

Evaluation Report of the Special Vial Holders from Automaxion SARL

The goal of the test performed in the FRITSCH application laboratory was to investigate the performance of the special vial holder for sample preparation.

All tests were done with the FRITSCH pulverisette-5 Planetary Ball Mill.

A. Geometry and design

The dimension and design of the vial holders are in accordance to the dimensions of the normal FRITSCH 500ml bowls. They can be used with the pulverisette-5 Clamping device 'Safe-Lock'.

B. Grinding test

We choose four different materials with different particle size distributions and different physical properties to test the milling effect in the test tubes.

1. Coarse sand – size approx. 1-2mm
2. Fine sand – size < 150 μ m
3. Alumina Al₂O₃ – size < 500 μ m
4. Zinc Oxide ZnO – size < 20 μ m

B.1. Coarse sand

The first test was done with the coarse sand. This was to evaluate the possibility of dry grinding. To reduce the size of this coarse material, we have to use large grinding media. Due to the dimensions of the vials, the maximum size of the grinding beads is 5mm.

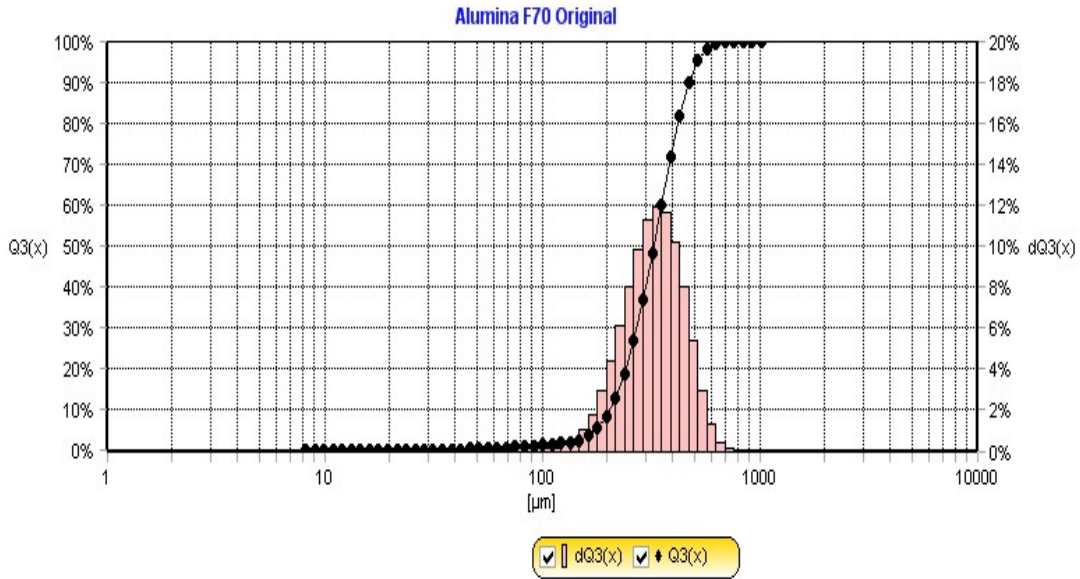
We filled the vials with a sufficient numbers of 5mm beads (50 pieces). The chosen material was agate and steel.

After 5 min. milling with a rotational speed of 400 rpm all the vials were destroyed by the beads. All material remains in the vial holder.

Result: For dry milling of coarse material we need at least beads of 5mm in diameter. Even with the lightest beads (agate) the vials were destroyed. Dry milling with the special vials is under these circumstances not possible.

