

# Statistical Aspects of Measuring Uncertainties in Laboratories and Interlaboratory Tests

What are telling us the different characteristic values of the statistics of measurements in our laboratories and in interlaboratory tests (Round Robins)

*„There a three kinds of lies:  
lies, damned lies and statistics“*

Benjamin Disraeli (1804 – 1881)

Results submitted by participants													
Lab <sub>i</sub> : individual results $x_{ij}$ + number of the test repetitions made by each lab ( $n_i$ ) + within laboratory means ( $\bar{x}_i$ ) and standard deviations ( $s_i$ ) + results of tests for outliers													
								Number of reporting laboratories $p$ *	6				
								Number of reported test results $\sum n_i$	12				
Lab Code No.	Test results in %						Statistical evaluation of the submitted test results $x_{ij}$			Outliers			
	Test replication No. (k)						$n_i$	$\bar{x}_i$	$s_i$	Cochran	Grubbs	t > 2	
1	2	3	4	5	6								
404	36,0	36,3					2	36,04	0,0183				
42	49,0	49,8					2	49,41	0,0283		**	X	
452	50,8	51,1					2	50,93	0,2006				
738	52,5	52,4					2	52,49	0,0707				
547	54,1	54,3					2	54,19	0,1373				
347	54,3	54,0					2	54,58	0,4283				
865	no results reported												X

\*\* ... statistical outlier (99%)      \* ... straggler (95%)  
 X ...  $z > 2$

Results of robust statistics		Convergence assumed at iteration number: 20	
Robust average: $x^* = 51,3$		assigned value for the proficiency assessment	
Robust standard deviation for the proficiency assessment: $s^* = 3,75$		OK	
Number of repeat measurements necessary due to $s_i/s^*$ -ratio: $n^* = 1$		see page 4 for the meaning of NOT OK	
Standard uncertainty of the assigned value: $u_x = 1,91115$		NOT OK	

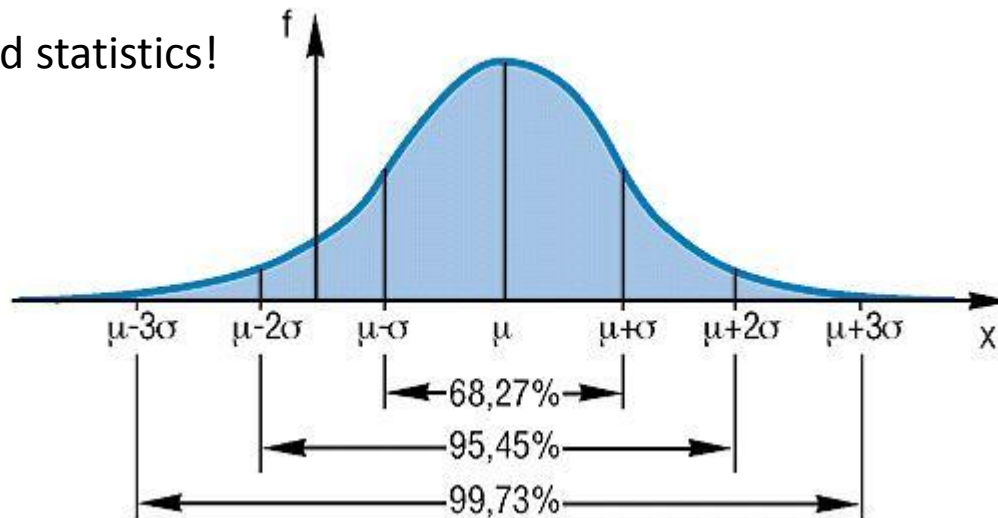
Additional check of the test method accuracy			
Do the input data come from a normal distribution ? (The results listed below shall be considered as really justified only if the input data come from a normal distribution)			YES
General mean $\sum n_i \bar{x}_{ij} / \sum n_i$	$\bar{m}$	52,4	%
Repeatability variance	$s_r^2$	0,0519600	%
Repeatability standard deviation	$s_r$	0,22795	%
Repeatability coefficient of variation	CV %	0,435	%
Between-laboratory variance	$s_L^2$	4,1437500	%
Between-laboratory standard deviation	$s_L$	2,03562	%
Between-laboratory coefficient of variation	CV %	3,884	%
Reproducibility variance $s_R^2$	$s_r^2 + s_L^2$	4,1957100	%
Reproducibility standard deviation	$s_R$	2,0483	%
Reproducibility coefficient of variation	CV %	3,909	%
Repeatability limit	$r$	0,6	%
Relative repeatability limit	$r_{rel}$	1,22	%
Reproducibility limit	$R$	5,7	%
Relative reproducibility limit	$R_{rel}$	10,94	%
Number of participants included in the accuracy evaluation	$p$	5	
Number of tests included in the accuracy evaluation	$\sum n_i$	10	

