Keynote lecture: EUROANALYSIS 2025

Why analysis needs the Theory of Sampling (TOS) – the importance of the 'before analysis' domain

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WHY analysis needs the Theory of Sampling (TOS) – What's behind 'Data Quality'

- There is an important past for analytical results ...
- The past has an impact on the total Measurement Uncertainty (MU_{total})
- This also impacts significantly on '**data quality**' ...

Always!!!

Uncertainty ← TSE + TAE

Analytical result = ”Data”+/-”Uncertainty” (TAE)

Total Analytical Error = TAE

Analysis: ONLY a **very small** fraction of a lot!

Sampling errors contribute with a **dominating** uncertainty, can be 10/25/50 X larger than TAE!

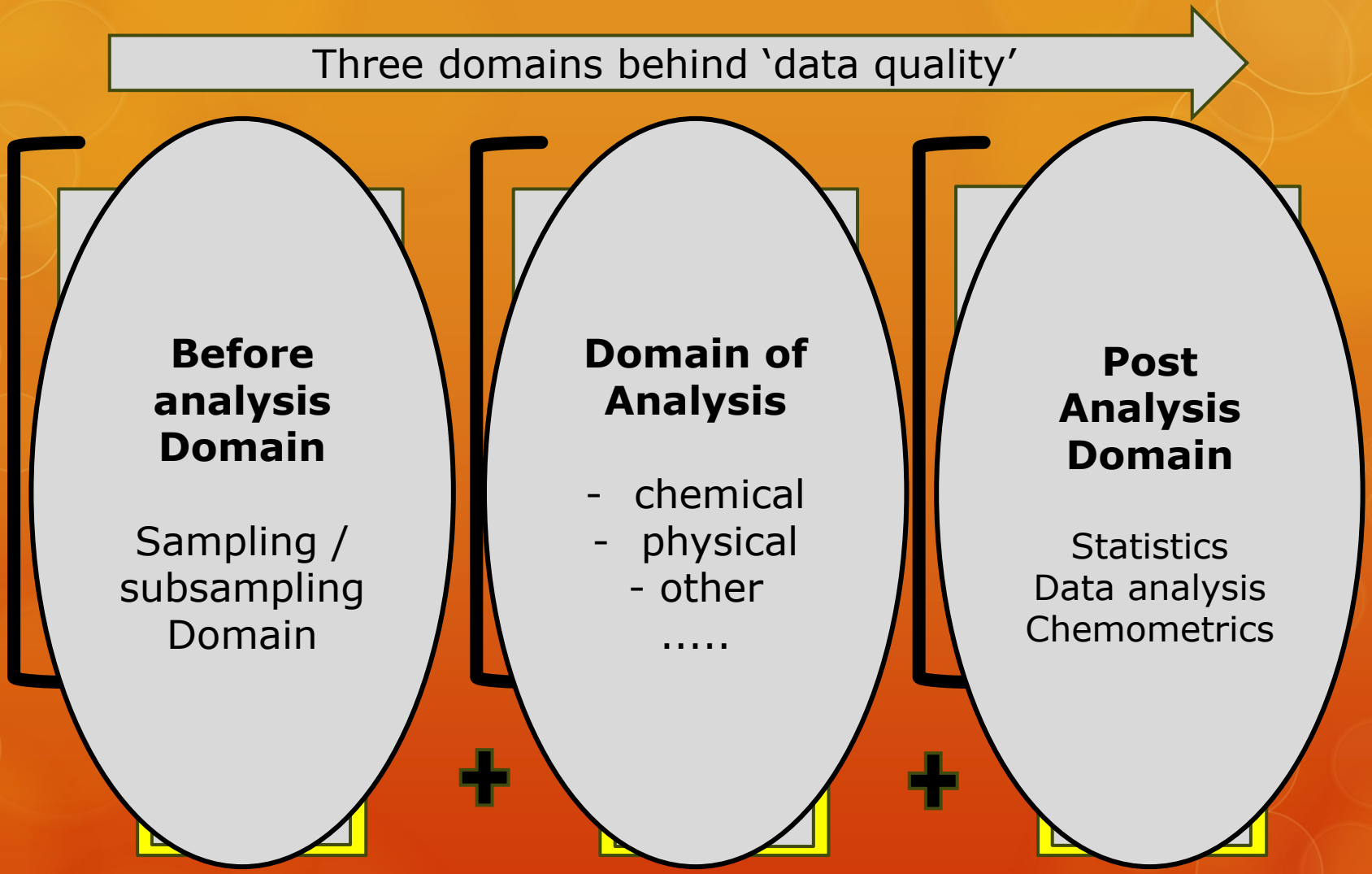
Total Sampling Error = TSE

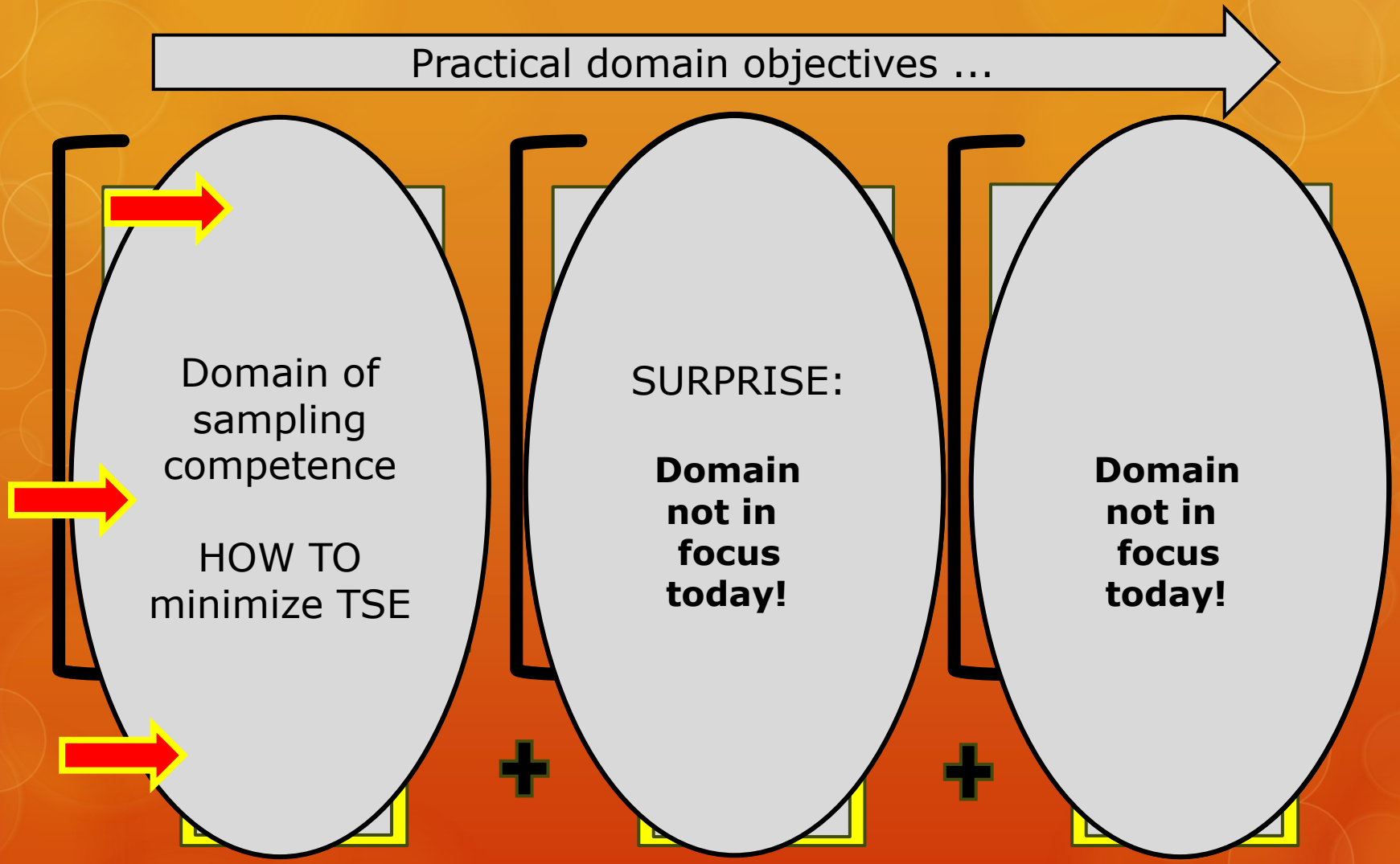
Today's take-home lessons … …

- **The analytical aliquot** is the physical manifestation of transgressing the boundary from the *before analysis* (sampling) domain to the domain of analysis
- **The analytical aliquot** is the only, ***miniscule*** portion of the original target material that is actually analysed

… It is **all** about the analytical aliquot …

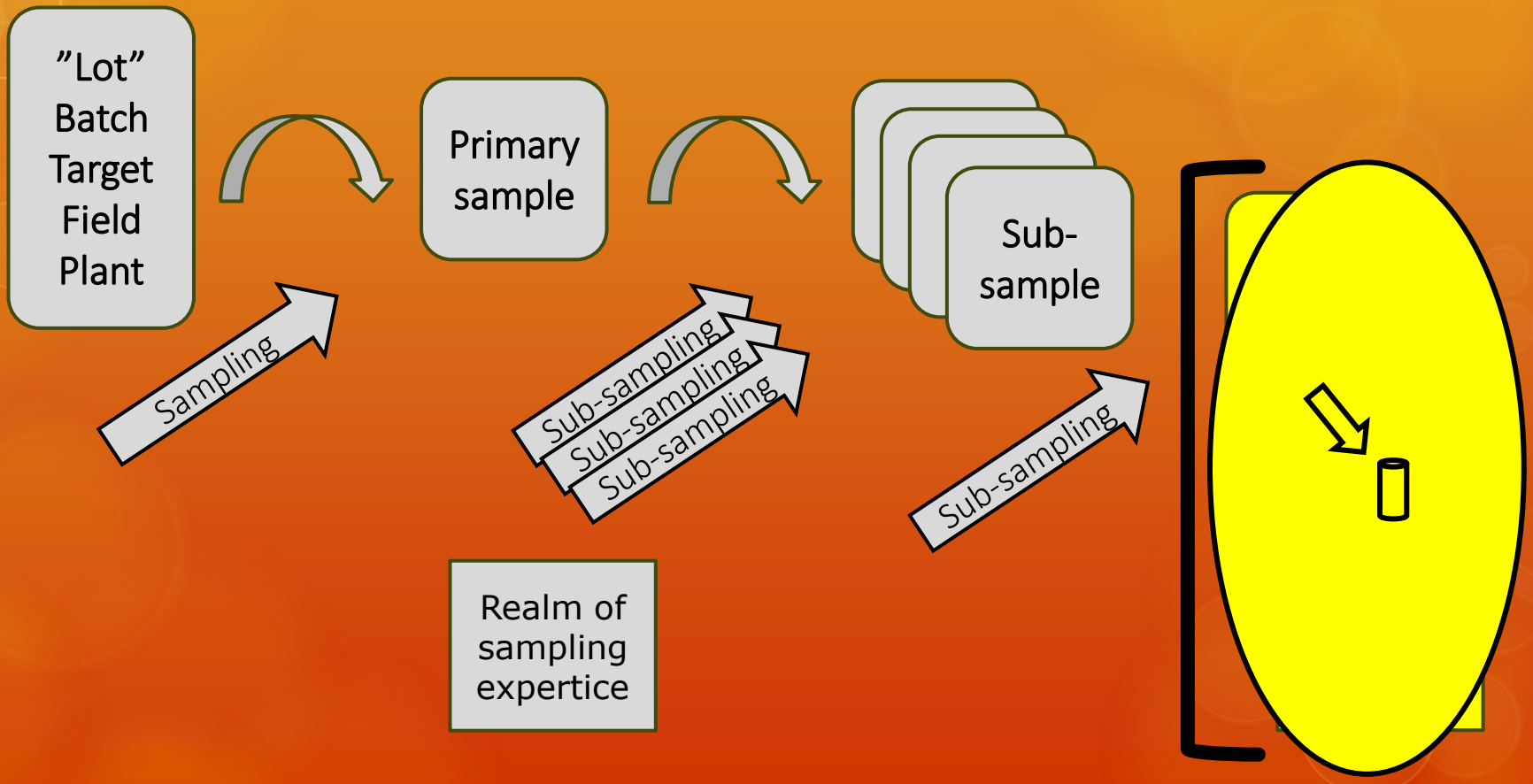
- It is only possible to guarantee representativity of the analytical aliquot, and thus of the analytical results w.r.t. the **original target batch/lot/process stream**
- … by invoking the necessary sampling competence described in the **Theory of Sampling (TOS)**


Three domains behind 'data quality'
Before analysis Domain
Sampling / subsampling Domain
Domain of Analysis
- chemical
- physical
- other
.....
Post Analysis Domain
Statistics
Data analysis
Chemometrics

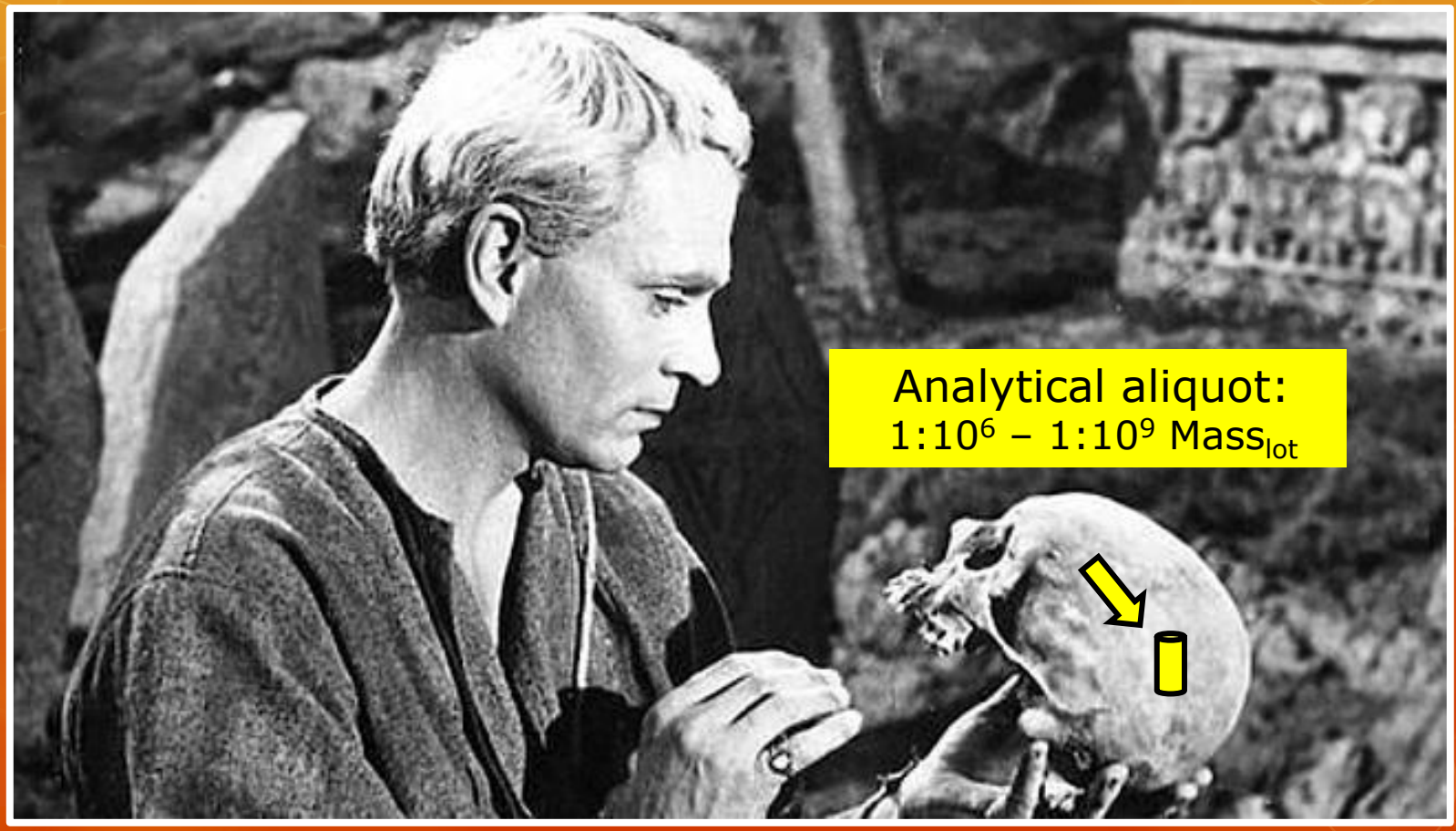

Practical domain objectives ...
Domain of sampling competence
HOW TO minimize TSE
SURPRISE:
Domain not in focus today!
Domain not in focus today!

