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ACCESSORIES FOR PROCESSING SMALL VOLUMES IN BALL MILLS

One of the challenges when using ball mills is the processing of small sample volumes. Food, tissues, pharmaceuticals, and many other samples are often limited to only a few grams in quantity. To homogenize such small amounts for analysis, or for developing new materials, matching jar volumes are required. In addition, small-volume jars allow for simultaneous processing of multiple samples. This leads to high sample throughput and enables effective parameter variation in material development.

RETSCH offers an extended range of accessories for the processing of small sample volumes in mixer mills. It should be taken into account that a simple downscaling from large volume to small volume is not always possible; in small jar volumes, for example, the power of the balls is lower or thermal transport processes are faster. This application note focuses on the homogenization of different materials in small-volume jars.

MULTI-CAVITY JARS FOR MIXER MILLS MM 500 CONTROL AND MM 500 NANO

Accessories for the models MM 500 control and MM 500 nano include multi-cavity grinding jars that have the same advantages as conventional Screw-Lock grinding jars: easy jar filling thanks to flat jar lid - even in a glove box; simple clamping of the jars; possibility to visually check the status during operation, and easy cleaning.

Two multi-cavity jar types are available: one with four 10 ml and the other with two 25 ml cavities. They are made of stainless steel and are supplied with pouring aids for taking out a sample from one cavity while the others remain plugged.



Models

- | MM 500 control
- | MM 500 nano

Multi-cavity jars

- | 4 x 10 ml
- | 2 x 25 ml
- | stainless steel
- | with pouring aid to plug individual cavities

In a multi-cavity jar, the oval shape of the cavities corresponds to the single-cavity Screw-Lock jars. This shape facilitates effective ball movement for crushing of hard, brittle, fibrous and dry materials, see example of grinding basalt. The multi-cavity jars are also suitable for applications in mechanochemical material development, for example, when dealing with small volumes of, e. g., expensive educts.

Cryogenic grinding is another application for which the jars can be employed. Samples are embrittled with liquid nitrogen in the MM 500 control or by using the CryoKit for MM 500 nano, see example of cryogenic grinding of plants. In addition, the multi-cavity jars are particularly suitable for wet grinding processes to obtain ultra-fine particles. Taking wet grinding of TiO₂ as an example, the results are comparable to those achieved with larger jars, see example below.

CRYOGENIC GRINDING OF FRESH PLANTS

| | 10 ml | 25 ml |
|------------|----------|----------|
| plant | 1.5 g | 4 g |
| 7 mm balls | 12 balls | 35 balls |

- | 30 Hz
- | -100 °C with 15 min pre-cooling
- | 9 cycles of 1 min grinding with 1 min intermediate cooling

RESULTS



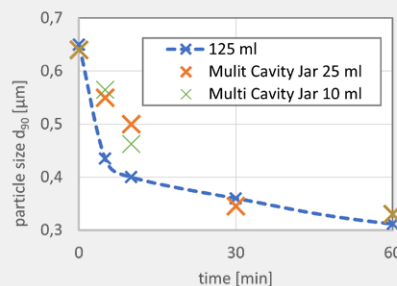
- | Good homogenization under cryogenic conditions.

WET GRINDING OF TiO₂

| | 10 ml | 25 ml |
|----------------------|-------|-------|
| TiO ₂ | 2 g | 5 g |
| 0.1 mm balls | 22 g | 55 g |
| 1% NaPO ₄ | 2 ml | 5 ml |

- | 30 Hz
- | 5, 10, 30 and 60 min

RESULTS



- | Fineness comparable to results with 125 ml jars.

GRINDING OF BASALT

| | 10 ml | 25 ml |
|--------------|--------|---------|
| Basalt split | 17 g | 7 g |
| 12 mm balls | 1 ball | 4 balls |

- | 30 Hz
- | 1, 2 and 5 min

RESULTS



- | Reproducible results in all cavity stations.
- | Fineness comparable to results in MM 400 screw-top jars with identical capacity.

2 ML STAINLESS STEEL REACTION VIALS FOR MM 500 CONTROL AND MM 500 NANO

For the MM 500 control and MM 500 nano an adapter is available that accommodates nine 2 ml reaction vials made of stainless steel or, alternatively, 18 single use reaction vials of 1.5 or 2 ml.



