



Validation of targeted methods: Where we are?

Presentation at

Eurachem workshop May 2019:

**“Validation of targeted and non-targeted
methods of analysis”**

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LAB Quality International**

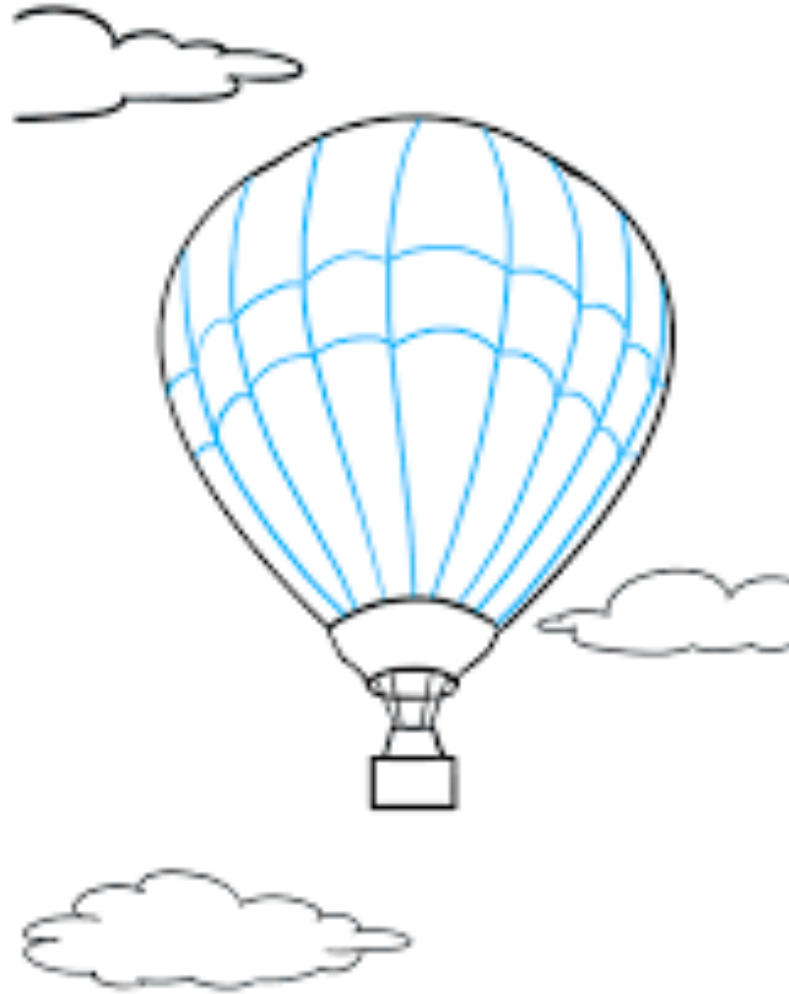


Who am I??

- ✓ Lorens P. Sibbesen (lp@labqa.dk)
- ✓ From Denmark!
- ✓ Chemical Engineer (spec. food chemistry)
- ✓ Add. degree in teaching (teaching analyt. and microbiol. techniques)
- ✓ Working with analyt./microbiol. lab.s and QA in lab.s since 1987
 - Quality Manager for a major Danish Laboratory 1988-2000
- ✓ Started **LAB QUALITY INTERNATIONAL** (April 2000)
- ✓ Member of EUROLAB, Eurolab TCQA, Eurachem GA a.o.
 - Secretary of Eurolab Denmark
- ✓ Chairman of Eurachem Working Group on Method Validation
- ✓ Working on projects for improvement of Quality Infrastructure in e.g. Turkey, Jordan, Syria, Rwanda, Jamaica, The Philippines a.o.

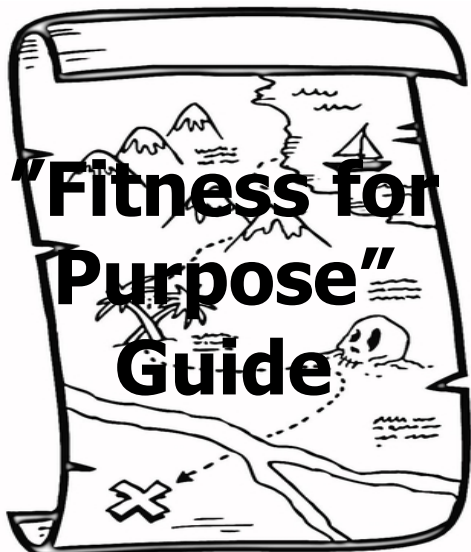
....just FYI ☺

Where are we?



Where are we?

MVWG



**Non-targeted
Methods**

**Measurement
Uncertainty**

**Measurement
Uncertainty**

**Analytical chemistry
(- and microbiology)**

**Measurement
Uncertainty**

Targeted Methods

Who are we: MVWG ?

