KUNSTSTOFF INSTITUT LÜDENSCHEID


# Mysterious ways - Paths to Measurement Uncertainty

Dr. Andreas Balster, Deutsches Institut für Ringversuche

## Facts and figures

KUNSTSTOFF INSTITUT LÜDENSCHEID

- The Kunststoff-Institut Lüdenscheid supports you with
  - the selection
  - the development
  - optimization and implementation

  of products, tools and process sequences in the entire field of plastics technology

- The institute finances itself exclusively through services in the form of consulting, collaborative and development projects.
- The sponsoring company with over 350 active members from Europe represents the majority shareholder

GOLD Cluster Management Excellence PROVEN FOR CLUSTER EXCELLENCE

go-cluster EXZELLENT VERNETZT!

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## Companies/participations

**Sponsoring society**
**354 companies, 76%**

- machine manufacturers
- raw material manufacturers
- Tool and mould makers
- Peripherals, Automation
- All industries: automotive industry, electrical industry, lighting industry, medical technology, .....
- Universities, technical colleges, institutes, clusters, ...

are represented in the sponsoring company



## Interlaboratory comparisons at the Kunststoff-Institut


- 2002: First round robin tests of the Kunststoff-Institut (own requirement)
- 2006: First participant outside Germany
- 2010: First participants outside Europe
- 2016: More than 400 participating laboratories for the first time
- 2017: Registration of the trademark "Deutsches Institut für Ringversuche" (German Institute for Interlaboratory Tests)
- 2017: Partnership with QuoData GmbH
- 2018: Accreditation according to DIN EN ISO/IEC 17043

Deutsches Institut für Ringversuche

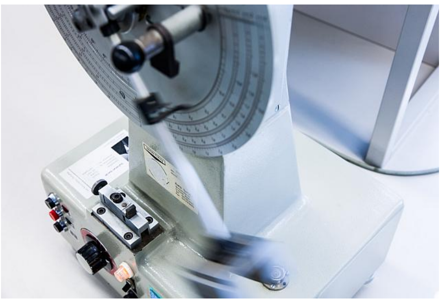
# WHAT ARE INTERLABORATORY COMPARISONS?



## Deutsches Institut für Ringversuche

**What are interlaboratory comparisons?**

- Interlaboratory tests represent a possibility of external quality assurance
- A group of laboratories is assigned a measurement, testing or analysis task
- Each participating laboratory will receive
  - the same (or the same) sample
  - same information
  - the same period for implementation