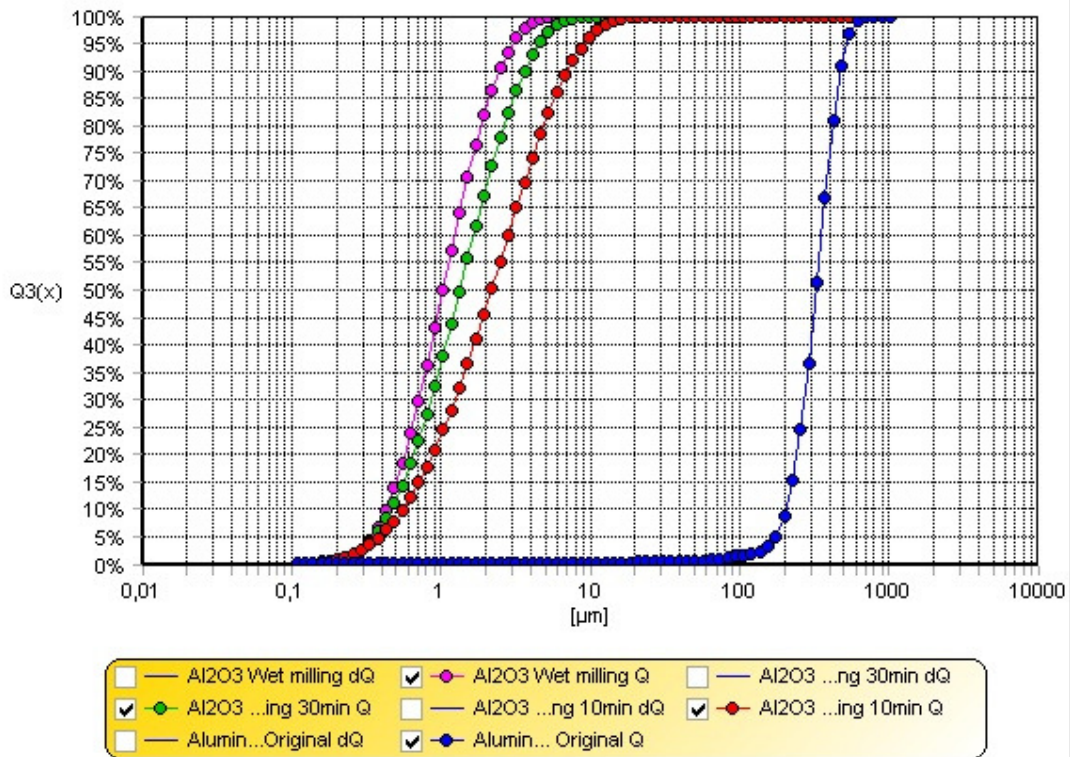
B.2. Alumina Al₂O₃

The next test was done with a rather hard material: alumina. The Original size is: D50= 327 μ m and D90= 473 μ m



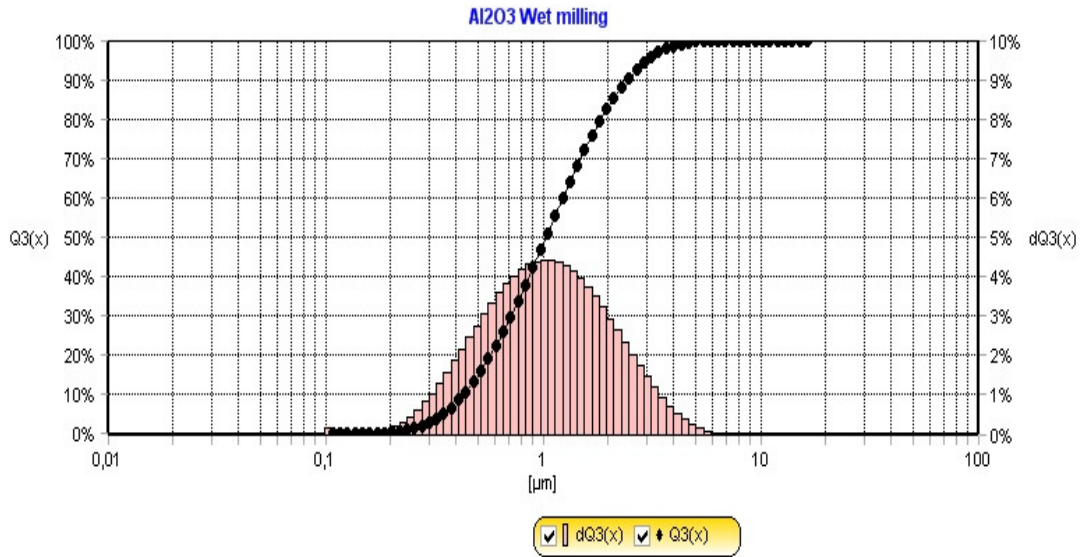
For the test the small vials (volume 15ml) were used. We put 20g of 1mm ZrO₂ beads into the vial together with 6g Alumina and 4ml of water. The Particle Size distribution was measured after 10 min. – 30 min. and 1 hour.

