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Measurement uncertainty evaluation in soil liquid limit testing

K L Lua[1], A J M S Lim[2*] and S Y Sim[3]

[1] Fakulti Kejuruteraan Awam dan Alam Bina, UTHM, [+]C.E.O. Ascendent Technology Sdn Bhd
[2] Research Center for Soft Soil, Big DATA and Advance Analytics Research, Department of Civil Engineering, Faculty of Civil Engineering and Built Environment, Universiti Tun Hussein Onn Malaysia.
[3] Department of Electrical Engineering, Faculty of Electrical and Electronic Engineering, Universiti Tun Hussein Onn Malaysia
*Corresponding author: alvin@uthm.edu.my

Abstract. Measurement uncertainty provides an indication of the quality of test result and very often also adopted in laboratory proficiency testing activities. Soil mechanical properties testing for example plastic and liquid limits conventionally do not provide uncertainty figure; however due to laboratory accreditation requirement, laboratory is required to provide it in the test report for some specific purposes for example Proficiency testing and quality validation. Conventional approach to derive measurement uncertainty is too difficult or mathematically unable to justify for the figure derived because liquid limit is not obtained experimentally instead it is estimated through linear regression method. Thus, it becomes a problem to report accurate and reliable measurement uncertainty figure in the test report. This research focuses into estimating the water content of liquid limit with relative uncertainty approach through estimation from the regression curve. It suggested the technique for estimation of water content and dry weight resulting in uncertainty component different treatment from conventional approach. Error of regression, a Type A component becomes one of the key components dominating the final answer. This study suggests the control of quality and improvement of measurement uncertainty evaluation through curve fitting observing the value of R^{2} should not be less than 0.95. This uncertainty figure can be used in proficiency testing and inter-laboratory comparison purpose.

1. Introduction

Accuracy of test result is reflected by the reported measurement uncertainty figure. This figure also indicates the presence of metrological traceability. Soil mechanical properties for example plastic and liquid limits are based on percentage of moisture content, the value of limit is derived from the regression curve fitting at 25^{th} blow for Casagrande method. In proficiency testing or Inter-laboratory comparison activities, the results would be meaningless to adopt Z-score [1] in statistical design when the number of participant "n" is low because the error of comparison is inversely proportionate to the number of participants and also Z-score does not require measurement uncertainty in the calculation. This study focuses into an alternative method adopting metrological approach where statistical design to include element of measurement uncertainty in the results would be meaningful in the interpretation of statistical information.

Standards Malaysia demands the evaluation of uncertainty shall be according to Guide to the Expression of Uncertainty in Measurement (GUM) [2]; however, testing report is not compulsory to report it except calibration certificate. It has become a common practice that laboratories performing testing activities do not pay serious attention to it. Another scenario is the variation of uncertainty model among the Skim Akraditasi Makmal Malaysia (SAMM) accredited laboratories due to Standards Malaysia does not provide models for specific tests for example soil mechanical properties liquid and plastic limits thus these laboratories exhibit large variations among them in measurement uncertainty evaluation. This study goes into developing a measurement uncertainty evaluation model for soil liquid limit for common interest.

2. Literature Review

ISO/IEC Guide 98-3 [3] in the introduction indicated that when reporting the result of a measurement of a physical quantity, it is obligatory that some quantitative indication of the quality of the result be given so that those who use it can assess its reliability. Without such an indication, measurement results cannot be compared, either among themselves or with reference values given in a specification or standard. It is therefore necessary that there be a readily implemented, easily understood, and generally accepted procedure for characterizing the quality of a result of a measurement, that is, for evaluating and expressing its uncertainty. It is very clear that measurement uncertainty denotes the accuracy of the result and the dispersion of value.

Measurement uncertainty is defined as non-negative parameter characterizing the dispersion of the quantity values being attributed to a measurand, based on the information used [4]. It is probabilistic in nature and reflects incomplete knowledge of the quantity value, thus this definition indicates that uncertainty is expressed in distribution and assuming normal distribution. Measurement uncertainty can be used to judge a laboratory's capability. In fact, calibration laboratories are required to publish Calibration and Measurement Capability (CMC) in the department's website [5]. ISO/IEC 17025 [6] clause 7.8.4 states that it is a requirement for calibration certificate to include this figure but clause 7.8.3 does not make it a requirement for testing laboratory to report in the test report. The side effect of this requirement causes testing laboratories pay less attention in this area.