Sampling/sub-sampling of .....

heterogeneous/lots materials



”To be, or not to be ... representative” THAT is the question!



Stationary lot

Moving lot / dynamic lot

Sampling rate (typical)

Primary sample

Secondary sample

Tertiary sample / aliquot

$10^3 – 10^6$

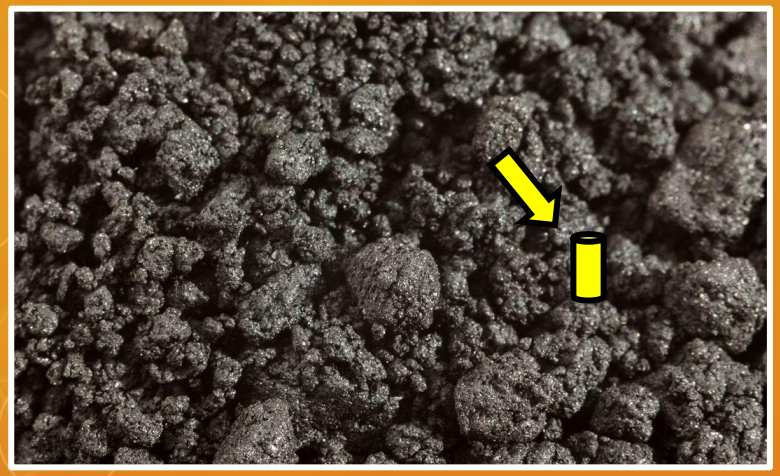
$10^1 – 10^2$

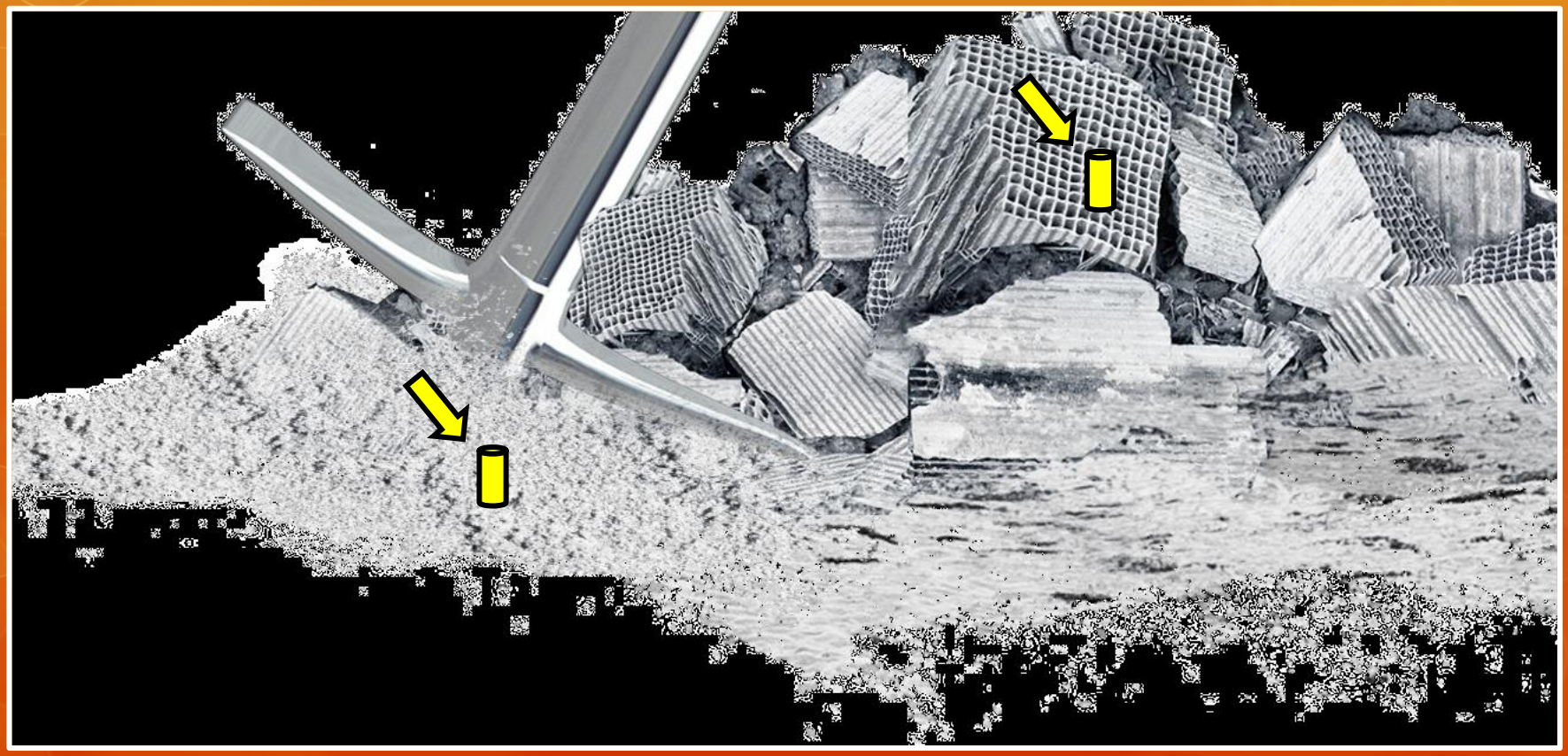
$10^1 – 10^2$

$10^3 – 10^6 - 10^9$

Traditional laboratory domain

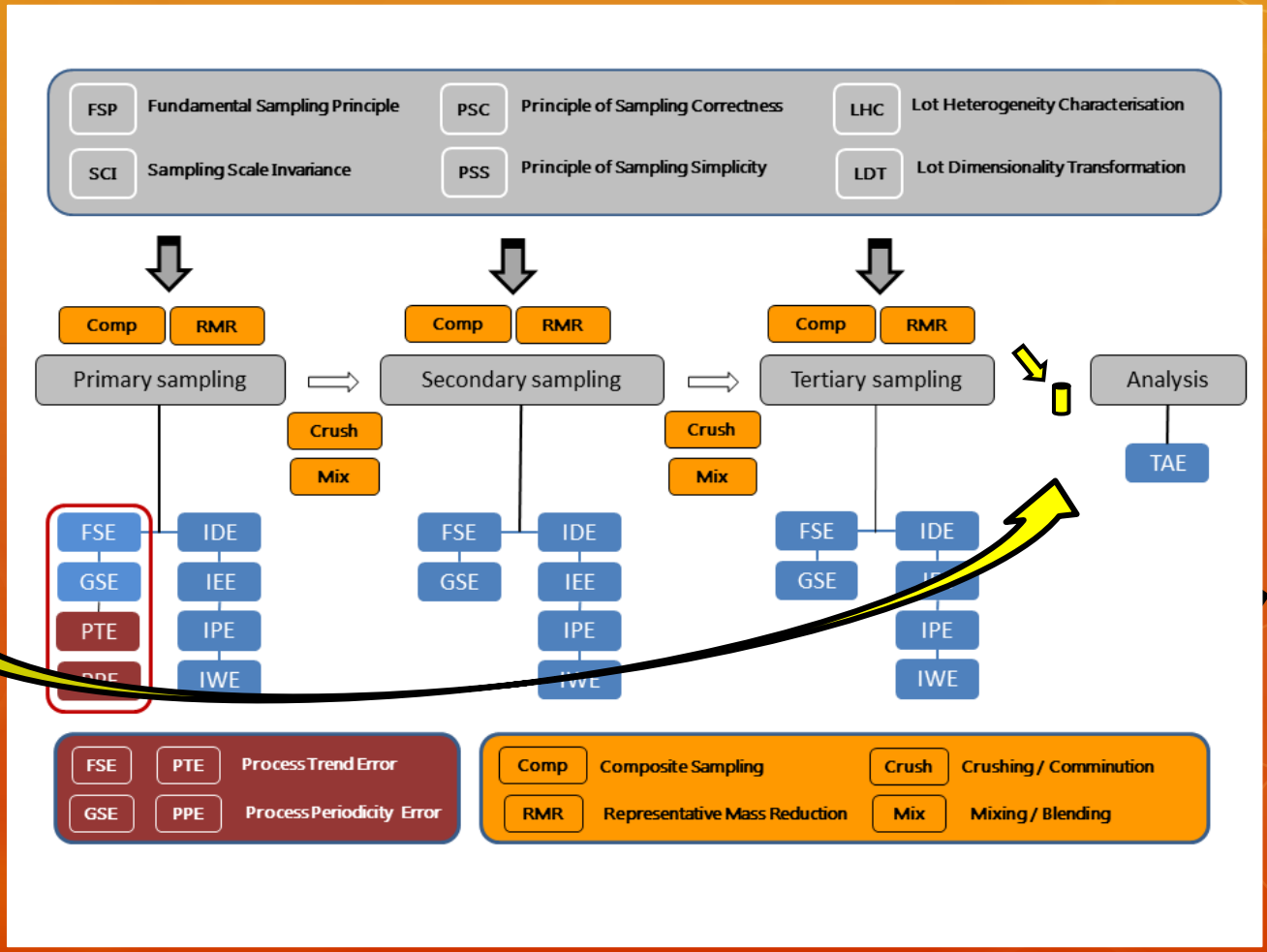
Imperative demand: Mass reduction must be representative !!!





Theory of Sampling (TOS) – everything in a glance

"Lot"
Batch
Target
Field
Plant



HOW TO GET A REPRESENTATIVE SAMPLE?

(Food, Feed, Pharma, Chemical, Polymer, Exploration, Environment, Mining, Metals - *process industries* o.a.)

HOW TO GET A REPRESENTATIVE ANALYTICAL ALIQUOT?

Theory of Sampling (TOS) – the critical success factor *before analysis*

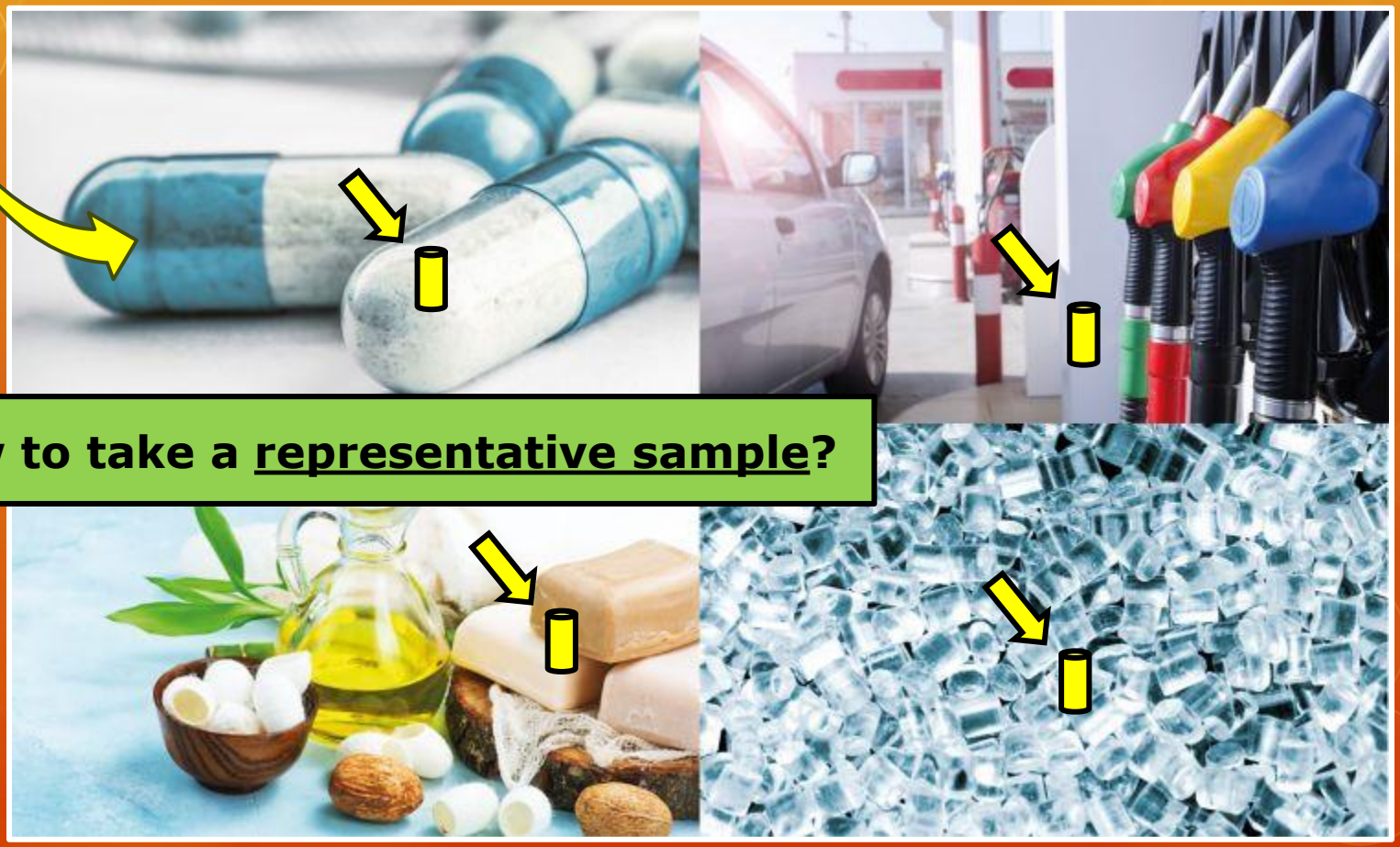
Critical issue ... Scope and Focus

- Accuracy – a tribute only related to analysis of the aliquot [TAE]
- Accuracy – a tribute related to the full, compound [sampling/subsampling/analysis] process [TSE + TAE]

- Precision – an attribute only related to analysis [~TAE]
- Precision – an attribute related the full, compound [sampling/subsampling/analysis] process] [TSE + TAE]

Need a shift in focus .....
From: Analytical responsibility (*only*) –
To: Sampling-and-Analytical responsibilities (*both*)