Result:



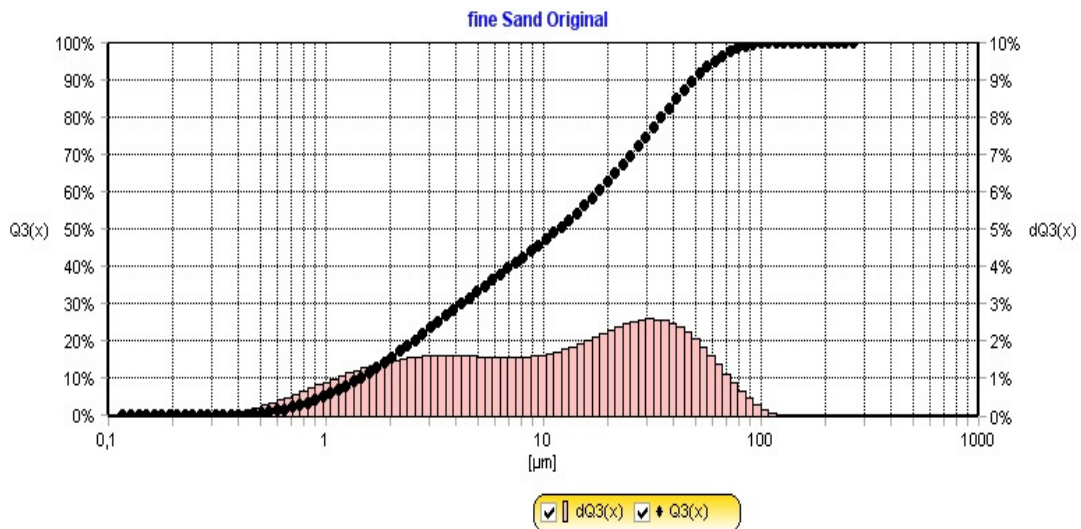
After 1 hour grinding time we got a very good grinding result. Now the d-values were:

D50 = 1,2 μm and D90 = 2,4 μm



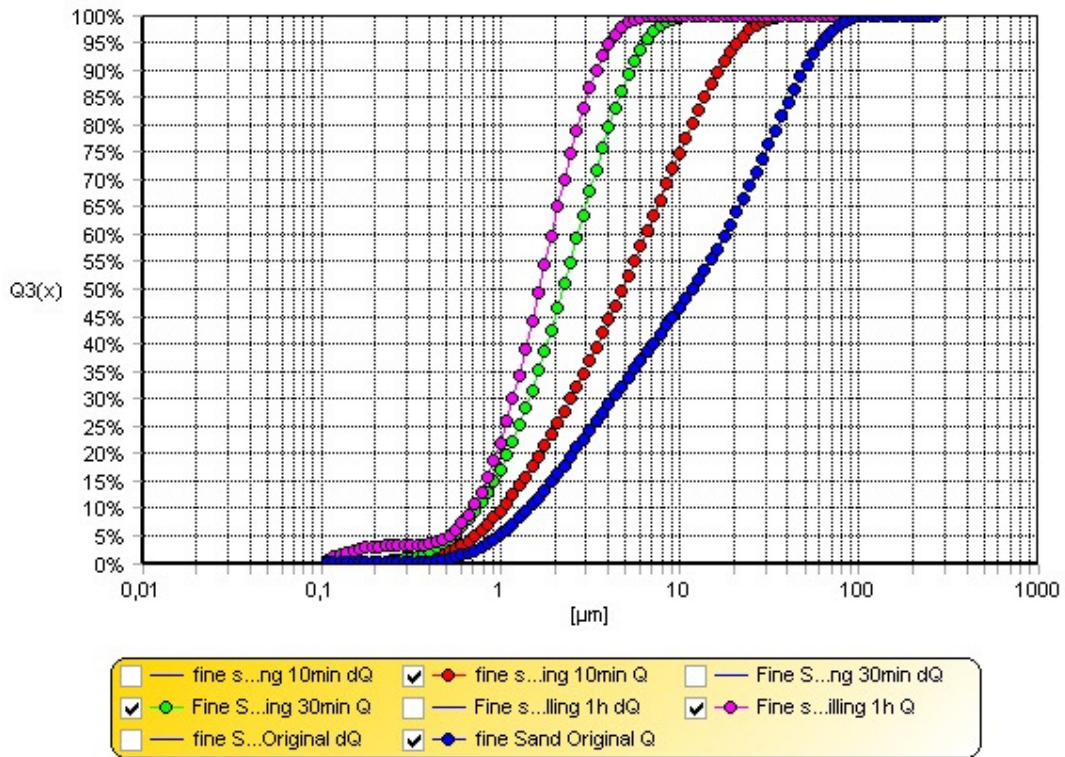
B.3. Fine Sand

The next test was done with a medium sized material: fine sand. The Original size is: D50= 11,7 μm and D90= 50 μm



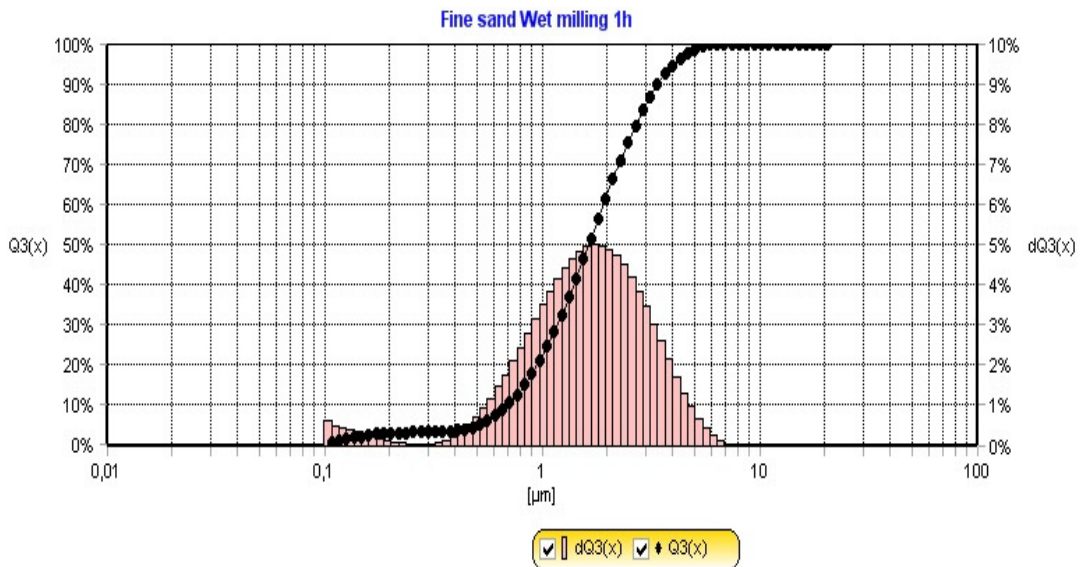
For the test the small vials (volume 15ml) were used. We put 20g of 1mm ZrO₂ beads into the vial together with 5g Alumina and 5ml of water. The Particle Size distribution was measured after 10 min. – 30 min. and 1 hour.

