Sampling bias in detrital zircon U-Pb analysis

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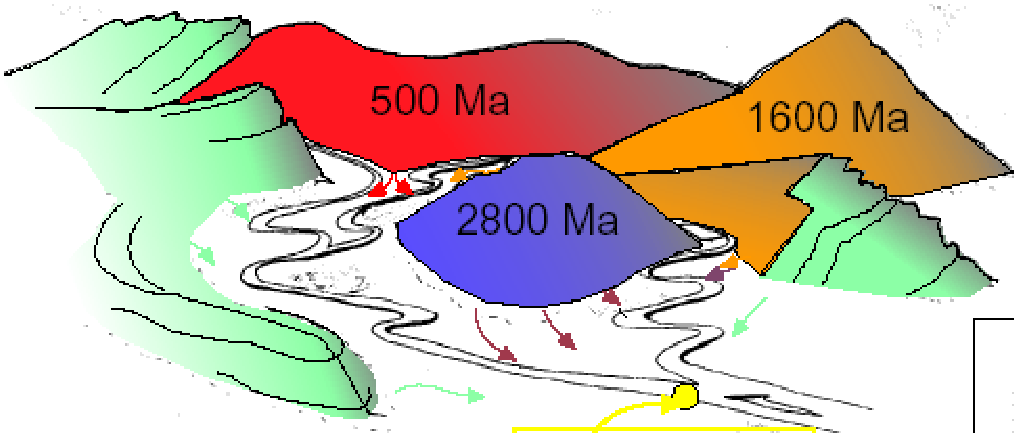
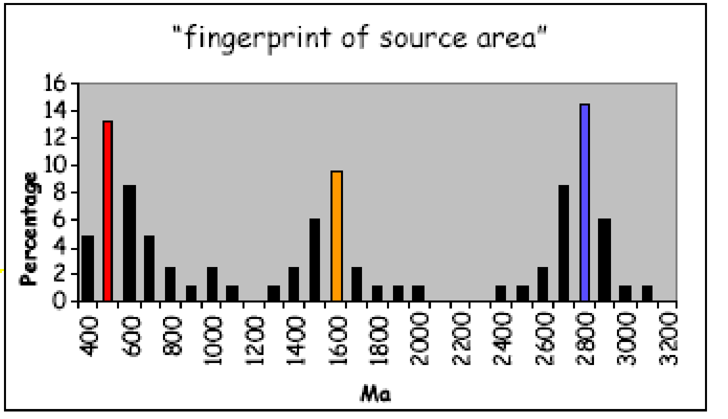
Deep Seafloor • Deep Biosphere • Deep Time • Roots of life

Detrital zircon workshop, Prague, 13.-14.8. 2011

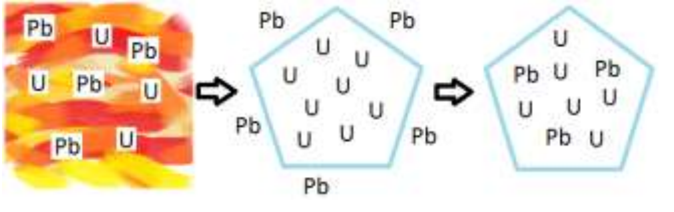
Sedimentary provenance - principles





Zircon: $ZrSiO_4$

Concept of U-Pb zircon dating



$^{235}U \rightarrow {}^{207}Pb$ 0.704*10^9 years

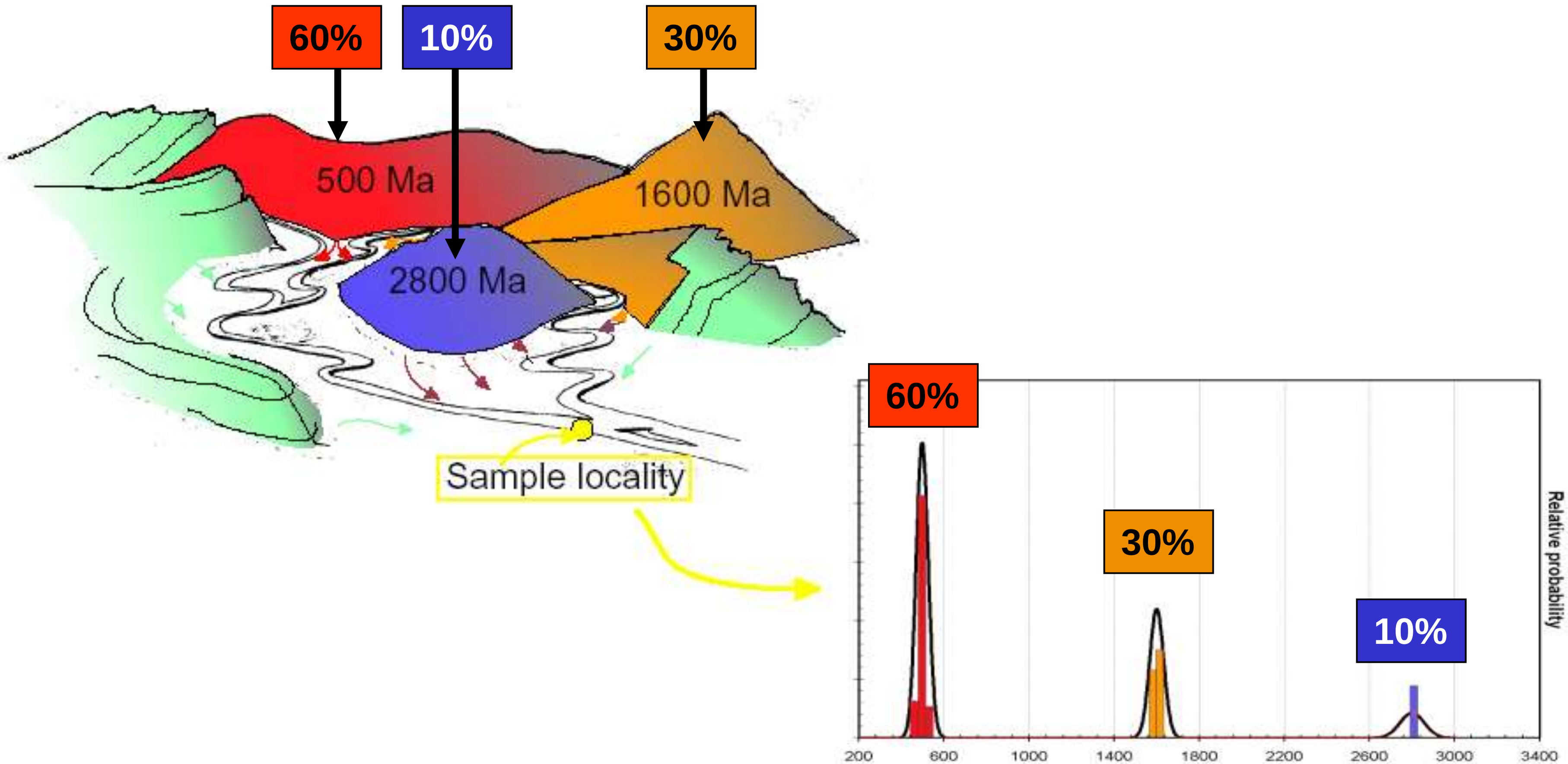
$^{238}U \rightarrow {}^{206}Pb$ 4.468*10^9 years

Quantification of sedimentary sources?

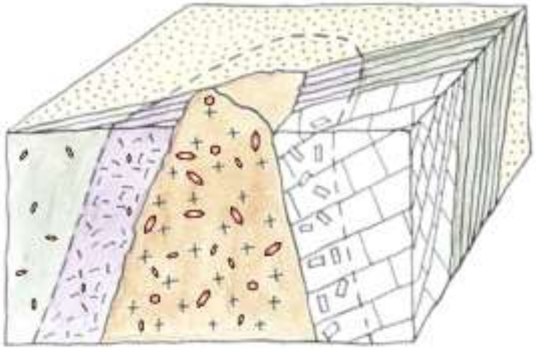
Is the age distribution proportional to the rock abundance in the source terrain?