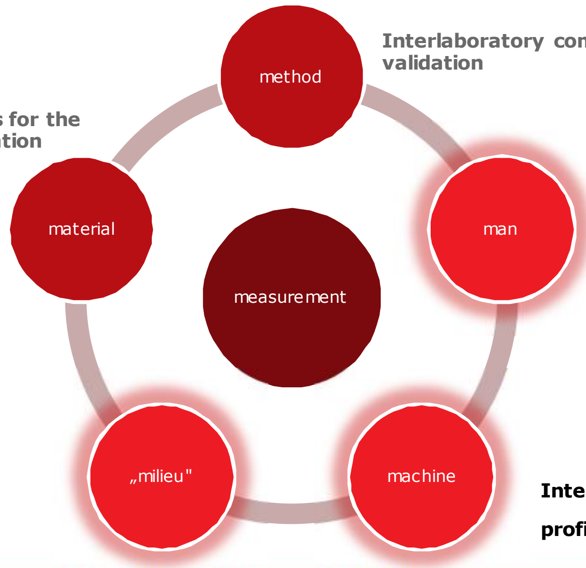


*Source: Kunststoff-Institut Lüdenscheid*

## Deutsches Institut für Ringversuche

**Interlaboratory tests for the material characterization**

**Interlaboratory comparisons for method validation**




**Interlaboratory comparisons for proficiency testing**



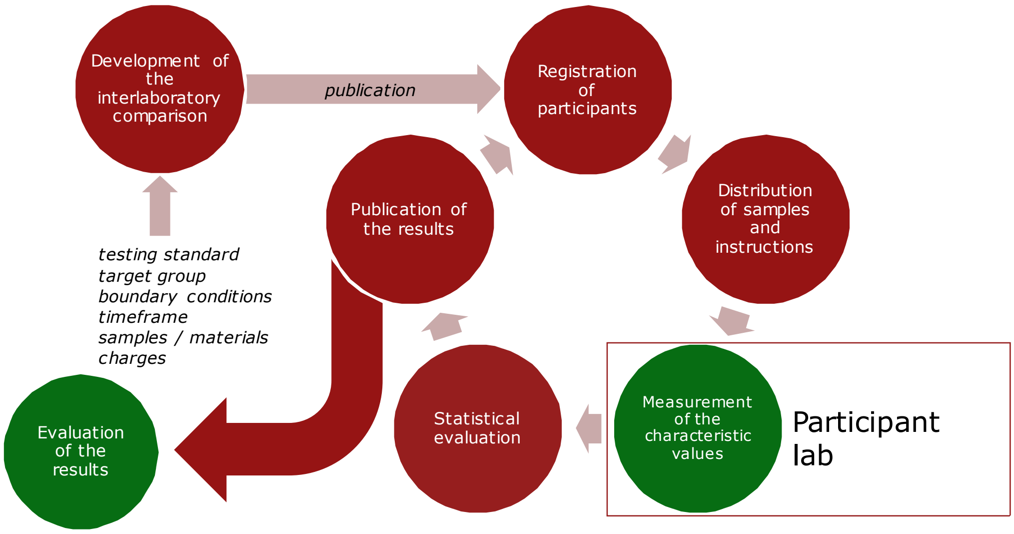
## Deutsches Institut für Ringversuche

An interlaboratory test is organised and managed from a central location. This one takes care of...

- the selection and technically correct description and organisation of the respective procedure
- the selection, preparation and dispatch of suitable sample material
- the registration and supervision of the participating laboratories, in particular with a view to ensuring that
  - neutrality and objectivity
  - the anonymity of the participants
- to ensure that data transmission is as error-free as possible
- to ensure a correct statistical evaluation of the results
- to ensure meaningful data preparation and -interpretation

*Source: Kunststoff-Institut Lüdenscheid*

## Deutsches Institut für Ringversuche






## Deutsches Institut für Ringversuche

- For DIN ISO/IEC 17025 accredited laboratories, regular participation in interlaboratory comparisons is mandatory.
- Results from round robin tests can be used (e.g. for customers) as proof of competence
- There is no better way to objectively verify your own laboratory performance
- Interlaboratory comparisons provide insight into the performance of processes and equipment
- Interlaboratory tests are an ideal opportunity to train staff

*Source: Kunststoff-Institut Lüdenscheid*

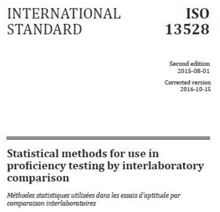
# PERFORMANCE EVALUATION



## Performance evaluation in proficiency tests

The "usual" performance evaluation is carried out by...

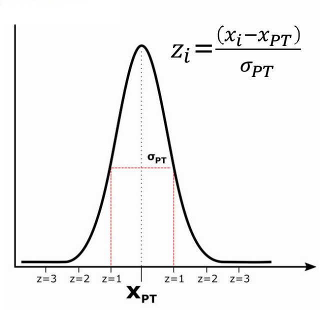
- The recording of *all* measurement results of the participants
- Checking this data for plausibility
  - Position of the measurement results
  - Distribution and dispersion of results
- Forming a (*robust*) mean $x_{PT}$ („Hampel estimator") -> consensus value
- Calculation of the (*robust*) comparative standard deviation $\sigma_{PT}$ ("Q method")
- Allocation of a score that uses the distance of the own laboratory result $x_i$ from the consensus value *in units of the standard deviation*: e.g. *z-Score*


INTERNATIONAL STANDARD

ISO 13528

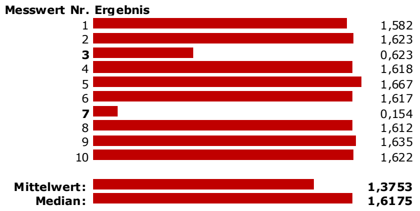
Statistical methods for use in proficiency testing by interlaboratory comparison

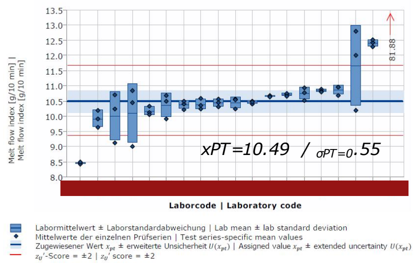
*Méthodes statistiques utilisées dans les essais d'aptitude par comparaison interlaboratoires*



## Performance evaluation in proficiency tests

- What does the term "*robust*" mean?
  - Mean value and standard deviation are *not* (or *only slightly)* affected by outliers
- What are *outliers*?
  - Results which, by objective standards, are so far removed from the consensus value that they are very likely to have been produced by measurement or documentation errors.
  - Typing errors, number shifts, misaligned equipment, unit, factor or scale errors...etc.