Models

- | MM 500 control
- | MM 500 nano

2 ml reaction vials

- | 316L steel
- | adapter for 9 vials (also suited for 18 x 1,5 ml and 18 x 2 ml single-use vials)

Models

- | CryoMill
- | MM 400
- | MM 500 vario



Homogenization of biological samples as well as cell disruption are often performed with small volumes, for example, in disposable reaction vials. The MM 500 nano allows high-energy grinding with a frequency of up to 35 Hz. With the MM 500 control, temperature-controlled processing is executed by connecting the mill to a chiller and cooling the sample during grinding. In both mills cryogenic grinding at very low temperatures is also an option, for example, to homogenize tissue. The MM 500 control provides an automated and convenient handling process to freeze the sample down to -100 °C. For cryogenic grinding in the MM 500 nano the CryoKit is available for manual pre-cooling of the fully loaded adapter in a liquid nitrogen bath prior to homogenization. As polymeric reaction vials may not withstand the impact of the balls and may break when frozen, the use of stainless-steel vials is recommended for cryogenic applications.

2 ML STAINLESS-STEEL REACTION VIALS FOR MM 400, MM 500 VARIO AND CRYOMILL

PTFE adapters are available for the MM 400 and MM 500 vario and a steel adapter is available for the CryoMill. The adapters accommodate 1.5 ml and 2 ml single-use reaction vials or 2 ml stainless-steel reaction vials.

2 ml reaction vials

- | 316L steel
- | PTFE adapter for 5 & 10 vials (MM 400 and MM 500 vario)
- | 4 x 10 & 2 x 5 PTFE adapter (MM 500 vario)
- | 6 vials steel adapter (CryoMill)



These adapters enable simultaneous preparation of up to 6 samples in the CryoMill, up to 20 samples in the MM 400 and up to 50 samples in the MM 500 vario. This is beneficial for sample preparation, cell disruption and mechanochemical processes alike, see example of grinding of tablets.

The CryoKit is used for cryogenic grinding in the MM 400 and MM 500 vario. Generally, as polymeric reaction vials may break when frozen, the use of 2 ml stainless-steel reaction vials is recommended for this application, see cryogenic grinding of tissue. If very cold temperatures are required, for example when grinding polymers, the CryoMill offers maximum cooling down to -196 °C, see example of cryogenic grinding of polyamid.

The images on the left show mixer mill types with corresponding adapters for 2 ml vials.

GRINDING OF TABLETS

MM 500 vario

- | 1 tablet cut into 4 mm pieces
- | 1 x 7 mm ball, stainless steel
- | 30 Hz
- | 1.5 min

RESULTS



- | Manual pre-crushing.
- | Fast and reproducible sample homogenization.

CRYOGENIC GRINDING OF TISSUE

MM 400

- | 3 x 4 mm tissue, pre-cooled
- | 4 x 5 mm balls, tungsten carbide, pre-cooled
- | Adapter for 10 PTFE vials, 10 min, pre-cooling of fully loaded adapter in CryoKit
- | 30 Hz
- | 30 sec grinding
- | 3 cycles with 5 min intermediate cooling in CryoKit

RESULTS



- | Heavy tungsten carbide balls create high impact forces.
- | Pre-cooling of adapter and sample prevents the sample from sticking to the walls.

CRYOGENIC GRINDING OF POLYAMIDE

CryoMill

- | 3 g polyamide pellets
- | 1 x 7 mm ball, tungsten carbide
- | 15 min pre-cooling
- | 30 Hz
- | 30 sec grinding
- | 9 cycles with 1 min intermediate cooling

RESULTS



- | To increase impact forces, larger ball sizes and jar volumes are required.
- | The temperature and impact forces required depend on the polymer type.

Models

- | MM 400
- | MM 500 vario



5 ML STAINLESS STEEL JARS FOR MM 400 AND MM 500 VARIO

In addition to 2 ml reaction vials, 5 ml stainless steel jars are available for use in the MM 400 and MM 500 vario. The adapter holds 4 jars, so that 8 samples can be processed simultaneously in the MM 400 and up to 24 samples in the MM 500 vario. The 5 ml jars are suitable to grind plants, pharmaceuticals, tissues, harder materials like basalt, as describes for the above-mentioned examples.

The images on the left show mixer mill types with corresponding adapter.

Find out more at www.retsch.com

5 ml grinding jars

- | stainless steel adapter
- | for 2 or 4 grinding jars

