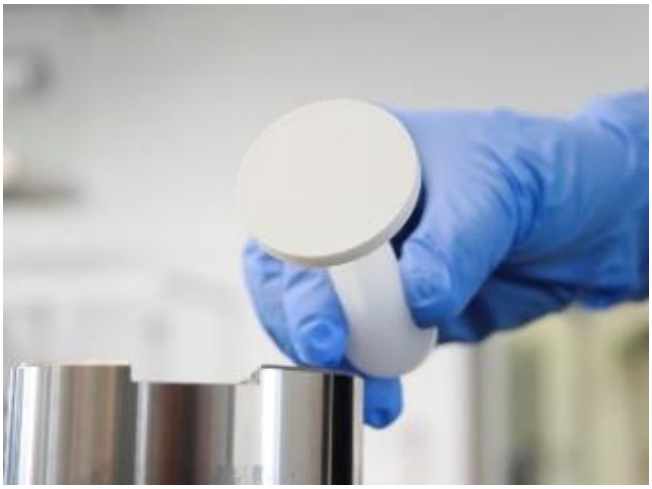
Sample Preparation: Making Pressed Pellets



1.) Introduction

Pressing a sample leads to a pressed pellet with a defined density thus ensuring reproducible sample preparation.

For X-ray fluorescence analysis, it is important that the pellet is also mechanically stable because when being introduced into the analytical instrument, the sample is subjected to evacuation and then aeration after the measurement. High stability is required so that the sample does not break during these procedures. A few materials display this stability which can be increased by pressing in aluminum cups or rings. However, mixing with a binder is obligatory for most materials.

Using a cement sample as an example, this Whitepaper presents the steps for creating the perfect pressed pellet and provides important tips for sample preparation.

2.) Making a Pellet Using a Cement Sample as an Example

Example: Preparation instructions for a 32 mm pressed pellet:

Weigh
4.00 g Sample
1.00 g CEREOX®
into a mixing cup, add 1 mixing ball and mix for 2 minutes in the MUK mixer. Then place a pellet film on the press plate, remove the mixing ball and press.

Sample Preparation: Making Pressed Pellets

2. 1) Selecting the Press

Comparison of press models:

	PR-15	PR-25N	PR-25A	PR-40
max. pressure	15 t	25 t	25 t	40 t
handling	manual	manual	electrical	electrical
die	extra	extra	extra	integrated
stroke	16 mm	16 mm	30 mm	60 mm
pressed pellet 32mm	yes	yes	yes	yes
pressed pellet 40mm	yes	yes	yes	yes
pressed pellet in ring	yes	yes	yes	yes
other diameters	yes	yes	yes	yes
different diameters	yes	yes	yes	yes
programable	-	-	-	yes
programs	-	-	-	10

Figure 1 Comparison of different presses

The 15 tons of the PR-15 are completely sufficient for making a pressed pellet with a 32 mm diameter. As described above, it is most important that the production of pressed pellets is reproducible. VANEOX presses ensure that each powder is pressed with exactly the same pressure; giving the pellets a defined sample density.

Because pressure is equal to force (tons) per area, at least 20 tons, e.g., from the PR-25A, PR-25N or PR-40Da, should be used to achieve comparable densities for larger diameters, such as 40 mm.

The PR-25A differs from the PR-25N in that the stroke of the cylinder is larger. This is required when samples have a very large volume and a very small mass so that pressing can be performed in one step. Otherwise, several steps are necessary.

A problem occurs when the pressing procedure is conducted too quickly, as trapped air causes parts of the pellet to flake later. Too much force can also lead to the pressed pellet remaining stuck to the pressing plate; this can only be helped by reducing the force or using pellet films.

Sample Preparation: Making Pressed Pellets

The programable 40-ton press, PR-40, overcomes these problems by slowly building up the pressure in up to three steps, whereby it is also possible to specify a waiting time after each pressing step.

The PR-40's integrated pressing die considerably simplifies cleaning, thus leading to higher throughput. The throughput can be further increased by using rings because the last step of pressing out of the pellet is eliminated.

