



## Standard Practice for Determination of a Pooled Limit of Quantitation<sup>1</sup>

This standard is issued under the fixed designation D 6259; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last approval. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

### 1. Scope

1.1 This practice covers the determination of a lower quantitative limit for a test method for an analyte. The determined lower limit is hereinafter referred to as the *pooled limit of quantitation*.

1.2 Applicable test methods will produce test results greater than zero. Examples are those test methods that measure sample composition.

### 2. Referenced Documents

#### 2.1 ASTM Standards:

E 456 Terminology Relating to Quality and Statistics<sup>2</sup>

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method<sup>2</sup>

### 3. Terminology

#### 3.1 Definitions:

3.1.1 *pooled limit of quantitation, n*—level of property or concentration of analyte above which quantitative test results can be obtained with a specified degree of confidence. See 3.2.1 for acronym.

3.1.2 *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 (see 10.3 of Practice E 691).

NOTE 1—The *same operator, same equipment* requirement means that for a particular step in the measurement process the same combination of operator and equipment is used for every test result. Thus, one operator may prepare the test specimens, a second measure the dimensions, and a third measure the mass in a test method for measuring density.

NOTE 2—By *in the shortest practical period of time* is meant that the 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 E 456.

#### 3.2 Acronyms: Acronym:

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

### 4. Summary of Practice

4.1 Determine the standard deviation of a test result, under repeatability conditions, at progressively higher levels of the analyte until the ratio of measured level to standard deviation becomes greater than ten and remains so.

### 5. Significance and Use

5.1 In a single laboratory, the limit of quantitation, LOQ, equal to ten standard deviations has been recommended.<sup>3</sup> A test result at this LOQ has an uncertainty of  $\pm 30\%$  at the 99% confidence level. Similarly here, as a general estimate, the PLOQ, equal to ten repeatability standard deviations is recommended. A test result at this PLOQ has an uncertainty of  $\pm 30\%$  at the 99% confidence level.

5.2 Values below the PLOQ are deemed to be too uncertain for meaningful use in commerce, or in regulatory activities.

5.3 Many test methods never find application outside their PLOQ. However, in the quest for ever more sensitive procedures, it can become difficult to distinguish an analytical response from background noise with the technology at hand. Test methods defective in design or poorly executed may also function outside their PLOQ.

### 6. Procedure

6.1 Make the preparations outlined in 6.2, then carry out one of the procedures described in 6.3.

#### 6.2 Preparations:

6.2.1 *Select Test Levels*—Decide the objective of the test method, the range of typical samples it is expected to cover. Name a set of test levels covering this range and spaced to bracket the PLOQ. In some cases, the PLOQ will be well below the useful range. Then, it is only necessary to determine a *less than* value.

NOTE 3—Mean and standard deviation data from experienced laboratories, archived research reports, and known limitations of the test method or equipment can give a preliminary notion of the PLOQ. An LOQ can be estimated in a single laboratory.

6.2.2 *Select Sample Materials*—Normally, use sample materials that are typical of those to which the test method is applied. In special cases, the method of standard additions

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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 14.02.

<sup>3</sup> Keith, L. H., et al, "Principles of Environmental Analysis," *Analytical Chemistry*, American Chemical Society, Vol 55, 1983, p. 227.