Representative Sampling

30 min crash course

Peter Paasch Mortensen (MSc. Chem. Eng, Ph.D.)
Senior Project Manager

Global Categories & Operation – Supply Chain Development

Initial questions one could ask

Why do we sample?

- Proces control,
- Quality,
- Legal,
- Economics,
- Mass Balances
- .....

Who is sampling?

Machines

Operators

What characteristics do we want the sample to have?

- Easy to extract,
- Composite sample,
- Representative,
- .....

Which characteristics are most important?

Representative Sampling ”Take home messages”

In general:

- Focus on **representative** sampling first and worry about the rest (e.g. sample size) afterwards.

But ask yourself:

Why do you want a sample?

- Proces control/adjustment, Quality, Legal, ...
  (different effort might be needed)

How to sample?

- At what frequency?
- Random, Systematic , Stratified?
- Instrumentation: In/On/At/Off-line evaluation?

Is the sample representative?

- This can (and should) be evaluated

Representative sampling – 30 min crash course

Outline:

- What is representative sampling
  - Correct sampling
  - 0-D and 1-D lots
- Sampling errors
  - Correct sampling errors
  - Incorrect sampling errors
- How to evaluate a sampling procedure

Representative sampling

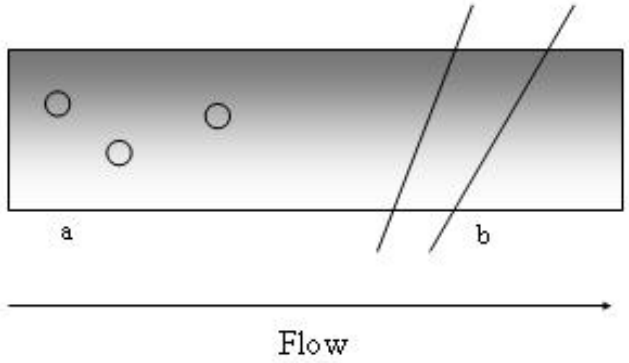
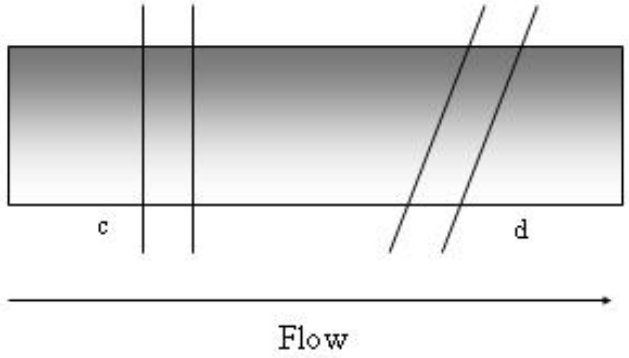
- The overall driver for sampling:
  - Analysis is only performed on a fraction of the complex material (the lot).
- But:
  - If the sample does not truly represent the lot, erroneous deductions and conclusions will invariably follow – *no matter how precise the actual analysis*
- So:
  - There has to be a balance between the sampling accuracy & precision and the assaying technique imposed on the sample.

When is a sample correctly extracted?

Basic requirements

1. ALL elements in a batch must have equal probability of being selected by the sampling tool.
2. All elements NOT part of the sample/increment defined by the sampling tool must have zero probability of ending up in the sample (e.g. no leftovers in sampling tool).
3. And that the sample is not altered after extraction.

Correct sampling

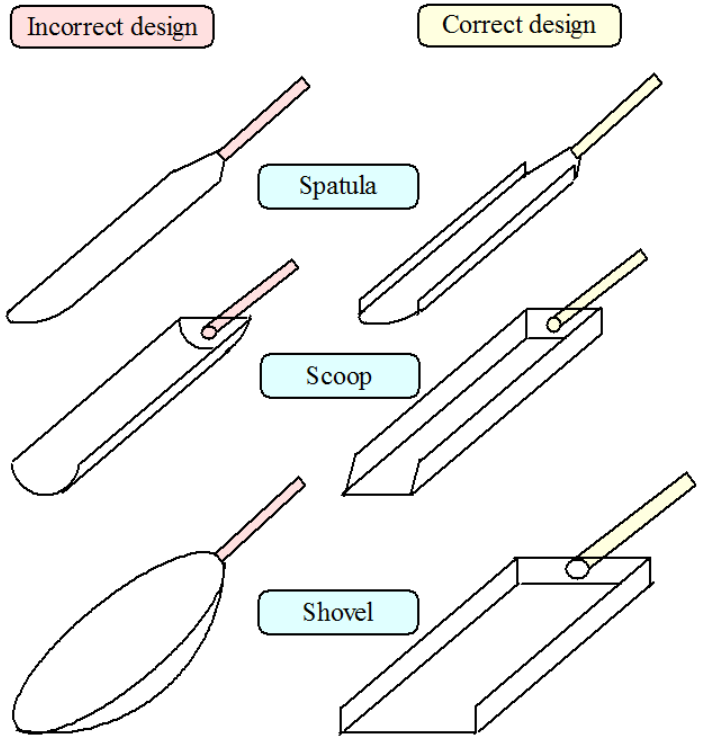
	0-D sampling	1-D sampling
Incorrect		
Correct		

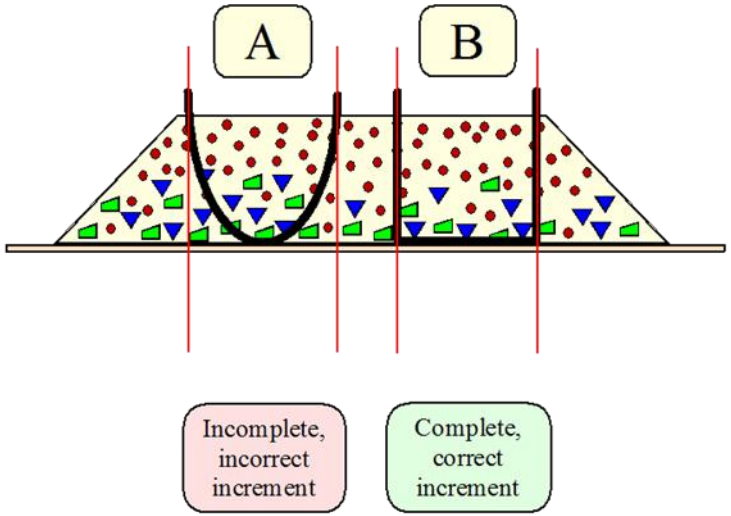
Basic rule:

All elements in the lot must have equal probability of being selected.