*Source: Kunststoff-Institut Lüdenscheid / QuoData*

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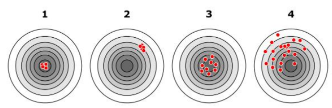
## Measurement uncertainty and interlaboratory comparisons

- Measurement uncertainty
  - As a rule, we do not know the true value of a quantifiable property.
  - We can only try to capture it as good (i.e. precise) as possible.
  - Whether it is *correct beyond that*, we must check with an external comparison (calibration).
  - The uncertainty of the measurement is fed from various sources
  - We don't have every one of these sources under control.




*Source: Kunststoff-Institut Lüdenscheid*

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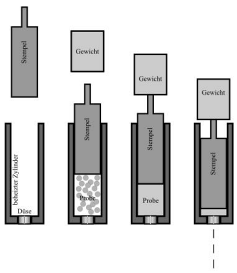
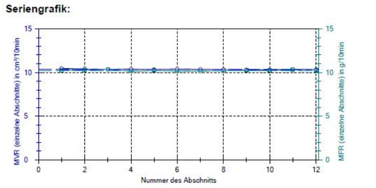
## CASE STUDY: MFR DETERMINATION



## Process description

- Melt Flow Rate or Melt Volume Rate determination according to ISO 1133
  - MFR: Operator weighs the strands
  - MVR: Volume determination of the strands by displacement transducers



**Prüfprotokoll**

Datum : 07.03.2019
Material : [redacted]
Art und Bezeichnung : [redacted]
Vorbehandlung : 3h bei 80°C im Vakuum
Prüfnorm : ISO 1133
Prüfgerät : Mflow Fließprüfgerät (WP-12)/ Waage ABS 80-4 (Wa-8)
Prüfer : [redacted]

Solltemperatur : 200,0 °C
Prüflast : 5,00 kg
Probeneinwaage : ca. 5 g
Messweg/-zeit Δs/Δt : 5 s

**Prüfergebnisse:**

| $MFR_n$ g/10min | $\overline{MFR}$ g/10min | s MFR g/10min | ν MFR | $MVR_n$ cm³/10min | $\overline{MVR}$ cm³/10min | s MVR cm³/10min | νMVR | $\rho_n$ g/cm³ | $m_n$ g |
|---|---|---|---|---|---|---|---|---|---|
| 10,26 | 10,23 | 0,06 | 0,60 | 10,46 | 10,37 | 0,04 | 0,42 | 0,981 | 0,0855 |
| 10,26 | | | | 10,40 | | | | 0,986 | 0,0855 |
| 10,38 | | | | 10,37 | | | | 1,001 | 0,0865 |
| 10,15 | | | | 10,40 | | | | 0,977 | 0,0846 |
| 10,21 | | | | 10,35 | | | | 0,986 | 0,0851 |
| 10,18 | | | | 10,38 | | | | 0,980 | 0,0848 |
| 10,18 | | | | 10,37 | | | | 0,981 | 0,0848 |
| 10,19 | | | | 10,34 | | | | 0,985 | 0,0849 |
| 10,21 | | | | 10,32 | | | | 0,989 | 0,0851 |
| 10,24 | | | | 10,28 | | | | 0,995 | 0,0853 |
| 10,28 | | | | 10,36 | | | | 0,993 | 0,0857 |
| 10,25 | | | | 10,35 | | | | 0,990 | 0,0854 |

**Seriengrafik:**



**Legende:**
$MFR/MVR_n$ = einzelne Abschnitte / $\overline{MFR}/\overline{MVR}$ = Mittelwerte / s MFR/MVR = Standardabweichung /
ν MFR/MVR = Varianzkoeffizient / $\rho_n$ = Schmelzdichte der einzelnen Abschnitte / $m_n$ = Gewicht der einzelnen Abschnitte

*Source: Kunststoff-Institut Lüdenscheid*

## Process influence factors

sample history
storage location
sampling tool
...

temperature
duration
homogeneity
Spec. surface
...

tidiness
quantity
...

