Big Issues in Analytical Chemistry—the Work of the Analytical Methods Committee

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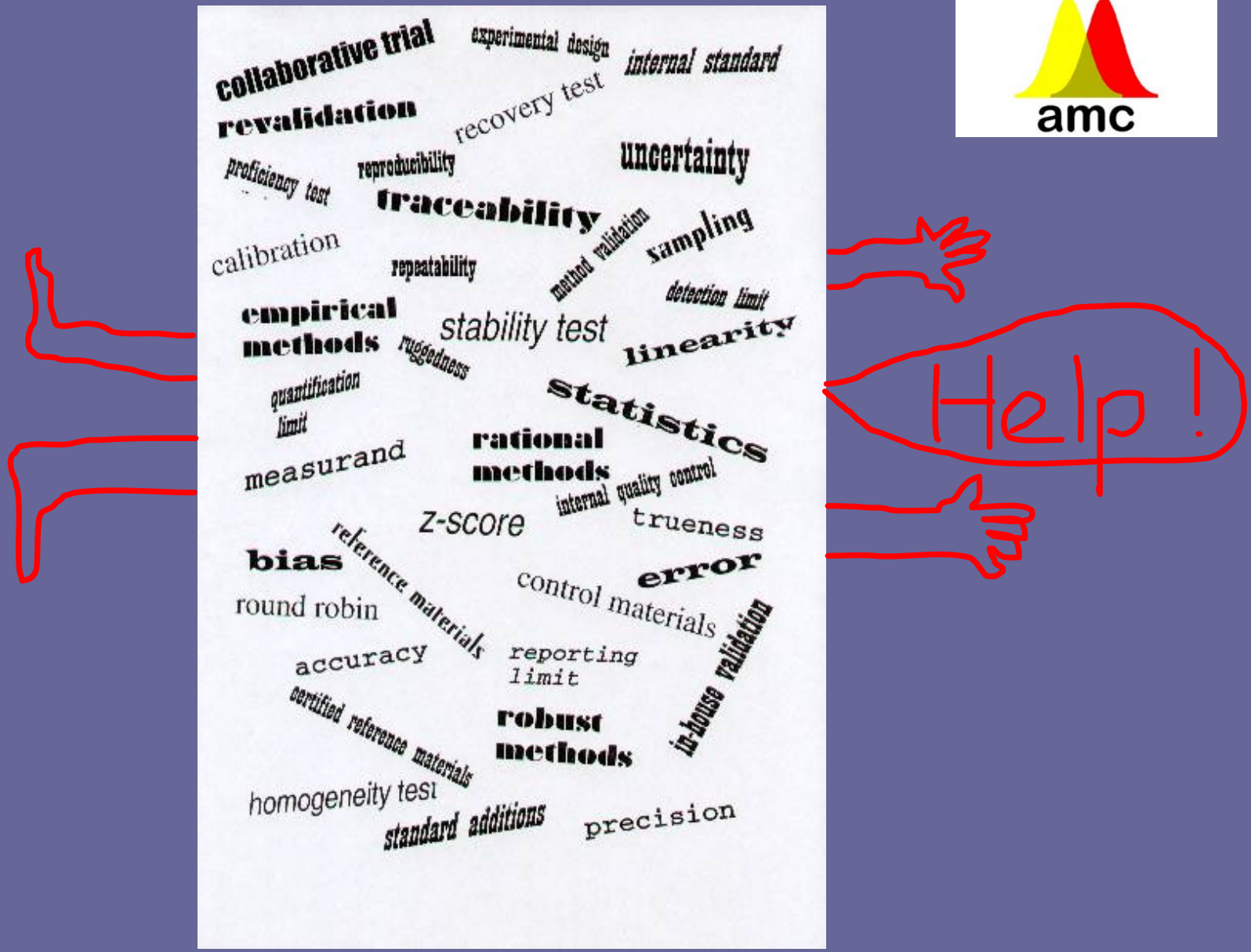
(Editor of *AMC Technical Briefs*)


collaborative trial
experimental design
internal standard
revalidation
recovery test
uncertainty
proficiency test
reproducibility
traceability
calibration
repeatability
method validation
sampling
detection limit
empirical methods
stability test
linearity
ruggedness
quantification limit
statistics
rational methods
measurand
internal quality control
z-score
trueness
reference materials
bias
error
round robin
control materials
accuracy
reporting limit
in-house validation
certified reference materials
robust methods
homogeneity test
standard additions
precision
Help !

AMC?
What? Who? How?