✓ Eurachem Method Validation Working Group

✓ One of 8 active working groups under Eurachem

✓ Terms of Reference

1. The EURACHEM Method Validation Working Group has been established and will operate in accordance with the Constitution agreed in the EURACHEM Memorandum of Understanding, and is as such working:
2. To provide guidance on method validation. This guidance will be applicable to all chemical analytical laboratories and meet the requirements for accreditation
3. To organize - and contribute to - international seminars and workshops on issues related to Method Validation within analytical chemistry

(cont.)

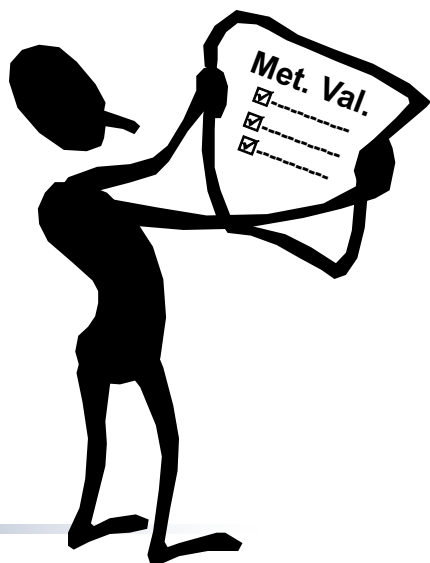
The Eurachem MVWG

✓ Terms of Reference (cont'd)

4. To collaborate and provide input on Method Validation into other Eurachem working groups
 5. To follow and discuss new developments within analytical chemistry, which may require new approaches regarding Method Validation
 6. To contribute to the "Reading List" with relevant references on Method Validation in analytical chemistry.
- ✓ 25 members from 13 different member countries
 - ✓ 1½ day meetings twice per year
 - ✓ Co-arranger of this workshop
 - And contributor to other events
 - E.g. NMKL symposium in Norway, June 3-4 2019

Our Guide

- ✓ **The Fitness for Purpose of Analytical Methods**
- ✓ **A Laboratory Guide to Method Validation and Related Topics**
- ✓ **2nd Edition 2014**



The Fitness for Purpose of Analytical Methods

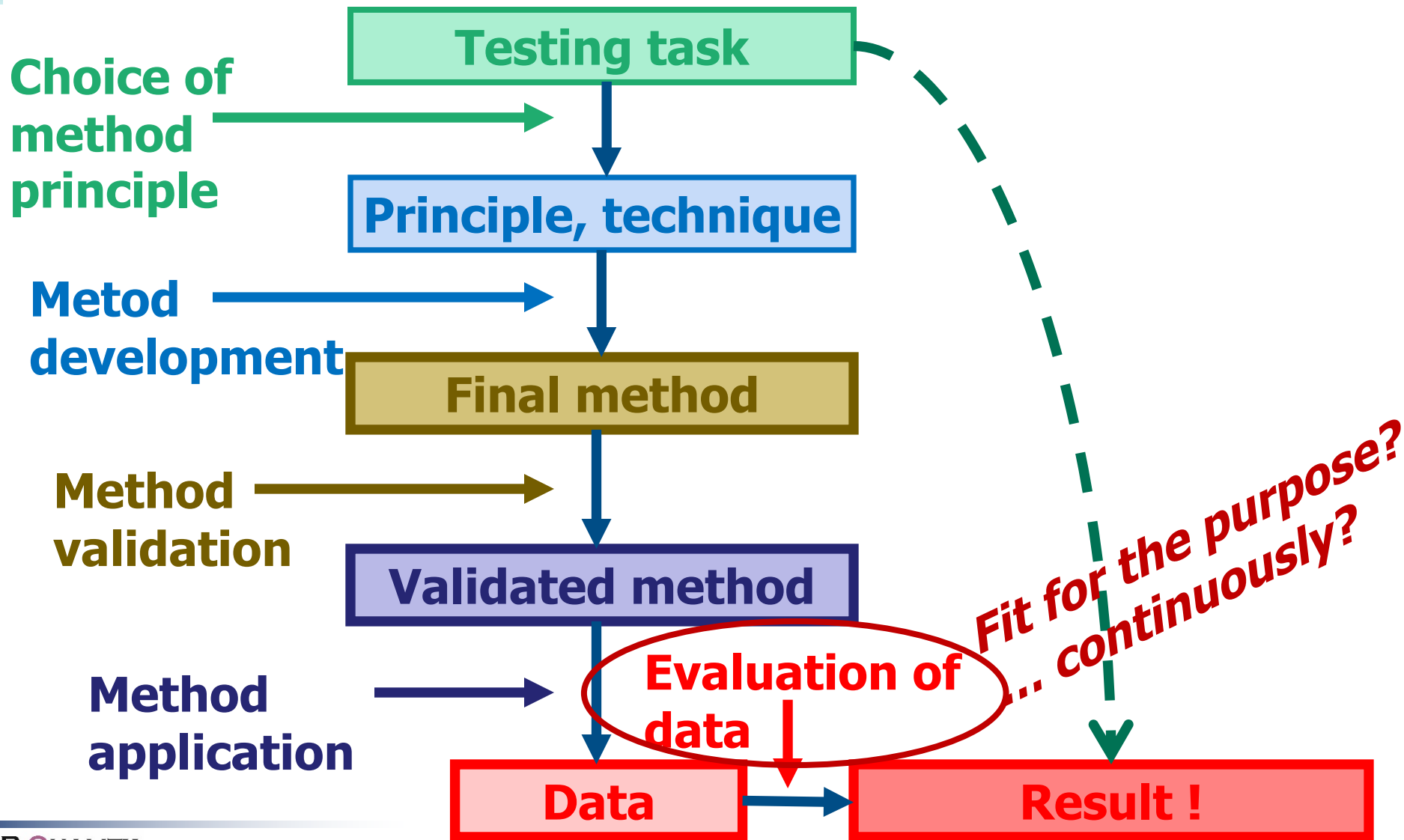
A Laboratory Guide to Method Validation and Related Topics

