

Making Pressed Pellets Using VANEON Presses

Introduction

Pressing a sample leads to a defined density in the pressed pellet; guaranteeing reproducible sample preparation.

For XRF, it is important that the pellet is mechanically stable. When being placed into the analytical instrument, the sample is subject to evacuation and then aerated after measurement. High stability is required so that the sample does not break during these procedures. Only very few materials display this stability, which can be increased by pressing in aluminum dishes. For most materials, mixture with a binding material is obligatory.

The Principle of Making Pressed Pellets



Grinding of the whole sample



weigh 4g sample



Add 1g binder



2 minutes mixing



Use pelletfilm or



Aluminium cup



Pour the mix into the die



And press:



Perfect pressed pellet

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Preparation Instructions for a, e.g., 32 mm pressed pellet

4.00 g sample

1.00 g CEREOX®

Weigh into a mixing cup, add 1 mixing ball and mix for 2 minutes in the MUK mixer.

Then place pellet film onto the press plate, pour in the mixture, remove the mixing ball and press.

Effective Mixing of the Sample with Binding Material



The binding material must be homogeneously mixed with the sample to ensure mechanical stability of the pressed pellet. This can be done manually in a mortar, for example, by grinding the sample and the binder with the pestle for a longer period of time.

More effective and time-saving is the use of an electric mixer such as, e.g., the MU-K-Mixer. The sample is homogeneously mixed with the binder and a mixing ball in a disposable cup within 1-2 minutes.

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

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Selection of the Press

Comparison of press models:

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

The 15 tons of the PR-15 are completely sufficient to make a pressed pellet with a 32 mm diameter. As described above, it is important that making of the pressed pellets is reproducible. VANEON Presses ensure that every 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 25 tons, e.g., the PR-25A, PR-25N or PR-25A, should be used for larger diameters, e.g., 40 mm in order to achieve a comparable density.

The stroke of the cylinder for the PR-25T is larger than for the PR-25N. This is required when samples have very large volumes and small masses so that the pressing can be performed in one step. Otherwise, several steps are necessary.

More power, such as 40 tons, is not a problem, but does not bring any additional benefit. A problem occurs when the pressing procedure is performed too quickly, because trapped air causes parts of the pressed pellet to flake later.

Here, our programmable PR-40 40-ton press is recommended. It slowly builds pressure in up to 3 steps, whereby a waiting time can also be specified after each pressing step.

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

Analytical Results

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

Table 1: 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

Summary

When making pressed pellets, selection of the tools is a deciding factor for the quality achieved. Mechanical stability is attained when a binding material such as CEREOX® is used. Efficient mixing of sample and binder using a mixer is crucial here. A whole range of different manual and automatic presses with pressing tools for various diameters are available for the pressing procedure.

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Finally, the good reproducibility in the example shown demonstrates what modern press technology can accomplish.

Literature

- [1] Rainer Schramm, Röntgenfluoreszenzanalyse in der Praxis, korrigierte Auflage II, FLUXANA (2017).
- [2] www.fluxana.com