Helmholtz-Institut Freiberg für Ressourcentechnologie

Accuracy of ^{9}Be-data and its influence on ^{10}Be cosmogenic nuclide data

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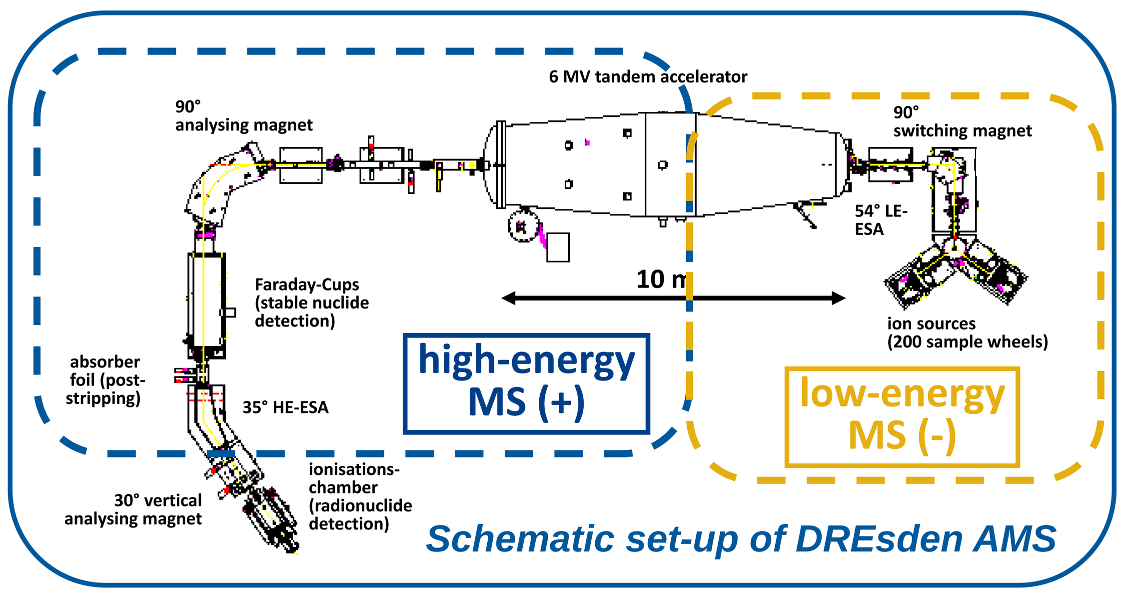
Introduction

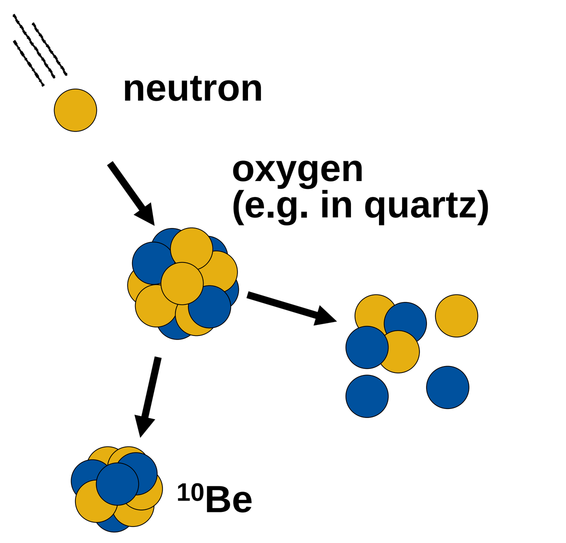
Accelerator mass spectrometry (AMS) & cosmogenic nuclides (CN)

