

Cryogenic Grinding of Polymers with the CG-200 Freezer/Mill®



SUBJECT: Cryogenic Grinding of Polymers into Fine Powders

APPARATUS: CG-200 Freezer/Mill® and 6751 Small Vial Set

Processing polymers into fine particles is critical for consistent performance in research, manufacturing, and recycling. However, traditional grinding methods at room temperature generate heat, leading to material degradation, inconsistent particle sizes, and potential contamination issues.

Cryogenic grinding, or cryo-milling, addresses these issues by using very low temperatures to embrittle polymers, enabling cleaner fracture and more controlled size reduction while preserving the sample's composition. Cooling materials below their glass transition temperature (T_g)—often cited as below $-150\text{ }^{\circ}\text{C}$ —promotes brittle behavior, though T_g alone is not the sole predictor of grinding performance. Additives, unknown fillers, and polymer structure can all affect embrittlement, and brittleness does not always equate to fine grindability. In practice, most polymer samples are heterogeneous and may not behave consistently.

This application note outlines the practical considerations involved in cryogenic grinding polymers, emphasizing common challenges, grinder selection, and process optimization.

Considerations When Selecting Materials

Polymers respond widely to low temperatures. Selecting materials for cryogenic grinding requires understanding how cooling affects their mechanical behavior, especially embrittlement.

Thermoplastics such as polyethylene (PE), polypropylene (PP), and polyamide (PA), are typically suitable candidates due to their clearer thermal transitions, which support embrittlement and size reduction. However, even for these materials, fine grinding performance depends on the formulation, filler content, and additive load.

Elastomers like natural rubber and styrene-butadiene rubber often require significantly lower temperatures (e.g., $-196\text{ }^{\circ}\text{C}$ using liquid nitrogen) to achieve embrittlement. These materials resist fracture unless fully frozen and may remain ductile if not sufficiently cooled.

Due to their thermal stability and toughness, high-performance polymers such as PEEK and PTFE present additional challenges. Cryogenic grinding can support efficient size reduction; however, reaching sub- $100\text{ }\mu\text{m}$ or finer particles requires further optimization of energy input and grinding duration.

It is important to note that brittleness does not always guarantee effective grinding to small particle sizes. Polymer blends, additives, and layered or reinforced structures can influence particle formation, irrespective of temperature.

Materials Suitable for Cryogenic Grinding

Material Category	Polymer Type	Common Applications
Thermoplastics	Polyethylene (PE)	Often recycled and reprocessed into films, bags, and medical tubing
	Polypropylene (PP)	Ground for repurposing in automotive parts and packaging
	Polyamide (PA, Nylon)	Processed into fine powders for coatings, composite reinforcement, and additive manufacturing
	Polyethylene Terephthalate (PET)	Ground for recycling into fibers, bottles, and packaging materials
Elastomers (NR, SBR, EPDM)	Natural Rubber (NR)	Used in recycled tire materials, shock-absorbing pads, and industrial seeds
	Styrene-Butadiene Rubber (SBR)	Processed into fine powders for asphalt modification, footwear, and impact-resistant materials
	Ethylene Propylene Diene Monomer (EPDM)	Ground for reuse in automotive seals, roofing materials, and specialty composites
High-Performance Polymers (PEEK and PTFE)	Polyether Ether Ketone (PEEK)	Reduced to fine powders for aerospace and medical applications
	Polytetrafluoroethylene (PTFE, Teflon®)	Finely milled for coatings, gaskets, and medical components
Food Materials	Spices and Herbs (e.g., pepper, cinnamon, turmeric)	Retain volatile oils and active compounds, preventing flavor loss and improving shelf life
	Coffee Beans	Processed at low temperatures to maintain the aroma and produce consistent grind sizes for optimal extraction
	Proteins and Functional Powders (e.g., whey protein, plant-based proteins)	Enhances solubility, bioavailability, and mixing efficiency in food supplements and pharmaceutical formulations
	Fat-rich Ingredients (e.g., chocolate, nuts, dairy powders)	Prevents clumping and oil separation, maintaining uniform texture for confectionery and baking applications
	Gummy and Sticky Foods (e.g., dried fruits, cheese, marzipan)	Processed at sub-zero temperatures to reduce adhesion and improve consistency in coatings and fillings
Pharmaceuticals	Active Pharmaceutical Ingredients (APIs)	Cryogenic milling improves solubility, dissolution rates, and bioavailability for poorly water-soluble drugs
	Excipients (e.g., lactose, microcrystalline cellulose)	Ensures uniform blending, enhances tablet compression, and improves drug stability
	Biodegradable Polymers (e.g., PLGA, PCL)	Milled for use in sustained-release drug delivery systems and biodegradable implants

Process Challenges and Optimization

Cryogenic grinding introduces specific challenges that require attention to detail:

- **Temperature Control:** Maintaining consistent cryogenic conditions prevents sample warming, which can lead to inconsistent fracture behavior and poor size distribution.
- **Agglomeration:** As particles become smaller, they cluster due to surface energy. Mechanical solutions (e.g., flow agents or chamber design) and environmental controls (e.g., humidity management) help mitigate this.
- **Process Tuning:** Grinding time, impact energy, and cooling rate must be adjusted to meet target specifications without compromising sample integrity.

Selecting the Right Cryogenic Grinder

Choosing a cryogenic grinder requires careful consideration of material properties, processing efficiency, contamination risks, and operation convenience. The right choice ensures consistent particle size reduction, sample integrity, and process repeatability. Below are the key factors to evaluate:

Temperature Control and Efficiency

Maintaining the correct low temperature prevents material softening, degradation, or phase transitions. Choose a grinder that efficiently integrates liquid nitrogen cooling to facilitate continuous embrittlement, allowing for clean, precise fractures.