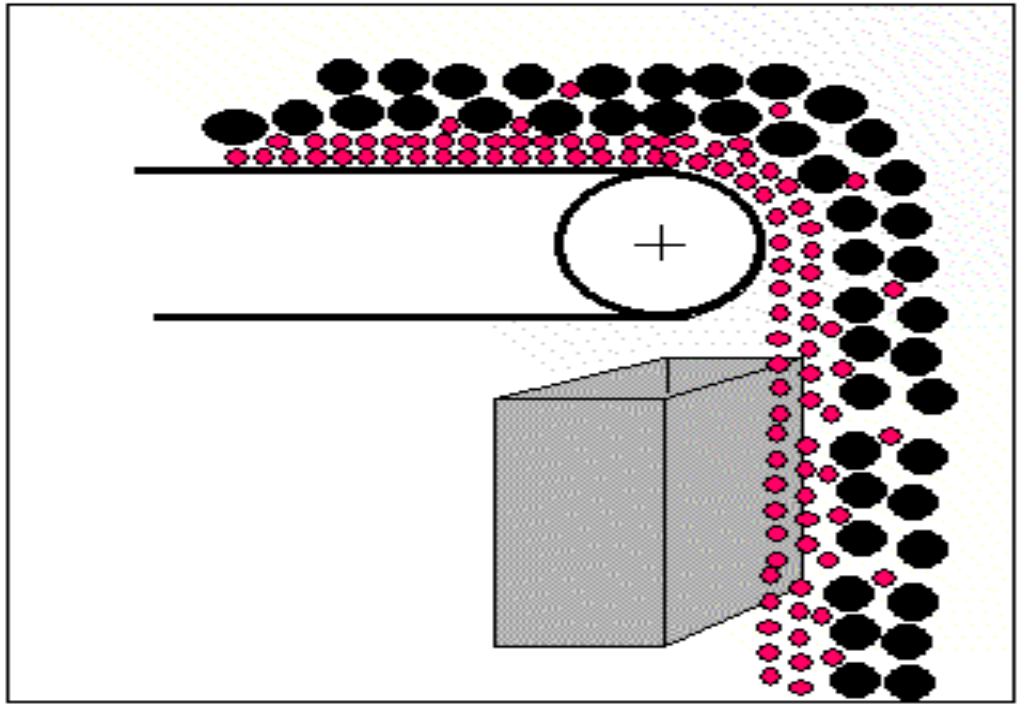
Why are ”samples” not always representative ?

