

TOS competence: critical **SUCCESS FACTOR**

# Theory and Practice of Representative Sampling ( $\tau_{OS}$ )

- ... of materials and processes (lots) of very different nature ...
- ... of lots of very different composition and size ...
- ... of lots of very different grain size distribution characteristics ...
- ... of materials lots from all over science, technology, industry and society...

1980 – 2015: professor (3 universities/gov. R&D institutions)

2015 → Consultant, independent researcher, owner (KHEC)

Associate, guest and affiliated professor @ 3 universities



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The business card features a background image of a geological cross-section with a blue bar chart and an orange line graph overlaid at the bottom. The contact information is listed on the left, and a small portrait of Kim H. Esbensen is on the right. A blue box at the bottom right of the card repeats the email address.

**“Usikkerheder i forureningsundersøgelser – hvad skal der til for at vi er sikre nok?”**

**Hvorfor er det relevant at diskutere datausikkerhed?**

→ Før vi gør dette, er det kritisk vigtigt at forstå at der er to slags usikkerhed:

→ **Måleusikkerhed**

**Total Analytical Error = TAE**

→ **Prøvetagningsusikkerhed**

**Total Sampling Error = TSE**

→ **Men det er næsten altid kun måleusikkerheden der optræder på dagsordenen ... KRITISKT PROBLEM !!**

**Always!!!**

**Uncertainty = TSE + TAE**

Analyse resultater = "Data" +/- "usikkerhed"

**Total Analytical Error = TAE**

Analyse af en forsvindende brøkdel af lot/mat

Prøvetagning bidrager med en **DOMINERENDE** usikkerhed, ofte 10/25/50 X større end TAE !!

**Total Sampling Error = TSE**

**Always!!!**

**Uncertainty = TSE + TAE**

Analyse resultater = "Data" +/- "usikkerhed"

**Total Analytical Error = TAE**

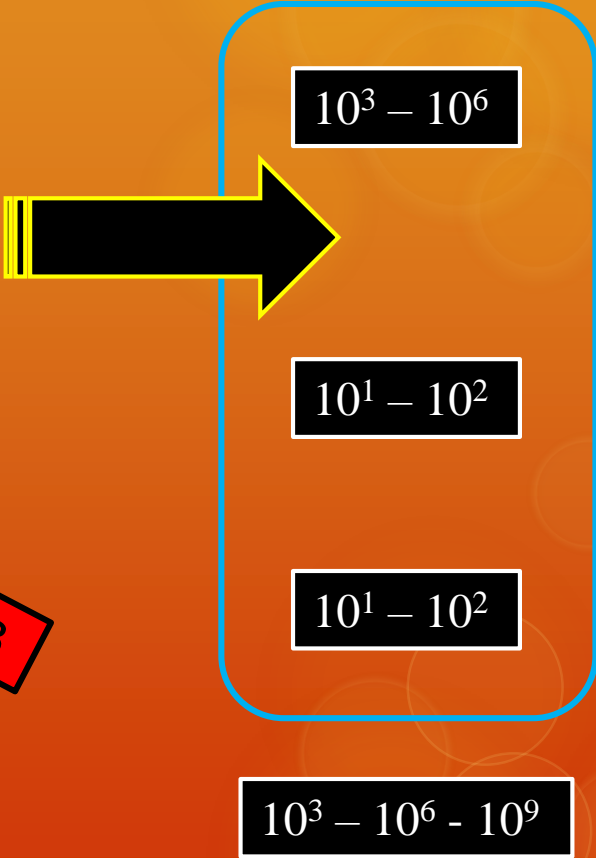
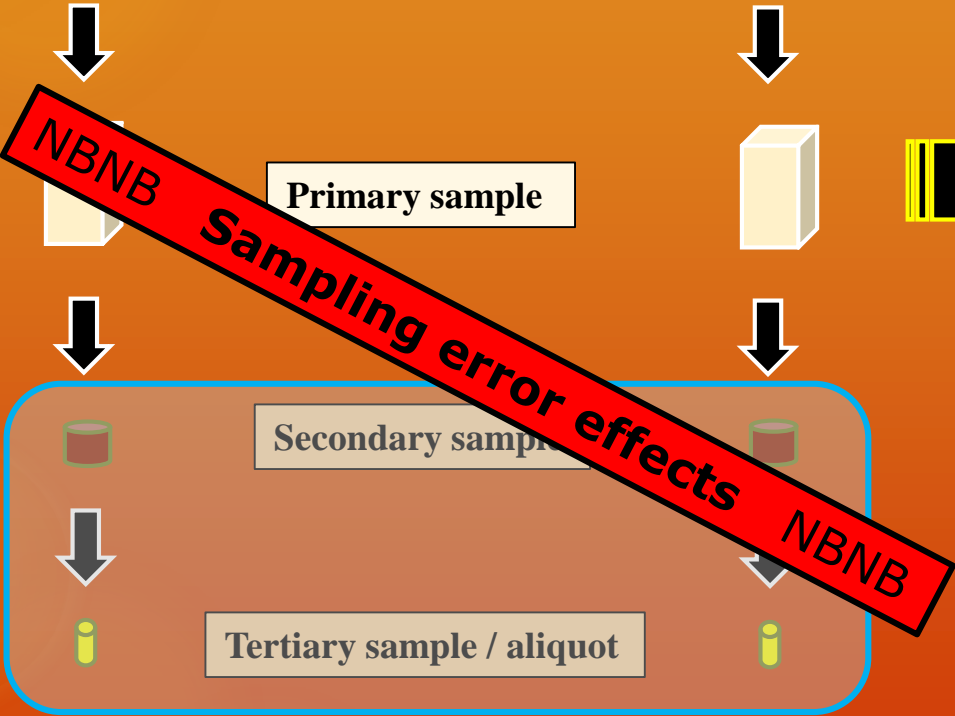
Analyse af en forsvindende brøkdel af lot/mat

Prøvetagning bidrager med en DOMINERENDE usikkerhed, ofte 10/25/50 X større end TAE !!

**Total Sampling Error = TSE**

**Adverse characteristic:  
Heterogeneity**

**Sampling rate  
(typical)**



# Sampling ... most common understandings

Sampling - priorities:

1. Sampling method, -plan
2. Equipment, containers, ID-tags, logbook, time
3. Economics, ergonomics, effectivity, logistics ...
4. Often very detailed plans covering "everything" ++



Sample size?  
(mass/volume)

# Sampling ... but there are often “problems”



## Sampling - problems:

1. Materials (lots) can be of *very* different composition and constitution e.g. solids, liquids aggregates, mixtures, slurries
2. Materials can be of extremely different grainsize distributions
3. Lots can be a extremely different size and accessibility





