



Food and Agriculture  
Organization of the  
United Nations

# Proficiency Tests and Interlaboratory Comparisons

Mrs. Nopmanee Suvannang,  
GLOSOLAN Chair

[nopmanee\\_su@hotmail.com](mailto:nopmanee_su@hotmail.com)

[suv.nopp@gmail.com](mailto:suv.nopp@gmail.com)

2<sup>nd</sup> meeting of the International Network on Fertilizer Analysis (INFA)

Virtual meeting | 29-30 June 2021 - 02:00 PM CEST



# Rationale

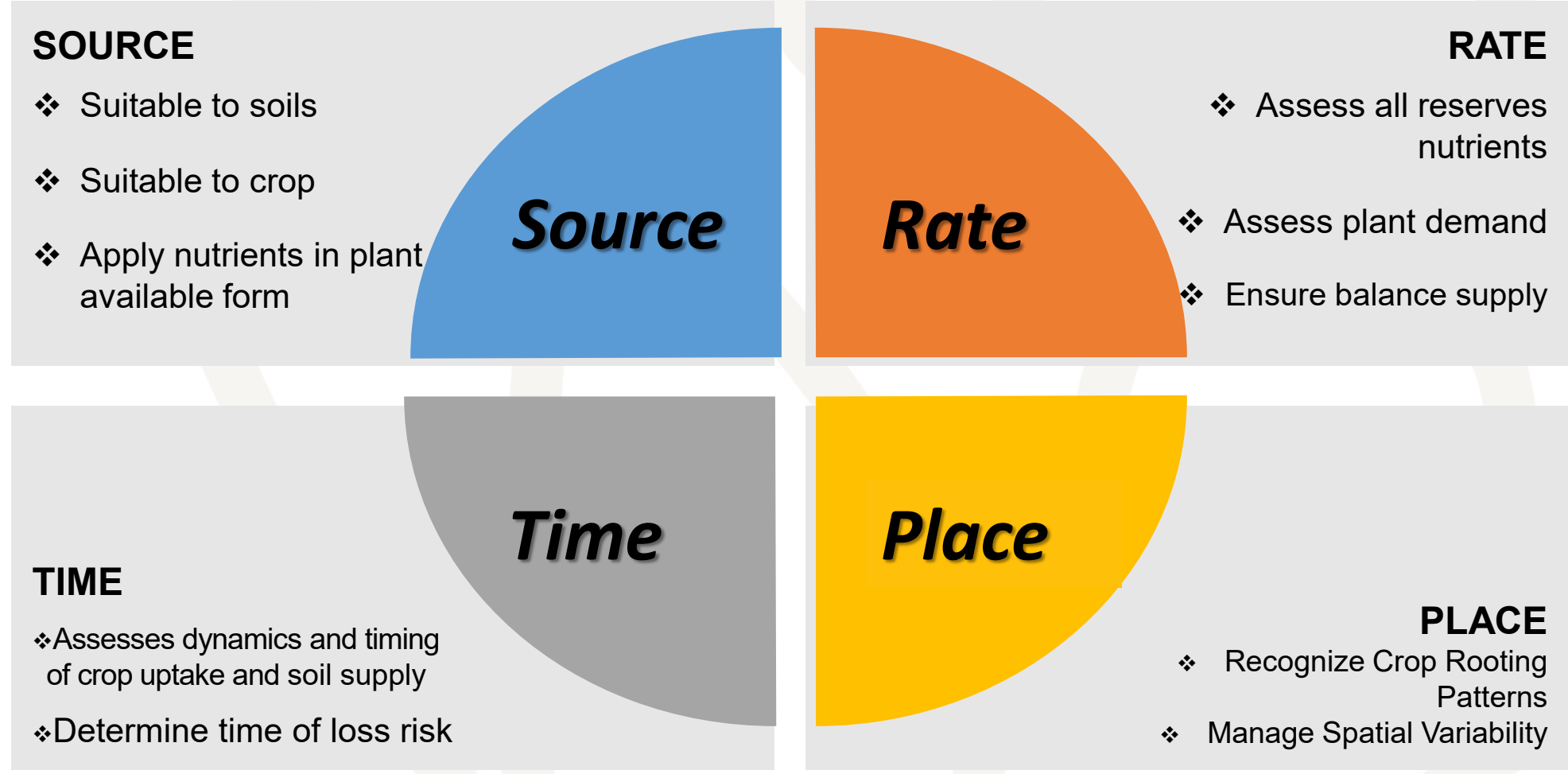
- The world's population increasing
- Increased agricultural production will be required to support the growth of the population, but .. reality it is decreasing due to soils degradation, miss management etc.
- Fertilizers are regarded as responsible for the pollution of water supplies, harming the soil structure, leading to intensive agriculture which disfigure the countryside
- to ensure future food supplies by promoting their correct use is the must consideration at this stage
- The consequences of injudicious fertilizer use are significant for the environment and have resulted in several jurisdictions of legislative tools to manage grower practice.

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# 4 R Nutrient management

*reducing negative environmental impacts due to improper fertilizer use?*



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## **TO BE ABLE TO :**

Facilitate exchange of scientific data, methodologies and research applications among various stakeholders to support national/country-level research

## **YOU NEED LABORATORY:**

To provide comparable and reliable fertilizer testing results

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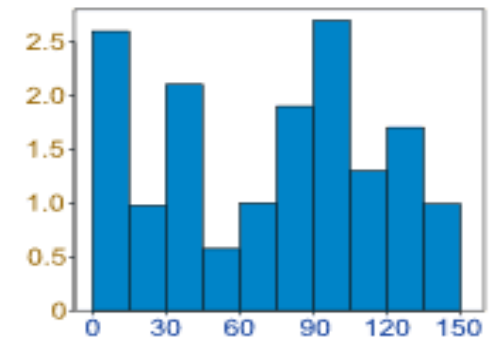
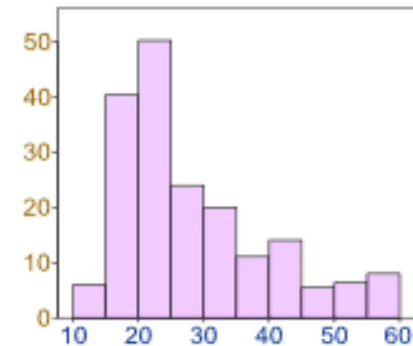
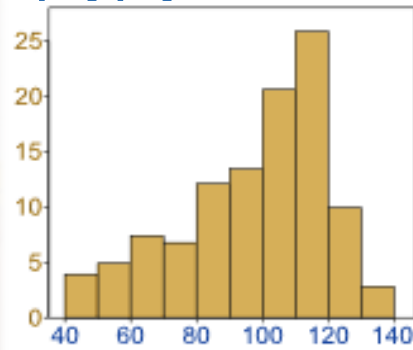


When sending a same fertilizer sample to any fertilizer testing laboratory...