However, for an increase in pressure it is necessary to take the load bearing capacity of the pressing die into account:

2.2) Selecting the Pressing die

Load limits for pressing dies without rings

Diameter	Maximum pressing force
ab 40 mm	40 t
35 mm – 39 mm	30 t
32 mm – 34 mm	25 t
28 mm – 31 mm	18 t
15 mm – 27 mm	5.0 t
10 mm – 14 mm	2.5 t
8 mm	1.5 t

Load limits for pressing dies with rings

With ring (outer/inner diameter in mm)	Maximum pressing force
PR-CD-6032R (40/32)	15 t
PR-CD-6016R (20/16)	5.0 t
PR-40-CD35R_b (40/35)	15 t
PR-40-CD35R (51,5/35)	30 t
PR-40-CD32R (40/32)	15 t

Sample Preparation: Making Pressed Pellets

2.3) Grinding the Sample

A grain size of less than 100 µm is recommended for XRF analysis of pressed pellets. The sample can be ground in different ways to achieve this grain size. The following lists show the different principles:

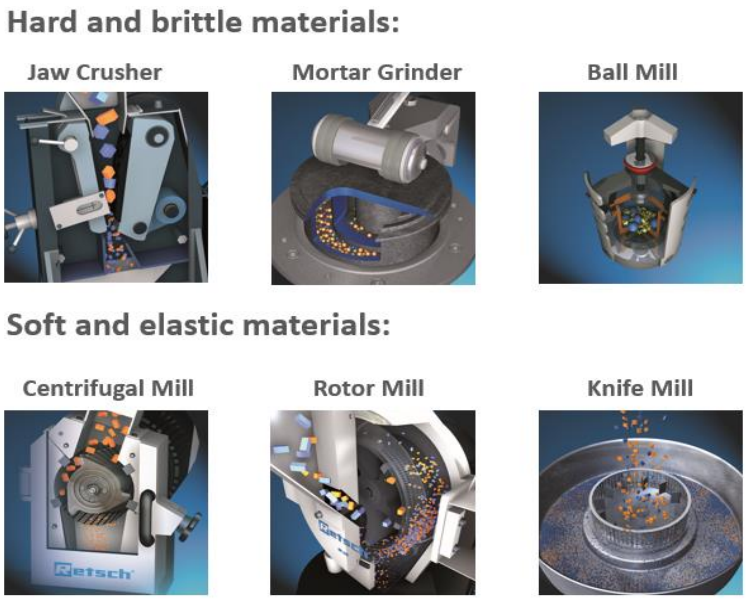


Figure 2 Principle: Grinding a sample

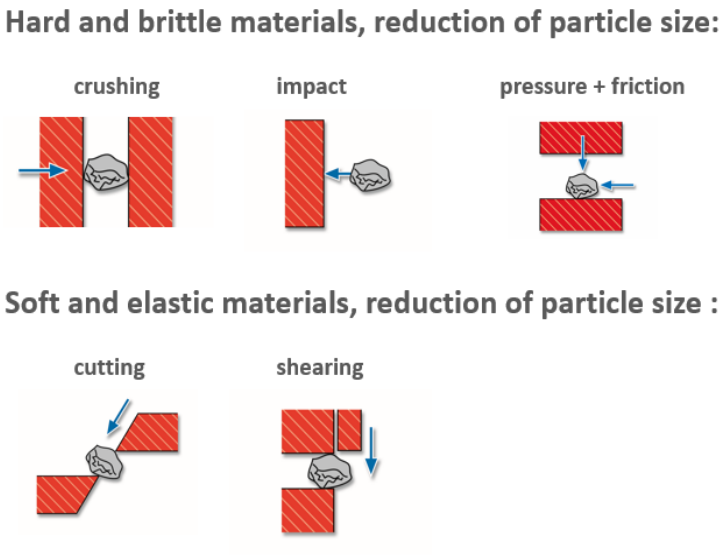


Figure 3 Principle: Grinding a sample

Sample Preparation: Making Pressed Pellets

2.4) Weighing in the Sample

When the sample has been ground, the necessary amount is weighed.

Figure 4: Weighing the sample

2.5) Binding Material: Overview of the binding materials for making pressed pellets, their suitability as grinding aids and their properties

Binder	Grinding aid	Properties
Cereox (Wax)	No	Excellent binder without impurities
Celleox (Cellulose)	Yes	Good binder, absorption agent for liquid waste
(Boric acid)	Yes	No binder, stabilizes the sample if used as backing (used for small quantity of sample), classified as toxic!
Boreox	Yes	Excellent binder, stabilizes the sample if used as backing (used for small quantity of sample), non toxic replacement for boric acid.
Boreox Tablets	Yes	Like Boreox, used as grinding aid in automatic dispensing

Figure 5: Binding materials

Sample Preparation: Making Pressed Pellets

2.6) Effectively Mixing the Sample with Binder

Figure 6 MU-K-Mixer

The binder must be homogeneously mixed with the sample to ensure a mechanically stable pressed pellet. Manually, this can be performed in a mortar, for example, by grinding the sample and the binder with the pestle for a longer period of time.

