

Injection Stretch Blow Molded Bottles

Ingeo biopolymer 7032D is a bottle grade resin designed for injection stretch blow molded applications where heat setting is needed. Ingeo biopolymer 7032D can be run on conventional and heat set ISBM equipment typically used for PET. Ingeo biopolymer 7032D is run at lower processing temperatures than PET.

Applications

Injection stretch blow molded bottles (ISBM) for single- and two-stage operations. Ideal for applications where heat setting is required, such as:

- Fruit juices
- Sports drinks
- Oils, jams, non-beverage

Preform Design & Injection Molding

Preform design is critical in making a container with good clarity and physical properties. Designing a preform for use as an Ingeo bottle is, to an extent, specific to the blow mold equipment, bottle design, and mold tooling. As a starting point, designing a preform with an areal (axial x hoop) stretch ratio (SR) of 8-11, an axial SR of 2-3, and a hoop SR of 3-4, should allow for the blow molding of the desired container. The natural stretch ratio for Ingeo biopolymer 7032D is a bit lower versus the Ingeo biopolymer 7001D bottle grade. As a result, a slightly lower areal stretch may be needed to help prevent pearlescence as compared to what may typically be required in a design using 7001D. As an estimate, the 7032D grade areal stretch ratio window may need to be decrease by 15-20% vs. the 7001D stretch ratios in order to prevent overstretching of the bottles. The use of a reheat additive will also improve preform re-heating and equilibration time in the blow molding ovens. A preform designed with a thinner endcap might also be desired in order

Typical Material & Application Properties ⁽¹⁾			
Physical Properties		Ingeo 7032D	ASTM Method
Specific Gravity		1.24	D792
MFR, g/10 min (210°C, 2.16kg)		7	D1238
Glass Transition Temperature		55-60°C	D3418
Crystalline Melt Temperature		155-170°C	D3418
Transmission Rates	Oxygen	675 cc-mil/ m ² -24hr -atm	D1434
	Carbon Dioxide	2,850 cc-mil/ m ² -24hr-atm	Internal
	Water Vapor	375 g-mil/m ² -24hr	F1249
Clarity		Transparent	
General Blow Molding & Heat Setting Guidelines ⁽²⁾			
Preform Temperature		80-100°C (176-212°F) Maximize temperature while keep good material distribution.	
Stretch Rod Speed		0.8-1.2 m/second (Balayage stretch rod required)	
Blow Mold Temperature		100-120°C (212-248°F) Optimize for production rate and heat set quality.	

(1) Typical properties; not to be construed as specifications.

(2) Typical conditions; an experimental approach may be required.

to prevent excess material accumulating in the base of the blown container. Less aggressive transitions may also help with controlling material distribution and prevent material accumulating in the base, since Ingeo stretches easier than PET due to its lower extensional viscosity.

Injection Molding of Preforms

A general-purpose screw designed to minimize residence time and shear is recommended. A screw designed for PET is a relatively good choice, as they are a low compression screw that minimizes shear. Processing conditions should be controlled accordingly in order to minimize melt temperature and residence time. The shot size and hot runner capacity should be sized accordingly to the extrusion barrel size. This helps to maintain a short barrel residence time and eliminate the chance of material degradation. It is important to

avoid prolonged exposure to shear or heat, in order to prevent polymer degradation.

Typical Processing Parameters*		
Melt Temp.	390-430°F	200-220°C
Feed Throat	70°F	20°C
Feed Section	355°F	180°C
Compression Section	410°F	210°C
Metering Section	410-430°F	210-220°C
Nozzle	410-430°F	210-220°C
Mold	70-100°F	21-38°C
Back Pressure	100-200psi	
Mold Shrinkage	0.004 in/in.+/- .001	

*These are starting points and may need to be optimized. It is important to optimize back-pressure, process temperature, mold temperature, and injection speed such that the cycle time and the internal stresses in the finished part are kept to a minimum. This will help keep the shrinkage of the preform upon re-heating to the lowest level possible while keeping preform production as high as possible. Using a hot runner system in the injection-molding step is also recommended to help keep shear stress and preform shrinkage to a minimum.