When sending a same fertilizer sample to any fertilizer testing laboratory...

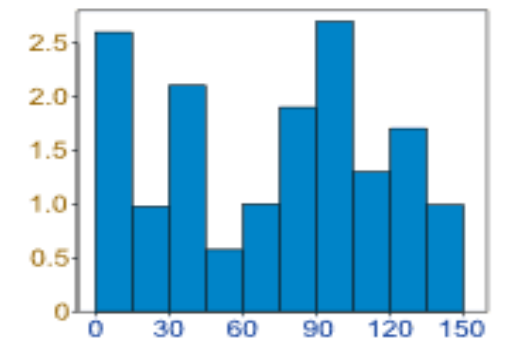
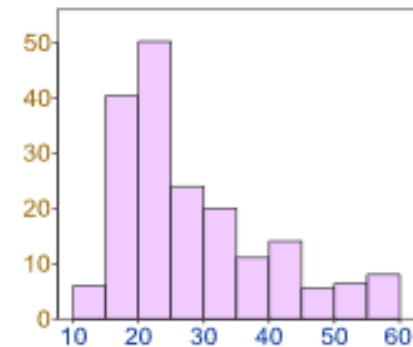
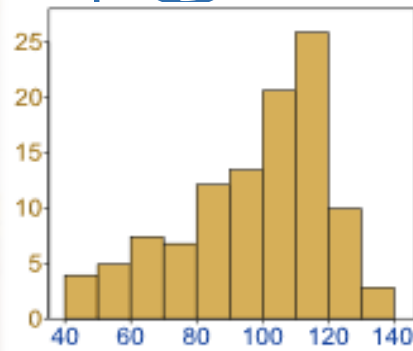
your expectation is...  
**...to get the same results! (+/- uncertainty)**



When sending a same fertilizer sample to any fertilizer testing laboratory...

...and thus...

to obtain the **same** or provide **similar fertilizer quality and recommendations, etc.**



# Laboratory with consistency comparable and reliable data

- ❑ Testing method : harmonized and validate
- ❑ Laboratory staffs : well trained and knowledgeable
- ❑ Quality control : internal and external quality control implemented
  - ❖ IQC : use RM/QC samples/quality control chart
  - ❖ EQC : Interlaboratory Comparison(ILC)  
Proficiency test (PT)



## ❑ **Interlaboratory Comparison (ILC)**

looks at measurement results by two or more laboratories on the same or similar items

## ❑ **A proficiency test (PT)**

determination of laboratory testing performance by means of interlaboratory comparison (ILC)

PT having a coordinating body and a formal report which clearly outlines the Z score

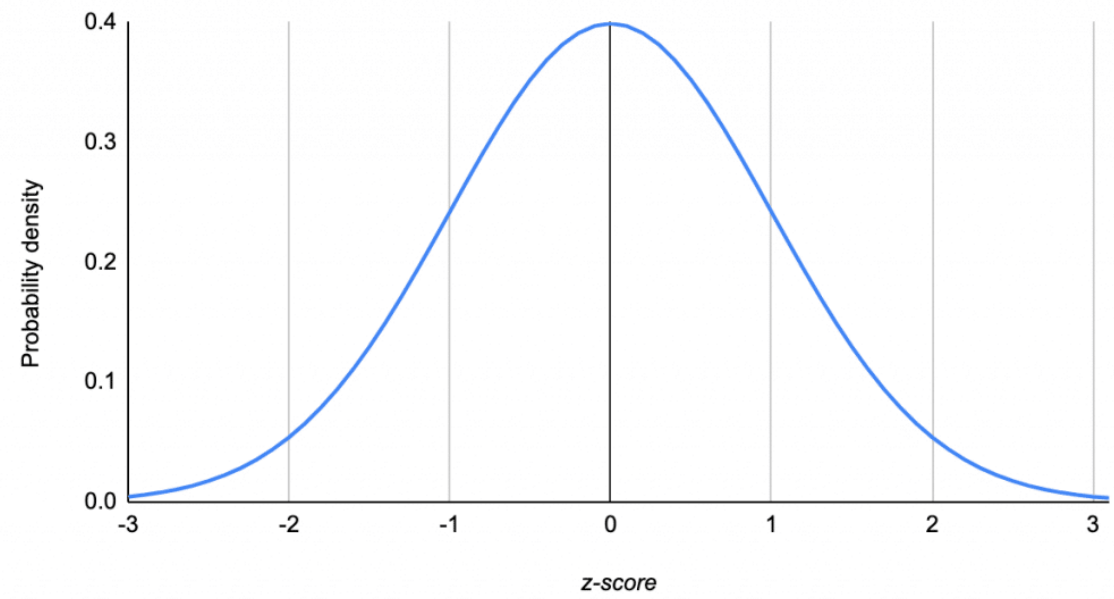


# What is Proficiency

- ❖ **Testing** the laboratory performance by inter laboratory comparison using the consensus value calculate from
- ❖ PT is the external quality control activity, it shall perform to evidence on laboratory performance and is one of the
- ❖ A program designed as a statistical quality control tool enabling participating laboratories to assess their performance



Standard normal distribution



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# Benefit of Laboratory joining Proficiency Testing

01

Support the technical validity of laboratory results

Protect your interest in trade transactions

02

03

Establishment of comparability of test or measurement methods

Identification of problems in laboratories and initiation of actions for improvement

04

05

Verify analyst proficiency and Use as laboratory quality control materials

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# *PT and Accreditation*

- ❖ To achieve initial accreditation a laboratory must be able to demonstrate successful participation in at least one PT event.
- ❖ - Corrective action must be taken for any outlying results.
- ❖ Unacceptable PT results may result in an adverse accreditation action such as suspension of that test from the scope until the lab can demonstrate acceptable performance

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29 January 2015



# Limitation of Proficiency Testing

01

It has to be carried out within the context of a complete system in each laboratory

