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Research Report: E28-1046

Interlaboratory Study to Establish Precision Statements for ASTM E1450, Standard Test Method for Tension Testing of Structural Alloys in Liquid Helium

Technical contacts:
Dr. William Luecke, Jolene Splett, and David McColskey
National Institute of Standards and Technology
Gaithersburg, MD 20899
U.S.A.
(301) 975-5744
William.luecke@nist.gov

ASTM International
100 Barr Harbor Drive
West Conshohocken, PA 19428-2959

1. Introduction:

An Interlaboratory Study was conducted to establish precision statements for E1450-09, Standard Test Method for Tension Testing of Structural Alloys in Liquid Helium.

2. Test Method:

The Test Method used for this ILS is E1450-09. To obtain a copy of E1450-09, go to ASTM's website, www.astm.org, or contact ASTM Customer Service by phone at 610-832-9585 (8:30 a.m. - 4:30 p.m. Eastern U.S. Standard Time, Monday through Friday) or by email at service@astm.org.

3. Study Information:

Six laboratories completed tensile measurements for test material SUS 316LN according to a protocol that was standardized as E1450. The material was selected to correspond to previous gage repeatability and reproducibility tests. The data, study participants, material and specimen fabrication, and equipment/apparatus are described in [1].

4. Statistical Data Summary and Analysis:

We computed repeatability and reproducibility for: tensile strength, yield strength, elongation at fracture, and reduction of area. We used ASTM E691-14 as a basis for analysis, however, the technique used in ASTM E691-14 requires that each laboratory produce the same number of measurements for each material. Since the six laboratories in the current study did not produce the same number of measurements, we used a restricted maximum likelihood method to estimate the repeatability and reproducibility standard deviations.

Because tensile testing is destructive, no repeat measurements are available. However, we assume that all samples in the study are homogeneous and can be treated as repeat measurements *of the same sample* so that a random-effects model can be used to compute variance components.

Note: Maximum likelihood estimation is a statistical technique that selects parameter values which maximize the likelihood function. For this study, the parameters of interest are variance components associated with a one-way random effects model. (The analysis in ASTM E691-14 is also based on a random effects model for the case where laboratory sample sizes are equal.) More information regarding maximum likelihood estimation can be found in [2]. Analysis of variance and random effects models are discussed in [3].

4.1 Tensile Strength

Data are available for six laboratories and one material. According to ASTM E691-14, six is the minimum number of laboratories that can be used to estimate repeatability and reproducibility standard deviations. The raw tensile strength data and summary statistics (average, standard deviation, and between-laboratory consistency, h, as defined in ASTM E691-14) for each laboratory are listed in Table 1.

Table 1. Tensile strength raw data and summary statistics for each laboratory.

Lab ID	Test Results (MPa)	$\bar{x}$ (MPa)	s (MPa)	h
H	1763, 1768, 1726, 1755	1753	19	1.37
I	1674, 1677, 1690, 1680, 1680, 1691, 1693, 1680	1683	7	-1.16
J	1706, 1701, 1721, 1691	1705	13	-0.38
K	1733, 1732, 1751, 1739, 1732	1737	8	0.80
L	1685, 1693	1689	6	-0.95
M	1710, 1730, 1730, 1730, 1720	1724	9	0.32

In a typical interlaboratory study, k statistics are used to demonstrate the consistency of the test method within a laboratory. Since we only have measurements for one material, SUS 316LN, the k consistency statistics will not be computed.

The raw tensile strength measurements are plotted in Figure 1. The average and ± 1 standard deviation (s) reference lines based on $n = 28$ measurements are also shown on the plot. The reference lines were calculated using the values in Table 1 and do not correspond to precision statistics.

