Measurement uncertainty analysis

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Measurement uncertainty in ISO/IEC 17025

7.6 Evaluation of measurement uncertainty

7.6.1 Laboratories shall identify the contributions to measurement uncertainty. When evaluating measurement uncertainty, all contributions that are of significance, including those arising from sampling, shall be taken into account using appropriate methods of analysis.

7.6.2 A laboratory performing calibrations, including of its own equipment, shall evaluate the measurement uncertainty for all calibrations.

7.6.3 A laboratory performing testing shall evaluate measurement uncertainty. Where the test method precludes rigorous evaluation of measurement uncertainty, an estimation shall be made based on an understanding of the theoretical principles or practical experience of the performance of the method.

Measurement uncertainty in ISO/IEC 17025

NOTE 1 In those cases where a well-recognized test method specifies limits to the values of the major sources of measurement uncertainty and specifies the form of presentation of the calculated results, the laboratory is considered to have satisfied 7.6.3 by following the test method and reporting instructions.

NOTE 2 For a particular method where the measurement uncertainty of the results has been established and verified, there is no need to evaluate measurement uncertainty for each result if the laboratory can demonstrate that the identified critical influencing factors are under control.

NOTE 3 For further information, see ISO/IEC Guide 98-3, ISO 21748 and the ISO 5725 series.

Measurement uncertainty

- the Measurement uncertainty theory states that all **<u>Measurement are estimation</u>** (since it is based on probabilities).

- Because of variation, there is **<u>no</u>** such thing as a **<u>true value</u>**

- **<u>To obtain the true value</u>**, all **<u>sources of variation</u>** (user, method,……environment) must be **<u>identified</u>** and controlled. since this is **<u>impossible</u>**, all measured values are estimation and a true value will never exist

- **True value:** it is the measured value which measured by the **most accurate method** but **no zero error value**.

- There is **no true value** but there is **the probability to find true value**.

- It is important to make enough control.

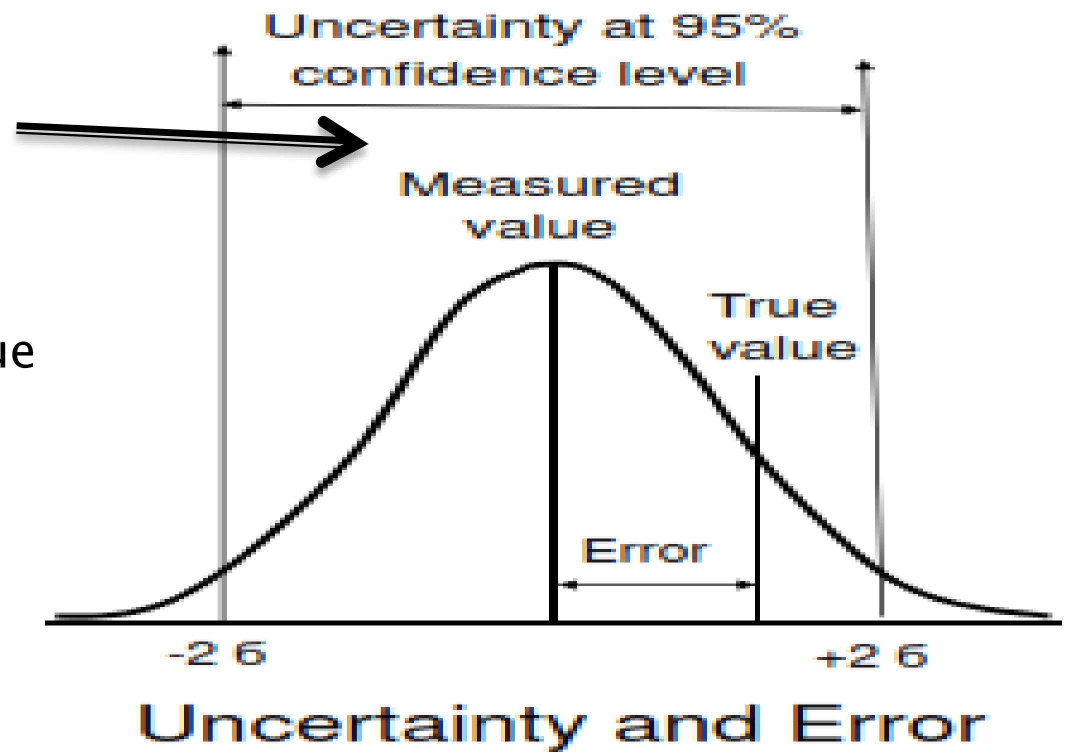
- The **main target** is to **control** the **variables** to **reduce** the **error** (the distance from the true values) as much as possible. Accordingly, **reducing** the **estimated uncertainty** value

