



Method validation and quality assurance course at the University of Jyväskylä

Ari Väisänen



Contents

- KEMS3040 Measurement Uncertainty and Validation of Analytical Methods 4 ECTS
- Course is based on the book Miller, J.N. & Miller, J.C., Statistics and Chemometrics for Analytical Chemistry, Prentice Hall.
- Error types and their sources
- Statistics of repeated measurements
- Significance tests
- The quality of analytical measurements
- Calibration methods in instrumental analysis





- Intended for 4- or 5-year students
- First lectures and then exercises (using Microsoft Excel)
- The same calculations are practiced in advanced laboratory works (10-18 ECTS)
- The course can include the written exercise (2-4 ECTS)
- Course is scheduled just before Masters thesis

Handwritten calculations on a piece of paper:

$$1000 \text{ ml } \text{H}_2\text{O}_2 = 607.2 \text{ g}$$
$$n(\text{H}_2\text{O}_2, 1000 \text{ ml}) = \underline{1741,}$$



Statistics of repeated measurements

- Mean and standard deviation
- Confidence limits of the mean
- Presentation of results (the accuracy with which the result is informed)

14.784 mg L⁻¹

14.784 ± 0.167 mg L⁻¹

14.8 ± 0.2 mg L⁻¹





Significance tests

- Probably the most important part of the course
 - Comparison of the experimental mean with the certified value
 - Comparison of two experimental mean
 - Paired t-test

1. Zn concentrations in carbide blade raw material was analyzed using FAAS (standard method) and ICP-OES (new method).

ICP	0.242	0.240	0.239	0.244	0.241	0.238 mg/g
FAAS	0.252	0.247	0.242	0.248	0.248	0.246 mg/g

Is the difference between the methods significant?

Zn is a metal that weakens carbide blades, the concentration of which should not exceed the 0.025 %. Is the copper concentration at the acceptable level?

$$t = \frac{|\bar{x} - \mu|}{s / \sqrt{n}}$$

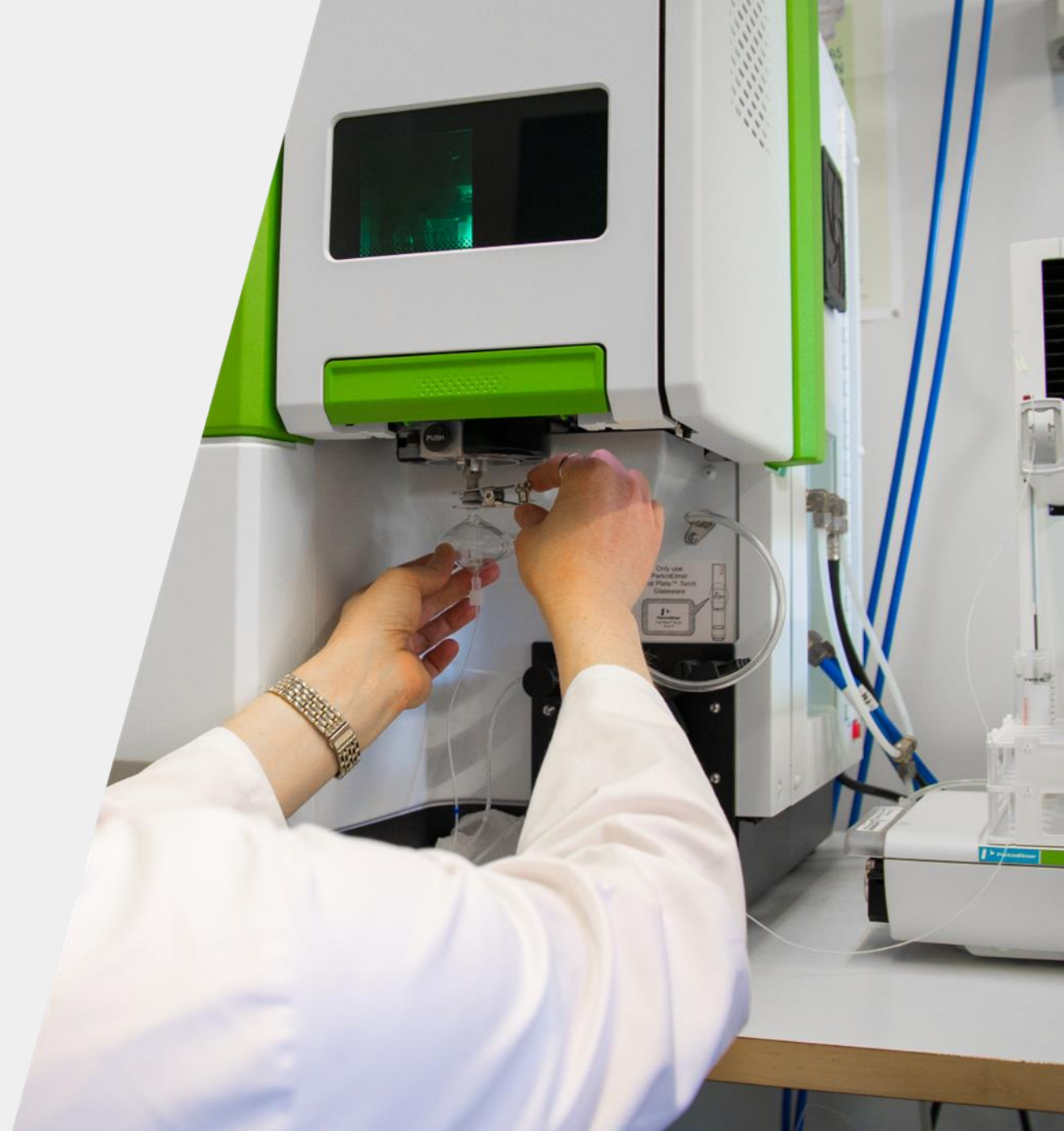
$$|t| = \frac{\bar{x}_1 - \bar{x}_2}{s \sqrt{\frac{1}{n_1} + \frac{1}{n_2}}}$$

$$|t| = \frac{\bar{d} \sqrt{n}}{s_d}$$



The quality of analytical measurements

- Sampling
- Quality control methods
 - standard reference materials
 - internal quality control standards
 - recovery tests
- Shewhart charts
- Proficiency tests
- Uncertainty
 - MUKIT taught by Teemu Näykki
- Other lecturer from the industry





Calibration

- Principles of trace element analysis
- Preparation of calibration standard solutions
- Linear regression and correlation coefficient
- LOD and LOQ

**Thank you for your
attention!**

