



Standard Practice for Determination of a Pooled Limit of Quantitation for a Test Method¹

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1. Scope*

1.1 This practice covers the use of standard regression techniques and data from an interlaboratory study to determine a lower quantitative limit for a test method. This determined lower limit represents the numerical limit at or above which the test results are considered to be quantitatively meaningful for commerce or regulatory activities by this practice. It is defined by this standard as the *pooled limit of quantitation (PLOQ) for the test method*.

1.2 This practice is applicable to test methods that are capable of producing numerical test results close to zero. Examples are those test methods that determine quantitatively the concentration of analyte(s) near zero.

2. Referenced Documents

2.1 ASTM Standards:²

D6300 Practice for Determination of Precision and Bias Data for Use in Test Methods for Petroleum Products and Lubricants

E456 Terminology Relating to Quality and Statistics

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

3. Terminology

3.1 Definitions:

3.1.1 *repeatability conditions*, n —conditions under which test results are obtained with the same test method in the same laboratory by the same operator with the same equipment in the shortest practical period of time using test units or test specimens taken at random from a single quantity of material that is as nearly homogeneous as possible.

NOTE 1—By *in the shortest practical period of time* is meant that the

¹ This practice is under the jurisdiction of Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.94 on Coordinating Subcommittee on Quality Assurance and Statistics.

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² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

test results, at least for one material, are obtained in a time period not less than in normal testing and not so long as to permit significant change in test material, equipment, or environment. See Terminology **E456**.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *pooled limit of quantitation (PLOQ)*, n —level of property or concentration for a test method at which the ratio: [10 $\times$ pooled repeatability standard deviation of results for a concentration level/concentration level] = 1.

3.2.1.1 *Discussion*—Quantitative test results obtained using a test method at or below its PLOQ is expected to have an uncertainty of $\pm 30\%$ or greater at the 95 % confidence level.

3.2.2 *pooled repeatability standard deviation*, n —the statistically pooled standard deviation for results obtained under repeatability conditions over multiple operators/laboratories for a sample that is obtained in accordance with Practice **D6300** or other equivalent Interlaboratory Study (ILS) analysis (for example, Practice **E691**, ISO 4259).

3.2.3 *laboratory limit of quantitation (LLOQ)*, n —level of property or concentration for a test method at which the ratio: [10 $\times$ repeatability standard deviation of results for a concentration level determined in a single laboratory/concentration level] = 1.

3.2.3.1 *Discussion*—Quantitative test results obtained by this single laboratory at or below its LLOQ is expected to have an uncertainty of $\pm 30\%$ or greater at the 95 % confidence level.

3.3 Acronyms:

3.3.1 *ILS*, n —interlaboratory study.

3.3.2 *LLOQ*, n —laboratory limit of quantitation.

3.3.3 *PLOQ*, n —pooled limit of quantitation.

4. Summary of Practice

4.1 Statistically pooled repeatability standard deviations from multiple laboratories for at least seven samples meeting the requirements in Section 6 are obtained.

4.2 An empirical power function is fitted to the plot Y versus X where:

Y = [10 $\times$ pooled repeatability standard deviation/mean] for each sample, and
 X = [mean] of each sample.

*A Summary of Changes section appears at the end of this standard