ISO/IEC 17025 clause 7.7.2 requires laboratories to conduct inter-laboratory comparison and one of the key information to submit is measurement uncertainty. ISO 13528 [7] provides guidelines for organization providing such service to adopt ζ-score, Z-score and En-score in statistical design. Both ζ-score and En-score require uncertainty figures. IUPAC/CITAC Guide [8] indicated that when the number of participants is less than 30 then traditional approach in statistical design is not suitable for the error of standard deviation too large and suggested metrological approach i.e. ζ-score and En-score. Both formulae adopted uncertainty as a dominator in the deviation calculation. On the other hand the traditional approach is adopting Z-score without considering the uncertainty instead taking standard deviation of the scheme as dominator in the equation.

3. Measurement Uncertainty Model

In the process of measurement uncertainty evaluation, there are bound to unveil many factors affect the result. These factors are combined together to derive a final figure reflecting the compounded effect. There are many mathematical models for this evaluation; however, according to ISO Guide 98-3 which is adopted by ISO/IEC 17025 accredited laboratories to present measurement uncertainty is GUM method.

According to GUM the theoretical model is based on $u_c{}^2 = \sum_{i=1}^{n} \left(\frac{\partial f}{\partial x_i}\right)^2 . u_n{}^2$, where u_c is the standard uncertainty which has the characteristic of 1σ. Normally the reported uncertainty is at 95% confidence level adopting this equation $U = u_c . k$, where k is the coverage factor, and U is the expanded uncertainty. Standard uncertainty, u, is the deviation components in the measurement process. It is derived from both empirical information and the estimate from knowledge and experience and is expressed in distribution, whereas $\frac{\partial f}{\partial x_i}$ is the weighting ratio of the component to reflect the actual effect of the compounded result.

3.1. Traditional model

Measurement uncertainty is not compulsory to be stated in accredited test report; however, at times customers do request for quality control and inter-laboratory comparison purpose. The common models developed by accredited laboratories are mostly direct from weighing process and errors due to personnel but not mention anything about regression least square error. The mathematical model can be written as follow

$$W_{LL} = W_{25th\,blow} + \delta_{ref} + \delta_{oven} + \delta_{W_1} + \delta_{W_2} + \delta_{W_3} \quad (1)$$

- W_{LL} is the water content at liquid limit

3.2. Problems associated with this traditional model

There are several problems associated with this model discuss below motivated this study to research into mathematically valid model.

- Equation (3.1) cannot be established for different units direct combination. Liquid limit is in % whereas the uncertainty components may not be expressed in the same unit %.
- It is very difficult to quantify reference deviation with respect to the percentage of water content without prior knowledge of the relation. Similarly, oven temperature too, requires to know the coefficient of temperature with respect to percentage of water content.
- The uncertainty components are independent of the 25th blow water content.
- No estimation of components at 25th blow

4. Liquid Limit using Casagrande Method Measurement Uncertainty Model

This research focuses into the uncertainty model for liquid limit of soil mechanical property using Casagrande method. Plastic limit and water content are not the main discussion topic here due to common procedures among them except that Casagrande method involves transposition of abscissa into logarithmic scale and regression least square curve fitting. On the other hand, liquid limit adopting fall cone does not require to transposed into logarithmic regression. Thus, this study on Casagrande method is also applicable to fall cone method.

Liquid limit by Casagrande method is based on BS EN ISO 17892-12: 2018 Clause 5.4 and BS 1377-2:1990 Clause 4.5, in fact both standards refer to similar technique, no difference between them. The Calculation for water content *W* for liquid limit is determined as follows:

$$w = \frac{100(W_2 - W_3)}{(W_3 - W_1)} \quad (2)$$

4.1. Brief description of the test

Dried soil sample was added with water to produce 4 different percentages of water content, specimen is ideally 2 at lower than expected liquid limit, ie. Less than 25 blows and 2 at higher than this limit along the linearity curve. The wet soil specimen water content is obtained by conventional oven dry method and the corresponding number of blows are recorded. The number of blows is then transposed into logarithmic value and treated as abscissa with corresponding water content in percentage as Y-axis. For this study, a least square mathematical relation of:

$$y = m\log_{10}x + c \quad (3)$$

is derived from this information. The Liquid Limit according to the standard BS EN 1377-2, percentage of water content is derived from this relation at the corresponding 25th blow.