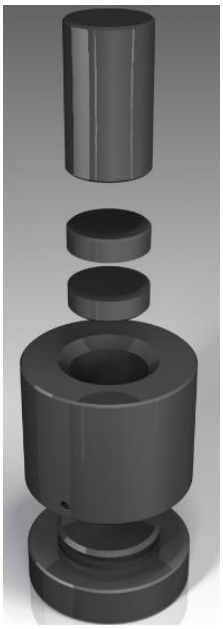
More effective and timesaving is the use of an electric mixer such as the MU-K-Mixer. The sample is homogenously mixed with the binder and a mixing ball in a disposable cup within 1-2 minutes. **It is absolutely necessary to remove the mixing ball from the mixture before pressing!**

This guarantees that the binding material is evenly distributed throughout the entire sample and, thus, that during pressing, the pressed pellet is stable in all areas.

Sample Preparation: Making Pressed Pellets

2.7) Assembly of the Pressing Die

The pressing die for the PR-15t, PR-25N and PR-25A press is assembled as follows:



First, the press housing is placed onto the base plate.

Then the first pressing plate is placed into the housing, the sample material introduced and then the second pressing plate is placed on top. Make sure that the polished sides face the sample. To prevent contamination, both pressing plates should be fit with pellet films (TF-PF-32500 for 32 mm or TF-PF-40500 for 40 mm).

Finally, the pestle is placed into the die and the entire pressing die is placed into the press. The press spindle is lowered until it touches the pestle and can no longer be lowered by hand. The pressing process can be started.

Figure 7: Assembly of the pressing die

When the pressing process is finished, remove the die from the press, turn it over and set it down. Remove the bottom plate and **carefully** press down on the pressing die housing with the balls of the hands. **Caution: Danger of pinching!**

It is also possible to place the pressing die upside down into the press, remove the bottom plate and replace it with the WZ-0024.

Figure 8: WZ-0001

Then carefully lower the spindle by hand until the pressed pellet is pressed out of the die.

Sample Preparation: Making Pressed Pellets

2.8) Using Accessories: Pellet Films, Alu-Cups and Pressing Rings

Our pressing films (pellet films) prevent contamination and sticking of pellets onto the pressing die.

The aluminum cups are ideal for unstable samples and for the labelling of samples.

The rings for pressed pellets are available in varying diameters and for different presses. They are suitable for the stabilization of samples and are used for automatic and manual sample preparation.

Figure 9: Pellet film

Figure 10: Alu-Cups

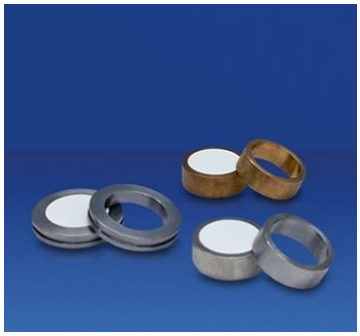


Figure 11: Pressing rings

2.9) Pressing Procedure

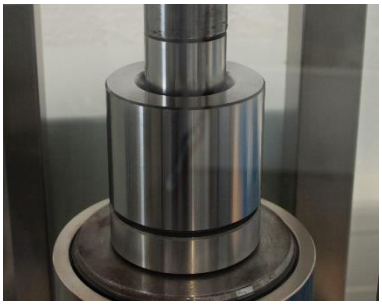


Figure 12: Pressing procedure

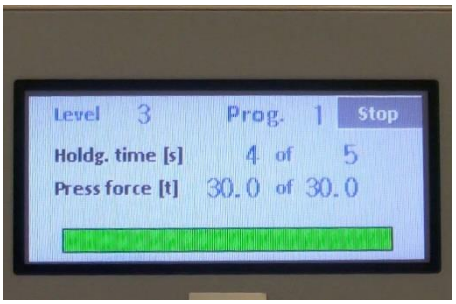


Figure 13: Software documents pressing procedure

Pressure is built up during pressing. This occurs either manually using a lever or automatically until the required pressure is reached.

Sample Preparation: Making Pressed Pellets

2.10) Removing the Sample

We recommend the WZ-0004a suction tool to easily and cleanly remove the sample from the pressing die.

