

Institut für Mineralogie und Petrographie**Contamination effects through grinding**

The following Information should be taken serious when attempting to grind rocks in the XRF sample preparation lab CO 25. For any questions, contact the lab assistant.

Note: The greatest systematic errors through contamination occur during rock crushing and grinding, not in later wet chemical digestion or analytical procedures! For powder pills, grain size must not be greater than 64 micrometers (a sieve with 250 mesh without residues). If the powders feels and looks like flower when rubbing between your fingertips (no grains felt), then the powder is right.

Oxidation effect.

When grinding rock pieces, Fe (II) -bearing minerals heat up and react with atmospheric O₂. To critically evaluate the effect on your sample, you might consider two ideas:

- treat all your samples the same way too keep the systematic error to the same level. This makes your results more reproducible, precise and comparable. For example, always use the same mortar of the disk mill for your samples, use the same amount of sample, and the same grinding time (if equality of composition allows it, see lab manual for composition-time relation).
- Separately determine FeO by spectrophotometry (contact V. Dietrich) on coarser powder. J. POTTS (1987) reports up to 20% increase of Fe₂O₃ relative to FeO in the same sample already, when grinding for less than 10 minutes!

Contamination through mortar material

Different mortar materials cause different contaminations. the best systematic study was done by THOMPSON & BANKSTON (1970). They compared different mortar and sieve materials by grinding high purity quartz. Some of their results are listed below:

grinding equipment	contaminating element	concentrations in	concentrations in
		SiO ₂ test sample	SiO ₂ test sample
		Batch 1(ppm)	Batch 2 (ppm)
Tungsten Carbide	Co	32	8.6
	Ti	124	102
	Ta	5	5
	W	n.d.	n.d.
Al-ceramic	Al	> 2000	> 2000
	Cu	2.6	2.0
	Fe	34	55
	Ga	21	54
	Li	1	1
	Ti	11	31
	Zn	2.9	<2
Agate	B	1.8	2.3
	Cu	1.1	3.9
	Si	n.d.	n.d.

Other useful references with similar data can be found in JOHNSON & MAXWELL (1981), p. 75, BENNET & REED (1971), p. 10ff, and in ZIEF & MITCHELL (1976), p.153.

In addition, especially Nd and Ta among other HFSE (high field strength elements) may show contamination by using tungsten carbide.

Cross-contamination effects

Cross contamination means: Contamination of sample B by traces of sample A, when sample B is ground after sample A, using the same container. This is probably the greatest contamination effect. To avoid or minimize this effect, consider two ideas:

- Carefully read and follow the lab manual instructions for cleaning the equipment. Rather run the mill with quartz twice than only once between samples. Note: cleaning the mortar with water, and acetone after (see manual) carefully and running it once with quartz can give better results than loosely cleaning it but running with quartz twice.
- Think of the right order of grinding your samples. Similar compositions give less contamination.

There is actually a *reason* why certain mortars are assigned to certain rock chemistries!

Literature

POTTS, J. (1987). *A handbook of silicate rock analysis*. Blackie London, 622pp.

JOHNSON, W. M., & MAXWELL, J. A. (1981). *Rock and Mineral analysis*. Chemical analysis series Vol. 47. J.Wiley, New York, 489 pp. (2nd ed.)

ZIEF, M., & MITCHELL, J. W. (1976). *Contamination control in trace element analysis*. Chemical analysis series Vol. 47. J.Wiley, New York, 262 pp.

BENNET, H., & REED, R. A. (1971). *Chemical methods of silicate analysis*. British Ceramic Research Association. Academic Press, London, 272 pp.

THOMPSON, G., & BANKSTON, D.C. (1970): Sampling contamination for grinding and sieving determined by emission spectroscopy. *Appl. Spectrosc.* **24**:210-219.

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ETH-IMP 1998**