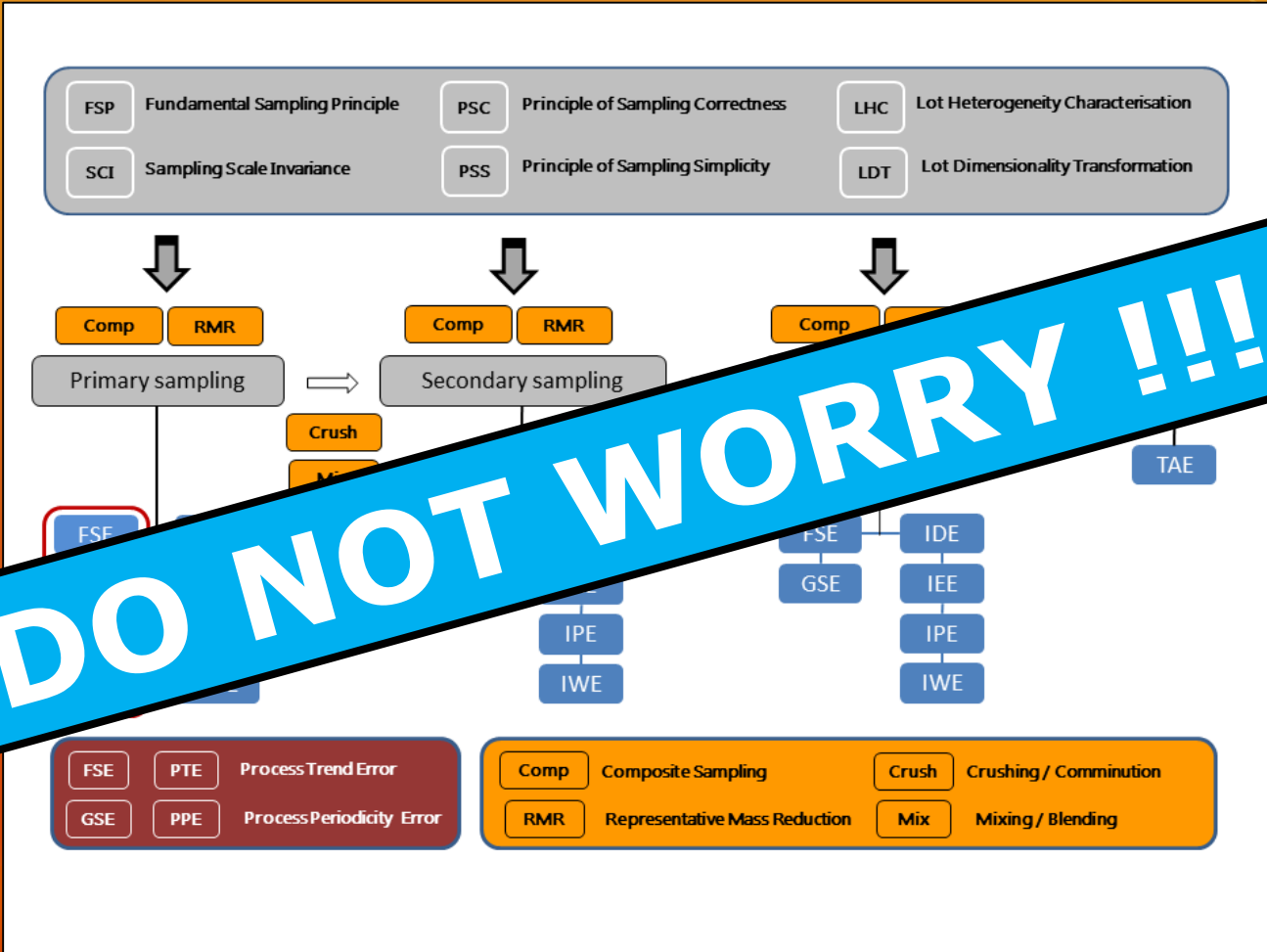
# Sampling ... imperative priority



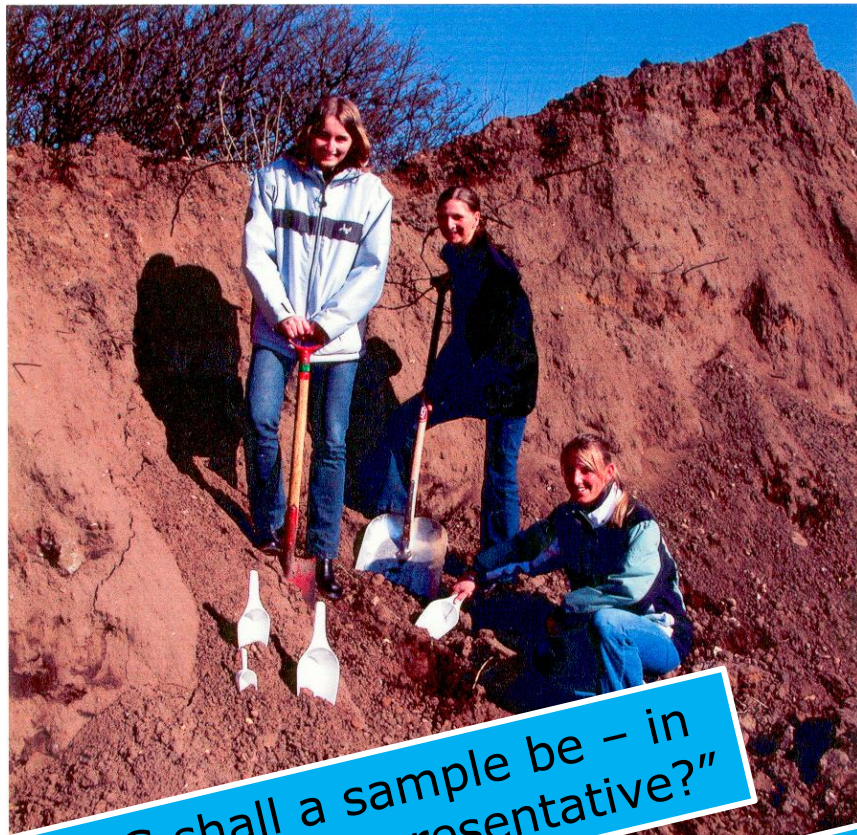
## Sampling priorities (TOS):

1. Representativity ... ..
2. Procedure, sampling plan (2-D), equipment, containers, ID-tags, logbook ....
3. Economics, ergonomics, effectivity, logistics ...
4. And what ever else have you ... ..

# DO NOT WORRY !!!



# The most often heard question ... ..



**"HOW BIG** shall a sample be – in order for it to be representative?"



**HOW small** can a sample be – in order for it to be representative?"

# Unfortunately ....

- This wishful thinking is **WRONG** ...
- 1. The **WRONG** question ... (TOS will tell you why!)
- 2. At the **WRONG** place (the lot) ...
- 3. At the **WRONG** time ...



**2. & 3. *should* have been resolved long before sampling commences ...**

- 4. But: TOS will of course give you an answer!  
- a very surprising answer ;- ) ;- ) ;- )**

# The right priority ... acc. to TOS

1. Primary samples **must** be REPRESENTATIVE  
- this is **non-negotiable!**

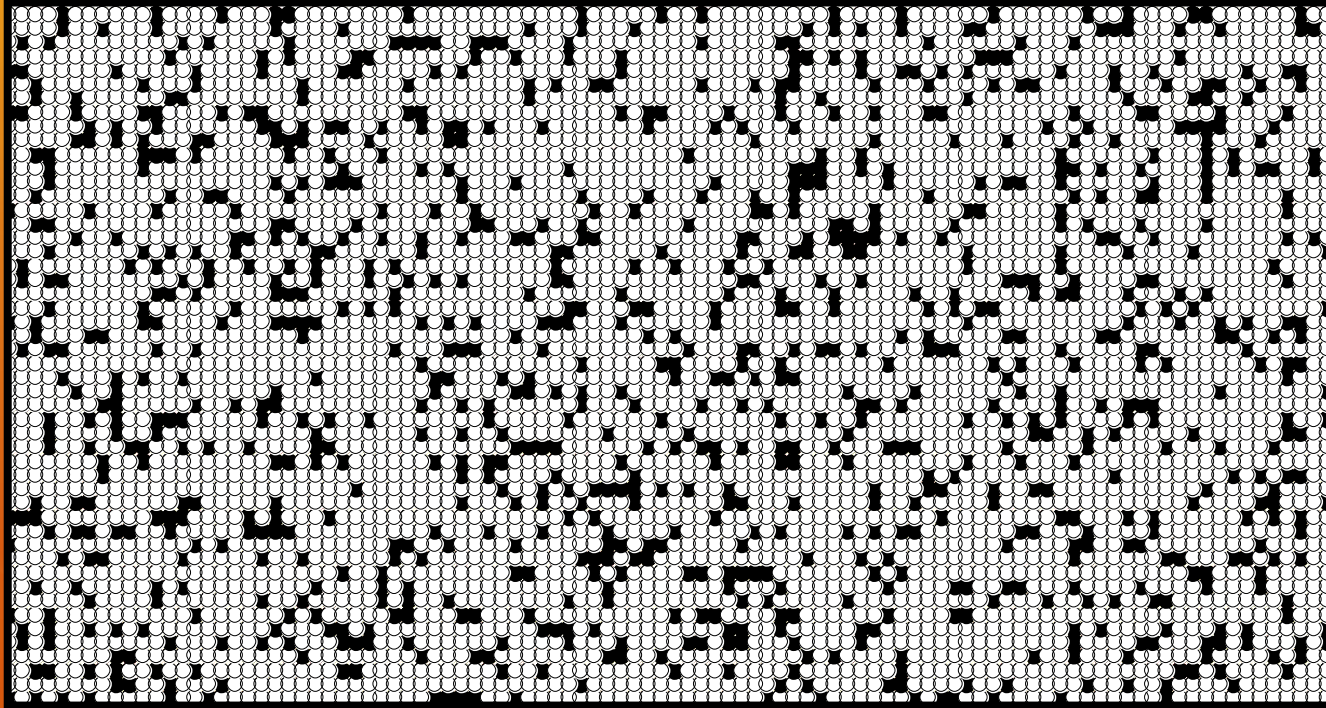
2. TOS is the only framework that stipulates procedures and design of equipment and sampling processes that **guarantees** representativity (accuracy *and* precision)

3. When representativity is achieved → optimal sample mass!

**4. Representativity is the driver for proper sampling !!!!!!!!!**

**5. Representativity is the driver for fixing sample mass !!!!!**

# SAMPLING vs. HETEROGENEITY

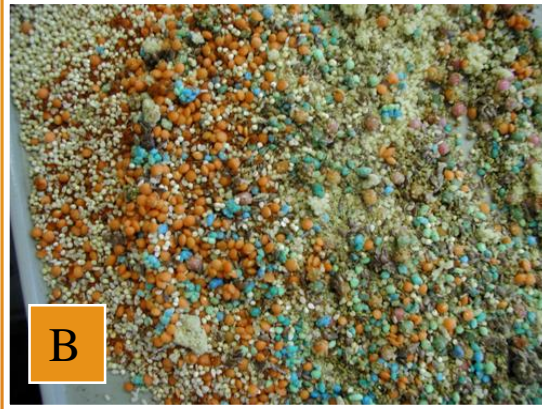


It is ALL about how to *control* the negative effects of heterogeneity on the sampling process