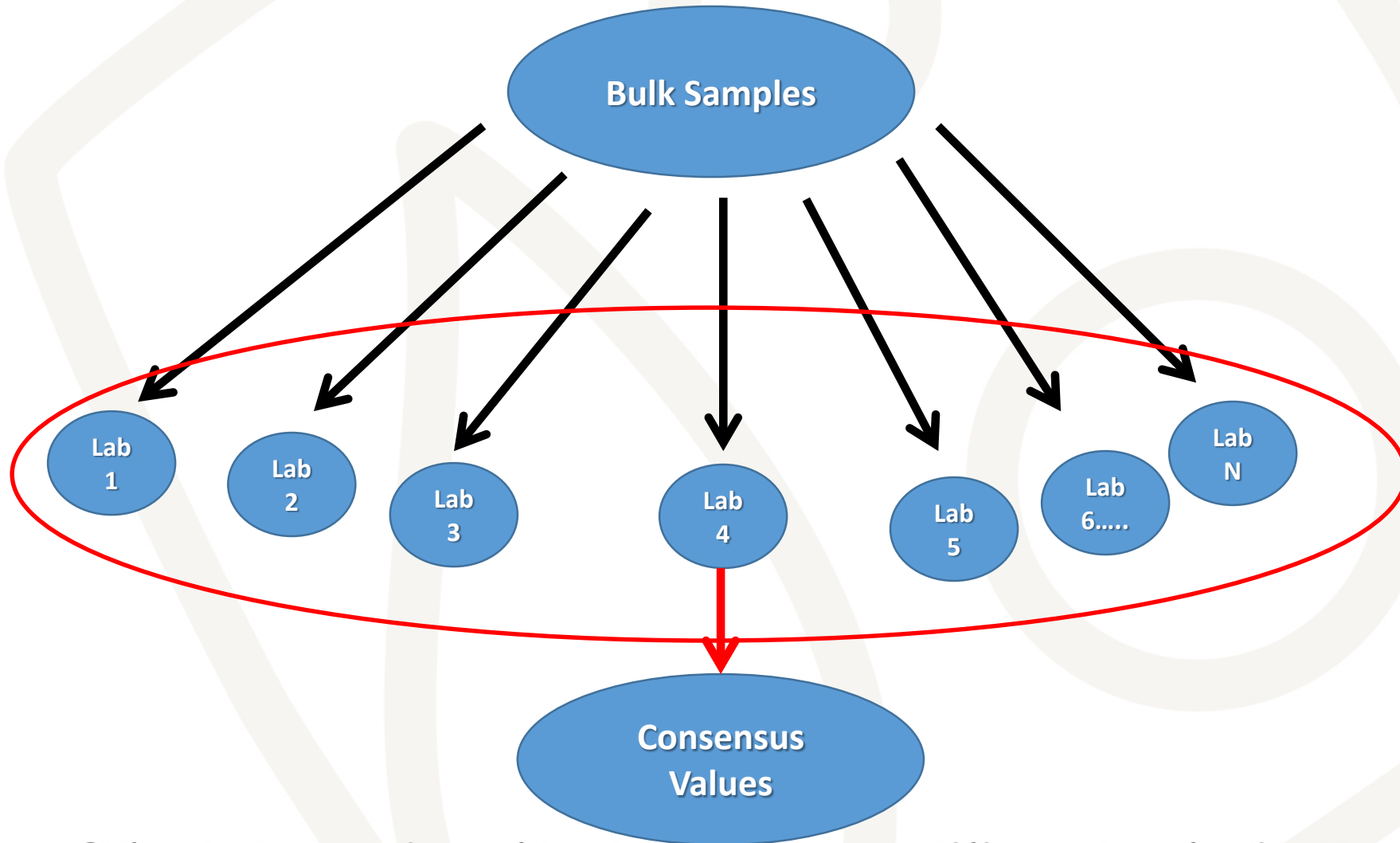
It provides a participating laboratory only with an indication of problems if they are present. But it does

02

03

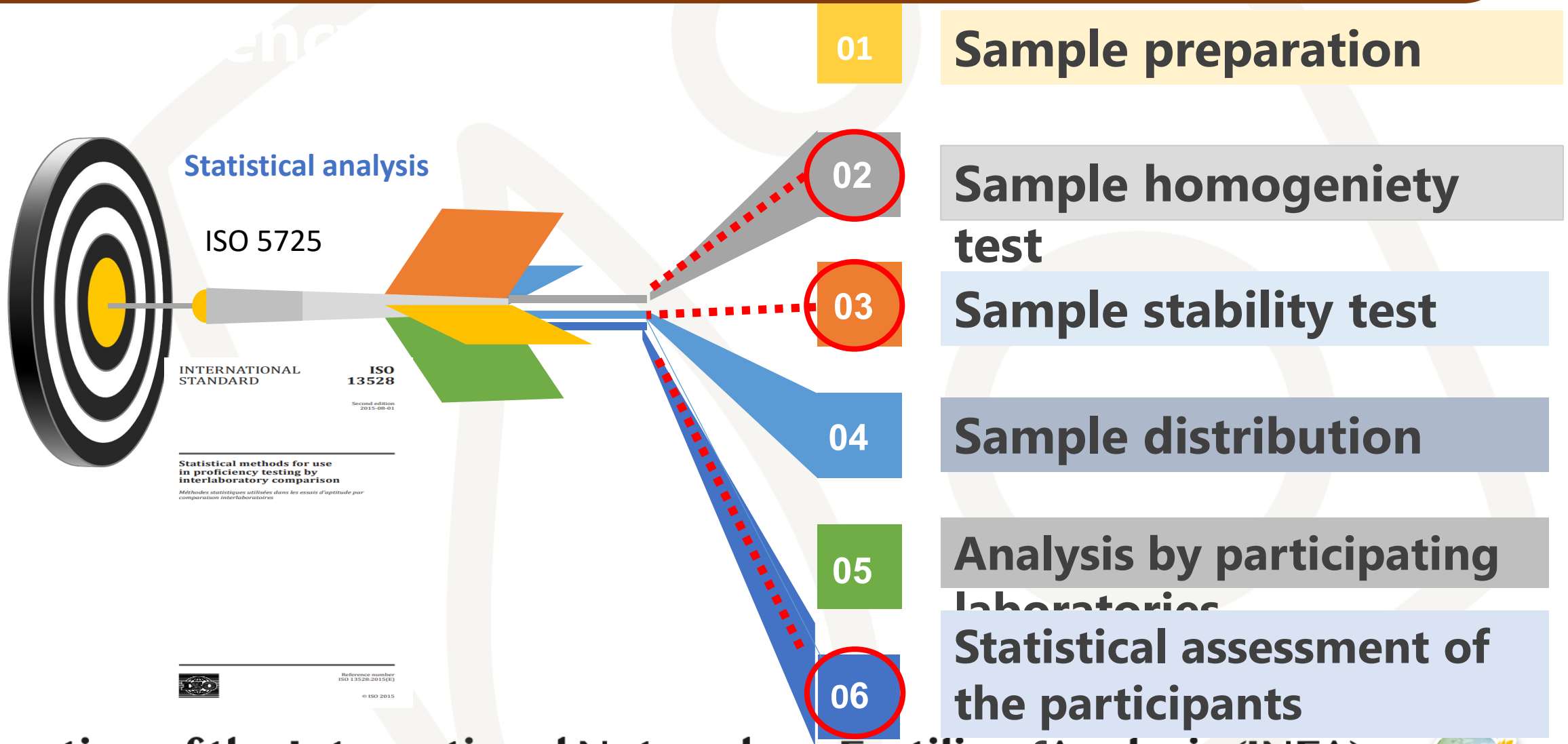
Success in a PT for one analyze does not mean that a laboratory is equally competent in other

