Efficient Grinding at –196 °C

Retsch's New CryoMill for Safe and Reproducible Grinding with Liquid Nitrogen

The CryoMill has been specially designed for cryogenic grinding. The mill features an **integrated cooling system** which continually cools the grinding jar with liquid nitrogen before and during the grinding process. Thus the sample is embrittled and volatile components are preserved.

The CryoMill does not require bothersome manual refilling with LN2. The liquid nitrogen circulates through the system and is continually replenished from an **autofill system** in the exact amount which is required to keep the temperature at –196°C. The automatic cooling system guarantees that the grinding process is not started before the sample is thoroughly cooled. This results in reduced consumption and guarantees reproducible grinding results.

The operation of the mill is exceptionally easy. The grinding parameters can be stored and called up for routine tasks. The display features LED lights which indicate the current state of operation, e.g. cooling or grinding. With a vibrational frequency of 25 Hz the CryoMill processes sample materials very effectively in a few minutes. The sample is mainly ground by impact but also by friction which allows for **substantially finer grind sizes compared to other cryogenic mills**. The CryoMill is equipped with one grinding station for no-loss screw-top grinding jars of 25 ml, 35 ml and 50 ml. Another option is the use of an adapter which holds up to four 5 ml grinding jars. The mill can also be operated without cooling which makes it suitable for a vast range of applications.

Application Example Polymers

In the polymer industry XRF (X-Ray Fluorescence) analysis is a very efficient multi-element method for the determination of heavy metals in plastic parts or granulates. In a modern laboratory not only the accuracy and reproducibility of the results are important, but also the time factor and the safety of the laboratory employees are crucial aspects. That is the reason why XRF has become the preferred method for many applications because ICP and AAS analyses require time-consuming digestion of the sample material with strong acids.

For XRF analysis, the polymers have to be pulverised first. To obtain reliable results, the powder should have a fineness of at least 500 microns. Smaller particle sizes are advantageous, because less disturbing reflections are detected and the material is also more homogeneous.

Trial Conditions

Retsch CryoMill
Grinding temperature: -196 °C
Frequency: 25 s-1
Time: 2 min

Retsch MM 400
Pre-cooled sample (-196 °C), grinding at room temperature
Frequency: 25 s-1
Time: 2 min

Standard cryogenic mill
Grinding temperature: -196 °C
Frequency: 10 s-1
Time: 5 min

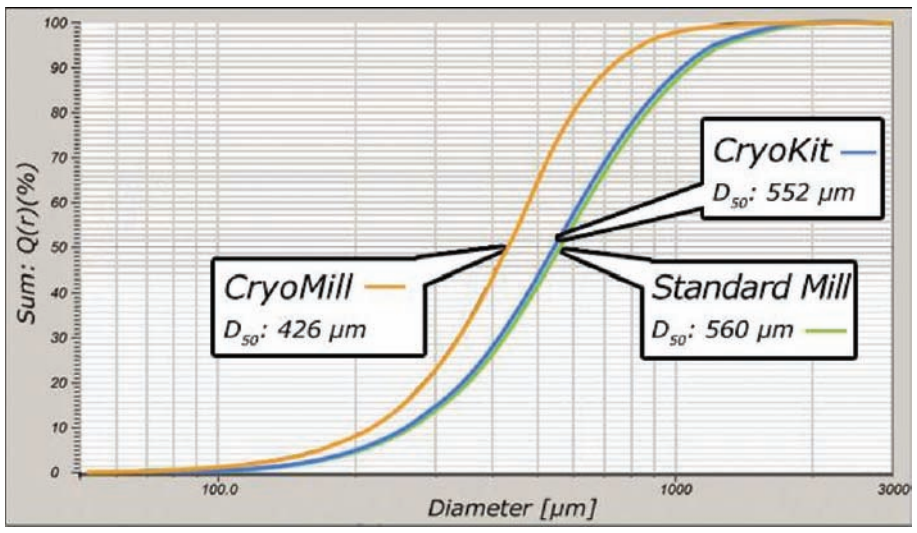

Figure 1. Grinding results car tyre

Results

The trials were carried out with 3 different mills: the Retsch CryoMill, the Retsch Mixer Mill MM 400 with CryoKit, and a standard mill for cryogenic grinding. The sample was a piece of commercial car tyre.

For the MM 400 the sample was pre-cooled in an insulated container (CryoKit) filled with liquid nitrogen and then ground at room temperature. *Figure 1* shows the results obtained with each mill. It can be clearly seen that the result of the MM 400 with CryoKit and the standard cryogenic mill are almost identical. The fineness achieved with the CryoMill is significantly better (>20 % finer).

Figure 2. Easy handling of the grinding jar

The sample for the second trial was a car seat filling. Usually, this type of material is done with a cutting mill which, however, hardly produces particles smaller than 1 mm, because the sample gets too hot during the cutting process. For applications like this the CryoMill is very effective – sizes down to 200 µm are easily achieveable.

Application Example Leather

In another trial a leather glove was tested. Leather is a material which is difficult to grind. A cutting mill would be the obvious choice, however, the final fineness is usually limited to 1 mm. To achieve a smaller grind size - e.g. for subsequent extraction or digestion for chromatographic or spectroscopic analysis - the sample was processed in the CryoMill and also in a standard cryogenic mill. The photos show the grinding results of both mills after 5 minutes. The CryoMill achieved a fineness of approximately 250 microns whereas the standard mill hardly ground the leather at all. The test results clearly show the superiority of the CryoMill.

Figure 3. Grinding results CryoMill

Figure 4. Grinding results standard cryogenic mill

Conclusion

For many materials cryogenic grinding is the only method to obtain particle sizes below 500 microns which are a prerequisite for reliable and meaningful XRF analysis results. The Retsch CryoMill has proven to be the perfect tool for grinding temperature-sensitive materials which are above the glass point at room temperature. Moreover, the grind sizes are significantly finer than those produced by comparative mills. Especially for difficult materials such as car tyres or leather the Retsch CryoMill is the ideal instrument for sample preparation. Since the mill works with a liquid nitrogen cycle, the system is especially safe and easy to use.

The CryoMill at a glance:

- Fast, efficient cryogenic grinding at –196°C
- Ideal for plastics, temperature-sensitive materials and samples with volatile components
- Safe and convenient thanks to an integrated cooling system with Autofill
- Programmable cooling cycles, digital parameter setting
- High final fineness down to approx. 5 microns
- Highly reproducible grinding results

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