Injection Molding Guide for Ingeo™ 3801X

This information is intended for use only as a guide for the injection molding of Ingeo 3801X resin. It will consist of generalized concerns for safety, process conditions, typical properties, and tooling.

Because injection molding covers a wide arena of applications and polymers, an experimental approach using Ingeo resins at your facility will have to be completed to determine what tooling and mode of operation will work best.

Testing of the molded Ingeo products is recommended to insure that customer requirements are met.

1.0 Safety and Handling Precautions

All safety precautions normally followed in the handling and processing of melted thermoplastics should be followed for NatureWorks’ Ingeo resins.

As with most thermoplastics, melt processing and the variability of those conditions may result in minor decomposition. Ingeo 3801X is composed of polylactide (PLA) and a variety of processing additives. Lactide, a non-hazardous gaseous irritant, is a minor by-product of PLA melt processing. Appropriate air testing should be completed to ensure an acceptable Threshold Limit Value (TLV) of less than 5 mg/m³ is maintained. The use of process area point source remediation measures such as fume hoods or exhausts near melt processing equipment are typically recommended.

Molten PLA sticks more readily to cloth, metal, brass and wood compared to other molten thermoplastics. Be prepared for this when cleaning die faces, purging equipment, collecting molten patties, and emptying purge containers. Unlike polyolefins, molten PLA will not release as cleanly from a gloved hand, so use caution when touching any stream or patty of PLA.

PLA is considered non-hazardous according to DOT (US Department of Transportation) shipping regulations. When handling PLA resin at room temperature avoid direct skin and eye contact along with conditions that promote dust formation. For further information, consult the appropriate MSDS for the PLA grade being processed.

Ingeo 3801X contains 8-12% (w/w) mineral talc, added as a reinforcing agent. As supplied, the talc is fully encapsulated in polymer and poses little risk from dust. See MSDS for further details.

As with any melted thermoplastic waste, melted PLA waste should be allowed to cool before being placed into any waste container to minimize fire risks.

Ingeo 3801X is not certified for food contact.

Ingeo 3801X is not currently certified for compostability.

2.0 Pellet Storage and Blending Recommendation

Ingeo resins should be stored in an environment designed to minimize moisture uptake. Product should also be stored in a cool place at temperatures below 122°F (50°C). Product that is delivered in cartons or super sacks should be kept sealed until ready for loading into the blending and/or drying system. Bulk resin that is stored in closed silos and hoppers for extended periods (more than 6 hrs) should be kept purged with dry air to minimize moisture gain. In the case of outside storage, if the product is supplied in boxes or other non-bulk containers, the unopened container should be brought into the extrusion production area and allowed to equilibrate for a minimum of 24 hours before opening to prevent excessive condensation.
3.0 Resin Properties

Ingeo 3801X Injection Molding Grade is specifically made for Injection Molding Applications that require high heat resistance and high impact strength. Typical properties of Ingeo 3801X are shown in the table below.

<table>
<thead>
<tr>
<th>Resin Property</th>
<th>Nominal Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Specific Gravity, g/cm³</td>
<td>1.33</td>
</tr>
<tr>
<td>Relative Viscosity1 or RV, unitless</td>
<td>3.1</td>
</tr>
<tr>
<td>Melt Temperature, °F (°C)</td>
<td>325 – 343 (166 – 173)</td>
</tr>
<tr>
<td>Glass Transition Temperature (Tg), °F (°C)</td>
<td>113 (45)</td>
</tr>
<tr>
<td>Crystallization Temperature, °F (°C)</td>
<td>158 – 248 (70 – 120)</td>
</tr>
<tr>
<td>Tensile Modulus, psi (MPa)</td>
<td>432,000 (2,980)</td>
</tr>
<tr>
<td>Tensile Yield Strength, psi (MPa)</td>
<td>3,750 (25.9)</td>
</tr>
<tr>
<td>Tensile Elongation at Break, %</td>
<td>8.1</td>
</tr>
<tr>
<td>Notched Izod Impact, ft-lb/in (J/m)</td>
<td>2.7 (144)</td>
</tr>
<tr>
<td>Flexural Strength, psi (MPa)</td>
<td>6,400 (44)</td>
</tr>
<tr>
<td>Flexural Modulus, psi (MPa)</td>
<td>413,000 (2,850)</td>
</tr>
<tr>
<td>Heat Distortion Temperature at 66 psi, °F (°C)</td>
<td>149 (65)</td>
</tr>
<tr>
<td>Heat Distortion Temperature at 16.5 psi2, °F (°C)</td>
<td>284 (140)</td>
</tr>
<tr>
<td>Thermal Conductivity3, BTU/ ft-hr-°F (cal / cm-sec-°C)</td>
<td>Amorphous 0.075 (3.1 x 10^-4)</td>
</tr>
<tr>
<td></td>
<td>Crystalline 0.11 (4.5 x 10^-4)</td>
</tr>
<tr>
<td>Specific Heat3, BTU/ lb °F (cal / g °C)</td>
<td>Below Tg 0.29 (0.29)</td>
</tr>
<tr>
<td></td>
<td>Above Tg 0.51 (0.51)</td>
</tr>
</tbody>
</table>

