A practical Approach towards Measurement Uncertainty of Tests Methods for Moisture Content

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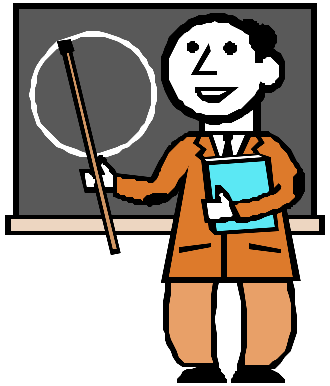
Agenda



- Introduction to measurement uncertainty
- Guidelines and procedures available
- Applied procedure
- Comparison with reproducibility
- Conclusion

What are we talking about?

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- Measurement uncertainty expresses the degree of doubt associated with a measurement
- There is always experimental variations when we make a measurement
- A result should be reported with a tolerance interval

Result: 28 ± 4mg

Requirement for accredited laboratories

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- **ISO/IEC 17025: 5.4.6 Estimation of uncertainty of measurement**

5.4.6.2 Test laboratories should have and should apply procedures for estimating uncertainty of measurement. In certain cases, the nature of the test method may preclude rigorous, metrologically and statistically valid calculation of uncertainty of measurement. In these cases the laboratory should at least attempt to identify all the components of uncertainty and make a reasonable estimation, and should ensure that the form of reporting of the result does not give a wrong impression of the uncertainty. Reasonable estimation should be based on knowledge of the performance of the method and on the measurement scope and should make use of, for example, previous experience and validation data.

Use of measurement uncertainty (MU)

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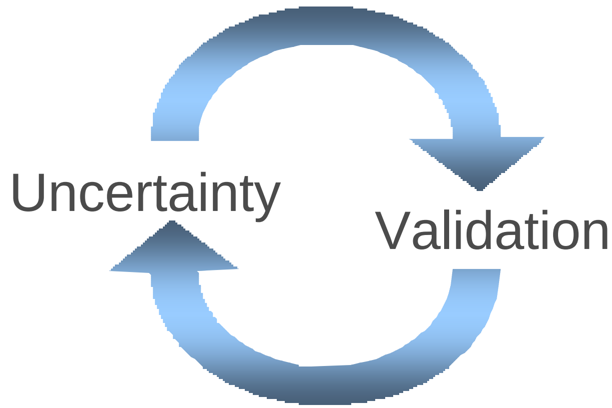
- To enable a result to be interpreted with respect to a limit
- To compare results obtained on the same material from different laboratories
- To reveal those part of a method having the greatest variability during method validation



Guidelines Available

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General

Specific

- **ISO Guide for the expression of uncertainty**
- **Eurachem Guide for quantifying uncertainty in analytical chemistry**
- **VAM Protocol for uncertainty evaluation from validation data**

General Approach of MU

Step 1
Description of the measurement procedure

Flow chart with the description of all steps of the analytical procedure

Step 2
Specification of the measurand

Relationship between the measurand and the variables of the analytical procedure

Step 3
Identification of uncertainty sources

The uncertainty of each variable is identified by means of a cause and effect diagram

Step 4 & 5
Quantification and combination of uncertainties

The above identified uncertainties are quantified and combined to give the total uncertainty on the measurand