Sampling → Drying → Filling — MFR MVR

*transfer*
duration
conditions

Machine - Material - Man - Environment - Method



## Process influence factors

temperature distribution
heat transfer
residual moisture
additivation
thermal load
...

tidiness
stamp weight
nozzle dimensions
Displacement transducer (MVR)
timing
Inclination of the device
melt viscosity
...

timing
knife condition
"Stickiness."
melt viscosity
cooling behaviour
...

preheating → extrusion → chopping — MFR MVR

Machine - Material - Man - Environment - Method

## process influences

duration
tidiness
hygroscopy
surrounding
...

tidiness
Inclination of the device
Precision/correctness
quantity
...

miscalculations
typing errors
...

sample transfer → weighing → documentation

MFR
~~MVR~~

Machine - Material - Man - Environment - Method



## Contributions to measurement uncertainty

past history
storage location
extraction tool
...

temperature
duration
homogeneity
Spec. surface
...

tidiness
quantity
...

temperature distribution
heat transfer
residual moisture
additivation
Therm. load
...

tidiness
stamp weight
nozzle dimensions
Displacement transducer (MVR)
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duration
tidiness
hygroscopy
surrounding
...

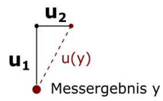
tidiness
Inclination of the device
Precision/correctness
quantity
...

miscalculations
typing errors
...

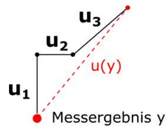
## Contributions to measurement uncertainty

- Each of the above factors $u_i$ (i>30) contributes to the overall uncertainty of measurement.
- Which ones can we control?
- Which ones are not under control?

- We may group factors together, make estimates, and possibly reasonably neglect certain factors.




$$u\,(y) = \sqrt{u_1^2 + u_2^2}$$




$$u\,(y) = \sqrt{\sum u_i^2(y)}$$

*Combined standard uncertainty*

*https://www.eurachem.org/index.php/publications/guides/quam*



## Contributions to measurement uncertainty

- Contributions of **type 1**
  - Laboratory specific
  - Not constant, i.e. potentially varying for each measurement
  - Contributions can be determined by measurements under repeat conditions -> $s_w$
- **Type 2** contributions
  - External cause (e.g. uncertainty of reference materials)
  - Constant, i.e. identical from measurement to measurement
  - Must be individually identified and *mathematically* included ("bottom-up approach")

$$s_w = \sqrt{\frac{1}{n-1}\sum_{i=1}^{n}(y_i - \bar{y})^2}$$

iterations

repeatability standard deviation

single value

Arithmetic Mean value from n Measurements

## Interlaboratory comparisons and uncertainty of measurement

- Interlaboratory comparisons solve the problem of Type 2 contributions
  - Each laboratory has "its" set of Type 2 contributions
  - If one evaluates the results of many laboratories (comparison conditions*, the previously constant but unknown Type 2 contributions become an accessible quantity ("top-down approach")

Repeat standard deviation (Type 1)

