Guideline for Sampling and Preparation

How to achieve high accuracy for quantitative analysis

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Introduction

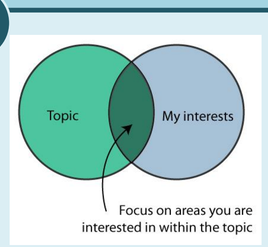
In modern instrumental analysis a high accuracy and precision combined with a preferably high sample throughput are important performance criteria. These criteria are essential to observe and quantify small differences of a parameter within a heterogeneous environment. To achieve highly accurate and precise measurement results, a well-considered sampling strategy as well as a thorough sample preparation is required. Depending on the research question different sample types need different sample pre-treatment and preparation steps.

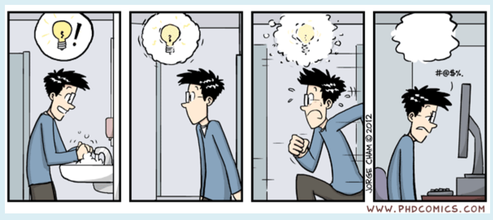
Insufficient sample treatments can lead to defective measurement results and incorrect assessments. This can be avoided by following a consistent guideline to attain comparable and reproducible results. Furthermore the technical advances in instrumental analysis allow the use of rather small sample amount. Therefore the most important thing is to achieve a homogeneous sub-sample [1].

For reproducible results thorough sample preparation is required!

Workflow

research questions





planning

- Did you consider, to announce your measurements to the service departments?
- Are they able to measure your samples?
- Check that you have everything you need or do you have to order things?
- How long will it take to prepare your samples for all measurements?

sampling

- sampling strategy and statistics [2]
- clear sample designation and data structure (Rules of Good Scientific Practice & GLP)
- tools (shovel, split tube, sample bags and so on)
- liquid N

→ *field sample*

sample pre-treatment

Pre-treatments need to happen at the field side or shortly after sampling:

- homogenize (make a representative lab sample from the field sample) subdividing or sample reduction (mechanical splitting, bowl and cut into quarters or Cross-Riffling)
- if needed: pre-classification (remove big stones and roots, sieving > 2 mm) & weigh the sample

Preservation, transport and storage temperature have to be appropriate due to the research question: **-20/-80 °C** for DNA/RNA analysis, PLFA analysis & long term storage
+ 4 °C for microbial biomass, enzyme activity, soil incubations
non for chemical & physical analysis

→ *lab sample*

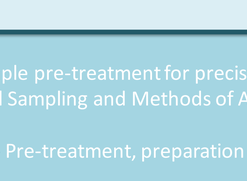
sample preparation

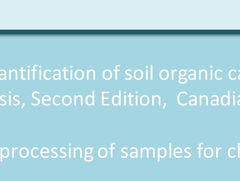
→ *test sample*

The preparation of the lab samples according to requested analysis:

	plant samples: isotope & routine measurements	soil samples: isotope & routine measurements	soil samples: enzyme analysis	soil samples: soil incubations
oven- or freeze drying	60 °C [4]	40 °C [2,3,4]	Do not dry your samples if you want to measure biological properties! [1,2,3,4]	
homogenizing	cutting or coffee mill	sieving < 2 mm & sub-sampling [3]		
fine grinding	ball mill → powder-like material			
drying	60 °C	Convert the results to the 105 °C-dry basis. [2,4]		

analysis





Devices

plant samples

Cutting mill

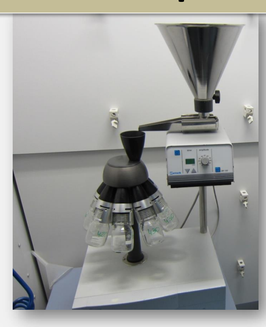
Coffee mill

soil samples

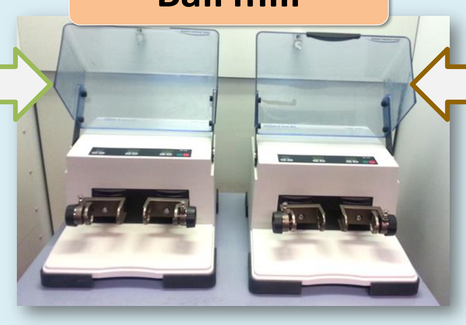
Sieving machine

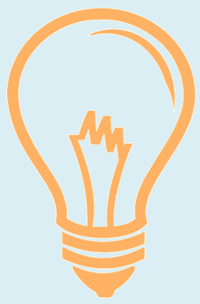
Jaw crusher

Sub-sampler



Ball mill





plant samples

(plants, leafs, wood, branches, litter, roots)

- In case of very resinous or fibrous material it could help to deep freeze your sample with liquid nitrogen or with dry ice before grinding. This should improve your grinding result.
- During the grinding process the sample material will heat up → **Just grind as long as necessary!**

soil samples

- During the grinding process the sample material will heat up which may cause carbon losses → **Just grind as long as necessary!**
- Silicate enriched soils have to be grind a bit longer but with less material (max. 8 min).
- With small grinding jars you will achieve satisfactory grinding results much faster.
- Determination of water content is required to convert the results to the 105 °C-dry basis. This has to be done at the same time of the weighing for analytical purpose. [2,4]

Conclusion

The state of the art of analytical instruments allows to measure samples very precisely, with very low detection limits. Insufficient care while sampling at the field site and sample preparation in the lab can influence analytical results considerably. Different sources of errors are possible and sum up to a total error: $c = \sqrt{a^2 + b^2 + c^2 + \ldots}$

For example : a) data of organic carbon originated from soils with different accuracy levels in root extraction can result in high valuation discrepancies of total carbon stocks, b) we proofed that soil samples, which have not been ground sufficiently, do not achieve reliable and precise results [1].

References:

(1) Hilke, I. & Henkel, K. (2014): Accurate sample pre-treatment for precise quantification of soil organic carbon.
(2) Carter, M.R. & Gregorich, E.G. (2007): Soil Sampling and Methods of Analysis, Second Edition, Canadian Society of Soil Science, ISBN: 9780849335860
(3) DIN 19747 (2009): Investigation of solids - Pre-treatment, preparation and processing of samples for chemical, biological and physical investigations, 2009-07
(4) VDLUFA (1991): Methodenbuch I, Die Untersuchung von Böden, 1.Teillieferung, ISBN: 3-922712-42-8

For further information see:

- Schlichting, E. et al. (1995): Bodenkundliches Praktikum, 2. Auflage, Blackwell Wissenschaftsverlag, ISBN: 3-8263-3042-0
- BGR (Bundesamt für Rohstoffe) – AG Boden
- SSSA (Soil Science Society of America), https://www.soils.org/

pictures by Ilka Mai, Iris Kuhlmann, Jessica Heublein