

## UNDERSTANDING AND LEARNING TO CALCULATE REPEATABILITY AND REPRODUCIBILITY USING ASTM D6300



### Introduction

**In the world of petroleum and lubricant testing, few qualities are as important as precision. Precision reflects the reliability of a procedure to produce consistent results under set conditions, and two key elements of precision are repeatability (r) and reproducibility (R). Repeatability refers to the variability of multiple test results by an individual analyst using a single apparatus to test replicate specimens from a single sample. Reproducibility measures the variability of test results among different analyses, each using a different apparatus, when testing specimens from sub-samples of a sample that have been shipped to multiple laboratories.**

Both types of variability are crucial for understanding the confidence limits for a test method and minimizing the risk of incorrectly assessing a property to either be out of specification when it is actually in specification (known as a Type I error) or, conversely, assessing the property to be in specification when it is actually not (known as a Type II error). These values then inform precision, which can establish the expected resolution between two values such that researchers performing condition monitoring tests can minimize the risk of either type of error. With an established range, researchers can more confidently test the results of an experiment and gauge whether the process itself is operating within specified parameters.

To numerically determine these parameters, ASTM International developed the ASTM D6300 standard, which provides guidelines for determining precision and bias for test methods involving petroleum products, liquid fuels, and lubricants<sup>1</sup>. ASTM D6300 has become an indispensable tool for gauging variability in test results. The formulas for calculating repeatability and reproducibility are of great importance to researchers in this field. This paper covers the calculation methods for both values, as outlined in the ASTM D6300 standard, explains the rationale behind key steps and provides a framework for manual calculation of repeatability (r) and reproducibility (R).

### The Significance of Repeatability and Reproducibility

The importance of repeatability and reproducibility should be reinforced significantly. While the two values do not govern the outcome of a procedure, they act as measures to examine the variability of a testing method. Repeatability and reproducibility allow analyses to establish tolerance thresholds by indicating when two replicate test results are and are not "within normal expectation," with a standard expectation cutoff representing 95% of the reference distribution for the "absolute difference" variable (2). This is necessary for researchers performing condition monitoring tests, as an observable measure of replicability is always welcome for applications machines can automate, as mechanisms performing outside of acceptable error ranges are typically in need of maintenance and inspection.

Without the ability to gauge the irregularity of the output, researchers would be venturing blindly into a maze of Type I and Type II errors, unable to gauge whether the machine is operating inside or outside specified ranges and thus unqualified to determine whether an apparatus is working or malfunctioning. This would make system maintenance a nightmare, as without a basis to gauge the expected range of products, it would be much more difficult to determine if a mechanism requires maintenance just by observing its output. This drastically increases maintenance time

and cost in factories, since each machine would then require manual inspection for errors at regular intervals to avoid the risk of catastrophic failure. With the application of repeatability and reproducibility, researchers can simply observe the precision of a baseline machine and compare it to the output of the machines under observation to gauge whether they are within acceptable margins.

### Preparing Samples for Calculations:

Before properly beginning calculations, we must prepare a valid and informative data set for the experiment. According to standard practice, one would prepare enough material to supply at least 10% more than necessary for each laboratory involved in the experiment<sup>2</sup>. Each unit will be labelled with a letter for the material and a sequential number. For instance, for 10 laboratories and 2 test results of a material B, the test samples would be labeled B1 through B22<sup>3</sup>. The samples themselves should include specimens in which the measured property is just above the detection limit, within the detection range, and just below the maximum measurable value without dilution.

While other precision metrics are tested and calculated within the experiment, this paper will focus solely on the formulas related to the calculation of repeatability and reproducibility.

### Formula for Repeatability (r):

Of the various measures of accuracy, the fundamental precision statistics are Repeatability Standard Deviation ( $S_r$ ) and Reproducibility Standard Deviation ( $S_R$ ). These two values are central to calculating repeatability and reproducibility of the subject, respectively. Repeatability concerns the results of multiple analyses by a single operator on specimens from a single sample. The expected data set will contain one or more samples undergoing multiple trials of the same procedure across multiple labs. As the calculations for reproducibility require repeatability, repeatability will be calculated first.

First, we must calculate the mean of test results from a single sample as tested by a single analyst. The formulas for computing the mean and standard deviation (S) of replicate tests are:

$$\text{Mean (A)} = \frac{1}{n} \sum_{i=1}^n a_i. \quad n = \text{number of trials, } a = \text{value.}$$

This gives us the average value of the sample within the relevant data set. With the mean, we can calculate the standard deviation of that sample with the next formula:

$$\text{Standard Deviation (S)} = \sqrt{\frac{\sum_{i=1}^n (a_i - A)^2}{n - 1}}$$

The standard deviation indicates how dispersed the data is relative to the mean. This serves as a general indicator of how precise the data is before any further calculations. We will then calculate the standard deviation for the same sample in each of the remaining labs in the data set using the same formulas above. With that set of standard deviations, we will calculate the repeatability standard deviation ( $S_r$ ) pooled over all labs using the following formula:

$$\text{Repeatability Standard Deviation (S}_r\text{)} = \sqrt{\frac{\sum_{i=1}^p (S_i)^2}{p}} \quad p = \text{number of labs}$$

Repeatability standard deviation is the standard deviation of the given test under repeatability conditions. However, this value does not account for 95% confidence. This will be rectified in the next step. This value will be further refined in the next step.