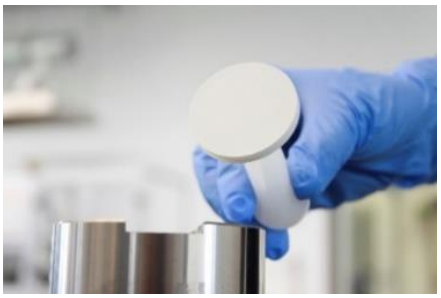


Figure 14: WZ-0004a suction tool

2.11) Analytical Results

The following table shows, as an example, the results for multiple preparations of a cement sample with binder.

Table: Preparation of the cement sample CEM V02 as a pressed pellet with CEREOX® in a ratio of 4:1.

Mass%	Na	Mg	Al	Si	P	S	Cl	K	Ca
Prep. #1	**0.206**	**2.60**	**3.70**	**12.38**	**0.040**	**1.35**	**0.045**	**0.79**	**39.24**
Prep. #2	**0.212**	**2.63**	**3.74**	**12.44**	**0.041**	**1.35**	**0.044**	**0.76**	**39.09**
Prep. #3	**0.221**	**2.66**	**3.78**	**12.47**	**0.042**	**1.36**	**0.044**	**0.75**	**38.92**
Prep. #4	**0.206**	**2.61**	**3.71**	**12.39**	**0.042**	**1.34**	**0.047**	**0.76**	**39.22**
Average	**0.211**	**2.63**	**3.73**	**12.42**	**0.041**	**1.35**	**0.045**	**0.76**	**39.12**
Std. dev.	0.007	0.03	0.04	0.04	0.001	0.01	0.001	0.01	0.15

Mass%	Ti	V	Cr	Mn	Fe	Zn	Sr	Ba
Prep. #1	**0.23**	**0.0091**	**0.0039**	**0.139**	**1.26**	**0.0123**	**0.069**	**0.040**
Prep. #2	**0.23**	**0.0093**	**0.0038**	**0.139**	**1.25**	**0.0128**	**0.067**	**0.045**
Prep. #3	**0.24**	**0.0098**	**0.0031**	**0.137**	**1.27**	**0.0133**	**0.067**	**0.045**
Prep. #4	**0.24**	**0.0099**	**0.0038**	**0.138**	**1.28**	**0.0132**	**0.069**	**0.045**
Average	**0.23**	**0.0095**	**0.0037**	**0.138**	**1.27**	**0.0129**	**0.068**	**0.044**
Std. dev.	0.01	0.0004	0.0004	0.001	0.01	0.0005	0.001	0.002

Sample Preparation: Making Pressed Pellets

3.) Special Applications:

3.1) Pressed Pellets for Small Quantities (BOREOX Backing)

This is suitable for small quantities (approx. 0.5 g sample or less available) when a normal pressed pellet is not possible.

The pressing die is assembled whereby the lower pressing plate is placed into it with pellet film. Use the filling tool (PR-CD-BA32/ PR-CD-BA40) to introduce the sample. Distribute the sample evenly with a careful twisting motion of the pestle. The aim is to achieve a flat and uniform layer of material. Then fill in 5 to 7 g BOREOX, remove the filling tool, put in the upper pressing plate and press.

A stable pressed pellet with a thin, but undiluted sample layer results and can be used for XRF like a normal pressed pellet.

Figure 15: Filling tool PR-CD BA32/40

Figure 16: Pressed pellet with a layer of sample

Figure 17: Breaking the sample shows the thin layer

3.2) Using WC Pressing Plates

When analyzing very hard samples such as quartz, pressing can lead to contamination of the sample by the elements Fe and Cr. In addition to employing pellet films, this can be prevented by using tungsten carbide (WC) pressing plates. The table below shows the results for Cr and Fe in sand pellets prepared once with hardened steel pressing plates and once with WC pressing plates. The comparison reveals how strongly the pressed pellet is contaminated with Fe and Cr.

Sample Preparation: Making Pressed Pellets

µg/g	PP of Steel		PP of WC	
	Cr_2O_3	Fe_2O_3	Cr_2O_3	Fe_2O_3
1	33	505	0	251
2	152	1090	0	271
3	5	333	5	250
4	25	403	5	251
5	0	280	0	273
Avg.	43	522	2	259
Std. Dev.	62	328	3	12

Table: Comparison of the Cr and Fe contamination when pressing sand with pressing plates made of steel versus tungsten carbide.

