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# TEST REPORT

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BiologiQ, Inc.
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| **Result:** | **Pass** | Report Date: 16-OCT-2012 |
|---|---|---|

| | |
|---|---|
| Customer Name: | BiologiQ, Inc. |
| Tested To: | ASTM Standard D6400-12 |
| Description: | Thermoplastic starch (TPS) resin |
| Test Type: | Chemical analysis, disintegration, biodegradation and phytotoxicity |
| Job Number: | J-00110670 |
| Project Number: | 9128405 |
| Project Manager: | Cheryl Navarro |

Executive Summary: The TPS resin submitted for testing fulfilled the requirements listed in ASTM D6400-12 "*Standard Specification for Labeling of Plastics Designed to be Aerobically Composted in Municipal or Industrial Facilities*" and can therefore be considered industrially compostable.

**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.

Digitally signed by Ata Ciechanowski
DN: cn=Ata Ciechanowski, o=NSF International, ou=Engineering Lab, email=ciechanowski@nsf.org, c=US
Date: 2012.10.18 11:13:09 -04'00'

**Report Authorization:**_____

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

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## Objective

The objective of this testing was to evaluate TSP resin to the requirements of ASTM D6400-12.

## Sample Description

Testing was performed on thermoplastic resin provided by BiologiQ, Inc. The samples submitted for testing included a bag of resin pellets for biodegradation testing and a milled plaque of resin for disintegration testing. Table 1 outlines the sample characteristics and Figure 1 shows the samples as received.


**Figure 1:** J-00110670 samples as received (left) and as prepared for disintegration testing (right)

**Table 1:** J-00110670 characteristics

| | |
|---|---|
| Total solids | 97.2% |
| Total volatile solids | 100% |
| Total organic carbon | 27.15% |
| Ash | 0% |
| Maximum thickness | 170 mils |
| Density | ~1.4 g/mL |

## Test Protocol

Samples were tested to all requirements of ASTM D6400-12. The biodegradation test was run according to ASTM D5338-11 "*Standard Test Method for Determining Aerobic Biodegradation of Plastic Materials Under Controlled Composting Conditions, Incorporating Thermophilic Temperatures*". The disintegration rate of the material was also

tested to ASTM D5338 without the carbon dioxide trapping. Only duplicates were run instead of triplicates for the disintegration test. This was because there was not enough milled sample to support a third flask of equal measure.

The chemical analyses were all performed on site with the exception of the measurement of total organic carbon. The phytotoxicity test was performed according to OECD 208 with the modifications found in Annex E of EN 13432. Data was collected to verify the identity of the sample which included a Fourier Transform Infrared Spectroscopy scan, and density analysis. Regulated metals content and percent ash were determined to ensure compost quality was preserved post disintegration.

## Test Results

*Regulated Metals*

The sample was subjected to microwave digestion and metal scan per the EPA 200.8 ICP-MS test method. The sample was evaluated for concentration of regulated heavy metals as prescribed in Table 3 of 40 CFR Part 503.13 (USA) and Table II of the Trade Memorandum T-4-93(Canada).

The heavy metal analysis indicates that the sample concentrations of regulated metals were below the limits set by U.S. and Canadian guidelines. Table 2 provides a comparison of the regulated metal limits for the United States and Canada with the sample results.

**Table 2:** Regulated metals content for J-00110670

| Metal | 50% of the U.S. limit (ppm) | 50% of the Canadian limit (ppm) | Sample results (ppm) |
|---|---|---|---|
| Arsenic | 20.5 | 37.5 | 0.015 |
| Cadmium | 19.5 | 10 | 0.012 |
| Copper | 750 | NA | 0.053 |
| Lead | 150 | 2.5 | 0.028 |
| Mercury | 8.5 | 250 | 0.008 |
| Nickel | 210 | 90 | 0.127 |
| Selenium | 50 | 7 | 0 |
| Zinc | 1400 | 925 | 1.986 |
| Cobalt | NA | 75 | 0.002 |
| Molybdenum | NA | 10 | 0.017 |

*FTIR analysis*

One resin pellet was scanned on a Fourier Transform Infrared Spectroscopy instrument between 4000 $cm^{-1}$ to 650 $cm^{-1}$ showing the major peaks of interest in regards to identifying the composition of the final product.

