

This bulletin is intended for use only as a tool to provide information and help address issues that may pertain to heat-setting injection stretch blow molded (ISBM) bottles made from Ingeo biopolymer. Since there are many factors to consider with preform and bottle design, development, and manufacturing, an experimental approach may be needed.

Preforms and bottles made from Ingeo biopolymer look and feel similar to ones made from PET, and they can be made on similar preform and bottle production equipment. However, there are differences between the two resins. These differences pertain mostly to the thermal behavior of the polymers. Ingeo biopolymer processes in the melt phase at much lower temperatures than bottle grade PET and the preforms are typically blown at lower temperatures. Because of these differences, an experimental approach may be required to achieve optimal results.

Products that are packaged under hot filling conditions usually require a bottle that can withstand the high temperatures to which the product is subjected during sterilization. A certain level of dimensional stability is required. Typical applications include fruit juices, sport drinks, ketchup, and jams. Ingeo biopolymer 7032D is a resin which is able to be injection-stretch blow molded for use in some hot fill applications where heat setting is required.

Ingeo biopolymer is a semi-crystalline polymer. Typical properties for Ingeo biopolymer 7032D resin can be found in the table below.

**Typical Ingeo Biopolymer 7032D Resin Properties**

Resin Property	Nominal Value
RV	3.9 – 4.1
Melt Temperature, °F (°C)	330 – 343 (165 – 173)
Glass Transition Temperature, °F (°C)	131 – 136 (55 – 58)
Crystallization Temperature, °F (°C)	212 – 248 (100 – 120)
Thermal Conductivity, BTU/ ft-hr-°F (cal / cm-sec-°C)	Amorphous 0.075 (3.1 x 10-4) Crystalline 0.11 (4.5 x 10-4)
Specific Heat, BTU/ lb °F (cal / g °C)	Below Tg 0.29 (0.29) Above Tg 0.51 (0.51)

At around its glass transition temperature of 55-60°C, Ingeo biopolymer begins to soften due to the mobility of the amorphous regions within the polymer chain. This causes problems for applications where dimensional stability is required at temperatures near or greater than this range. Hot filling or excessive shipping and storage conditions are such examples. Typical hot fill temperatures are in the range of 85-90°C, however this temperature will depend upon the application. When a conventional, non-heat set bottle is filled with product at these temperatures, the bottle becomes soft, is unable to maintain its shape, and shrinks. Bottles that are shipped or stored at excessive environmental conditions may also result in such deformation, however to a lesser extent.

To create a bottle that has a certain level of temperature resistance, a number of factors have to be taken into consideration: the shape and size of the container, its weight, filling temperature, production machine type and configuration, and product filling specifics. Heat setting, bottle design, and process conditions are the most important areas of focus in order to produce heat set bottles made out of Ingeo biopolymer.

## Heat Setting

Compared to conventional blow molding where a cold mold is used, heat setting utilizes a hot mold and a balayage stretch rod, which helps create a more temperature resistant bottle. This is due to the following polymer changes occurring:

- Reduction of amorphous phase in the polymer
- Development of crystallinity
- Relaxation of residual stresses

Using Ingeo biopolymer 7032D resin, typical heat setting parameters are as follows:

Parameter	Run Conditions	Specifics
Ingeo biopolymer 7032D	---	Bottle heat setting resin grade
Preform temperature (°C)	85-95	Try to maximize while maintaining material distribution throughout bottle
Mold temperature (°C)	100-120	Observe for mold sticking and bottle shrinkage
Mold residence time	Try to maximize	Optimize based on production rates
Balayage stretch rod	Required	Cooling and to prevent bottle shrinkage away from mold

## Bottle Design

In order to prevent dimensional stability problems during hot filling, a bottle's design, shape, and weight must also be taken into consideration to help improve mechanical stability and strength. 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 and thus, not subjected to stress-induced crystallization. These areas are mostly amorphous in nature, but because they are thicker, hold up to a certain level of thermal deformation. Stability of the amorphous neck and base areas will also depend upon preform design, bottle design, and material distribution. For example, bottles used for hot fill applications usually have a high push-up on the base and ribbing for stabilization. Hot fill bottles are typically higher in weight to help improve heat resistance.

The natural stretch ratio for Ingeo biopolymer 7032D is a bit lower as compared to Ingeo biopolymer 7000D bottle grade. This is because 7032D strain-hardens sooner than 7000D, and is able to develop more crystallinity. As a result, a slightly lower axial and hoop stretch may also be needed to help with preventing pearlescence as compared to what may typically be required in a conventional design when using 7000D. In terms of preform and bottle design, an experimental approach may be needed to find the right balance of processing and properties.

For further information on blow molding Ingeo bottles, preform design, injection molding, and drying, please refer to the 'Ingeo ISBM Bottle Guide.'

## Process Conditions

Processing Ingeo biopolymer 7032D for heat setting requires attention to the following unit operations and conditions:

- Correct preform conditioning and temperature
- After stretching, stabilization of the bottle against the hot mold using a balayage
- Optimum mold contact time to maximize stress relaxation
- Crystallinity development and level
- Adequate cooling to prevent mold sticking
- Thicker neck and base areas since they are amorphous

In terms of production rates, maximizing mold residence time improves heat set bottle performance. Acceptable heat set bottle performance has been observed when operating at typical heat setting rates on lab-scale equipment. Process optimization will most likely need to take place, especially on large-scale equipment, in order to find the most efficient production rates where a quality bottle can be made.

Since Ingeo biopolymer has a lower glass transition temperature as compared to PET, not all Ingeo bottles that are heat set may be suitable to package all products requiring hot fill, due to excessive temperature filling requirements. After hot filling, even though the bottles do not appear to shrink or deform, a loss of panel modulus may be observed until the product is cooled. However, this effect will depend upon the application, fill temperature, and bottle design. An experimental approach may be needed in order to ensure proper capping of the hot filled bottle and to ensure bottle performance.

## Summary

Ingeo biopolymer 7032D is a resin grade being evaluated for use in heat setting applications. Ingeo biopolymer has a lower melt profile so this needs to be kept in mind during preform conditioning, bottle blowing, and heat setting. Optimum preform temperature is in the range of 85-95°C. For heat setting, this temperature should be maximized so the material can be as close to the polymer's crystallization temperature as possible in order to minimize any internal stresses, which may lead to shrinkage. Heat setting occurs in the temperature range of 100-120°C, with the use of a balayage stretch rod to help promote cooling and bottle stability against the hot mold. Mold dwell time should be maximized, but this will be dependent upon the machine set-up and production rate targets. Proper bottle ejection out of the mold will depend upon correct mold temperature and enough cooling from the balayage. Using heat setting, Ingeo bottles can be hot filled at typical filling temperatures of 85°C without drastic bottle deformation. However, a loss in modulus or stiffness of the bottle may occur due to Ingeo biopolymer's lower glass transition temperature. Since there are many factors to consider with preform and bottle design, heat setting development, and manufacturing, and sterilization, an experimental approach may be needed. With regards to a heat set Ingeo bottle, no improvements in water or gas barrier properties are realized after bottle blowing and heat setting versus a conventional bottle blowing process which uses a cold mold.

## 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.

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