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ASTM D8272 Guide for developing and optimizing methods at D19 on Water

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ASTM - Voluntary Consensus Standard Development Organization (SDO)

- AKA – VCSB
- Standard Method = A method of known and demonstrated precision issued by an SDO
- Standard Reference Method = A Standard Method with demonstrated accuracy

Reference = John K. Taylor, Quality Assurance of Chemical Measurements, Lewis Publishers, 1987

D8272 is a Guide To Task Groups To Gather All Information Needed During Method Development and Optimization

- “In accordance with the National Technology Transfer and Advancement Act (NTTAA), EPA considers Voluntary Consensus Standards Bodies (VCSB), such as Standard Methods and ASTM in regulatory actions when periodically updating the list of approved methods.”

- The guide provides guidelines to method developers as to information needed to optimize a method

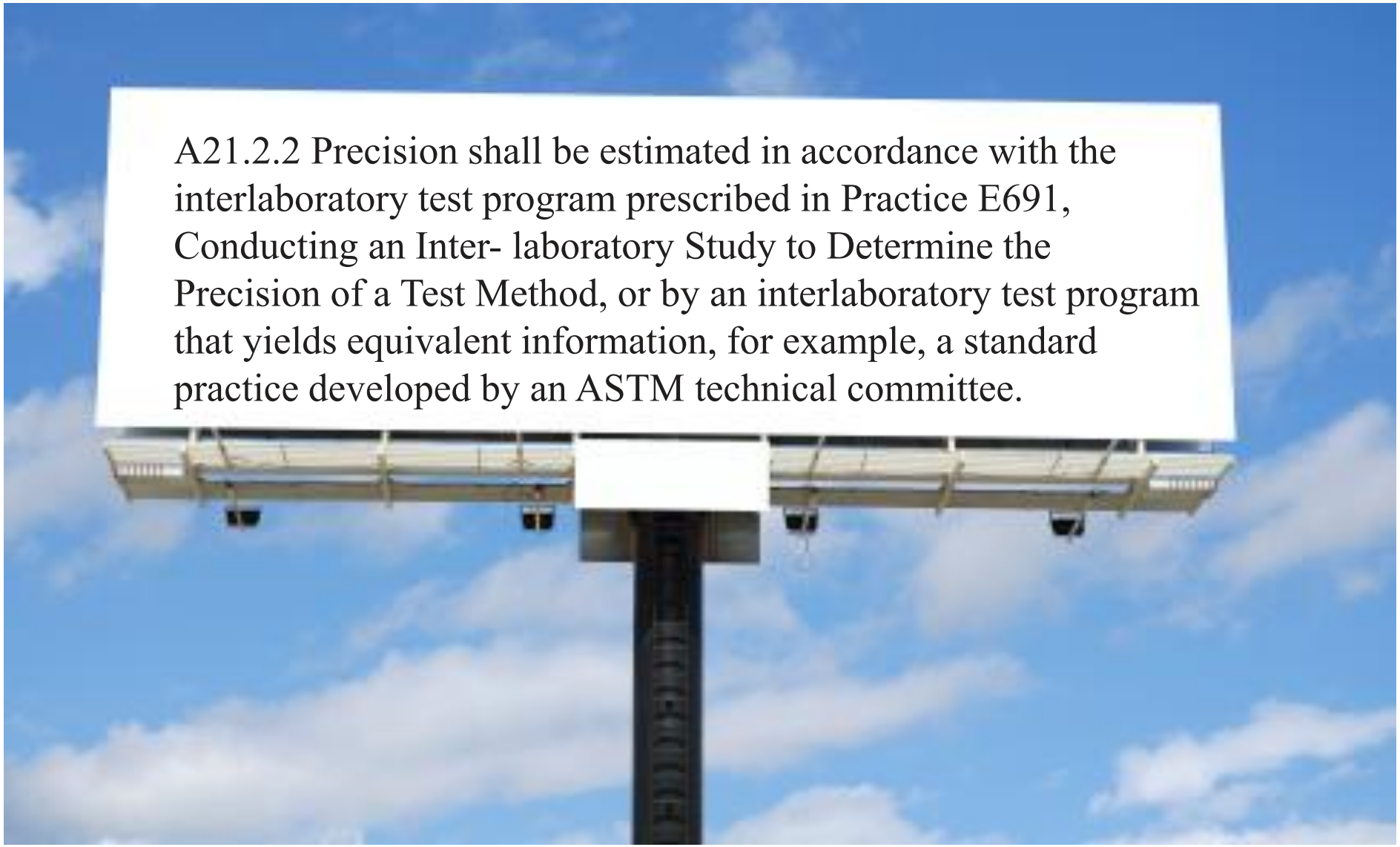
- so that ASTM can easily submit new, and updated methods for use in wastewater, drinking water, and RCRA compliance