Second Edition 2014

Validation of **TARGETED** methods

- ✓ Giving ongoing challenges
 - Securing that the method actually IS **targeted**
 - and that we get **a valid result for the target**
- ✓ In focus in the validation of the traditional Performance Characteristics
 - Selectivity
 - not letting the target getting interferred
 - LOD/LOQ
 - the target being present on lower and lower levels
 - Accuracy (trueness & precision)
 - that we can get reliable results for the target
 - being in control of the measurement uncertainty

Challenge: Getting from task to result



What are the new challenges?

- ✓ The process from **Task** to **Result** is undergoing constant changes at an increasing pace!!
 - **New tasks / applications**
 - new needs for analysis
 - new parameters
 - complicated matrices
 - lower levels
 - bulk analysis (routine) / specific analysis (non-routine)
 - prices / competition
 - **More advanced (and sensitive) technologies**
 - new principles of preparation (separation) and detection
 - micro-systems ("Lab on a chip")
 - automation
 - computer power (FT-IR, chemometrics ...)

... the new challenges (cont'd)

- **New methods**
 - multiparameter methods
 - rapid methods (test kits)
 - automated methods (micro-, in-process etc.)
 - bioanalytical methods
 - **Non-Targeted Methods**
- ✓ Can we follow up by **Validating Methods** properly?
 - What does "Fitness for purpose" mean now?
 - **Valid & Reliable results** (currently!)
 - .. but also
 - **Manageable methods** (currently!)
 - **Quick Methods**
 - **Affordable methods**



A few (!) examples of the new challenges!

✓ *Technological development*

- ❑ Electrochemical preparation of **polytiron modified pencil graphite electrode** for **voltammetric detection** of dopamine
- ❑ Detection of hen egg lysozyme by a **labelfree apta-sensor** and **Maldi-TOF**
- ❑ **Sophisticated evolved gas analysis** for the characterization of polymers by means of **STA-FT-IR** and **STA-GC-MS**
- ❑ Analytical determination for copper by **extraction using aliquat 336/TBP based supported liquid membrane**
- ❑ Chemically **modified DNA nano-biosensor** for the **label free detection** of hybridization reaction

Examples! (cont'd)

✓ Applications

- Nanotechnology in the flow domain of **process analysis**
- Analytical investigations in evaluation of the peculiar properties of **polymers with special applications**: optical and medical devices
- **Multiparameter and non-targeted methods** (mostly based on new biotechnological principles) especially very relevant in relation to dealing with food fraud
 - Suggestion for new CEN/TC dealing with “..standardization of validation concepts for non-targeted methods regarding food authenticity”
- Etc. etc.

Do all these fascinating methods provide valid and reliable results in routine use?

Current work in the MVWG

- ✓ The MVWG is currently considering and working on supplementary guidance on individual aspects of method validation (probably (?) to be included in a coming version of the "Fitness for Purpose" guide as annexes)
- ✓ The MVWG is currently assembling issues for revision of the guide (due around 2019). Readers are welcome to provide inputs for this (see web-page)
- ✓ The MVWG is working on illustrative examples (validation protocols) on application of the principles recommended in the "Fitness for Purpose" guide.
- ✓ The MVWG will over time elaborate shorter leaflets pointing to application of the guide and specific issues described in the guide.