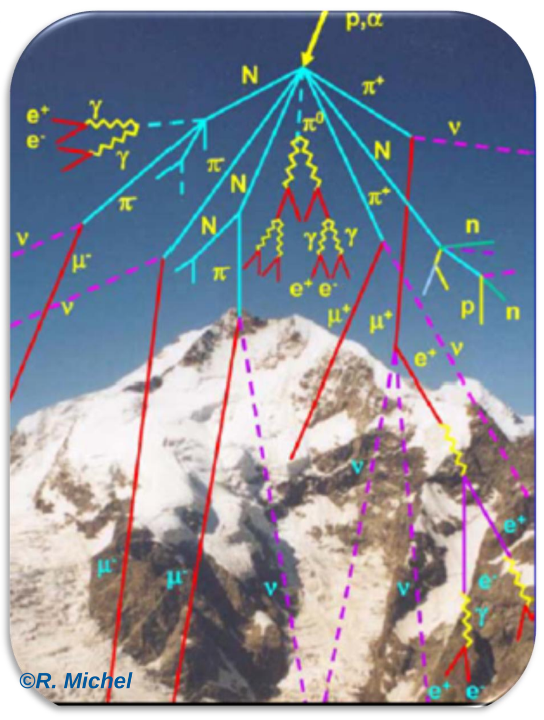
- method of choice for determination of ^{10}Be ($t_{1/2}$ = 1.378 Ma): AMS [1,2]
- ^{10}Be/^{9}Be as low as 10^{-16}



Schematic set-up of DREsden AMS

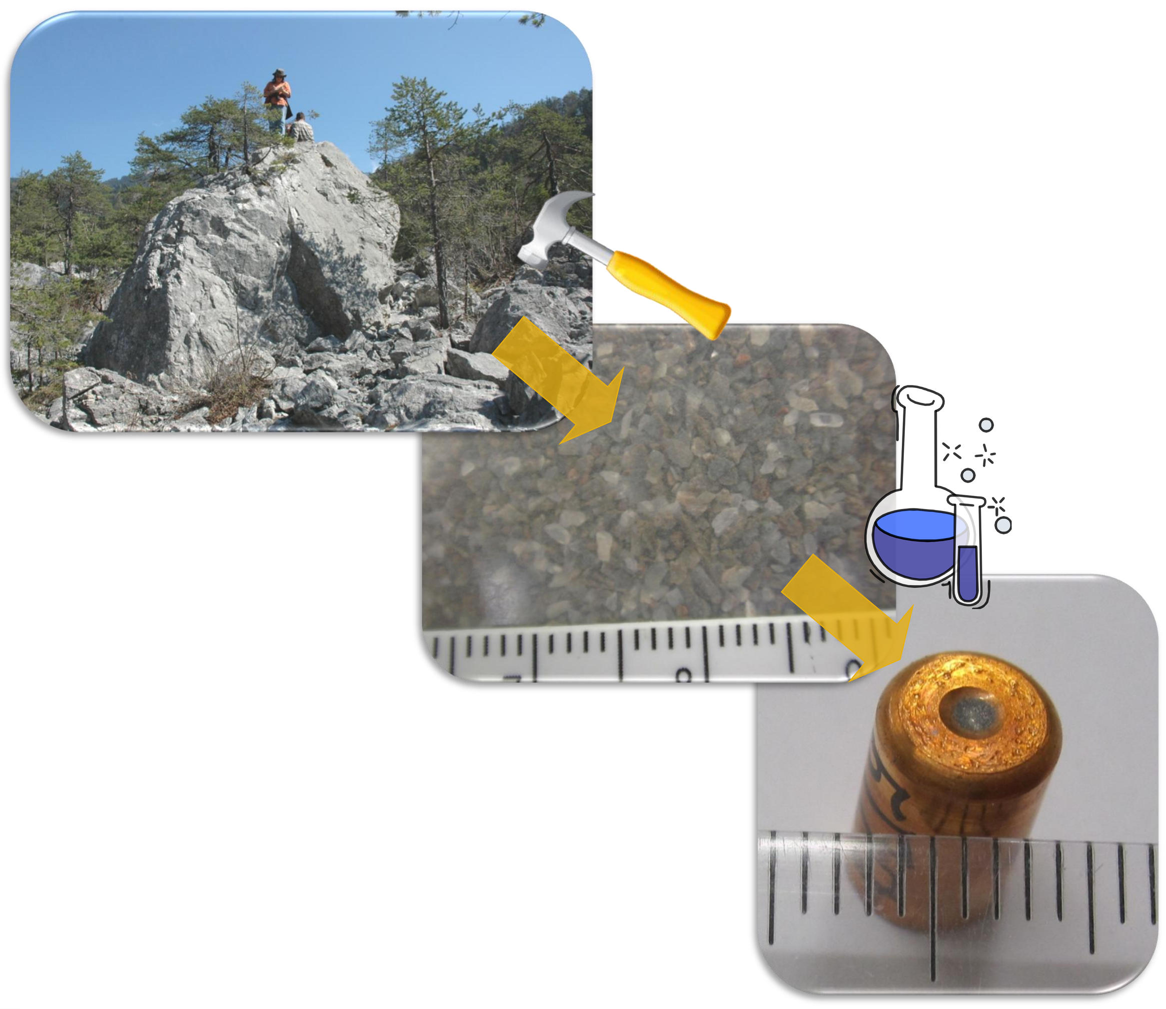
- ^{10}Be (=CN) produced by nuclear reactions in space or on Earth ››› 10^{-9} – 10^{-14}