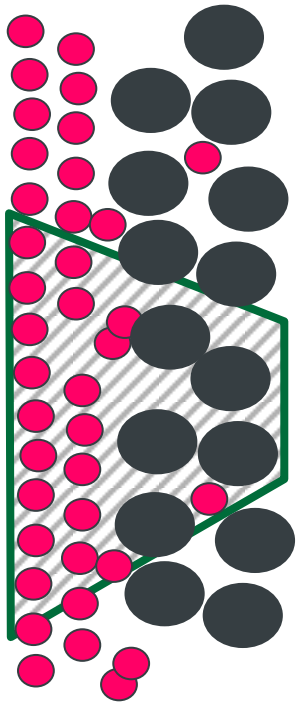
Sample tools are not designed properly





The effect of incorrect sample delimination

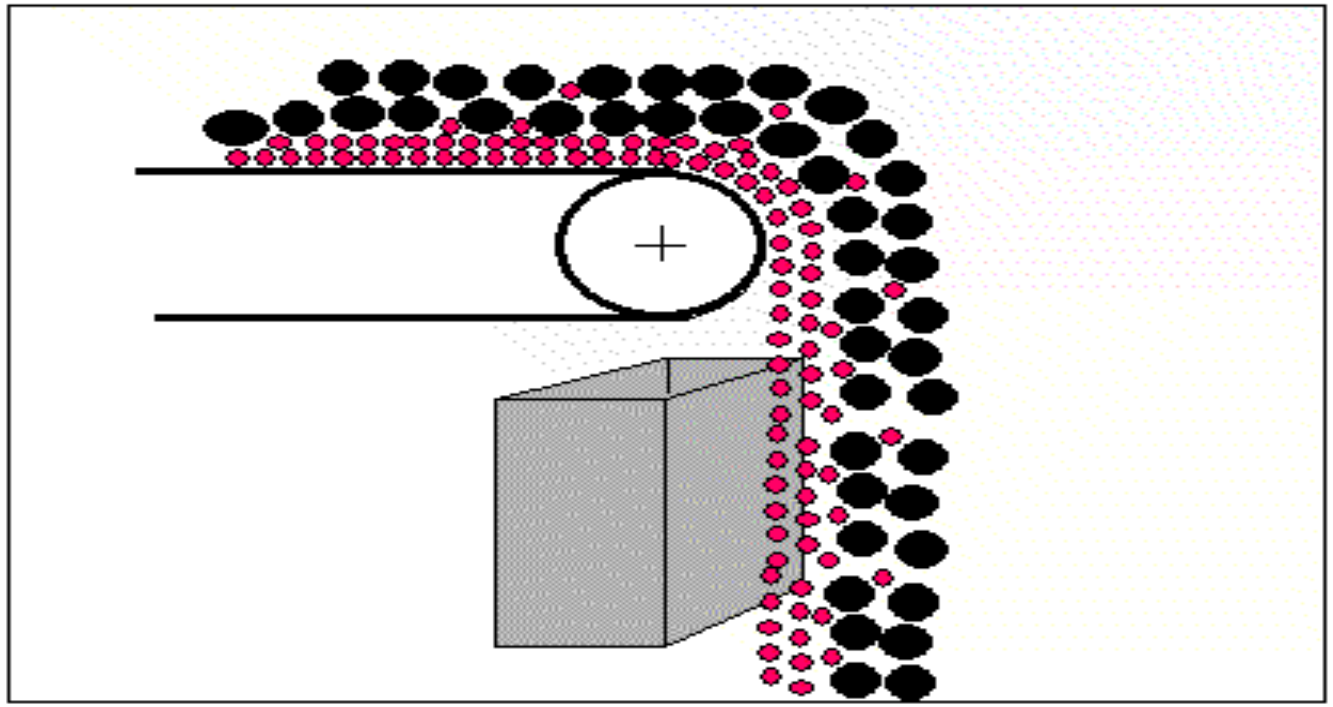


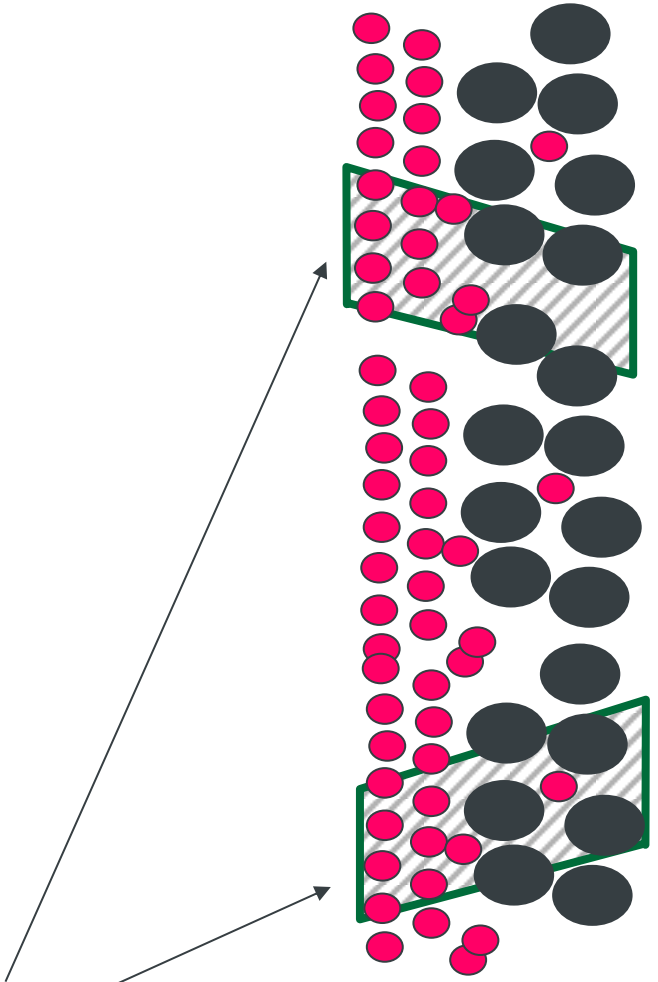


Incorrect sample profile

Minkkinen 2006

The effect of correct sample delimination



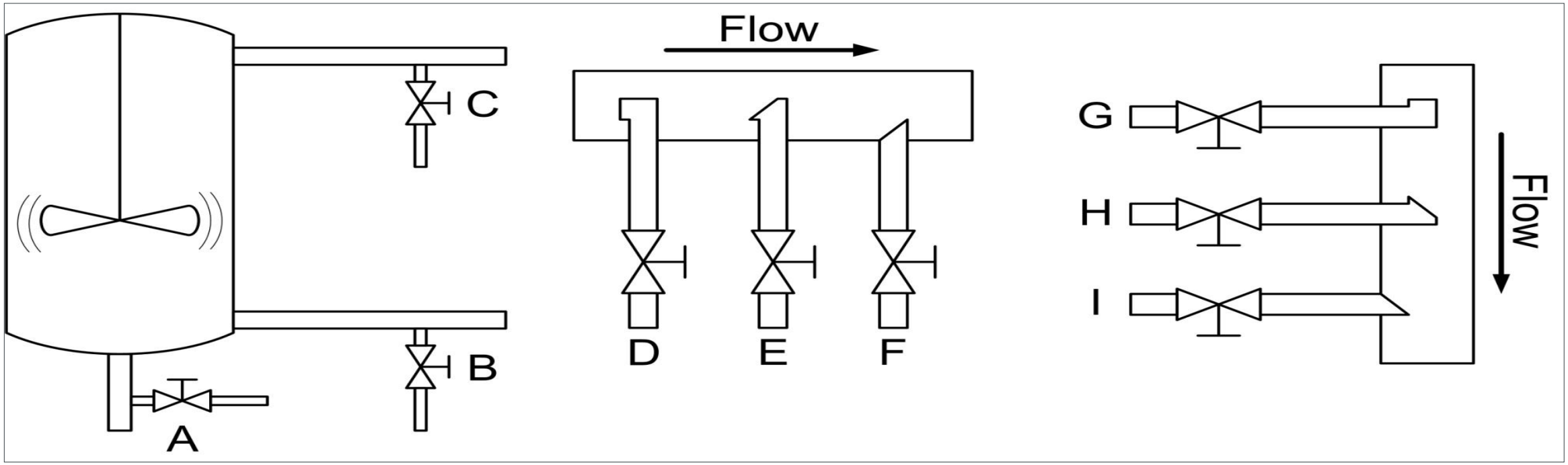


Correct sample profiles

Minkkinen 2006

Why are ”samples” not always representative ?

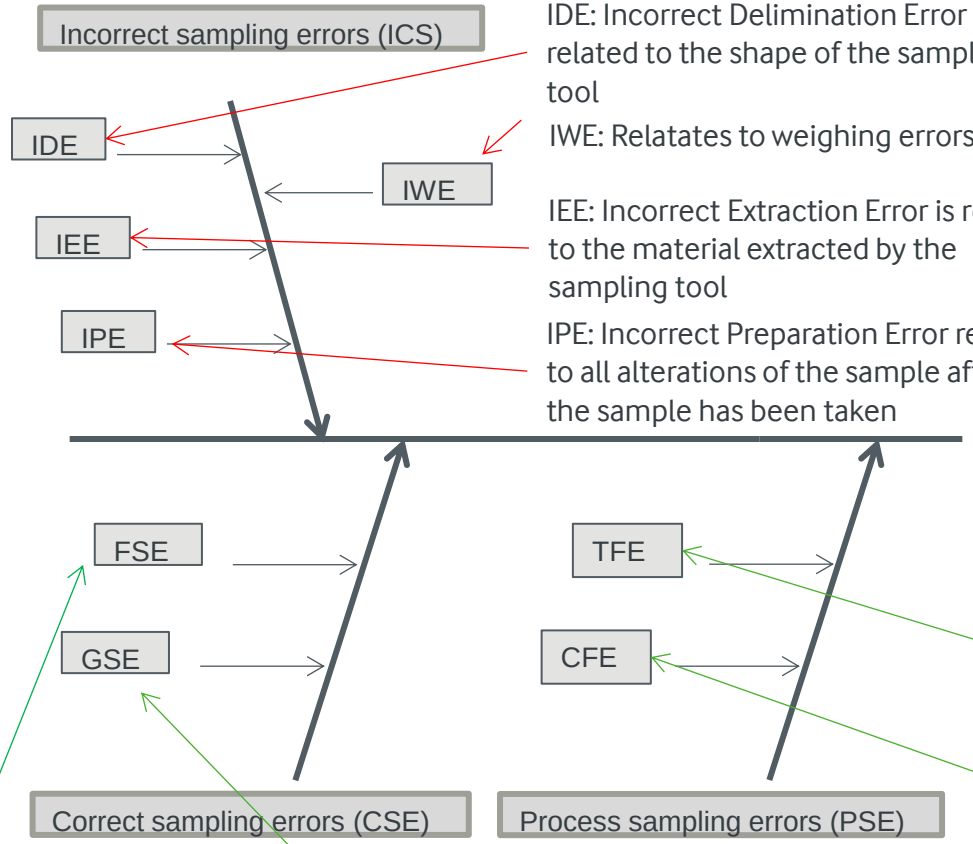
Sample tools are not designed properly continued



Theory of Sampling (TOS)

The global estimation error is made up of the total sampling error and the total analystical error