Case study: determination of water content by Karl-Fischer

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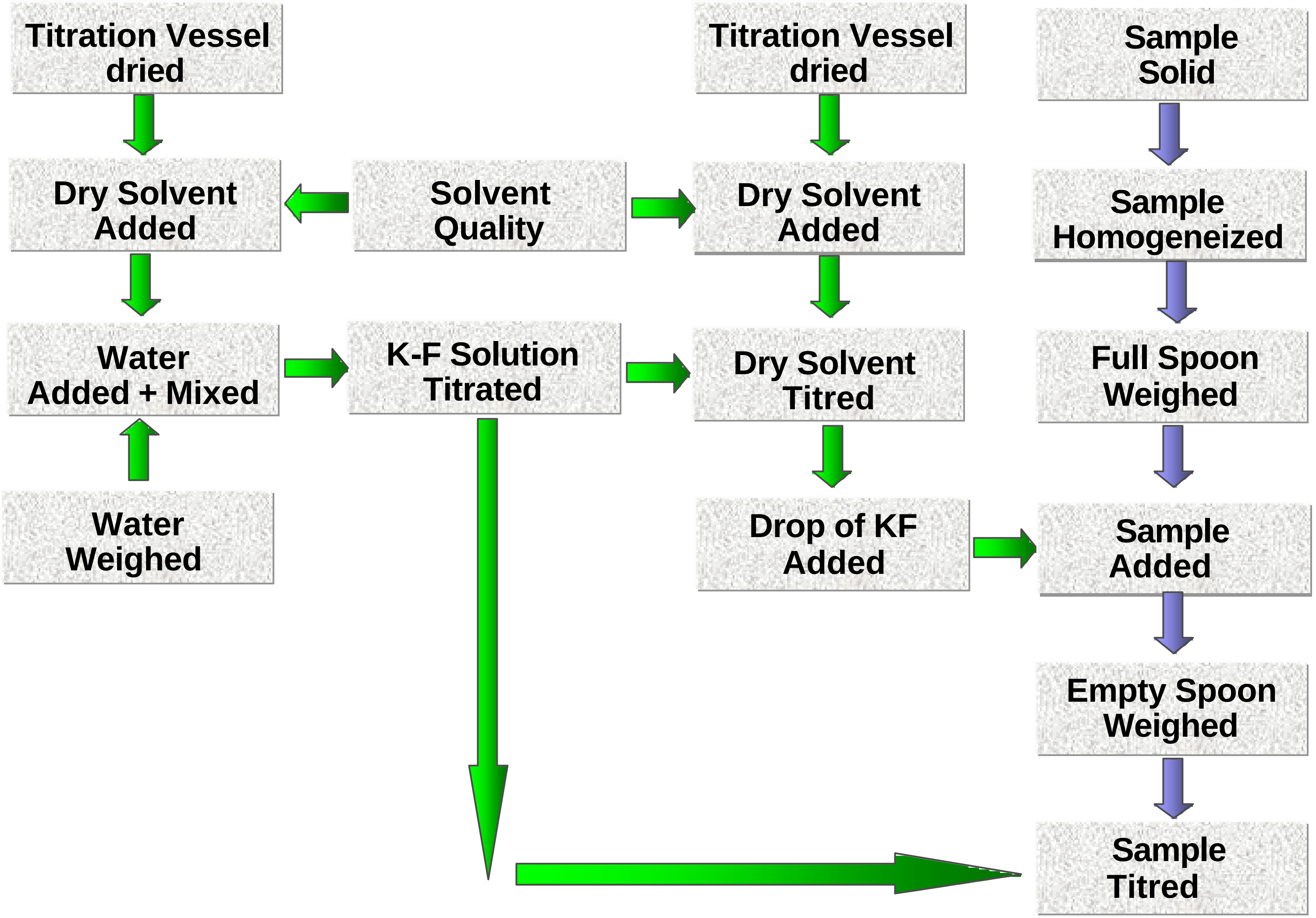

Main relationship of the K-F method

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$$\left(\frac{Vol_{KF} \times t \times 100}{m_{ech} \times 1000}\right) - bias = \text{Mass } H_2O \text{ percent}$$

(g/100g; %)

m_{ech}	**Mass of sample [g]**
Vol_{KF}	**Volume of Karl Fischer solution added for the titration of the sample [ml]**
t	**Water content of the Karl Fisher Solution [mg/ml]**
1000	**Conversion of mg to g**
100	**To express the result in %**
bias	**Bias from the reference value**