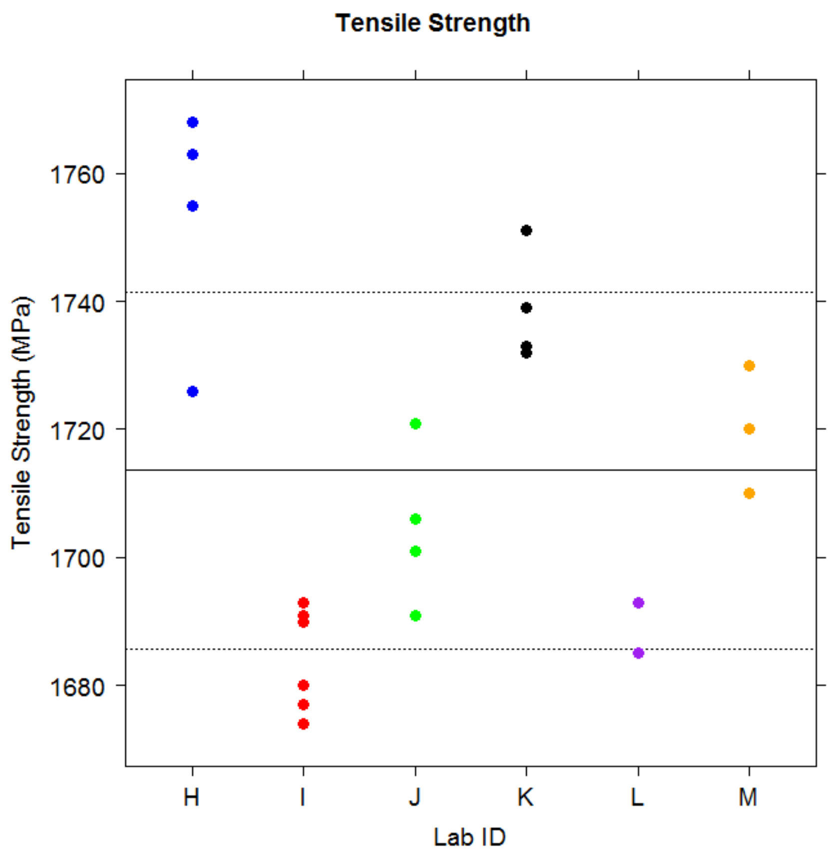

Figure 1. Tensile strength data from Table 1. The solid horizontal line represents the overall mean, and the dashed lines represent $\pm 1s$ of the measurements.

The *h* statistics, indicating between-laboratory consistency, are plotted in Figure 2 for each laboratory. The horizontal lines at -1.92 and 1.92 represent the critical value associated with the 0.5 % significance level and six participating laboratories.

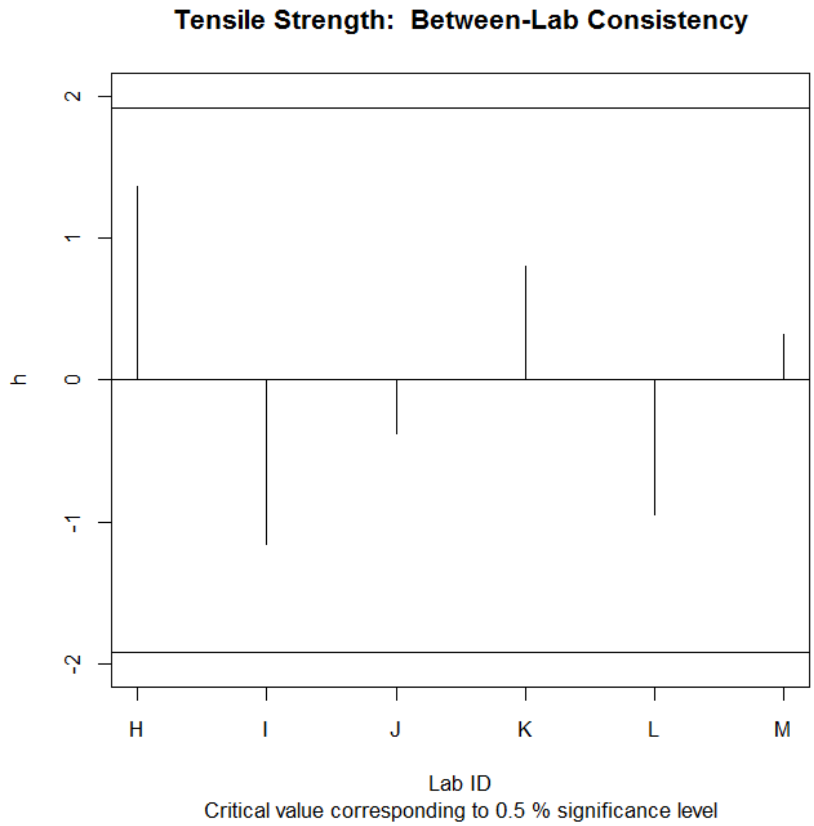

Figure 2. Tensile strength h consistency statistics for material SUS 316LN.

