CHEMICAL SCIENCES DIVISION: Cryogenic Reference Material Production Facility

The NIST Cryogenic Reference Material Production Facility at the Hollings Marine Laboratory produces raw material for developing Standard Reference Materials (SRMs®) and control materials (CMs) used in the measurement of analytes of interest. This facility consists of ISO Class 7 clean room space and the specialized equipment required for producing fresh-frozen cryohomogenized materials.

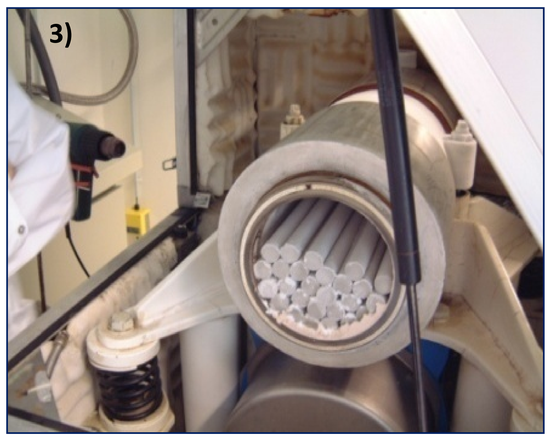
CRYOHOMOGENIZATION

Cryohomogenized SRMs are produced using the Palla VM-KT Vibrating Cryomill (Cryomill) creating a fresh, frozen powder sample.

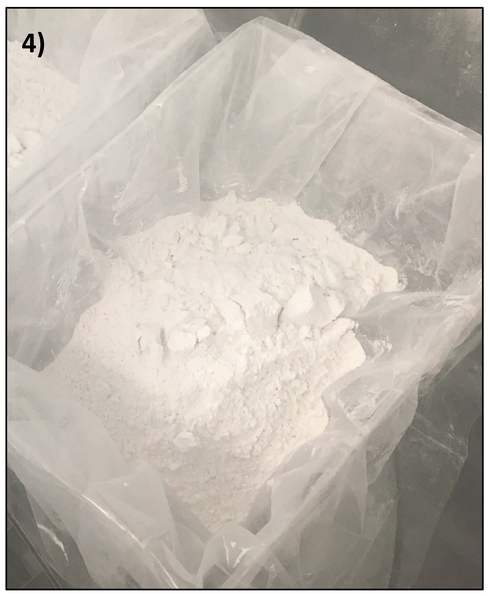
1.) Before using the Cryomill samples must be cut or crushed (if already frozen) into small fragments (< 2.5 cm) in order to fit properly into the Cryomill feeder.

The Cryomill is cooled using liquid nitrogen (LN_2). The motor begins vibrating and rotating 26 titanium rods located within the drum chamber.

2.) The pre-frozen sample fragments are introduced into the feeder at the top of the Cryomill.



3.) The sample travels through the vibrating chamber where the titanium rods are located.



4.) Cryohomogenized powder sample falls from a distribution tube at the bottom of the Cryomill. The sample is caught in a container and quickly placed in an LN_2 vapor-phase freezer (-150° C).

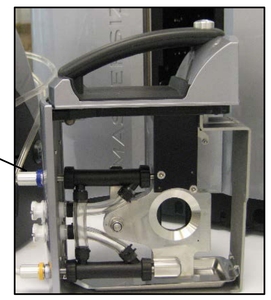
The cryohomogenized powder is then passed through the Cryomill following the steps above 2 to 3 more times to ensure homogeneity.

After homogenization is complete, the sample is bottled and ready for value assignment and certification.

PARTICLE SIZE ANALYSIS OF SRMs®/CMs

Laser-diffraction particle size analysis (PSA) using wet dispersion can measure the equivalent spherical diameter in an average volume distribution.

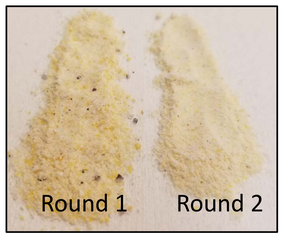
Particles are recirculated via wet dispersant. Lasers produce scatter as particles pass through beams. Light scattering and optical properties of the material are used to determine the equivalent spherical diameter.

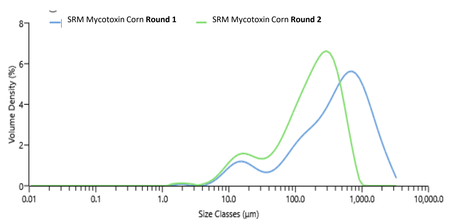


PSA utilized for multiple purposes

- During production to determine diminishing returns of milling or if material is sufficiently milled past a pre-determined threshold.
- Determine fraction that is below the respiratory hazard threshold (< 10 µm)
- Size characterization of a material to determine homogeneity of size across batches
- Whether material is too large/small for critical environments or instrumentation.

Example 1. **SRM 1565 Mycotoxins in Corn**
PSA used to track progress in cryomilling operations





Example 2. **SRM 2682c Subbituminous Coal**
Particle size characterization reported as an Information Value on the Certificate of Analysis

Information Values: Particle size measurements were made using a laser-based light scattering system. Approximately 0.5 g of material (refractive index: 2.42, absorption index: 1.0) was measured using water as the dispersant, (refractive index: 1.33) and 0.1 % Triton X-100 as a pre-wetting surfactant. Calculated 10th percentile ($d_{0.1}$) and 90th percentile ($d_{0.9}$) particle sizes (percent volume of particles smaller than the value) are $d_{0.1}$ = 4 µm and $d_{0.9}$ = 55 µm. The volume weighted mean is 14 µm. The fraction of material smaller than 10 µm in diameter is 36 %. The particle size distribution is shown in Figure 1.

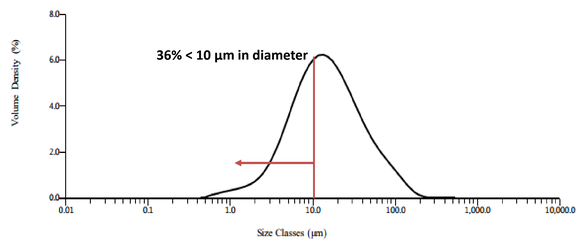


Figure 1. Particle size distributions in SRM 2682c