Cause and effect diagram for K-F method

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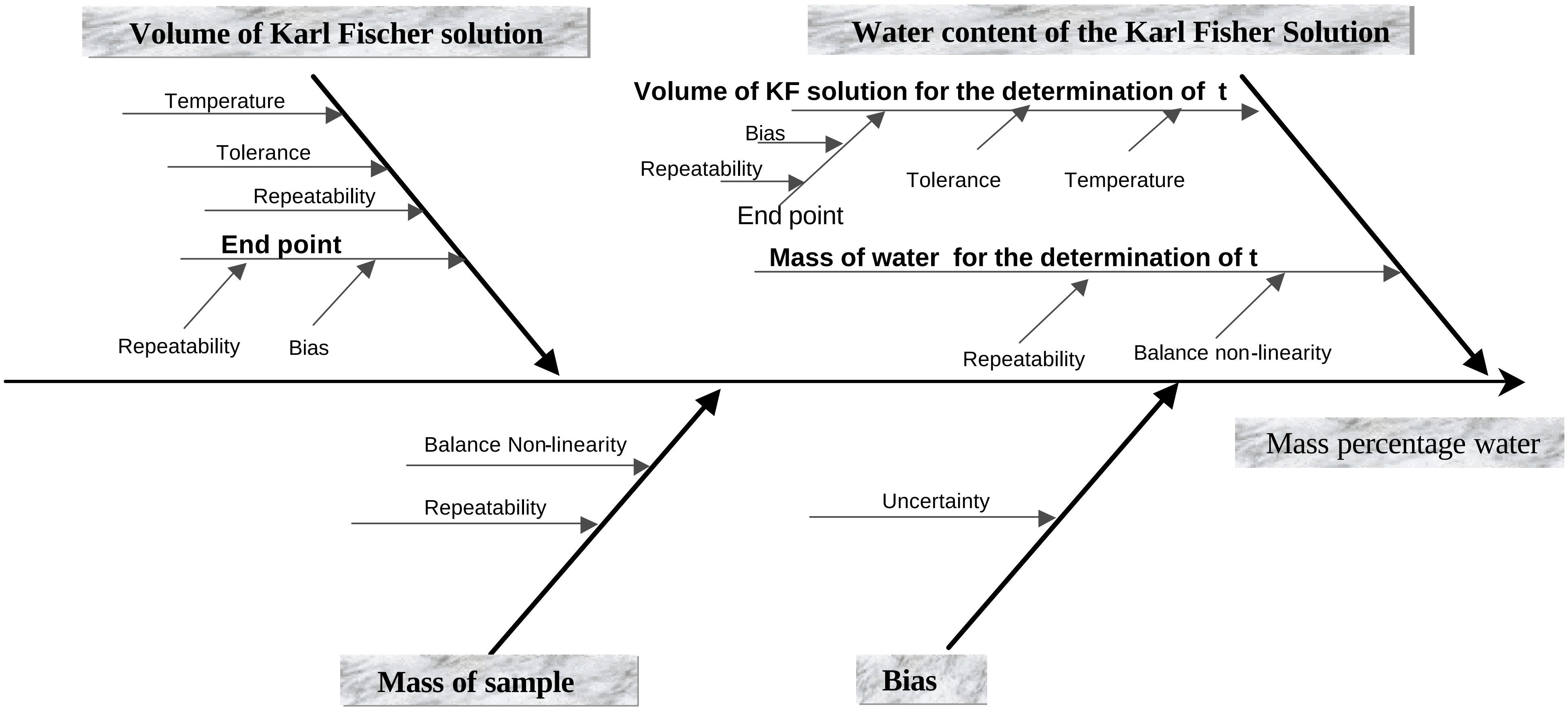

Validation data available for K-F method

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- Precision study: repeatability conditions analysis in duplicates
- Trueness study: experiments with references