Can we relate the age spectrum to the amount of detritus from a source?

How reproducible are the age abundances derived from a single source?

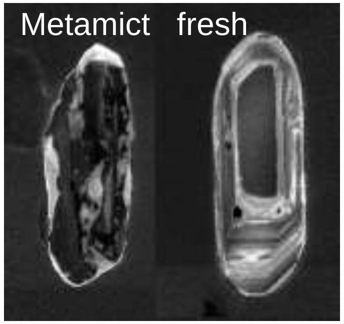


Quantification of sedimentary sources?

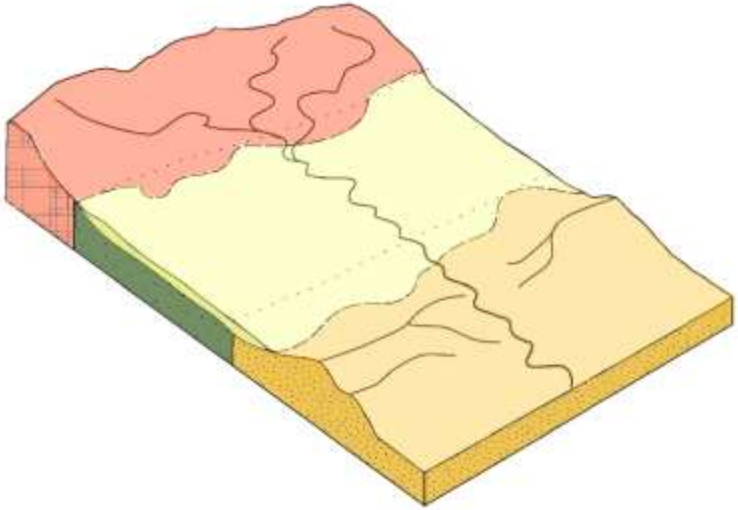


• Natural variability of zircon content in the source
(Moecher & Samson, 2006)

• Quality of zircon in the source



• Change of topography in the source area - active (erosion) or passive (deposition) behaviour of stream *(Cawood et al., 2003)*

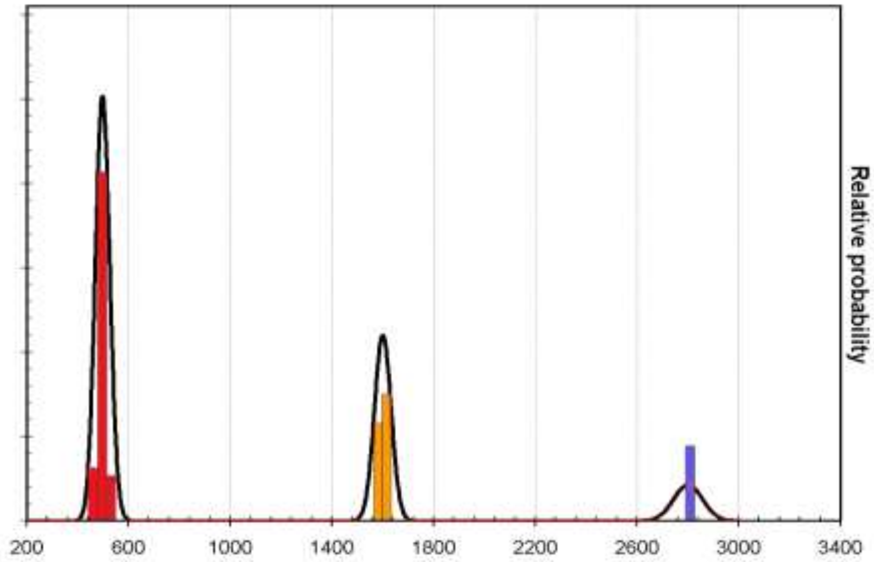


• Mixing of components, size sorting and disintegration of zircon during transport

Reproducibility of age spectra?

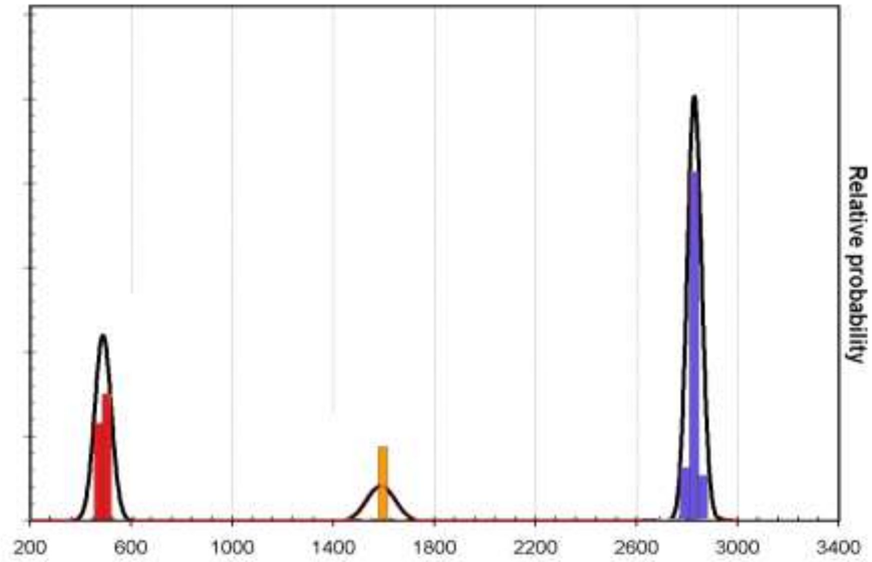
Are we able to reproduce zircon age distribution from a sample?

True age distribution in the sediment



Age (Ma)

Measured age distribution



Age (Ma)

See the results of the ILC exercise.

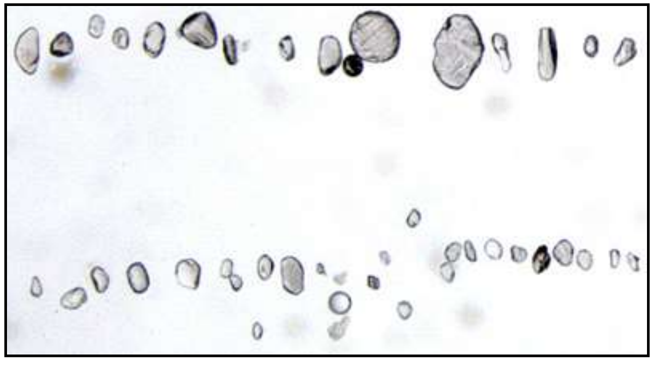
Reproducibility of age spectra?