*Inherent Biodegradability and Disintegration*

Inherent biodegradation and disintegration over 12 weeks was determined per section 6.3 and 6.2 of ASTM D6400-12 using test method ASTM D5338-11. The compost used in testing was obtained from Tuthill Farms and sourced from a mixture of agriculture, yard and municipal solid wastes. Prior to testing, the compost was passed through a 12 mm sieve to remove any large particles. A sample of compost was analyzed for pH, percent dry and volatile solids, C:N ratio and $CO_2$ evolution during the first 10 days of testing. Table 6 lists the results of these analyses.

**Table 6:** Compost properties

| Property | Standard Recommendations | Actual |
|---|---|---|
| Total solids (%) | 50-55 | 52 |
| Total volatile solids (%) | < 70 | 31.4 |
| C:N ratio | 10-40:1 | 15.4:1 |
| pH | 7.0-8.2 | 7.5 |
| Compost activity (mg CO2/g volatile solids) | 50-150 | 100.6 |

Each vessel was charged with approximately 600 grams of compost and mixed with an additional 100 grams of sample (dry weight). Table 7 lists the experimental charge of each sample test vessel.

**Table 7:** Experimental charge for biodegradation test

| Test Vessels | | Compost (g) | Sample Material (g) |
|---|---|---|---|
| **J-00110670** | 1 | 1154.6 | 103.2 |
| | 2 | 1154.0 | 102.9 |
| | 3 | 1154.3 | 102.9 |

Cellulose was used as a positive control to demonstrate the ability of the compost media to consume a known biodegradable material. The test cellulose was a high purity chromatography grade powder of size 20 micron or less. The cellulose was purchased from Sigma Aldrich from lot 108K0113. The blank controls, positive controls and samples were all tested in triplicate. Polyethylene was not used as a negative control as it was determined that this is an unnecessary step in determining the biodegradation of sample materials.

Composting vessels were kept at 58°C and maintained under diffuse light. Samples were exposed to a continuous moist air flow. Test vessels were temporarily removed from the incubator on a weekly basis in order to shake the contents which promoted aeration and reduced channeling throughout the vessel. During this time, visual observations of the vessels' contents were recorded to observe the progress of the samples under test. After 12 weeks of testing, all of the composting material was removed from the vessels to determine the level of disintegration.

A Micro-Oxymax respirometer from Columbus Instruments was used to monitor levels of oxygen and carbon dioxide in the head space of each composting vessel. A 10% carbon dioxide sensor was used for the experiment. A measurement of evolved carbon dioxide and consumed oxygen occurred approximately every two and half hours. The data was used to determine the amount of carbon dioxide evolved over the entire testing period. This value was then compared to the amount of carbon dioxide evolved from the blank controls to determine the percent of sample carbon conversion to carbon

dioxide. For detailed calculations, refer to ASTM D5338 section 12. Respirometry continued until the samples reached 90% carbon conversion when compared to the positive reference or at least until the 45 day mark when it can be determined that the testing was valid.

A higher than normal amount of biodegradation was observed in both the sample and positive control vessels. The total biodegradation observed from the cellulose and test samples exceeded that which is theoretically possible. This can be attributed to the priming effect which is common in compostablity tests that use compost as the medium instead of vermiculite. The priming effect occurs because the cellulose and test sample vessels contain more organic matter than the blank vessels. This increased food source permits a greater microbial population to exist and therefore, more carbon mineralization activity occurs within these vessels.

Tables 8 and 9 below outline the results of the disintegration test and the calculations for the biodegradation test, respectively. Figures 2-7 show the progress of the sample throughout the disintegration and biodegradation tests, including graphs which compare the sample to the positive and blank controls.