Particle Size Reduction Capabilities

Cryogenic grinders have distinct purposes. Some are designed for fast, coarse reduction, while others provide controlled grinding at cryogenic temperatures, ideal for tough or elastic polymers. When selecting a system, consider the sample type, target particle size, and consistency of results. Grinders with enclosed chambers, programmable cycles, and stable low-temperature performance offer advantages in applications where material integrity and repeatability are essential.

Material Compatibility

Different materials have unique grinding challenges. Some, like elastomers and high-performance polymers, require specialized handling to avoid heat-induced deformation. The selected grinder must be versatile enough to process thermoplastics, elastomers, pharmaceuticals, and composite materials effectively.

Sample Capacity and Lab Space

Laboratories and industries require grinders that can handle their sample volume without taking up too much lab space. Compact grinders are ideal for everyday research applications that require minimal bench space.

Contamination Control and Purity

A well-designed cryogenic grinder must minimize exposure to unwanted contaminants and maintain sample integrity to reduce the risk of sample oxidation, contamination, or loss of volatile compounds.

Automation and Process Control

Advanced models offer programmable grinding cycles, allowing for consistent pre-cooling, impact energy, and cycle duration. Automation reduces user error, improves process repeatability, and enhances safety in handling cryogenic conditions.

Why Choose the Cole-Parmer SamplePrep CG-200 Freezer/Mill®?

The Cole-Parmer SamplePrep CG-200 Freezer/Mill stands out among other cryogenic grinders due to its precision engineering, superior process control, and versatility in handling various materials.

What Sets us Apart?

- ***Unmatched temperature control for consistent embrittlement***

Unlike some cryogenic grinders that struggle with fluctuating temperatures, the CG-200 Freezer/Mill precisely maintains cryogenic conditions using liquid nitrogen, ensuring continuous embrittlement for effective grinding.

The closed-system design minimizes sample warming between cycles, preventing inconsistent grinding results.

- ***Optimized for the most challenging materials***

The CG-200 Freezer/Mill effectively handles thermoplastics, elastomers, and high-performance polymers (Table 1), while other grinders may struggle with these materials.

Particle Size Distribution Data

Particle size distribution data following cryogenic grinding with the Cole-Parmer CG-200 Freezer/Mil. The D10 value represents the particle size below which 10% of the sample falls, while the D90 value represents the size below which 90% falls.

All samples were processed using the following parameters:

- Pre-cool – 10 minutes
- Run time – 2 minutes
- Cycles – 4
- Cool time – 1 minute
- Rate – 12 cps

Table 1. Particle Size Distribution Data

Sample	Sample Amount (g)	Particle Size (median) (µm)	D10 (µm)	D90 (µm)
Polypropylene (PP) Pellets	3	331	95	660
Polycarbonate (PC) Pellets	3	559	231	1026
Polystyrene (PS) Pellets	3	167	40	464
Polyethylene (PE) Pellets	3	445	119	847
Polymer Pellets	3	48	9	116
Acrylonitrile Butadiene Styrene (ABS) Pellets	3	93	19	176
Polylactic Acid (PLA) Pellets	3	340	86	624
Polyamide (PA) Pellets	3	274	69	499
Polyurethane (PUR) Foam	0.5	175	57	561
Carpet	1	72	9	119
Nylon 6 Filter	0.5	172	39	484

- **Advanced programmable grinding cycles**

Many cryogenic grinders require manual adjustments, leading to variability between runs.

The CG-200 Freezer/Mill allows precise customization of pre-cooling time, grinding time, impact energy, and cycle duration, ensuring consistent, reproducible results across multiple batches.

- **Compact, space-efficient design**

Unlike larger industrial cryogenic grinders, which can be bulky and complex, the CG-200 Freezer/Mill is designed for research and small-scale applications – making it ideal for laboratories with limited bench space.

- **Reduced contamination and sample integrity protection**

Many conventional grinders risk sample oxidation and cross-contamination due to open processing systems.

The fully enclosed grinding chamber of the CG-200 Freezer/Mill prevents exposure to air and moisture, ensuring high-purity sample processing – a critical factor in pharmaceutical, polymer, and analytical research applications.

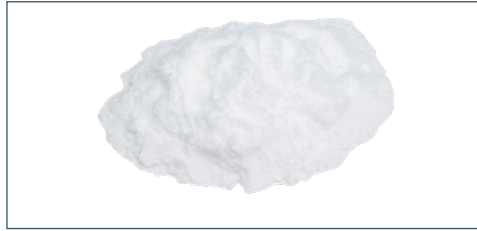
- **Improved safety and user-friendly interface**

Lid interlock system to prevent accidental opening operation. Liquid nitrogen sensor to ensure proper cryogenic conditions. User-friendly touchscreen interface for intuitive operation and storage of grinding protocols.

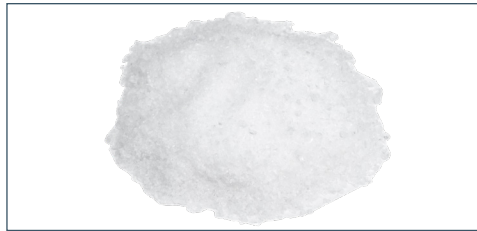
Note on Performance: The Freezer/Mill is well-suited for embrittling and fracturing tough or elastic polymers under controlled cryogenic conditions. While particle size outcomes depend on the specific material, formulation, and application goals, the system provides a reliable platform for consistent size reduction within a wide range of target sizes.

Before and After Samples

Polymer



Nylon



Contact and Additional Resources

For more details on cryogenic grinding solutions, application notes, and product specifications, visit cpsampleprep.com.

For technical support and inquiries:

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