# What's our goal when measuring a property?

- to find the **true value** of what we have to measure !!!  
(that's what your customer expects from you because he pays for it)

Unfortunately the **true value** will only be obtained by the average of an **infinite** numbers of single results performed by an **infinite** number of laboratories.

That's why we need statistics!



## Gaussian Bell Curve or Normal Distribution

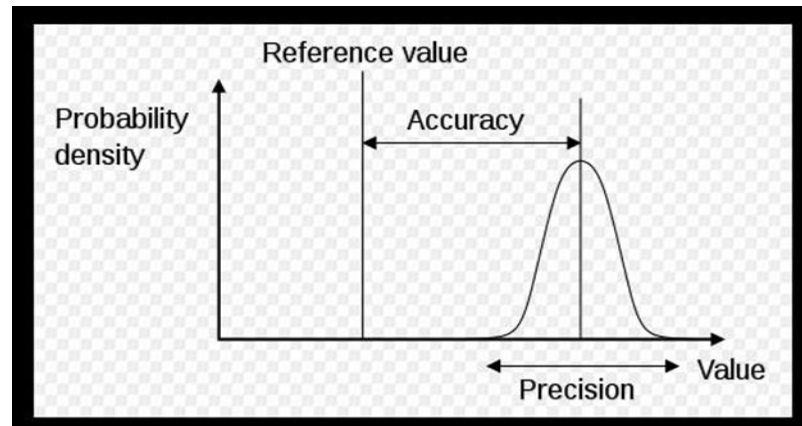
Laboratory statistics and interlaboratory statistics (Round Robins) give us the following idea about what we are measuring:

(1) How close is my measured value to the **true value** within certain (statistical) borders when measuring one, two or three times as it is usual in our daily laboratory routine!

(2) When does a property of a measuring object do not meet the requirements, means failing the specification!!

So what must an **accurate** measurement procedure **always** fulfill?

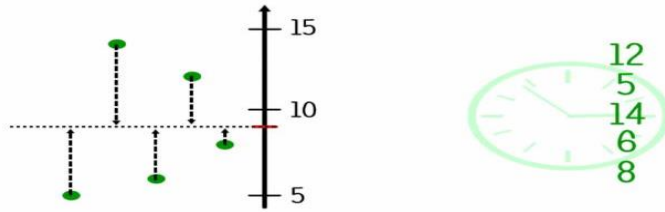
**accuracy** = trueness (systematical errors) **and** precision (random errors)



Dr. Dirk Stegemann: Statistical Aspects of Measuring Uncertainties in Laboratories and Interlaboratory Tests  
- Technical Meeting ISSS - Florence 27.10.16 - 28.10.16

## Some necessary definitions to characterize measured data

(1) The **Standard Deviation** (SD)  $s$  ( $\sigma$  = true value in Normal Distribution)



$$s = \sqrt{\frac{\sum_{i=1}^n (x_i - \bar{x})^2}{n - 1}}$$

$n-1$  = estimates the SD of the universe

$n$  = estimates the SD of the test

(!) The **SD** is a measure of the distribution of measured values

(2) The **Variance** (VAR)

$$s^2 = \frac{1}{(n - 1)} \sum_{i=1}^n (x_i - \bar{x})^2$$

(!) The **VAR** is a measure of how the measured values are distributed around the mean value