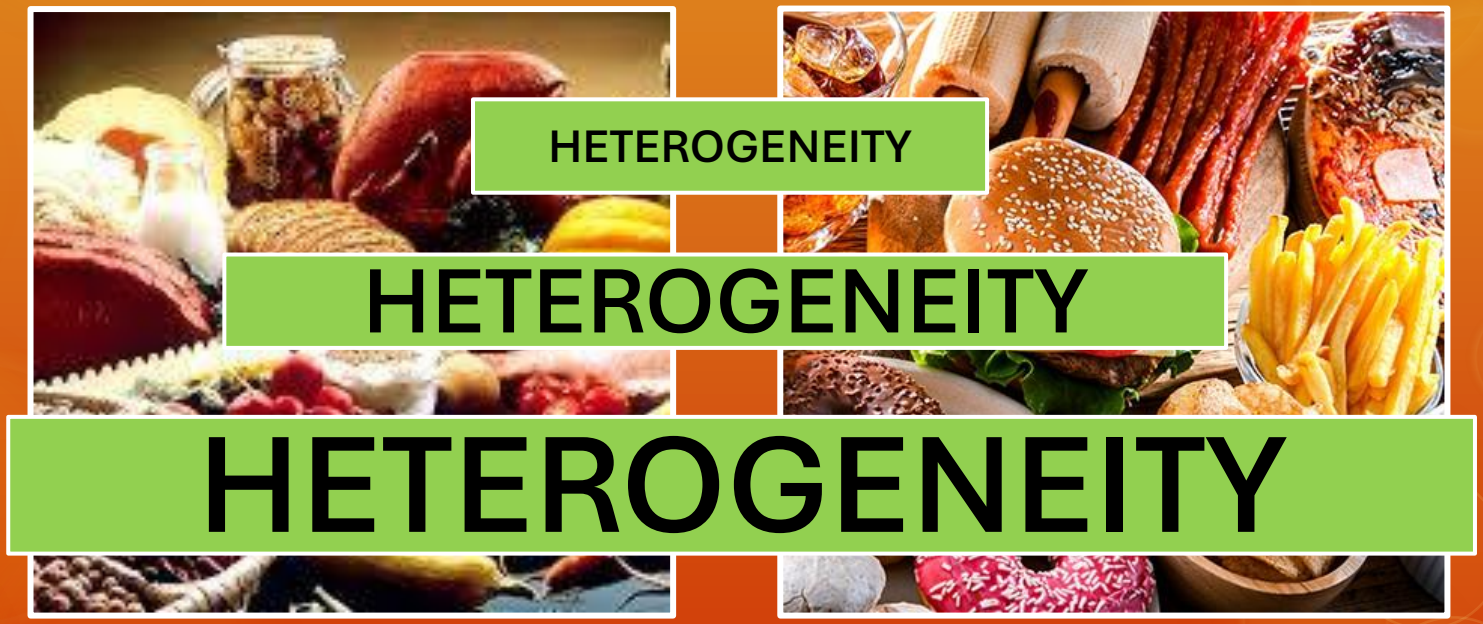
”Take a sample of the lot / target material, bring it to the lab + some sample preparation – present the aliquot to the analytical instrument” - et voila”



How to take a <u>representative sample</u>?


What’s the common characteristic of most materials destined to be analysed?
How to take a representative sample?

WHAT characterises e.g. food/feed/agri/pharma/chemical/ polymer/mineral industry materials, and 99.50% of <u>all</u> others?



All materials met with in science, technology and industry are **heterogeneous**, it is only a matter of to which degree ... ...

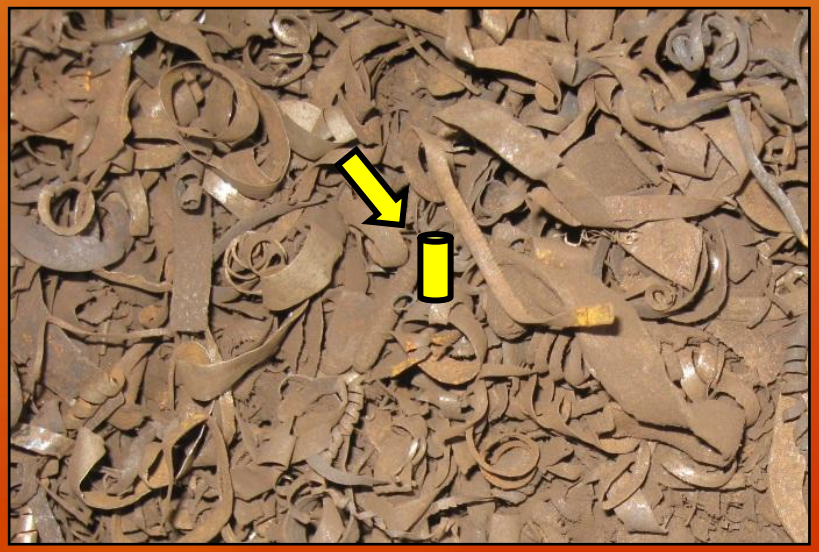
Need a shift in focus .....

From: Analytical responsibility (*only*) –
To: Sampling-and-Analytical responsibilities (*both*)

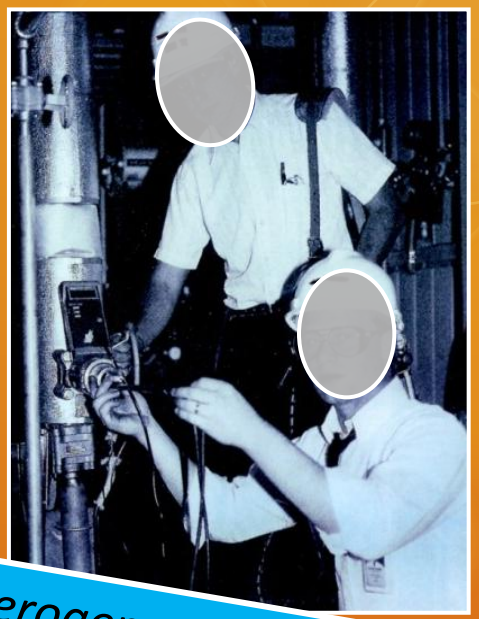
The sampling process must be able to deal appropriately with heterogeneity

Nothing is gained by sallying forth with the aim to get the analytical aliquot - in one step ...

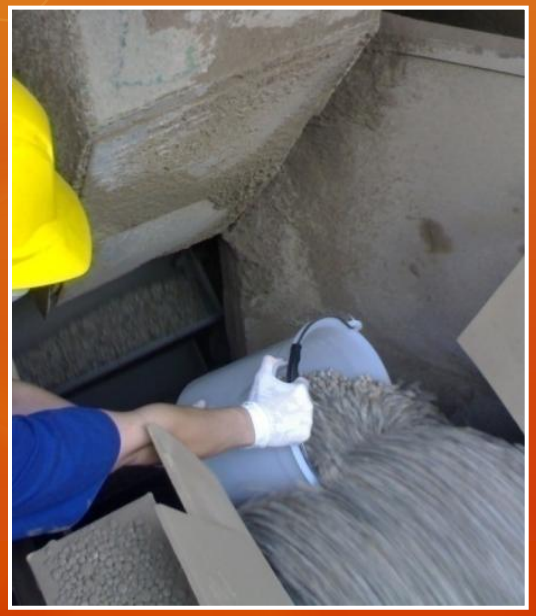
Nothing is gained by sallying forth with the aim to get the analytical aliquot in one step ...



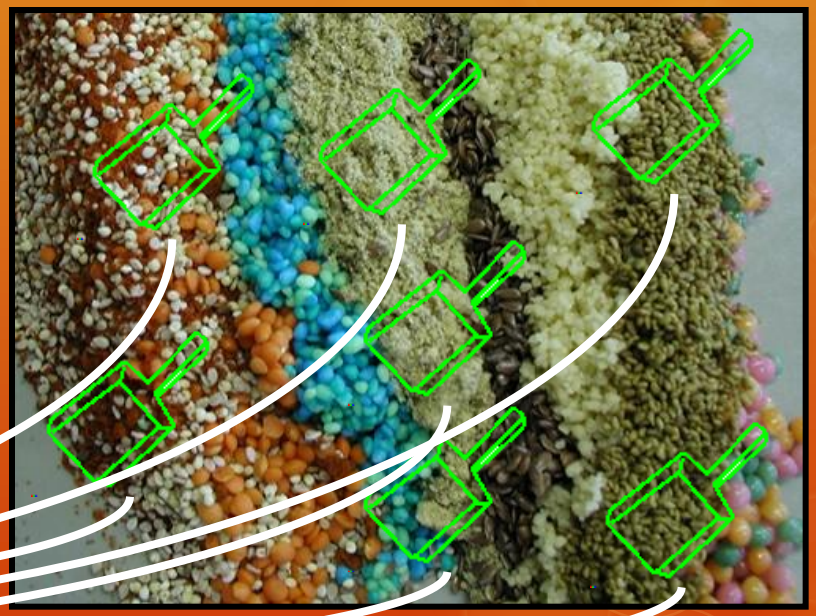
Examples of the futility of the most often met with one-step approach:
”grab sampling”



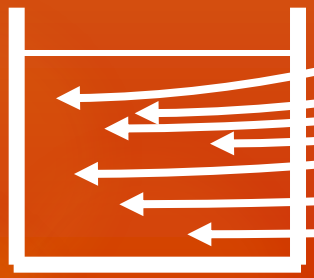
A **grab sample** can never be representative of a *heterogeneous* material



Sampling Unit Operation 1: Composite Sampling

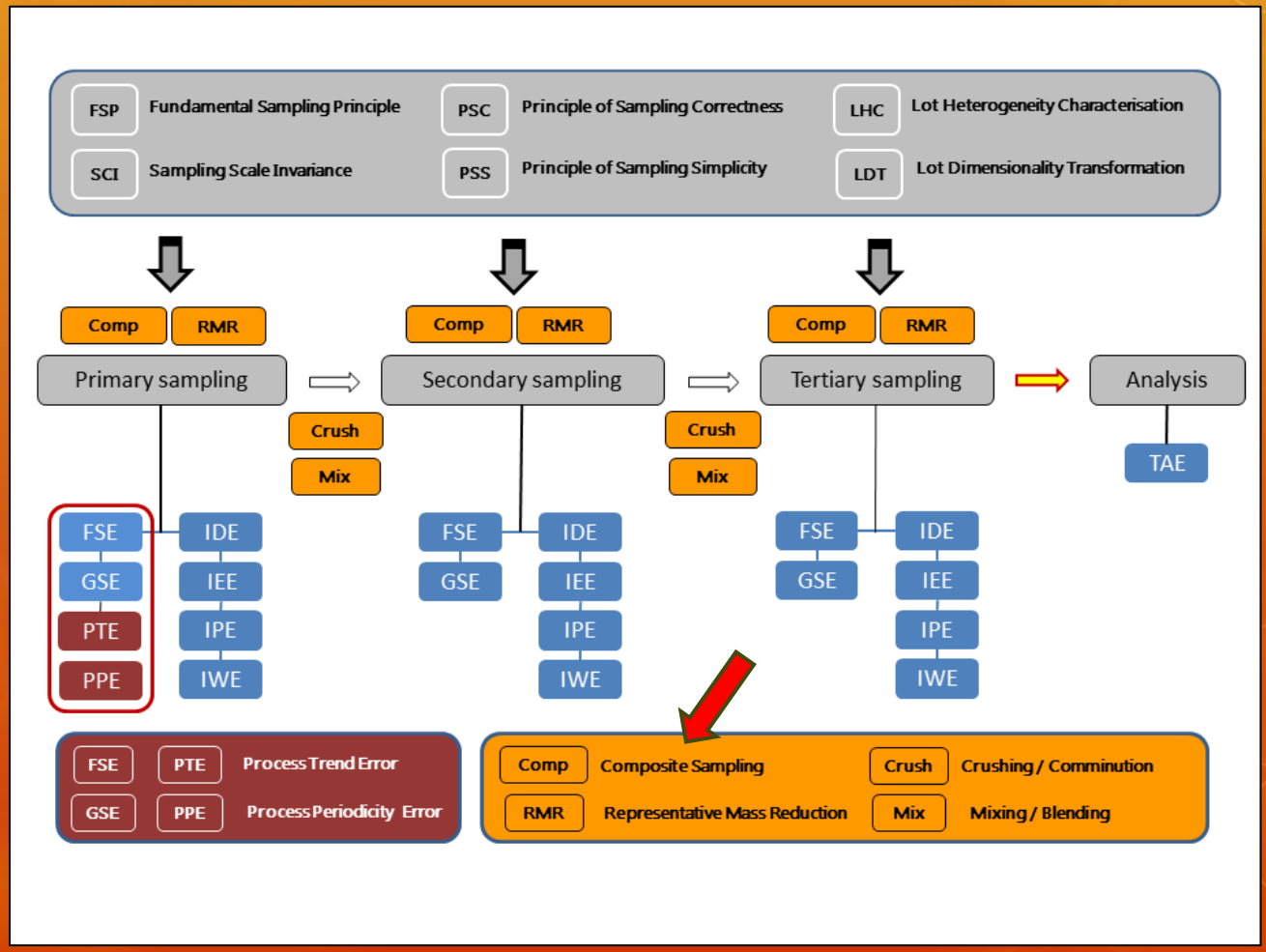


"Grab Sampling" - "not thinking"



… … Composite Sampling … …

Theory of Sampling (TOS) – everything in a glance



Lot-to-aliquot: Need for representative sampling !!!

1. Sampling is never a one-shot operation (grab)

2. Sampling is always a multi-stage process (comp):

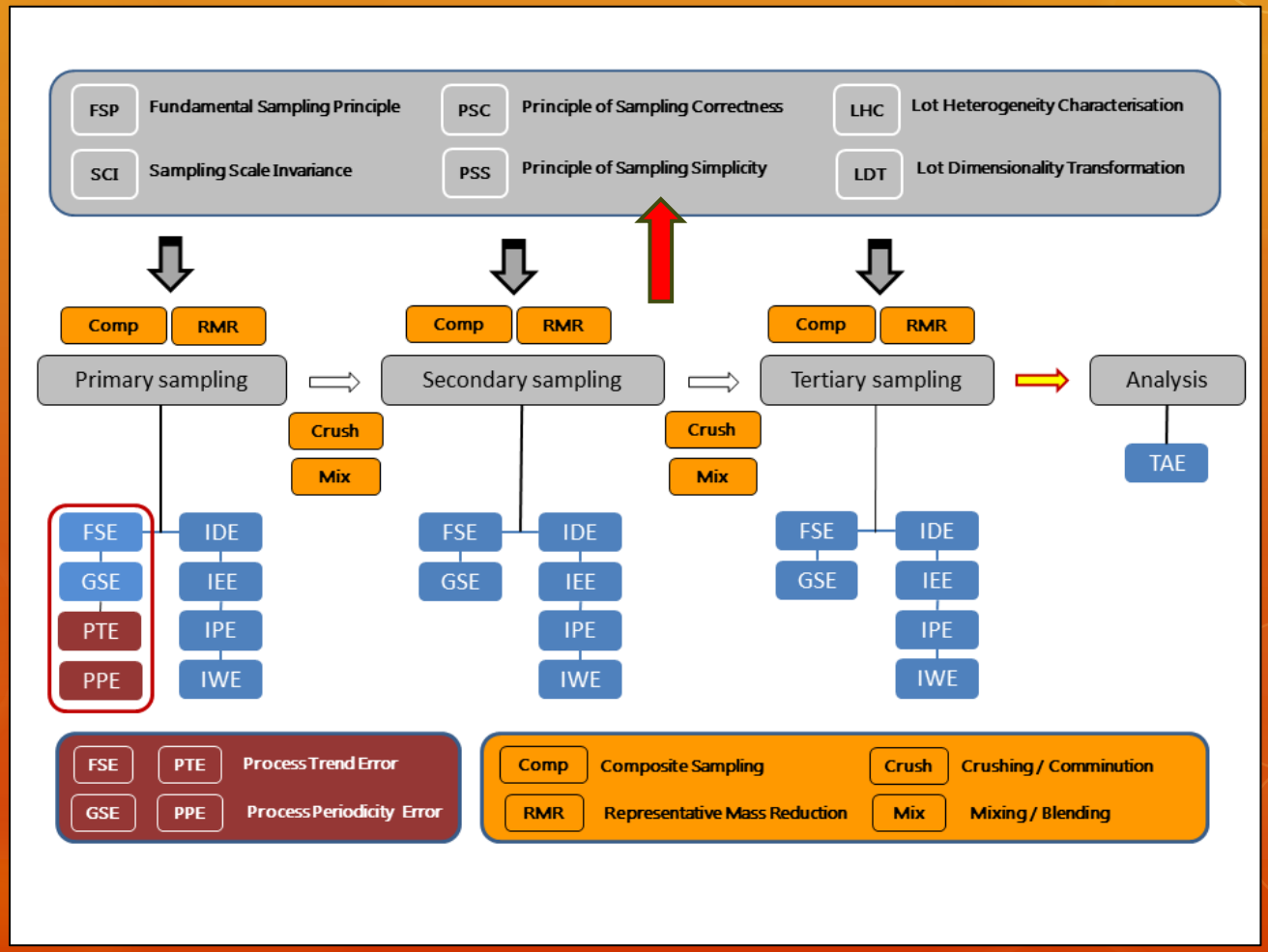
   i) Primary sampling +

   ii) Representative Mass-reduction

Sample splitting/division/sub-sampling ...