Sample preparation may bias the measured zircon age distribution

- Sample crushing - loss of low-temperature zircon population *(Hay & Dempster, 2009)*

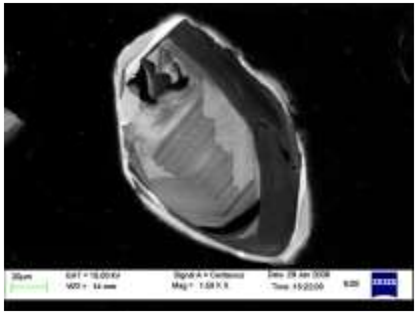
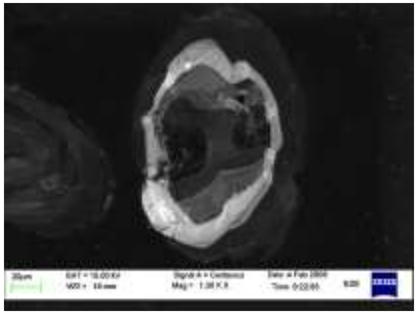
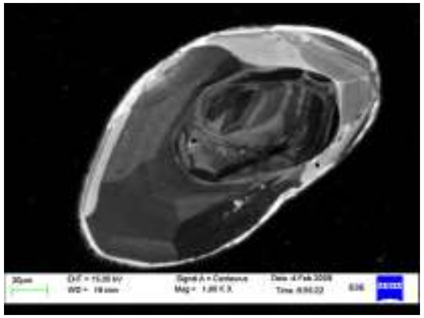
- Preference for less magnetic zircon grains during magnetic separation *(Sircombe & Stern, 2002)*

- Sieving limits the size of zircon grains that can be analyzed *(Fedo et al., 2003)*
- Preference for larger zircon grains during handpicking



Reproducibility of age spectra?

- Biases caused by more likely analysis of grain cores compared to the rims *(Moecher & Samson, 2006)*

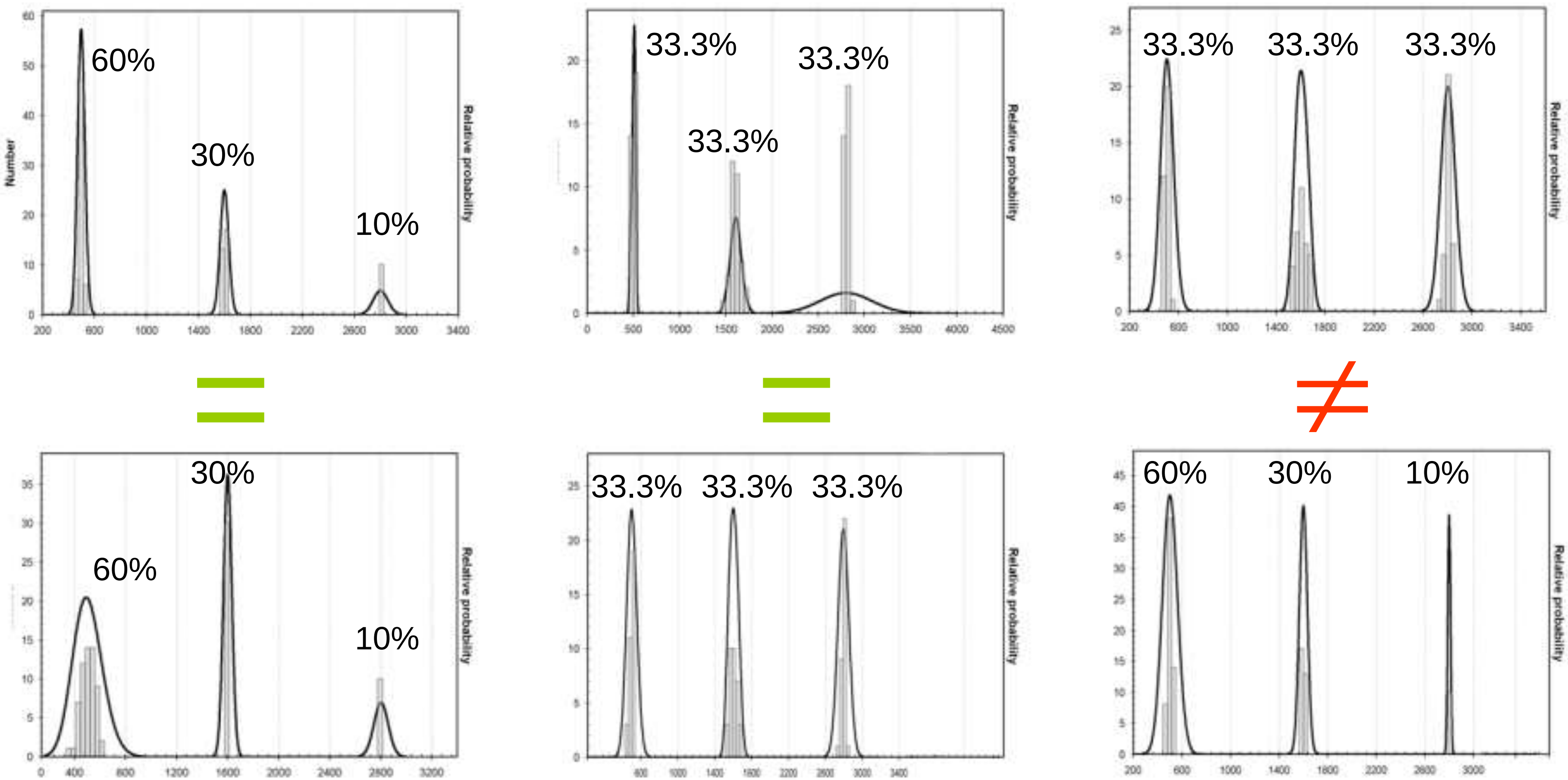




- The effects of Wilfley table and heavy liquid separation on detrital zircon age distribution - not yet studied in detail

- Number of zircon grains that needs to be analyzed in order to avoid loss of a zircon population during the analysis *(Dodson et al., 1988; Vermeesch, 2004; Andersen, 2005; Link et al., 2005)*

Data handling and comparison of age spectra

Can we use age spectra for direct comparison of samples?