Figure 2 shows strong evidence that the measurements are consistent among laboratories, so the precision statistics can be calculated (Table 5) based on all the reported data.

The results of similar analyses performed for yield strength, elongation at fracture, and reduction of area follow.

4.2 Yield Strength

The raw yield strength measurements are listed in Table 2 and are plotted in Figure 3. The average and $\pm 1s$ reference lines based on $n = 26$ measurements are also shown on the plot. The reference lines were calculated using the values in Table 2 and do not correspond to precision statistics.

Table 2. Yield strength raw data and summary statistics for each laboratory.

Lab ID	Test Results (MPa)	$\bar{x}$ (MPa)	s (MPa)	h
H	1075, 1096, 1059	1077	19	0.90
I	1048, 1045, 1062, 1054, 1046, 1046, 1043	1049	7	-1.55
J	1059, 1049, 1059, 1059	1057	5	-0.89
K	1090, 1073, 1065, 1075, 1060	1073	11	0.53
L	1071, 1066	1069	4	0.17
M	1070, 1070, 1060, 1090, 1090	1076	13	0.84

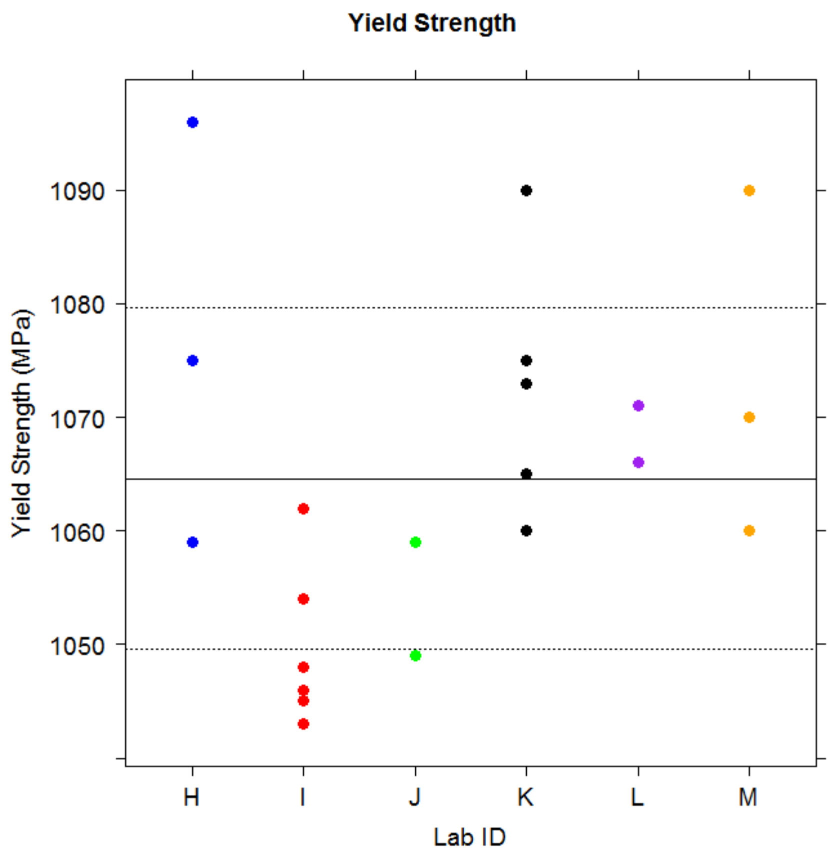

Figure 3. Yield strength data from Table 2. The solid horizontal line represents the overall mean, and the dashed lines represent $\pm 1s$ of the measurements.

The h statistics, indicating between-laboratory consistency, are plotted in Figure 4 for each laboratory. The horizontal lines at -1.92 and 1.92 represent the critical value associated with the 0.5 % significance level and six participating laboratories.