Total sampling error prior to Laboratory



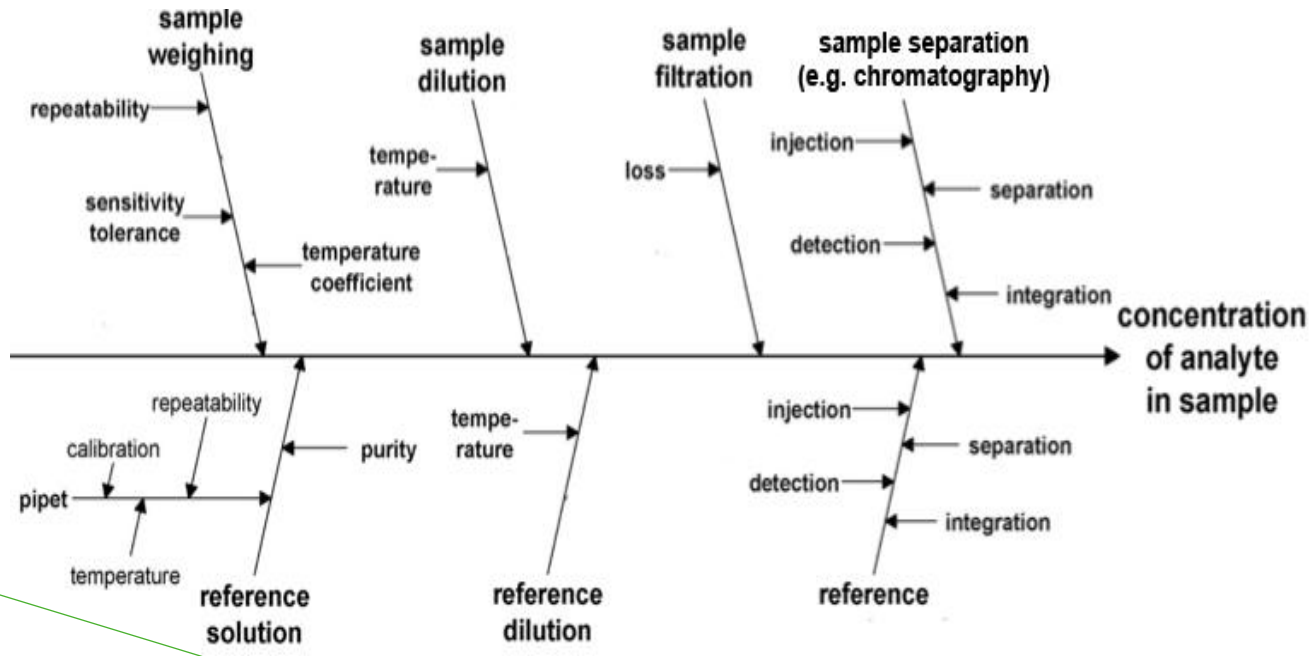
IDE: Incorrect Delimination Error is related to the shape of the sampling tool

IWE: Relatates to weighing errors

IEE: Incorrect Extraction Error is related to the material extracted by the sampling tool

IPE: Incorrect Preparation Error relates to all alterations of the sample after the sample has been taken

Total Analytical Error



FSE: Fundamental Sampling Error originates from differences between fragments – crush material to minimize.

GSE: Grouping and Segregation Error originates from particles tendency to segregate within the lot – blend material to minimize.

CFE: Cyclic Fluctuation Error originates from periodic changes over time. Do varigraphic analysis to determine sampling frequency.

TFE: Time Fluctuation Error originates from long term trends. Do varigraphic analysis to determine sampling frequency.

Why should I care about sampling errors?

Because - There is no point in being precisely wrong!

How do we measure representativeness?

First let us define the relative sampling error from the analytical grade a in the lot L and the sample s:

$$e = \frac{a_s - a_L}{a_L}$$

- A sampling process is said to be accurate when the average error m_e is practically zero
- A sampling process is said to be reproducible if the variance of the relative error s_e^2 is small.