# Theory and Practice of Representative Sampling

Material/lot **heterogeneity** – where it all begins  
(and often ends, sadly)

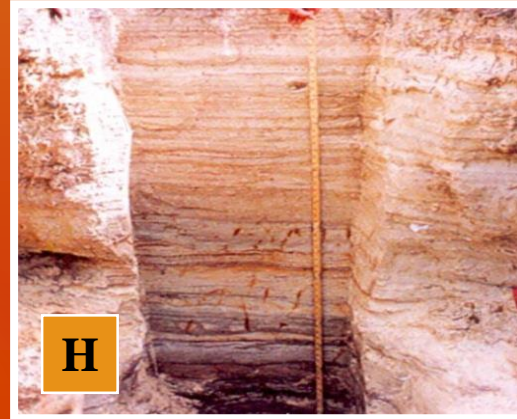


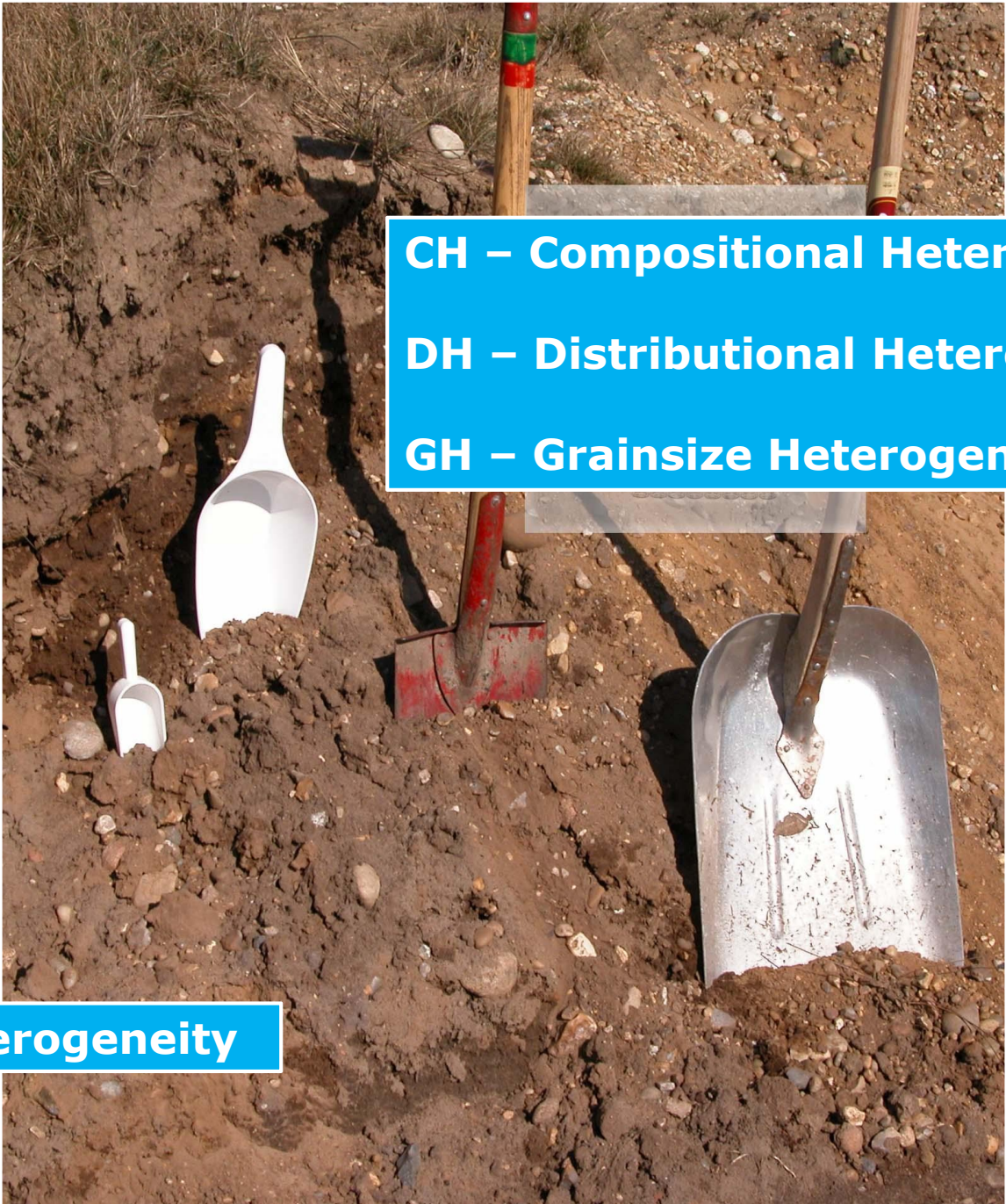


**CH – Compositional Heterogeneity**

**DH – Distributional Heterogeneity**

**GH – Grainsize Heterogeneity**





**CH – Compositional Heterogeneity**  
**DH – Distributional Heterogeneity**  
**GH – Grainsize Heterogeneity**

**Visual Heterogeneity**



**CH – Compositional Heterogeneity**

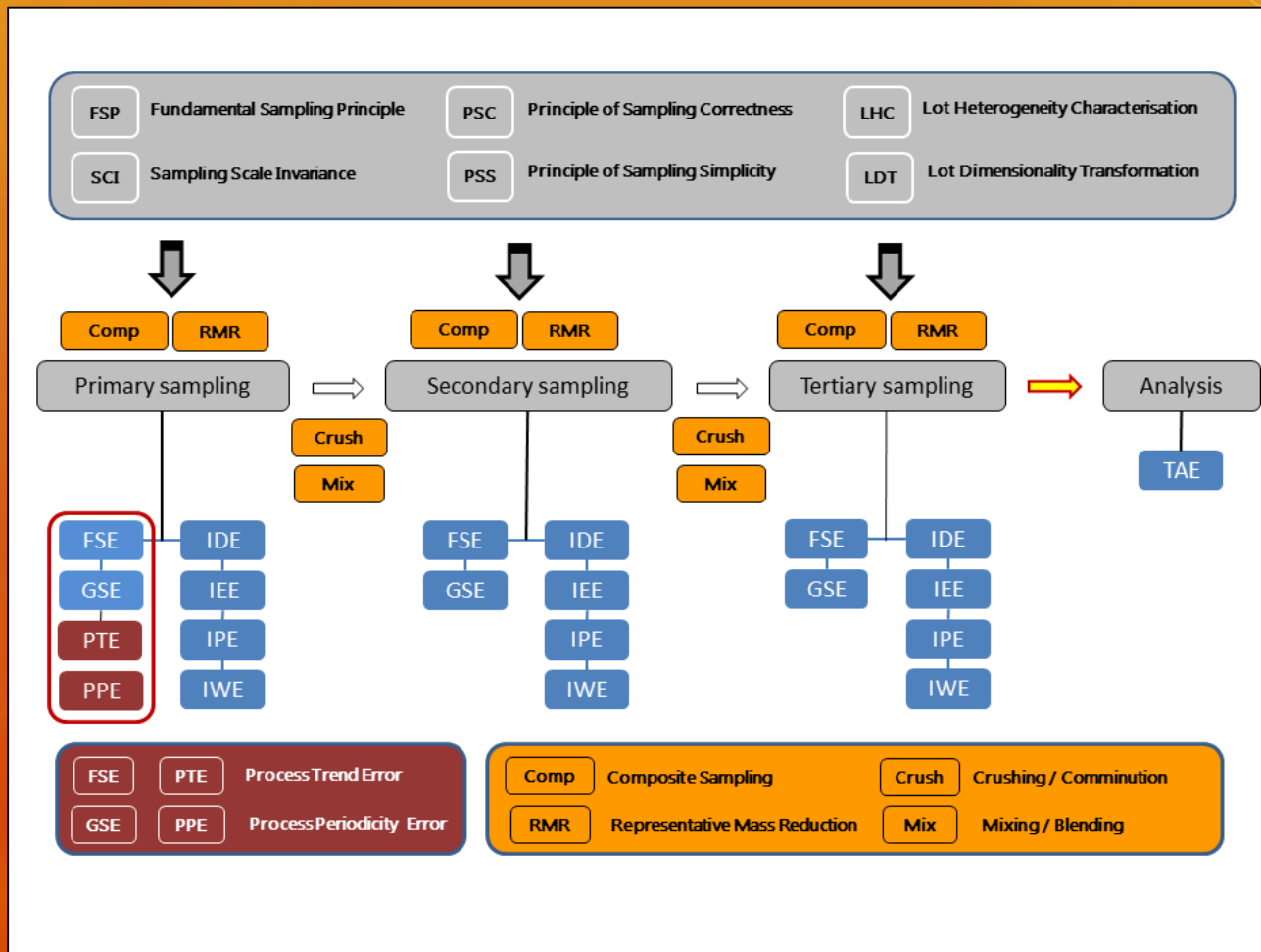
**DH – Distributional Heterogeneity**

**GH – Grainsize Heterogeneity**

**Not-so-visual Heterogeneity**

# Theory of Sampling (TOS)

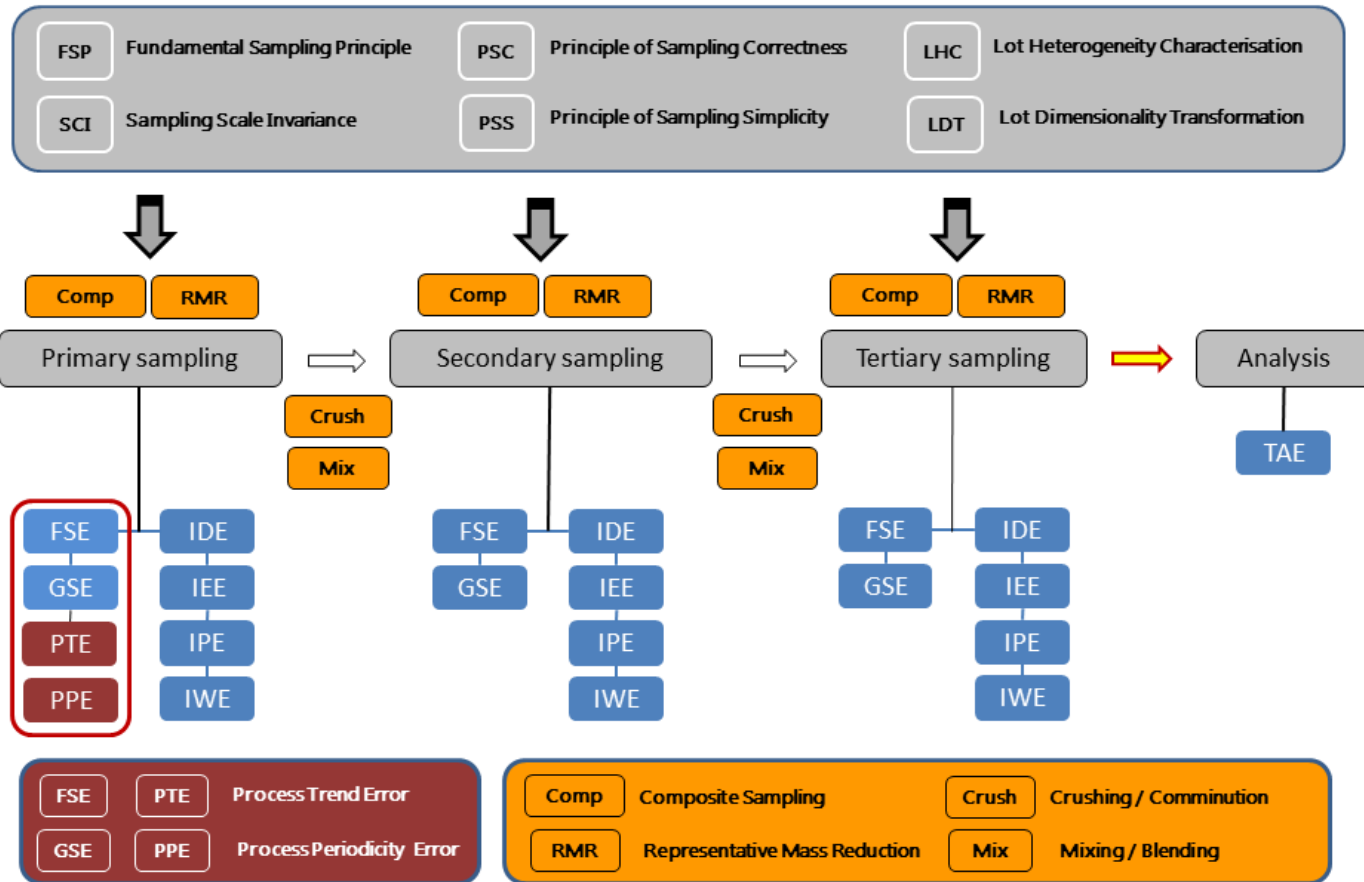
## – your very first overview



# TOS' systematic framework ...

- Six Governing Principles (GP)
- Four Sampling Unit Operations (SUO)
- Sampling is always a multi-stage process
- Each stage can be, shall be, addressed individually
  
- Sampling Errors (SE) *will* be induced at all stages !!!