# Typical Proficiency Test



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# ☑ Important steps for the



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# Sample Preparation



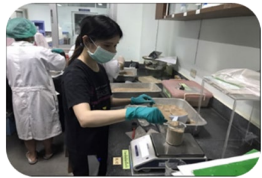
Dry soil sample



Grind the soil sample



Sieve through 2 mm and then through 0.5 mm



Weighting



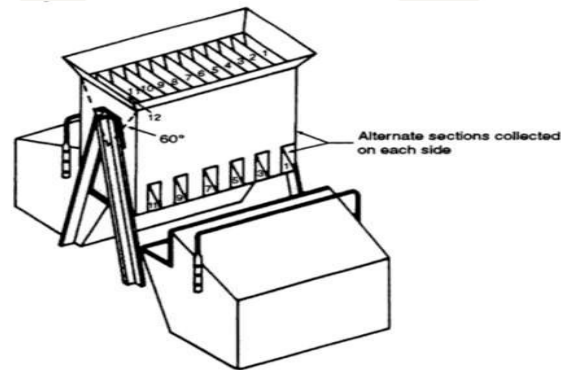
Packing



Sealing



Label



• Riffle box



Soil mixing

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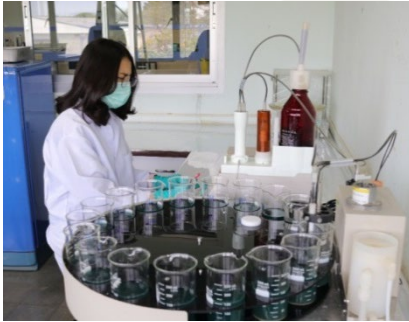
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"Standardization of Soil Testing Methods and Good Laboratory





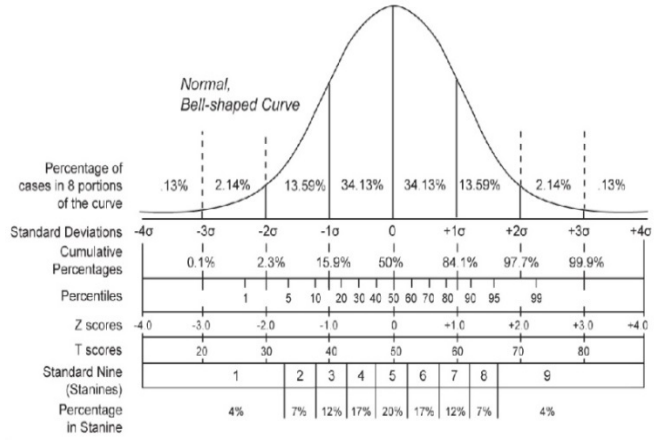
# Homogeneity and stability test



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# Statistical design and the evaluation of the



Homogeneity test

01

Stability test

02

- Assigned value;  $X_{pt}$
- Standard uncertainty of the assigned value  $u_x$

03

Standard deviation for proficiency assessment;

04

$\hat{\sigma}/\sigma_{pt}$   
Calculate Z-Score

05

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# Homogeneity Test



Homogeneity test  
within sample variation

Homogeneity test  
Between Sample Variations

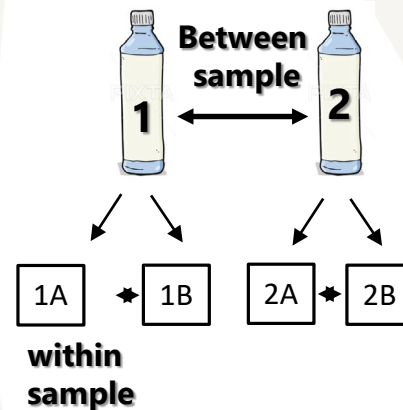
Duplicate analysis:  
Cochran's maximum range test

Calculate the ratio:  $\frac{D_{\max}^2}{\sum D_i^2}$

where

$D_{\max}$  = the maximum difference of the duplicates

$D_i$  = difference of each pair of duplicates



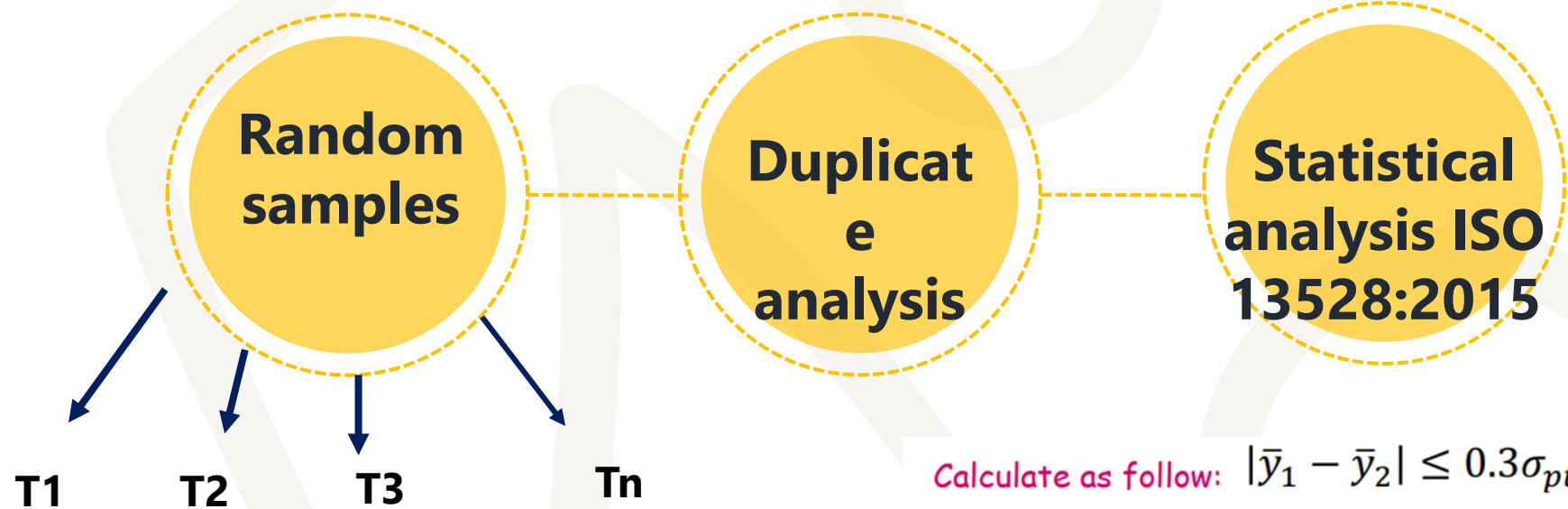
- ❖ One way ANOVA
- ❖ ISO 13528:2015

$$s_s \leq 0.3 \sigma_{(pt)}$$

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# Stability Test



Calculate as follow:  $|\bar{y}_1 - \bar{y}_2| \leq 0.3\sigma_{pt}$

$\bar{y}_1$  = Mean value from homogeneity of test material

$\bar{y}_2$  = Mean value from stability each storage time

$\sigma_{pt}$  Robust Standard Deviation for Assessment

**If not adequately stable  
: use expand criteria**

$$|\bar{y}_1 - \bar{y}_2| \leq 0,3\sigma_{pt} + 2\sqrt{u^2(\bar{y}_1) + u^2(\bar{y}_2)}$$

