

DETERMINATION OF MEASUREMENT UNCERTAINTY IN A COMPLEX TESTING PROCESS FOR TEXTILE TESTS

Tibor GREGÁSZ, Veronika PÁL, Vera GÖNDÖR

Abstract: *All standards dealing with colorfastness testing of textiles avoid the issue of measurement uncertainty. However, this does not exempt accredited testing laboratories from the obligation to assess the factors of measurement uncertainty required by ISO/IEC 17025:2017. This is where the abstract text comes. This is a sample. This paper identifies and classifies the uncertainty components of the most frequent textile colorfastness testing procedures. As the test method comprises chemical, mechanical, and visual elements, the results are subject to complex uncertainty factors. Using experimental data, measurement results, and their statistical evaluation, we inquire into factors that allow defining a numerical uncertainty value for the respective test method. The result obtained allows continuous improvement of the procedure.*

Keywords: *measurement uncertainty, colour fastness testing, visual assessment, R&R*

INTRODUCTION

This research addresses the current and complex problem of determining measurement uncertainty in an accredited and designated textile laboratory. The research focuses on colour fastness testing, which has several different subtypes. As the subjective factor is significant in each test method, the measurement uncertainty is also difficult to quantify. For this reason, it has not yet been accurately defined and evaluated in similar laboratories. The ISO 17025:2017 standard places greater emphasis on the assessment of measurement uncertainty, the determination of its components, their evaluation, and, in the case of a complex method, its estimation as a general requirement of testing laboratories. [1] Currently, there is no method to adopt for this type of test method to provide measurement uncertainty. Our research will help the laboratory meet the requirements of laboratory accreditation, and for this test type, we will need to define sources of measurement uncertainty and quantify it using the appropriate analytical method. The purpose of the research is to support the textile testing laboratory in this topic by systematizing and evaluating the measurement uncertainty factors for the most common colour fastness tests. Furthermore, experimental quantification of the variance components of a particular test method will give us a specific uncertainty value.

PRINCIPLE AND STRUCTURE OF COLOR FASTNESS TEST METHODS

Throughout this research, we analyzed the six colorfastness tests most in demand: which are namely to water, perspiration, rubbing, baby saliva and perspiration, washing, and dry cleaning. The principle of the above tests is to subject the dyes or prints on fabrics or fibers to various chemical and/or mechanical stress simulations. Consequently, we can obtain two kinds of results in the so-compared to grayscales:

- classification of the specimen's change of color due to wet and chemically active medium,
- the intensity of the staining on the white adjacent fabrics attached to the specimen. (The latter is generally expected to be more relevant.)

With the use of the greyscale, the darkness of the adjacent fabrics is quantified by one of the values 1/2 on a scale of 5. Here, 1 is the worst (meaning the greatest darkness), and 5 is the best (meaning remaining white/unchanged), but half grades can be given. [2] [3] [4]



Figure 1: Part of the grayscale (grades 1-3) to assess staining (top) and colour change (bottom)

The steps of the test methods in question are illustrated by the following figure. The model of action used in each study depends on the type of exercise tested. For each specimen, either a standard single or multifibre (a special weave of six different components) adjacent fabric is attached, as required by the standard. For practical reasons, the former was used in the experiments of this study. The sample fixed between the adjacent fabrics is called a composite specimen.

The most sensitive part of colour fastness tests - thus, the most regulated part by standards - is the visual evaluation of pre-treated composite specimens. This is done under strict conditions in a 6500K colour temperature, special grey cabinet (colour viewing booth) with D65 sunlight to provide the observer with a neutral ("inert"), reflex-free, and always the same angle of sight. [2] [3]