- But can be minimised in *similar fashion* at each stage ...
  
- *Stationary* lots and *moving* lots (*process sampling*) are governed by the exact same GP, SUO, stages, errors ...



# Beginning to see an overview ... ;-)



# Representative Sampling: Theory of Sampling (TOS)

## TOS - Axiomatic exposé

### Governing Principles (GP) – Sampling Unit Operations (SUO)

1. FSP: Fundamental Sampling Principle
2. SSI: Sampling Scale Invariance 
3. PSC: Sampling Correctness (bias-free sampling)
4. PSS: Sampling Simplicity (primary sampling + mass-reduction) 
5. LDT: Lot Dimensionality Transformation
6. LHC: Lot Heterogeneity Characterization (0-D, 1-D)
7. SUO: Composite Sampling
8. SUO: Comminution
9. SUO: Mixing / Blending
10. SUO: Representative Mass Reduction (Sub-sampling)



## **Repræsentativ prøvetagning – Horizontal standard**

Representative sampling – Horizontal standard

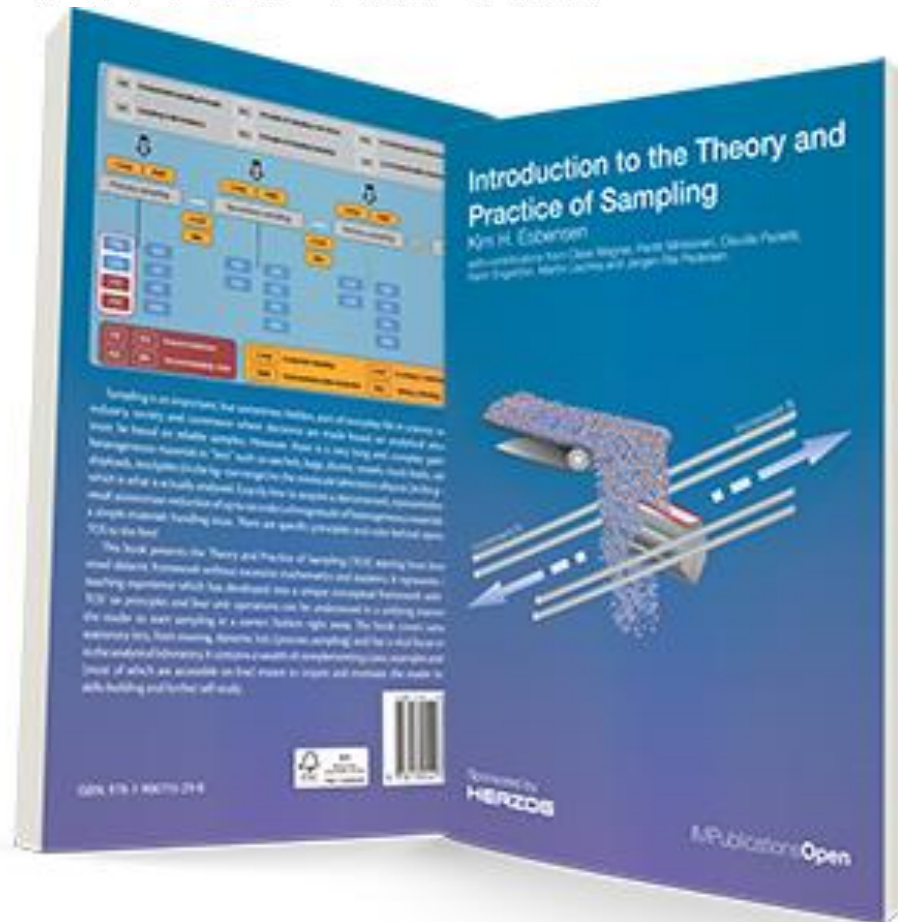
[www.ds.dk](http://www.ds.dk)