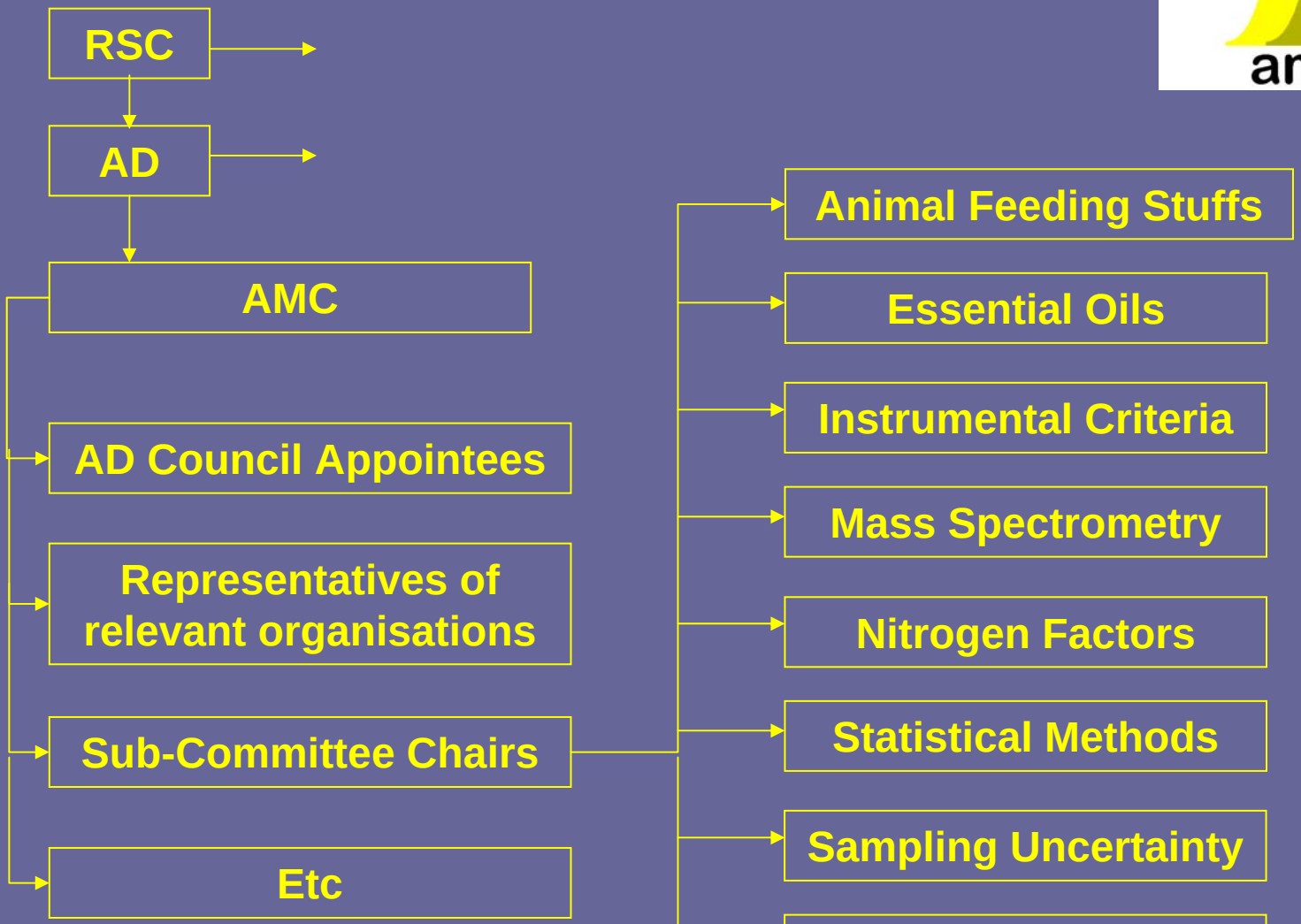
- A subcommittee of the Analytical Division Council.
- Voluntary (unpaid) members.
- Activities supported by donations from industry and research contracts.
- Recent finance principally from the Food Standards Agency.

Aims

- The broad aim of the AMC is to participate in national and international efforts to establish a comprehensive framework for appropriate quality in chemical measurement, and to keep the analytical science community informed of developments.
- It achieves this aim through the activities of its expert sub-committees as well as the parent Committee, which fulfil the following functions:

Intentions

- Development, revision and promulgation of validated, standardised and official methods of analysis
- Development and establishment of suitable performance criteria for methods and instruments
- Use and development of appropriate statistical methods
- Identification and promulgation of best analytical practice, including aspects relating to sampling, equipment, instrumentation and materials
- Generation of validated compositional data of natural products for interpretative purposes.