4.2. Uncertainty model

Liquid limit is derived from equation (2), $w = \frac{100(W_2 - W_3)}{(W_3 - W_1)}$ where

- W is the Liquid limit at 25th blow water content in %
- W_1 weight of container in g

- W_2 weight of wet specimen with container in g
- W_3 weight of dried specimen with container in g

Replacing $r = W_2 - W_3$, $s = W_3 - W_1$

Therefore, equation (2) can be rewritten into:

$$w = \frac{100r}{s} \tag{4}$$

Based on equation (4) the relative uncertainty model as follow

$$\left(\frac{u_W}{W}\right)^2 = \left(\frac{u_r}{r}\right)^2 + \left(\frac{u_s}{s}\right)^2 + \left(\frac{u_{slope\ error}}{W}\right)^2 \tag{5}$$

Rewrite equation (5), standard uncertainty of liquid limit u_W

$$u_W = W\sqrt{\left(\frac{u_r}{r}\right)^2 + \left(\frac{u_s}{s}\right)^2 + \left(\frac{u_{slope\ error}}{W}\right)^2} \tag{6}$$

4.3. Uncertainty evaluation

Uncertainty components for weighing process consists of 2 components, they are:

$$W_{1,2,3} = I + \delta ref + \delta c \tag{7}$$

where

- I is the weighing indication by the weighing machine,
- δref is the deviation cause by the reference, weighing machine. The figure is obtained from expanded uncertainty reported in the calibration certificate,
- δc is the deviation caused by correction of the weighing machine, it is obtained from the correction or error figure at corresponding weight reported in theu_W calibration certificate. Note that in the process of weighing, there is no compensation for the correction of the balance.

5. Casagrande Method Measurement Uncertainty Evaluation

This section is to verify the measurement uncertainty model discussed in Table 1. A sample collected from natural soil, dry weight about 300g was sieved through 425μm according to BS EN 1377-2. the first specimen was wet with little water and homogenized specimen to generate water content below 25th blow water content according to Casagrande procedure. A good guide is to get more than 30 blows for the first specimen. The weight of container was initially weighed as W_1. The specimen was taken for about half cup of Casagrande cup and was put in the container and together represented as yield W_2. Calculate the wet soil value X then put in oven at 105±5 °C to dry for overnight. The dried specimen was let to cool in desiccator chamber then weigh as W_3.

A second specimen follow the same procedure as the first specimen but with more water added to yield slightly higher water content then the first specimen higher than 25 blows. The number of blows were recorded and the soil specimen with weight to X±0.2g was collected. The process was continued until the fourth specimen where the water content expected to exceed liquid limit. The number of blows should be lower but near to the 25th blow for specimen 3rd and 4th specimen expected to yield even lesser number of blows than 3rd specimen.

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The uncertainty budget is as shown in Table 1.

Table 1. Measurement Uncertainty Budget for Soil Liquid Limit.

component	Standard uncertainty formula	Component value	u	u_c	U
W_1	$u_{reference} = \frac{S}{k}$	S is the expanded uncertainty from calibration certificate of weighing machine, k also obtains from the certificate	u_1=		
	$u_{correction} = \frac{S}{2x\sqrt{3}}$	S is the correction stated in the calibration certificate, it is treated as rectangular distribution with semi range included.	u_2=		
	$u_{W1} = \sqrt{u_{reference}{}^2 + u_{correction}{}^2}$				
W_2	$u_{reference} = \frac{S}{k}$	S is the expanded uncertainty from calibration certificate of weighing machine, k also obtains from the certificate	u_3=		
	$u_{correction} = \frac{S}{2x\sqrt{3}}$	S is the correction stated in the calibration certificate, it is treated as rectangular distribution with semi range included.	u_4=		
	$u_{W_2} = \sqrt{u_{reference}{}^2 + u_{correction}{}^2}$				
W_3	$u_{reference} = \frac{S}{k}$	S is the expanded uncertainty from calibration certificate of weighing machine, k also obtains from the certificate	u_5=		
	$u_{correction} = \frac{S}{2x\sqrt{3}}$	S is the correction stated in the calibration certificate, it is treated as rectangular distribution with semi range included.	u_6=		
	$u_{W_3} = \sqrt{u_{reference}{}^2 + u_{correction}{}^2}$				
$r = W_2 - W_3$	$u_r = \sqrt{u_{W_2}{}^2 + u_{W_3}{}^2}$				
$s = W_3 - W_1$	$u_s = \sqrt{u_{W_3}{}^2 + u_{W_1}{}^2}$				
Standard error of least square, n=4	$u_{slop\ error} = \frac{\sigma}{\sqrt{4}}$	The slop error is obtained from the least square error which is in 1σ level. Therefore the standard uncertainty is to be divided by square root of n, where n is the number of plot point on the least square.			
$w = \frac{100r}{s}$	$u_W = W\sqrt{\left(\frac{u_r}{r}\right)^2 + \left(\frac{u_s}{s}\right)^2 + \left(\frac{u_{slop\ error}}{W}\right)^2}$	u_W is the standard uncertainty for Liquid Limit			
U	$U = u_W.k$ *where k=2*	The expanded uncertainty is expressed as confidence level at 95%			

The results of weighing were recorded in Table 2 as shown below.