Representativeness r_e^2 is a composite property of the relative error

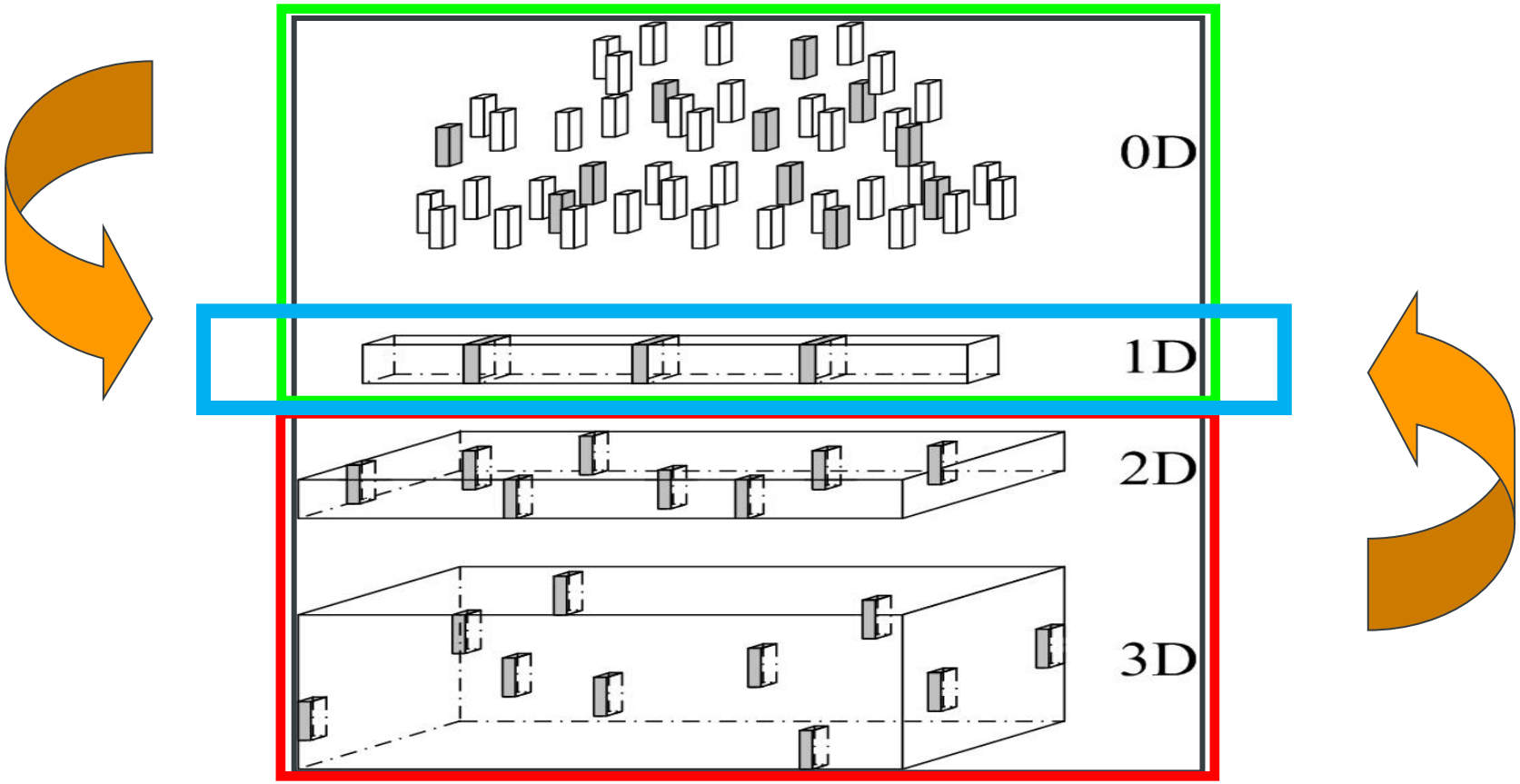
$$r_e^2 = m_e^2 + s_e^2$$

- Only a correct sampling procedure secures that samples are both accurate and reproducible
- The random component of r_e^2 tend to reduce when averaging over a large number of sample.
- This is not the case with the systematic part and this is why a sampling bias is problematic.

Sampling Unit Operations

1. Always perform a heterogeneity characterization of new materials
2. Mix (homogenize) well before all further sampling steps.
3. Use composite sampling.
4. Only use representative mass reduction.
5. Comminute (chrush) whenever neccesary.
6. Perform a varigraphic characterization of 1-D heterogeneity
7. Whenever possible turn 0-D, 2-D and 3-D lots into 1-D equivalents

LDT: Lot Dimensionality Transformation

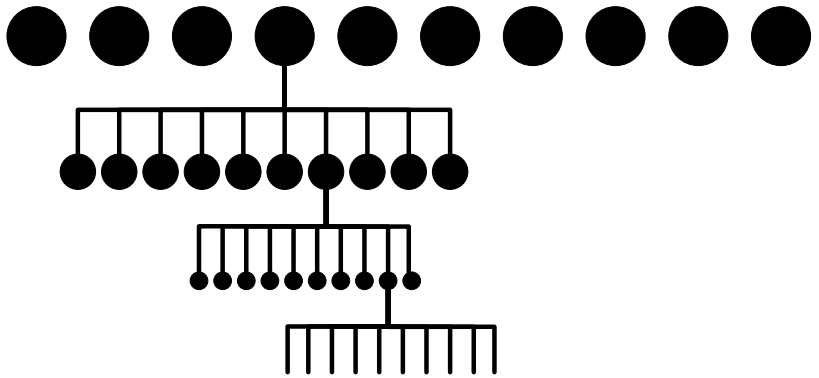


How to take a representative sample of the curd in a cheese VAT?

By transforming 0-dimensional lot to a 1-dimensional lot

How to evaluate a sampling process?

The Replication Experiment (0-D case)



Primary sampling

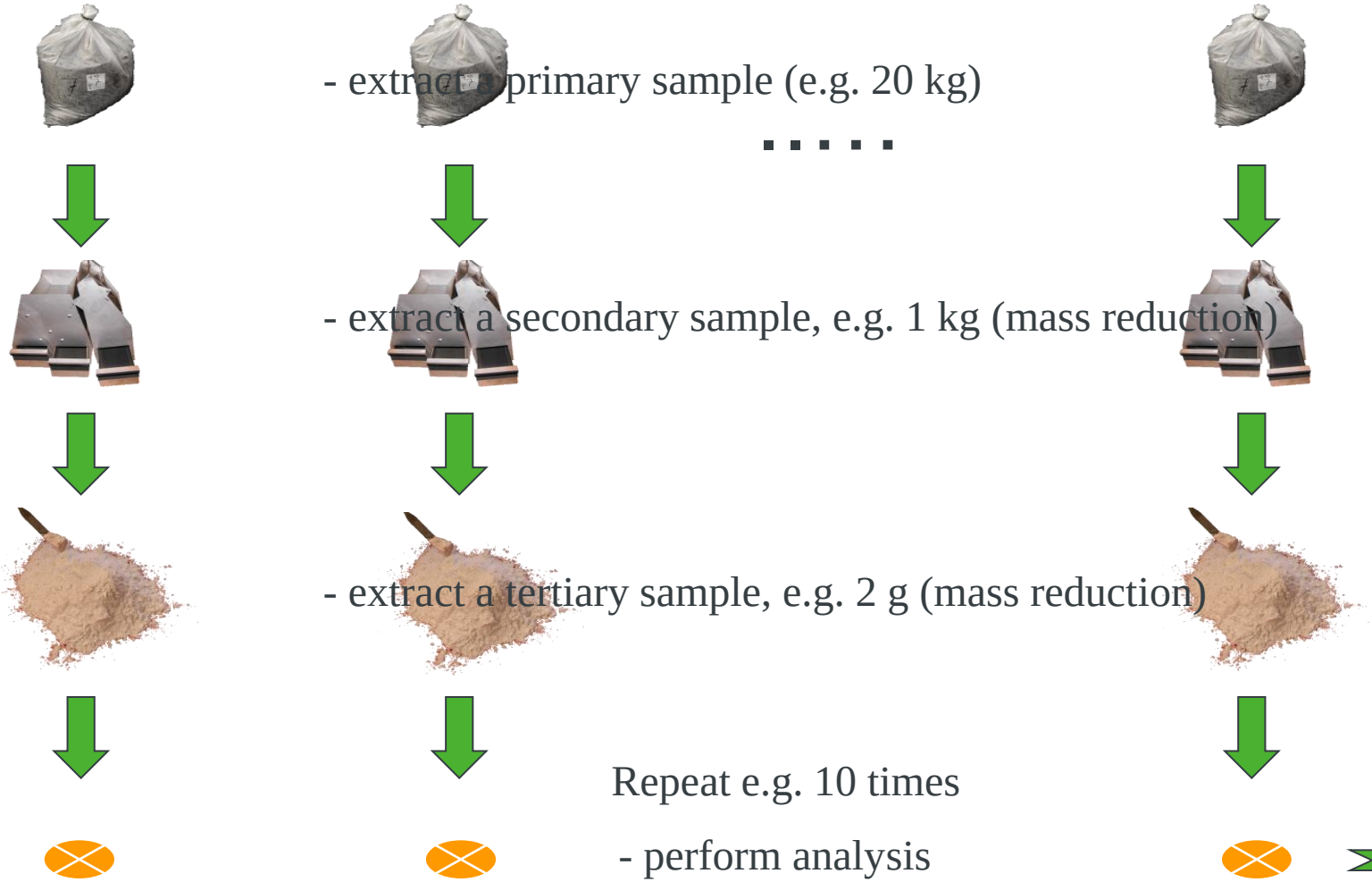
Secondary sampling / Mass Reduction

Tertiary sampling / Sub-sampling

Analysis s.s.