Princple of Sampling Simplicity (PSI)

Theory of Sampling (TOS) – everything in a glance



FSP Fundamental Sampling Principle
PSC Principle of Sampling Correctness
LHC Lot Heterogeneity Characterisation
SCI Sampling Scale Invariance
PSS Principle of Sampling Simplicity
LDT Lot Dimensionality Transformation

Comp RMR
Primary sampling
Crush
Mix
Comp RMR
Secondary sampling
TOS
Crush
Mix
Comp RMR
Tertiary sampling
Analysis
TAE

FSE GSE PTE PPE
IDE IEE IPE IWE
FSE GSE
IDE IEE IPE IWE
FSE GSE
IDE IEE IPE IWE

FSE PTE Process Trend Error
GSE PPE Process Periodicity Error

Comp Composite Sampling
Crush Crushing / Comminution
RMR Representative Mass Reduction
Mix Mixing / Blending

Intended to cover ALL types of materials (stat. and moving)

Competence building / philosophy

Governing Principles (GP): 6

Sampling Unit Operations (SUO): 4(5)

- **"Once a principle has been acquired it becomes a part of you and is never lost. It can be applied to novel problems and never becomes obsolete as do all facts in a changing world (science, technology, industry, commerce, regulation)**

TOS

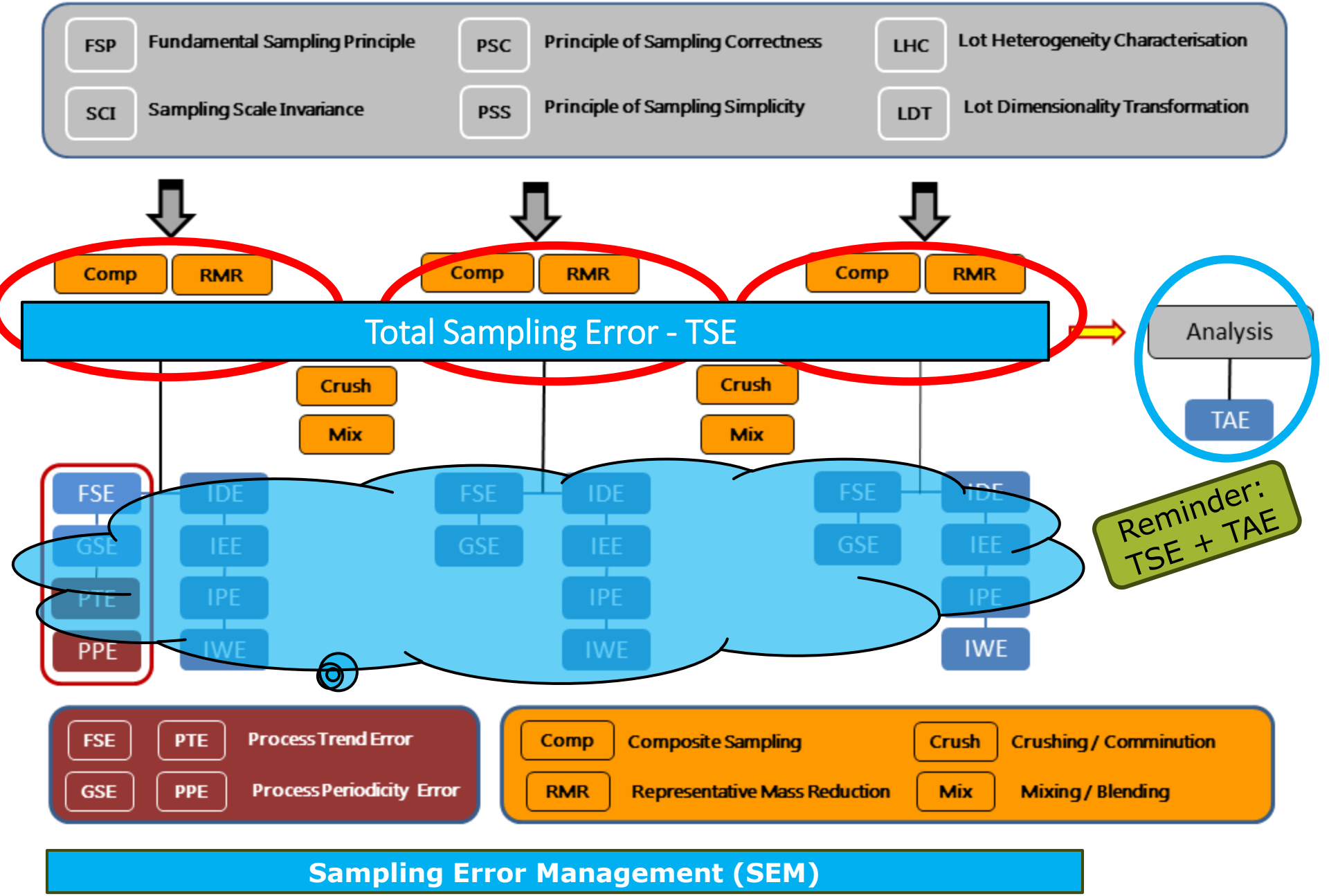
- **A well-trained mind can cope with many unforeseen problems**

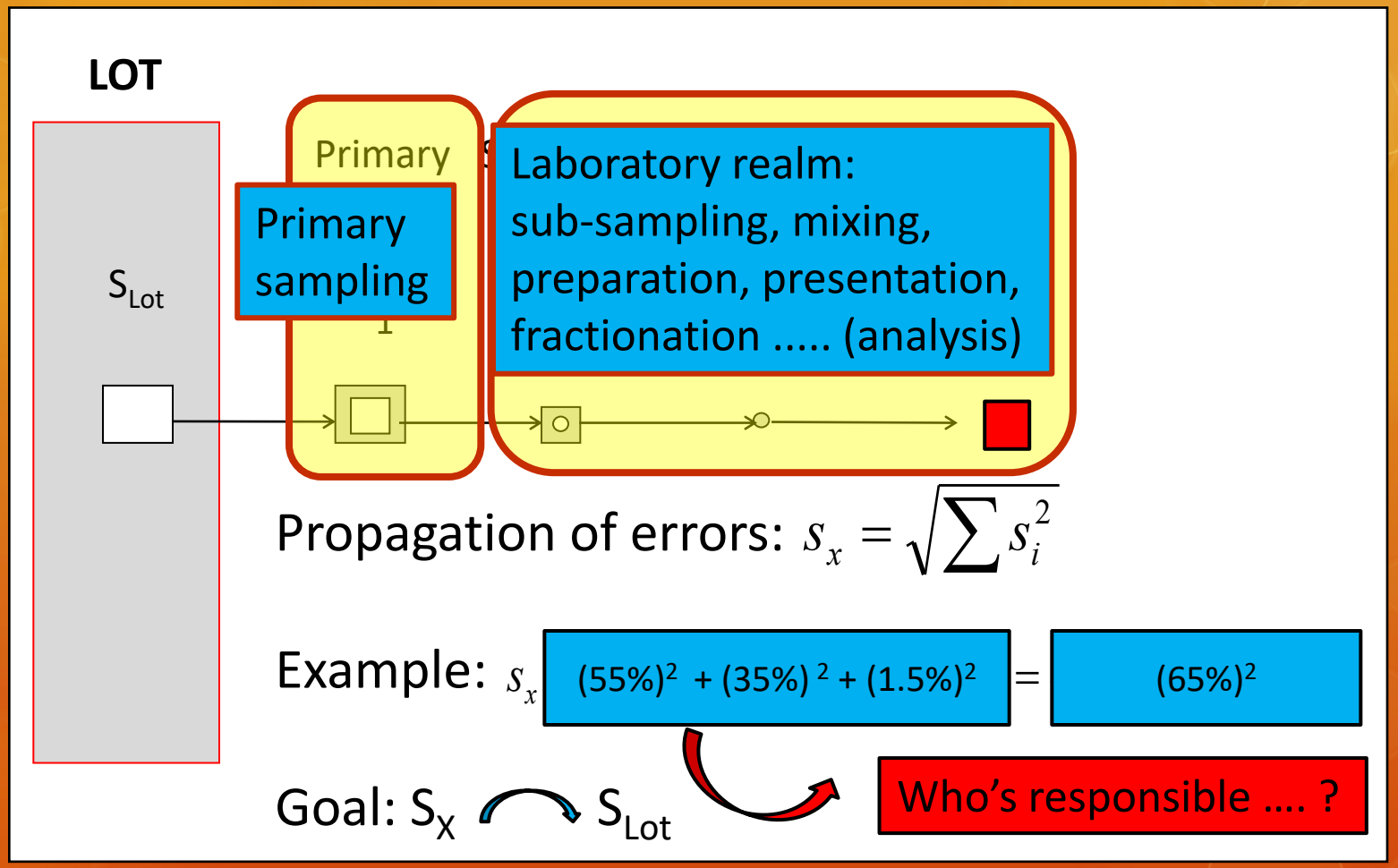
TOS

- **In a rapidly changing world, no one can foresee what future problems will have to be met"**

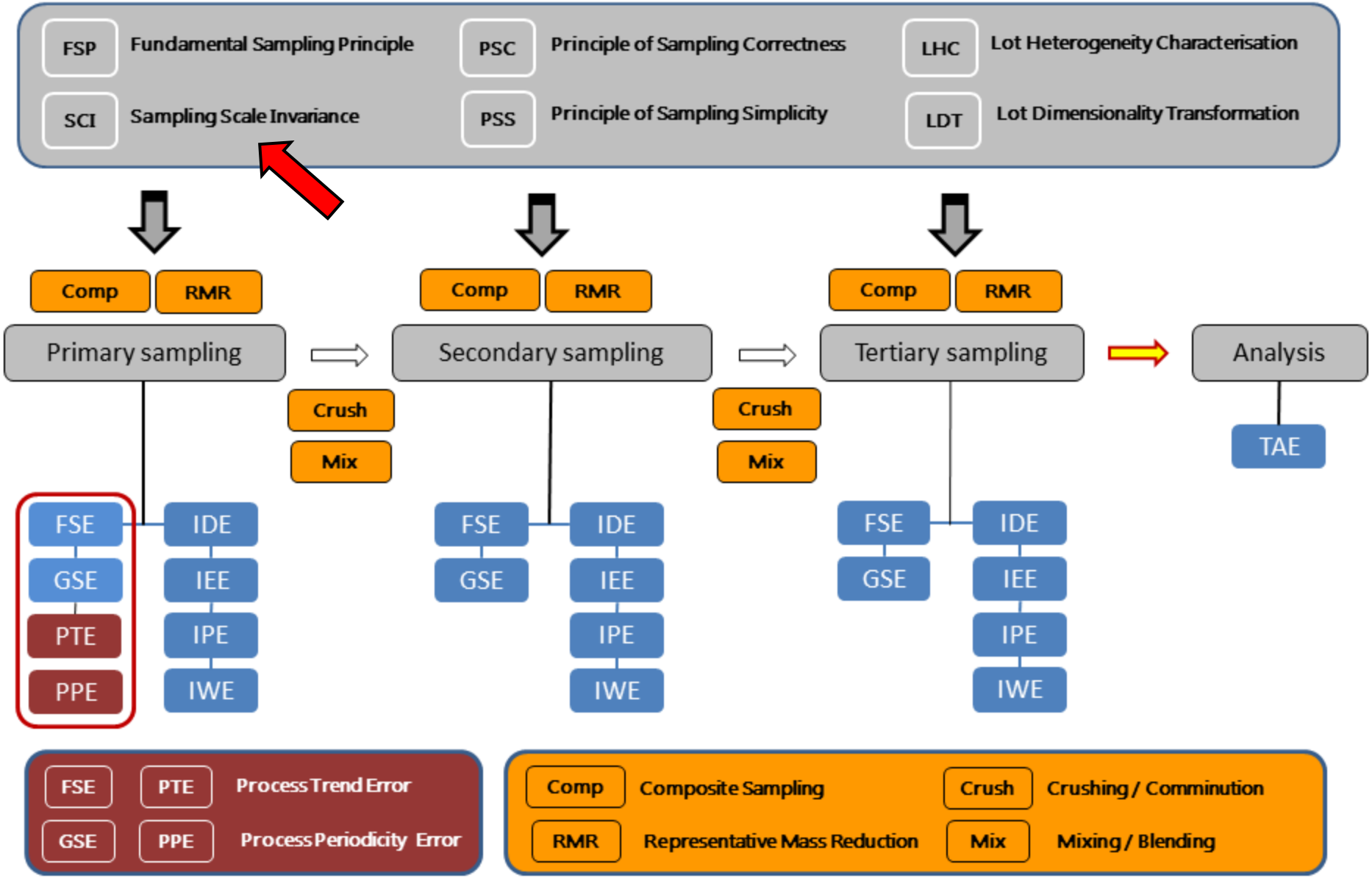
TOS

Sampling Error Management rules (SEM)


FSP
Fundamental Sampling Principle
PSC
Principle of Sampling Correctness
LHC
Lot Heterogeneity Characterisation
SCI
Sampling Scale Invariance
PSS
Principle of Sampling Simplicity
LDT
Lot Dimensionality Transformation
Comp
RMR
Comp
RMR
Comp
RMR
Total Sampling Error - TSE
Crush
Mix
Crush
Mix
Analysis
TAE
FSE
GSE
PTE
PPE
IDE
IEE
IPE
IWE
FSE
GSE
IDE
IEE
IPE
IWE
FSE
GSE
IEE
IPE
IWE
Reminder:
TSE + TAE
FSE
PTE
Process Trend Error
GSE
PPE
Process Periodicity Error
Comp
Composite Sampling
Crush
Crushing / Comminution
RMR
Representative Mass Reduction
Mix
Mixing / Blending
Sampling Error Management (SEM)



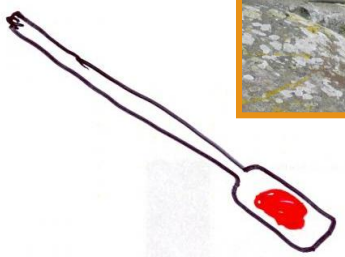
The analytical process always contains several sampling and preparation steps, but usually primary sampling dominates



... TOS in the analytical laboratory ...