Table 2 Liquid Limit by Casagrande method

Container No	A1	E	5	A2
Number of blows	12	20	41	68
Mass of Container (W_1)g	23.27	20.29	20.37	23.15
Mass of Container + wet soil (W_2)g	52.95	53.05	52.91	53.96
Mass of Container + oven dry soil (W_3)g	45.38	44.85	44.89	47.44
Mass of water, $r = (W_2 - W_3)$g	7.57	8.2	8.02	6.52
Mass of dry soil, $s = (W_3 - W_1)$g	22.11	24.56	24.52	24.29
Water content $w = \frac{100(W_2 - W_3)}{(W_3 - W_1)}$	34.24	33.39	32.71	26.84

Table 3 Summary of regression information

The abscissa was transposed into $\log_{10}$	x, number of blows	$\log_{10}$x	y, water content %
	12	1.0792	34.24
	20	1.3010	33.39
	41	1.6128	29.42
	68	1.8325	26.84
Liquid limit y, $y = m log_{10} 25 + C$	31.57%		
m	-10.2460		
C	45.8945		
Error of Least Square	0.7479%		
R^2	0.9689		

Table 4 Reverse calculation for r_{25} and s_{25}

Specimen liquid limit	31.57%	From $y = m log_{10} 25 + C$
Average wet soil weight	31.22g	Average wet soil of 4 specimen
Range of wet soil weight	3.08g	Range from the 4 specimens
Estimated dry soil s_{25} weight	23.90g	Calculated from $w = \frac{100r}{s}$
Estimated water weight r_{25}	7.55g	Calculated from wet weight - dry weight

The uncertainty evaluation is shown as in Table 5.

Table 5. Measurement Uncertainty Budget for Soil Liquid Limit.

component	Standard uncertainty formula	Component value S	u	u_c	U
W_1	$u_{reference} = \frac{S}{k}$	0.02	$u_1 = 0.01$		
	$u_{correction} = \frac{S}{2x\sqrt{3}}$	0.01	$u_2 = 0.00288$		
	$u_{W1} = \sqrt{u_{reference}{}^2 + u_{correction}{}^2}$			0.0104	
W_2	$u_{reference} = \frac{S}{k}$	0.02	$u_3 = 0.01$		
	$u_{correction} = \frac{S}{2x\sqrt{3}}$	0.01	$u_4 = 0.00288$		
	$u_{W_2} = \sqrt{u_{reference}{}^2 + u_{correction}{}^2}$			0.0104	
W_3	$u_{reference} = \frac{S}{k}$	0.02	$u_5 = 0.01$		
	$u_{correction} = \frac{S}{2x\sqrt{3}}$	0.01	$u_6 = 0.00288$		
	$u_{W_3} = \sqrt{u_{reference}{}^2 + u_{correction}{}^2}$			0.0104	
$r = W_2 - W_3$	$u_r = \sqrt{u_{W_2}{}^2 + u_{W_3}{}^2}$			0.0147	
$s = W_3 - W_1$	$u_s = \sqrt{u_{W_3}{}^2 + u_{W_1}{}^2}$			0.0147	
Standard error of least square, n=4	$u_{slop\ error} = \frac{\sigma}{\sqrt{4}}$			0.3740	
$w = \frac{100r}{s}$	$u_W = W\sqrt{\left(\frac{u_r}{r}\right)^2 + \left(\frac{u_s}{s}\right)^2 + \left(\frac{u_{slop\ error}}{W}\right)^2}$			0.3795	
U	$U = u_W . k$ *where k=2*	The expanded uncertainty is expressed as confidence level at 95%			0.76

6. Discussion

The calculation above yields liquid limit of the soil sample is (31.57 ± 0.76) %. The expanded uncertainty constitutes 2.4% of the reported liquid limit which is reasonable and acceptable.; however, there are precautions need to be observed.

The main equation is equation number (2); however, the equation is rewritten into equation (4) for convenience, where r is the water content and s represent the dry soil weight but the liquid limit does not arrive from this figure instead, it is an estimate from regression least square relation, equation (3) thus this result is an estimate not empirical information.