(1) tested in chloroform at 30°C, 1% (w/v) concentration
(2) modified ASTM test E2092 to simulate low load
(3) estimated values

4.0 Drying

Ingeo resins can be successfully dried using most standard drying systems. Recommended conditions are provided for standard desiccant based column dryers. For other drying system designs, additional information can be provided upon request.

To prevent equipment corrosion, it is not recommended to dry or store hot Ingeo resin in carbon steel vessels (see Section 2.0).

In-line drying is essential for Ingeo resins

It is recommended that Ingeo should be dried to a maximum of 250 ppm of moisture as measured by Karl-Fischer coulometric titration method. A moisture level lower than 250 ppm will help keep the melt viscosity stable over time at elevated temperatures. Processes that have unusually long residence times or result in melt temperatures greater than 240°C should only extrude Ingeo resin at moisture levels less than 50 ppm for maximum retention of molecular weight and physical properties. Ingeo resin is supplied in foil lined containers dried to less than 400 ppm as measured by NatureWorks LLC’s internal method. The resin should not be exposed to atmospheric conditions after drying. Keep the package sealed until ready for use, and promptly dry and reseal any unused material. The drying table below can be used to estimate the drying time needed for Ingeo resins. Air or nitrogen based desiccant drying systems can be used at the recommended temperatures. It is not recommended to dry Ingeo 3801X above 85°C.
Typical Ingeo Resin Drying Conditions

<table>
<thead>
<tr>
<th>Drying Parameter</th>
<th>Amorphous</th>
<th>Crystalline</th>
</tr>
</thead>
<tbody>
<tr>
<td>Residence Time, hours</td>
<td>4</td>
<td>2</td>
</tr>
<tr>
<td>Air Temperature, °F (°C)</td>
<td>113 (45)</td>
<td>176 (80)</td>
</tr>
<tr>
<td>Air Dew Point, °F (°C)</td>
<td>-40 (-40)</td>
<td>-40 (-40)</td>
</tr>
<tr>
<td>Air Flow Rate, CFM/lb resin (m³/hr - kg resin)</td>
<td>&gt; 0.5 (1.85)</td>
<td>&gt; 0.5 (1.85)</td>
</tr>
</tbody>
</table>

Typical desiccant dryer regeneration temperatures exceed the melt point of Ingeo resins. To prevent issues with pellet bridging, sticking or melting, the drying system should be tested to ensure temperature control is adequate during operation as well as during regeneration cycles since valve leakage is common in many systems. Installation of a water cooled after-cooler may be necessary to prevent the drying air temperature from exceeding the recommended set point when drying amorphous materials. Ingeo resins are semi-crystalline products that can come in either amorphous or crystalline form and can be dried accordingly as per the table above.

5.0 Melt Processing

Prior to introducing Ingeo resin into any melt processing system, the system should be properly cleaned and purged to prevent any polymer cross contamination. Ensure that the feeding & blending equipment is thoroughly cleaned & free from dust / contamination and all metal magnates have been wiped clean. Ensure that granules are completely removed from all hang-up areas such as elbows, transitions and slide gates. The purging procedures below are recommended for optimal removal of other polymers.

5.1 PLA Purging Procedure

Following PET, PA, or HDPE in your system

1. Purge with low MFR (<1) PP at normal PET operating temperatures. Purge 10-30 minutes as necessary. Let system empty as much as possible. Clean out hopper as much as possible.
2. Introduce a high melt flow PP (5-8 MFR) and change to normal Ingeo resin operating temperatures.
3. Purge 10-30 minutes as necessary. Let system empty as much as possible.
4. Alternatively, you can purge with a high flow PETG (similar to Eastman copolymer 6763) and then reduce temperatures to Ingeo resin conditions.
5. Stop injection molder and completely clean all hoppers, elbow, slide gates, dryers, hopper loaders bins, hopper loader filters and material conveying lines of residual PET, PA or HDPE and PP. Load Ingeo resin into material handling system.
6. Transition to Ingeo resin and purge, and check that melt is clear of any contamination.
7. At the completion of the run, purge all Ingeo resin from the extrusion system, using a moderate to low melt index PP.