**Table 8:** Disintegration test results after 12 weeks

| Test Vessels | | Initial Sample Loading (g) | Final Weight Retrieved on 2 mm Sieve (g) | Percent of Original Weight | Average Disintegration | Standard Requirement |
|---|---|---|---|---|---|---|
| **J-00110670** | 1 | 102.7 | 0 | 0 | 100 % | > 90 % |
| | 2 | 102.7 | 0 | 0 | | |

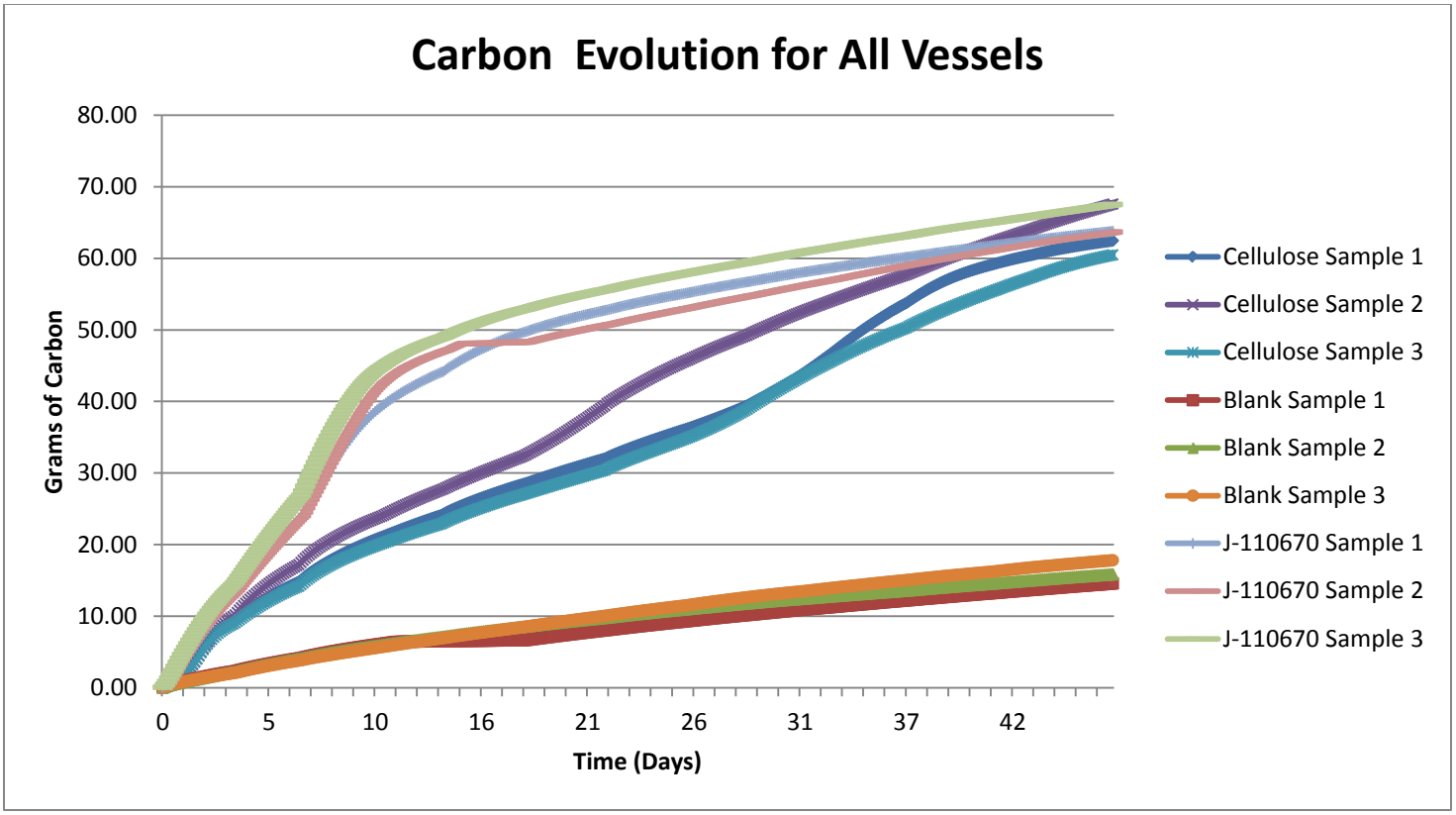



**Figure 6:** Evolved $CO_2$ for the blank and positive controls as well as J-00110670

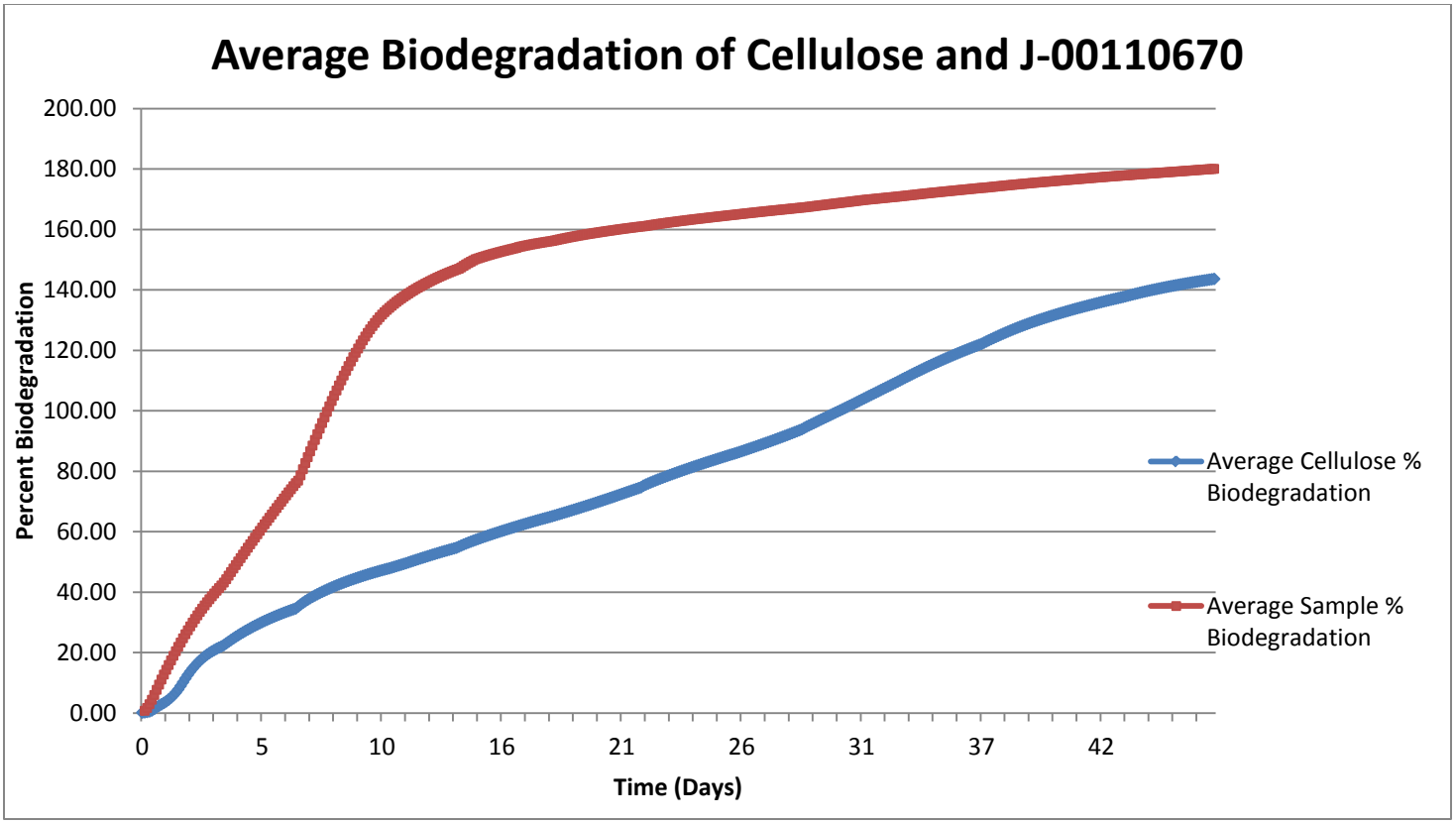



**Figure 7:** Percent biodegradation of the positive control and J-00110670

**Table 9:** Statistical measurements of sample data

| Statistical Measure | J-00110670 |
|---|---|
| Average carbon evolution (g) | 48.89 |
| Standard deviation | 2.19 |
| Biodegradation (%) | 180 |
| Standard error | 4.7 |
| 95 % confidence limit | 180 +/- 18.67 |
| % Biodegradation for positive control | 144 |
| % Biodegradation of the sample relative to the positive control | 125 |

*Phytotoxicity (2 Species)*