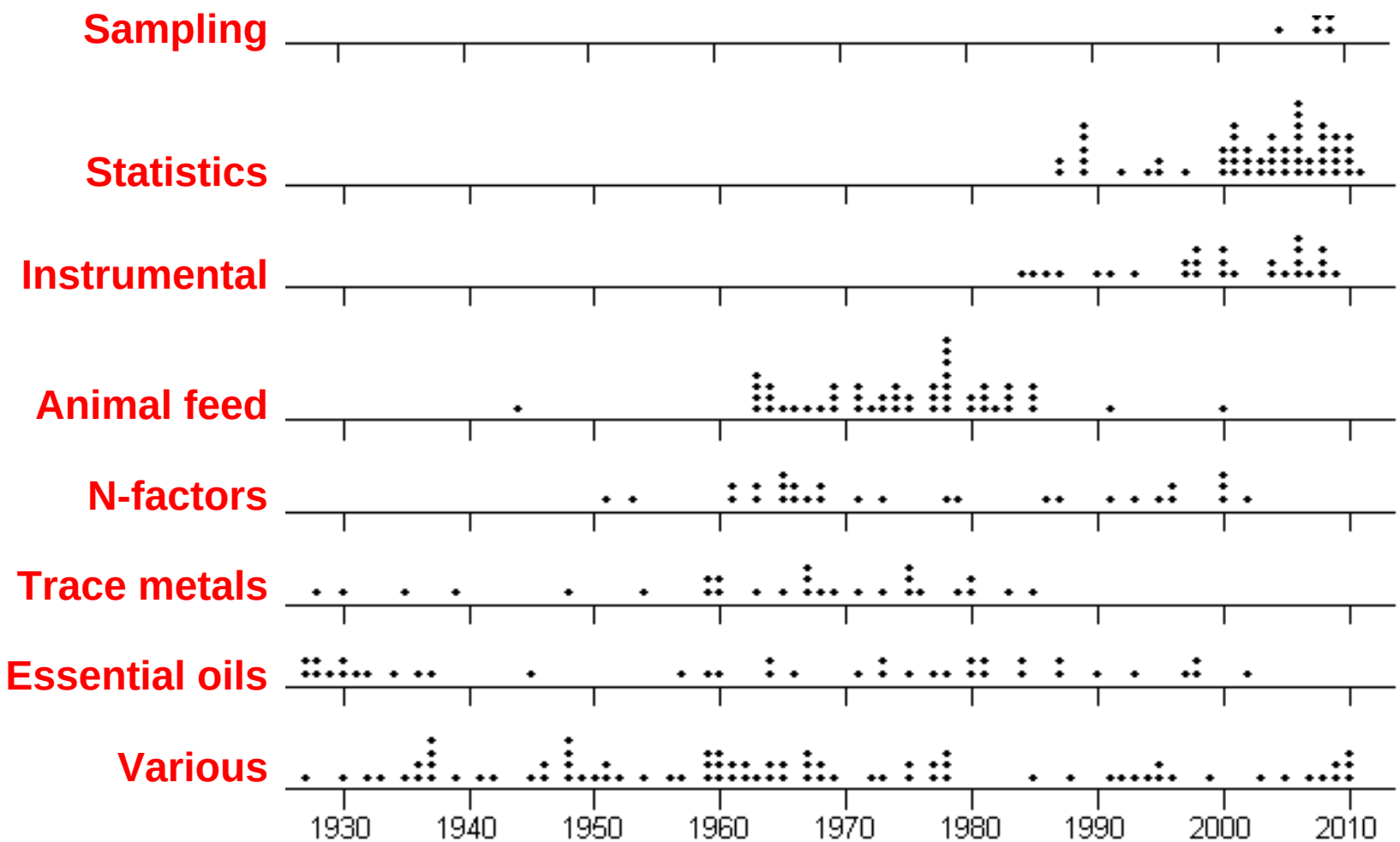

RSC
AD
AMC
AD Council Appointees
Representatives of relevant organisations
Sub-Committee Chairs
Etc
Animal Feeding Stuffs
Essential Oils
Instrumental Criteria
Mass Spectrometry
Nitrogen Factors
Statistical Methods
Sampling Uncertainty
Method Validation

AMC outputs

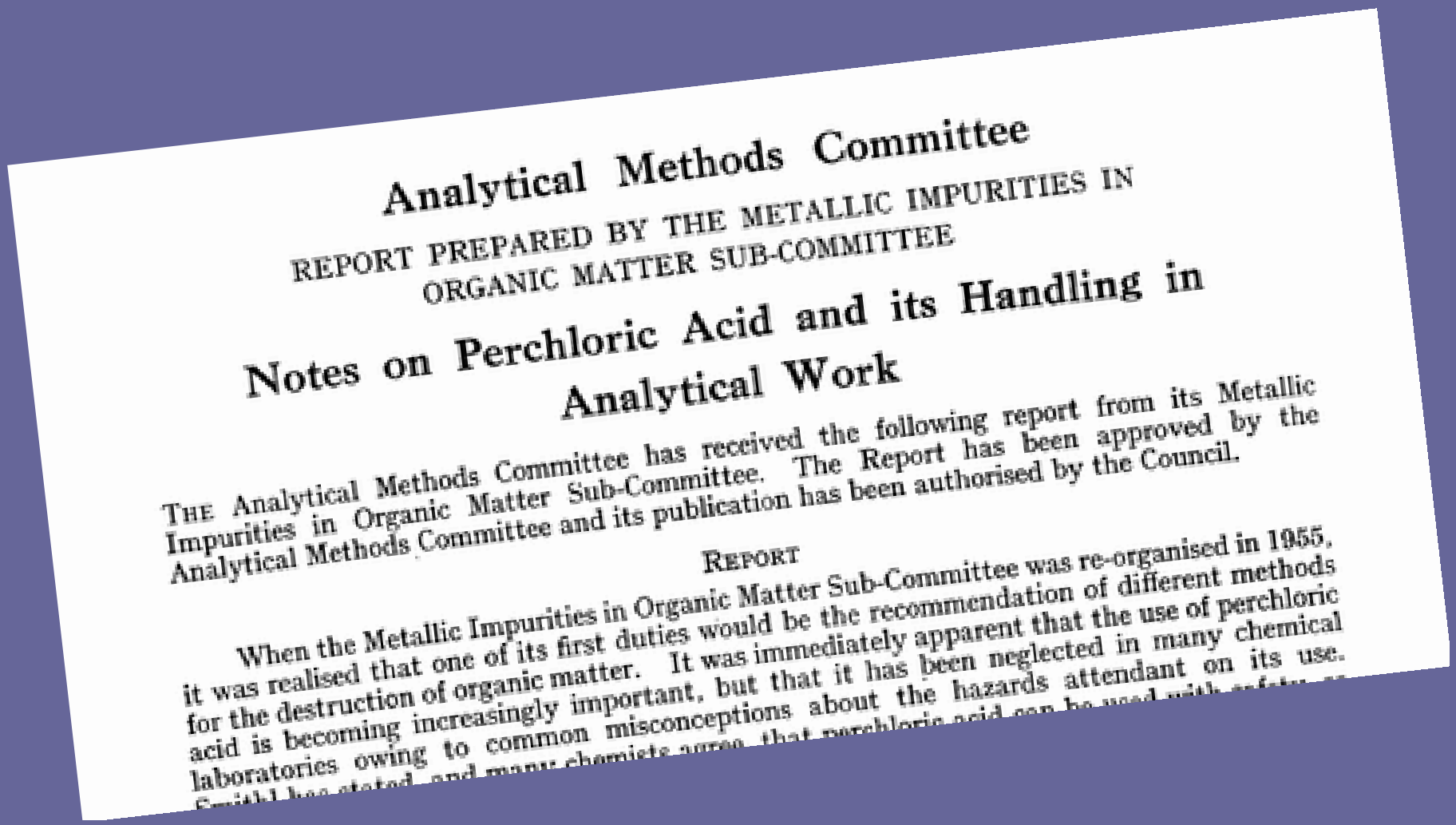
- Reports
  AMC Technical Briefs
  Datasets
  Presentations
  Software?