Notes

1. It is critical that all drying and conveying/receiving systems be free of all PET or PP and is vacuumed to ensure that there is no remaining polymer dust, before adding Ingeo resin. PET will not melt at Ingeo resin operating temperatures and will block screens if it is present in the system.
2. The brand of PP used for purging is unimportant, so long as it does not thermally cross-link.
5.2 Injection Molding Machine and Condition Recommendations

Ingeo resin will injection mold on most conventional equipment but there could be some torque limitations if the screw design has a high compression ratio. Compression ratios of 2.5-3 should be adequate for Ingeo resin. Typical molding conditions are listed below.

### Typical PLA Molding Conditions

<table>
<thead>
<tr>
<th>Zone</th>
<th>Barrel Temperature Setting, °F (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Feed throat</td>
<td>75 (24)</td>
</tr>
<tr>
<td>Feed Section</td>
<td>350 (177)</td>
</tr>
<tr>
<td>Compression Section</td>
<td>370 (188)</td>
</tr>
<tr>
<td>Metering Section</td>
<td>370 (188)</td>
</tr>
<tr>
<td>Hot Runner</td>
<td>400-410 (205-210)</td>
</tr>
<tr>
<td>Nozzle Tips</td>
<td>400-415 (205-215)</td>
</tr>
<tr>
<td>Mold</td>
<td>185-221 (85-105)</td>
</tr>
<tr>
<td>Screw Speed</td>
<td>125 rpm</td>
</tr>
<tr>
<td>Back Pressure</td>
<td>250 psi</td>
</tr>
<tr>
<td>Mold Shrinkage</td>
<td>0.012 in/in +/- 0.001</td>
</tr>
</tbody>
</table>

Please note that these are recommended starting parameters and may need to be optimized.

Successful molding of ASTM test specimens using a cold runner and cold sprue design was performed on a Sumitomo 110 ton press. Successful mold temperature was achieved by using either water or oil heat exchange units. Machine specifications were:

- Sumitomo SE100DU C250M 32
- Clamping force: 110 (U.S. ton)
- Distance between tie bar: 460 x 410 mm
- Overall size of platens (H x V): 650 x 600 mm
- Injection volume: 113 cm³
- Screw diameter: 32 mm

Because Ingeo 3801X has a relatively long crystallization time, it takes longer to set up in the mold than many commercial materials. Finding an optimum mold coolant temperature is recommended in order to get the best quality parts and the shortest cooling times. In addition, the Izod impact strength is sensitive to mold temperature. The following two plots illustrate the influence of mold temperature on the properties of an Ingeo 3801X when molded into ASTM test specimens. Barrel temperature = 190°C.
Influence of mold temperature on the cycle time and impact strength of Ingeo 3801X

**Cycle time vs. mold temperature**  
**Notched Izod impact vs. mold temperature**

Hot runners and heated sprues are generally recommended for the injection molding of Ingeo resins. Coat hangers were successfully molded from Ingeo 3801X using a heated sprue and mold designed for PP with a Toshiba 114 ton press. With water coolant medium at 90°C a cycle time of about 45 seconds was achieved.

In the event that Ingeo 3801X is not molded at temperatures facilitating crystallization of the matrix (for instance if a mold temperature of 30°C is used), the heat distortion temperature will be dominated by the glass transition temperature (Tg), and the material is not expected to withstand loads above 40°C. It is the responsibility of the converter to ensure that molding conditions are used that can meet the product requirements.

The stiffness of parts made from Ingeo 3801X decreases in a manner consistent with many typical semi-crystalline thermoplastics as the material is heated. Below the Tg of the matrix the material is quite stiff and rigid. As the material is warmed through the Tg, the modulus drops with temperature, and settles into a plateau region, above the matrix Tg and below Tm where modulus decreases to a smaller degree. The heat distortion temperature of the plastic is a strong function of load throughout this region. Finally the material melts at about 150°C. This behavior is in sharp contrast to amorphous Ingeo 3251D, where the modulus decreases very quickly at the glass transition, to a low level that is unable to bear a significant mechanical load.