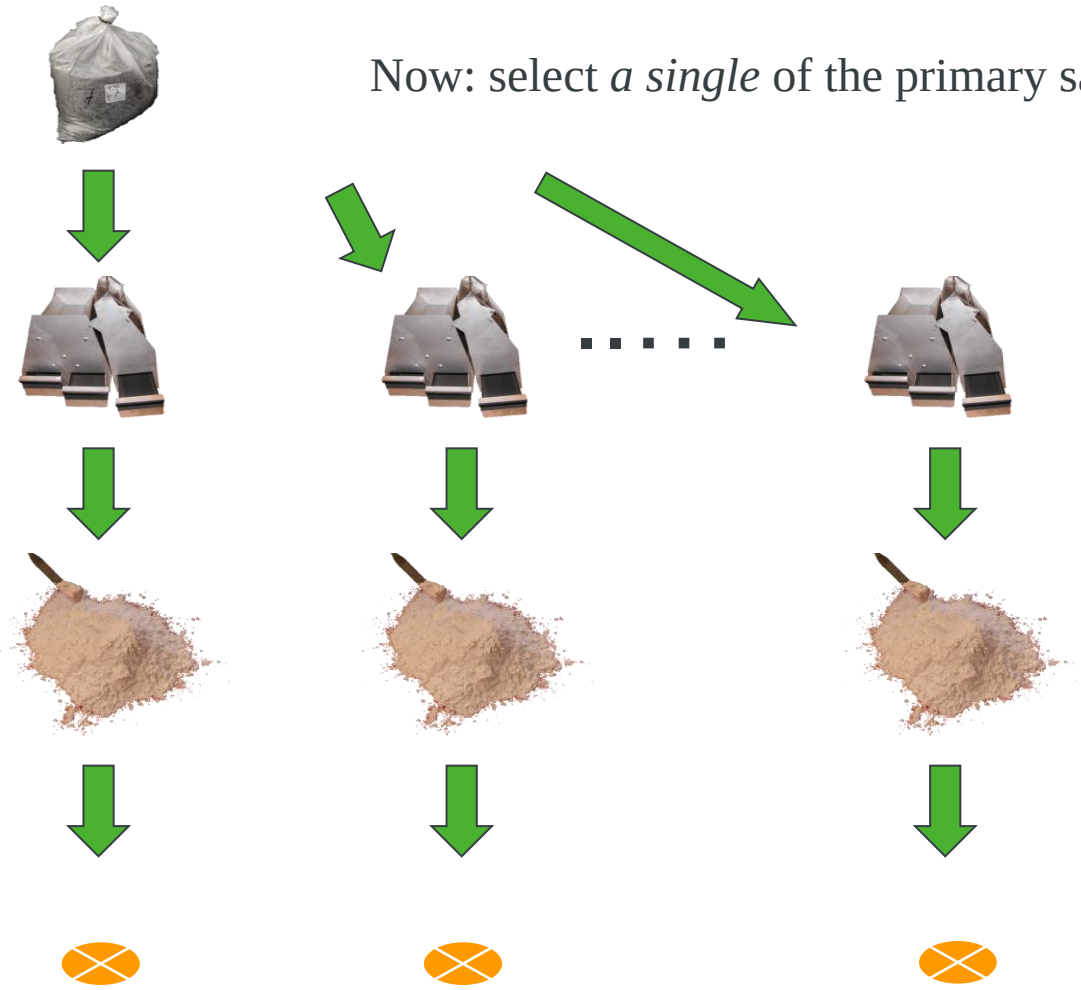
The Replication Experiment (0-D case)

Primary Sampling Error (PSE)



The Replication Experiment (0-D case)

Secondary Sampling Error (SSE)



Now: select *a single* of the primary samples

Repeat sub-sampling (mass reduction) from the *same* primary sample 10 times

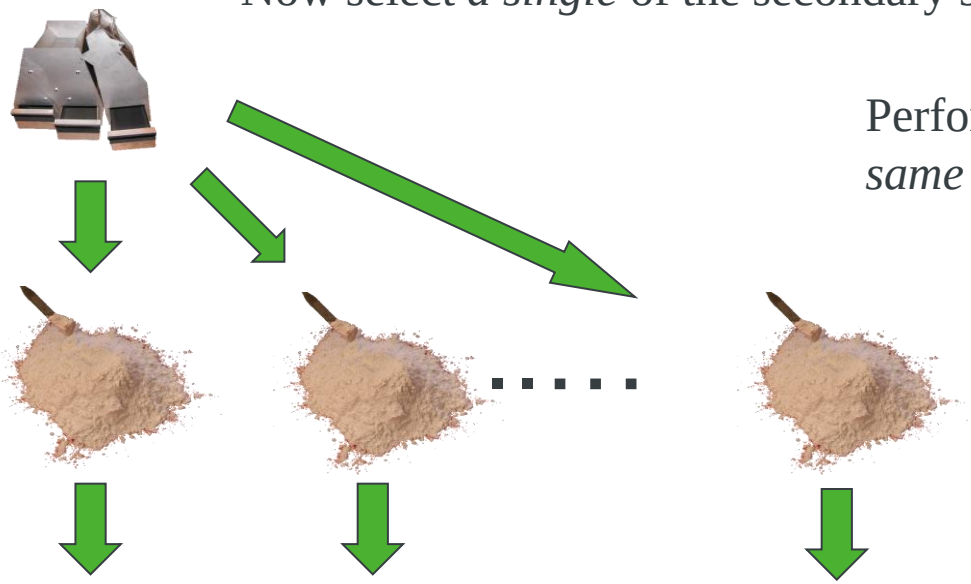
s_{SSE}^2

m_{SSE}^2

The Replication Experiment (0-D case)

Tertiary Sampling Error (TSE)

Now select *a single* of the secondary samples



Perform <u>tertiary sampling</u> 10 times from the *same* secondary sample

s_{TSE}^2

m_{TSE}^2

The Replication Experiment (0-D case)

Total Analytical Error (TAE)

At the bottom line:

