Top down MU method – ISO 21748 explained simply

One of the most popular top down approaches for the estimation of measurement uncertainty is referred to ISO 21748:2010: *Guidance for the use of repeatability, reproducibility and trueness estimates in measurement uncertainty estimation*.

As usual, all standard documents are being written by experts who tend to write in much greater details. But for us as practitioners, we would like to be practical and to catch only the main points to do our work smoothly. Hence, this article is trying to dissect and simplify the approach.

ISO 21748 advocates the estimate of reproducibility from an inter-laboratory method validation study (including continued participation in established proficiency testing PT program(s)) can be used as an estimate of measurement uncertainty (MU).

This requires the laboratory to demonstrate competence with the test method concerned and such uncertainty estimate is suitable for the laboratory's use. If so, no additional data need to be generated, other than to estimate repeatability and bias (trueness), which should have done anyway by the laboratory in its method validation process.

The evaluation of repeatability, reproducibility and bias (trueness) is basically checking the method performance whilst measurement uncertainty relates to individual result. Hence, this international standard suggests that these process-performance figures derived from method performance studies are relevant to all individual measurement results produced by the process. Hence, this assumption requires supporting evidence in the form of appropriate QAQC data for the measurement process.

Basic principles of ISO 21748 approach

The statistical model for this approach is based on the following equation used in collaborative study for a test method:

$$y = \mu + \delta + B + \sum c_i x_i' + e \qquad \text{Eq [1]}$$

Where

- y is the measurement result, assumed to be calculated from an appropriate function;

μ is the (unknown) expectation of ideal results (i.e. true value);

δ is a term representing intrinsic *method bias*, assumed normally distributed with variance σ_L^2 having s_L^2 as estimate

B is the laboratory component of bias, Δ;

x_i' is the deviation from the nominal value of x_i;

c_i is the sensitivity coefficient, equal to $\partial y / \partial x_i$;

e is the random error term under repeatability conditions, assumed normally distributed with variance σ_r^2 having s_r^2 as estimate

and,

$\Sigma c_i x_i'$ is the summation of deviation effects other than those incorporated in δ, B, and e.

Given the model described by Equation [1] above, the uncertainty $u(y)$ associated with an observation can be estimated using Equation [2]:

$$u^2(y) = u_{ref}{}^2 + s_L{}^2 + \sum c_i{}^2 u^2(x_i) + s_r{}^2 \qquad \text{Eq [2]}$$

where

s_L^2 is the estimated variance of B;

s_r^2 is the estimated variance of *e*;

$u(ref)$ is the uncertainty associated with δ due to the uncertainty of estimating δ by measuring a reference measurement standard or reference material with certified or reference value;

$u(x_i)$ is the uncertainty associated with x_i'.

Given that the reproducibility standard deviation s_R is given by

$$s_R^2 = s_L^2 + s_r^2$$

the equation [2] can be further reduced to equation [3] below:

$$u^2(y) = u_{ref}{}^2 + s_R{}^2 + \sum c_i{}^2 u^2(x_i) \qquad \text{Eq [3]}$$

Important points to be noted

1. This ISO standard assumes there was an appropriate validation study design and data analysis (including removal of outliers), and that the estimates of repeatability, r and reproducibility, R are suitable for use in the laboratory. It takes the estimate of reproducibility standard deviation, s_R as a provisional estimate of measurement uncertainty, u'.

2. The estimates of repeatability in the form of repeatability standard deviation (s_r) and reproducibility in the form of reproducibility standard deviation (s_R) are found in the reports of inter-laboratory collaborative or PT studies. Today, most, if not all, standard or official test methods would normally state its s_r and s_R as well.

3. If those study reports state the values of repeatability r and reproducibility R, instead of s_r and s_R, we can make use of the following relationships for convenience:

   $r = 2.8 \times s_r$
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4. It follows from equation [3] that the between-laboratory standard deviation s_L can be calculated from equation [4] as below:

   $$s_L = \sqrt{s_R^{\ 2} - s_r^{\ 2}} \qquad \text{Eq [4]}$$

5. If the laboratory has previously carried out its own method validation process, it would have carried out replicate measurement on reference standard and estimated the within laboratory repeatability s_w with n replicates in this internal study. Where practical, n should be chosen such as $\sqrt{s_w^{\ 2}/n} < 0.2s_R$. Ideally the study would have carried out at least 10 replicates.

6. The estimate of experimental standard deviation of results obtained by repeated measurement on a reference material used for checking control of bias, s_D is then calculated by the following equation [5]:

   $$s_D = \sqrt{s_L^{\ 2} + \frac{s_w^{\ 2}}{n}} \qquad \text{Eq [5]}$$

7. The laboratory bias (B_L) can be estimated from repeated measurements of reference materials, comparison with a reference laboratory, or from PT testing, using the following equation [6]:

$B_L = \Delta = |\text{Laboratory Mean} - \text{Reference Value}|$ Eq [6]

8. According to ISO 21748, the acceptable criterion for the laboratory bias is to satisfy the following **bias limit** condition:

$$\Delta \leq 2s_D \qquad \text{Eq [7]}$$

If the B_L is larger than the limit, then the method cannot be used. The reason for the observed bias must be investigated and corrective action is to be taken.

9. Also, according to ISO 21748, the acceptance criterion for repeatability is to satisfy the following **precision limit** condition:

$$S_w \leq 1.5s_r \qquad \text{Eq [8]}$$

If s_i is larger than the precision limit, the method may still be used, but the provisional estimate must be expanded as follows:

$$u' = \sqrt{s_L^{\,2} + s_w^{\,2}}$$

10. When the above two criteria (points 8 and 9) are satisfied, the combined standard uncertainty is then:

$$u = \sqrt{u'^2 + s_a^{\,2} + s_b^{\,2}} \qquad \text{Eq [9]}$$

where,

s_a, s_b are any other components of variability that were not included in the validation experiment, such as uncertainty in sub-sampling or sample preparation or pre-treatment

11. The expanded uncertainty U with 95% confidence using coverage factor k =2 is therefore,

$$U = 2\text{x}u.$$

12. Usually, u_{ref} value tends to be very much smaller than the other standard uncertainties and can thus be neglected.