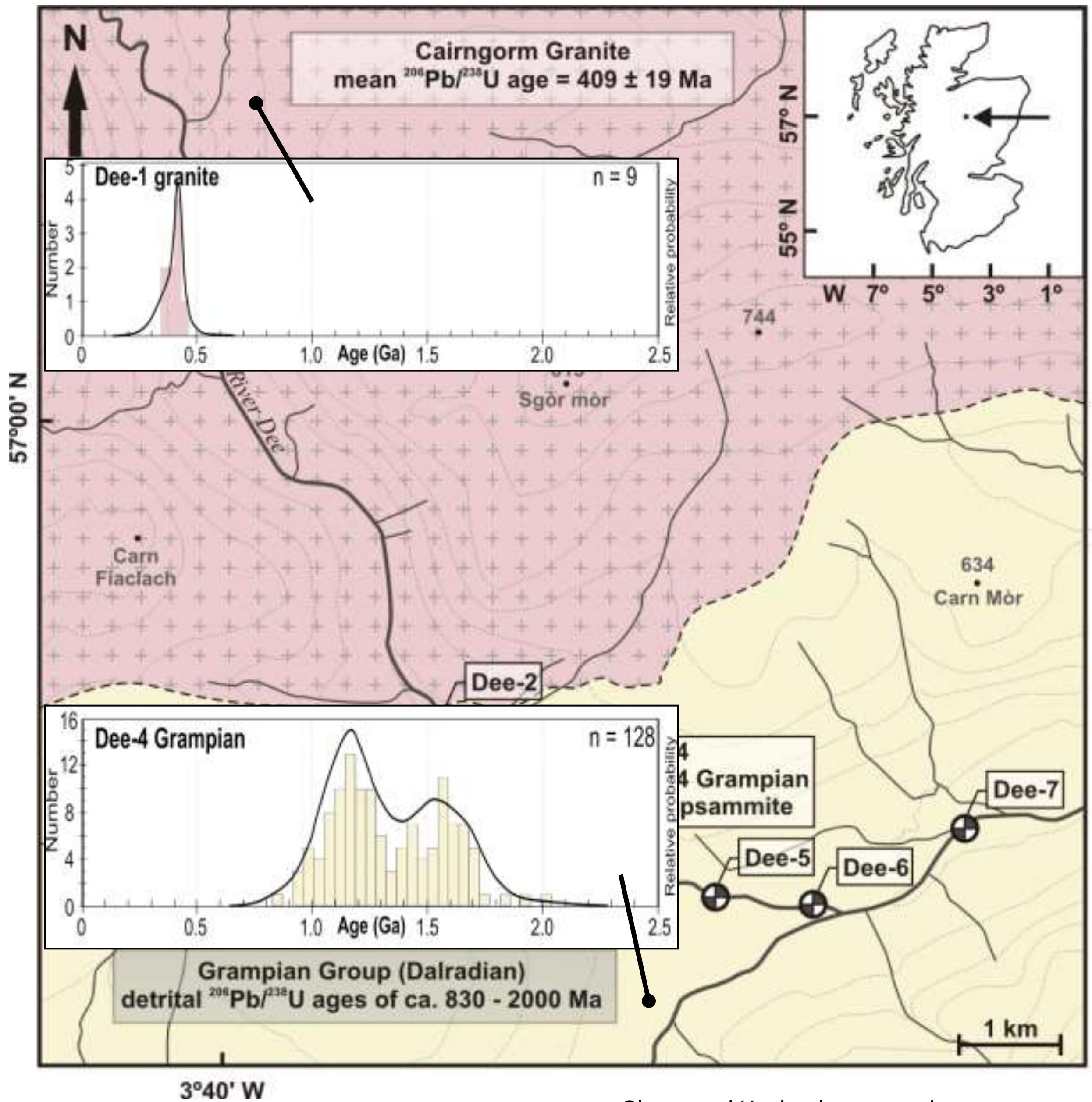


Two case studies

1) **Effects of natural zircon variability on detrital age spectra**
Active source-to-sink system, River Dee in the Cairgorm Mts, Central Scotland

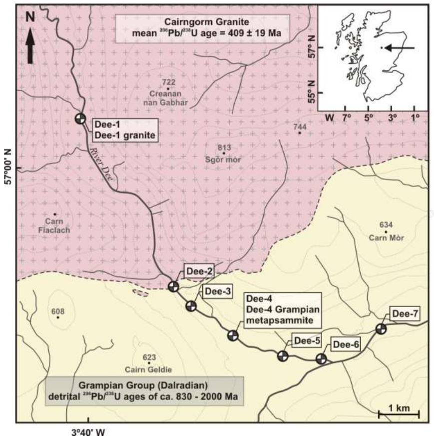
2) **Effects of mineral separation on detrital age spectra**
U-Pb analysis of "detrital" zircons from a synthetic sediment