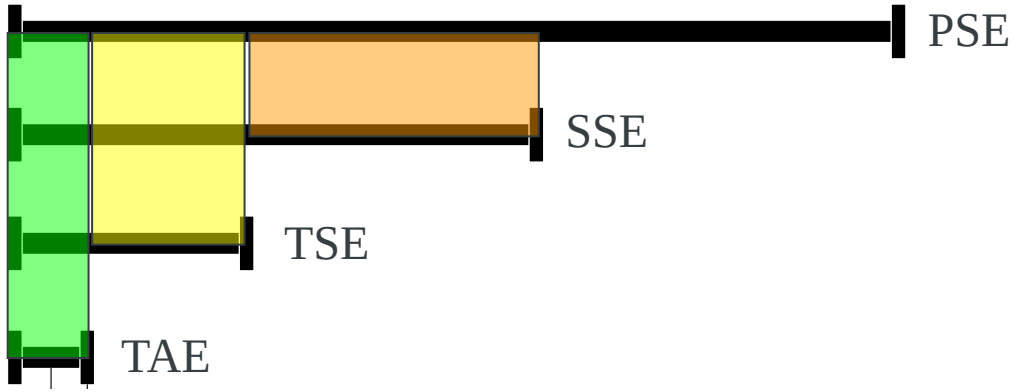
Repeat *only* the analysis 10 times

“Press the button” 10 times! (if analysis is *non*-destructive)

$$s_{TAE}^2$$
$$m_{TAE}^2$$

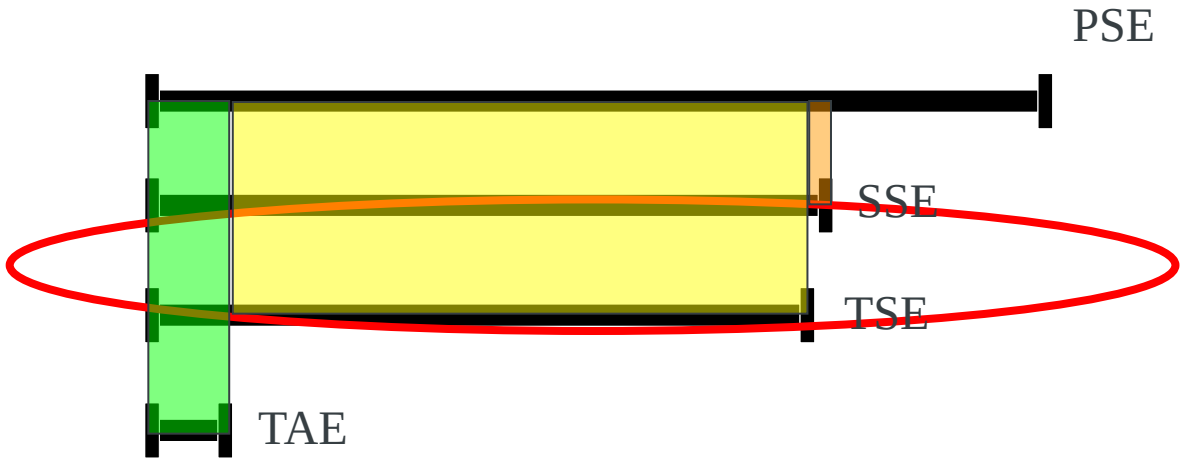
The Replication Experiment (0-D case)

Comparison of sampling stages



Typical relative magnitude of sampling variances:

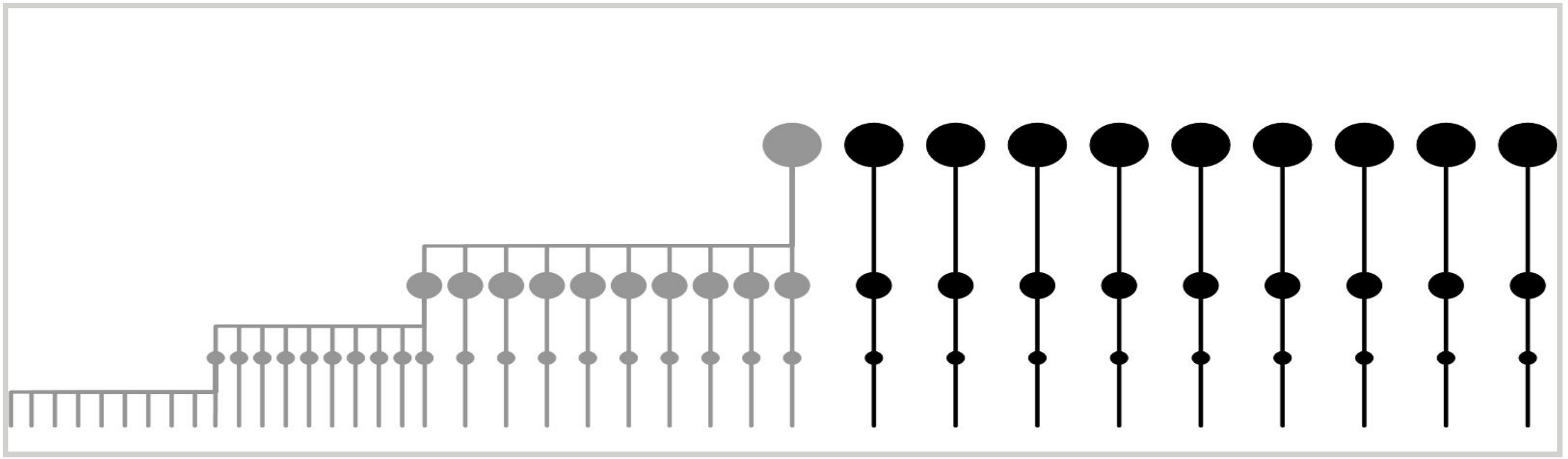
var(PSE),var(SSE),var(TSE),var (TAE)



Something is clearly WRONG regarding the third sampling level - we may want to have a closer look at the *third stage* in our sub-sampling procedure …