# Introduction to the Theory and Practice of Sampling

Kim H. Esbensen

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BOOK

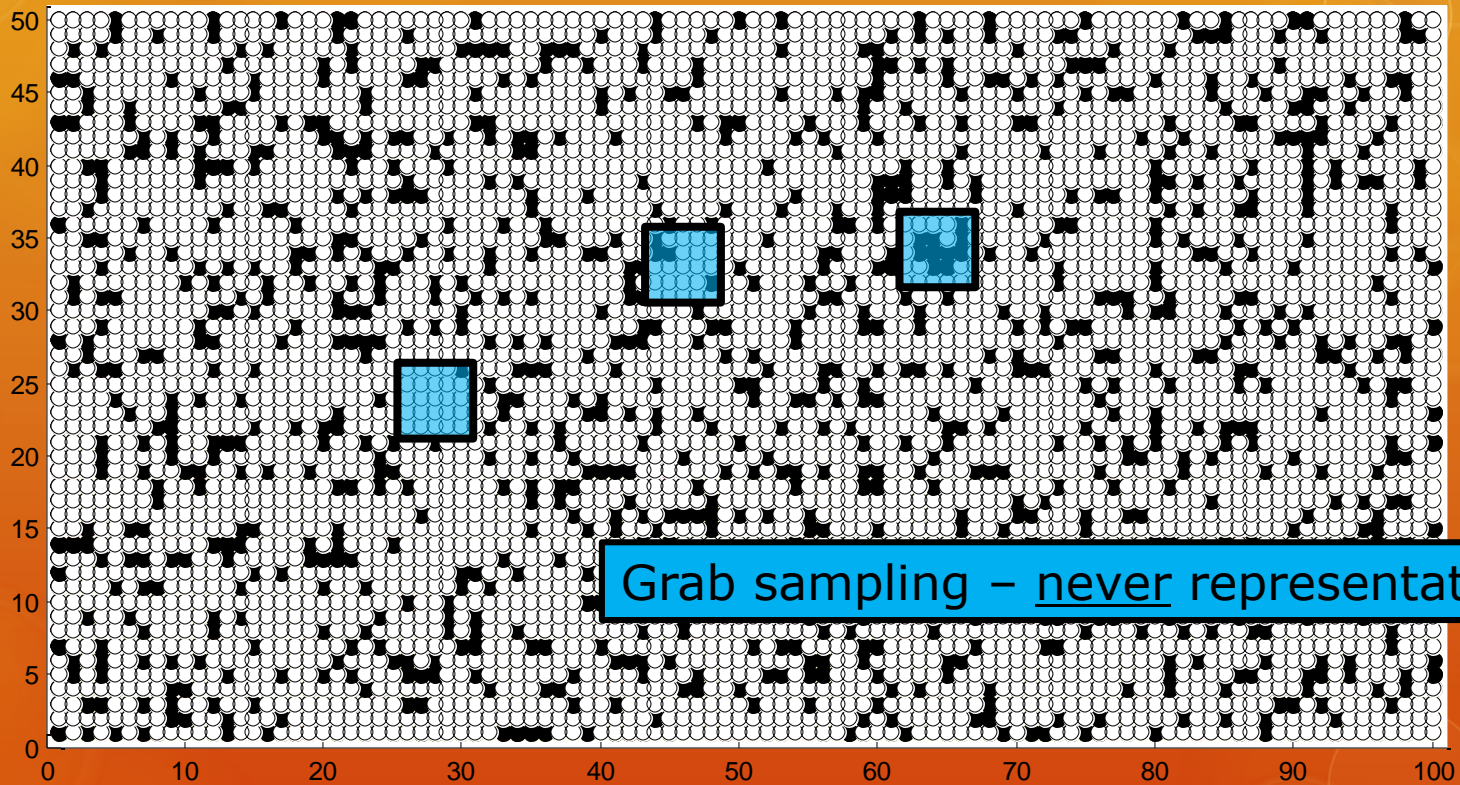


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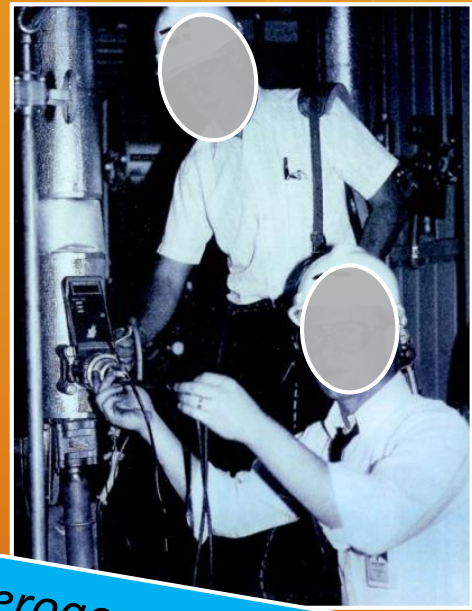
<https://www.impopen.com/sampling>

# Heterogeneity - the unifying characteristic for all types of material



Grab sampling - never representative





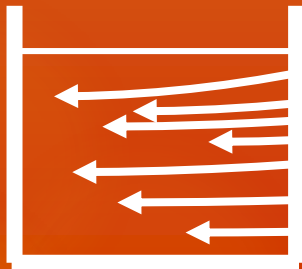
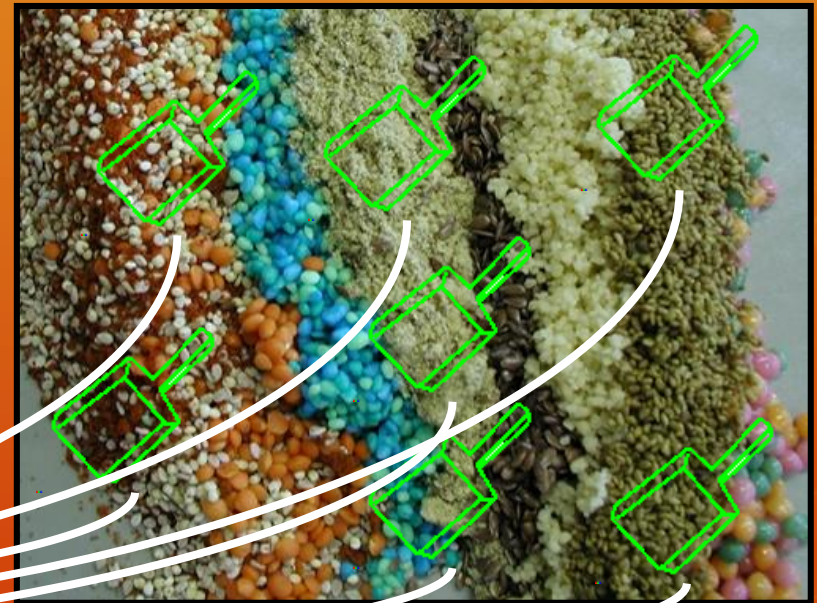
A grab sample can never be representative of a heterogeneous material



# Sampling Unit Operations: Composite Sampling

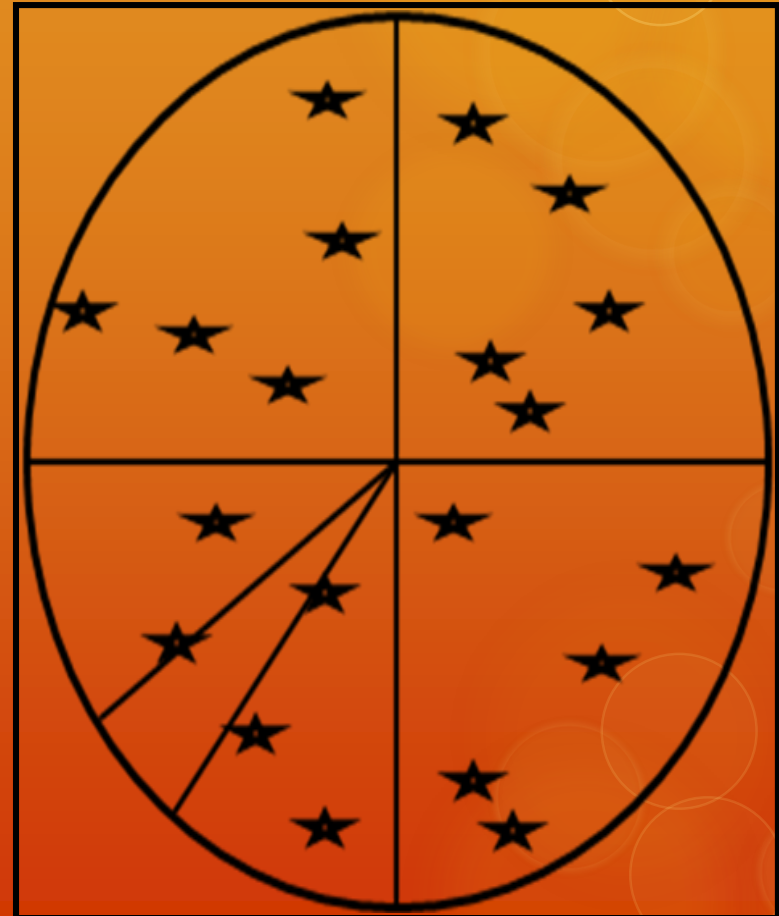


"Grab Sampling" - "not thinking"



... .. vs. Composite Sampling ... ..