- The **qualitative** laboratories such microbiology labs (that give +ve or –ve) or physics labs (go or not go) **may not** can estimate the uncertainty value of the test but only estimated for **quantitative** labs only.

- The uncertainty value of the test may **differ from range to range** (in some cases especially in **calibration labs**) according to the variations in the system.

Measurement uncertainty

متوسط القراءه



If measured value = true value
Then error is zero

To estimate the uncertainty value the measured value must fit the normal distribution curve.

Measurement uncertainty

Measurement Error

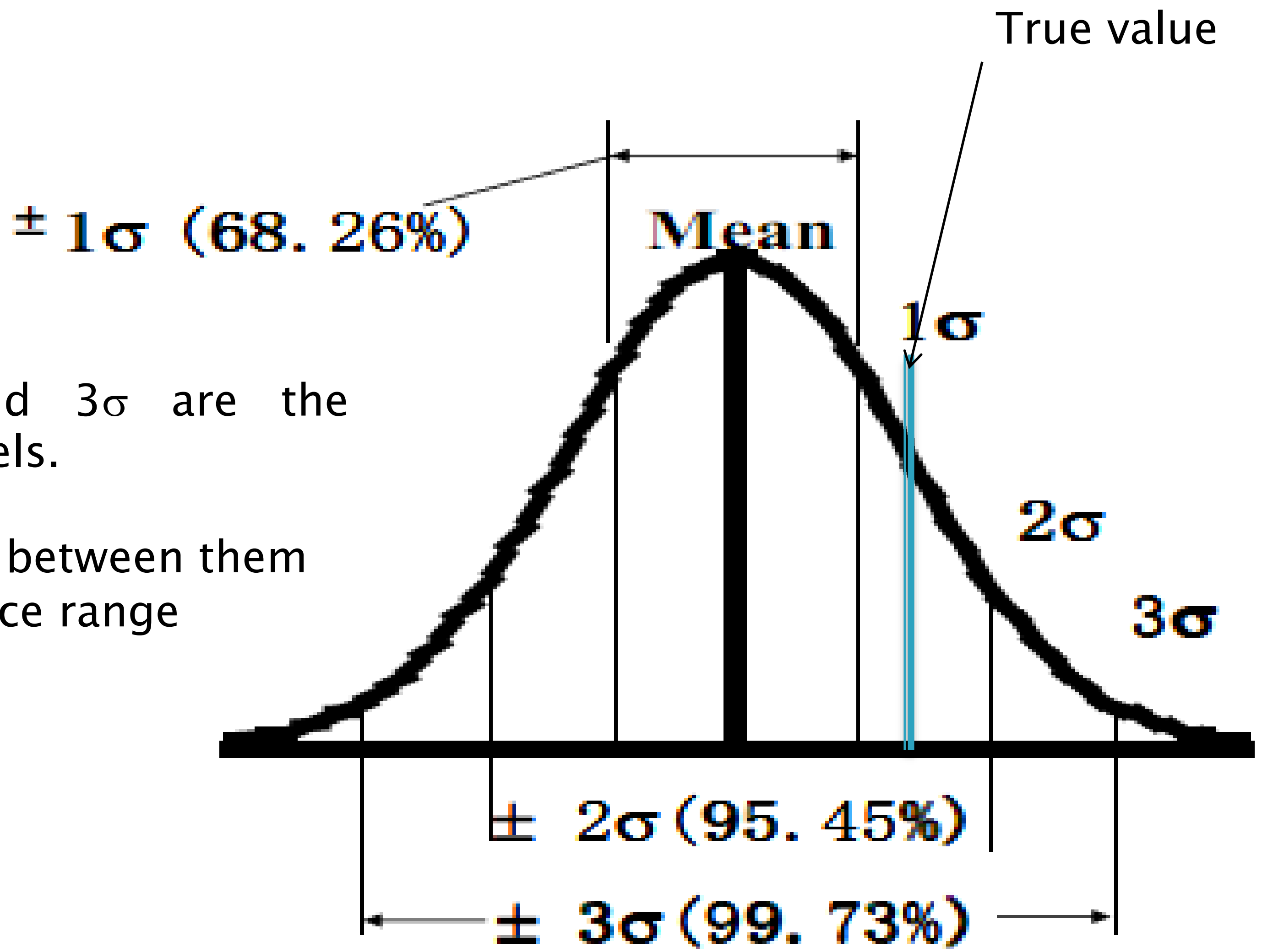
- **Error** is the **difference** between the result of the **measurement** and the value of the quantity **true measured**.