Plant growth mediums were prepared with the test compost and Growdan spun rock wool in ratios of 1:3 and 1:1 by volume. Similarly, control groups were prepared by mixing the same concentrations and using the blank test compost. The growing medium was then distributed into a 72 cell growing flat with three seeds planted per cell for a total of 108 seeds per concentration and species. Two seedling species were planted: rodeo oats and pacific gold mustard. The two species shared a single flat equally, and triplicates were not performed due to a lack of test compost. A thin layer of topsoil was sprinkled over the top of each flat to cover the seedlings.

The flats were kept in a temperature controlled greenhouse. The temperature range remained within 65-95 °F over the course of the test. The test beds were lightly watered twice daily with reverse osmosis water and rotated twice weekly to ensure equal sun exposure. The test was terminated after three weeks. Final seedling emergence was tallied, mortality was documented and biomass was measured on a dry weight basis. The results of this testing can be found in Table 8. Figure 4 shows an example from the phytotoxicity test.

**Table 8:** Phytotoxicity test results*

| Sample | Concentration | Seed Species | % Germination | % Biomass (dry wt) |
|---|---|---|---|---|
| J-00110670 | 1:3 | mustard | 106.9 | 117.4 |
| J-00110670 | 1:1 | mustard | 100.0 | 97.1 |
| J-00110670 | 1:3 | oats | 90.3 | 101.8 |
| J-00110670 | 1:1 | oats | 108.0 | 139.3 |
| **AVERAGE** | | | **101.3** | **113.9** |
| **Requirement per ASTM D6400-12** | | | > 90 | > 90 |

*Values are in comparison to the control group


**Figure 4:** 50% concentration blank tray (left) and sample tray (right)

## Test Validity

ASTM D5338-11 sections 11.4.2 and 13.2 require a review of the final test results in order to justify the validity of the test data. This includes the pH of the vessels at the end of the test, the performance of the positive control at 45 days and the deviation of biodegradation between positive controls. Furthermore, OECD 208 requires at least 70% emergence of the control seedlings for the test to be considered valid. Table 10 outlines the validation data for the biodegradation and phytotoxicity tests.

