Summary of ASTM D6400-99 Test Method and Specifications

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Correlation of Test Methods to Real World Composting Results

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Background

The development of the test methods and specifications (criteria for passing) found in ASTM D6400-99 "Standard Specification for Compostable Plastics" is the result of almost 8 years of scientifically based research and discussion under the direction of ASTM's Institute for Standards Research (ISR). The work was conducted by the IRS from 1991 through 1996 with the final standard resulting from this research published in May, 1999 and is summarized as the "ASTM-ISR Degradable Polymeric Materials Program". The complete results of testing activities are available for purchase from ASTM (phone 610-832-9555: Product Code Number 33-000019-19).

ASTM (American Society for Testing and Materials) is one of the largest organizations for establishing standards in the world. Its goal is to develop "voluntary full consensus standards for materials, products, systems, and services by providing a forum for producers, users, ultimate consumer and those having a general interest." The ISR is the research arm of ASTM and conducts projects at the request of specific ASTM committees and its members.

The impetus for the Degradable Polymeric Program stemmed largely from the marketplace confusion, surrounding claims like "biodegradable" and "compostable", which existed since the introduction of starch impregnated polyethylene bags in the mid-1980s.

> "The U.S biodegradable's industry fumbled at the beginning by introducing starch filled (6-15%) polyolefins as true biodegradable materials. These at best were only biodisintegrable and not completely biodegradable. Data showed that only the surface starch biodegraded, leaving behind a recalcitrant polyethylene material. Starch entrapped within the PE matrix did not appear to be degraded."[1]

The ISR project was a public and private cooperative effort to establish the test methods and practical scientific criteria for determining and substantiating these controversial claims.

Program Participants & Purpose

The participants for this program, included resin suppliers, consumer product marketers, governmental agencies and educational institutions. Additionally, resources from The Composting Council were made available for the in-situ composting activities. Program members included

Cargill	Dow	DuPont
Eastman Chemical	Ecochem	Exxon
Kimberly Clark	Mobil	Novamont
Novon	Procter & Gamble	US Army
Zeneca	Nat. Corn Growers	INDA (Non-Wovens Assoc.)

Also contributions were received from Michigan Biotech Institute (MBI), Organic Waste Systems (OWS), E&A Environmental and Phil Leege.

The original Scope and Mission of the program were as follows:

> **Mission:** "To provide the basis for the scientific substantiation of disposability statements for degradable polymeric materials."
>
> **Scope:** "To determine the behavior of degradable polymeric materials in real disposal systems, and how those results correlate with laboratory results, in order to assure that such materials are safe for disposal and effectively degraded."

While ISR group was tasked with reviewing a variety of disposal avenues, the first and most relevant to be studied was composting. During the course of its investigation, the group reviewed available data and information from both North America as well as Europe. Sources for their work included

- 2 literature databases on composting;
- 16 laboratory programs for testing biodegradability and compostability, covering a variety of natural and synthetic materials;
- 3 pilot scale trials to develop representative test methods
- 3 trials in full sized operating facilities, to develop testing procedures and to confirm laboratory and pilot scale results.
- Developing a correlation between laboratory, pilot and real-world, full-scale composting conditions.

> "In these selected test procedures (ASTM D5338-P&G pilot-Recomp II full scale), for all the materials tested without a single exception the degradation results obtained from the higher-level test equaled or exceeded those obtained in a lower-level test…The results mean that the laboratory –scale ASTM D5338 is more conservative than the pilot-scale P&G test which in turn is more conservative than the full-scale Recomp II test."

This document draws upon the results and conclusions from this ISR effort, in order to address the questions raised at the September 12th Board of Directors Meeting of the USCC. The fundamental issues raised were "How representative of real world conditions are the test conditions and how predictive are the tests?"