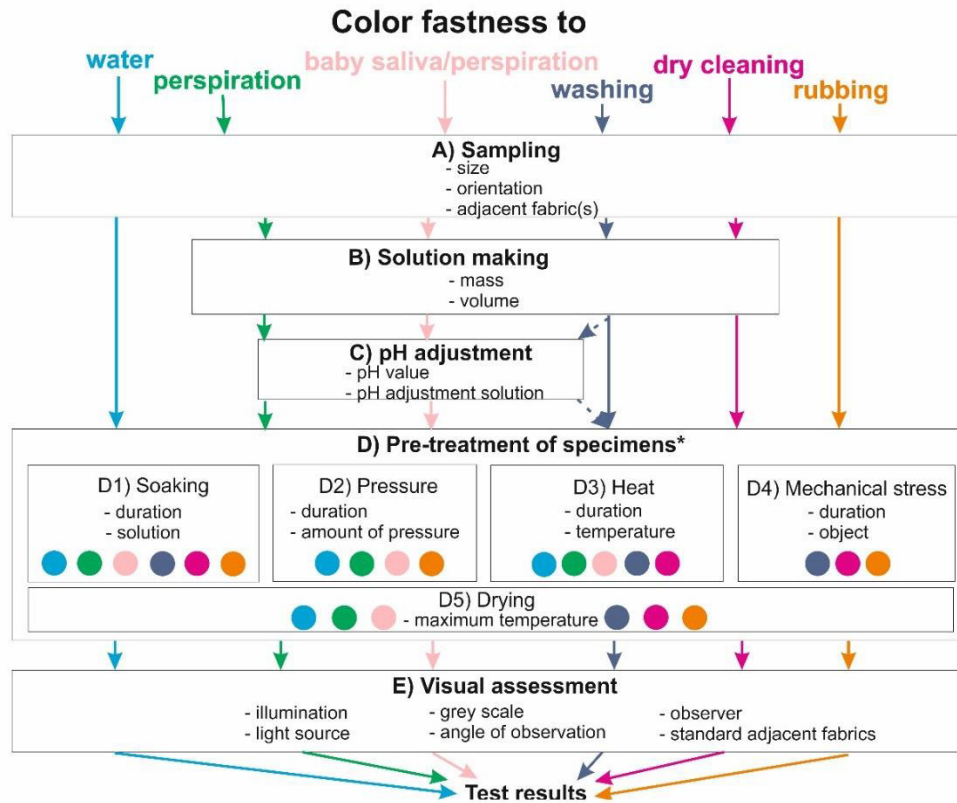


Figure 2: Process of six colour fastness tests and the most important parameters

INTERPRETATION OF MEASUREMENT UNCERTAINTY

It is acceptable even for laymen that a measurement result, due to its many influencing factors, can never be completely identical and will deviate from its true value; that is, it is loaded with an error. The phenomenon and this property of the measurement procedures are, according to the most commonly used wording: "parameter, associated with the result of a measurement that characterizes the dispersion of the values that could reasonably be attributed to the measurand." It can be given by the variation characteristic calculated from the measurement results (Type A determination) or by other scientifically valid information (Type B determination). [6] [7] [8]

It can be expressed as an accurate numerical value using various statistical features; in a direct, "one-dimensional" measurement where we want a relatively easy-to-measure parameter (such as weight measurement), the results obtained are called repeatability standard deviation can also be given as "s." This can be considered the same as the "standard uncertainty," which is called "u" (u - uncertainty). Another solution is the so-called "extended uncertainty" U, for which

$$U = k \cdot u \quad (1)$$

Where "k" being the coverage factor, 2 or 3 times the standard deviation is used to extend it to the standard 95-99% statistical coverage. Things become more complicated when we deduce the value we need from other measurement results in an indirect way (e.g., using a formula).

One of the possible ways to do this is a Type B determination. The situation is even more complex in the case of colour fastness tests because the response of a given specimen to the pre-treatment procedure can be obtained only once – which essentially means that this is a destructive test method. Naturally, we can make more specimens, but in this case, we immediately suspect that our measured characteristics are also influenced by the inequalities of the manufacturing process. Which makes it difficult to tell whether to what extent our own measurement process is responsible for uncertainty. And when the testing process is made up of many different, more or less controlled parameters, one cannot really neglect to carefully analyse how much these parameters can contribute to the swings of our measurement results.

In our case, we use Type B to determine measurement uncertainty because, after estimating uncertainties ($U_A, U_B, \dots U_E$) that cumulate during the test, they are considered as variance components and summed to determine the combined uncertainty (U). Context to determine combined uncertainty indexed by letter of test phases (A-E)