Drying

In-line drying capabilities are essential to process Ingeo biopolymer 7032D, which is supplied with a moisture content of less than 0.04% (400 ppm). The recommended moisture content to prevent viscosity degradation and possible lactide reformation is less than 0.010% (100ppm). Typical drying conditions for Ingeo biopolymer 7032D are 4-6 hours at 149°F-185°F (65-85°C) with desiccated air that has a dew point of at least -40°F (-40°C), and an airflow rate of greater than 0.5 cfm/lb of pellets. The resin should not be exposed to atmospheric conditions after drying. Transfer lines and hoppers should be sealed or padded with inert gas. Keep resin package sealed until ready to use and promptly reseal packages of unused material. See our NatureWorks LLC 'Crystallizing and Drying' guide for more information. Before introducing Ingeo biopolymer into any vessel, the vessel should be clean and free of any cross-polymer contamination.

Process Details

Startup and Shutdown

Ingeo biopolymer 7032D is not compatible with a wide variety of resins, and special purging sequences should be followed:

1. At normal operating temperatures for the current polymer in the injection molder, purge with polypropylene (PP), preferably starting with a low MI (high viscosity) PP, switching to a high MI (low viscosity) PP.
2. Bring injection molder to steady state at normal operating temperatures for Ingeo while running PP.
3. Vacuum out dryer, hopper system, hoses, valves, and any other potential points where contamination could occur. Dry Ingeo resin according to recommending drying conditions & times. Ingeo needs to be dried before thermal processing/extrusion.

4. Introduce Ingeo resin into the injection molder at the recommended operating temperatures. (See operating temperatures listed.)
5. It will be obvious when pure Ingeo is being extruded, as it gives a clear, steady melt.
6. At shutdown, purge machine with a high-viscosity resin, such as PP.

Note: If transitioning from PET to Ingeo polymer, it is advised to first transition from these materials to a low melt index (higher viscosity), PP. Follow this transition to a higher MI (lower viscosity) PP. All PET polymers must be cleaned and expelled from vessels and the extrusion system before Ingeo is introduced. Next, lower temperatures to recommended starting conditions for Ingeo before continuing the startup process.

Blow Molding the Container

Processing Ingeo biopolymer 7032D for heat setting requires attention to preform temperature, mold temperature, and material distribution. Maximizing mold residence time improves Ingeo heat set bottle performance. However, one will need to optimize production rate while maintaining the dimensional stability requirements of the filling process. The use of a balayage stretch rod is required for cooling the bottle against the hot blow mold.

Re-heating of Preforms

The re-heating of the preforms is critical in getting a container with good clarity and material distribution. Typical preform temperatures for a two-stage blow molding process is in the range of 80-100°C. This temperature may be lower or higher depending on the preform design, bottle design, and re-heating equipment that are being used. It is recommended that a reheat additive be used in order to minimize

pearlescence and optimize material distribution.

Preforms made out of Ingeo biopolymer can be run on both single – and two-stage conventional blow molding equipment. Ingeo biopolymer has a lower extensional viscosity than PET, and so is easier to stretch. The use of pre-blow pressure and timing are important control parameters to help manage material distribution throughout the bottle.

Bottle Design

The performance of a heat set bottle utilizes the attributes of a good bottle design, which considers the following:

- Vacuum relief
- Reinforcement
- Base structure
- Finish stability
- Weight, wall thickness, and material distribution

All of these features help improve vacuum resistance, prevents bottle panels from collapsing and ovalization to occur, minimizes shrinkage, and improves top load strength.

The finish and base areas of the bottle may still be sensitive to heat since they haven't been stretched. For example, bottles used for hot fill applications usually have a high push-up on the base and ribbing for stabilization. Ingeo bottles have been found to work well with typical PET heat set bottle designs. The base of any given bottle has a thicker material distribution than the sidewall. This thick area in the bottle needs to be distributed well and cooled quickly. A base design that includes ribs and a high pushup for reinforcement is recommended. Sharp and aggressive edges and corners should be avoided in the bottle design, as Ingeo biopolymer stretches easier than PET and obtains good resolution of mold details.

