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An American National Standard

Standard Practice for Determination of a Pooled Limit of Quantitation¹

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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:²

E456 Terminology Relating to Quality and Statistics

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

2.2 ASTM Adjuncts:

D2PP, Determination of Precision and Bias Data for Use in Test Methods for Petroleum Products³

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 E691.)

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 E456.

3.2 Acronyms:

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.⁴ A test result at this LOQ has an uncertainty of ± 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 ± 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.

¹ This practice is under the jurisdiction of Committee D02 on Petroleum Products and Lubricantsand 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.

³ Withdrawn and no longer available from ASTM International.

⁴ Keith, L. H., et al, "Principles of Environmental Analysis," *Analytical Chemistry*, American Chemical Society, Vol 55, 1983, p. 227.