$$U_{combined} = \sqrt{(U_A^2 + U_B^2 + \dots + U_E^2)} \quad (2)$$

AN ALTERNATIVE WAY TO DEFINE MEASUREMENT UNCERTAINTY FOR COLOUR FASTNESS TESTING

In case of lacking scientific curiosity to determine the value of measurement uncertainty specifically for our research and our laboratory, or urgently need to provide an uncertainty value for a given test method, a less resource-intensive process is available due to international round-robin tests. Laboratories accredited for the tests in question shall participate in interlaboratory round-robin tests several times a year. The organizing institute provides the same sample (which is made of 100% wool in the case of this example) and then evaluates the submitted results. The statistical characteristics of the results submitted for a given test method (in this case, the colour fastness to perspiration) are evaluated by the type of adjacent fabrics and type of solution. After averaging the standard deviations of the more than 320 results classified by the adjacent fabric used, the overall value of standard deviation is 0,535. This can be converted into expanded uncertainty using Formula 3, resulting in uncertainty of 1,07 scale grades, with a 95% confidence interval. [9] As it is expected, this result would show a higher value than the value which is to be defined experimentally through the next chapters, specifically for our own laboratory. Naturally, as the total number of uncertainties in the 61 laboratories carrying out the round-robin test accumulates in this numeric value, in which different types of test devices, grey scales, adjacent fabrics, and laboratory staff may play a significant role. This makes the "responsibility" of our own measurement system very difficult to distinguish and define in terms of numerical values. Therefore, if appropriate resources are available, it may be more useful to quantify the value of our own system and keep track of changes in order to continuously improve the work of the laboratory.

SYSTEMATIZATION OF UNCERTAINTY COMPONENTS

To determine measurement uncertainty, we have broken down the process of colour fastness tests leading up to the measurement result as shown in Figure 2, since during these steps, the measurement result may be distorted, and the groups of measurement uncertainty components can be identified at these points. These factors will be included or omitted in our model of calculation, depending on their degree of relevance. The table in the annex summarizes the test parameters to be set up for the process. The sources of uncertainty in a particular test phase will also be these parameters. In the columns of the table, we classified the tracked parameters according to the different kinds of elements of the measurement system, and the colour codes indicate the classification of the factors in the critical analysis. This fully complies with paragraph 7.6.1 of ISO 17025:2017, which is one of the objectives of the research. [1] In addition to accurately monitoring the uncertainty parameters, the table provides the opportunity to illustrate the proportions of the factors based on the three pillars of the measurement system (i.e., calibrated and regularly checked measurement instruments, accredited suppliers, and proficiency of operators). Thus, in the event of a change of any of these factors, the table provides transparency in the exploration of causation and in the management of risks arising from measurement uncertainty.

DESIGN AND IMPLEMENTATION OF EXPERIMENTS ON CRITICAL FACTORS

Uncertainties in a particular process phase (see Figure 2) are that the error in the setting of a given parameter will also be a probability variable (temperature, duration of pre-treatment, the concentration of solutions, quantities added, amount of solution absorbed by the textile material, etc.). These random variations during the test process can escalate into smaller or larger differences in staining on the composite specimens when the results are compared to the greyscale. This is why the uncertainty of the final result is interpreted as a sum, that is, the sum of the uncertainties (as components of variance) generated in the previous steps of the process. (see Formula 2 – Figure 3)

In the course of our work, we had to examine these components of uncertainty about how they may increase uncertainty values. First of all, the laboratory's council of metrology examined at least critically – according to good engineering judgment – whether this factor could change at all and whether it could influence the results significantly or they can be logically excluded. At the meeting, we planned experiments on the suspected influencer factors. Where the practice or expert opinion is considered significant, we need to perform an experimental analysis.

- The process begins with sampling, where we have already found that, due to the inhomogeneity of the specimens obtained from the fabric, its ability to bind and release dye, as well as the resulting color change and staining, can be significant. Therefore, we decided to design an experiment for sampling differences between samples in the degree of uncertainty in scale grades.

- The pH range of the chemical content of sweat solutions to be prepared as standard is ± 0.2 , which can be reliably adjusted on the indicator paper's colour scale, so the uncertainty of this factor is considered to be negligible and is not yet analyzed.
- Visual assessment on the pre-treated and dried samples based on the greyscale, is considered to be a significant component due to its key role.

Sampling uncertainty

The standard deviation of this factor was estimated from the differences (as ranges) between the results of the pairs of specimens treated with exactly the same treatment, and then the value of "U" was given. A total of 75 completely identical composite specimen pairs were subjected to fully standardized treatment. After averaging the ranges between the results of the pairs of specimens, the standard deviation was estimated by the following formula:

$$\hat{s}_{sampling} = \frac{\bar{R}}{d_2} = \frac{0,15}{1,128} = 0,133 \quad (3)$$

where the actual value of the statistical factor d_2 is 1,128. Based on this, the extended measurement uncertainty inherent in the sampling procedure is obtained using formula 4, by using the ranges between the results of two identical samples treated with the same procedure.

$$U = k \cdot s \quad (4)$$

This gives us the expanded measurement uncertainty for a 95% confidence interval (where $k=2$): $U_{sampling95} = 0,266$ grades.

