



**A**s in most analytical methods, good sample preparation is a key factor in obtaining reliable, precise, and representative results using X-ray fluorescence techniques (XRF). The final sample, usually in the form of a pressed pellet or fusion bead, must contain on its surface layer all the elements that are present in the initial material and be homogeneously distributed in their true concentration levels.

In samples that exhibit both compositional and particle size inhomogeneity, the initial sample must be representative before any attempt is made to comminute the material. If the volume of the original bulk sample is large, care has to be taken to ensure that sufficient material is sampled so that all elements present will be accurately represented in the final sample that is analyzed. The amount of sample taken will depend on the maximum particle size and its homogeneity; sampling techniques used should include a reliable rotating sample divider.

### Size Reduction

The size reduction technique used will depend on the nature of the material (hard, brittle, soft, etc.). Hard, brittle materials are relatively easy to comminute using impaction techniques used in ball and vibratory disc mills. For reliable XRF analysis results, a particle size of less than 100 microns is usually required and preferably closer to 50 microns. To some extent, the fineness achieved will depend on the material and how easy it is to comminute since some plastics, for example, are very difficult to get below 200  $\mu\text{m}$ . Final sample preparation usually requires a pellet press such as a Retsch PP 25 or PP 40.

The Retsch RS 200 is an advanced design version of a vibratory disc mill or ring and puck mill. Options include a wide selection of grinding sets in different materials including stainless steel, zirconia, agate and tungsten carbide and can accommodate samples sizes up to 250 cc.

Since sample throughput is critical, these units must be able to process samples quickly yet grind them to the correct size and do so precisely and repeatedly each and every time. Therefore, the drive kinetics of the mill are critical if the desired results are to be achieved.

## Sample Preparation of Solid Materials for X-Ray Fluorescence Analysis

Retsch, Inc.

During the grinding process, the grinding set (jar, ring, and puck) complete with sample is subjected to extreme circular and horizontal vibrational forces. The key design feature of any mill is that these forces must be controlled and directed in a single plane, otherwise the jar will gyrate and tumble causing unreliable grinding results.

Two demanding tests were used in order to illustrate the superior design and effectiveness of the RS 200. The material chosen was cement clinker of initial size 0.85–3.15 mm with initial sample dividing using a Retsch PT 100. In the first test, the mills were run for only 15 s and the particle size was evaluated using laser diffraction. After this very short time, the RS 200 produced results that were up to 62% finer than existing mills. In the second test, the same mills were run for 60 seconds and the RS 200 not only produces the finest grind, but also produces a standard deviation that is at least half produced by other mills.



Figure 1: RS 200 Vibratory Disc Mill.

Table 1: RS 200 grinding results

Mill	Grinding results $d_{90}$ [ $\mu\text{m}$ ]				Mean Value	Standard deviation	
	1	2	3	4		[ $\mu\text{m}$ ]	[%]
RS 200	26.3	25.7	27.8	27.2	26.7	0.9	3.4
Comparative mill 1	25.5	28.8	25.5	28.8	27.0	1.9	7.1
Comparative mill 2	33.4	33.2	33.4	38.5	34.5	2.6	7.4

**Retsch, Inc.**  
 74 Walker Lane  
 Tel. (866) 473-8724,  
 Fax (267) 757-0351  
 info@retsch-us.com, www.retsch.com

For More Information Circle 406