Sampling equipment – must be strictly c...

"UNBIASED" ;-)



Sampling Scale Invariance (SSI)

Sampling <u>in the laboratory</u>: What's the difference w.r.t. <u>field/plant sampling</u>?

Field samplers and laboratory personnel → <u>same</u> tasks !

Theory of Sampling (TOS)

Why is TOS the critical success factor before analysis?

- for the **seller**; for the **buyer**; for the **middleman**, for the **arbiter**?
- for the **company**; **customer**; **scientist**; **technician – for management**?
- for **science, technology**, **industry, compliance**; **risk management**; **society?**

- indeed: also for the analyst!


DS
DANSK STANDARD
Dansk standard
DS 3077:2024
2024-10-13
Repræsentativ prøvetagning – Horisontal standard
Representative sampling – Horizontal standard
www.ds.dk
© Fonden Dansk Standard – Eftertryk uden tilladelse forbudt

Horizontal – Representative Sampling

DS-3077

This standard outlines a practical, iterative, self-controlling approach with minimal complexity, based on the Theory of Sampling (TOS). The generic sampling process described and all elements involved are sufficient and necessary for the stated objective, with the consequence that no exceptions can be allowed in order to be able to document the intended sampling representativity. It is necessary to consider the full pathway from primary sampling to analytical results in order to be able to guarantee a reliable and valid analytical outcome. This standard, including normative references, annexes (and further, optional references) constitute a complete and sufficient competence basis for this purpose. The present approach will ensure appropriate levels of accuracy and precision for both primary sampling as well as for all sub-sampling procedures and mass-reduction systems at the subsequent laboratory stages before analysis.

DS
DANSK STANDARD

Dansk standard

DS 3077:2024

2024-10-13

3.43
theory of sampling
TOS
framework of *governing principles (GP)* (3.16), *sampling unit operations (SUO)* 3.41and *sampling error management* rules (SEM) together with normative practices needed to overcome adverse effects of material heterogeneity and incorrect sampling procedures

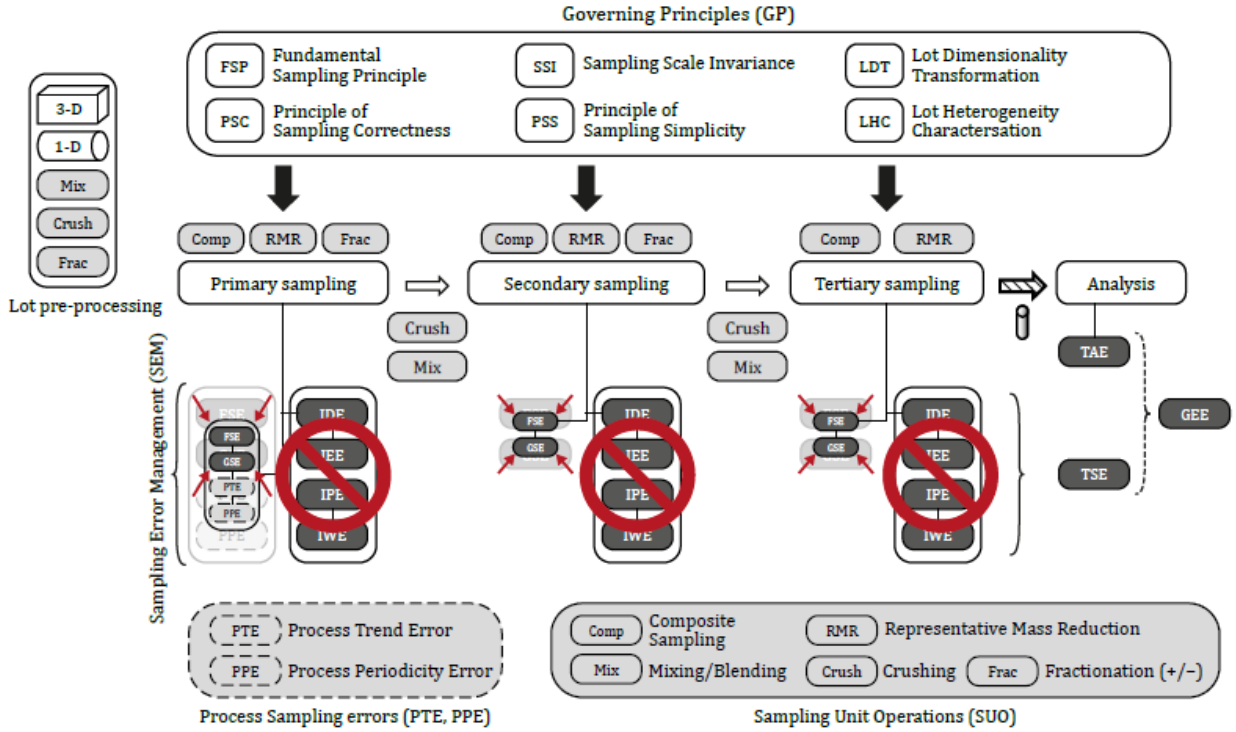
Note 1 to entry: TOS allows to design, implement, use, and maintain proper sampling processes and equipment ensuring sampling representativeness.

Note 2 to entry: TOS history: Body of theoretical developments initiated in 1950 by the French scientist Pierre Gy, who, over the period of the following 25 years, developed a complete theory of heterogeneity, necessary and sufficient principles and operations guaranteeing representative sampling, including appropriate sampling equipment requirements (design principles, specific operational and maintenance requirements), see the Bibliography [1, 3-7, 9, 12, 13, 15, 17 and 24].

3.44
total analytical error
TAE
sum of all uncertainty effects due to a specific analytical measurement made on an analytical aliquot

Note 1 to entry: Aliquots can be extracted as physical sub-samples, or can be intact virtual samples, e.g., a volume of a flux of matter interacting with a spectroscopic analytical instrument, or similar (PAT). TAE reflects the uncertainty effects involved in analytical method validation *per se*. TAE is specifically singled out in TOS to avoid overlapping, or confusion with respect to TSE.

3.45
total sampling error
TSE
combined uncertainty effects resulting from material extraction along the full sampling pathway from-lot-to-aliquot TSE = ISE + CSE, see Figure 1.



Key

FSP Fundamental Sampling Principle (GP1)
PSC Principle of Sampling Correctness (GP2)
SSI Principle of Sampling Scale Invariance (GP3)
PSS Principle of Sampling Simplicity (GP4)
LDT Principle of Lot Dimensionality Transformation (GP5)
LHS Principle of Lot Heterogeneity Characterisation (GP6)
SUO 1 Composite sampling (Comp)
SUO 2 Representative Mass Reduction (RMR)
SUO 3 Crushing (Crush) (comminution, milling, grinding)
SUO 4 Mixing/blending (Mix)
SUO 5 Fractionation (Frac)
3-D stationary lots
1-D dynamic lots

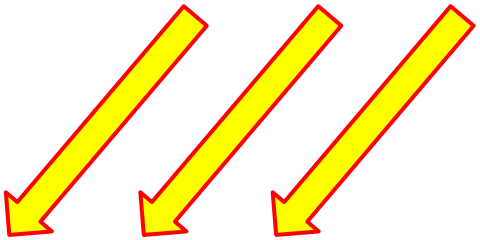
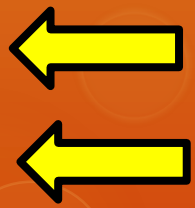


Figure 11 – TOS' requirements for fit-for-purpose representativity (compare with Figure 1)

New insights ...

- Analytical results ... [the end-result of a very long, often tortous pathway 'from-lot-to-aliquot'] form the basis for important decision-making across all of society, industry, commece, trading, compliance regulation – decisions of great political, economic, health, resource importance.

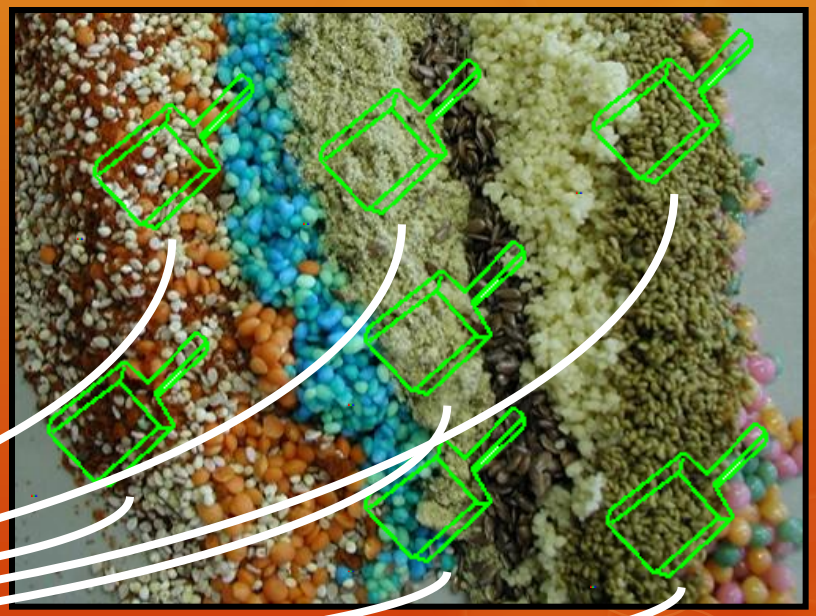
- Analytical results *depend* on the sampling pathway ...
- Analytical results *depend* on the sampling process:
  - representative, or – non-representative sampling?



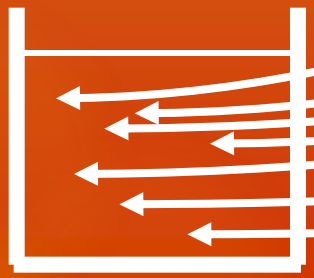
This may at first cause confusion and consternation 'in the management, legal and accounting offices' – but there is only one scientific and logical conclusion

Analytical results – depend on the sampling process

$a_{S(grab)} \neq a_{S(comp1)} \neq a_{S(comp2)}$



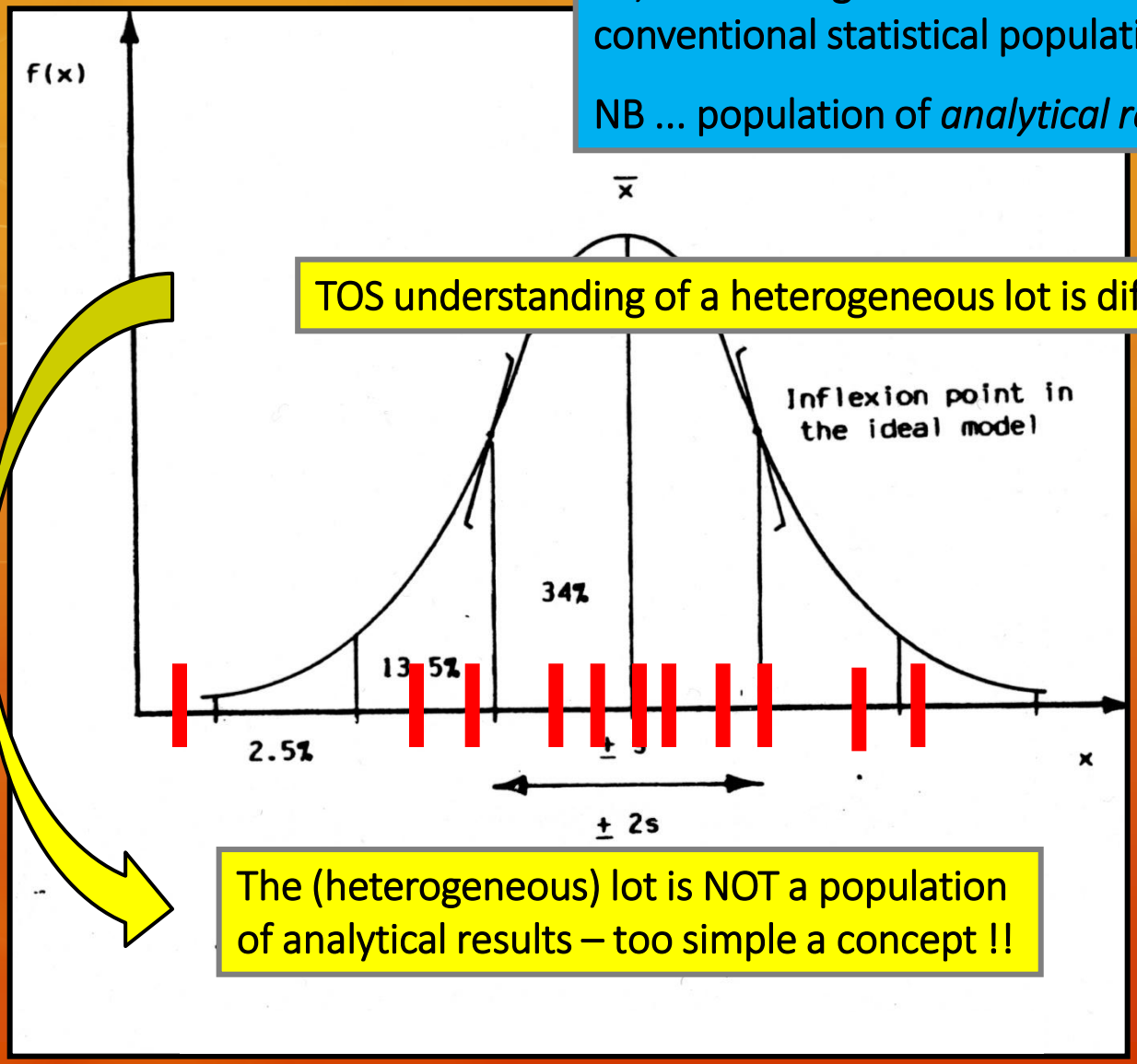
"Grab Sampling"



"Composite Sampling"

So, even though is is often difficult to avoid conventional statistical population thinking