This verifies our idea that even with the highest degree of control over the testing process, the result may be significantly affected by the unevenness of the manufacturing of textiles. However, it is worth mentioning that as this value completely falls outside the control of the laboratory and the test method itself, including it within the final expanded uncertainty value could be arguable and debatable.

The uncertainty caused by visual assessment of staining - Gage R&R method

R&R analysis helps quantify the repeatability and reproducibility of measurement systems originally applied by automotive suppliers. Repeatability is the measure of the difference within a measurement sequence expressed in standard deviations. This can give you the difference in measurement results over a short period of time, leaving all elements of the measurement system intact. Reproducibility defines the vital differences between measurement sequences created in different circumstances (e.g., measuring person, instrument, location), also expressed in standard deviation. [10] Our aim is to quantify the accuracy with which the "measuring instruments" created by the eyes, experience, and judgment of operators can work together with the specific round-robin test methodology adopted by the automotive industry. After drying, the pre-treated specimens are visually evaluated using a nine-level greyscale (graded from 1 to 5 in half degrees) with colour differences as defined in ISO 105-A03. We compare the

difference between the standard adjacent fabric and the pre-treated specimen's staining with the shades shown in *Figure 1*.

We started with 12 samples of each of the 6 most common adjacent fabrics (cotton, wool, polyester, polyamide, viscose, and cotton used for testing fastness to rubbing). Each sample was graded 3 times with 3 operators, in random order to avoid the memory effect. Taking into account the results of all three operators on all types of adjacent fabrics, the repeatability of the measuring system (estimated standard deviation from the mean value of the ranges taken per operator) was 0,593 scale grades, its reproducibility (the standard deviation estimated from the mean result values of operators) was 0,275; thus **the R&R standard deviation value counts 0,654 grades of scale**. The latter value can also be used as a factor to estimate measurement uncertainty. Where there is a difference of opinion between two operators' results is the question of reproducibility; when one is not consistent with one's own results, it is a question of repeatability. It is clear from our numerical values obtained during the examination that the latter factor, in this case, contributes more than twice as much to the measurement uncertainty as to the former.

Since the color differences for each grade are non-linear, there is a much smaller, less perceptible difference between the intensities of staining at the better grades than the worse ones. In the future, it may be interesting to determine the varying detection accuracy. It would also be important to study this topic further because, in reality, the focus is not on the measurement results given by the grades but on whether the sample qualifies for a given certification, i.e., fails or passes. The average (reproducibility) and range (repeatability) control charts (well known from SPC and Gage R&R) show the overall uncertainty of the measuring system, as shown in the following figures. The horizontal axis shows the mean of the samples, while the vertical axis shows the average of repeated measurements and the ranges between such measurements.