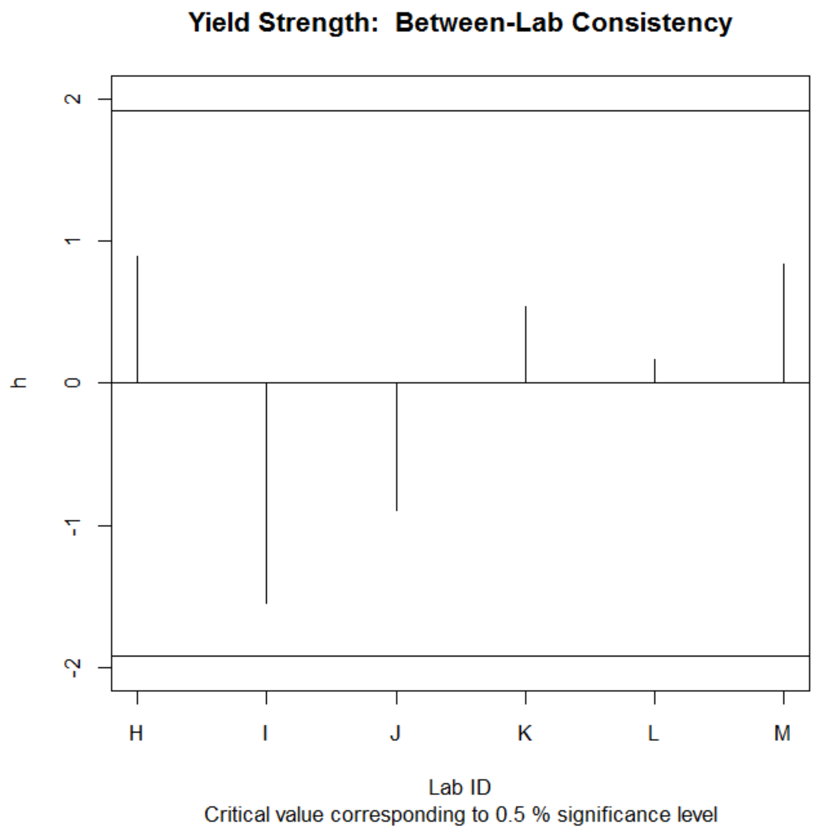

Figure 4. Yield strength h consistency statistics for material SUS 316LN.

Figure 4 shows strong evidence that the measurements are consistent among laboratories, so the precision statistics can be calculated (Table 5) based on all the reported data.

4.3 Elongation at Fracture

The raw elongation at fracture measurements are listed in Table 3 and are plotted in Figure 5. The average and $\pm 1s$ reference lines based on $n = 26$ measurements are also shown on the plot. The reference lines were calculated using the values in Table 2 and do not correspond to precision statistics.

Table 3. Elongation at fracture raw data and summary statistics for each laboratory.

Lab ID	**Test Results (%)**	$\bar{x}$ (%)	***s*** (%)	***h***
H	47.4, 47.8, 46.0, 46.3	46.9	0.9	-1.04
I	45.7, 48.0, 45.7, 50.0, 51.0, 49.0, 48.0, 47.0	48.1	1.9	-0.34
J	49.6, 50.0, 48.5, 50.0	49.5	0.7	0.54
K	46.3, 46.7, 48.5, 46.2, 47.3	47.0	0.9	-0.96
L	51.4, 51.2	51.3	0.1	1.60
M	46.9, 46.0, 51.1, 51.1, 49.7	49.0	2.4	0.20

