

# Specac

## Application Note

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High throughput, high accuracy  
sample prep for XRF

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Rapid, high throughput pellet preparation using the Autotouch semi-automatic pellet pressing system and Apex™ Quick Release Die.

In high volume XRF laboratories, pellet preparation is essential for quick turnover of samples and ease of sample handling.

But different sample preparations also yield results that are both quantitatively and qualitatively different: how different?

Read on to discover how pressed pellet sample preparation even improves the quality of your XRF spectra and enables accurate quantification to be obtained.



*Specac's Atlas® Autotouch Press & APEX™ Quick Release Die*

### Acknowledgement

Dr Tom Knott, XRF Geochemist at the University of Leicester School of Geography, Geology and the Environment provided the XRF spectra shown here

## Factors affecting X-ray analysis

The analytical signal in XRF spectroscopy contains two major components: 1) X-rays emitted at characteristic wavelengths corresponding to electron transitions within the sample atoms; superimposed over 2) a continuous background of X-rays scattered by the outer electrons.

The characteristic X-rays generally emerge from depths on the order of 1-1000  $\mu\text{m}$  below the surface of the sample, with the depth dependent on atomic weight; lighter elements are harder to detect than heavier ones.

Particle size, mineral composition, or density can all affect the intensity of the characteristic emission peaks and increase background scattering. These effects rule out reliable quantification of sample composition (1).

Grinding sample powders to a fine particle size and pressing them into a smooth, flat pellet should reduce scattering and improve the detection of light elements.

## Experimental

Samples of Type 1 Ordinary Portland Cement (Dragon Alfa, UK) were used as-purchased.

### Loose powder preparation

10 g of sample was placed into an open-ended sample cup and covered with a 6  $\mu\text{m}$  Mylar® film window.

### Pressed pellet preparation

The sample was milled and homogenized with 20wt% of cellulose binder (SpectroBlend®) using a planetary ball mill. Pellets were pressed in aluminium sample cups at 20 tonnes using a 25 T Atlas® Autotouch Press and a 40 mm APEX™ Quick Release Die.

### Spectral acquisition

Spectra were recorded on a high-throughput wavelength dispersive XRF instrument with vacuum capability.

## Results & Discussion

### Heavy and light elements

XRF spectra for the loose powders and pressed pellets show clear differences (Figure 1). The lightest element Na is undetectable in the powders, while the signals from Mg, Al, and Si are much reduced. As expected, the signal from the heavier elements such as Fe is not affected.

The samples prepared as pellets show higher signal-to-noise and allow the lightest elements to be detected easily above the background. The detection of light elements is further improved by avoiding the use of thin film coverings, which allows measurement under a vacuum.

### Quantification of composition

The ability to clearly detect all the elements in the sample becomes critical for accurate quantification, as can be seen in Table 1. In the powder samples, underestimation of the lighter Al, Mg, and Na elements leads to overestimation of Fe and Ca in the cement. The pellet samples result in a quantification which is within the range established by averaged laboratory experiments.

## Conclusions

Loose powder sample preparation is a low-intensity method for the detection of heavy elements, however, quantification is unreliable due to the inability to detect lighter elements.

Therefore, pellet preparation is essential for accurate quantification of sample composition. It allows detection of the lightest elements and prevents underestimation of the other light elements.

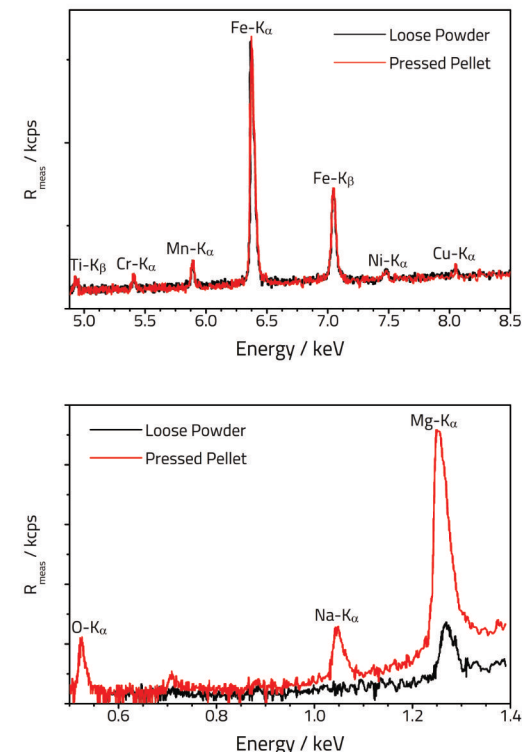


Figure 1: Spectral comparison for (A) heavy elements and (B) light elements in cement samples

Compound	Powder (%)	Pellet (%)	Expected [2]
SiO <sub>2</sub>	7.75	18.90	19.0-21.8
Al <sub>2</sub> O <sub>3</sub>	1.16	4.35	3.9-6.1
Fe <sub>2</sub> O <sub>3</sub>	5.70	2.32	2.0-3.6
MgO	0.12	1.06	0.8-4.5
CaO	78.57	65.60	61.5-65.2
Na <sub>2</sub> O	Not Detected	0.25	0.2-1.2

Table 1: Results of Portland Cement fundamental parameter quantification

## References

- [1] Jenkins, Ron. Quantitative X-Ray Spectrometry, Second Edition. New York : Marcel Dekker, Inc., 1995.
- [2] Kosmatka, Steven H., Kerkhoff, Beatrix and Panarese, William C. Design and Control of Concrete Mixtures, 14th Edition. Skokie : Portland Cement Association, 2002.