- www.rsc.org/amc

AMC Reports and TBs by year



Analyst, 1959, **84**, 214-216



Analytical Methods Committee

REPORT PREPARED BY THE METALLIC IMPURITIES IN ORGANIC MATTER SUB-COMMITTEE

Notes on Perchloric Acid and its Handling in Analytical Work

THE Analytical Methods Committee has received the following report from its Metallic Impurities in Organic Matter Sub-Committee. The Report has been approved by the Analytical Methods Committee and its publication has been authorised by the Council.

REPORT

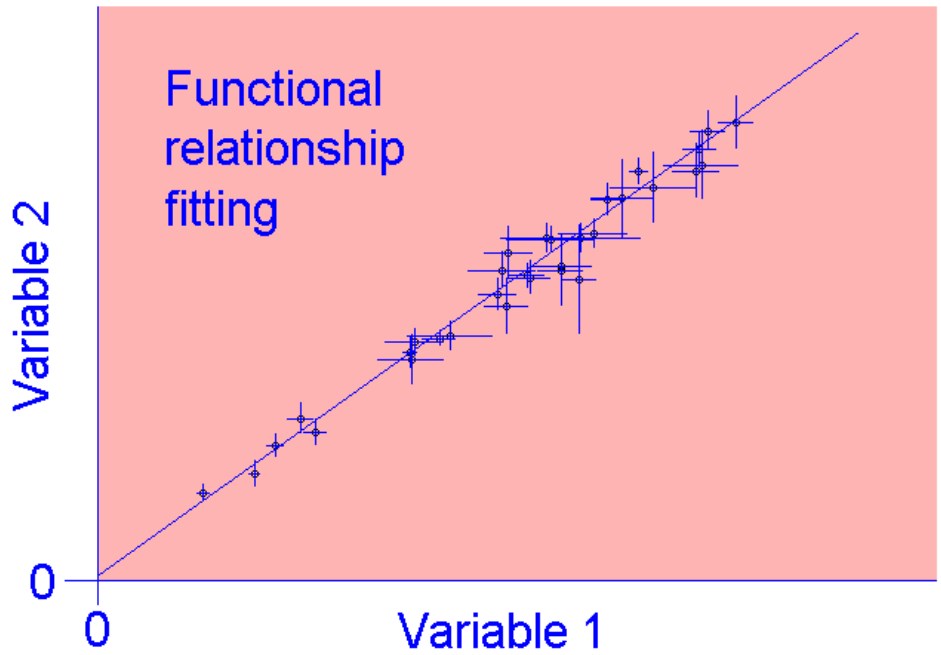
When the Metallic Impurities in Organic Matter Sub-Committee was re-organised in 1955, it was realised that one of its first duties would be the recommendation of different methods for the destruction of organic matter. It was immediately apparent that the use of perchloric acid is becoming increasingly important, but that it has been neglected in many chemical laboratories owing to common misconceptions about the hazards attendant on its use.

Statistical Subcommittee

Example 1—functional relationship

- *AMC Technical Briefs* No 10:
  "Fitting a linear functional relationship to data with error on both variables".
- *AMC Software*: Excel Add-in.
- *AMC Datasets No 24*:
  "Dissolved oxygen method comparison".

Don’t use regression!



Example 2: Robust methods

- A robust method for the estimation of mean and standard devation is “Huber’s H15” .
- The statistics (*e.g.*, mean and standard deviation) are defined by an ***algorithm,*** not by equations.
- *AMC Technical Briefs* No 6: “Robust statistics: a method of coping with outliers”.
- *AMC Software*: Excel add-in for mean, standard deviation, and ANOVA.

Huber's H15

$$\mathbf{x}^{\mathrm{T}} = \begin{bmatrix} x_1 & x_2 & \cdots & x_n \end{bmatrix}$$

amc

Set $1 < k < 2, \quad p = 0, \quad \hat{\mu}_0 = \text{median}, \quad \hat{\sigma}_0 = 1.5 \times \text{MAD}$

$$\tilde{x}_i = \begin{cases} x_i & \text{if} \quad \hat{\mu}_p - k\hat{\sigma}_p < x_i < \hat{\mu}_p + k\hat{\sigma}_p \\ \hat{\mu}_p - k\hat{\sigma}_p & \text{if} \quad x_i < \hat{\mu}_p - k\hat{\sigma}_p \\ \hat{\mu}_p + k\hat{\sigma}_p & \text{if} \quad x_i < \hat{\mu}_p + k\hat{\sigma}_p \end{cases}$$

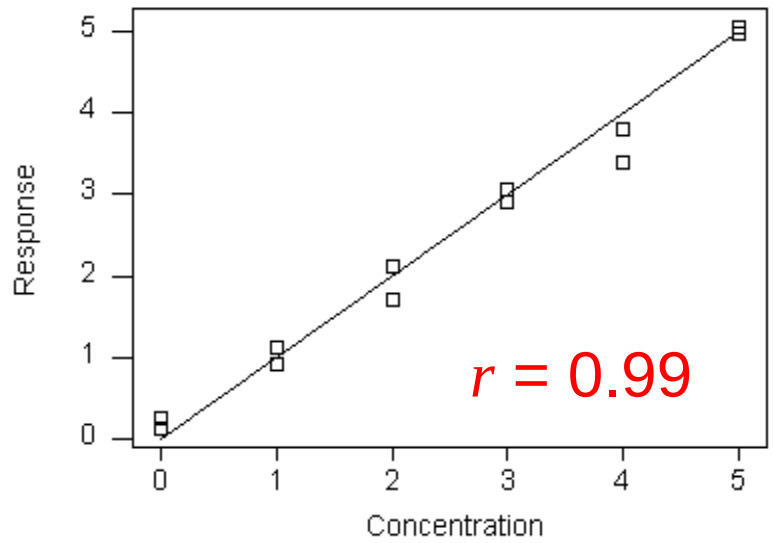
$$\hat{\mu}_{p+1} = \text{mean}(\tilde{x}_i)$$