Radiochemistry

- need for radiochemical separation to enrich ^{10}Be & reduce matrix: 0.1 - 300 g sample ››› 0.5 mg BeO



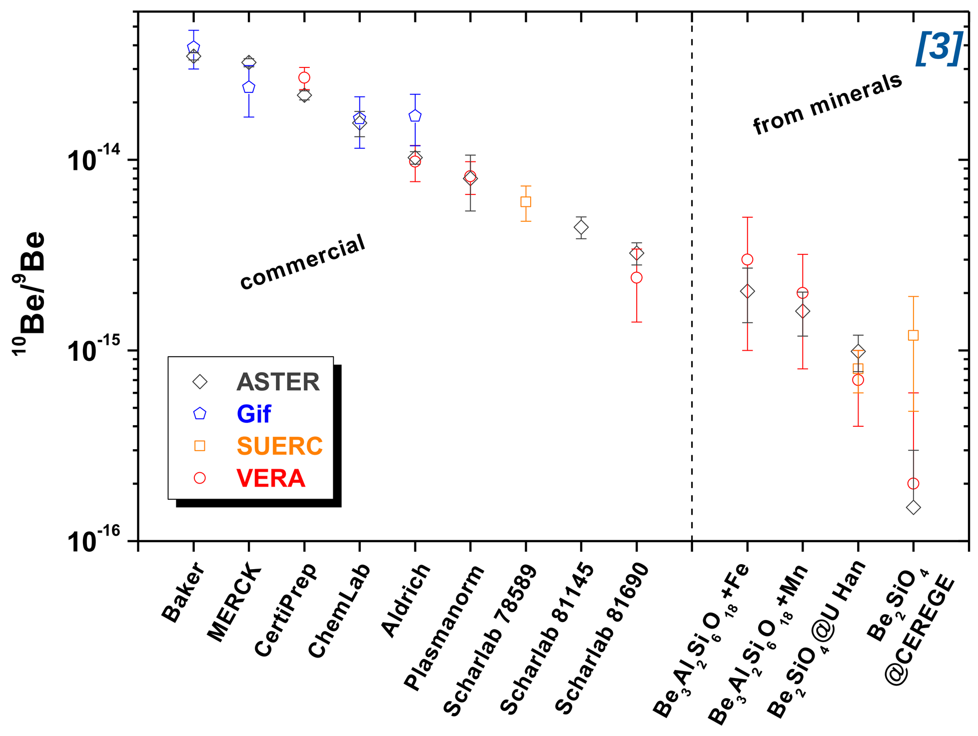
- most samples are too low in natural ^{9}Be ››› addition of ^{9}Be-carrier solution of known concentration for sample preparation

››› ^{9}Be-atoms taken into account to calculate the number of ^{10}Be-atoms in sample

If ^{9}Be is wrong, ^{10}Be is wrong!

Problem

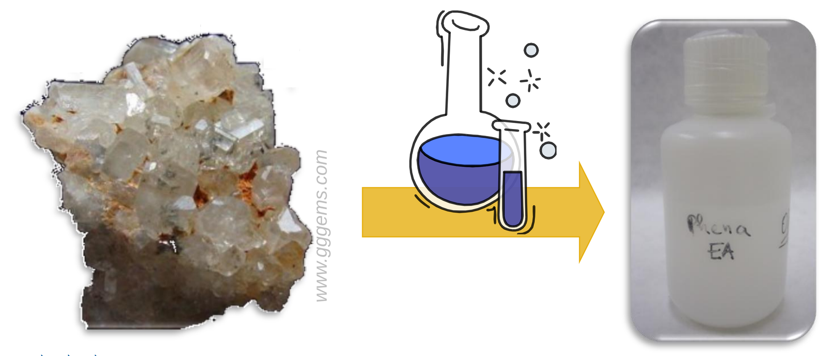
- commercial ^{9}Be-carrier contains intrinsic ^{10}Be @ the level of 0.3 - 4 ·10^{-14}