Combined Standard uncertainty of y

$$u(y) = \sqrt{\frac{s_w}{n_r} + \sum u_{B,i}^2(y)}$$

iterations the measurement

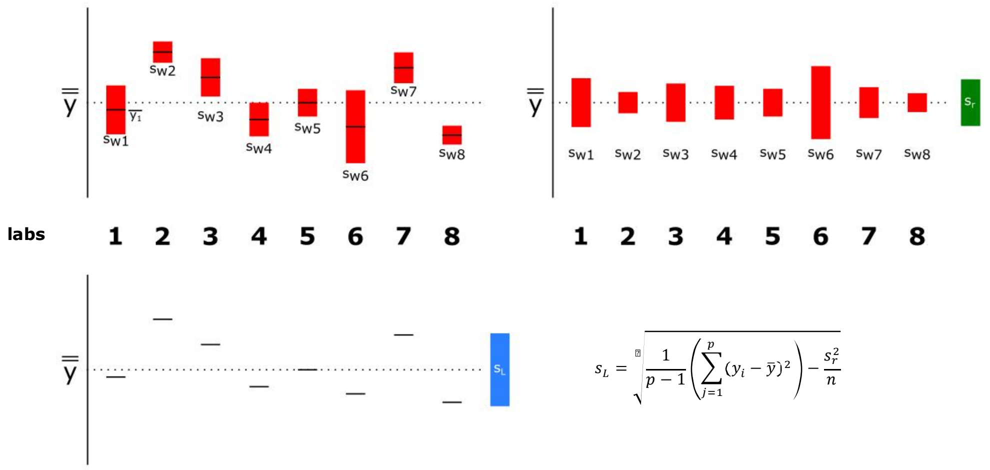
Type 2 contributions

*Repeat conditions: Identical person, laboratory, method, devices, short time period
: "Identical" sample, identical method - laboratory, person, devices, points in time different



## Contributions to measurement uncertainty

*Top down approach* according to ISO 21748:2010:
"Guidance for the use of repeatability, reproducibility and trueness estimates in measurement uncertainty estimation"




$$s_r = \sqrt{\frac{1}{p}\sum_{j=1}^{p} s_{w,j}^2}$$

$$s_L = \sqrt{\frac{1}{p-1}\left(\sum_{j=1}^{p}(y_i-\bar{y})^2\right) - \frac{s_r^2}{n}}$$

## Contributions to measurement uncertainty

| error components | Recommendation ISO 21748 | classification |
|---|---|---|
| Systematic differences between laboratories | "Laboratory standard deviation" according to ISO 5725-2 ($s_L$) | Identical for all laboratories that participated in the interlaboratory test |
| Laboratory specific scattering within the laboratory | Laboratory internal standard deviation ($s_w$) | Specific to the laboratory for which the MU is determined |
| Sum (quadratic) **= Uncertainty of measurement** | u(y) | |

- In this case, the laboratory standard deviation is the same for each participant.
- Experience now shows that the contribution of laboratory specific scattering is significantly smaller than $s_R$
- Good" laboratories in particular therefore often have to "live" with an overestimated measurement uncertainty.

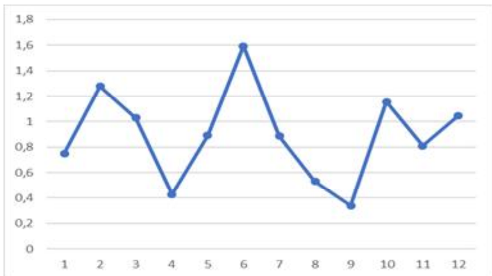


## Contributions to measurement uncertainty

- Internal laboratory standard deviations are also subject to large fluctuations from interlaboratory comparison to interlaboratory comparison: the differences between the laboratories are overestimated.