$$\hat{\sigma}^2_{p+1} = f(k)\,\text{var}(\tilde{x}_i)$$

If not converged, $\quad p = p + 1$

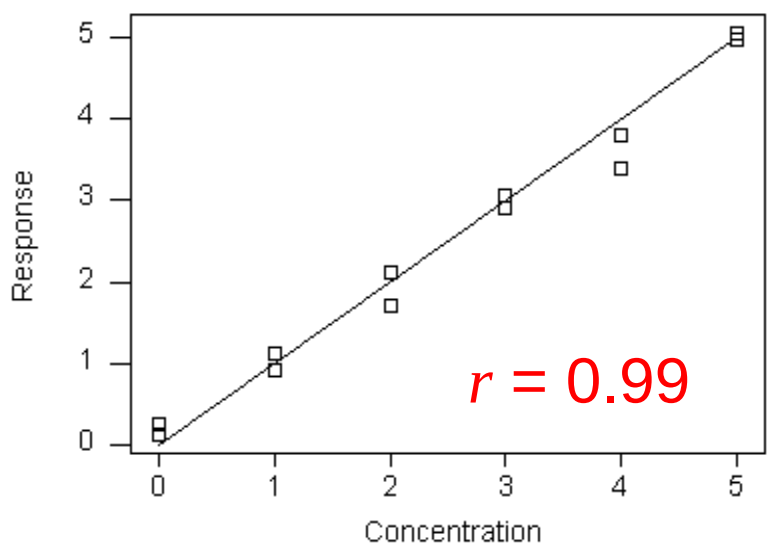
Example 3—
Is my calibration linear?

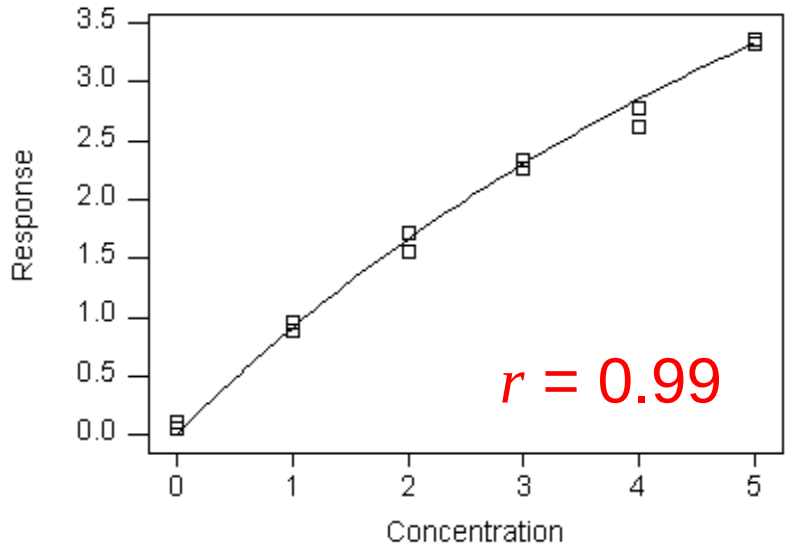
AMC Technical Briefs No 3



Is my calibration linear?