- Error viewed is being of two kinds: **random error** (**type A**) which related to **precision** component and **systematic error** (**type B**) which related to **accuracy** component.

- **Uncertainty**

is the **range of values**, usually centered on the measured value, that contains the true value with stated probability

Measurement uncertainty



- 1σ, sσ and 3σ are the confidence levels.

- The distance between them is the confidence range

Measurement uncertainty

	Level of confidence	% error	Confidence
1σ	68.26	31.74%	Very low
2σ	95.45	4.55	moderate
3σ	99.73	0.27	Very high

So 95% was globally taken as level of confidence

Measurement uncertainty

- We would say “the **true value** can be expected to lie within **± X units** of the measured value with **95% confidence**

- uncertainty is estimated by an uncertainty analysis that take into consideration the effect of systematic and random errors in all the measurement processes

Why is measurement uncertainty Important?

- The uncertainty is **quantitative indication** of the **quality** of the **results**.

- Knowledge of the uncertainty shows the **acceptance** of the result within the **limits defined** in **specification** or regulation.

- Various **standards** and **guides** require analysis of uncertainty in **measurement** processes.

- Assessment of the uncertainty is one of the techniques use for **validation of methods** according to **ISO 17025**

Sources of measurement uncertainty

- Incomplete **definition** of measured quantity.
- Non representative **sampling**.
- **Environmental condition** effects.
- Instrument **resolution** .
- **Personal bias** in reading analogue instrument
- Inexact values of measurement **standards** and **references materials**
- Approximate and assumptions in **measurement methods** and **procedure** variation in repeated measurement

Calculating measurement uncertainty

Before starting

- Great deal of **understanding principles** of the **measurement and tractability** is required to estimate measurement uncertainty

- Basic knowledge of **mathematics** and **statics** required

- Ensure that the process of **determining** uncertainty is under **statistical control**

Calculating measurement uncertainty

Steps for calculating measurement uncertainty

1. Identify the **uncertainties** in the measurement process
2. Classify **type** of uncertainty(A or B)
3. Quantify (evaluate and calculate) **individual uncertainty** by various methods
4. **Combine uncertainty** by root **sum square method** (RSS methods)
5. Assign appropriate **K factor** multiplier to combined uncertainty to report expanded uncertainty
6. **Document** in an uncertainty budget
7. Document an uncertainty in the **test report**

Calculating measurement uncertainty

1-Identify the uncertainties in the measurement process

Determine **contribution** of **factors** affecting measurement

- Environment
- Operators
- Measuring instruments
- Measuring methods and procedures
- Calculation methods
- Other constrains

Calculating measurement uncertainty

2. Classify type of uncertainty (A or B)

- Uncertainty **components** are **grouped** into two major categories depending on the source of data . The categories are **type A** and **type B** uncertainties

Calculating measurement uncertainty

2.1.Type A uncertainty

- Uncertainty of measurement evaluated by statistical analysis of **series of observations (repeatability)**. In this case the **standard uncertainty** is the experimental **standard deviation** of **the mean** that follows from an averaging procedure

إعادة النتائج على فترات زمنيه قصيره سواء بشخص واحد او بعدة أشخاص ونأخذ (SD) حيث يعتبر هذا هو قيمة اللايقين الناتج من النوع A.