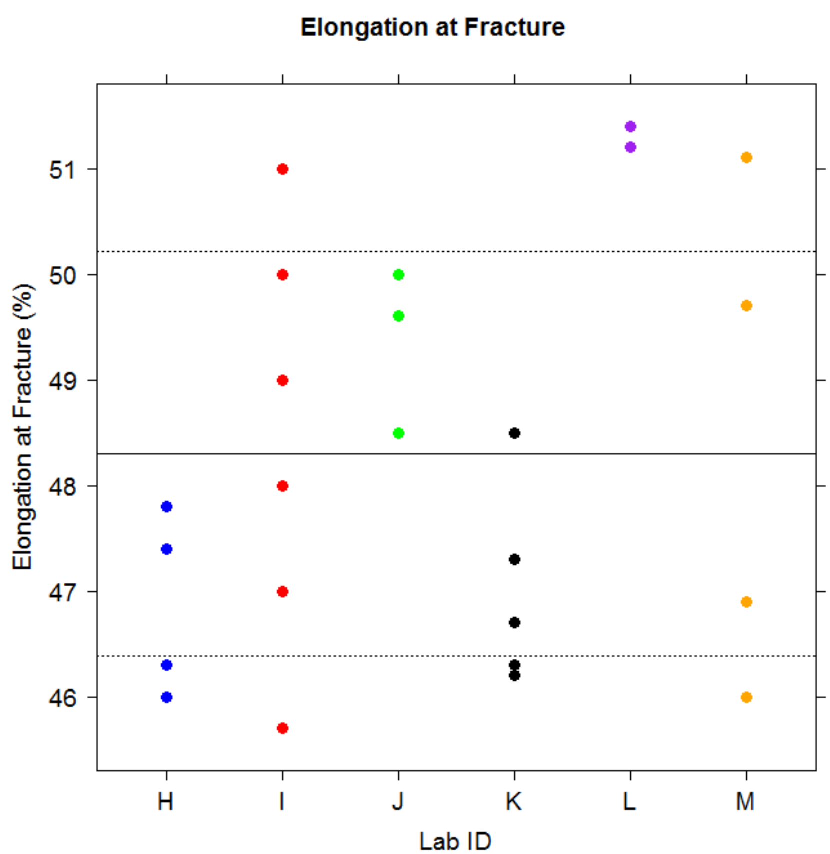

Figure 5. Elongation at fracture data from Table 3. The solid horizontal line represents the overall mean, and the dashed lines represent $\pm 1s$ of the measurements.

The h statistics, indicating between-laboratory consistency, are plotted in Figure 6 for each laboratory. The horizontal lines at -1.92 and 1.92 represent the critical value associated with the 0.5 % significance level and six participating laboratories.

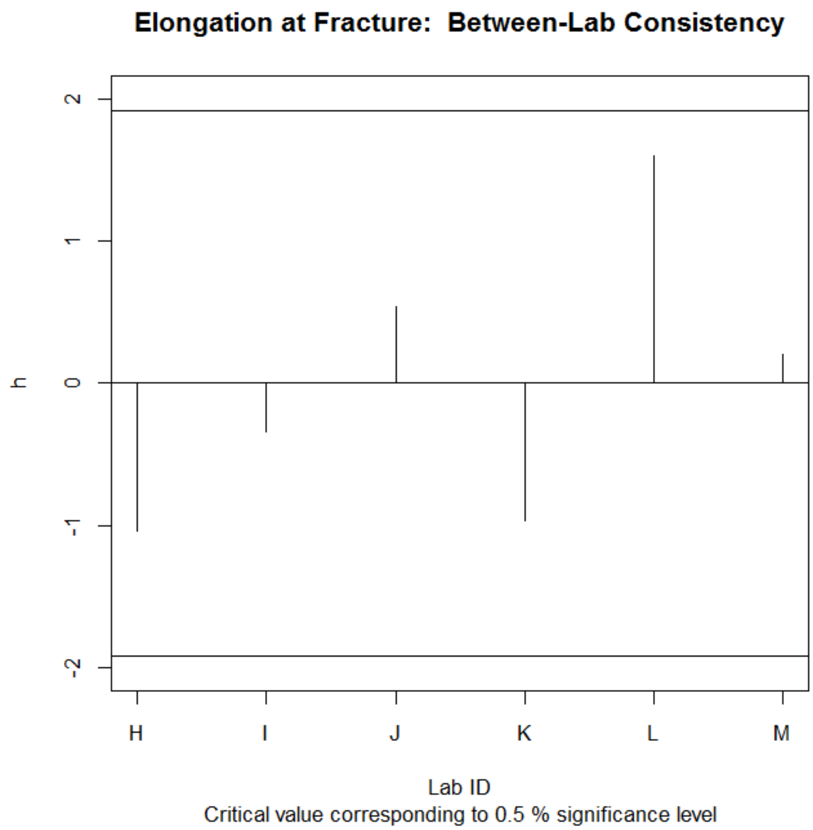

Figure 6. Elongation at fracture h consistency statistics for material SUS 316LN.

Figure 6 shows strong evidence that the measurements are consistent among laboratories, so the precision statistics can be calculated (Table 5) based on all the reported data.