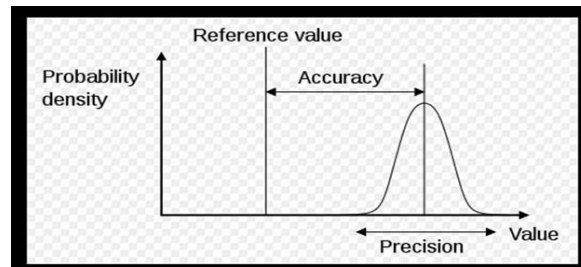
We always have to deal with two kinds of statistics:

(a) the laboratory statistics in your own lab

(b) the interlaboratory statistics between different laboratories (Round Robins)

Please keep in mind that both statistics about your measurement uncertainties are based on same principles!

trueness (systematical errors) and precision (random errors)



(a) In your own lab: GUM =  $u = \sqrt{u_{R/W}^2 + u_{bias}^2}$

(b) In Interlaboratory Tests (Round Robins) =  $s_R = \sqrt{s_I^2 + s_L^2}$

## Let us now talk about the interpretation of interlaboratory tests (Round Robins)

key figure	calculation	meaning
$u_x$	$u_{C_{ref},i} = 1,25 \cdot \frac{s_{R,i}}{\sqrt{n_{p,i}}}$	Uncertainty of the assigned value $x^*$ $1,25 \times 3,75/\sqrt{6} = 1,911 \rightarrow 0,3 \times s^* = 1,12$
$x^*$	ISO 13528 DIN ISO 5725-5	<b>robust mean</b> regarding all individual means (assigned value!)
$s^*$	ISO 13528 DIN ISO 5725-5	robust <b>mean standard deviation</b> regarding all individual SD's (target value for analytical performance called <b>z-score</b> )
$s_r$	$s_r^2 = \frac{\sum s_i^2}{p}$	SD of all individual SD's (repeatability)
$s_L$	$s_L^2 = \frac{1}{n} \left[ \frac{1}{p-1} \sum_{i=1}^p n_i (\bar{x}_i - \bar{\bar{x}})^2 - s_r^2 \right]$	SD of all differences from the individual mean to the assigned value $x^*$
$s_R$	$s_R = \sqrt{s_r^2 + s_L^2}$	the overall SD taking into account the precision and trueness

# Why are these characteristic values $s_r$ and $s_R$ so important?

$r = 2,8 \cdot s_r$  (repeatability limit 95 % confidence interval)

$R = 2,8 \cdot s_R$  (reproducibility limit 95 % confidence interval)

$$CD = 2,8 \cdot s_r \cdot \sqrt{\frac{1}{2n_1} + \frac{1}{2n_2}} \quad \text{How do your employees work?}$$

$$CD = \sqrt{(2,8 s_R)^2 - (2,8 s_r)^2 \left(1 - \frac{1}{2n_1} - \frac{1}{2n_2}\right)} \quad \text{Do two labs have the same performance?}$$

$$CD = \frac{1}{\sqrt{2}} \cdot \sqrt{(2,8 s_R)^2 - (2,8 s_r)^2 \left(\frac{n-1}{n}\right)} \quad y' - m_0 : \text{Has the test object failed in your lab?}$$

$$CD = \frac{1}{\sqrt{2p}} \cdot \sqrt{(2,8 s_R)^2 - (2,8 s_r)^2 \left(1 - \frac{1}{p} \sum_{i=1}^p \frac{1}{n_i}\right)} \quad y'' - m_0 : \text{Has the test object failed in different labs?}$$

2 employees making  $n_i$  determinations / 2 labs making  $n_i$  determinations

$p$  labs making  $n_i$  determinations to confirm a reference value  $m_0$



## Now, what to do when the critical differences (CD) are exceeded??

Don't worry be happy and ask the **DIN ISO 5725** or the **EN ISO 4259 Annex 1**, because they will tell you what to do to avoid a lawsuit with your customer or to fire one of your employees.

Thank you for having so much patience with statistics and the guy who was talking about it.

For further Informations:

Darell Huff, Irving Geis, ***How to Lie with Statistics***, W.W. Norton & Company Ltd, New York, 1954