››› ^{10}Be too high for several applications

››› need for home-made ^{9}Be-solution from phenakite (Be_2SiO_4) or beryl ($Be_3Al_2Si_6O_{18}$) crystals

- after several weeks of chemistry [3]: ^{9}Be-solution



››› need for accurate ^{9}Be-measurement

^{9}Be-measurements & -data

First idea: Replicate "French" work

- Three independent measurements:
  - gravimetry
  - flame atomic absorption spectrometry (AAS)
  - graphite furnace AAS (GFAAS)

of earlier ^{9}Be-solution (Phena-DD) @ CEREGE: (3025 ± 9) µg/g ››› 0.3 % uncertainty ☺

- New Phena-EA solution analysed:
  - gravimetry: (2214 ± 84) µg/g
  - inductively coupled plasma-mass spectrometry (ICP-MS): (2038 ± 128) µg/g
  - inductively coupled plasma-optical emission spectrometry (ICP-OES): (2400 ± 14) µg/g

(2217 ± 181) µg/g ››› 8 % uncertainty ☹

››› Find additional labs for round-robin exercise!

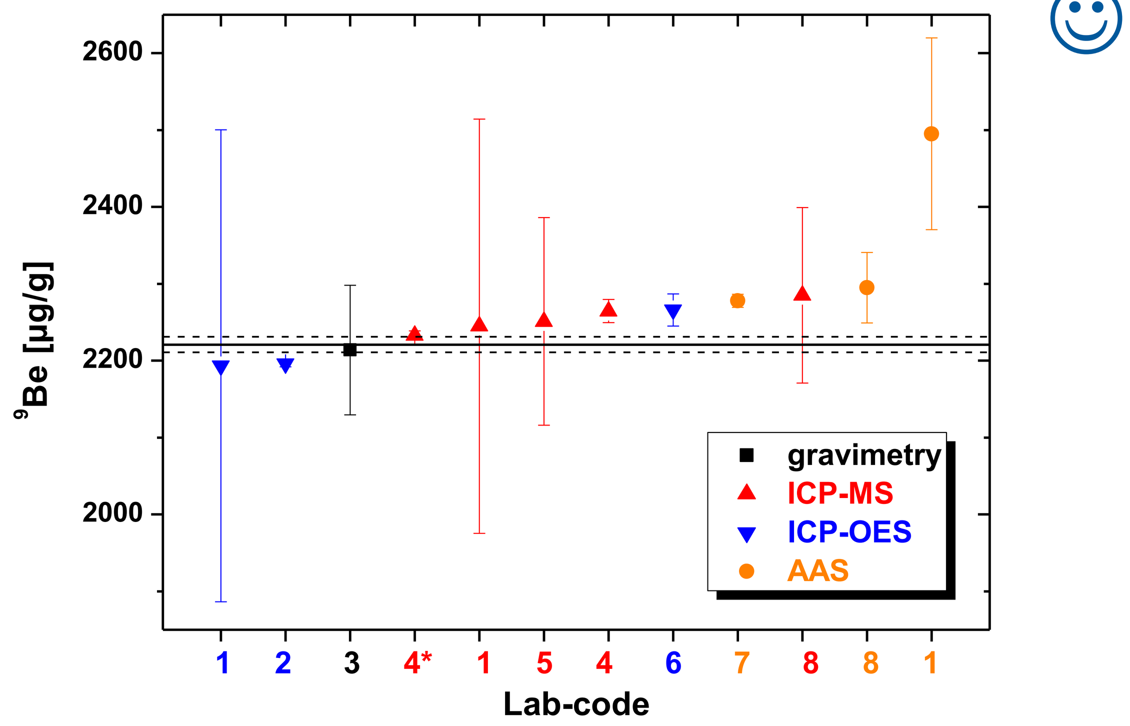
Round-robin labs, methods & data

- 2 out of 3 labs remeasured/-calculated (-9 % & +6 %)
- additional labs: + 4 research, + 1 commercial ☺