6.1. Uncertainty components

Equation (4) provides the basic equation to derive water content. Therefore, the relative uncertainty model becomes equation (5) and is rewritten into equation (6). This study adopted relative uncertainty model in favor of theoretical weighted combined standard uncertainty to avoid complication due to differentiation process and easily acceptable by general Malaysian laboratory personnel.

Equation (6) unveils 3 main uncertainty components, they are:

- Deviation caused by r, water content
- Deviation caused by s, dry soil weight, and
- Deviation caused by least square error.

6.2. Deviation caused by r

Water content, r, obtained from the difference of wet and dry weights of specimen, therefore the main causes for deviation are the balance and the correction factor because corrections were not compensated in the process of weighing. The whole process only used 1 balance thus the first component is the uncertainty reported in the calibration certificate. The other component is the correction reported in the calibration certificate too.

The uncertainty components for r thus as indicated in Table 1 consists of uncertainty due to reference (Balance) and correction which was not compensated while weighing wet specimen and dry specimen.

6.3. Deviation caused by s

Deviation for s, as indicated in Table 1 too, similar to r with no exception on weighing of dry and blank container weights. The combined standard uncertainty for s is similar to r as shown in Table 5.

6.4. Deviation caused by least square error

Liquid limit is obtained through 25^{th} blow derived from the transposed logarithmic regression curve. Thus, it is an estimation and affected by the curve fitting error. Table 3 shows the data to derive equation (4.1.1). The standard uncertainty is calculated from the error of the regression equation divided by square root of the number of points determined experimentally.

6.5. The influencing components

Notice that the standard uncertainty of both s and r are similar and only affected by the reference equipment, balance and the correction factors. Both values are Type B and can be obtained from the calibration certificate. They are not affected by weights and even the process of weighing. Note that residue of weighing does not come into picture because the weighing process is to weigh not to weigh to some limits to control the weight.

Another factor, least square error, a Type A component affects the result through curve fitting. Indirectly it is the result affected by the competence of the personnel. The data of this model shows curve fitting contributes the most to the uncertainty in Table 5 in fact it is the single largest component.

6.6. Values of r and s

The method to derive both r and s values are the key issues of this research. Liquid limit is an estimated figure derived from regression least square not from experiment thus the values of r and s cannot be ascertained through experiment; however, they can be derived through estimation as shown in Table 4. The value of r is estimated from the average of 4 wet specimen. Whereas the value of s is estimated through equation (3). This procedure manages to satisfy equation (6) hence derive the standard uncertainty of liquid limit.

6.7. Care and attention on estimated values of r and s and R^2

There are 2 main areas of care need much attention in order to achieve accuracy in the experiment. The average value of wet specimen comes from the 4 specimen and the range is controlled to 3.08g. Equation (3) shows there is no difference due to ratio constant; however, the weighing process still control to small range possible for the values are estimated and if possible, estimation should be based on small variation data.

Table 5 shows the most influential factor that is curve fitting error. This error comes from the experimental data of the 4-points water content corresponding to the number of blows; thus it is a good practice to monitor the R^2 value which is commonly adopted to check the quality of regression relation. Table 3 shows R^2 is controlled to 0.968 considered highly good fitting. The resultant effect is the expanded uncertainty constitute 2.40% of the liquid limit. The suggested value should not less than 0.95. The procedure to achieve 4 points water content may need to change to include more than 4 specimen from 6 or even 8 specimen and pick only 4 specimen having resultant R^2 nearest to 1 to improve curve fitting error.

6.8. Laboratory accreditation scheme usage

The R^2 curve fitting indication can be used to indicate the quality of the laboratory activity. The figure nearer to 1, the smaller the is curve fitting error and eventually resultant in smaller measurement uncertainty reported. This uncertainty figure can be used in proficiency testing and inter-laboratory comparison purpose.

7. Conclusion

The conventional approach to derive the measurement uncertainty may not be suitable for the Soil liquid limit indication as the value of limit is only an estimate, thus a different approach is needed to yield accurate and reliable result is of paramount importance. The Soil liquid limit by Casagrande method is derived from the estimate at 25^{th} blow correspond to the water content from logarithmic regression curve. The regression relation comes from the 4-points water content around 25^{th} blows. This study emphasizes estimating the weight of water content and the dry weight in a measurement uncertainty model. These components are then combined together using relative uncertainty model.

Curve fitting error is the key component in the estimation of soil liquid limit because it contributes to the largest amount. The model proposes control of the error through R^2 value that can be capitalized to improve quality of testing.

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