With  $S_r$  calculated, we then multiply the answer by 2.8 to calculate repeatability ( $r$ ). This is to account for 95% confidence, as 1.96 standard deviations multiplied by  $\sqrt{2}$  is roughly equivalent to 2.8. We now must account for the "normal expectation" cutoff, which falls within the 95th percentile, or 95% of all data. To do so, we multiply the repeatability standard deviation by 2.8, which brings the value within those bounds and thus provides our value for repeatability.

$$\text{Repeatability } (r) = S_r * 2.8$$

With this procedure, you can now calculate repeatability for each of the samples in the data set by simply inputting the data from that specific sample.

### Formula for Reproducibility (R):

Reproducibility is found after repeatability, as Repeatability Standard Deviation is required for calculations. First, we calculate the mean of a sample in each lab within the set, as well as the total average of all the sample data across all labs ( $\bar{A}$ ). With the means, we calculate the Standard Deviation of the Averages ( $S_A$ ) using the following formula:

$$\text{Standard Deviation of Averages } (S_A) = \sqrt{\frac{\sum_{i=1}^p (A_i - \bar{A})^2}{p - 1}}$$

This serves as a measure of spread across multiple labs using the average values as a baseline, and we can use it to further extrapolate precision results across labs. With  $S_A$  calculated, we then determine the Reproducibility Standard Deviation ( $S_R$ ) using the following formula:

$$\text{Reproducibility Standard Deviation } (S_R) = \sqrt{(S_A)^2 + [(S_r)^2 * (\frac{n-1}{n})]}$$

Just like Repeatability Standard Definition, this is the standard deviation of the test results obtained under reproducibility conditions.

Again, this value is multiplied by 2.8 to account for normal expectation cutoffs, leaving us with this formula for reproducibility.

$$\text{Reproducibility } (R) = S_R * 2.8$$

Like with repeatability, you can repeat this sequence to calculate the reproducibility value of other samples in the set.

### Example Calculations

To demonstrate these calculations, we generated a series of random numbers. In these sample calculations, we generated 10 different samples, labelled A through J, and 5 different labs, with 6 trials per lab. The test data for the first sample is shown below, with the calculated average and standard deviation per lab set.

Lab #	Trial #	Sample A	Lab Average	Lab Standard Deviation
Lab 1	Trial 1	89.1	87.41667	1.596768
	Trial 2	87.9		
	Trial 3	86.6		
	Trial 4	84.6		
	Trial 5	88.1		
	Trial 6	88.2		
Lab 2	Trial 1	87.3	86.55	1.703819
	Trial 2	85.3		
	Trial 3	84.3		
	Trial 4	88.6		
	Trial 5	85.7		
	Trial 6	88.1		
Lab 3	Trial 1	85.2	85.63333	1.910672
	Trial 2	83.1		
	Trial 3	85.2		
	Trial 4	86.8		
	Trial 5	88.7		
	Trial 6	84.8		
Lab 4	Trial 1	89.3	85.15	3.46742
	Trial 2	89.5		
Lab 5	Trial 3	82.8	84.81667	3.321696
	Trial 4	84.7		
	Trial 5	83.3		
	Trial 6	81.3		
	Trial 1	82.7		
	Trial 2	88		
Trial 3	80.9			
Trial 4	87.5			
Trial 5	87.9			
Trial 6	81.9			

The repeatability ( $r$ ) of Sample A is calculated below:

$$S_r = \sqrt{\frac{\sum_{i=1}^5 (S_i)^2}{5}} = \sqrt{\frac{(1.596768)^2 + (1.703819)^2 + (1.910672)^2 + (3.46742)^2 + (3.321696)^2}{5}}$$

$$S_r = \sqrt{\frac{32.16}{5}} = \sqrt{6.432} \approx 2.536$$

$$\text{Repeatability } (r) = S_r * 2.8 = 2.536 * 2.8 \approx 7.101$$

The Repeatability of Sample A is roughly equal to 7.101. For repeatability, a lower value is preferred, as numerical measures of variability act as inverse measures of precision. High precision would indicate that repeatability is very low.

The reproducibility ( $R$ ) of Sample A is calculated below:

$$\text{Total Average } (\bar{A}) = \frac{87.41667 + 86.55 + 85.63333 + 85.15 + 84.81667}{5} = \frac{429.57164}{5}$$

$$\approx 85.91333$$

$$S_A = \sqrt{\frac{\sum_{i=1}^p (A_i - \bar{A})^2}{p - 1}} = \sqrt{\frac{(1.51)^2 + (0.64)^2 + (-0.28)^2 + (-0.76)^2 + (-1.09)^2}{4}} \approx 1.064$$

$$S_R = \sqrt{(S_A)^2 + [(S_r)^2 * (\frac{n-1}{n})]} = \sqrt{(1.064)^2 + [(2.536)^2 * (\frac{5}{6})]} \approx 2.548$$

$$\text{Reproducibility } (R) = S_R * 2.8 = 2.548 * 2.8 \approx 7.134$$

The Reproducibility of Sample A is roughly equal to 7.134. Again, reproducibility is a measure of variability, with lower values indicating higher precision.

### Conclusion

ASTM D6300 offers an accurate method to determine both repeatability and reproducibility, which are both invaluable to the reliability and production efficiency of many procedures and production lines. With the formula outlined in this report, the repeatability and replicability values can be calculated by hand, saving valuable time and money from manufacturing errors and benefitting the field of petroleum and lubricant testing.

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**Mr. Beau Eng** is part of a thriving internship program at Koehler Instrument company in Holtsville and is a student of Chemical and Molecular Engineering at Stony Brook University, Stony Brook, New York where Dr. Shah is on the external advisory board of directors.

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