NB … population of *analytical results* …. NB



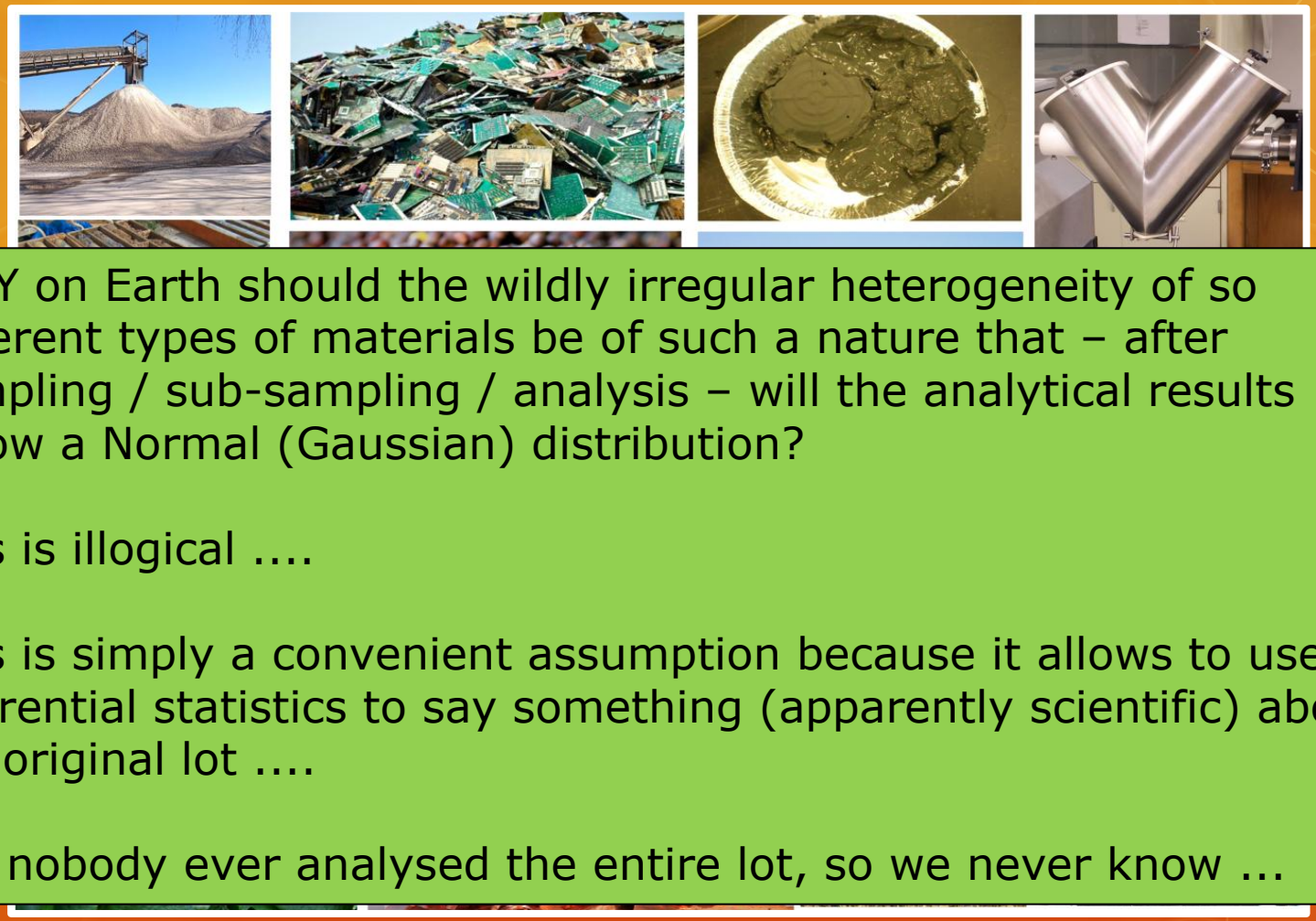
TOS understanding of a heterogeneous lot is different !

The (heterogeneous) lot is NOT a population of analytical results – too simple a concept !!

Understanding the heterogeneity of a material lot is <u>not</u> a task where trying to throw some statistics in the pot will help …

Because:

The complete lot-to-aliquot pathway has been 100% ignored!



WHY on Earth should the wildly irregular heterogeneity of so different types of materials be of such a nature that – after sampling / sub-sampling / analysis – will the analytical results follow a Normal (Gaussian) distribution?

This is illogical ....

This is simply a convenient assumption because it allows to use inferential statistics to say something (apparently scientific) about the original lot ....

But nobody ever analysed the entire lot, so we never know ...

All materials met with in science, technology and industry are **heterogeneous**, it is only a matter of to which degree ... ...

And word (of caution) …

- One does not 'collect' a sample ….
- One employs a **TOS-compliant sampling process**!

- One does not 'take' a sample …
- One employs a **TOS-compliant sampling process**!

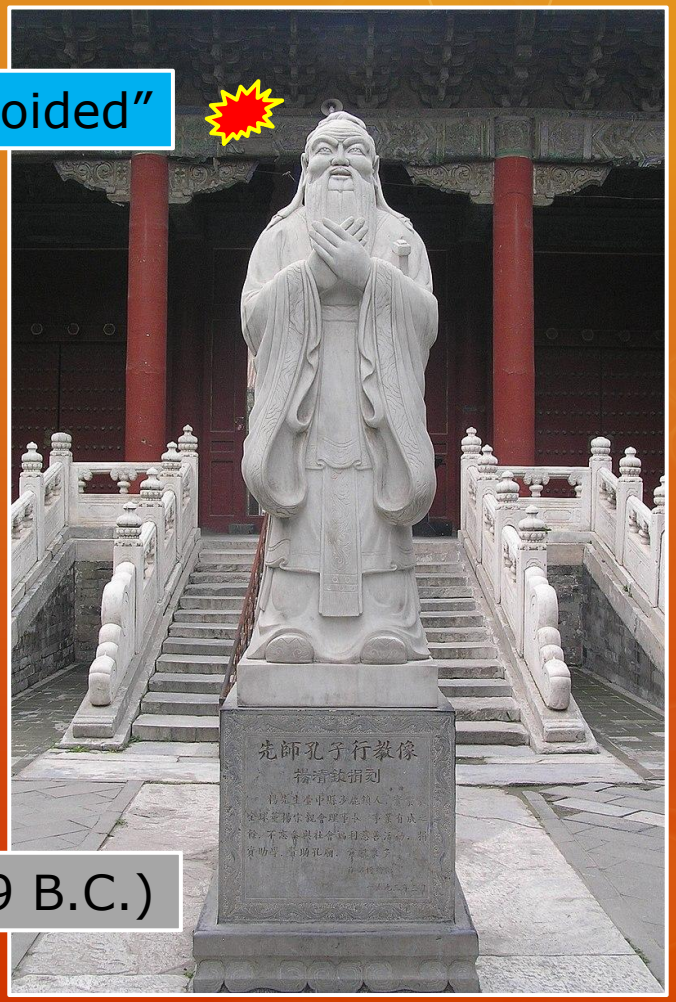
- Explanation: 'Samples' do not reside in the lot, ready to be 'taken, 'collected', or 'extracted' ….

One employs a **TOS-compliant sampling process**!

For this purpose, (also) analysts must have a minimum of TOS competence [**sorry for a little hard work**]

TOS – Definitions...

"Speak precisely – and wars can be avoided"



Kunfutze [Kong Futzi] (551 B.C. – 479 B.C.)

- Lot – material, size (mass), geometrica form, CH, DH, H_{grz}
- Fundamental unit – original particle, grain, fragment
- Fragment - result(s) of fragmentation of original unit(s)

'Fragments'

- Group (of fragments) – theoretical, virtual sampling increment

- Sub-sample – result of a division process/sub-process
- Increment – result of a aggregation process/sub-process

- Grab sample –
- Composite sam

TOS

$$SE = \frac{(a_S - a_L)}{a_L}$$

'Representativity' (def)

$$r^2(SE) = m^2(SE) + s^2(SE)$$

- Sample – result of a representative sampling process (grab, comp.)
- Specimen – result of a non-representative sampling process

(Under development) … … …

CRITICAL UNDERSTANDING … …

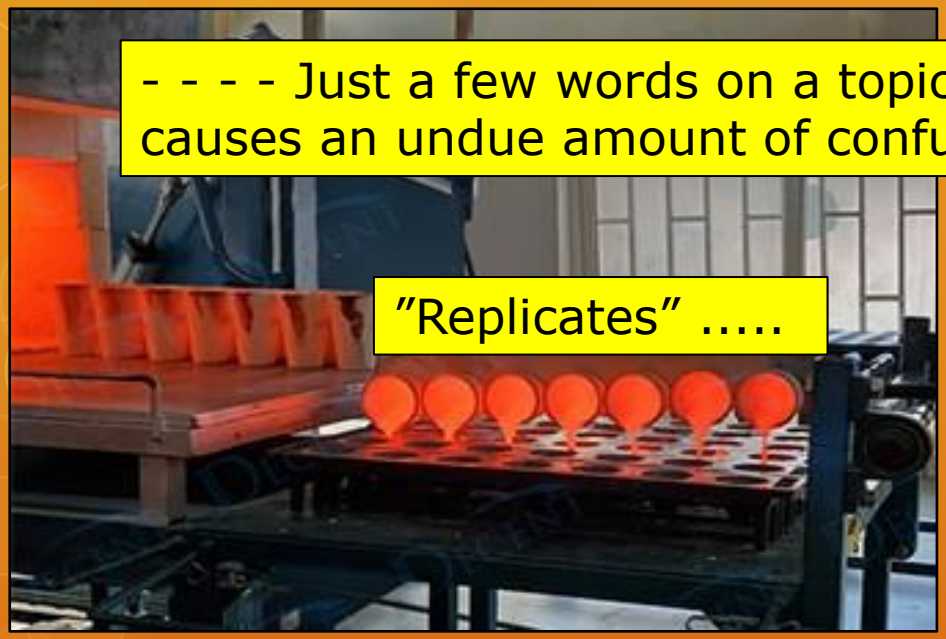
It is not possible to ascertain whether a sample is representative, or not – from any characterisation or evidence based on the sample itself !!!

It is only possible to demonstrate whether a *specific sampling process is* representative, or not …

… a specific sampling process

Sampling expert's job:

Audit the **sampling process(es)** – TOS compliance, or not …



- - - - Just a few words on a topic that causes an undue amount of confusion!

”Replicates” .....

Theory and Practice of Representative Sampling:

Duplication / Replication

Enormous confusion in the scientific literature !!

… Duplicate analysis / Duplicate analyses

… Duplicate sampling / Duplicate samples

… Replicate sampling / Replicate samples

… Replication from whichsampling stage … ???

TOS

FSP Fundamental Sampling Principle

PSC Principle of Sampling Correctness

LHC Lot Heterogeneity Characterisation

SCI Sampling Scale Invariance

PSS Principle of Sampling Simplicity

LDT Lot Dimensionality Transformation

Comp RMR

Primary sampling

Comp RMR

Tertiary sampling

Analysis

TAE

Replication Experiment

FSE GSE PTE PPE IDE IEE IPE IWE

FSE GSE IDE IEE IPE IWE

FSE GSE IDE IEE IPE IWE

FSE PTE Process Trend Error

GSE PPE Process Periodicity Error

Comp Composite Sampling

RMR Representative Mass Reduction

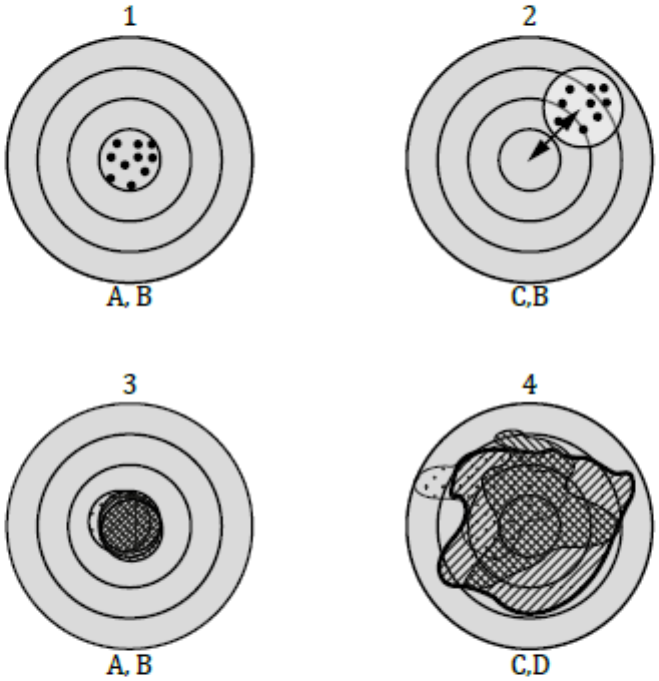
Crush Crushing / Comminution

Mix Mixing / Blending

The **sampling bias** –is not (at all) like the analytical bias. Fundamentally different !!

A systematic overview of issues that distinguish the sampling bias from the analytical bias starts with acknowledging the dual set of accuracy & precision uncertainties:

- analytical accuracy and precision
- sampling accuracy and precision

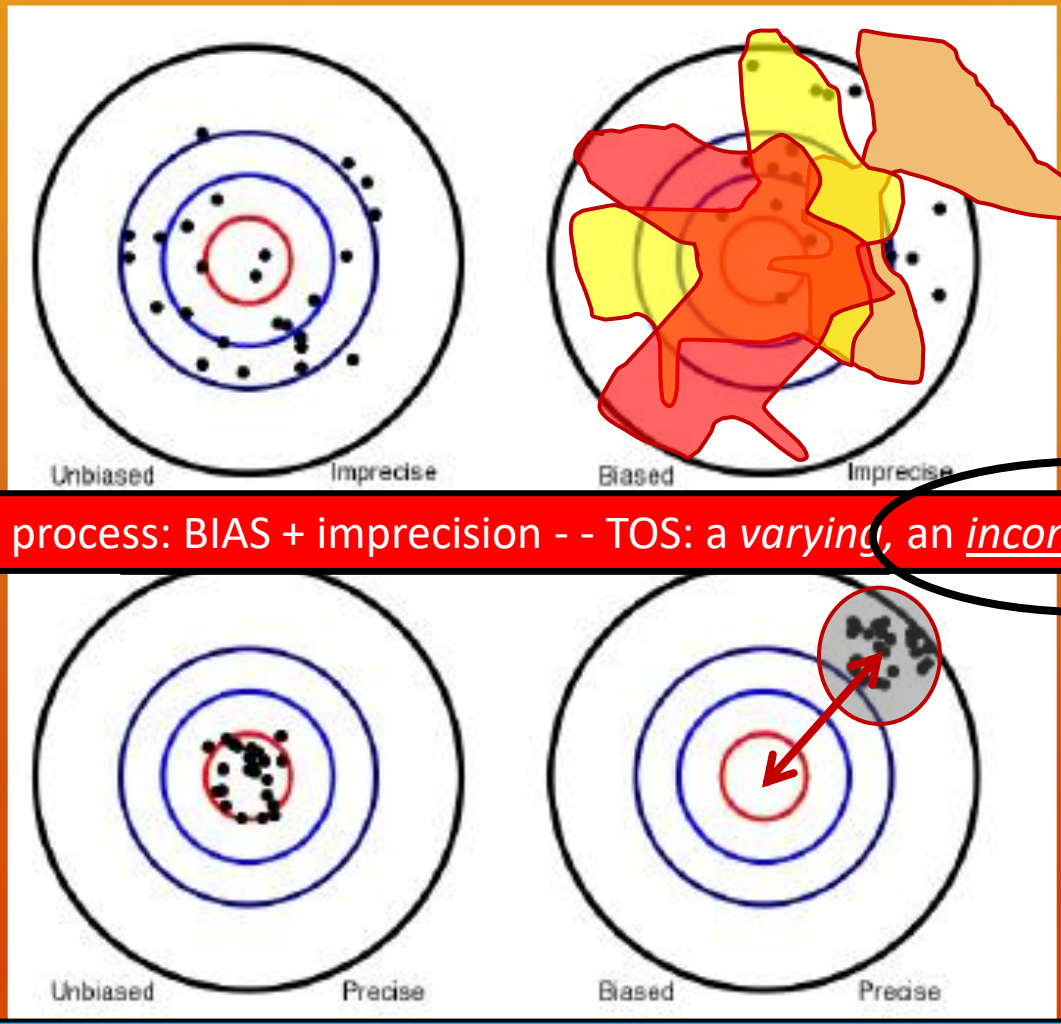


Key

1, 2	characteristics of the analytical bias
3, 4	characteristics of the sampling bias (renderings of RE envelopes)
A	accurate
B	precise
C	biased, inaccurate
D	biased, imprecise