On the present agenda of the MVWG

✓ Supplement to the FfP Guide

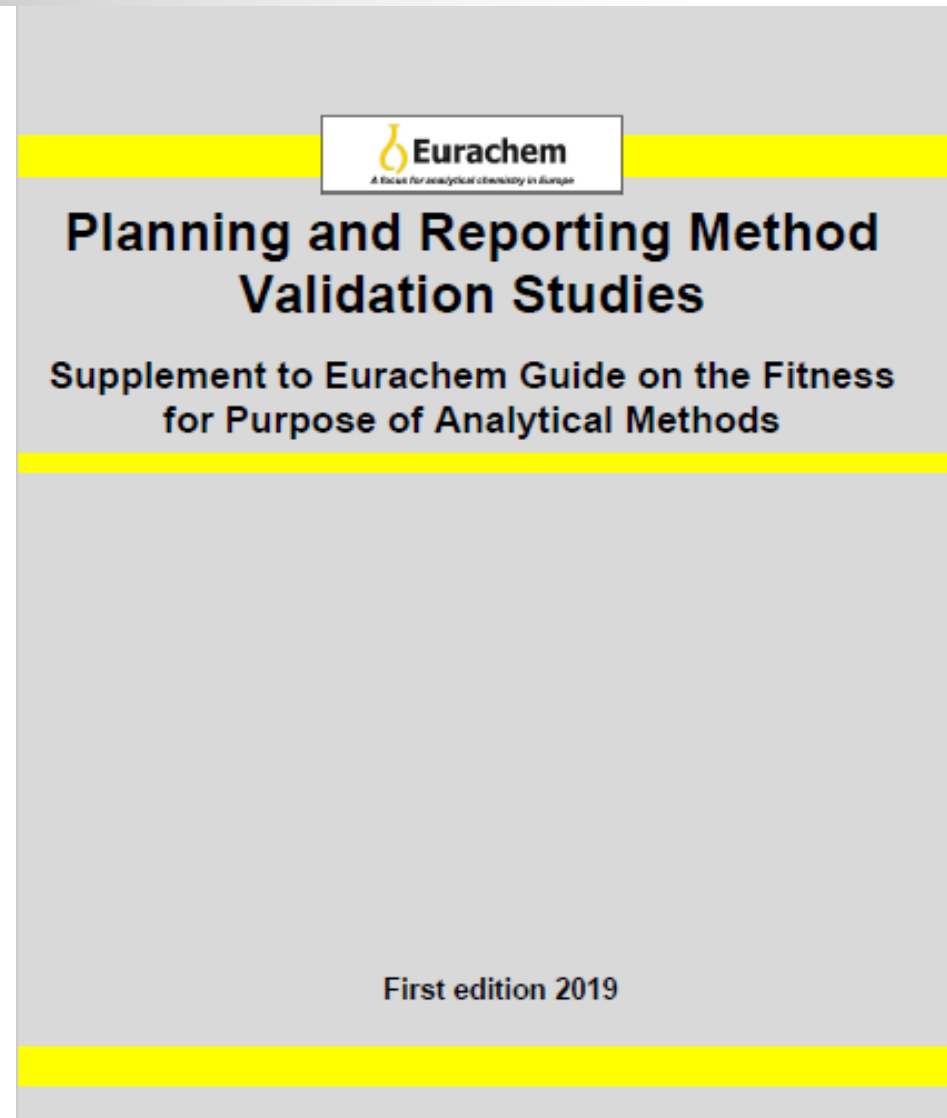
- For final approval at Eurachem GA this week

⇒ Available on web-page

✓ Main content

- General considerations re. planning of validation (& verification) studies in the lab. (i.e. inhouse)
- Protocol for every Perform. Characteristic (ref. to FfP Guide)
- Checklist for preparation

Aiming at covering a need for practical guidance in the laboratory



On the present agenda of the MVWG

- ✓ Supplement to the FfP Guide
 - For final approval at Eurachem GA this week
 - ⇒ Available on web-page
- ✓ Main content
 - Defining meaning – and use of – different types of blanks in relation to Met. Val.
 - Calibration blank
 - Procedural blank
 - Reagent blank
 - Solvent blank
 - Sample blank
 - Incl. situations where no suitable sample blank is available



Blanks in Method Validation

Supplement to Eurachem Guide The Fitness for Purpose of Analytical Methods

First Edition 2019

To be used in conjunction with the FfP Guide

On the present agenda of the MVWG

- ✓ More supplements to the FfP Guide (?):
 - Handling of Bias in Method validation
 - Necessary extent of a validation/verification study
 - Dealing with linearity in Method Validation studies
 - Selection of appropriate Test Kits+ a number of other subjects in "the pipeline"
- ✓ Next revision of the "Fitness for Purpose" Guide
 - Reviewing the present version
 - Asking for opinions about present version
 - ...
 - Handling the many new challenges for -
Targeted Methods

- and Non-Targeted Methods

?

.... thank you for your attention!