# Grab sampling vs. Composite sampling



It so easy to do it **WRONG!** And so easy to do it **RIGHT!**



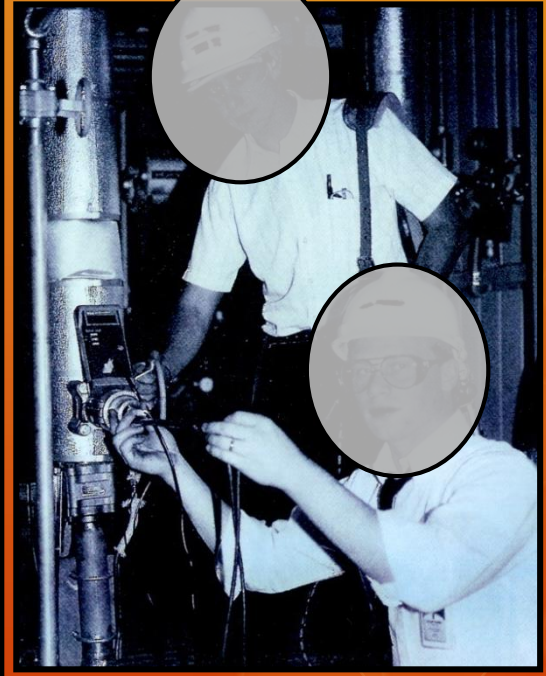
Model photo: with permission

# Three key elements of sampling

Sampling  
competence

The sampling *fool* -I

The sampling *fiasco!*



Sampling is only dependent upon three key elements !!







## Governing principles (GP) & Sampling Unit Operations (SUO)

1. FSP: Fundamental Sampling Principle
2. PSC: Sampling Correctness (bias-free sampling)
3. PSS: Sampling Simplicity (primary sampling + mass-reduction)
4. SSI: Sampling Scale Invariance
5. LDT: Lot Dimensionality Transformation
6. LHC: Lot Heterogeneity Characterization (0-D, 1-D)
7. SUO: Composite Sampling
8. SUO: Comminution
9. SUO: Mixing / Blending
10. SUO: Representative Mass Reduction (sub-sampling/splitting)

TOS' six **Governing Principles** describe how to conduct representative sampling of heterogeneous materials. The four **SUO's** are the only active agents at disposition.

# Four practical Sampling Unit Operations (SUO)

1. Composite Sampling
2. Particle Size Reduction (comminution)
3. Mixing / blending
4. Representative Mass Reduction (- sample preparation)

Theory & Practise of Sampling (TOS)

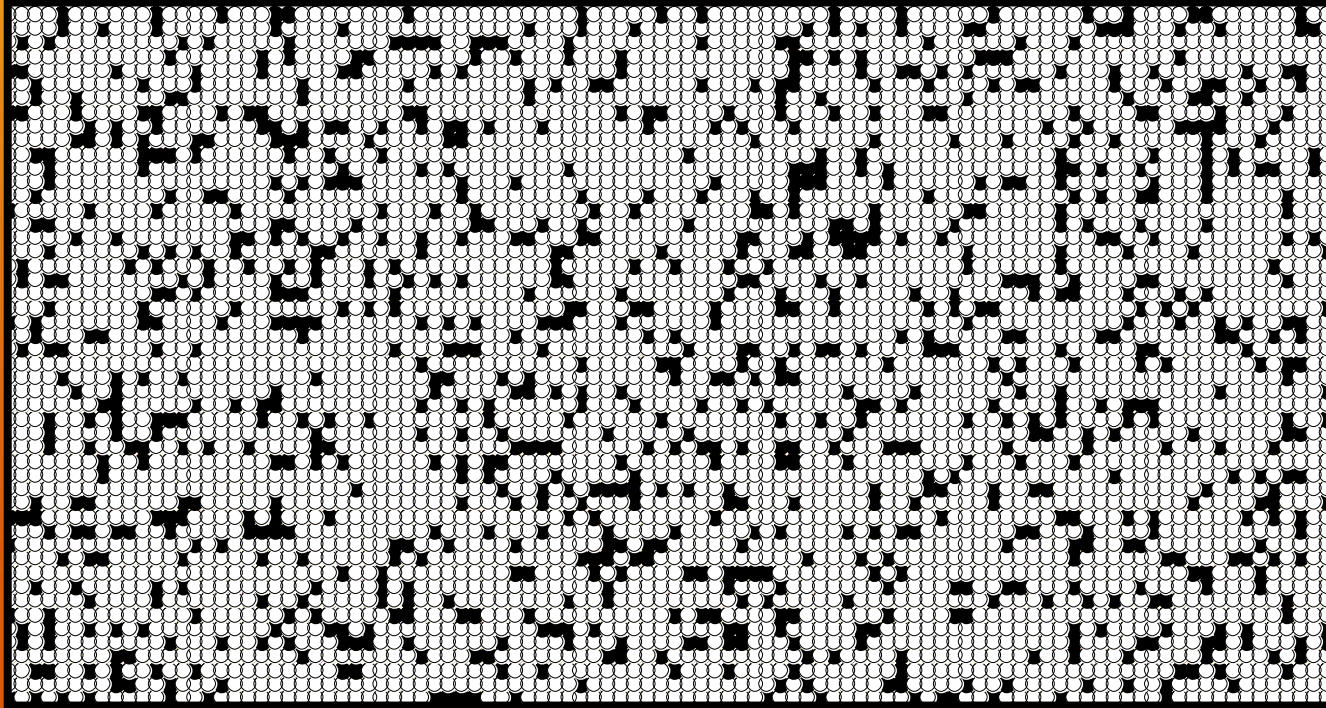
Practical procedures ... ..

Four Sampling Unit Operations (SUO)



Used as active steps in  
the sampling process  
(often used several  
times, in combination)

# SAMPLING vs. HETEROGENEITY



If there is no *control* the negative effects of heterogeneity on the sampling process ...