Variability of zircon in natural rocks



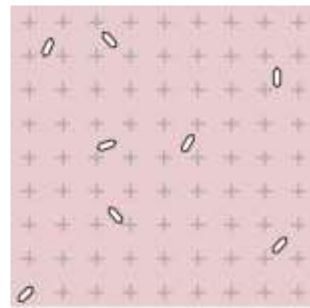
Slama and Kosler, in preparation

Variability of zircon in natural rocks



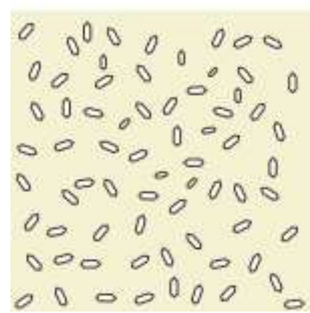
Zircon abundance

Cairngorm granite

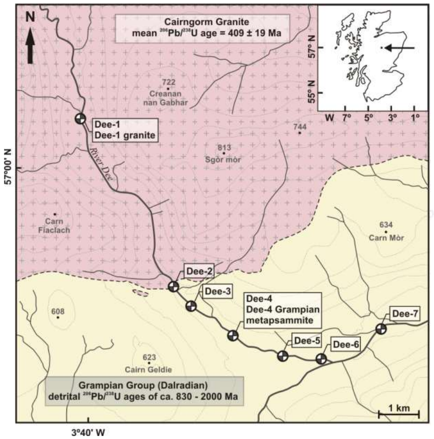


20x more

Grampian meta-psammite



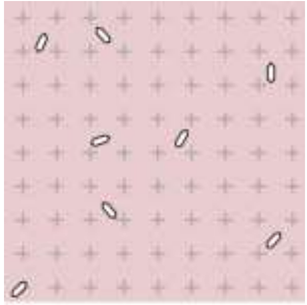
Variability of zircon in natural rocks

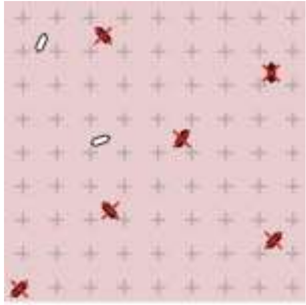


Zircon abundance | Zircon quality

3/4 metamict

Cairngorm granite

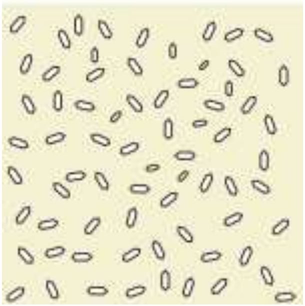


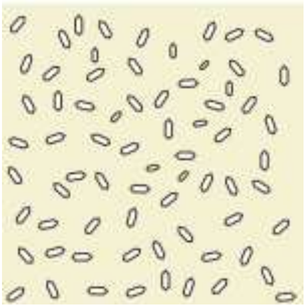


+

20x more

Grampian meta-psammite

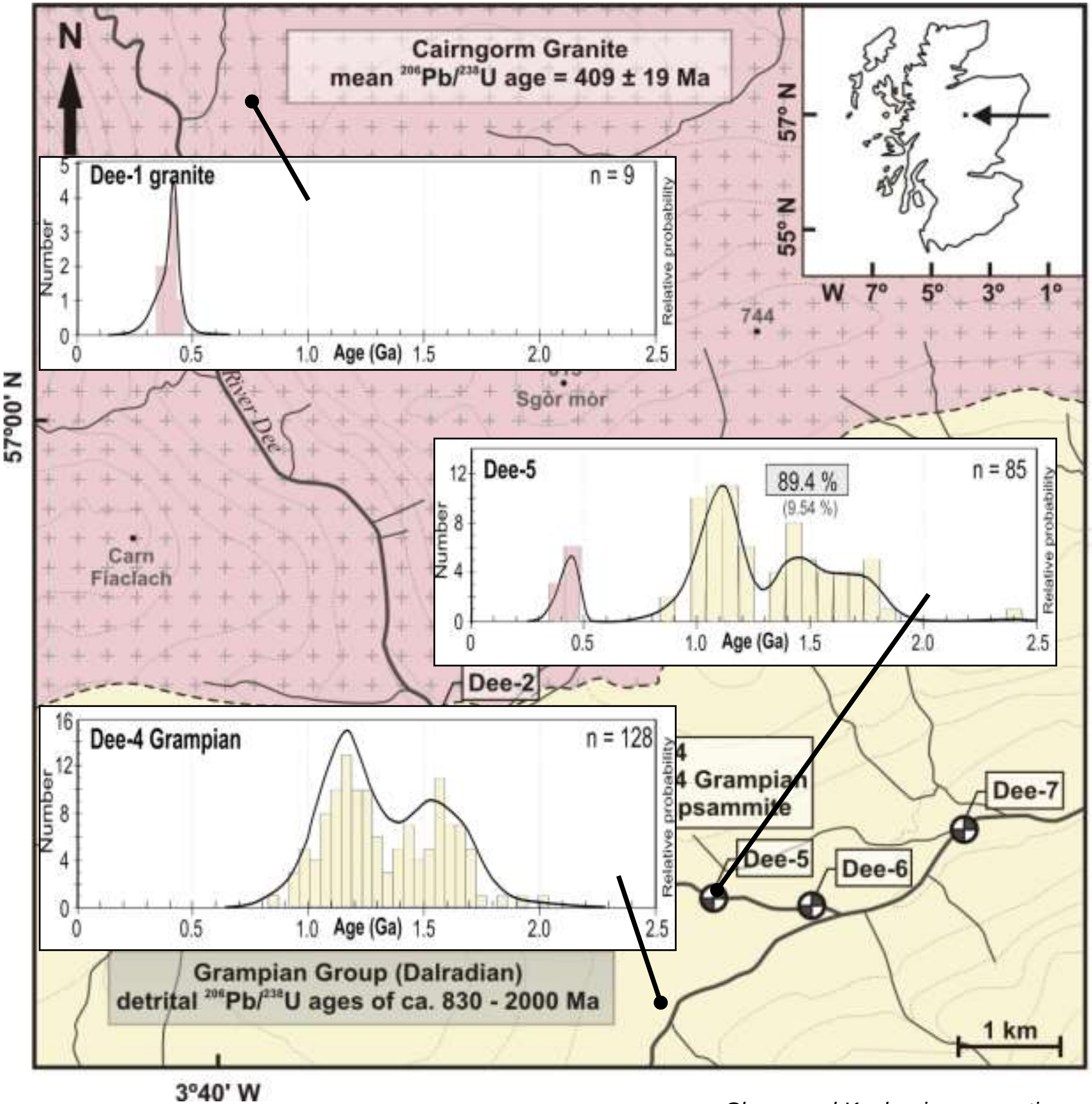




≈

Grampian metapsammite provides ca. 100 times more zircon to the sediment compared to the Cairngorm granite

Variability of zircon in natural rocks



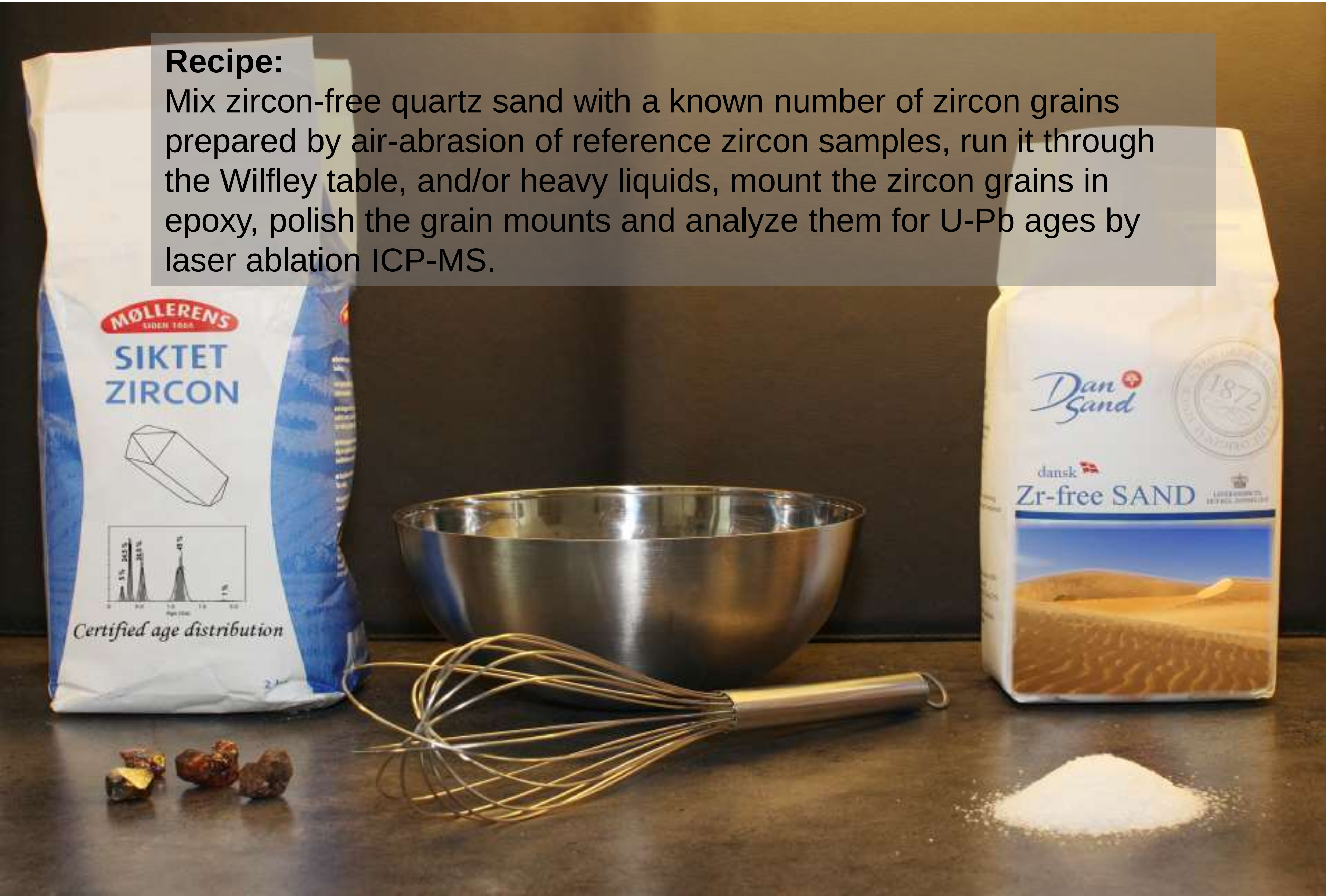
Slama and Kosler, in preparation

Ca. 90 % of detrital zircons in the sediment corresponds to only 10% contribution from the respective source rocks

Quantification of sedimentary sources is only possible for well-defined geological systems

Preparation of synthetic samples

Recipe:
Mix zircon-free quartz sand with a known number of zircon grains prepared by air-abrasion of reference zircon samples, run it through the Wilfley table, and/or heavy liquids, mount the zircon grains in epoxy, polish the grain mounts and analyze them for U-Pb ages by laser ablation ICP-MS.



Preparation of synthetic samples

- Six natural zircons (reference materials) were crushed and air-abraded to resemble detrital zircon population. The grains were sieved to >100 and <100μm size fractions.

- The abraded grains were mixed with zircon-free quartz sand as follows: samples ART-1 and ART-2 were prepared by mixing 2000 zircon grains with 500g of sand, samples ART-3 and ART-4 were prepared by mixing 400 zircon grains with 100g of sand.

- Samples ART-1 and ART-2 were separated using Wilfley table and heavy liquids (TBE and DIM) and the sample mounts were prepared in duplicate. Samples ART-3 and ART-4 were only separated in heavy liquids.

- Imaging techniques were used to measure the zircon grain recovery.