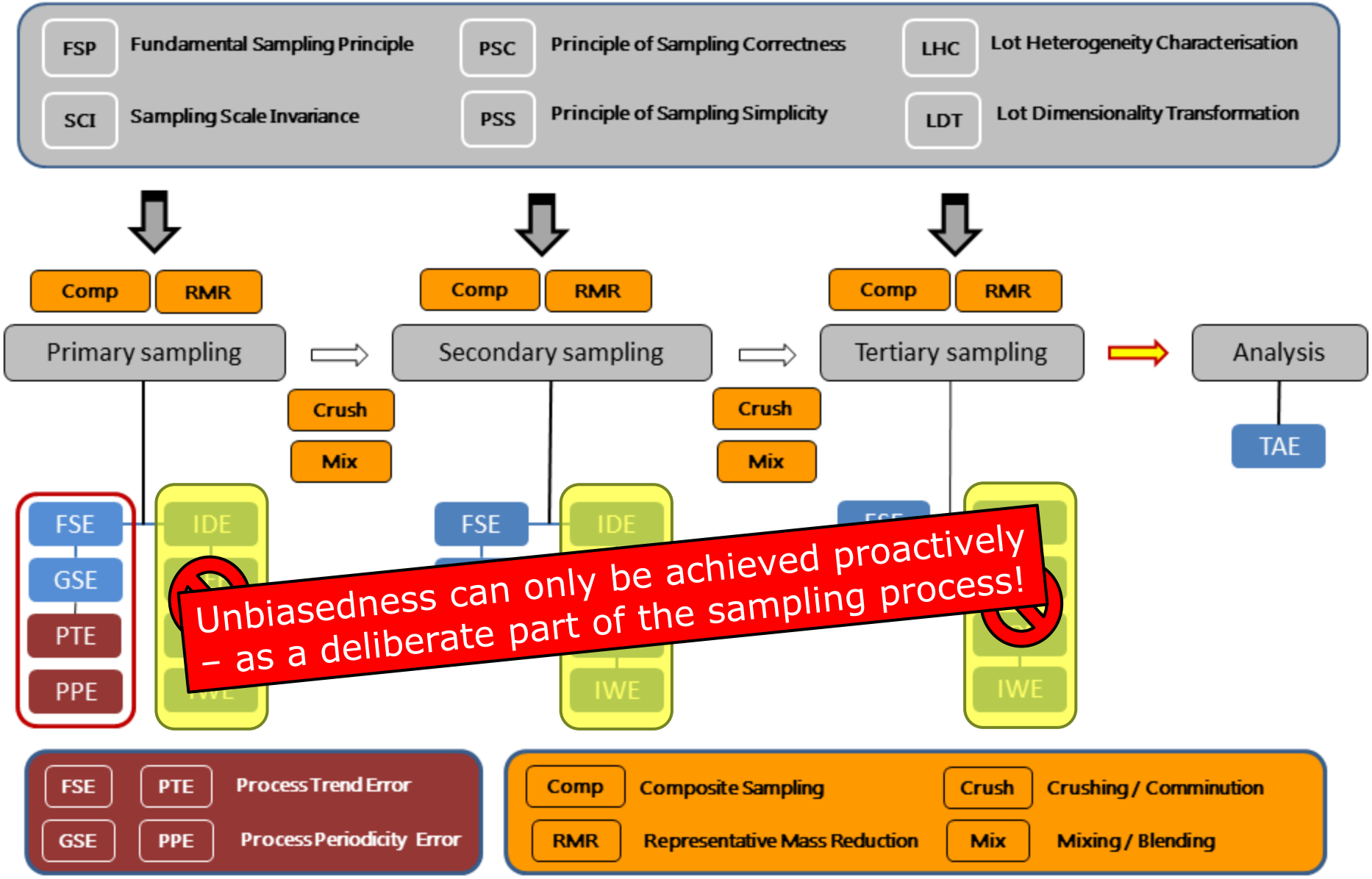
Figure 12 – Characteristics of the analytical bias versus the sampling bias

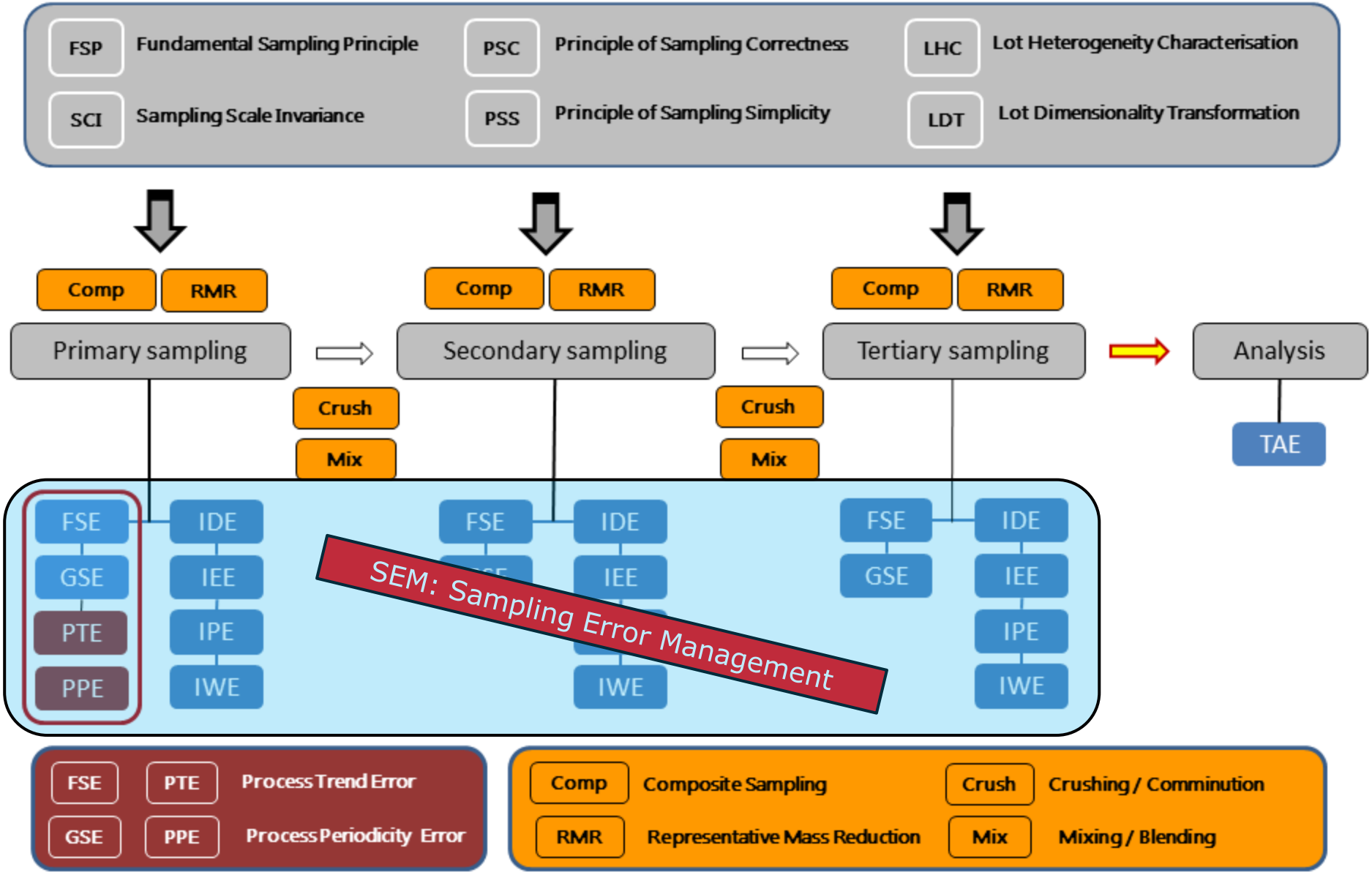
Critical distinction between analysis (*sensu strictu*) – and sampling_plus_analysis! TOS' unique understanding!



Sampling process: BIAS + imprecision - - TOS: a *varying*, an *inconstant* bias!

Analysis: bias + imprecision (statistical concept: a constant bias)


FSP
Fundamental Sampling Principle
PSC
Principle of Sampling Correctness
LHC
Lot Heterogeneity Characterisation
SCI
Sampling Scale Invariance
PSS
Principle of Sampling Simplicity
LDT
Lot Dimensionality Transformation
Comp
RMR
Primary sampling
Crush
Mix
Secondary sampling
Tertiary sampling
Analysis
TAE
FSE
GSE
PTE
PPE
IDE
IWE
Unbiasedness can only be achieved proactively – as a deliberate part of the sampling process!
Process Trend Error
Process Periodicity Error
Composite Sampling
Crushing / Comminution
Representative Mass Reduction
Mixing / Blending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PTE
PPE
IDE
IEE
IPE
IWE
FSE
IDE
IEE
IWE
FSE
GSE
IDE
IEE
IPE
IWE
SEM: Sampling Error Management
FSE
PTE
Process Trend Error
GSE
PPE
Process Periodicity Error
Comp
Composite Sampling
Crush
Crushing / Comminution
RMR
Representative Mass Reduction
Mix
Mixing / Blending

