

CryoMill - Cryogenic Grinding was Never More Convenient or Efficient

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Some sample materials have properties which make size reduction at ambient temperature impossible. If, for example, very elastic materials need to be ground or volatile components have to be preserved for further analysis, it is essential to carry out cryogenic grinding. The use of liquid nitrogen helps to embrittle the sample, thus improving its breaking properties, and preventing volatile substances from escaping due to the frictional heat produced by the grinding process.

Retsch has developed the CryoMill (*Figure 1*) especially for this application. An integrated cooling system ensures that the grinding jar is continually cooled with liquid nitrogen before and during the grinding process. Thus the sample is embrittled and volatile components are preserved. Thanks to the Autofill System, liquid nitrogen is continually refilled in the exact amount which is required to keep the temperature at –196°C.

Grinding in the CryoMill is effected by impact and friction. The grinding ball moves freely inside the grinding jar which allows for thorough pulverisation of the sample, in contrast to other cryogenic mills with differently shaped grinding tools.

Based on an extensive dialogue with users of the CryoMill, Retsch was able to integrate a variety of suggestions into the further development and optimisation of the new product generation. As a result, the new CryoMill makes grinding with liquid nitrogen as convenient and effective as never before.



Figure 1. Retsch's CryoMill with 50 I liquid nitrogen tank

Benefits

- Increased oscillating frequency of up to 30Hz allows for 50% higher energy input
- Reinforced housing and optimised liquid nitrogen duct

Example Plastic (PE-LD)

LD polyethylene is a highly elastic material which is very difficult to grind with impact and friction, even at very low temperatures. Compared to a grinding process at 25 Hz, the results at 30 Hz have improved by 50%.

Parameters:

6 g PE-LD granulate in grinding jar 50 ml, stainless steel, 1 grinding ball Ø 25 mm, stainless steel,

5 cycles of 2 min each, with 1 min intermediate cooling at 25 Hz (blue curve) and 30 Hz (red curve)



Example Leather

Leather is a very tough and fibrous material. These properties make it very robust for usage but also fairly difficult



- Clearly structured user interface, memory for 9 SOPs
- Additional accessories such as adapter for 6 reaction vials

Improved grinding results thanks to increased vibrational frequency

The maximum vibrational frequency of the new CryoMill generation is 30 Hz. This allows for a considerably higher energy input which results in smaller grind sizes. This is particularly true for extremely tough and elastic materials.

to grind. Processed at 25 Hz for a short interval, the material is only slightly torn up whereas at 30 Hz, after the same grinding time, it is completely pulverised.

Parameters:

8 g leather (approx. 8x8 mm) in grinding jar 50 ml, stainless steel, 1 grinding ball Ø 25 mm, stainless steel,

3 cycles of 2 min each, with 1 min intermediate cooling at 25 Hz (left) and 30 Hz (right)

Convenient operation

Thanks to the improved liquid nitrogen duct and electronic monitoring, direct contact of the user with LN_2 is virtually impossible. The new CryoMill offers a maximum of operation safety while ensuring low LN_2 consumption.

The operating interface has become more user-friendly; the clear structure allows for intuitive programming of grinding cycles.

The new product generation features memory for 9 Standard Operating Procedures (SOPs), allowing to carry out routine applications at the push of a button.

Inserting and releasing the grinding jar has become even easier and safer.

Wide range of accessories

The screw-top grinding jars of hardened steel and stainless steel have been specifically designed for use in the CryoMill. The fact that no sample material can escape makes them particularly safe. They are available with volumes of 5 ml, 10 ml, 25 ml, 35 ml and 50 ml. For applications where steel jars cannot be used due to possible sample contamination, Retsch offers a 25 ml grinding jar of zirconium oxide and matching grinding balls.

Various adapters make the CryoMill a versatile instrument. If only very small sample amounts need to be processed, an adapter for 2 resp. 4 grinding jars of 5 ml is recommendable. It is also possible to use 2 ml reaction vials with the CryoMill thanks to another adapter which can hold up to 6 tubes.

For safe and comfortable operation of the CryoMill, the unit is equipped with an autofill system for liquid nitrogen which can be obtained with a 50 litre container (for processing approximately 20 to 30 samples). It is also possible to connect existing cryo tanks to the mill.

Summary

For tough and elastic materials as well as for samples which contain volatile components, cryogenic grinding is the only method to obtain the required analytical fineness. The Retsch CryoMill has proven to be the perfect tool for grinding temperature-sensitive materials with a glass point below room temperature. The grind sizes are significantly finer than those produced by comparative mills and have further improved with the new product generation. Especially for difficult materials such as polyethylene or leather the Retsch CryoMill is the ideal instrument for sample preparation.

More information at www.retsch.com/cryomill

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Launch of a Mechanical Tablet for Routine Checks of Tablet Hardness Tester Functionality



Measuring product breaking force is a standard procedure in every tablet manufacturing facility. Like all instrumentation, regular qualification of a hardness tester is mandatory. In order to ensure that the instrument is functioning within specifications, **Sotax** has developed the MT, a simple device that is designed for frequent routine verifications of the functionality of tablet hardness testers between qualifications.

Supplier-independent, the MT is designed to work with all hardness testers. The tablets are engineered so that a predetermined amount of force triggers a mechanical breaking point which is recorded by the hardness tester.

MTs are manufactured at five different breaking forces with an accuracy of ±0.5 Kp or better (5 Kp, 10 Kp, 15 Kp, 20 Kp, 30 Kp). They can also verify six different physical dimensions ± 0.03mm (5mm, 10mm, 14mm, 15mm, 20mm, 30mm).

The devices are all calibrated, verified, and tested according to a strict quality process using a NIST traceable calibration instrument. A full internal test report along with a calibration certificate is provided with each MT.

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Launch of Next-Generation Capillary Electrophoresis (CE) Platform

Beckman Coulter Life Sciences offered products across a





Solutions in Milling & Sieving

New Dimensions in Sample Preparation and Particle Analysis





range of biotech, analytical chemistry and industrial applications at Pittcon 2013.

One example of new applications is found with the PA 800 plus next-generation capillary electrophoresis (CE) platform, which provides automated, quantitative analysis of protein purity, charge isoform distribution and glycan structure. Originally designed with biopharmaceutical analysis in mind, the system can also be used in food and beverage quality studies for monosaccharide and protein analysis, and in the quantitative characterisation of ions, organic acids and other charged or polar molecules of importance.



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