Compostability

Composting is a method of waste disposal that allows organic materials to be recycled into a product that can be used as a valuable soil amendment. Ingeo biopolymer is made of polylactic acid, a repeating chain of lactic acid, which undergoes a 2-step degradation process. First, the moisture and heat in the compost pile attack the polymer chains and split them apart, creating smaller polymers, and finally, lactic acid. Microorganisms in compost and soil consume the smaller polymer fragments and lactic acid as nutrients. Since lactic acid is widely found in nature, a large number of organisms metabolize lactic acid. At a minimum, fungi and bacteria participate in this degradation process. The end result of the process is carbon dioxide, water and also humus, a soil nutrient. This degradation process is temperature and humidity dependent. Regulatory guidelines and standards for composting revolve around four basic criteria: Material Characteristics, Biodegradation, Disintegration, and Ecotoxicity. Description of the requirements of these testing can be found in the appropriate geographical area: DIN V 54900-1 (Germany), EN 13432 (EU), ASTM D 6400 (USA), GreenPla (Japan). This grade of Ingeo biopolymer meets the requirements of these four standards with limitation of maximum thickness 2.0mm.

Food Packaging Status

U.S. Status

On January 3, 2002 FCN 000178 submitted by NatureWorks LLC to FDA became effective. This effective notification is part of list currently maintained on FDA's website at

<http://www.cfsan.fda.gov/~dms/opa-fcn.html>

This grade of Ingeo biopolymer may therefore be used in food packaging

materials and, as such, is a permitted component of such materials pursuant to section 201(s) of the Federal, Drug, and Cosmetic Act, and Parts 182, 184, and 186 of the Food Additive Regulations. All additives and adjuncts contained in the referenced Ingeo™ biopolymer formulation meet the applicable sections of the Federal Food, Drug, and Cosmetic Act. The finished polymer is approved for all food types and B-H use conditions. We urge all of our customers to perform GMP (Good Manufacturing Procedures) when constructing a package so that it is suitable for the end use. Again, for any application, should you need further clarification, please do not hesitate to contact NatureWorks LLC.

European Status

This grade of Ingeo biopolymer complies with Commission Directive 2002/72/EC as amended by 2004/19/EC, 2005/79/EC, 2007/19/EC, 2008/39/EC, and 2009/975/EC. No SML's for the above referenced grade exist in Commission Directive 2002/72/EC or as amended by 2004/19/EC, 2005/79/EC, 2007/19/EC, 2008/39/EC, and 2009/975/EC. NatureWorks LLC would like to draw your attention to the fact that the EU-Directive 2002/72/EC, which applies to all EU-Member States, includes a limit of 10 mg/dm² of the overall migration from finished plastic articles into food. In accordance with EU-Directive 2002/72/EC the migration should be measured on finished articles placed into contact with the foodstuff or appropriate food simulants for a period and at a temperature which are chosen by reference to the contact conditions in actual use, according to the rules laid down in EU-Directives 93/8/EEC (amending 82/711/EEC) and 85/572/EEC

Please note that it is the responsibility of both the manufacturers of finished food contact articles as well as the industrial food packers to make sure

that these articles in their actual use are in compliance with the imposed specific and overall migration requirements.

This grade as supplied meets European Parliament and Council Directive 94/62/EC of 20 December 1994 on packaging and packaging waste heavy metal content as described in Article 11. It is recoverable in the form of material recycling, energy recovery, composting, and biodegradable per Annex II point 3, subject to the standards of the local community. Again, for any application, should you need further clarification, please do not hesitate to contact NatureWorks LLC.

Bulk Storage Recommendations

The resin silos recommended and used by NatureWorks LLC are designed to maintain dry air in the silo and to be isolated from the outside air. This design would be in contrast to an open, vented to atmosphere system that we understand to be a typical polystyrene resin silo. Key features that are added to a typical (example: polystyrene) resin silo to achieve this objective include a cyclone and rotary valve loading system and some pressure vessel relief valves. The dry air put to the system is sized to the resin flow rate out of the silo. Not too much dry air would be needed and there may be excess instrument air (-30°F dew point) available in the plant to meet the needs for dry air. Our estimate is 10 scfm for a 20,000 lb/hr rate resin usage. Typically, resin manufacturers specify aluminum or stainless steel silos for their own use and avoid epoxy-lined steel.