Result:



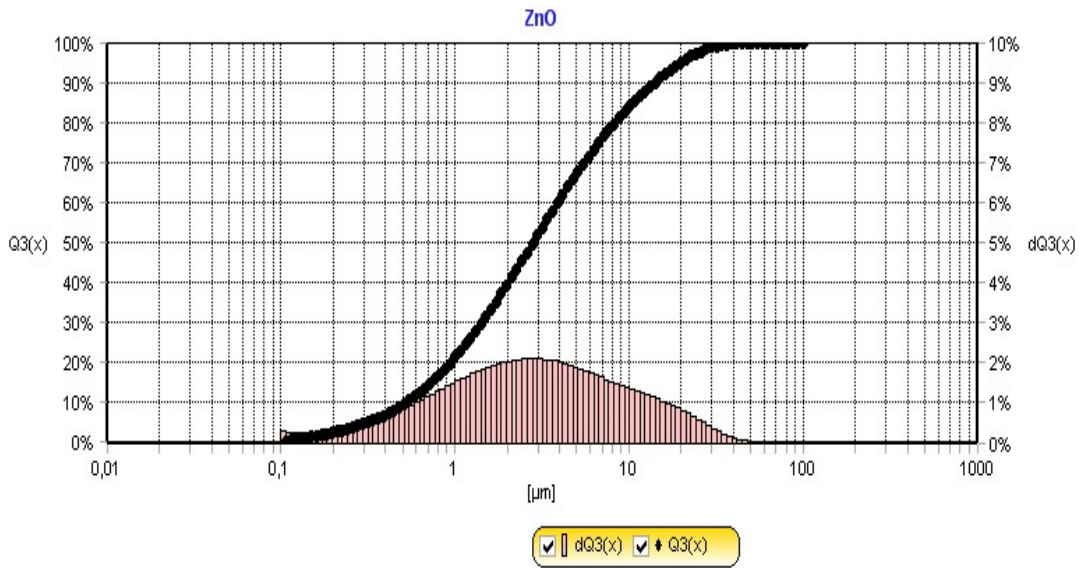
Again the grinding development is obvious. After 1h we found the result:

D50 = 1,65 μm and D90 = 3,4 μm



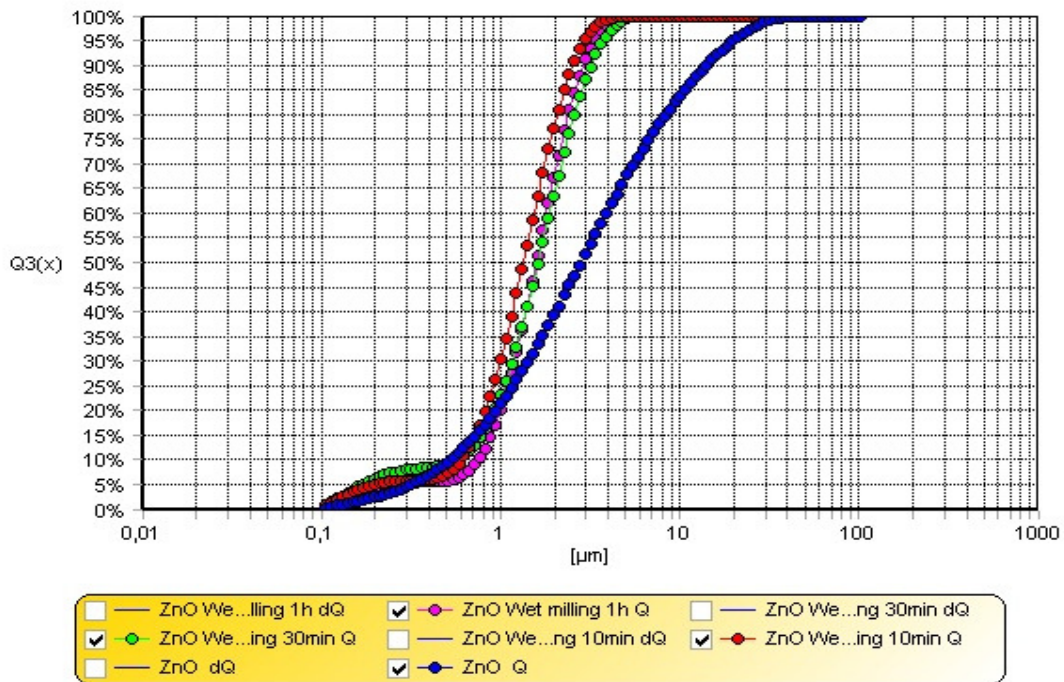
B.4. Zinc Oxide ZnO

The last model sample was Zinc Oxide. The Original sample is already very fine. So the was to show, that even very fine powder can be used for a size reduction.



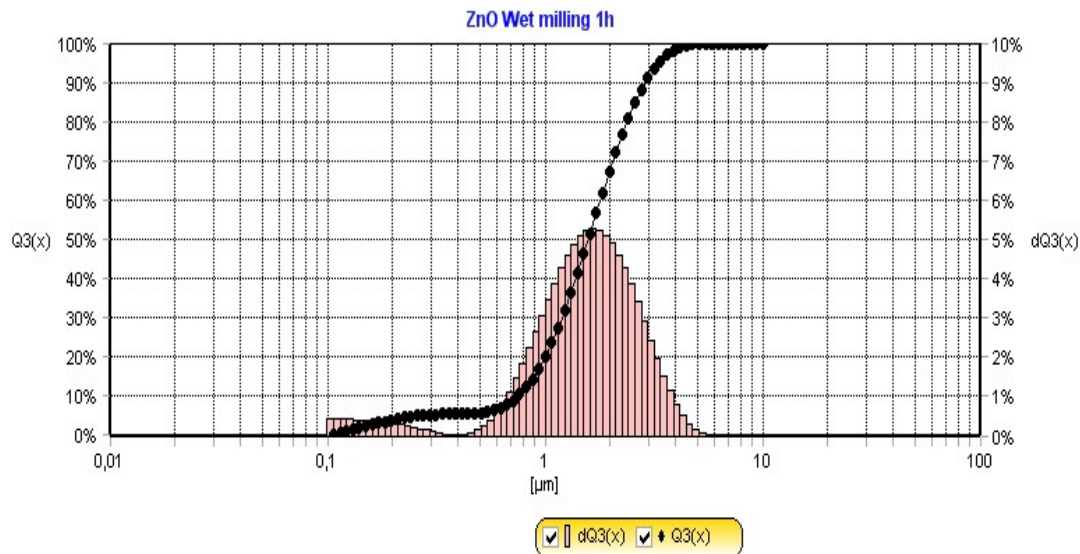
D50 = 2,8 μm – D90 = 14,1 μm

For the test the small vials (volume 15ml) were used. We put 20g of 1mm ZrO₂ beads into the vial together with 2g ZnO and 4ml of water. The Particle Size distribution was measured after 10 min. – 30 min. and 1 hour.



Already after 10 min. we saw a significant size reduction. Longer milling did not further decrease the size.

D50 = 1,5 μm – D90 = 2,8 μm



Result of the Wet Milling test:

For all three different test samples a very good and significant size reduction was observed. We therefore can recommend the usage of the special vial holder with the test vials in our pulverisette-5.

Other observations:

During the milling period a very low increase in temperature was observed! This is in contrast to the use of our normal grinding media. Especially for temperature sensitive samples like Pharmaceuticals this may be a very positive side effect.

Additional test that need to be done:

- we should equip the vial holder completely with test tubes and sample – to show the reproducibility of the result in all test tubes.
- we should measure the temperature increase during milling and compare it with conventional milling
- we should use for example an oil and water mixture with some beads to test the possibility to mix liquid-liquid or liquid-solid systems

Wolfgang Simon, 11.09.2008