| Quelle | Materialbezeichnung | Mittelwert ($x_{PT}$) | $s_r$ | $s_R$ | $s_r$ Rel | $s_R$ Rel |
|---|---|---|---|---|---|---|
| RV 2008 | [redacted] | 6,24 | 0,35 | 0,36 | 5,61% | 5,77% |
| RV 2008 | | 9,96 | 0,24 | 0,56 | 2,41% | 5,62% |
| RV 2009 | | 1,85 | 0,04 | 0,06 | 1,95% | 3,08% |
| RV 2009 | | 10,31 | 0,19 | 0,32 | 1,84% | 3,10% |
| RV 2009 | | 20,67 | 0,58 | 0,91 | 2,81% | 4,40% |
| RV 2010 | | 1,87 | 0,03 | 0,04 | 1,45% | 1,89% |
| RV 2010 | | 9,76 | 0,20 | 0,37 | 2,05% | 3,79% |
| RV 2010 | | 20,13 | 0,26 | 0,56 | 1,29% | 2,78% |
| RV 2011 | | 1,89 | 0,03 | 0,07 | 1,70% | 3,86% |
| RV 2011 | | 8,61 | 0,23 | 0,47 | 2,67% | 5,46% |
| RV 2011 | | 18,56 | 0,33 | 0,30 | 1,78% | 1,62% |
| RV 2012 | | 1,89 | 0,03 | 0,06 | 1,59% | 3,17% |
| RV 2012 | | 10,66 | 0,17 | 0,50 | 1,59% | 4,69% |
| RV 2012 | | 18,58 | 0,21 | 0,75 | 1,13% | 4,04% |
| RV 2013 | | 1,87 | 0,02 | 0,07 | 1,07% | 3,74% |
| RV 2013 | | 10,47 | 0,16 | 0,62 | 1,53% | 5,92% |
| RV 2013 | | 20,60 | 0,21 | 1,11 | 1,02% | 5,39% |
| RV 2014 | | 1,86 | 0,02 | 0,06 | 1,08% | 3,23% |
| RV 2014 | | 10,46 | 0,10 | 0,38 | 0,96% | 3,63% |
| RV 2014 | | 24,02 | 0,48 | 2,75 | 2,00% | 11,45% |
| RV 2015 | | 1,87 | 0,03 | 0,07 | 1,60% | 3,74% |
| RV 2015 | | 10,45 | 0,17 | 0,77 | 1,63% | 7,37% |
| RV 2015 | | 8,10 | 0,27 | 1,28 | 3,33% | 15,80% |
| RV 2016 | | 1,87 | 0,02 | 0,07 | 1,07% | 3,74% |
| RV 2016 | | 10,32 | 0,11 | 0,63 | 1,07% | 6,10% |
| RV 2016 | | 13,65 | 0,26 | 1,00 | 1,90% | 7,33% |
| RV 2017 | | 1,84 | 0,04 | 0,09 | 2,17% | 4,89% |
| RV 2017 | | 10,49 | 0,23 | 0,55 | 2,19% | 5,24% |
| RV 2017 | | 13,43 | 0,19 | 1,12 | 1,41% | 8,34% |
| RV 2018 | | 10,60 | 0,19 | 0,48 | 1,79% | 4,53% |
| RV 2018 | | 1,89 | 0,03 | 0,08 | 1,59% | 4,23% |
| RV 2019 | | 12,00 | 0,22 | 1,09 | 1,83% | 9,08% |
| RV 2019 | | 10,26 | 0,23 | 0,34 | 2,24% | 3,31% |

*Source: Kunststoff-Institut Lüdenscheid*




*Source: QuoData*

*Laboratory internal standard deviation in 12 PT rounds, based on three measurement repetitions each*

*Material- and field-dependent differences in interlaboratory comparisons, 2008-2019*

Each laboratory is different

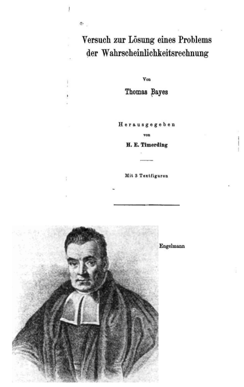
## OPTIMIZATION OF THE DETERMINATION OF THE MEASUREMENT UNCERTAINTY



## AI-based uncertainty of measurement calculation

- Use of the AI methodology based on Bayes' work to determine the
  - Bayesian-adapted laboratory standard deviation as well as a
  - Bayesian laboratory standard deviation

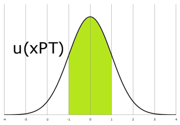
| error components | QuoData Concept | classification |
|---|---|---|
| Systematic differences between laboratories | *Bayes-adjusted* interlaboratory standard deviation | Identical for all laboratories that participated in the interlaboratory test |
| Laboratory specific scattering within the laboratory | *Bayesian* laboratory specific standard deviation | Specific to the laboratory, but taking into account the results of previous rounds and information on the results of other laboratories. |
| Sum (quadratic) **= Uncertainty of measurement** | | |

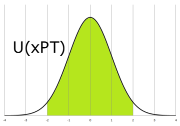


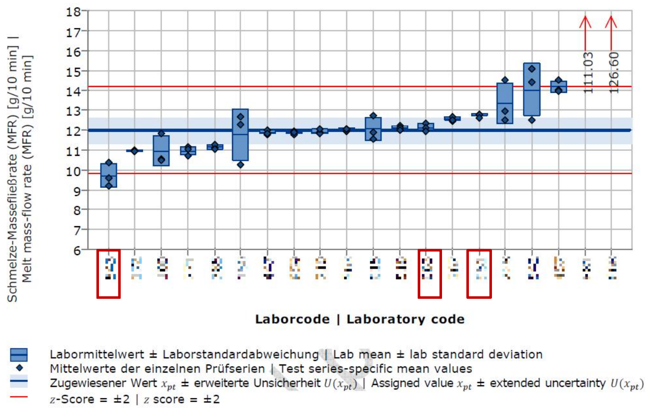

*Source: Public domain, digitisation: Bennett Kanuka, Mikhail Ryazanov*

## AI-based uncertainty of measurement calculation

- Example: EP Series 2019
- MVR/MFR determination on two granulate samples
- MFR to SAN: 20 records
- $x_{PT}$ = 12.00 g/10min
- Extended uncertainty $s_R$ = 1.09 g/10min
- $U(x_{PT})=2\cdot u(x_{PT}))=0.64$ g/10min