^{9}Be [µg/g]	method	lab-code
2193 ± 307	ICP-OES	1: commercial lab
2196 ± 4	ICP-OES	2: research lab
2214 ± 84	gravimetry	3: research lab
2233 ± 6	ICP-MS (st.ad.)	4: research lab
2245 ± 269	ICP-MS	1: commercial lab
2251 ± 135	ICP-MS	5: research lab
2265 ± 15	ICP-MS	4: research lab
2266 ± 21	ICP-OES	6: research lab
2278 ± 8	AAS (flame)	7: research lab
2285 ± 114	ICP-MS	8: research lab
2295 ± 46	GFAAS	8: research lab
2495 ± 125	GFAAS	1: commercial lab

Data evaluation - Simple

- Grubbs outlier at significance level of α = 0.01: commercial lab "1" (GFAAS) ››› removed!
- distribution basically normal
- mean and median not significantly different
- weighted mean metrologically the very best estimate: (2221 ± 10) µg/g ››› 0.44 % uncertainty ☺



^{9}Be-data
Data evaluation - Sophisticated

- mutual agreement values E_n

E_n between two individual labs given by

$$E_n = | x_a - x_b | / \sqrt{u^2(x_a) + u^2(x_b)}$$

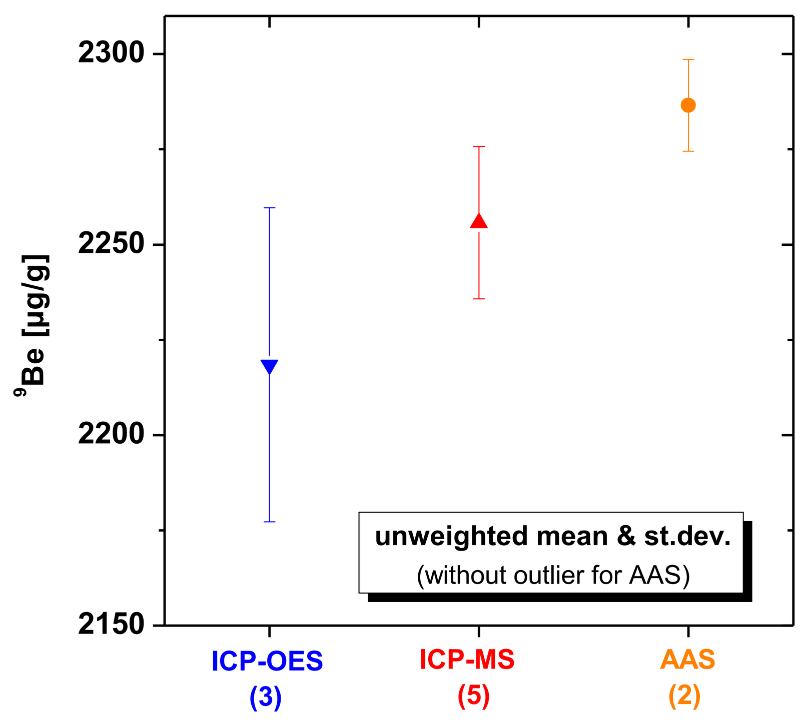
with x_i = individual lab result

$u(x_i)$ = uncertainty of ^{9}Be as stated by lab

^{9}Be result from lab a compatible with ^{9}Be result of lab b, if E_n <2

››› labs 2 and 4 underestimate grossly their uncertainty

- methods are slightly, but not significantly different (also proven by ANOVA)



Conclusions

- maximum deviation of single lab result from weighted mean ~3.3 % ››› need for all labs using non-commercial ^{9}Be-carrier to have it analyzed at more than one lab (outlier lab-Δ=12 %!)
- very likely that same problem arises if measuring individual samples››› constant quality assurance checks by e.g. taking part in round-robin exercises necessary
- differences might be even more prominent at the ng/g-level

No accurate ^{10}Be-data without accurate ^{9}Be-data (carrier & samples)!

Acknowledgments

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References

[1] Talk S. Merchel, Thursday, 28.02.@12 h.
[2] Akhmadaliev et al., NIMB 294 (2013) 5.
[3] Merchel et al., NIMB 266 (2008) 4921.

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