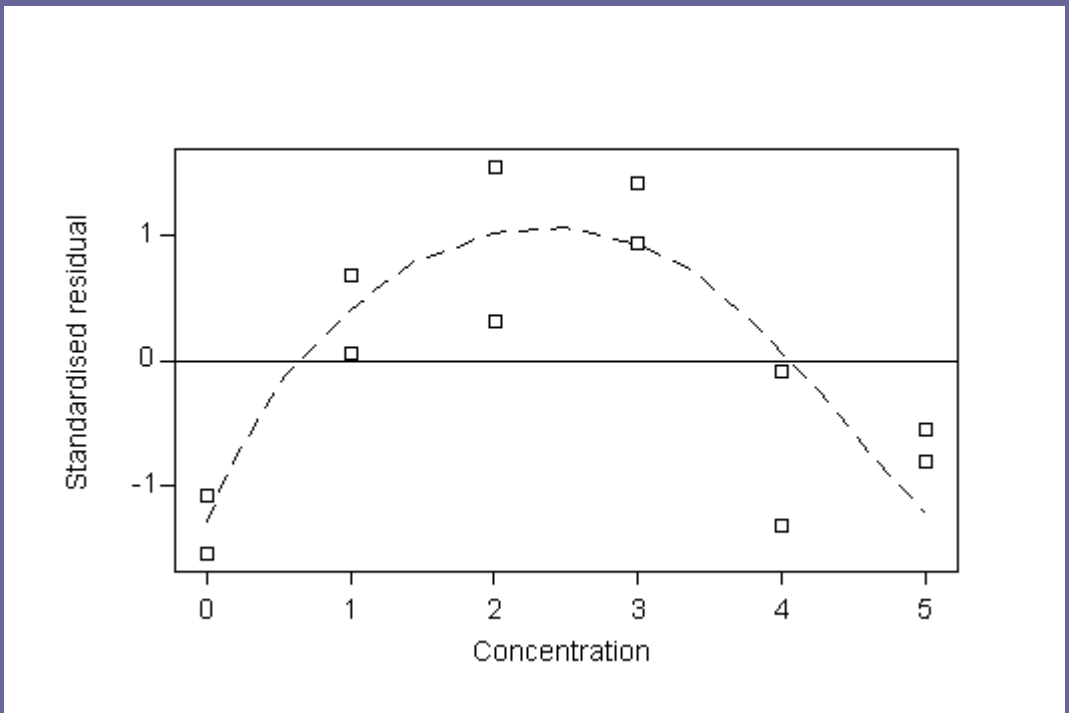
AMC Technical Briefs No 3





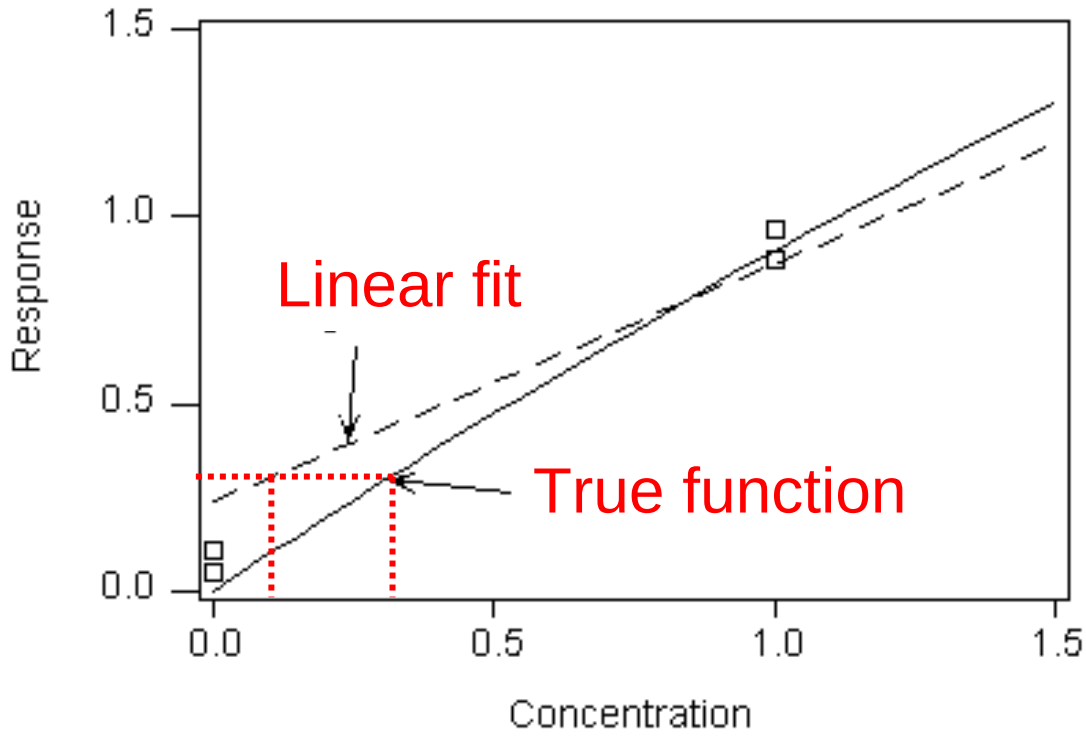
Problem with the correlation coefficient