Concentration

No control

Single grab sampling variance

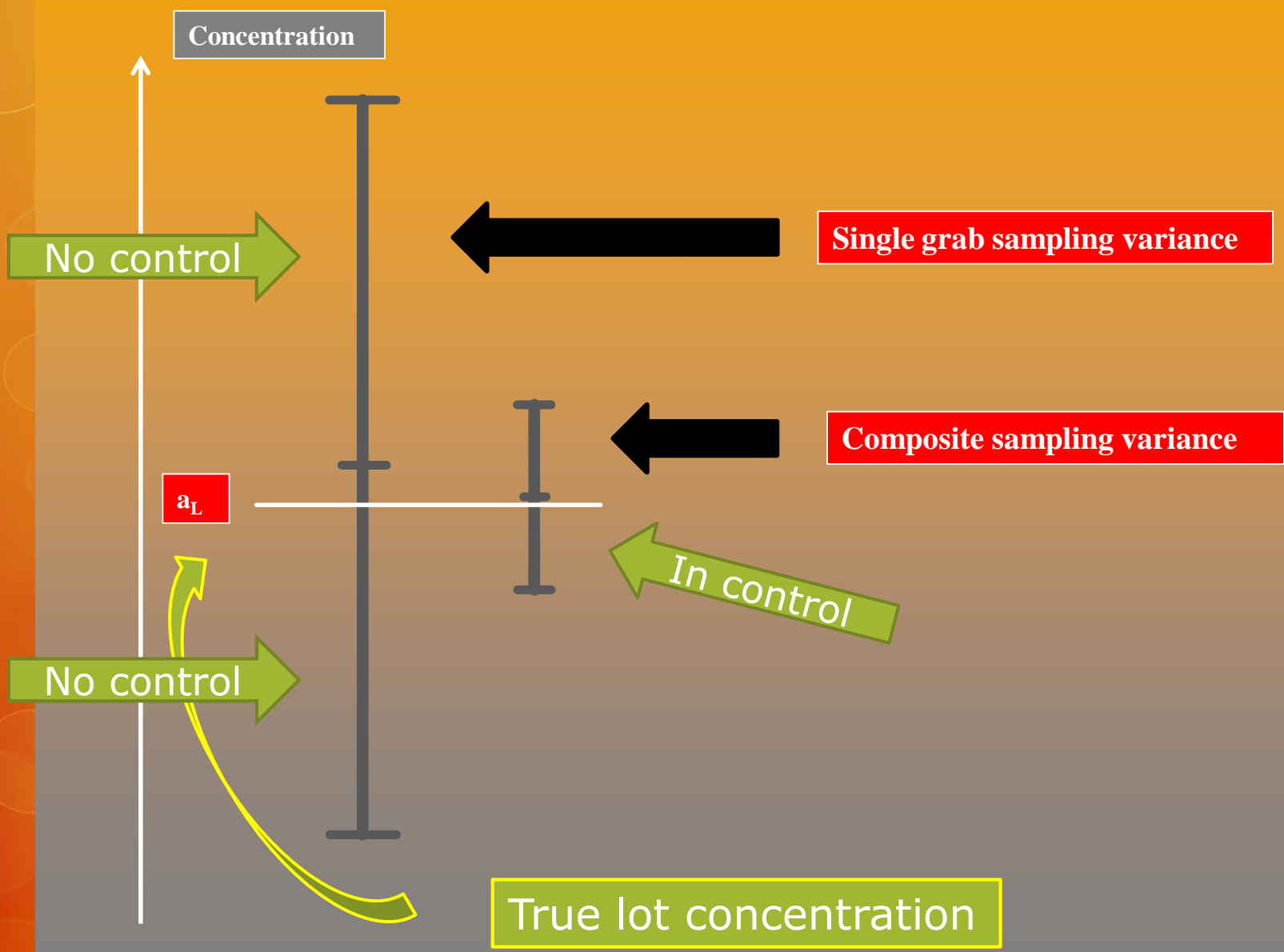
Composite sampling variance

$a_L$

In control

No control

True lot concentration



Buyer

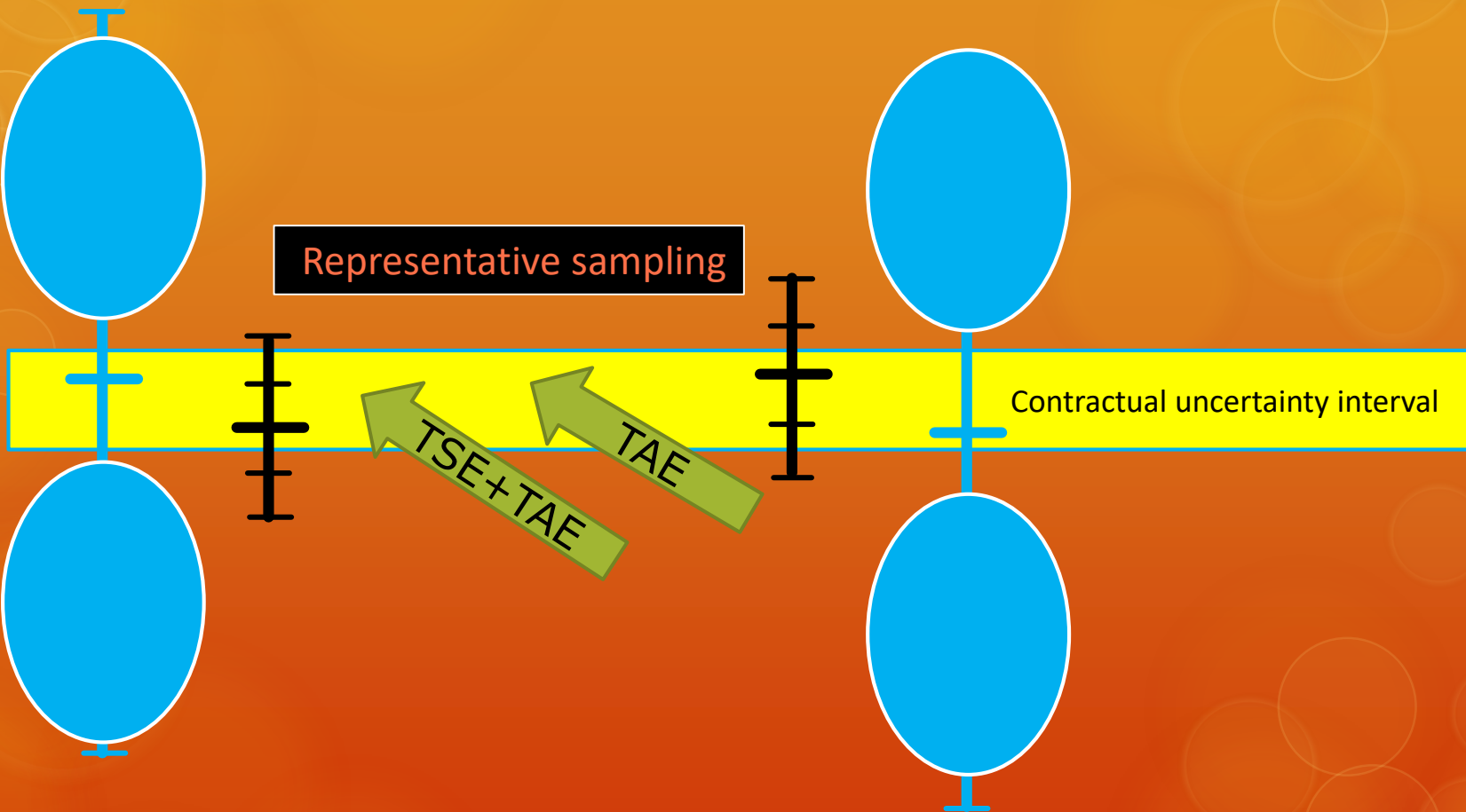
Seller

Client

vs.

Laboratory

Representative sampling



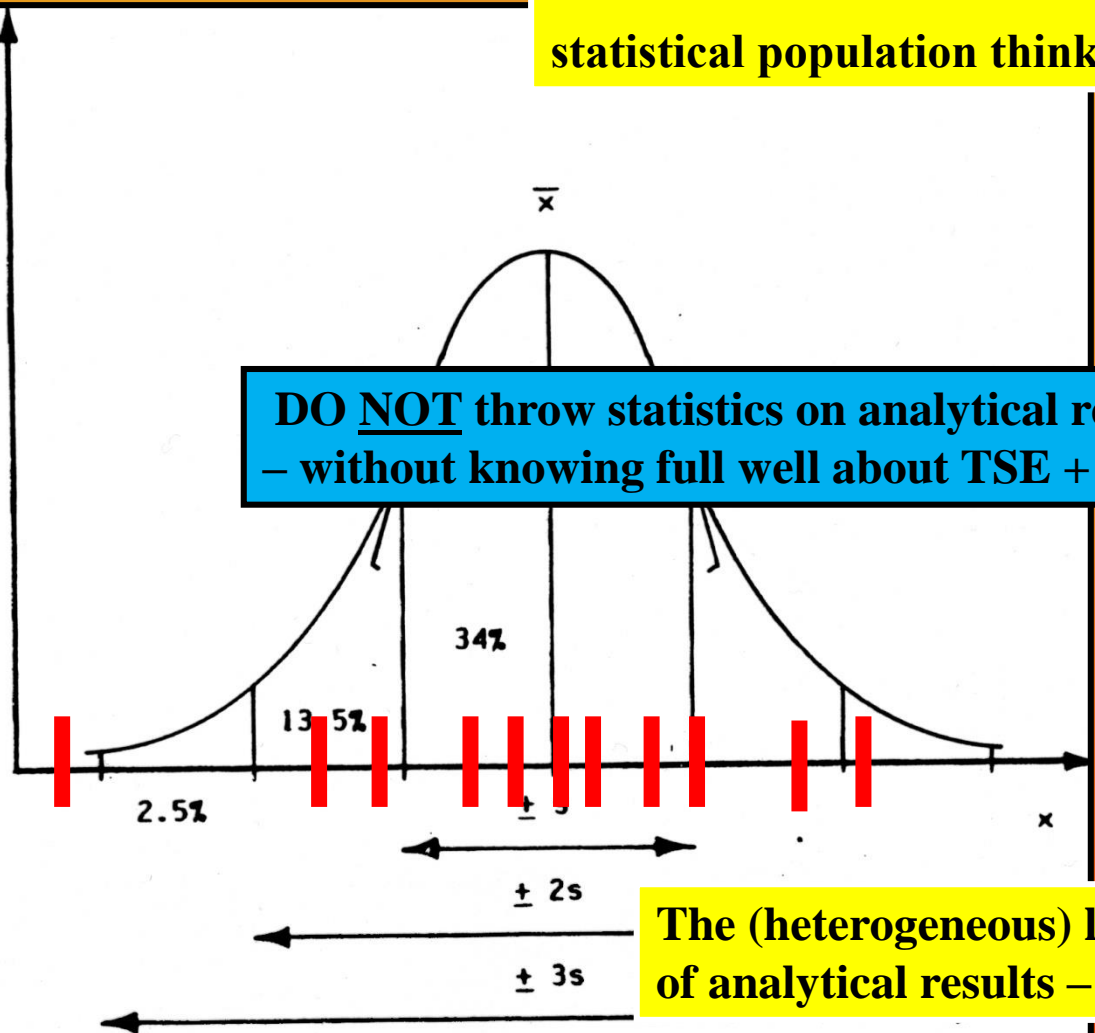
Contractual uncertainty interval

Non-representative sampling

Non-representative sampling

Very difficult to avoid conventional statistical population thinking ... ..