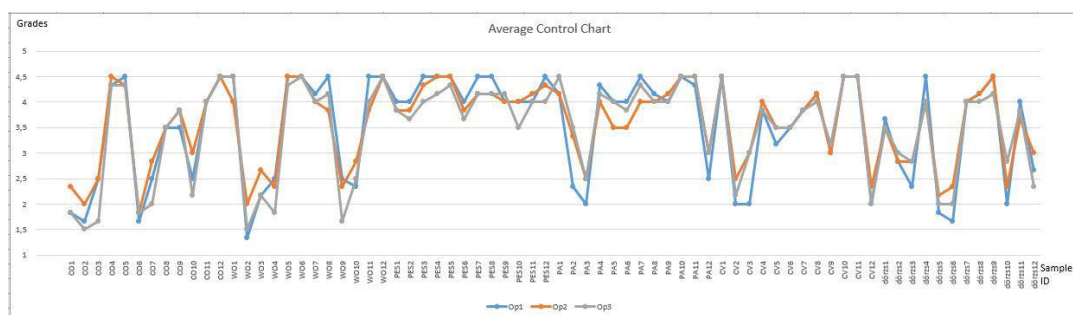


Figure 3: The "average control chart" to illustrate reproducibility

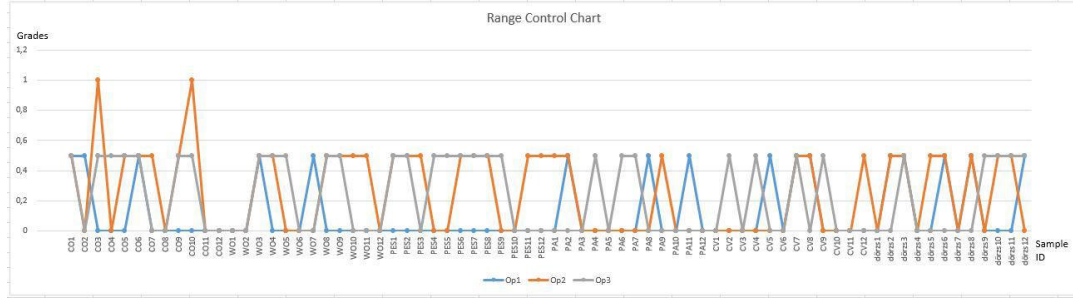


Figure 4: The “range control chart” to illustrate repeatability

DETERMINATION OF EXPANDED MEASUREMENT UNCERTAINTY

The combined standard uncertainty can be calculated using Formula 2:

$$U_{combined} = \sqrt{(U_A^2 + U_B^2 + \dots + U_E^2)} \quad (2)$$

As there are only two uncertainty values were judged statistically significant during the experiments and left in the model, the combined expanded uncertainty can be represented by the following formula:

$$U = \sqrt{U_{sampling}^2 + U_{visual\ assessment}^2}; \quad U = \sqrt{0,266^2 + 0,654^2} = 0,706 \text{ scale grades.} \quad (5)$$

We can state accordingly that the extended measurement uncertainty of colour fastness test procedures to water and perspiration in this laboratory counts 0,706 grades. Due to the high degree of similarity between the selected and examined factors, we can apply the established value to all colorfastness test methods in which we would be able to exclude any other factor of uncertainty except for sampling and visual assessment. As a further step in its development, the Council of Metrology has decided that the next step is to determine the sampling uncertainty of the colour fastness test to rubbing and to provide a more accurate uncertainty value based on the indicators of the special adjacent fabric used for rubbing.

SUMMARY

As mentioned above, currently, there is no systematic procedure to apply for measuring uncertainty for colour fastness tests, so in order to improve the method, our research has determined a measurement uncertainty that most accurately reflects the random variations caused by local conditions and techniques. According to the method now developed, we will identify uncertainty factors for further investigations, decide on their relevancy, calculate local uncertainty results as a component of variance, and consider them as a starting parameter for subsequent developments of the test method.

Color fastness to perspiration	Measuring instrument	Environment	Method	Measurand object		Operator
A) Sampling				size of specimen	(40±2) mm x (100±2) mm	** It is the responsibility of the operator to monitor
				adjacent fabric	in accordance with fiber	Legend:
B) Solution making	Standard adjacent fabrics			ingredients of alkaline solution (l):	0,5 g L-histidine 5 g sodium-chloride 2,5 g disodium-hydrogen ortophosphate-	could be a source of uncertainty
	graduated cylinder			ingredients of acidic solution (l):	0,5 g L-histidine 5 g sodium-chloride 2,2 g sodium-dihydrogen ortophosphate-	Guaranteed by the regular calibration of the measuring instrument/device
C) pH adjustment	pH test paper			alkaline	pH 8 (±0,2)	ensured by the
	0,1 mol/l sodium-hydroxide-solution			acidic solution	pH 5,5 (±0,2)	purchased from an accredited source/supplier
D) Pre-treatment of specimens						trivial capability
D1) Soaking		temperature	room	duration	30 minutes	object of measureme
	glass rod	removal of excess liquor	liquor ratio	approx. 50:1	moving of the specimen	until evenly wetted
D2-D3) Pressure and thermal treatment	perspirator	temperature	(37±2) °C	duration	4 h	
	glass/acrylic sheet	pressure	(12,5±0,9)			
	drying oven					
D5) Drying	clothespin					
	drying oven	temperature	max. 60 °C	suspension	contact of layers	
E) Visual assessment	colorimeter OR	source of light	D65	angle of observation	approx. 90 °	
	the naked eye and standard adjacent	illumination	min. 600			color change of staining on the
* - Parameters defined by the standards but unusual and/or exceptional are not covered in this table; it is only intended to provide a general overview of the uncertainty components that occur during the testing process.						

Figure 4: Table of uncertainty factors for colour fastness to perspiration [5]

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