ليس معنى أن type A صفر ان نتائج المعمل بالتأكيد صحيحه ولكن يمكن ان يكون ال resolution فى المعمل لا يستطيع حساب الخطأ.

Calculating measurement uncertainty

2.2.Type B uncertainty

- Type B evaluation of standards uncertainty is the evaluation of the uncertainty by means **other than the statistical analysis of a series of observations**.
- Type B is all uncertainty components **except** repeatability and/or reproducibility.
- It is based on **experience** and **general knowledge**. It is a skill that can be leaned with practice.

Repeatability and reproducibility

Comparison	Repeatability	Reproducibility
Period of observations	Short period	Long period
Personnel observed	One personnel	Different personnel

Calculating measurement uncertainty

2.2.Type B uncertainty

Values of this type of uncertainty are driven at least from:

- **Previous measurement data**.
- Experience of general knowledge of the behavior of the **equipment**.
- **Manufactures specifications.**
- Data in **calibration**.
- Data in hand **books** .
- **Environmental conditions** effect.

Calculating measurement uncertainty

3. Evaluate and calculate individual uncertainty by various methods

- **Compute a type A** standard uncertainty for **random sources of error** from series of measurement observations
- **Compute** a standard uncertainty for each **type B** component.

Calculating measurement uncertainty

3.1.Type A uncertainty evaluation

- Arithmetic mean (الوسيط الحسابى) or average of repeated measurements of one analyst in the same instrument in short time

مجموع القراءات

$$\bar{x} = \sum x_i / n$$

التكرار

مجموع القراءات

المتوسط

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

Experimental standard deviation $$S = \sqrt{S^2}$$

Calculating measurement uncertainty

3.1. Type A uncertainty evaluation

Experimental standard deviation of the mean:

$$S(\bar{x}) = \frac{S}{\sqrt{n}}$$

Standard Deviation

عدد القراءات

Standard uncertainty is the experimental standard deviation of the mean

$$U(\bar{x}) = S(\bar{x})$$

Standard Uncertainty of type A

Calculating measurement uncertainty

3.2.Type B uncertainty evaluation

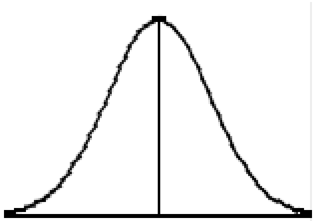
- The standard uncertainty is calculated from known characteristics of the **distribution**.
- There are 3 distributions that can be considered, **rectangular, triangle and normal.**
- One **can't combine** normal and non normal distributions when combining uncertainties
- Apply **correlation factors** when combining normal and non normal distribution

Type B

لا يمكن جمع الانواع الثلاثه معاً إلا بعد الضرب فى factor معين

Normal

- Has the **smallest standard deviation**
- characterized by **the mean** and the **standard deviation**
- represents the **statistical** behavior of much of **quantities**
- Example: Repeatability and reproducibility

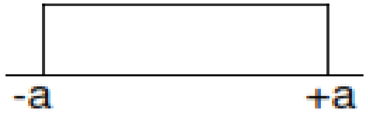


دائماً شهادات المعايره والمواد المرجعيه تكون عند
لذا يجب قسمة قيمة اللايقين الاتيه منها على 2 (K=2)

Rectangular

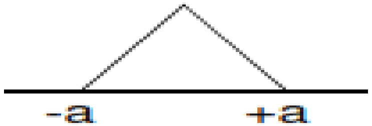
- Gives the **largest** standard deviation
- used if only **upper and lower limits values** +a,-a of the quantity **are known**
- It is often used when information is driven from **manufacture's specifications such as resolution.**
- Example: resolution of a given instrument which is a fixed value in the catalogue of instrument

$U(x) = 1/\sqrt{3}\ a$



Triangle

- Smaller standard deviation than the rectangular distribution
- It is often used in evaluation of noise and vibration
- $U(x) = 1/\sqrt{6}\ a$



Calculating measurement uncertainty

3.2.Type B uncertainty evaluation

- the uncertainty assigned to **the calibration** of the **references equipment** will be stated on the **certificate of calibration** with certain **confidence level** and **coverage factor** and it is has normal distribution
- U(X1)= Expanded uncertainty/K, where, k is the coverage factor.