Identification of the Key Processes of Composting

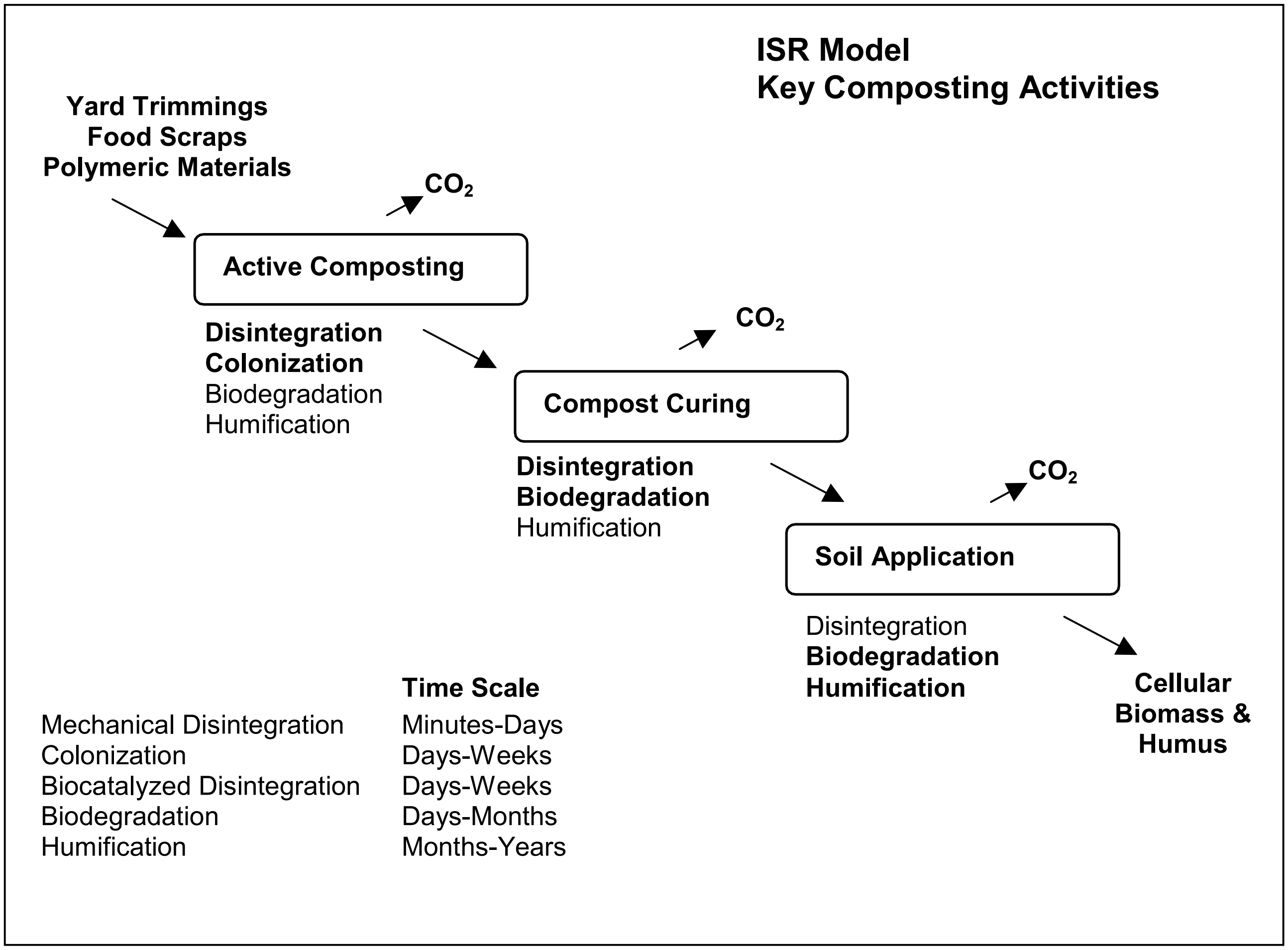
The initial stages of the ISR program studied and modeled the composting process, so as to better understand the performance of natural materials, commonly thought of as "compostable". This work identified the key variables that needed to be measured and established benchmarks for polymeric products based on commonly composted materials.

During composting and soil application, the transformation of organic materials (yard trimmings and food scraps as well as polymeric materials) into a useful amendment can be characterized in the following manner:

Active Composting	The starting point of the process, where under appropriate conditions intense microbial activity begins. This phase typically takes days or weeks. The critical characteristics for materials in this phase are their ability to safely support microbial growth and to start disintegration.
Compost Curing	This is the continuation of the Active phase. However temperatures are lower and degradation frequently occurs at a slower rate. This phase usually encompasses months to a year or more. Compost is typically screened and sold at the end of this phase to minimize the number of "inerts" and attain the desired particle size. During curing, materials need to continue to support microbial activity, humification and to disintegrate adequately; thereby not being distinguishable from other components in the compost.
Soil Application	After land application, compost components are expected to continue their degradation process and enhance soil productivity. Materials need to be able to be safely converted into carbon dioxide by the microorganisms in the environment.

Note: The above definitions were created by the ASTM Environmentally Degradable Plastics Sub-Committee (D20.96) and are distinct from those used in the USCC's Test Methods Guide (TMCC).

The diagram on the next page outlines the critical steps in the process, including time frames and activities.