# Evaluating the performance of laboratory and statistical treatment of data

- ❖ The **assigned value** to each measurement is represented by the **consensus mean** calculated either according to algorithm "A" ISO 13528:2015 or other statistical tools that fit to the data (arithmetic mean etc)
- ❖ The uncertainty measurement of the assigned value is calculated based on standard deviation by the formula:

$$u_x = 1.25 \times \frac{s^*}{\sqrt{p}}$$

Reference: ISO 13528:2015

$u_x$  = Uncertainty of assign value

$s^*$  = Robust standard deviation

$p$  = number of results

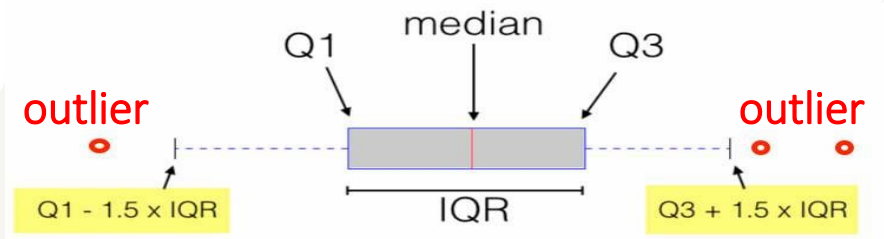
# Determination of Assigned value; $X_{pt}$

Assigned value, is a mean or median value that serves as an agreed-upon reference for comparison; normally derived from or based upon

- ❖ the CRM reference value
- ❖ a single laboratory using a ref method on PT item and CRM, RM
- ❖ expert laboratories
- ❖ **participant results**

exclude outliers using algorithm A

calculate consensus mean from Robust mean ( $x^*$ ) according to Algorithm A (ISO 13528:2015)



exclude outliers using IQR or others to identify statistical etc)

Free open-source alternative to Matlab, SAS, Excel, and SPSS

- Statistical software
- Language
- Environment



<http://www.r-project.org>

if the data is normal distribution ==> the mean can be used !  
no need of 'robust' (= using median) statistics

# Determination of Z-Score or Z'-Score

- ❖ calculated Z-Score when the measurement uncertainty is negligible or  $u_{x(pt)} \leq 0.3\sigma_{pt}$
- ❖ calculated Z'-Score when the measurement uncertainty is not negligible or  $u_{x(pt)} > 0.3\sigma_{pt}$

$$Z - Score = \frac{x_i - x_{pt}}{\sigma_{pt}}$$

$$Z' = \frac{(x_i - x_{pt})}{\sqrt{\sigma_{pt}^2 + u^2(x_{pt})}}$$

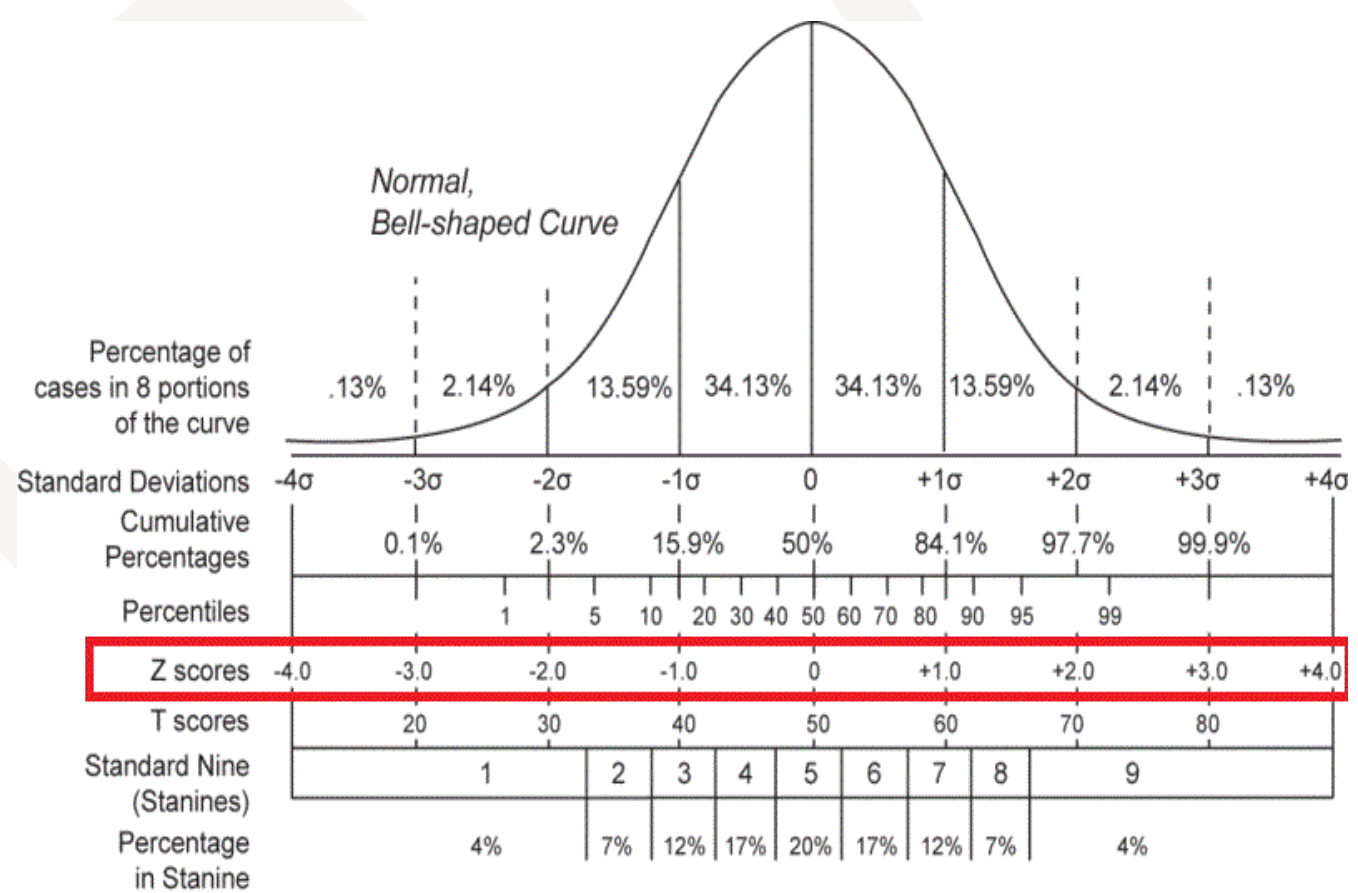
$x_i$  = Participant report's result  
 $x_{(pt)}$  = Robust mean  
 $\sigma_{(pt)}$  = Robust Standard Deviation