Refining the cause and effect diagram:
• Repeatability branch

Refined cause and effect diagram for K-F method

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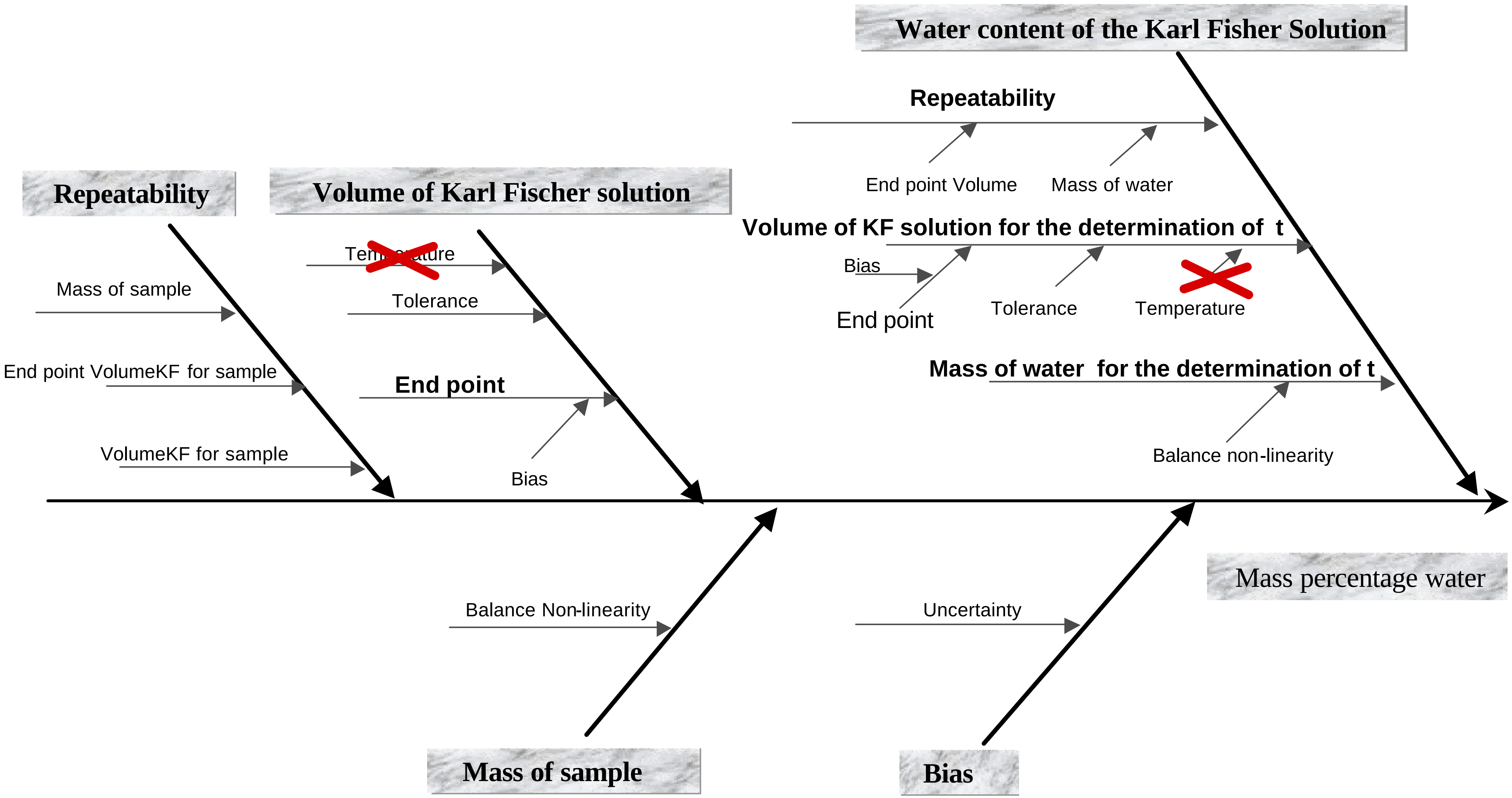

Uncertainty budget for K-F Method: example dietetic milk powder

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Description	Value	Units	Standard Uncertainty u(x)	Relative standard uncertainty u(x)/x
Repeatability (sample)	3.19	g/100g	0.057	0.018
Repeatability (KF water content)	5.2	mg/ml	0.029	0.006
Volume KF (KF water content)	14.3	ml	0.0084	0.0006
Mass of water (KF water content)	75	mg	0.173	0.0023
Volume of KF for sample	0.74	ml	0.0028	0.0037
Mass of sample	0.12	g	0.000173	0.0014
Intermediate standard uncertainty	3.19	g/100g	0.064	0.020
Bias	0.0	g/100g	0.011	na
Standard uncertainty	**3.19**	**g/100g**	**0.065**	**0.021**