The same rule is applied to the certified reference materials

Calculating measurement uncertainty

3.2.Type B uncertainty evaluation

All uncertainty components (standard deviations) of type B are combined by **root sum square** (RSS).

U(B)= $\sqrt{U_1^2 + U_2^2 + \ldots\ldots}$

Calculating measurement uncertainty

4. Combined uncertainty

- **Combine type A and type B** standard uncertainties into combined standard uncertainty by RSS

$$Uc = \sqrt{U_A^2 + U_B^2}$$

- This is the combined standard uncertainty of the measurement statistically **one standard deviation**

Calculating measurement uncertainty

5. Expanded uncertainty

- The uncertainty is expressed in **more than one standard deviation basis**
- Expanded uncertainty provide high level of confidence
- Expanded uncertainty should be calculated by **multiplying** the **combined uncertainly** by a coverage factor **K** (confidence level).

$$U_m = K\ U_c$$

Calculating measurement uncertainty

5. Expanded uncertainty

Coverage factor can be 1 or 2 or 3

Assuming normal distribution

- For K=1, the confidence level is 68%
- For K=2, the confidence level is 95%
- For K=3, the confidence level is 99%

Coverage factor 2 is often used for approximately confidence level is 95.45%

Calculating measurement uncertainty

6. Uncertainty budget

All results are tabulated to include :

- All uncertainty components
- Standard uncertainties
- Probability distribution
- Correction factors
- Coverage factors
- The effective degree of freedom

Calculating measurement uncertainty

6. Uncertainty budget

- The combined uncertainty
- The expanded uncertainty

Uncertainty components taken to be negligible must **still be documented and justified**

Calculating measurement uncertainty

7. Uncertainty reporting

The minimum requirements when reporting measurement results :

- Description of the measured quantity
- State the result of measurements as **y= Y ±U** with the **units** of y and U
- Value of **coverage factor K** used to obtain U
- Approximate **level of confidence** associated with interval

Y ± U and how it is was determined

It is usually sufficient to report uncertainty estimate to **no more than two significant digits**

Calculating measurement uncertainty

7. Uncertainty reporting

Example

- The reported measured value and the uncertainty are written in the calibration / Test certificate as

$$\underline{\mathbf{Y \pm U}}$$

- With the following statement :

<u>The reported expanded uncertainty of measurement is stated as the standard uncertainty of measurement multiplied by the coverage factor k=2 which for a normal distribution corresponds to confidence level of 95%</u>

Calculating measurement uncertainty

7. Uncertainty reporting

In the case of using K_p the following statement is used:

The reported expanded uncertainty of measurement is stated as the standard uncertainty of measurement multiplied by the coverage factor Kp =XX which for t-distribution corresponds to the effective degree of freedom V_{eff}=z z with confidence level of 95%

Calculating measurement uncertainty

- **Reasonability**

Every uncertainty estimate should be subjected to a "**<u>Reasonability check</u>**"

- Is this estimate **<u>reasonable</u>** ?

<u>Uncertainty estimates that look strange – either too big or too small should be re-evaluated</u>

Case study 1

Using Down top approach (GUM)

One of the lab tests requires weighting of sample with defined uncertainty

The measurement has been repeated 10 times using digital balance with resolution 1 mg

The mean value and the standard deviation for the 10 measurements have been calculated as 250 mg and 1.2 mg respectively

The calibration certificate for the digital balance includes measurement uncertainty 1.5 mg for the measurement range with K=2 and confidence level 95%

Report the measured value with the measurement uncertainty

Source of uncertainty	Value	Shape	Divisor	Standard uncertainty	Square
Repeatability					
Calibration					
Resolution					

Source of uncertainty	Value	Shape	Divisor	Standard uncertainty	Square
Repeatability	1.2	Normal	1	1.2/√10 = 0.38	0.38 * 0.38 = 0.144
Calibration	1.5/2	Normal	1	0.75	0.75*0.75 = 0.5625
Resolution	1.0/2	Rectangular	√3	0.5/√3 = 0.29	0.29*0.29 = 0.0833