$u_{x(pt)}$  = uncertainty of measurement

# Z score interpretation

**$|z| \text{ score} \leq 2$  is satisfactory**  
 **$2 < |z| \text{ score} < 3$  is questionable**  
 **$z \text{ score} \geq 3$  is unsatisfactory**

(see ISO 17025:2017, 9.4.1.1, ISO 13528:2015):



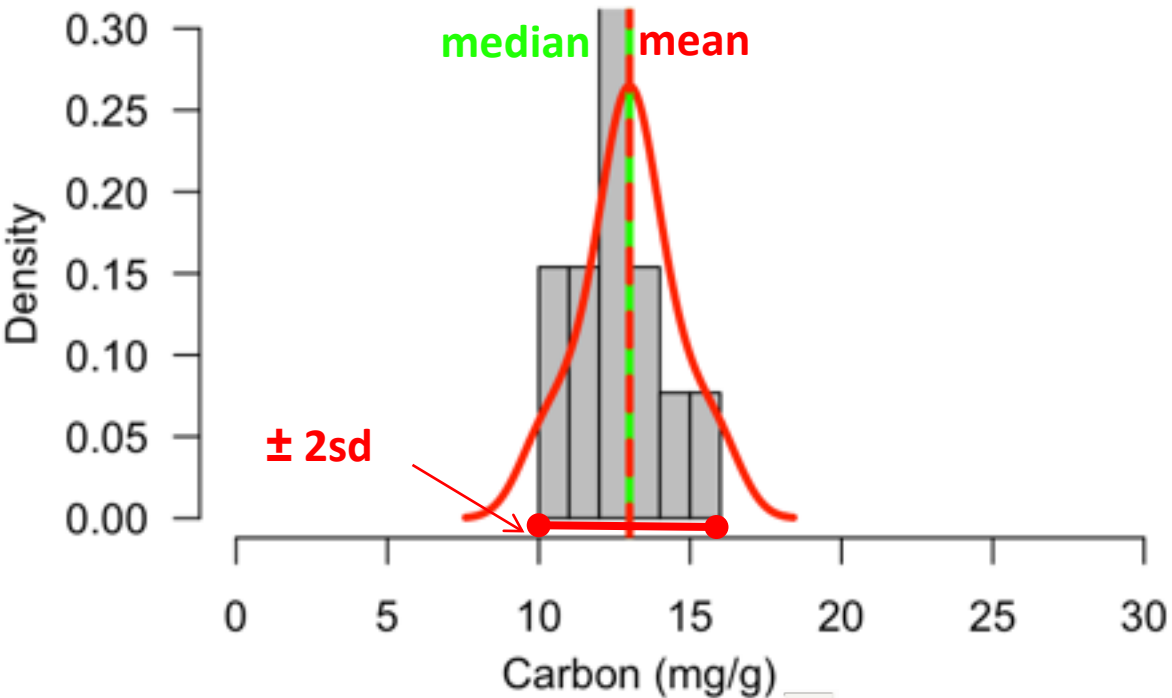
Z-score of -1 is that about 34.13% of the laboratory got result **below** the consensus mean. Similarly, the Z-score of +2 implies that 47.42% of the laboratory got result **above** the consensus mean.





