:

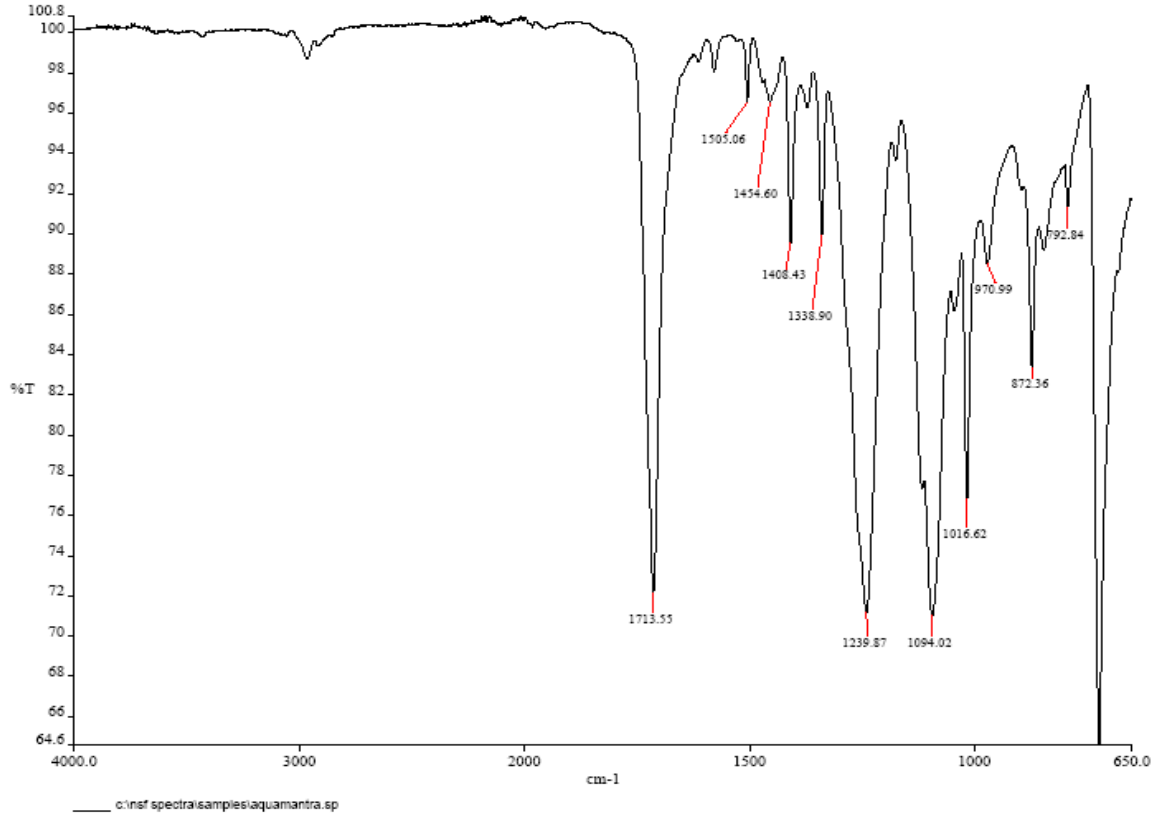
Testing was ended at the 60 day mark as the overall rate of biodegradation had stopped, as indicated by the plateau of the test samples curve on page 4. Based on the results obtained after 60 days of incubation, the level of degradation for the positive control (cellulose) was 44.31%, the negative control (polyethylene) level was 0.39%, and the plastic bottle samples level was 4.47%, 10% relative to the positive control.

FTIR Analysis:

FTIR analysis was performed on a plastic bottle sample. When compared with FTIR spectra stored in the library of the equipment, it was found to have a match of 82.8 % with PET. Attached is copy of FTIR spectrum in Annex A.



Annex A: FTIR Spectrum of Plastic Bottles Sample



Annex B: Comparison of Plastic Bottle Samples Before and After Testing

The following figures show photographs of the samples before and after 60 days of testing.

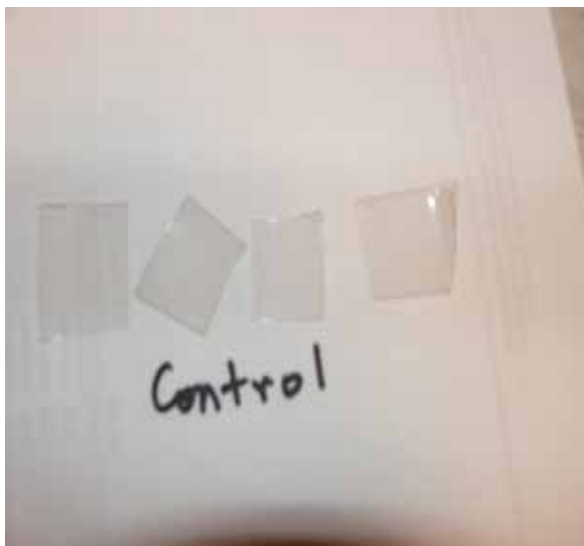


Figure 5: The Plastic Bottle Sample Prior to Testing



Figure 6: The Plastic Bottle Sample after 60 Days of Testing