- $U_C = \sqrt{0.144 + 0.5625 + 0.0833} = 0.888$
- $U_A/U_C = 0.144/0.888 = 0.162 < 0.5$
- $U_{exp} = 2*0.888 = 1.78$

Reporting

250 ± 1.78 mg

The reported expanded uncertainty of measurement is stated as the standard uncertainty of measurement multiplied by the coverage factor k=2 which for a normal distribution corresponds to confidence level of 95%

Measurement uncertainty implementation in testing laboratories

Key issues

- Measurement uncertainty is estimated for **quantitative and semi quantitative**

- It is the **lab responsibility** to determine the **tests or examinations** that requires **measurements uncertainty estimation**

- Existing **lab information** is converted to standard uncertainty which are **combined** to give **total** uncertainty estimate

Main Approach

	The bottom up approach	The Top Down approach
Principle	GUM (Guide to Uncertainty Measurement) principles	lab test performance :
Based on	uncertainty expressed as standard deviations	method validation ,intra lab, inter lab QC data information
Mathematics	complex mathematics	Easier mathematics
Suitability	may not be suitable for routine chemical lab tests	It is suitable for routine chemical lab tests

- The main target is to increase the precision and accuracy

- **Standard deviation** is the indication of **precision**

- **Bias** is indication of **accuracy**

- Bias, B= Test Result – Reference value

Measurement uncertainty by Top Down approach

- Use performance data from both **internal QC** (intera lab) and **PT program** (inter lab)
- A stable or well established methods or procedures for test
- Due to time , **multiple users** , **reagents** and **calibrators** lots are captured.

Type A = Reproducibility and or Repeatability

Measurement uncertainty by Top Down approach

- For new methods, a **minimum of 30 replicate** determination of appropriate control or **RM** is required to calculate the interim (المؤقته) standard deviation
- If **Bias** is significant, calculate the **combined uncertainty**
- **Precision and accuracy** data from **method validation** can be used when no significant change to procedures following validation.
- Do not **judge** the **significant** of precision and accuracy without making **complete calculation** and having the combined type A and type B uncertainty.

Measurement uncertainty by Top Down approach

5. Steps calculations

1. Calculate the overall SD of the method from monthly SD's (imprecision) for at least two levels of QC

$$SD = \sqrt{SD_{L1}^2 + SD_{L2}^2 / 2}$$

Where SD_{L1} and SD_{L2} are the average SD of each control level for the past 6 months

Assume number of monthly QC points n is the same for each level

Measurement uncertainty by Top Down approach

- For more than 2 levels

$$SD = \sqrt{n_1 SD_1^2 + n_2 SD_2^2 + \ldots\ldots n_x SD_x^2 / (n_1 + n_2 + \ldots n_x)}$$

Where n is the number of monthly QC points

X is the number of levels

(n_1 at level 1,n_2 at level 2 ,….etc

Calculate standard uncertainty associated with the method

US=SD average

Measurement uncertainty by Top Down approach

For some methods , with varying **imprecision** at different decision limits or control levels, measurement uncertainty must be **calculated** at **each** decision or control level.

Measurement uncertainty by Top Down approach

2. Calculate the bias, B associated with the method (intra laboratory & inter laboratory bias)

Bias, B = Test Result – Reference value

Where the **reference value** can includes any **assigned value** obtained from a higher order references measurement procedure, from **CRM**, from **peer group** and all methods mean values from PT program

Peer group: when group uses the **same test method** for analysis of **PT** samples then there will be **2 means** (z scores) one for that group and the other one for all the group.