DS
DANSK STANDARD

Dansk standard

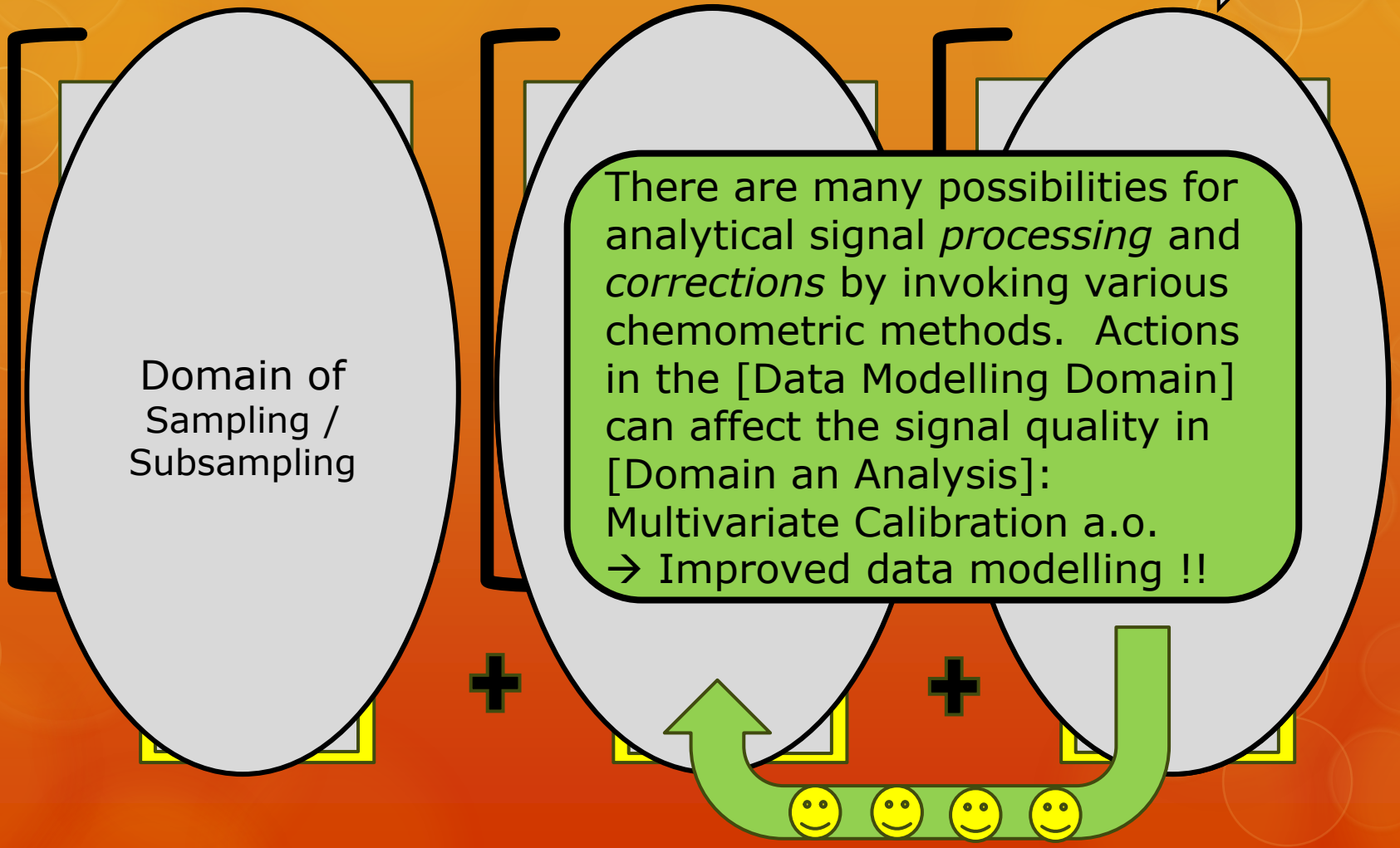
DS 3077:2024

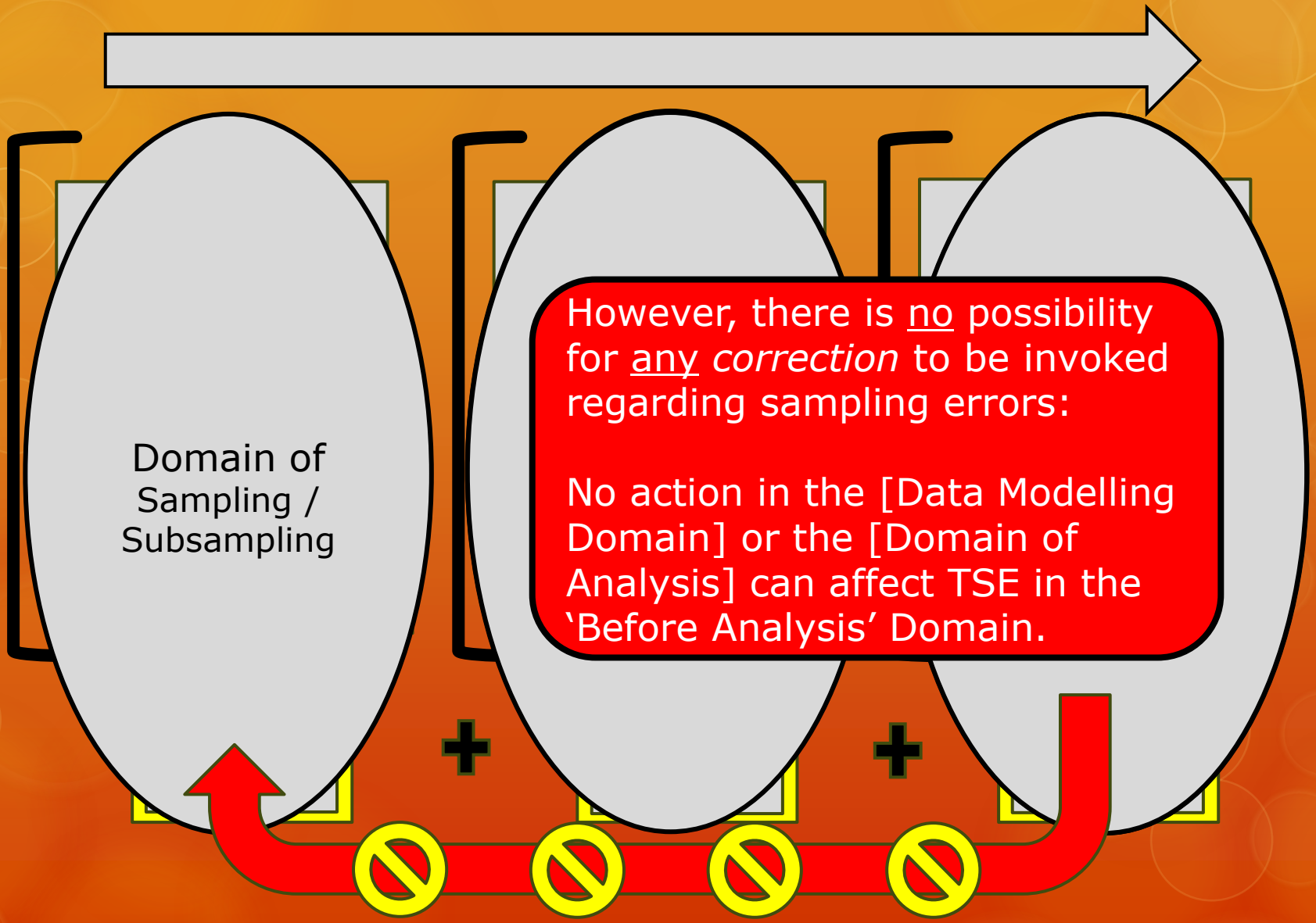
2024-10-13

Repræsentativ prøvetagning – Horisontal standard

Representative sampling – Horizontal standard

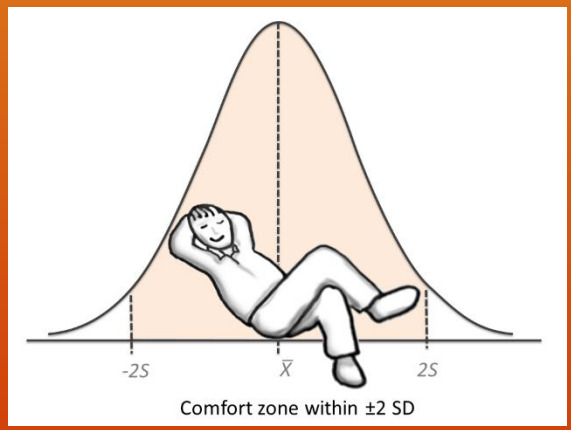
... Back to the three domains of reckoning ...

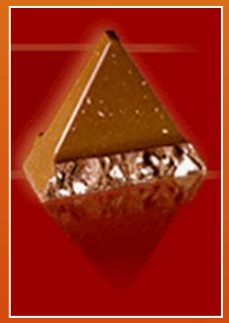



Domain of
Sampling /
Subsampling
However, there is no possibility for any *correction* to be invoked regarding sampling errors:
No action in the [Data Modelling Domain] or the [Domain of Analysis] can affect TSE in the 'Before Analysis' Domain.

THE real problem: assume normality (analytical results)

It most emphatically is **not** – heterogeneity does not follow a normal distribution (.. of analytical results)!





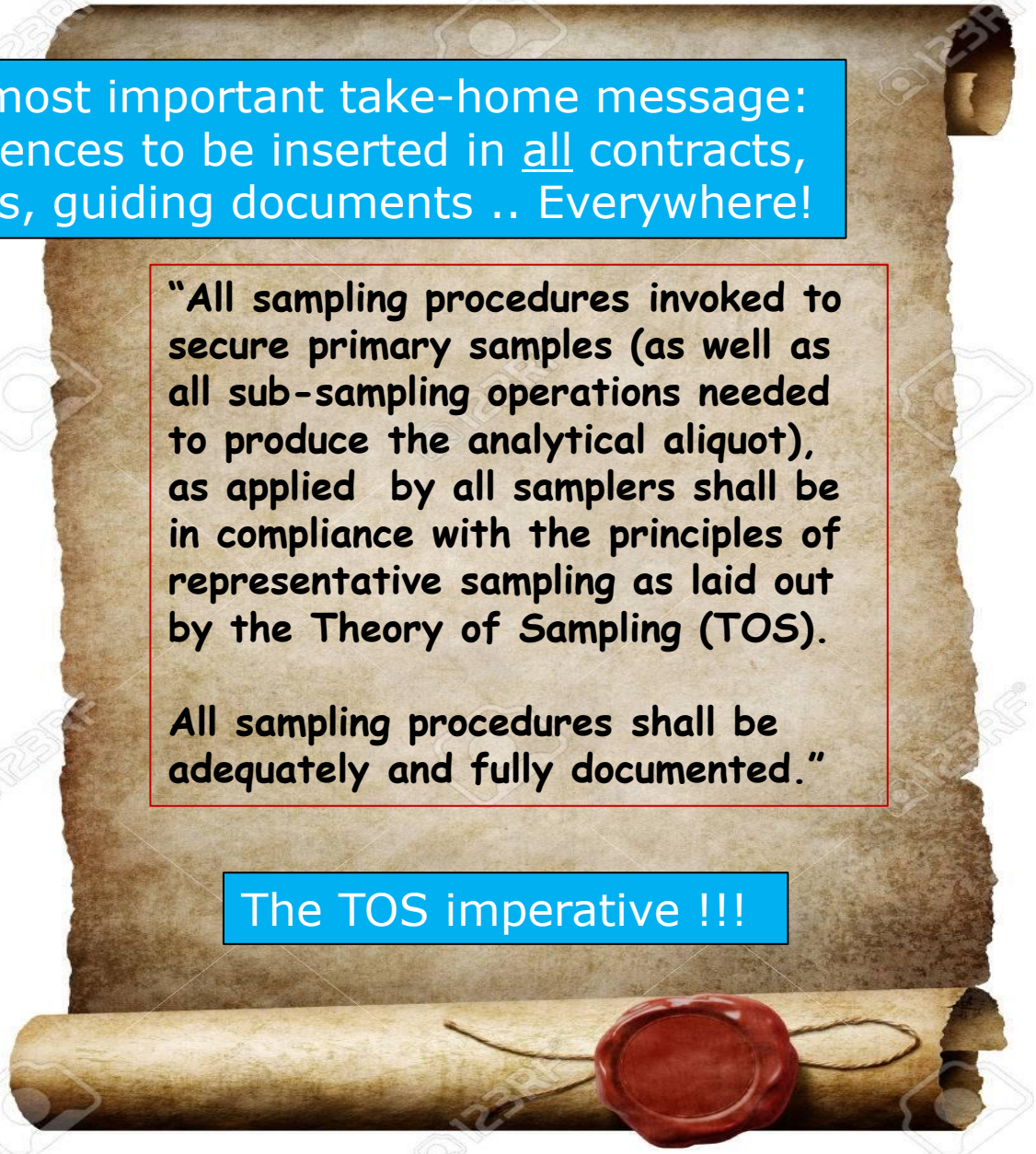
Sampling solutions – technology; sampling conditions; economy; complexities; risk management

Framing TOS in Risk Assessment - an outreach perspective

Claudia Paoletti & Kim H. Esbensen

Proceedings of WCSB10: TOS Forum Issue 11, 419–423 (2022)

Today's most important take-home message:
Two sentences to be inserted in all contracts, standards, guiding documents .. Everywhere!



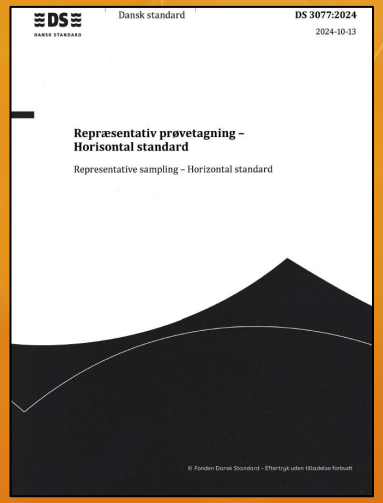
"All sampling procedures invoked to secure primary samples (as well as all sub-sampling operations needed to produce the analytical aliquot), as applied by all samplers shall be in compliance with the principles of representative sampling as laid out by the Theory of Sampling (TOS).

All sampling procedures shall be adequately and fully documented."

The TOS imperative !!!

WHAT can be accomplished in 30 minutes?

More than enough!



You are now well aware of **DS 3077:2024**
You can download it from here: **www.ds.dk**

A bonanza of **Open Access** literature is available …

1. F.ex. from Interntional Pierre Gy Sampling Association:
   https://www.intsamp.org/

2. Center of Sampling Competence (CSC)
   https://www.sampling-competence.com/

IPGSA
INTERNATIONAL PIERRE GY SAMPLING ASSOCIATION

ABOUT IPGSA | EDUCATIONAL OUTREACH | SST-MAGAZINE | WCSB CONFERENCE | NEWS | CONTACT

International Pierre Gy Sampling Association

The International Pierre Gy Sampling Association, IPGSA, promotes the development and application of the Theory of Sampling (TOS). TOS was founded by Pierre Gy in the 1950s to address sampling issues in mining and related industries, and since then the IPGSA has applied TOS in manufacturing, pharmaceuticals, agriculture, and waste management.

Learn more about the IPGSA

Sampling Science and Technology

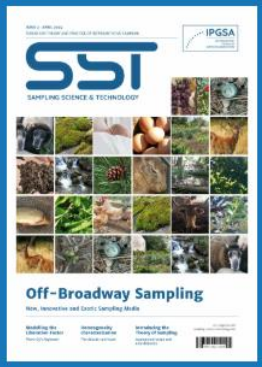
Sampling Science and Technology (SST) is a free publication for the sampling community, providing a communications platform for all interested in the theory and practice of representative sampling and blending. SST aims both to serve the world sampling community and to disseminate awareness of proper sampling practices to all interested parties in science, technology, industry, and commerce, as well as to supervising and regulating authorities.

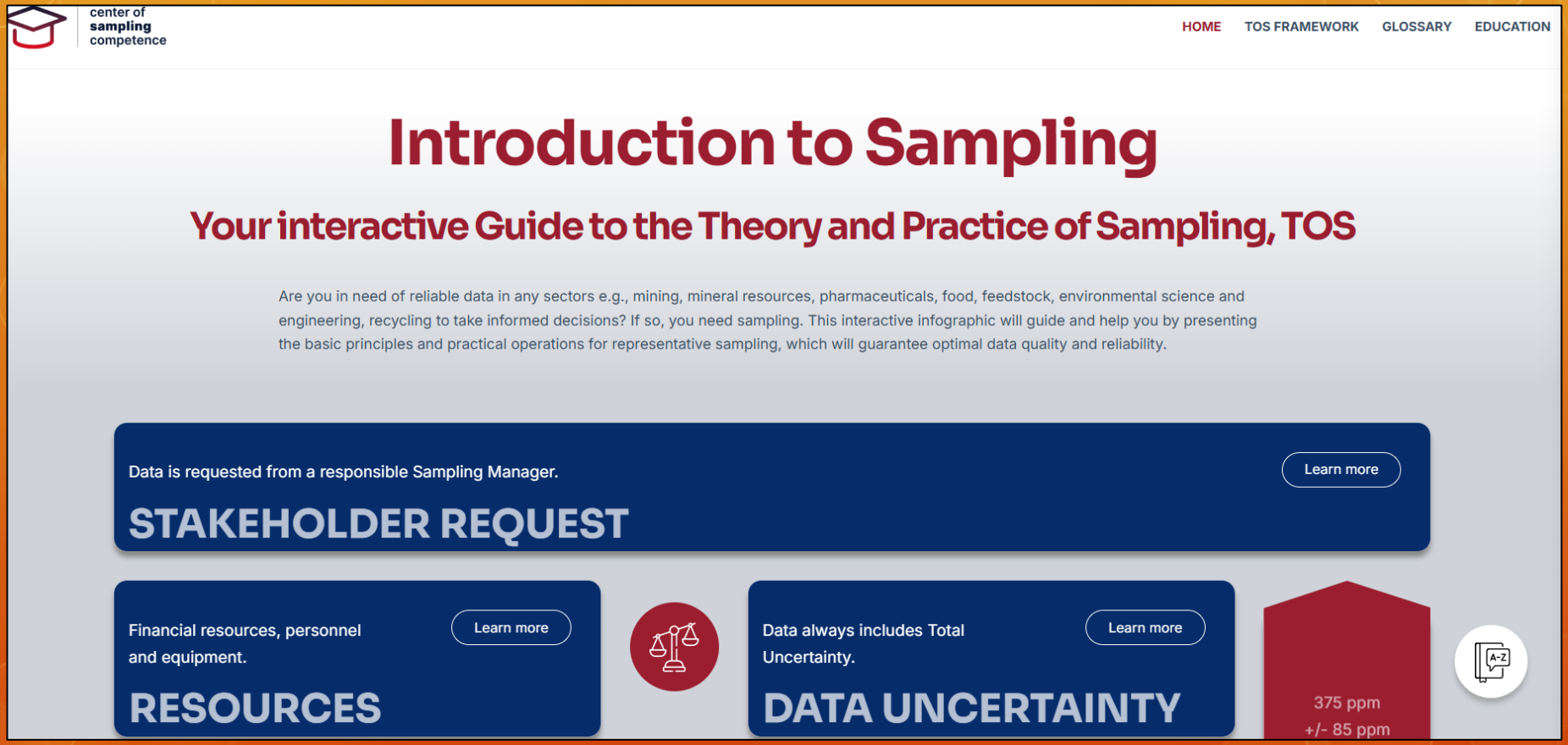
SST is the official communication platform of the International Pierre Gy Sampling Society (IPGSA). SST is offered as a forum for scientific and technological interaction among all members of the world sampling community and beyond. The aim of SST is to share knowledge on the science and practice of sampling and blending. As such, SST welcomes any-and-all research, practical perspectives, exemplary case histories, as well as the occasional theoretical article aimed at all sampling practitioners.

Read the latest issue of SST

Read the articles in the current issue or visit sst-magazine.info to learn more about the magazine and back issues. There you will also find the complete TOS forum archive.

- Read Issue 3 · April 2025
- sst-magazine.info




center of
sampling
competence
HOME
TOS FRAMEWORK
GLOSSARY
EDUCATION
Introduction to Sampling
Your interactive Guide to the Theory and Practice of Sampling, TOS
Are you in need of reliable data in any sectors e.g., mining, mineral resources, pharmaceuticals, food, feedstock, environmental science and engineering, recycling to take informed decisions? If so, you need sampling. This interactive infographic will guide and help you by presenting the basic principles and practical operations for representative sampling, which will guarantee optimal data quality and reliability.
Data is requested from a responsible Sampling Manager.
Learn more
STAKEHOLDER REQUEST
Financial resources, personnel and equipment.
Learn more
RESOURCES
Data always includes Total Uncertainty.
Learn more
DATA UNCERTAINTY
375 ppm
+/- 85 ppm

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Introduction to the Theory and Practice of Sampling

by Kim H. Esbensen

Thank you for your attention!

Kim H. Esbensen, KHE Consulting (KHEC), Copenhagen, Denmark

Associate-, visiting-, and external professor (Norway, Puerto Rico, Quebec)