To successfully pass the steps outlined in the above model a polymeric material (and for that matter "compostable" natural materials) must demonstrate 3 characteristics

Biodegradability-	Ability to be utilized as a carbon source by microorganisms and converted safely into carbon dioxide, biomass and water
Disintegration-	Ability to fragment, so as not to be distinguishable after screening, while biodegradation continues
Safety (No Ecotoxicity)-	Not detracting from the ability of the finished compost to support plant growth

Definition of Biodegradability

One of the cornerstones of this work was the recognition that conversion of the polymeric carbon in the test material to carbon dioxide was the end point of biodegradation and demonstrated assimilation and utilization of the polymeric materials by microorganisms. Earlier work and claims relied on a "loss of properties" as a sign of biodegradation. However this research concluded

> "The only direct measurements of biodegradability are measurements of mineralization, the conversion of carbon from the test substance into

gaseous carbon: CO_2 in aerobic processes or CO_2 plus CH_4 in anaerobic processes."[2]

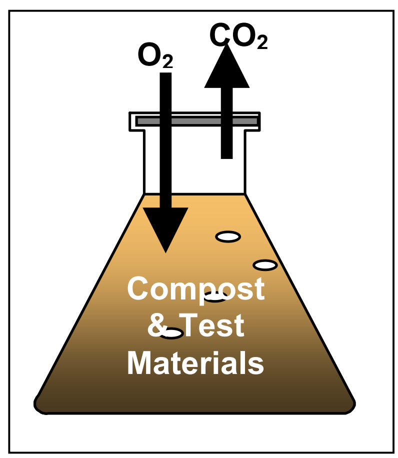
While changes in physical properties are useful indicators, they may be generated by means other than microbial attack (such as high temperatures or moisture). Only microbial assimilation of the polymeric carbon as demonstrated by the carbon dioxide end point measurement would assure of no adverse impact on the ecosystem or the creation of recalcitrant, persistent or toxic materials during the process.

Predictive Laboratory Tests and Conditions

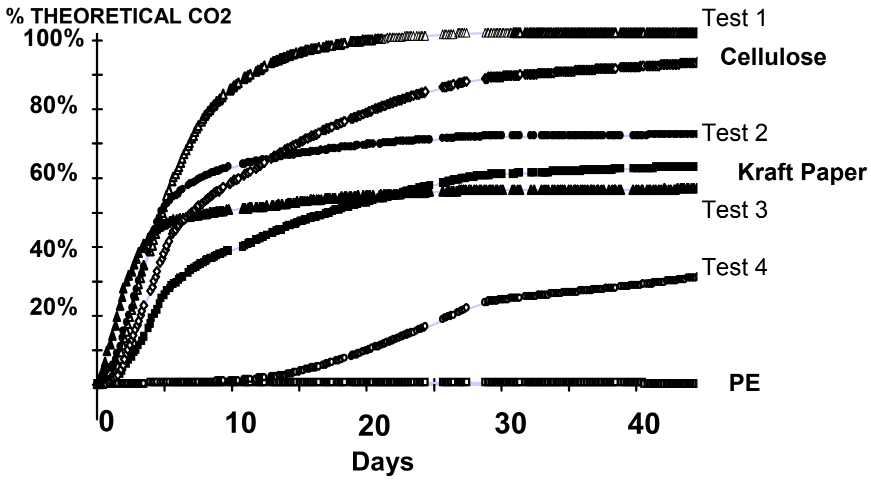
With an understanding of the composting process and its key requirements, the ISR program evaluated the results of natural as well as man-made materials in a variety of laboratory tests, pilot scale conditions as well as real world situations. The goal was to attempt to identify test methods and specifications that would be predictive of compostability. The program focused on measuring the 3 key characteristics identified in the composting model: Biodegradability; Satisfactory Disintegration and Lack of Ecotoxicity. It should be noted that the test methods refered to in this document are different than those used in the USCC's Test Methods for Examining Compost (TMECC). The ASTM tests and TMECC tests are not interchangable and were developed to meet different objectives.

Measurement of Biodegradability
The ISR program concluded that measurement of both the rate and extent of biodegradation occurring during composting *can be* predicted using ASTM D5338-98: *Test Methods for Determining Aerobic Biodegradation of Plastic Materials Under Controlled Composting Conditions*.

The test is conducted in the following manner- A known amount of active compost and the test material is placed in sealed flasks, as is the case with blank and control materials. These are maintained at thermophilic temperature (58^0C) along with adequate oxygen and moisture to sustain microbial activity. By measuring the carbon dioxide generated from the test and control flasks, the amount of carbon converted to carbon dioxide gas is calculated. Then the percentage of material, which is mineralized, can by determined. To the right is a schematic diagram of the test apparatus. Attached is a complete description of the test method.



The test results of D5338 are typically presented in the following fashion-as a graph of the percentage of total carbon dioxide evolved over time.



Note: Test materials are for illustrative purposes. Hence are not identified

It is important to note the curves for cellulose, kraft paper (40#) and polyethylene (PE). The first and last items typically represent positive and negative controls for this test. Kraft paper (40#) typically achieves a 60% conversion ratio, which now serves as the threshold (passing) specification for "homopolymers and random copolymers".