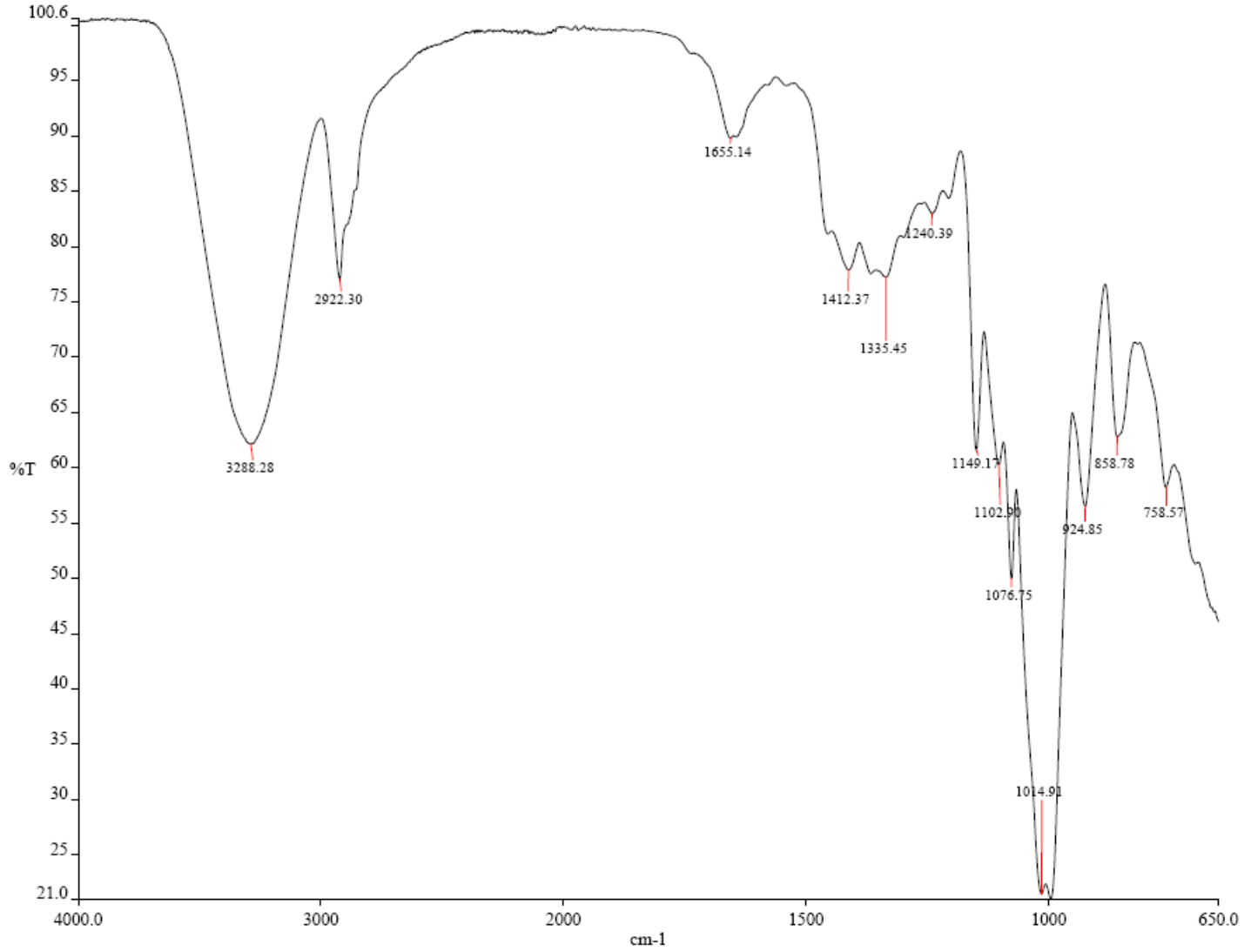
**Table 10:** Validation data

| Property | Requirement | Actual |
|---|---|---|
| pH of vessels | > 7 | 8.0 |
| Positive control carbon evolution at 45 days | ≥ 70 % | 140% |
| Deviation of positive control | < 20% | 3.7% |
| Seedling emergence of controls | >70% | 95.1% |

## Conclusion

TPS samples provided by BiologiQ, Inc. were tested to all requirements of ASTM D6400-12 including heavy metals analysis, disintegration while composting over a 12 week period, inherent biodegradability and phytotoxicity. Complete disintegration and biodegradation of the TPS samples occurred extremely quickly, with no phytotoxic effects observed from the subsequent compost. Based upon the results of all testing outlined in this report, the TPS resin meets all requirements of ASTM D6400-12 and can therefore be considered industrially compostable when used at a maximum thickness of 170 mils (0.170”).

**Annex A**




FTIR spectrum of J-00110670

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