Always look at a residual plot



Duplicate readings and use a significance test for lack of fit

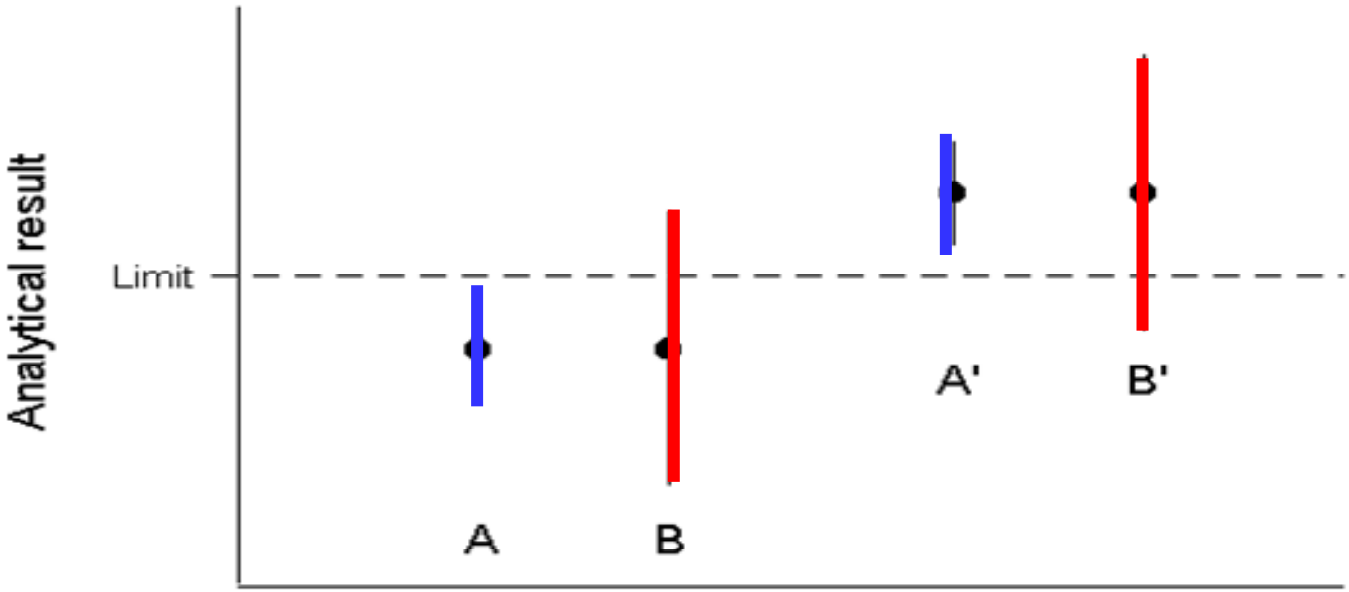
Potential errors at low concentrations



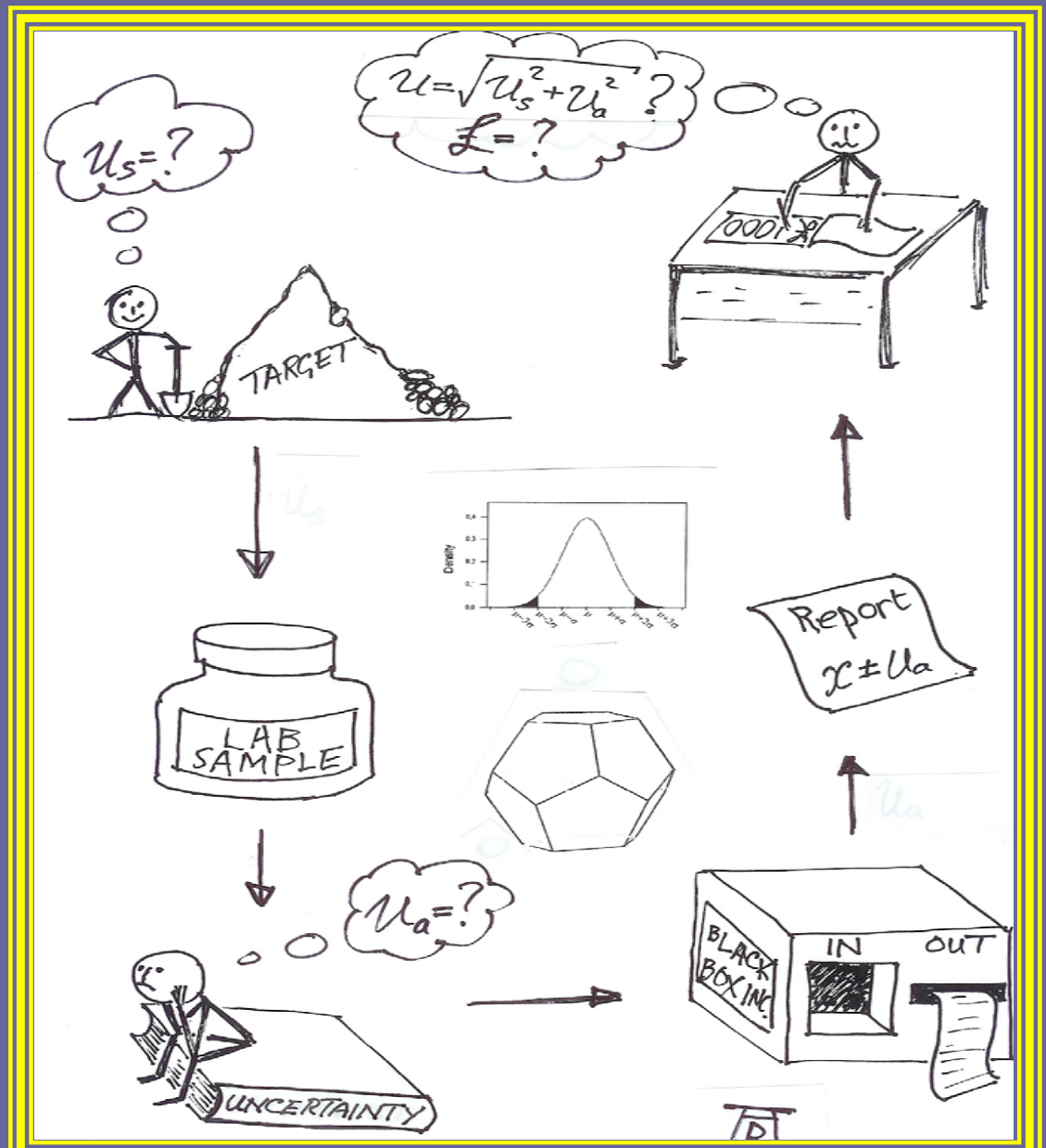
Uncertainty from Sampling

Why do analysts need to bother with sampling uncertainty?

