

掺 2%钼钇铝的石榴石

行 业:

进料尺寸: < 3 mm (thickness)

最终精度: d50 < 30 nm

样 品 量: 5 g

研磨建议: For finest grinding results, we recommend using a Planetary Micro Mill PULVERISETTE 7 premium line.



PLANETARY MICRO MILL PULVERISETTE 7 PREMIUM LINE

Main disk speed: 1100 rpm (10'-120')

- 20ml grinding bowl made of zirconium oxide (ZrO₂)
- + 10x ZrO₂ grinding balls with 10 mm Ø (0-10 min)
- + 30 g of ZrO₂ grinding balls with 1 mm Ø (10-30 min)
- + 30 g of ZrO₂ grinding balls with 0,1 mm Ø (30-120 min)

Feed quantity: 2,5 g (total provided sample)
Feed Size: < 3 mm (pre comminuted)
Additive: + 13ml EtOH (after 1 min dry grinding)
Grinding time: 120 min
Final fineness: d50 < 80 nm (after 60 min)
Comments: Original start size (~ 3 cm) is way too long for placing it into a grinding bowl directly.

We have placed the glass like sample into a plastic bag and pre ground sample with a few strokes of a hammer into pieces < 4 mm length.

Such pre ground sample could be fed into the grinding bowl of Planetary Micro Mill PULVERISETTE 7 premium line. With 10 mm Ø grinding balls, the pre ground sample got comminuted successfully. After 1 minute of dry grinding, sample is sticking lightly to bowl and grinding balls.

This uses to happen after the majority of particles reached a fineness of < 20 µm. Interacting forces between fine ground particles will become bigger as their own g-force. Therefore, particles will stick to each other and become compressed by the used grinding balls. These clusters of particles also contain bigger particles which will not be ground any further.

A longer dry grinding is not recommended and we added about 6.5 ml of ethanol (EtOH) to the sample and proceeded grinding in suspension. We recommend adding enough of liquid to maintain a motor oil like viscosity. This way, good grinding results and less abrasion on grinding materials should be found. DMSO was not available to the application laboratory.

To avoid over pressure, we ground the sample in steps of 3 minutes, followed by a programmed pausing time of 8 minutes. After several cycles, the outside temperature of the bowl should be checked (remain below 75 °C); grinding time or programmed pausing time might be readjusted afterwards.

After a total of 10 minutes, we changed grinding balls to 1 mm Ø to improve the present grinding result. Also further 6.5 ml EtOH have been added to the sample. After 20 minutes, a $d_{50} < 180$ nm was determined with our Laser Particle Sizer ANALYSETTE 22 NanoTec plus. After a total of 30 minutes, $d_{50} < 163$ µm was only lightly improved. Therefore, we performed a further change to 0.1 mm Ø zirconium oxide grinding balls after 30 minutes. With smallest grinding balls, finest grinding results should be achieved.

Small sample portions (after 20 / 30 minutes) have been picked during the grinding process and will be found in ~2 ml Eppendorf tubes for demonstration.

After a total of 60 minutes of grinding, a $d_{50} < 85$ nm was detected. For a proper detection of such small particles, Mie parameters should be given for a more precise result. When Mie parameters of sample will be provided, a recalculation of grinding results is still possible. A longer grinding time (for a total of 120 minutes) hasn't improved the determined grinding result any further. Grinding has been interrupted. It is possible that finer results might be achieved when grinding agents (in form of surfactants, in water also acids, bases or salts) might be added to the sample (to hinder flocculation of nano sized particles).

Finally we like to mention that nano sized particles tend to flocculate more or less rapidly. This can happen during the grinding process and will, most likely happen during transportation (after several hours or days). Therefore, we recommend to pre treat the sample after arrival for several minutes with ultrasonic before sample will be used again.



Start size of particles before grinding has been performed.



After only one minute of dry grinding, sample is already sticking to bowl and grinding balls. Longer dry grinding is not possible to improve fineness significantly.