Shown above are results from dynamic mechanical analysis (DMA) of a molded ASTM type I test bar, analyzed in three point bending geometry using a TA Instruments RSA III rheometer. Ingeo 3251D was molded with a mold temperature of 30°C in order to “quench” the parts in the amorphous state. Ingeo 3801X was molded with a 90°C mold in order to facilitate crystallization, enabling greater heat resistance / higher modulus above Tg.
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The image to the right shows ASTM dog bone style type I injection molded test bars that have been subjected to a heating rate of 0.5°C/min from room temperature to 118°C. At 118°C, the clarified polypropylene, polystyrene and amorphous Ingeo 3251D have all softened and bent significantly. Ingeo 3801X retains enough stiffness at this temperature to hold distortion to a minimum under this load.

The shear viscosity of Ingeo 3801X was characterized by both parallel plate and capillary rheometers. Shown to the right is the compilation of testing at 190°C (recommended melt temperature). Parallel plate was tested using 25 mm diameter aluminum plates, run in steady shear with a Rheometrics ARES strain rheometer. Capillary rheology was tested using 32, 16 and 4 mm length x 1 mm diameter capillaries, using a Rosand RH7, dual barrel rheometer. Tests were made under all conditions with a zero length x 1 mm diameter die in the second barrel, to easily enable Bagley and Rabinowitch corrections.

5.3 Mold/Tooling Recommendations

Hardened stainless steel is recommended for mold tooling. In most cases it has been found that thermal gates are preferred over valve gates and a hot-runner system designed for PET or PS can be used for Ingeo polymer. The shrink of Ingeo 3801X is relatively high (0.012 in/in) compared to amorphous PLA which is quite low (0.004 in/in), and tooling design should be carefully considered.

5.4 Additives

Colorants and slip agents can be added as a masterbatch at 15-30-wt% in PLA by dry blending with the neat resin in the required amount and adding the blend to the injection mold. The additions of colorants have been successfully done using liquid injection technology as well. Since PLA is not compatible with most incumbent materials, it is important that all additive masterbatches use PLA as the carrier. Some potential additives are inappropriate for extrusion with PLA because they are hygroscopic or hydrated salts (e.g. calcium carbonate) that would lead to severe PLA molecular weight degradation and
property loss. Note that adding any additives to Ingeo 3801X will likely dilute the other components in the formulation and could lead to processing and property changes. It is recommended that additive masterbatches to be used in Ingeo 3801X be compounded into Ingeo 3801X as a masterbatch, to ensure that the concentration of critical additives remains constant relative to the polymer concentration. It is recommended that all additive changes are thoroughly investigated for effects on process and property changes.

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Safety and Handling Considerations

Safety Data Sheets (SDS) for Ingeo biopolymers are available from NatureWorks. SDS’s 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. SDS’s are updated regularly; therefore, please request and review the most current SDS’s 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. Pellets or beads may present a slipping hazard.

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. 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.

Good general ventilation of the polymer processing area is recommended. At temperatures exceeding the polymer melt temperature (typically 175°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 (or goggles) to prevent exposure to particles, which could cause mechanical injury to the eye. If vapor exposure causes eye discomfort, improve localized fume exhausting methods or use a full-face respirator.

The primary thermal decomposition product of PLA is acetaldehyde, a material also produced during the thermal degradation of PET. Thermal decomposition products also include carbon monoxide and hexanal, all of which exist as gases at normal room conditions. These species are highly flammable, easily ignited by spark or flame, and can also auto ignite. For polyesters such as PLA, thermal decomposition producing flammable vapors containing acetaldehyde and carbon monoxide can occur in almost any process equipment maintaining PLA at high temperature over longer residence times than typically experienced in extruders, fiber spinning lines, injection molding machines, accumulators, pipe lines and adapters.

As a rough guideline based upon some practical experience, significant decomposition of PLA will occur if polymer residues are held at temperatures above the melting point for prolonged periods, e.g., in excess of 24 hours at 175°C, although this will vary significantly with temperature.

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 option is to recycle into the process 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. Disposal must be in compliance with Federal, State/Provincial, and local laws and regulations.

Environmental Concerns

Generally speaking, lost pellets, while undesirable, are benign in terms of their physical environmental impact, but if ingested by wildlife, 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 environment.

Product Stewardship

NatureWorks has a fundamental duty to all those that 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...
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environmental information on our products and their intended use, and then take appropriate steps to protect the environment and the health of our employees and the public.

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