Graphical presentation of measurement uncertainty for K-F method

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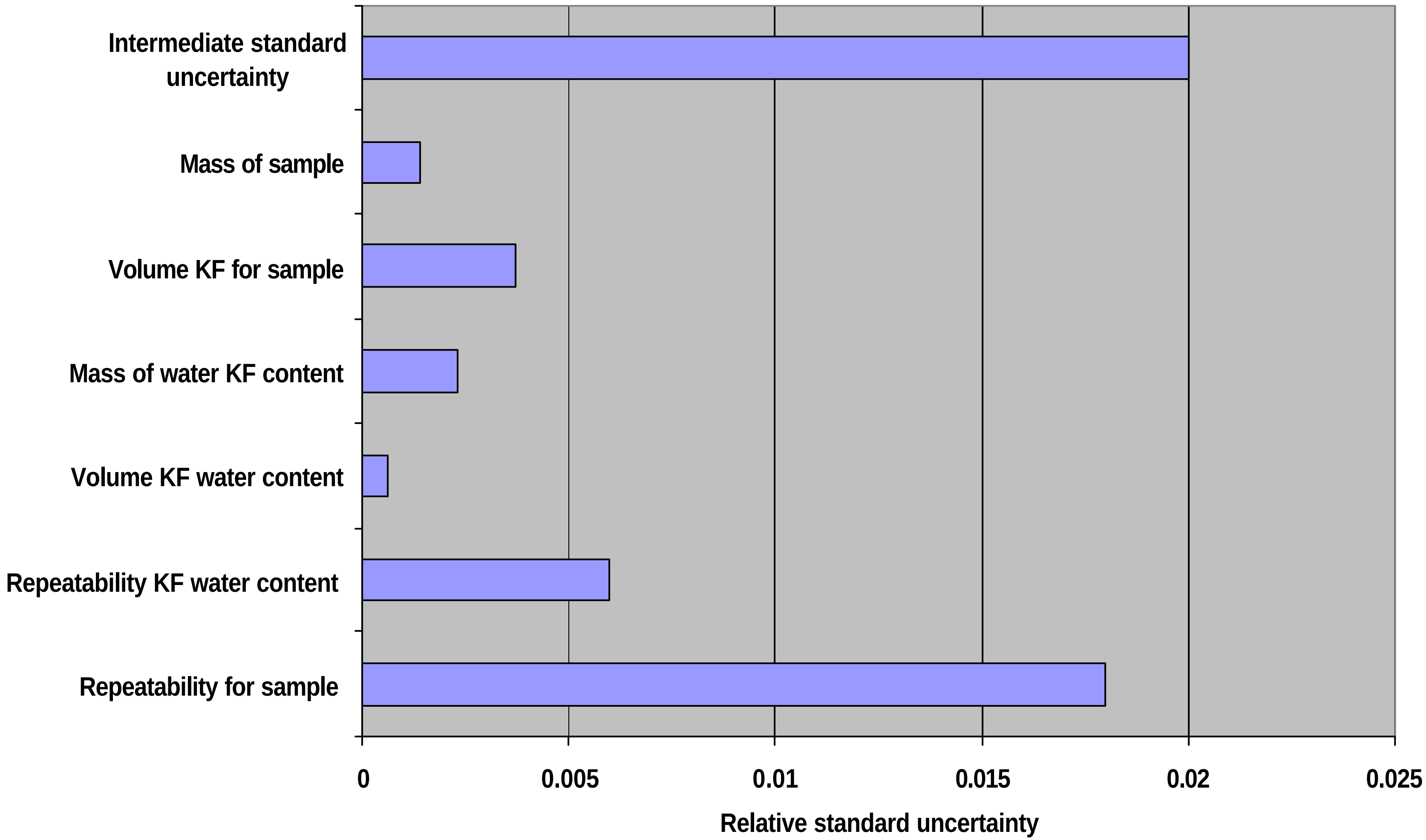
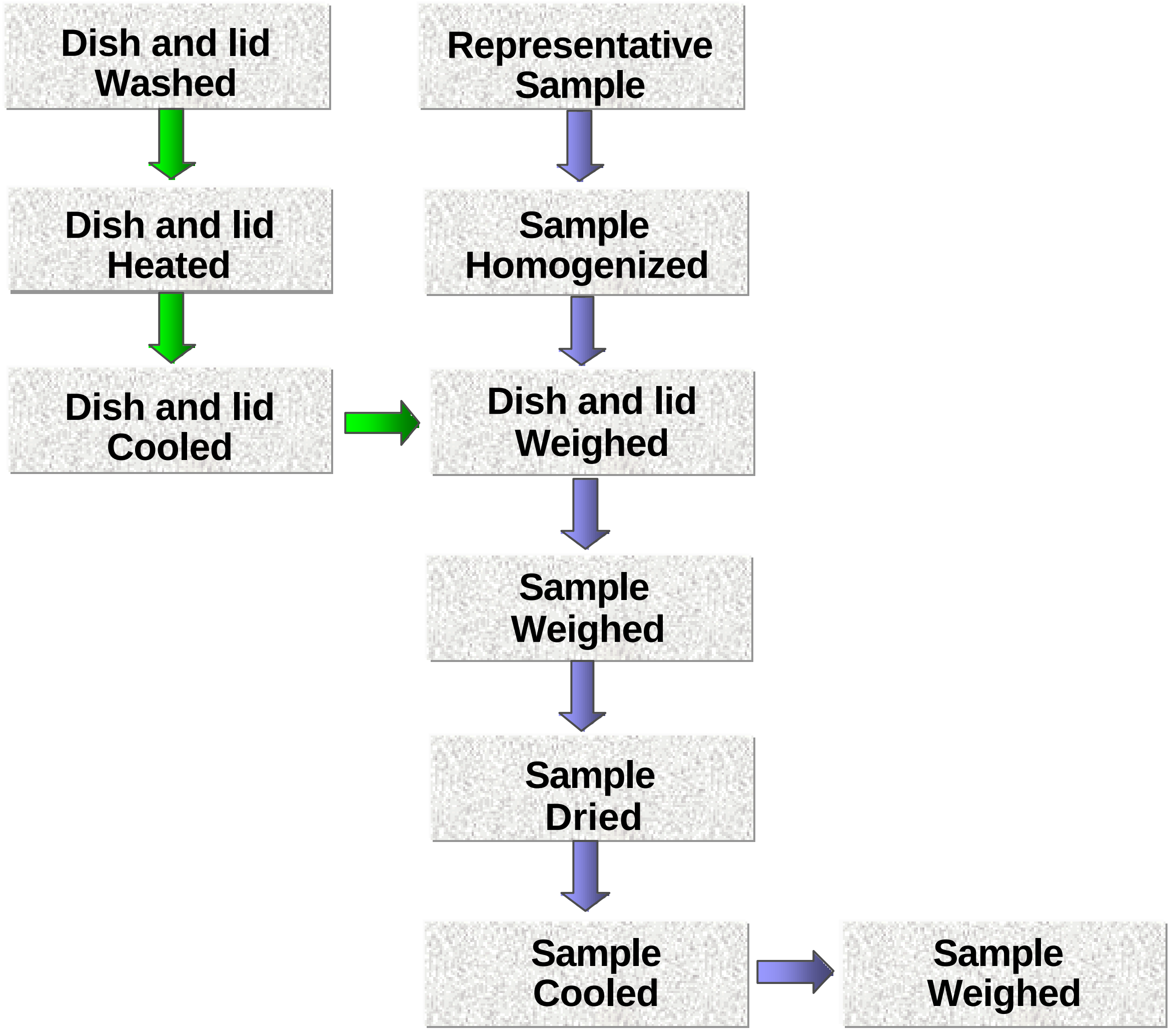