Safety and Handling Considerations

Material Safety Data (MSD) sheets for Ingeo biopolymers are available from NatureWorks LLC. MSD sheets are provided to help customers satisfy their own handling, safety, and disposal needs, and those that may be required by locally applicable health and safety regulations, such as OSHA (U.S.A.), MAK (Germany), or WHMIS (Canada). MSD sheets are updated regularly; therefore, please request and review the most current MSD sheets before handling or using any product.

The following comments apply only to Ingeo biopolymers; additives and processing aids used in fabrication and other materials used in finishing steps have their own safe-use profile and must be investigated separately.

Hazards and Handling Precautions

Ingeo biopolymers have a very low degree of toxicity and, under normal conditions of use, should pose no unusual problems from incidental ingestion, or eye and skin contact. However, caution is advised when handling, storing, using, or disposing of these resins, and good housekeeping and controlling of dusts are necessary for safe handling of product. Workers should be protected from the possibility of contact with molten resin during fabrication. Handling and fabrication of resins can result in the generation of vapors and dusts that may cause irritation to eyes and the upper respiratory tract. In dusty atmospheres, use an approved dust respirator. Pellets or beads may present a slipping hazard. Good general ventilation of the polymer processing area is recommended. At temperatures exceeding the polymer melt temperature (typically 170°C), polymer can release fumes, which may contain fragments of the polymer, creating a potential to irritate eyes and mucous membranes. Good general ventilation should be sufficient for most conditions.

Local exhaust ventilation is recommended for melt operations. Use safety glasses if there is a potential for exposure to particles which could cause mechanical injury to the eye. If vapor exposure causes eye discomfort, use a full-face respirator. No other precautions other than clean, body-covering clothing should be needed for handling Ingeo biopolymers. Use gloves with insulation for thermal protection when exposure to the melt is localized.

Combustibility

Ingeo biopolymers will burn. Clear to white smoke is produced when product burns. Toxic fumes are released under conditions of incomplete combustion. Do not permit dust to accumulate. Dust layers can be ignited by spontaneous combustion or other ignition sources. When suspended in air, dust can pose an explosion hazard. Firefighters should wear positive-pressure, self-contained breathing apparatuses and full protective equipment. Water or water fog is the preferred extinguishing medium. Foam, alcohol-resistant foam, carbon dioxide or dry chemicals may also be used. Soak thoroughly with water to cool and prevent re-ignition.

Disposal

DO NOT DUMP INTO ANY SEWERS, ON THE GROUND, OR INTO ANY BODY OF WATER. For unused or uncontaminated material, the preferred options include recycling into the process or sending to an industrial composting facility, if available; otherwise, send to an incinerator or other thermal destruction device. For used or contaminated material, the disposal options remain the same, although additional evaluation is required. (For example, in the U.S.A., see 40 CFR, Part 261, "Identification and Listing of Hazardous Waste.") All disposal methods must be in compliance with Federal, State/Provincial, and local laws and regulations.

Environmental Concerns

Generally speaking, lost pellets are not a problem in the environment except under unusual circumstances when they enter the marine environment. They are benign in terms of their physical environmental impact, but if ingested by waterfowl or aquatic life, they may mechanically cause adverse effects. Spills should be minimized, and they should be cleaned up when they happen. Plastics should not be discarded into the ocean or any other body of water.

Product Stewardship

NatureWorks LLC has a fundamental duty to all those that make and use our products, and for the environment in which we live. This duty is the basis for our Product Stewardship philosophy, by which we assess the health and environmental information on our products and their intended use, then take appropriate steps to protect the environment and the health of our employees and the public.

Customer Notice

NatureWorks LLC encourages its customers and potential users of its products to review their applications for such products from the standpoint of human health and environmental quality. To help ensure our products are not used in ways for which they were not intended or tested, our personnel will assist customers in dealing with ecological and product safety considerations. Your sales representative can arrange the proper contacts. NatureWorks LLC literature, including Material Safety Data sheets, should be consulted prior to the use of the company's products. These are available from your NatureWorks LLC representative.

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