*Source: Kunststoff-Institut Lüdenscheid / QuoData*



## AI-based uncertainty of measurement calculation

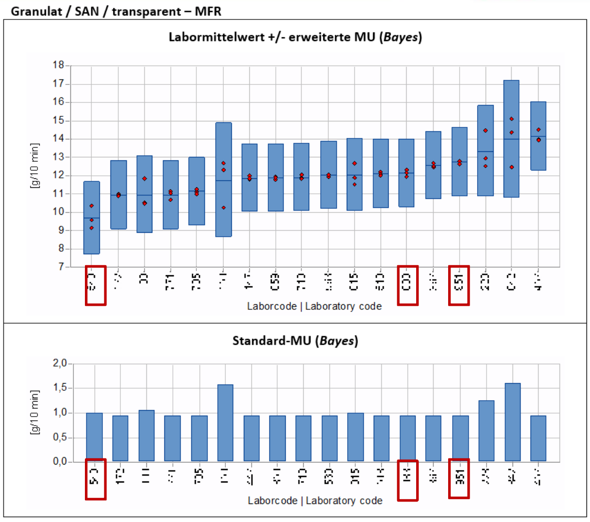
- $s_L$ becomes smaller in this example
- $s_W$ changes on the basis of additional information

| Labor | Laborspezifische SD ($s_L$) klassische Statistik | Bayes'sche Intra-Labor-SD | Bayes-Adjustierte Labor-SD ($s_L$) | u($x_i$) | U($x_i$) |
|---|---|---|---|---|---|
| [illegible] | 0,61 | 0,35 | 0,93 | 1,00 | 1,99 |
| [illegible] | 0,04 | 0,11 | 0,93 | 0,94 | 1,88 |
| [illegible] | 0,78 | 0,51 | 0,93 | 1,06 | 2,13 |
| [illegible] | 0,22 | 0,14 | 0,93 | 0,94 | 1,89 |
| [illegible] | 0,13 | 0,12 | 0,93 | 0,94 | 1,88 |
| [illegible] | 1,31 | 1,26 | 0,93 | 1,57 | 3,14 |
| [illegible] | 0,11 | 0,12 | 0,93 | 0,94 | 1,88 |
| [illegible] | 0,09 | 0,11 | 0,93 | 0,94 | 1,88 |
| [illegible] | 0,12 | 0,12 | 0,93 | 0,94 | 1,88 |
| [illegible] | 0,06 | 0,11 | 0,93 | 0,94 | 1,88 |
| [illegible] | 0,60 | 0,35 | 0,93 | 1,00 | 1,99 |
| [illegible] | 0,11 | 0,12 | 0,93 | 0,94 | 1,88 |
| [illegible] | 0,20 | 0,13 | 0,93 | 0,94 | 1,88 |
| [illegible] | 0,12 | 0,12 | 0,93 | 0,94 | 1,88 |
| [illegible] | 0,09 | 0,11 | 0,93 | 0,94 | 1,88 |
| [illegible] | 1,04 | 0,83 | 0,93 | 1,25 | 2,49 |
| [illegible] | 1,35 | 1,31 | 0,93 | 1,61 | 3,22 |
| [illegible] | 0,30 | 0,17 | 0,93 | 0,95 | 1,90 |

*Source: QuoData*

## AI-based uncertainty of measurement calculation

- A Bayesian laboratory internal standard deviation can also be determined on the basis of *own* test series using *other* materials and then be combined with the Bayesian-adjusted comparison standard deviation from the interlaboratory comparison.
- The calculation of this measurement uncertainty will be possible using a web tool.




Source: QuoData



## Thank you very much.

- Interlaboratory comparisons are a versatile, effective means of quality assurance.
- We offer more than 150 tests in the plastics sector
- Together with our partner we offer a real added value in the analysis of your laboratory data!

**Deutsches Institut für Ringversuche**

**www.dir-kimw.de**
**www.quodata.de**

quo data
*QUALITY & STATISTICS!

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