Case study: determination of water content by Oven method



Main relationship for the Oven method

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$$\left[\frac{(m_2 - m_3)}{(m_2 - m_1)} \times 100\right] - bias = \text{Weight loss percent}$$

(g/100g; %)

m_1	**Mass of the dish and its lid [g]**
m_2	**Mass of the dish, the lid and the test portion before drying [g]**
m_3	**Mass of the dish, the lid and the test portion after drying [g]**
bias	**Bias from the reference value**

Uncertainty budget for Oven method: example dietetic milk powder

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Description	Value	Units	Standard Uncertainty u(x)	Relative standard uncertainty u(x)/x
Repeatability	2.77	g/100g	0.04	0.014
M2-M3*	0.068	g	0.000087	0.0013
M2-M1	2.45	g	0.000087	0.000036
Intermediate standard uncertainty	2.77	g/100g	0.04	0.014
Bias	-0.04	g/100g	0.014	0.35
Standard uncertainty	2.81	g/100g	0.042	0.015

* with M1:83.2341, M2:85.6884, M3:85.6208

Comparison of standard measurement uncertainty with precision of P-tests

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Example for dietetic milk powder

Method	Karl-Fischer	Oven
Result Nestlé Research Center	**3.19**	**2.81**
Standard uncertainty	0.065	0.015
Number of tests	8	8
Result P-test	**3.12**	**2.84**
Reproducibility	0.095	0.083
Number of laboratories	37	129

Expression of total uncertainty as an interval

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The standard measurement uncertainty which is a standard deviation is expressed as an interval by multiplying it by a factor k. This factor has been set by convention as 2 for a 95% confidence level and 3 for a 99% confidence level. By default we use k=2

Thus for water content of dietetic milk powder measured by K-F method:

$$3.19 \pm (2 \times 0.065) = 3.19 \pm 0.13$$

Thus for weight loss of dietetic milk powder measured by Oven method:

$$2.81 \pm (2 \times 0.015) = 2.81 \pm 0.03$$

Conclusion

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- The approach is systematic and easy to understand and to verify
- It is pragmatic and based on validation data
- It can be integrated in validation study
- It can be transferred to other laboratories

→ **Fulfills ISO 17025**

Thank you for your attention !!!