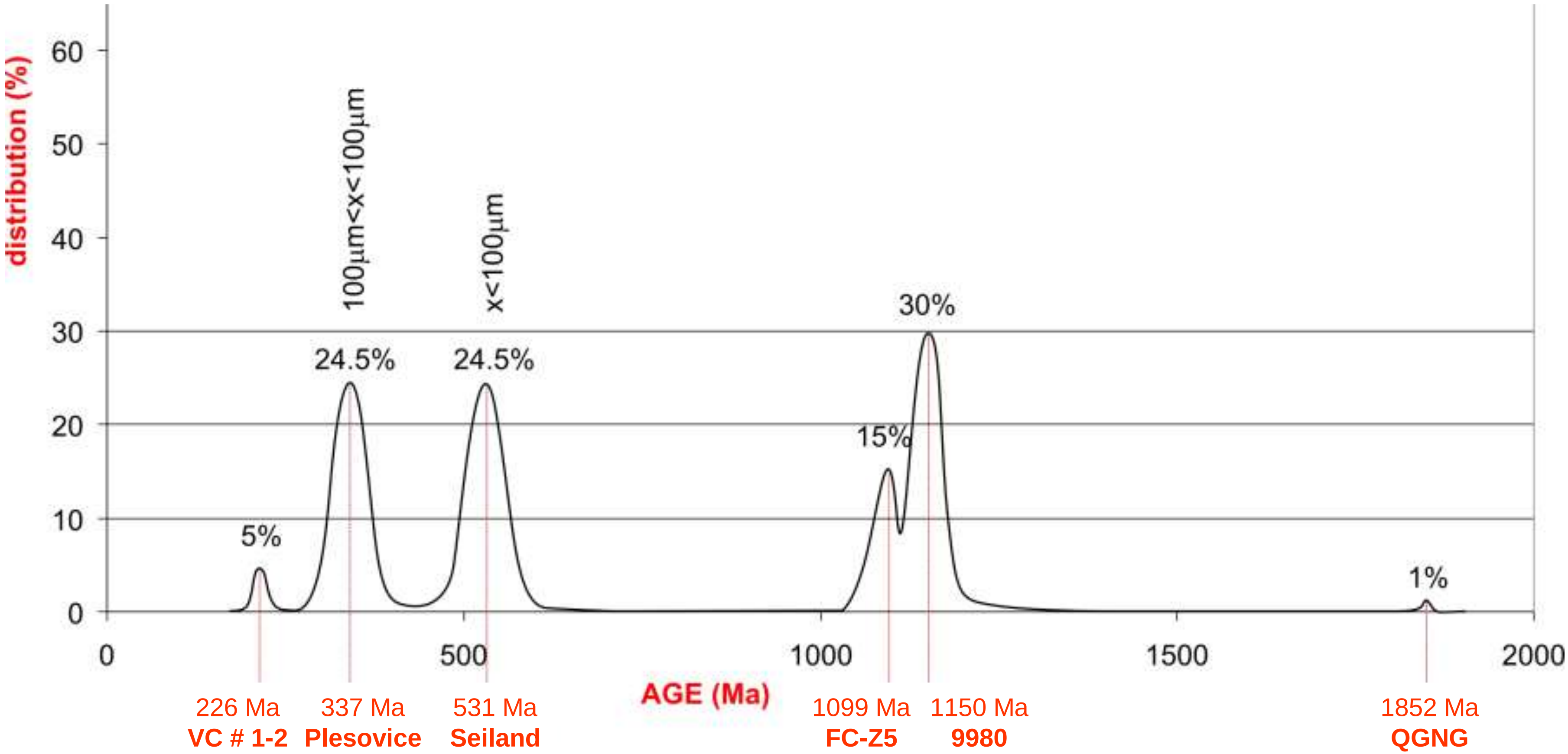
- Approximately 200 grains were hand-picked, embedded in epoxy and polished.

- More than 160 grains were analyzed in each of the six sample mounts by LA ICP-MS.

Reference zircons used in synthetic detrital samples

Sample	ART-1, ART-2		ART-3, ART-4	
Volume of sand	500 g		100g	
Total number of grains	2000		400	
Zircons proportions:	number of grains (**%** [number])		number of grains (**%** [number])	
	< 100 µm	> 100 µm	< 100 µm	> 100 µm
VC # 1-2 (226±2 Ma)	**5 %** [100]		**5 %** [20]	
	2.5 % [50]	**2.5 %** [50]	**2.5 % [10]**	**2.5 % [10]**
Plešovice (337±1 Ma)	**24.5 %** [490]		**24.5 %** [98]	
	12.25 % [245]	**12.25 %** [245]	**12.25 %** [49]	**12.25 %** [49]
Seiland (531±2 Ma)	**24.5 %** [490]		**24.5 %** [98]	
	24.5 % [490]	**0%** [0]	**24.5 %** [98]	**0%** [0]
FC-Z5 (1099.3±0.3 Ma)	**15 %** [300]		**15 %** [60]	
	7.5 % [150]	**7.5 %** [150]	**7.5 %** [30]	**7.5 %** [30]
9980 (1150±2 Ma)	**30 %** [600]		**30 %** [120]	
	15 % [300]	**15 %** [300]	**15 %** [60]	**15 %** [60]
QGNG (1852±1 Ma)	**1 %** [20]		**1 %** [4]	
	0.5 % [10]	**0.5 %** [10]	**0.5 %** [2]	**0.5 %** [2]

Age distribution in synthetic detrital samples



Evaluated parameters

- Zircon grain recovery
- Size-dependent loss
- Preference for large grains during handpicking
- Reproducibility of age spectra
- How many grains are needed
- Separation of age peaks

…

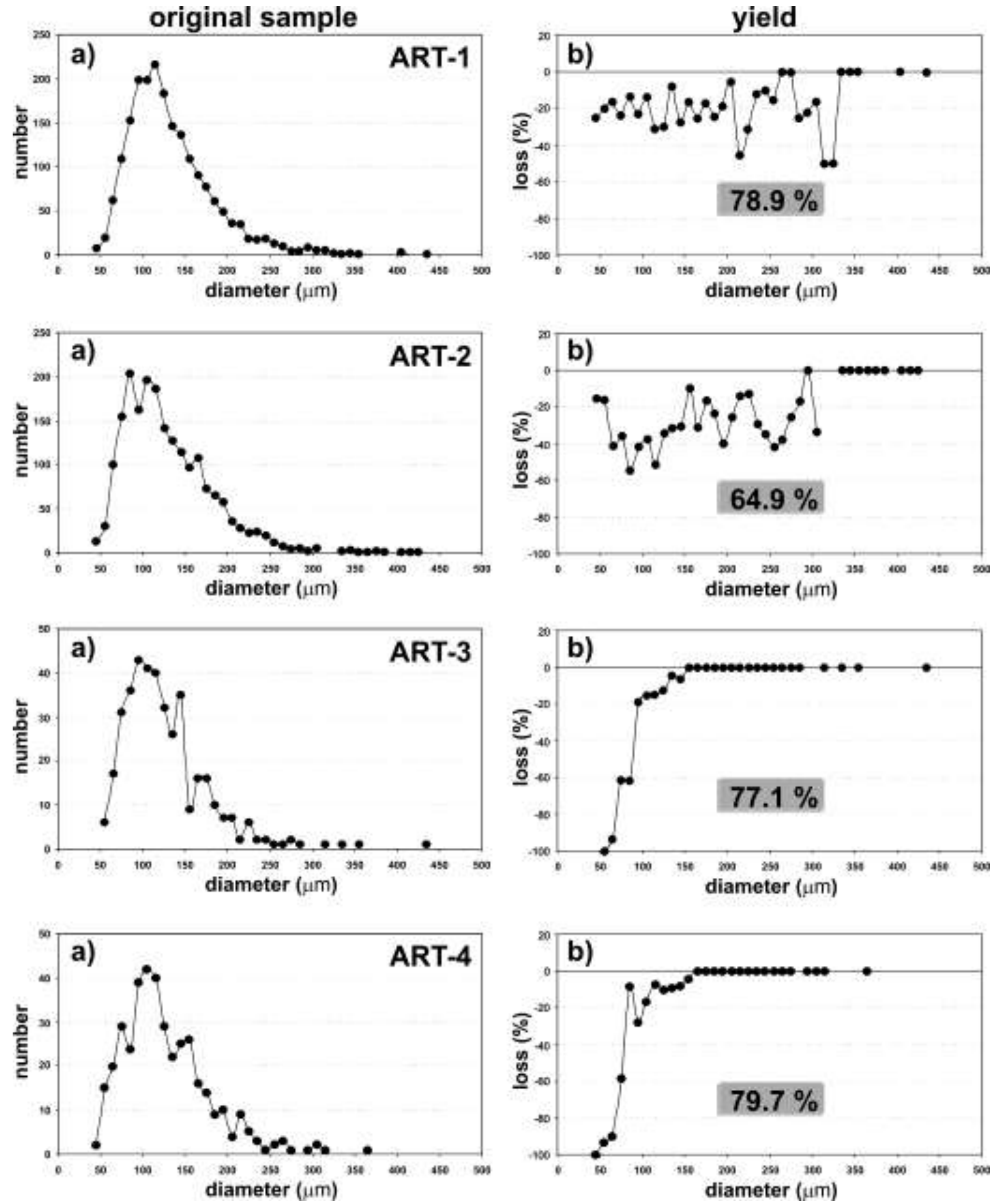
Zircon grain recovery and size-dependent loss