# Determination of the Assigned value: mean vs median

For normal distribution:  
mean = median

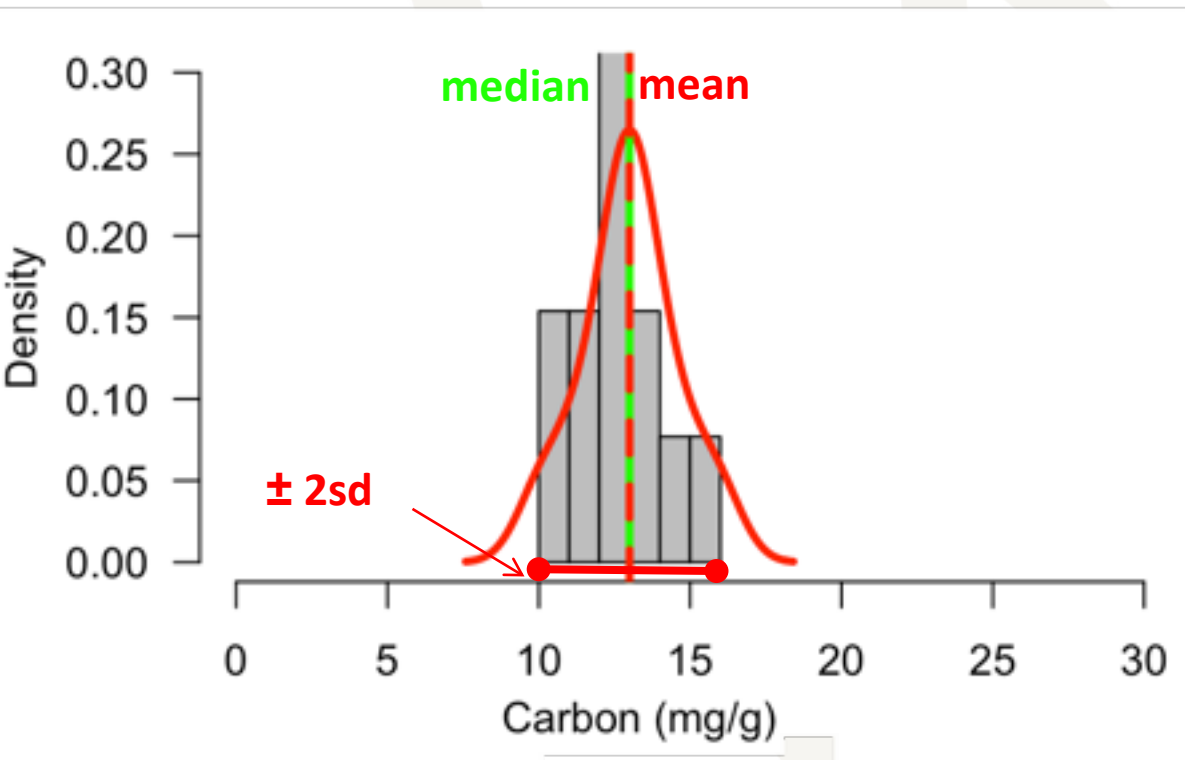


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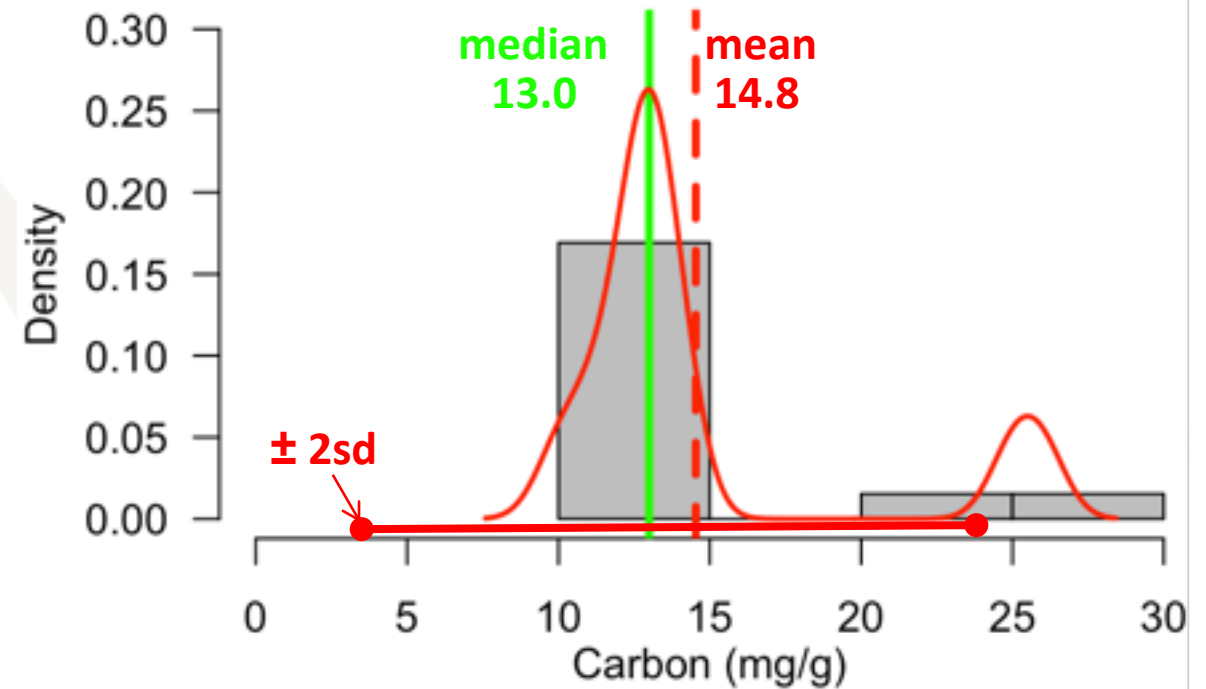


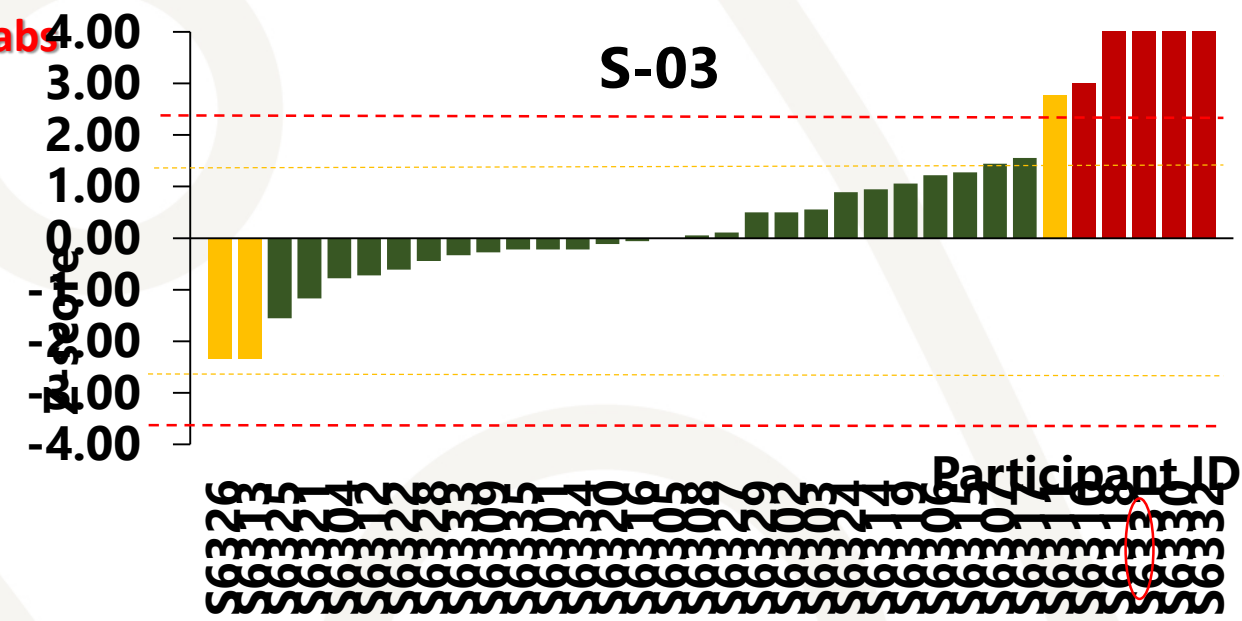
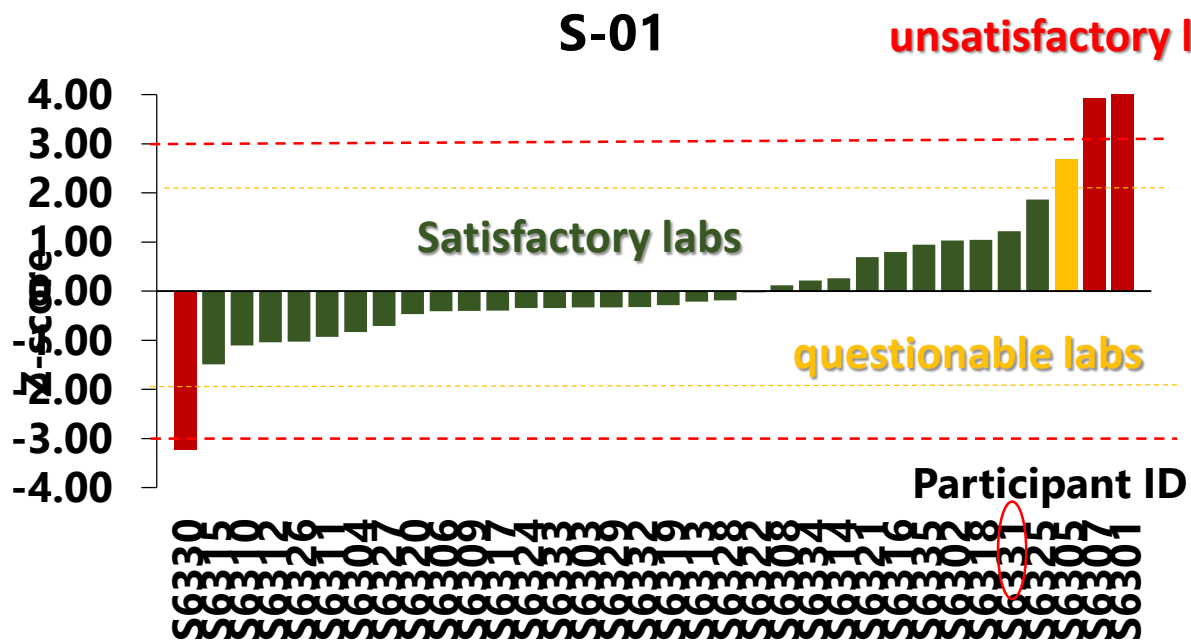
# Determination of the Assigned value: mean vs median