Measurement uncertainty by Top Down approach

3. Calculate the combined standard uncertainty of the method

$$U_C = \sqrt{U_S^2 + U_B^2}$$

4. Calculate the expanded uncertainty of the method U

$$U = U_c \times K$$

K = 2 for confidence level approximately 95%

Measurement uncertainty by Top Down approach

5. Report the results as (**<u>measured value ± U</u>**) (units)

- State the results of measurement as **<u>Y ± U</u>** with the **<u>units</u>** of y and U
- Value of coverage factor **<u>K</u>** used to obtain **<u>U</u>**
- Approximate level of confidence associated with the interval **<u>Y ± U</u>** and how it was determined

Y is the measured value

U is the expanded uncertainty

EX 60 ± 0.5 µg/L zinc

Measurement uncertainty by Top Down approach

- Calculation of the standard uncertainty associated with the bias

$$U_B = \sqrt{U_{ref}^2 + SR_g^2 + SEM^2}$$

standard uncertainty associated with a traceable **references material** or **calibrator**, or to an assigned / **references value**

→ Expanded **uncertainty** of **CRM** (**divided by 2**)

standard uncertainty associated with a **specified references**, estimated from all-method group reproducibility PT data.

→ Standard deviation of the **scheme** of the **PT**

standard **error of mean** uncertainty of the method derived from in laboratory replicate measurements of a reference material

→ **CRM** measured **10 times** and the **mean** is taken

Measurement uncertainty by Top Down approach

5. Report the results as (**measured value ± U**)(**units**)

EX.

A standard solution of Pb in water is stated by the supplier to contain **10 ±0.05 µg/L**

The reported expanded uncertainty **U = ±0.05 µg/L**

Us = ± 0.05 µg/L

When a confidence level is not stated, a 95 % confidence level is assumed (with K=2)

Case study 2

- XYZ lab has received the PT report for participation in the PT scheme for determination of Zinc concentration in a certain solution, the report indicates that the mean value is 69µg/L

- The standard deviation is 0.03 µg/L
- The long – term SD from six months in house quality control is 2.24 µg/L

- The method is regularly verified and the testing equipment is calibrated with a calibration solution of expanded uncertainty 0.5 traceable to a certified international standard as per manufacture

- The standard deviation of 10 replicate measurement of the standard solution is 0.024 µg/L

Calculate the expanded uncertainty and report it as standard deviation or coefficient of variations

Case Study 2 Continued.

- Calculate CV%.
- Calculate Z score of the lab as a result of PT participation when the value of zinc conc. Measured by lab and sent to the PT provider is 68.97 µg/L , what is your opinion about The lab performance.

Component	S components		B components	
	Value	$(Value)^2$	Value	$(Value)^2$
S1 (long term SD)	2.24	5.0176		
SEM (SD of ten replicate)			0.024	0.000576
U_{ref}			0.5/2 =0.25	0.0625
SR_g			0.03	0.0009
SUM individual		5.0176		0.063976
ΣSquare of US+UB	5.0176 + 0.063976 = 5.081576			
Square root of the sum	√5.081576 = 2.254			
$U_{combined}$	2.254			
$C_{expanded}$	2.254 * 2 = 4.508			
CV%	**(U/mean)*100 = (4.508/69)*100 = 6.53**			
Z Score	(measured value - Mean)/SD = (68.7 – 69) / 0.03 = -1			
Satisfaction	1< Z < 2 which is Satisfactory			

Case study 3

-The determination of nickel (Ni) in low alloy steel with x- ray fluoresce indicates that the mean value for running the quality control material with high concentration of nickel over 6 months is 6.8% and the impression SD is 0.14 %

-The certified references material CRM used for the calibration is provided with a certificate that include the expanded uncertainty of 0.2 % at k=2

-The replicate measurement of CRM are carried out by the lab for bias estimate using the complete measurement procedure the calculated standard deviation for the replicates is 0.1%

- Calculate and report the expanded measurement uncertainty

Component	S components		B components	
	Value	(Value)^2	Value	(Value)^2
S1 (long term SD)	0.14	0.14*0.14 =0.196		
SEM (SD of ten replicate)			0.1	0.1*0.1=0.01
U_{ref}			0.2/2 =0.1	0.1*0.1=0.01
SR_g				
SUM individual		0.0196		0.02
ΣSquare of US+UB	0.0196 + 0.02 = 0.0396			
Square root of the sum	√0.0396 =			
$U_{combined}$	0.199			
$C_{expanded}$	0.199* 2 = 0.4			
CV%	**(U/mean)*100 = (0.4/6.8)*100 = 5.88**			

Thank you & any question