Grain size analysis of the original sample

- Total recovery (yield) of zircon following the separation was ≈ 79%

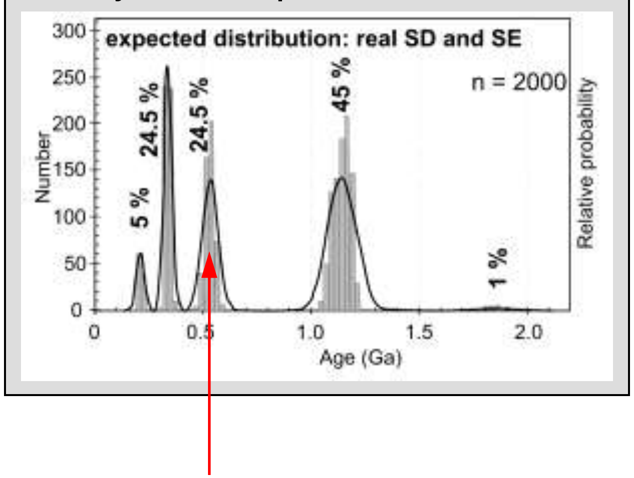
- Size-dependent loss was different for samples run through Wilfley table and samples run processed only in the heavy liquids

- This can affect zircon populations containing small grains



Size-dependent loss of zircon grains

Synthetic sample initial distribution

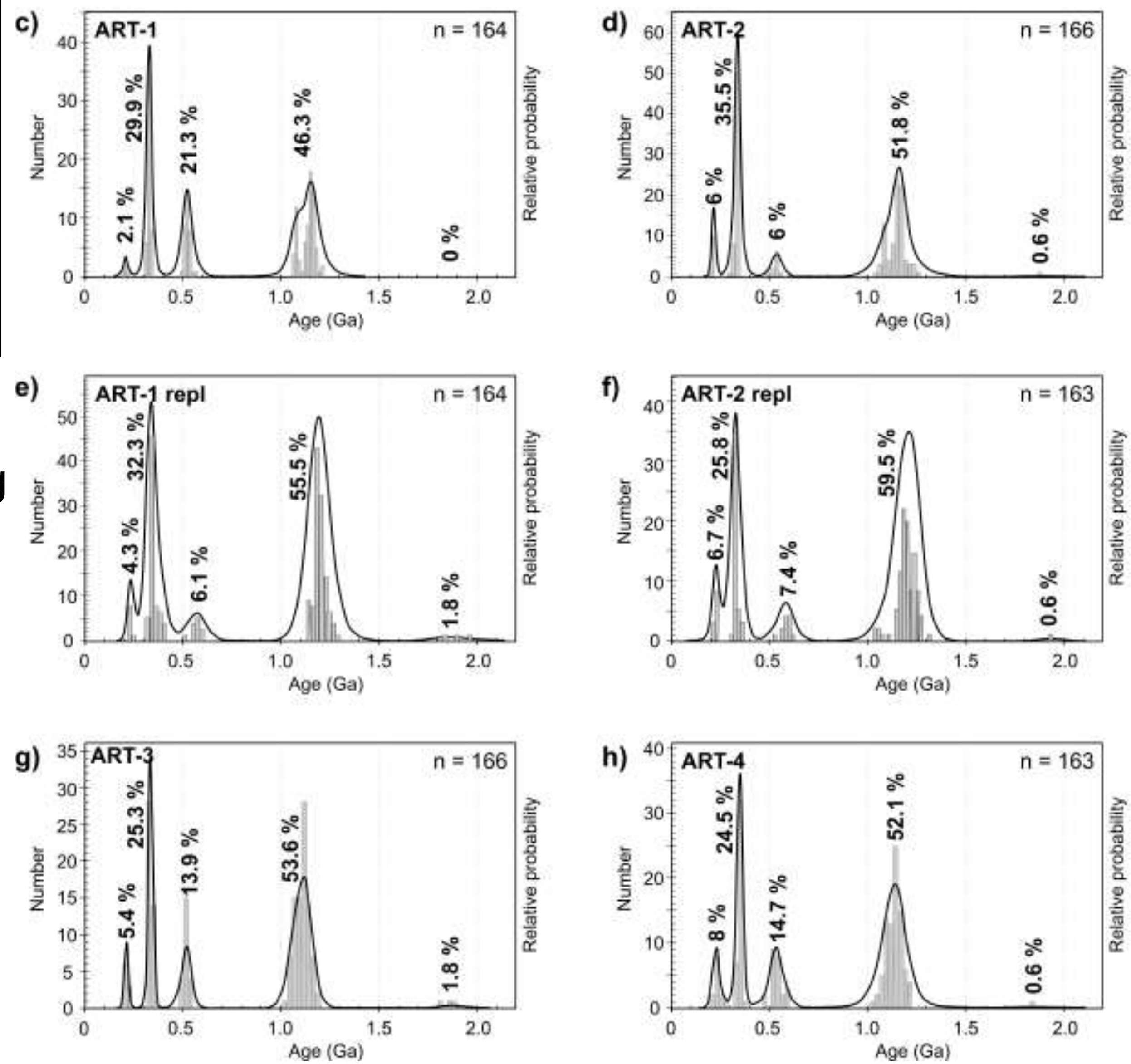


Zircon population consisting of grains smaller than 100 microns.

Measured abundance reduced by as much as 4x.

There may be additional factors, such as hand-picking, during grain mounting and analysis.