For normal distribution:  
mean = median



For NOT normal distribution:  
mean  $\neq$  median

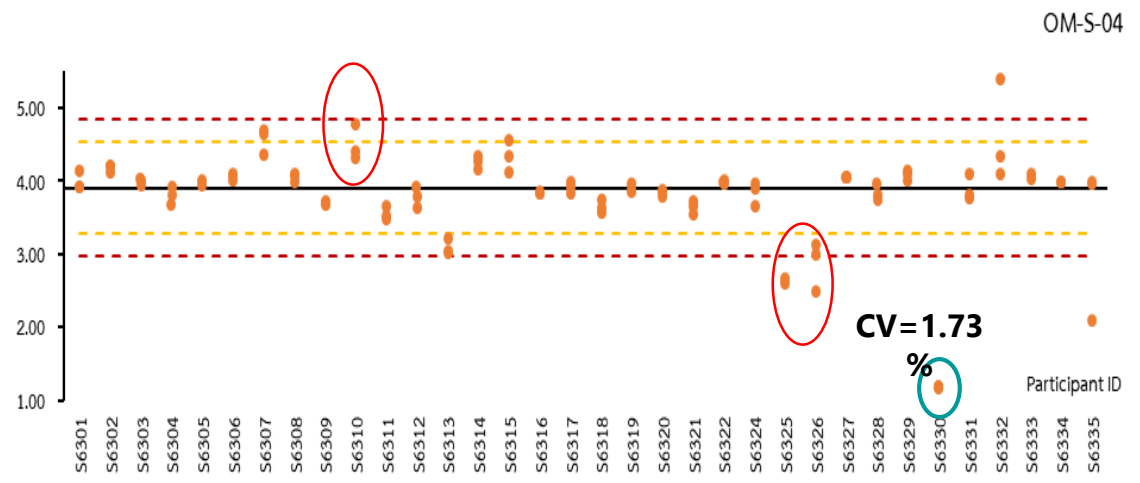
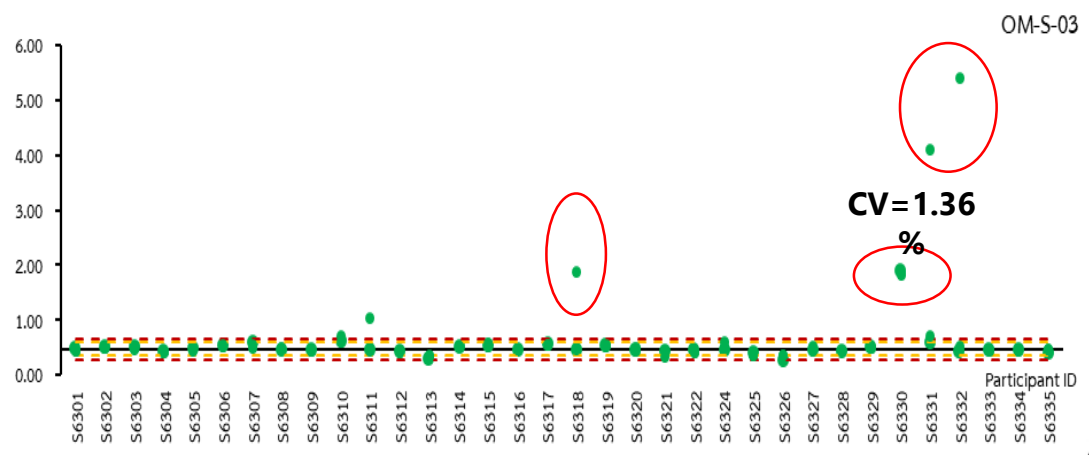
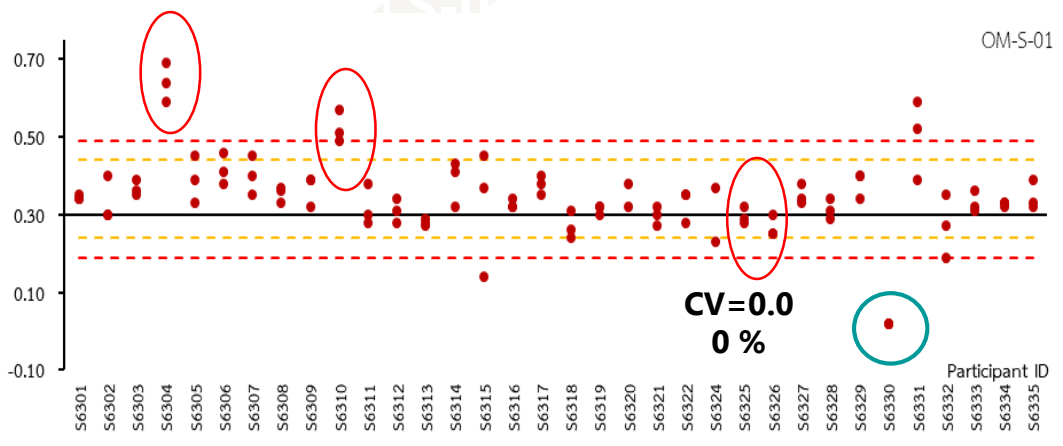




wrong data,  
 wrong conclusion,  
 wrong decision making in field and in political decision



# Precision



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# Conclusion

- ❖ A mandatory external quality control as interlaboratory comparison (ILC) or proficiency test (PT) is important for all laboratories in order to ensure for their comparable and reliable data
- ❖ With a participating to the ILC or PT programme, the quality of the laboratory and comparability of lab data will be test continuously
- ❖ ILC or PT consolidates or provides guidance for strengthening the approach to quality control of the laboratory.
- ❖ The results from the performance assessment of laboratory can be used for laboratory quality improvement

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Thank you for your kind attention

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