u_{an} and u_{sam} combined

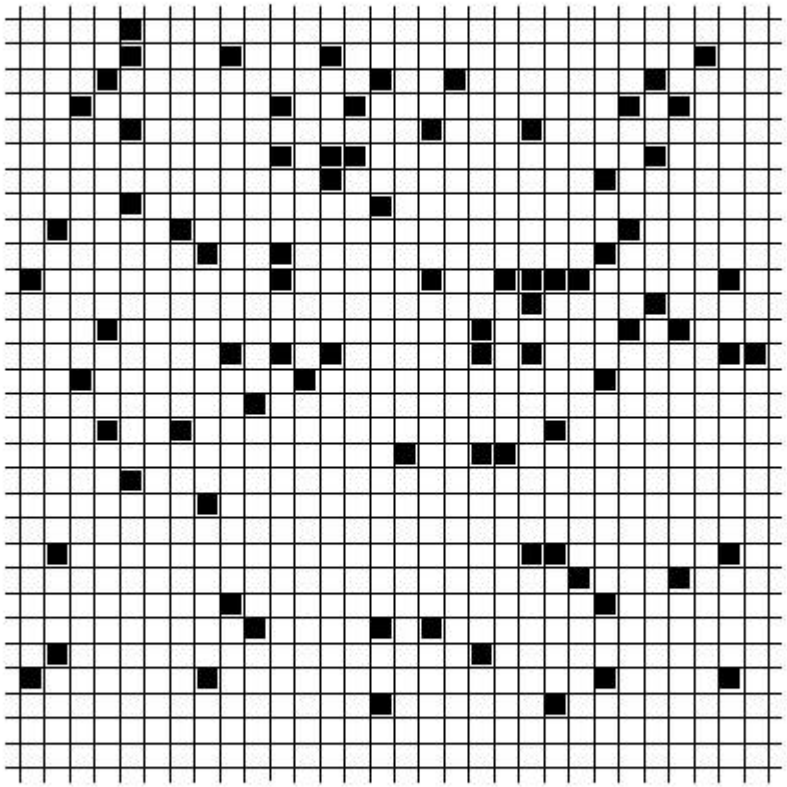


u_{an} alone

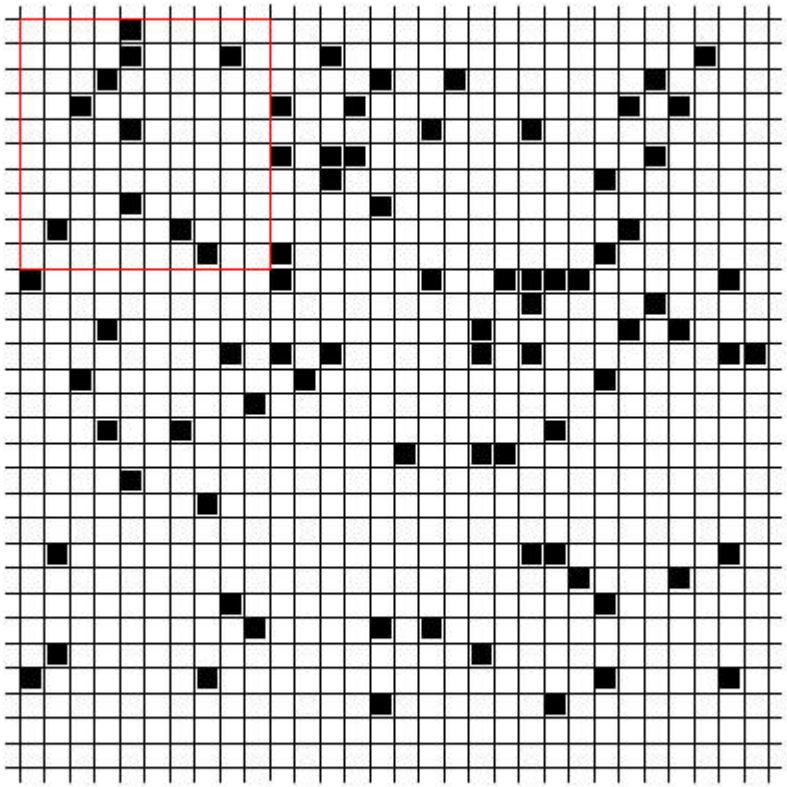


Uncertainty (after Dürer)

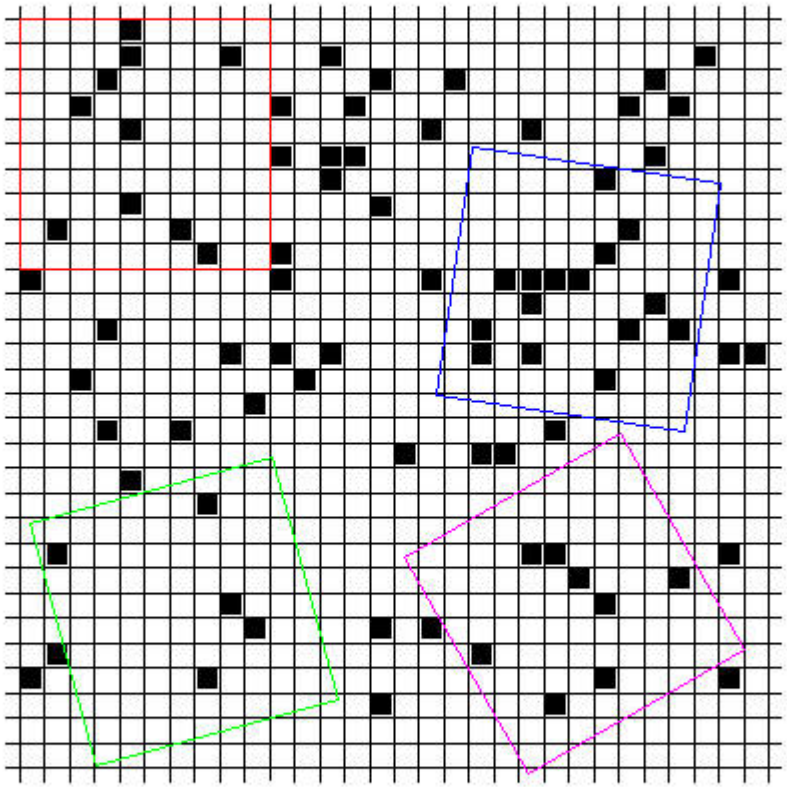
True = 10 %



True = 10 %



True = 10 %



10 % 15 % 8 % 5 %

Sampling uncertainty

- “Measurement uncertainty arising from sampling—a guide to methods and approaches”. Eurachem/CITAC/Eurolab/Nordtest/***AMC***, 2007.

- How UfM arises
- How you estimate it
- Sampling quality control

Lettuce—13-increment sample

Essential oils

Establishing authenticity via gas chromatograms obtained under standardised conditions.

UKAS role of the AMC

- United Kingdom Accreditation Service.
- The AMC has recently been appointed by UKAS as the Advisory Board for chemical measurement.

Could I get involved?

- Contacts via the AMC Chair or Subcommittee Chair.
- Comments, suggestions, ideas *via* MyRSC.