Measurement of Disintegration

Disintegration is measured by placing finished articles in the same conditions as found in the biodegradation tests. Once the test period ends, the residues are sieved. If less than 10% of the test material remains on a 2mm screen, then the product passes. Disintegration tests ensure that the finished polymeric article or product (in the shape and thickness as it would be used) will disintegrate rapidly enough during the composting process so that any non-degraded pieces would be indistinguishable in the compost.

In this test, as was true with measurements of biodegradability, kraft paper was a reference material. Therefore, if in real world situations, the disintegration rate (as well as biodegradation rate) will be impacted by the "intensity of the composting operation". Products, which pass the laboratory disintegration test, will perform similarly to kraft paper or wood chips, depending on form. It is reasonable to expect that "compostable plastics" will remain intact longer in piles or composting operations (as will kraft paper), where pile temperatures and microbial activity are low and there is little if any mechanical agitation. Conversely, degradation and disintegration will occur more rapidly in facilities where conditions and feedstocks are maximized.

[Note: the Mineralization Test establishes that the disintegrated material will be truly assimilated by the microorganisms.]

Determination of Ecotoxicity

Seedling germination and plant growth tests (including cress) are used to determine the potential phyototoxic impacts of test materials. Typically, 3 concentrations of compost and soil mixtures are used in these trials, ranging from 1% to 10%. If the plant growth and germination results of the test materials are significantly lower than those of the control compost (at 95% confidence level) then the material will not pass. To the right is a typical set of results from the ISR study.

	DRY WEIGHT YIELD-Radish		
	1.1% AR	3.3% AR	9.9% AR
Reference Materials			
Soil Control	100%	100%	100%
Control Compost	119%	108%	122%
Cellulose Compost	117%	122%	118%
Test Materials			
PE Compost	109%	116%	109%
Test 1	107%	93%	112%
Kraft Paper Compost	98%	116%	111%
Test 2	106%	119%	122%
Test 3	111%	101%	108%
Test 4	112%	107%	104%

Correlation of Laboratory to Operating Facility Results

While the ISR study tested 31 materials during the course of its work, 6 materials were tested in all phases of the program: laboratory tests, pilot scale (P&G) and operational facilities (Recomp II). The following conclusion were drawn:

> "In these selected test procedures (ASTM D5338-P&G pilot-Recomp II full scale), for all the materials tested without a single exception the degradation results obtained from the higher-level test equaled or exceeded those obtained in a lower-level test. As discussed at the beginning of the chapter, weight loss is not a measurement of biodegradation. However, the results mean that the laboratory –scale ASTM D5338 is more conservative than the pilot-scale P&G test which in turn is more conservative than the full-scale Recomp II test."[3]

Below is a summary of the results of the 6 materials, under the lab, pilot and full-scale tests.

	Lab-scale ASTM D 5338		**Pilot Scale P&G**		**Full-scale Recomp II**	
	Biodegradation %	Disinte-gration	Biodegradation %	Disinte-Gration	Biodegradation %	Disinte-gration
Kraft Paper	52%	Complete	66%	Complete	100%	Complete
PE	0%	None	0%	None	4%	None
Test 1	107%	Complete	99%	Complete	100%	Complete
Test 2	45%	None	100%	Complete	100%	Complete
Test 3	62%	None	72%	None	72%	None
Test 4	25%	N/A	46%	Moderate	88%	Extensive

The key implication here is that materials and products which pass the previously outlined laboratory tests will compost satisfactorily in actively managed compost facilities.

Anecdotal Observations

There is a growing body of first hand evidence demonstrating that biodegradable plastics, which have passed the previously described, tests (either ASTM's or comparable European ones) also compost satisfactorily.

In December 1997, *BioCycle* summarized the work of Rod Tyler, who composted bags under controlled conditions, from 6 organizations in 3 separate trials.

> "In summary, there were no visible residuals from four of the bags tested in our system: EcoPLA, Envar, Biocorp MaterBi reSource and MaterBi Linden Group bags. On the other hand, Plastigone, Bio-Solo and Degra-Novon bags tested were easily found and identified. These results were repeated in all three trials."[4]

It is important to note that EcoPLA and MaterBi bags have both passed standards comparable to ASTM D6400-99 in Europe.

References and Attachments

1. Narayan, R. Doi, Y. and Fukada, Impact of Governmental Policies, Regulations, and Standards Activities on an Emerging Biodegradable Plastics Industry. Biodegradable Plastics and Polymers. Elsevier, New York, 1994, page 261

2. De Wilde, B and Tillinger, R "1996 ISR Degradable Polymeric Materials Research Program, Final Program Report". American Society for Testing and Materials. page 23.

3. Ibid. page 95

4. Tyler, R. "Breaking Down Biodegradable Bags". BioCycle, December, 1997.

Appendix

- **ASTM Standards**
  -D6400-99
  -D6002-98
  -D5338-92

- **Composting Process Model & Key Process Variables supplied by P. Leege**