4.4 Reduction of Area

The raw reduction of area measurements are listed in Table 4 and are plotted in Figure 7. The average and $\pm 1s$ reference lines based on $n = 23$ measurements are also shown on the plot. The reference lines were calculated using the values in Table 4 and do not correspond to precision statistics.

Table 4. Reduction of area raw data and summary statistics for each laboratory.

Lab ID	**Test Results (%)**	$\bar{x}$ (%)	s (%)	h
H	47.4, 47.8, 41.0, 41.0	44.3	3.8	-0.98
I	42.0, 51.0, 62.0	51.7	10.0	0.34
J	47.0, 48.0, 39.0, 48.0	45.5	4.4	-0.76
K	44.0, 51.0, 55.0, 53.0, 55.0	51.6	4.6	0.33
L	58.7, 59.6	59.2	0.6	1.69
M	45.0, 45.3, 44.8, 50.1, 46.2	46.3	2.2	-0.62

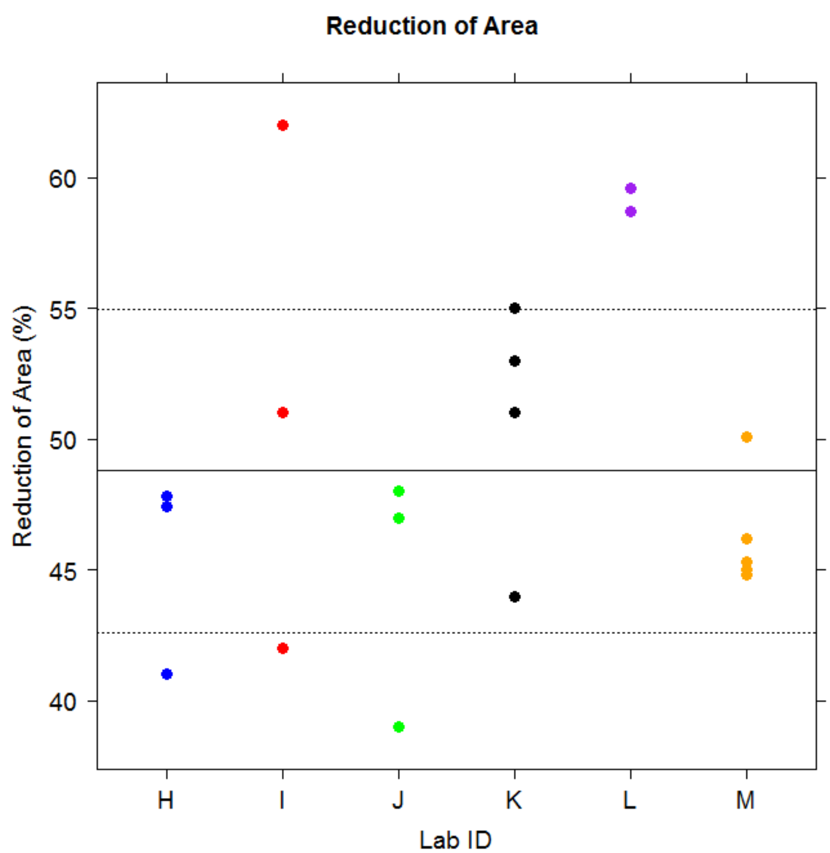

Figure 7. Reduction of area data from Table 4. The solid horizontal line represents the overall mean, and the dashed lines represent ±1s of the measurements.

The h statistics, indicating between-laboratory consistency, are plotted in Figure 8 for each laboratory. The horizontal lines at -1.92 and 1.92 represent the critical value associated with the 0.5 % significance level and six participating laboratories.