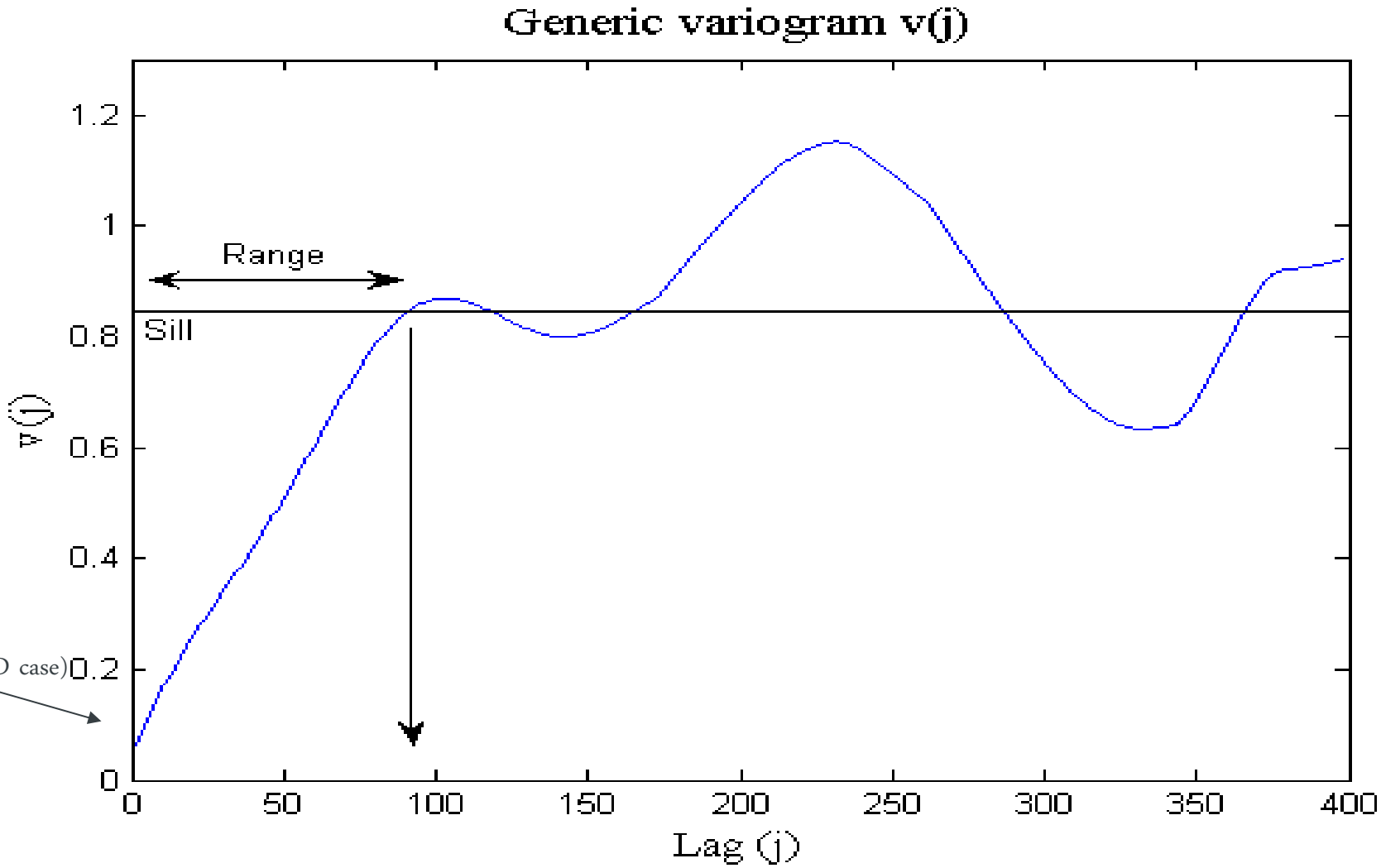
The Replication Experiment (0-D case)

Now was the sampling procedure correct ? The averages will tell



$$m_{TAE} \approx m_{TSE} \approx m_{SSE} \approx m_{PSE}$$

1-D sampling



Activity

0.6

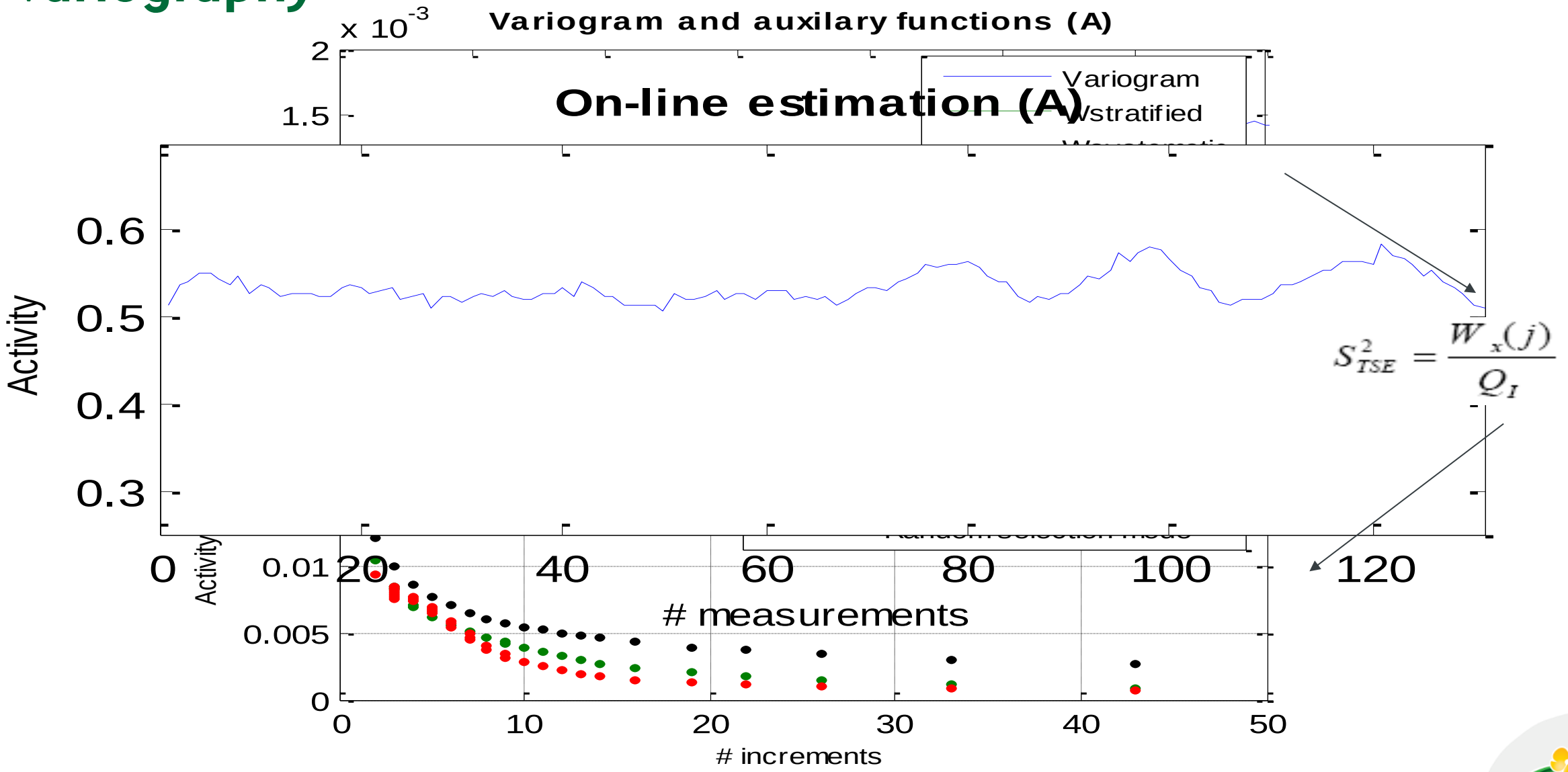
0.5

0.4

0.3

Minimal Practical Error (0-D case)

Variography



In conclusion

- Representative sampling is crucial for data reliability and the conclusions that follows.
- Theory of samling offers guidance on how to characterize materials and sampling procedures.
- By simple tests you can get an idea of how representative your sampling procedure is – its **not rocket science.**

Thank you for your attention

Contact information: pemoe@arlafoods.com