4.) Additional Information:

4.1) Storage and Use of Pressing Dies

The pressing die is made of hardened stainless steel to ensure its robustness. To prevent the surface from corroding, it is important to remove all fingerprints, sample material or similar.

The pressing die must be stored in a dry environment. Any corrosion that does occur is only on the sample surface and can be easily removed by polishing. It does not constitute a quality defect.

The surface quality of the pressing plates has only a limited influence on the XRF results. As a rule, the grain size of the powder is larger than impairments to the surface quality, i.e., as long as scoring and pitting on the surface are smaller than the grain size of the powder, they have no influence on the XRF measurement. Even in cases in which the pressed pellet displays patterns or color progressions, we have not been able to establish any analytical influence.

Sample Preparation: Making Pressed Pellets

4.2) Reusing the Rings for Cost Optimization

The rings can be reused for cost optimization and to improve sustainability. To do this, place the ring into the tool provided, place the pestle on the sample and then carefully knock out the sample with a hammer.

Figure 18: Place the ring in the tool

Figure 19: Placement of the pestle

Figure 20: Knock out the sample with a hammer

5.) Summary

When making pressed pellets, selection of the tools is a deciding factor for the quality achieved. Mechanical stability is attained when a suitable binding material is used, whereby efficient mixing of the sample and binder using a mixer is crucial. A whole range of manual and automatic presses with pressing dies for various diameters is available for the pressing procedure.

Ultimately, the good reproducibility in the example presented here demonstrates what modern press technology can accomplish today.

Sample Preparation: Making Pressed Pellets

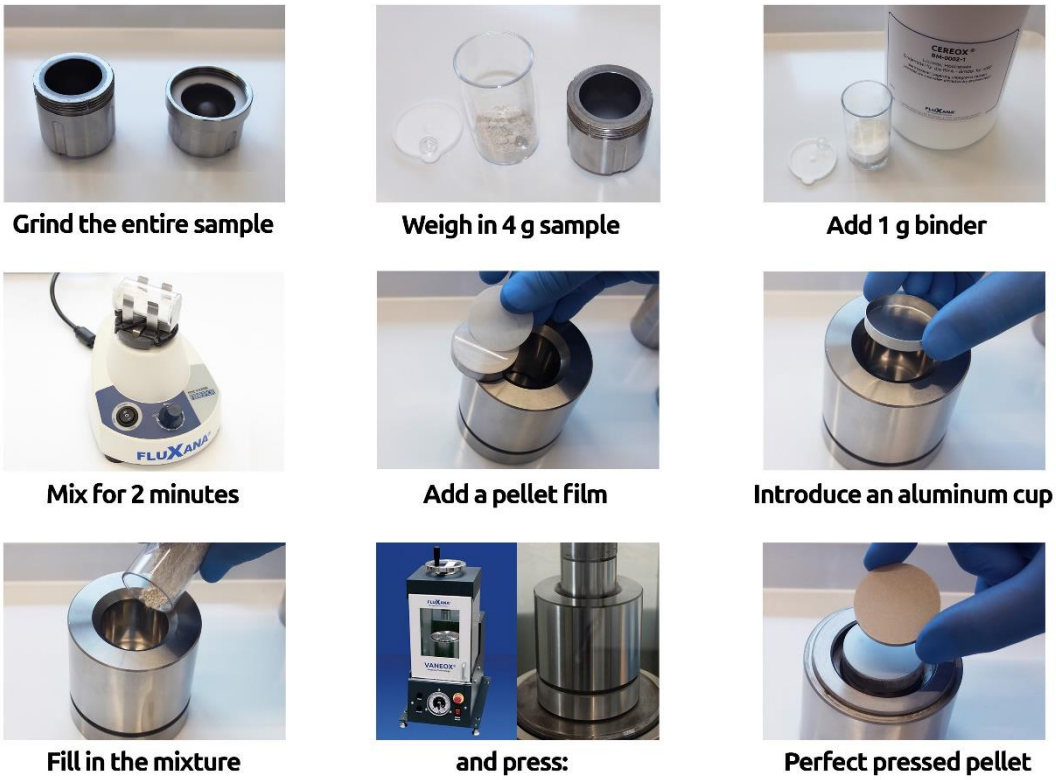


Figure 21: Summary of a pressing procedure

Literature

[1] Rainer Schramm, Röntgenfluoreszenzanalyse in der Praxis, korrigierte Auflage II, FLUXANA (2017).

[2] www.fluxana.com