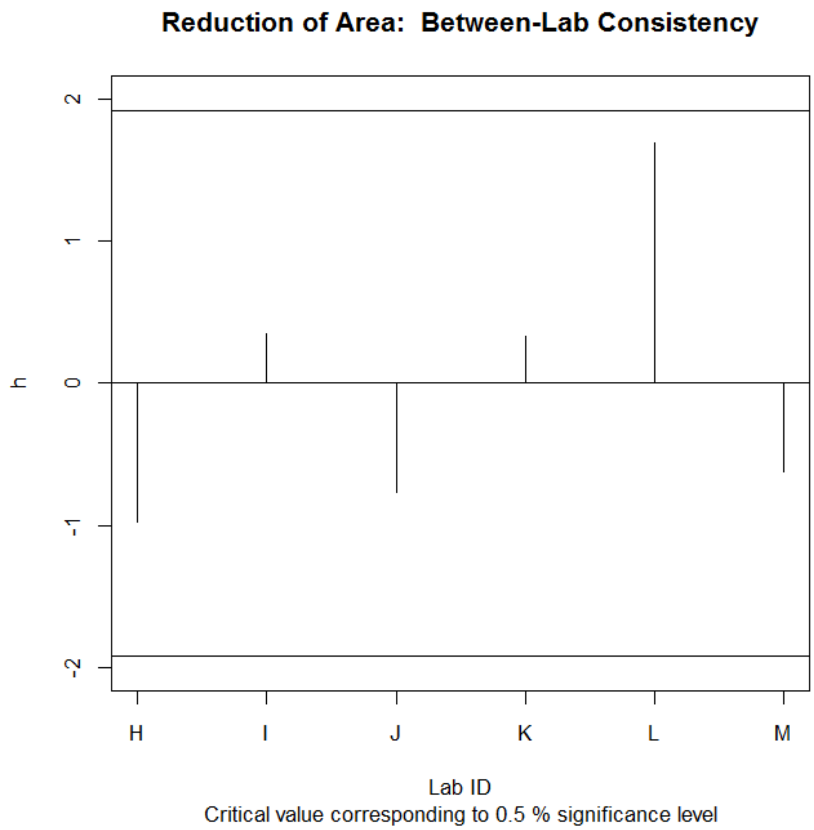

Figure 8. Reduction of area h consistency statistics for material SUS 316LN.

Figure 8 shows strong evidence that the measurements are consistent among laboratories, so the precision statistics can be calculated (Table 5) based on all the reported data.

5. Precision and Bias Statement:

5.1 Precision

An interlaboratory study [1] for the determination of the precision of Test Method E1450 for tensile strength, yield strength, elongation at fracture, and reduction of area, was conducted. Because only one material was used in the interlaboratory study, the precision statement does not necessarily apply to all other materials and the repeatability and reproducibility limits should be considered as general guides.

Precision statistics for the four tensile testing methods are shown in Tables 5.

Table 5. Precision statistics for four tensile testing methods.

	Tensile Strength (MPa)	Yield Strength (MPa)	Elongation at Fracture (%)	Reduction of Area (%)
$\bar{\bar{x}}$	1715	1067	48.6	49.7
s_r	11	11	1.6	4.9
s_R	29	15	2.1	6.6
$r = 2.8 \cdot s_r$	31	31	4.5	13.7
$R = 2.8 \cdot s_R$	81	42	5.8	18.5
$CV\ \%_r$	0.64 %	1.03 %	3.29 %	9.86 %
$CV\ \%_R$	1.69 %	1.41 %	4.32 %	13.28 %

Table 5 contains the following statistics described in ASTM E691-14:
1) the average of the lab averages ($\bar{\bar{x}}$),
2) the repeatability standard deviation (s_r),
3) the reproducibility standard deviation (s_R),
4) the approximate 95 % repeatability limit (r), and
5) the approximate 95 % reproducibility limit (R).

The terms "repeatability limit" and "reproducibility limit" are used as specified in ASTM E177-14. Also included in Table 5 are the coefficient of variation for the repeatability standard deviation ($CV\ \%_r$) and the coefficient of variation for the reproducibility standard deviation ($CV\ \%_R$). The coefficient of variation is 100 times the standard deviation divided by the average.

5.2 Bias

Since there is no accepted reference material, method, or laboratory suitable for determining bias using the procedure in this annex, no statement of bias is being made.

6. References:

[1] Ogata, T., Nagai, K., Ishikawa, K, and Shibata, K., (1992) "VAMAS Second Round Robin Test of Structural Materials at Liquid Helium Temperature," Advances in Cryogenic Engineering, V. 38, pp. 69-76.

[2] Mood, A. M., Graybill, F. A., and Boes, D. C., (1974) *Introduction to the Theory of Statistics*, 3rd Edition, McGraw-Hill, New York, New York.

[3] Montgomery, D. (2012) *Design and Analysis of Experiments*, 8th Edition, John Wiley & Sons, New York, New York.