**DO NOT** throw statistics on analytical results – without knowing full well about TSE + TAE!



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

FIGURE 2.6. Illustration of a typical standard normal distribution.



## **Repræsentativ prøvetagning – Horizontal standard**

Representative sampling – Horizontal standard

[www.ds.dk](http://www.ds.dk)



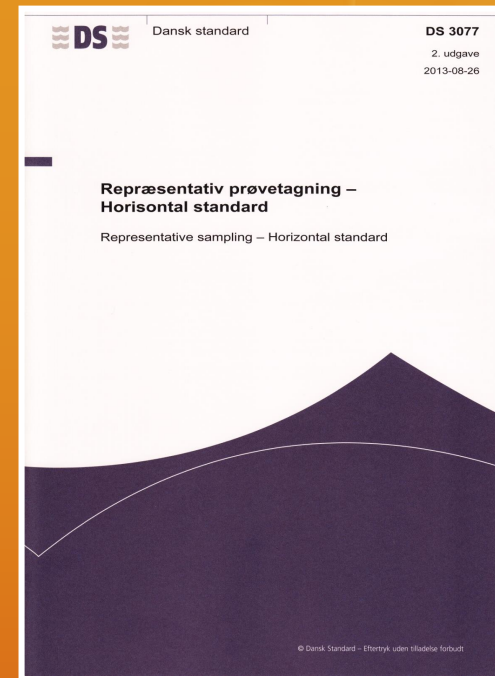
# DS3077 (2013)

## “Representative Sampling – Horizontal Standard”

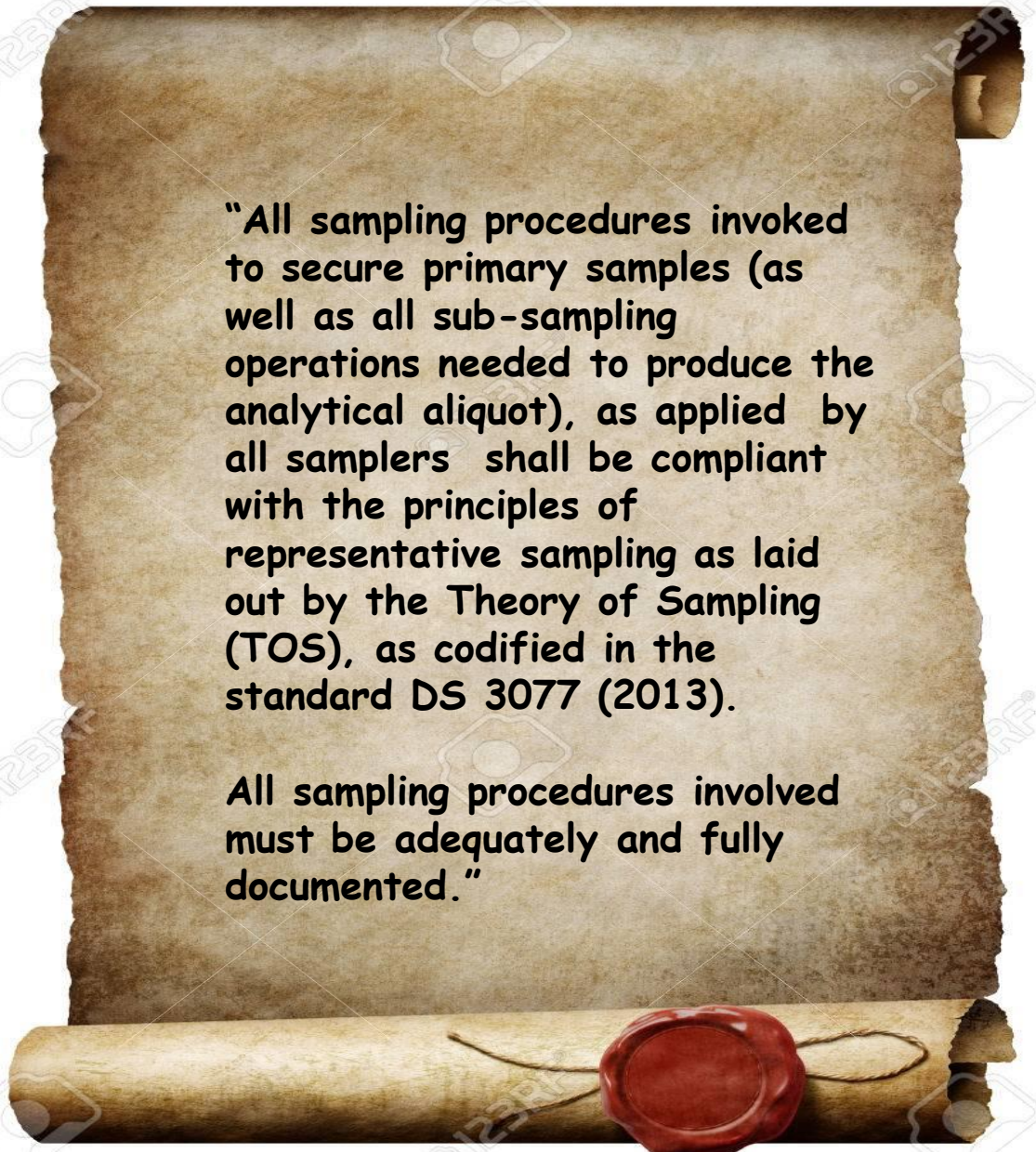
1. Danish Standard, published 2013
2. The only standard on *universal* sampling principles
3. The only comprehensive standard on all necessary and sufficient principles guaranteeing representative sampling
4. The **de facto** international sampling standard
5. Soon to be revised (2021) ...
6. Hereafter to be introduced to become an ISO standard

## 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.



**"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 compliant with the principles of representative sampling as laid out by the Theory of Sampling (TOS), as codified in the standard DS 3077 (2013).**

**All sampling procedures involved must be adequately and fully documented."**

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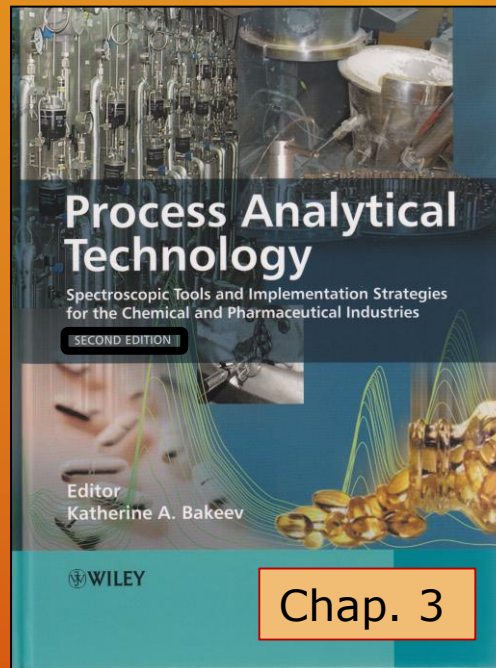
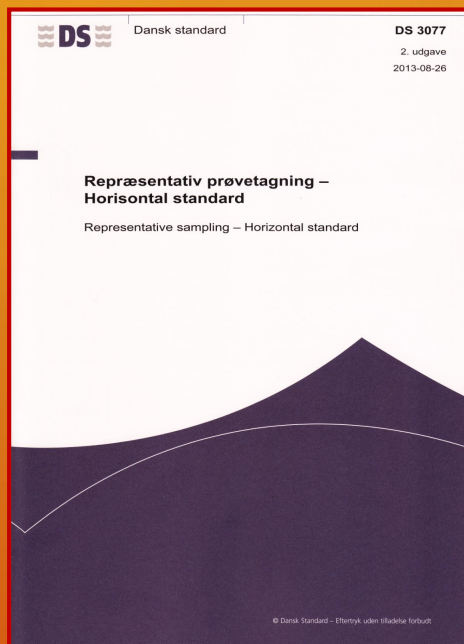


Take a look here:

Find out more and pre-order now from

<https://www.impopen.com/sampling>

# Normative resources: TOS education starts here ..



## REPRESENTATIVE **MASS REDUCTION** IN SAMPLING *- a critical review of techniques and hardware*

*Lars Petersen, Casper K. Dahl & Kim H. Esbensen*

Chemometrics and Intelligent Laboratory Systems, vol. 74 (2004) 95-114

# Digital resources – ATV Soil sampling “Mødemappe”

ENJOY your reading !!!

- INTRO to TOS:
- SE 32-3 Economic costs vs. technical understanding
- The third way to introduce TOS - The legal argument
  
- Illustrative case histories, examples:
- SE 32-4 Sampling of heterogeneous soils, part 1
- SE 32-5 Sampling of heterogeneous soils, part 2



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Thank you for your attention!