Facts and figures

- 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
Companies/participations

- 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
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
- Measurement
- Method
- Man
- Machine
- „milieu"

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
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.
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
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.

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.
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
Process influence factors

- Sample history
- Storage location
- Sampling tool
- Temperature
- Duration
- Homogeneity
- Specific surface

Sampling → Drying → Filling

MFR MVR

Sampling conditions:
- Transfer duration
- Conditions

Machine - Material - Man - Environment - Method

Temperature distribution
Heat transfer
Residual moisture
Additivation
Thermal load

Preheating → Extrusion → Chopping

MFR MVR

Process influence factors

- Tidiness
- Stamp weight
- Nozzle dimensions
- Displacement transducer (MVR)
- Timing
- Inclination of the device
- Melt viscosity
- Knife condition
- "Stickiness"
- Melt viscosity
- Cooling behaviour

Temperature distribution
Heat transfer
Residual moisture
Additivation
**process influences**

- duration
- tidiness
- hygroscopy
- surrounding
- ...

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

- miscalculations
- typing errors
- ...

**Contributions to measurement uncertainty**

- past history
- storage location
- extraction tool
- ...

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

- temperature
- duration
- homogeneity
- Spec. surface
- ...

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

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

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

- miscalculations
- typing errors
- ...

**Machine - Material - Man - Environment - Method**

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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.

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

Contributions of type 1

► Laboratory specific

► Not constant, i.e. potentially varying for each measurement

► Contributions can be determined by measurements under repeat conditions $\rightarrow s_w$

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")

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

Combined standard uncertainty

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

\[
u(y) = \sqrt{\frac{S_w}{n_r} + \sum u_{B,i}^2(y)}
\]

*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"

\[
s_i = \frac{1}{p-1} \left( \sum_{j=1}^{p} (y_j - \bar{y})^2 \right)^{-\frac{1}{2}}
\]
Contributions to measurement uncertainty

<table>
<thead>
<tr>
<th>error components</th>
<th>Recommendation ISO 21748</th>
<th>classification</th>
</tr>
</thead>
<tbody>
<tr>
<td>Systematic differences between laboratories</td>
<td>&quot;Laboratory standard deviation&quot; according to ISO 5725-2 (sL)</td>
<td>Identical for all laboratories that participated in the interlaboratory test</td>
</tr>
<tr>
<td>Laboratory specific scattering within the laboratory</td>
<td>Laboratory internal standard deviation (sAn)</td>
<td>Specific to the laboratory for which the MU is determined</td>
</tr>
<tr>
<td>Sum (quadratic) = Uncertainty of measurement u(y)</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

- 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 sL.
- Good" laboratories in particular therefore often have to "live" with an overestimated 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.

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

Source: QuoData
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

<table>
<thead>
<tr>
<th>error components</th>
<th>QuoData Concept</th>
<th>classification</th>
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<tbody>
<tr>
<td>Systematic differences between labs</td>
<td>Bayes-adjusted interlaboratory standard deviation</td>
<td>Identical for all laboratories that participated in the interlaboratory test</td>
</tr>
<tr>
<td>Laboratory specific scattering within the lab</td>
<td>Bayesian laboratory specific standard deviation</td>
<td>Specific to the laboratory, but taking into account the results of previous rounds and information on the results of other laboratories.</td>
</tr>
<tr>
<td>Sum (quadratic) = uncertainty of measurement</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

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
- \( x_{PT} = 12.00 \text{ g/10min} \)
- Extended uncertainty \( \gamma = 1.09 \text{ g/10min} \)
- \( U(x_{PT}) = 2\cdot u(x_{PT}) = 0.64 \text{ g/10min} \)

**Source:** Kunststoff-Institut Lüdenscheid / QuoData

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**AI-based uncertainty of measurement calculation**

- \( s_L \) becomes smaller in this example
- \( s_w \) changes on the basis of additional information

<table>
<thead>
<tr>
<th>Labor</th>
<th>Labor- spezifische SD (( \bar{q} ))</th>
<th>Bayesian Intra Labor SD</th>
<th>Bayesian- Adjustierte Labor-SD (( u_s ))</th>
<th>( u(x_{PT}) )</th>
<th>( U(x_{PT}) )</th>
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<tr>
<td>xpt</td>
<td>0.01</td>
<td>0.05</td>
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<td>( \sigma_x )</td>
<td>0.04</td>
<td>0.11</td>
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<td>0.12</td>
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<td>( \gamma_x )</td>
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<td>0.53</td>
<td>0.95</td>
<td>1.70</td>
</tr>
</tbody>
</table>

**Source:** QuoData

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**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.

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**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!