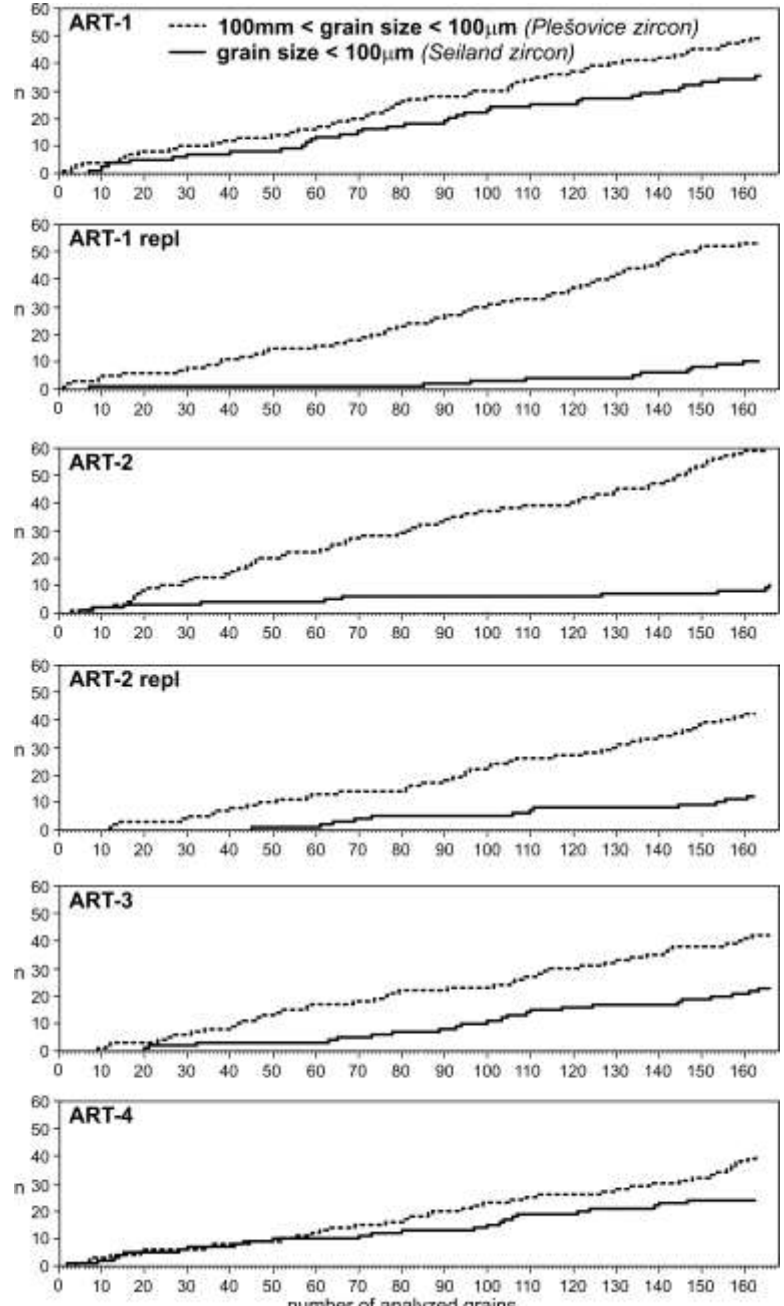
Measured distributions in synthetic samples:



Hand-picking bias

Age spectra suggest that a bias towards larger grains during hand-picking is also likely

Appearance of two equally represented age populations (24.5%) in the cumulative plots - note the deficiency of the *Seiland* zircon fraction which only contained small grains.



How many grains are needed?

How many grains need to be analysed in order to detect small population at a given level of confidence?

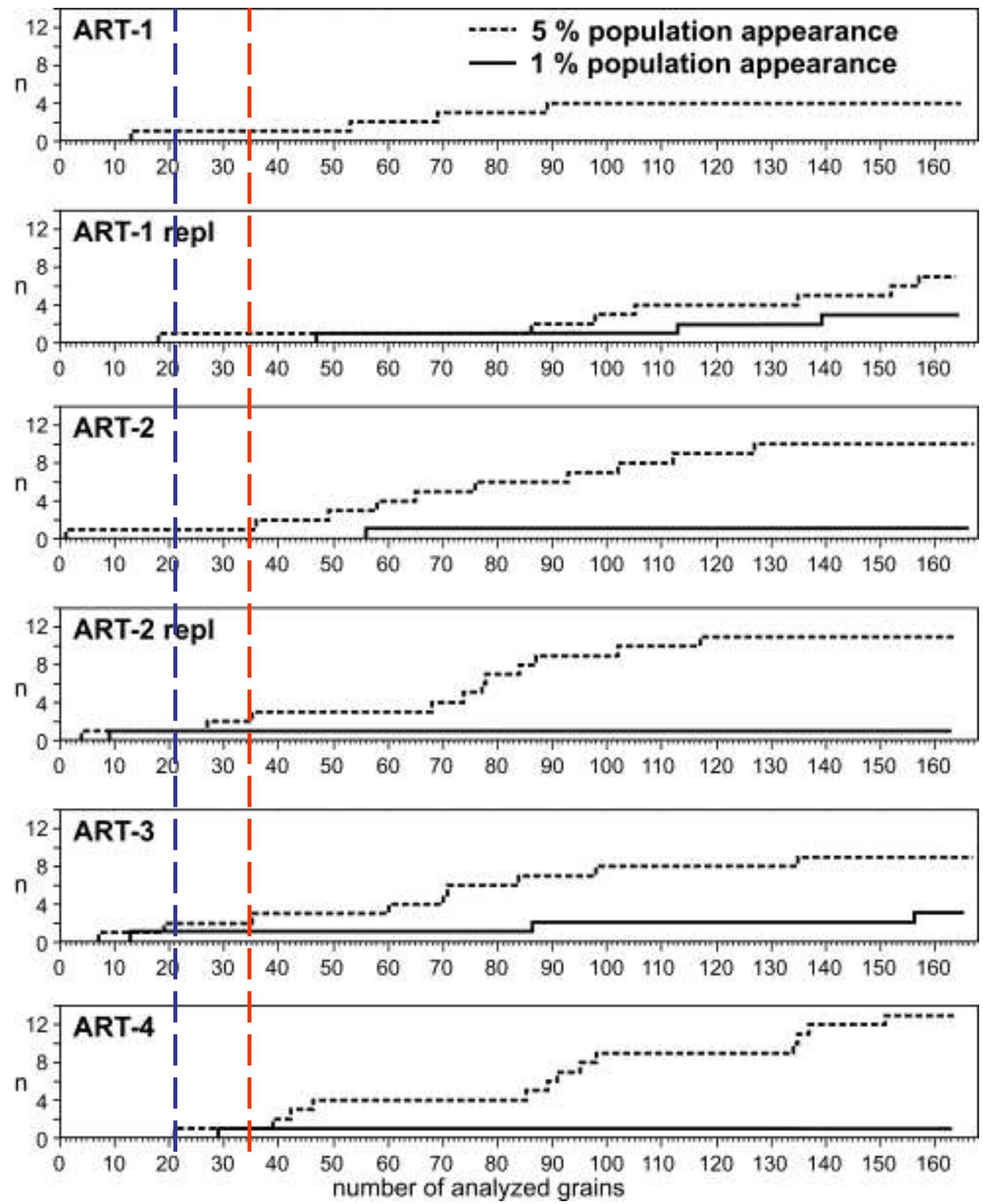
(Dodson et al., 1988):
„At least 60 grains need to be measured to be 95% confident that 5% population is detected“

(Andersen, 2005):
„random selection of 35-70 grains“

Experiment:
21 analyses was sufficient in all samples

(Andersen, 2005):
„probability of finding at least one grain of 1% population within 35 analyzed grains is 50% “

Experiment:
1 % population has been detected in 3 out of 6 (50%) samples within 35 analysis. In one sample (ART-1) the 1 % population has not been detected even after 164 analyses.



Summary

- Quality of natural zircon and its abundance in the source rocks can result in several orders of magnitude variations in sedimentary abundance. Quantification of the source terrain based on age distribution in sedimentary samples is difficult, often impossible, except for for well-defined geological systems.

- Sample preparation can (it often does) result in bias of zircon age spectra. The age spectra are usually biased towards larger zircon grains due to loss of small grains during heavy mineral separation and handpicking.

- We have only looked at few parameters that affect the age spectra, there surely are more.

Q & A

1) What are the potential biases involved in separating accessory minerals from rock?

Natural – selective weathering, depositional system, grain quality, …
Man-made – sampling, separation, analysis, data interpretation

2) What are the best procedures for making unbiased selections of zircon grains for analysis?

Ideally – do not touch the samples at all. And if you do, avoid any procedures that can fractionate the grains based on their quality. Use as few steps as possible.

3) Should the grains be selected/mounted randomly or should we use another strategy (e.g. Anderson 2005) to ensure that all age populations are mounted and analyzed?

The procedure is not random. Anderson’s approach is better suited to a non-random procedure.

4) Should all the grains be imaged prior to the LA-ICPMS/SIMS dating to reveal their internal structures?

Preferably yes.

5) For grains with multiple age zones, which zones should be analyzed – Cores? Rims? Both?

Subject to the questions we want to answer. Rims correspond to the latest history of the grains.