Requirements on ASTM task groups

standard, *n*— *as used in ASTM International*, a document that has been developed and established within the consensus principles of the Society and that meets the approval requirements of ASTM procedures and regulations.

test method, *n*— a definitive procedure that produces a test result.

ASTM Test Methods must have a precision statement according to E691 or similar



ASTM D19, follows Practice D2777

1.1 This practice establishes uniform standards for estimating and expressing the precision and bias of applicable test methods for Committee D19 on Water.

Statements of precision and bias in test methods are required by the Form and Style for ASTM Standards, “Section A21. Precision and Bias (Mandatory).”

EPA 624 Method Study

The data obtained from the interlaboratory study were analyzed employing a series of computer programs known as the Interlaboratory Method Validation Study (IMVS) system which was designed to implement ASTM procedure D2777.

17. KEY WORDS AND DOCUMENT ANALYSIS		
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EPA Form 2220-1 (Rev. 4-77) PREVIOUS EDITION IS OBSOLETE

An extensive single laboratory study establishes repeatability and other factors

D2777 states

6.1 Considerable pilot work on a test method must precede the determination of its precision and bias. This pilot work should explore such variables as preservation requirements, reaction time, concentration of reagents, interferences, calibration, and sample size. Potentially significant factors must be investigated and controlled in the written test method in advance of the collaborative test.

ASTM D8272 defines the single laboratory study work that must be done prior to determining precision and bias.

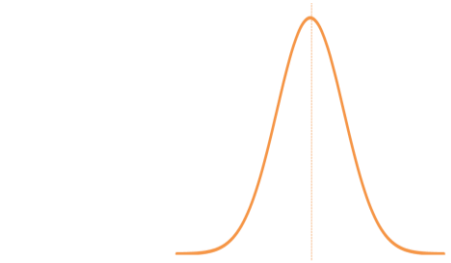
The Single Lab Study Of A New Method Establishes:

- Selectivity → prove new method measures what it says it measures
- Calibration/Standardization technique → required curve fitting or standardization protocols
- Reagent recipes, preparation, containers, shelf life
- Holding time and preservation
- Quantitation limit and range
- Applicable matrices
- Interferences and mitigation
- MDL, ML, and calibration range
- Repeatability on standard solutions
- Bias using reference materials
  - RM if available
  - Or recovery using spiked matrices (if possible)
- Ruggedness

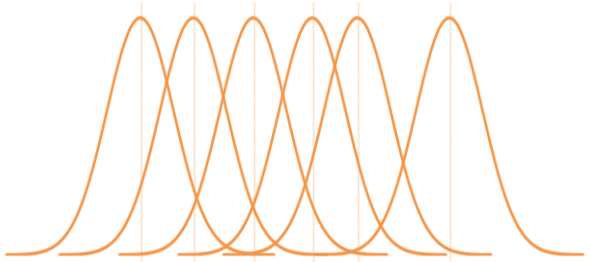
Reproducibility can only be determined by a multiple laboratory study

reproducibility, n—precision under reproducibility conditions.
3.1.11 *reproducibility conditions, n*—conditions where test results are obtained with the same method on identical test items in different laboratories with different operators using different equipment.

What is reproducibility?

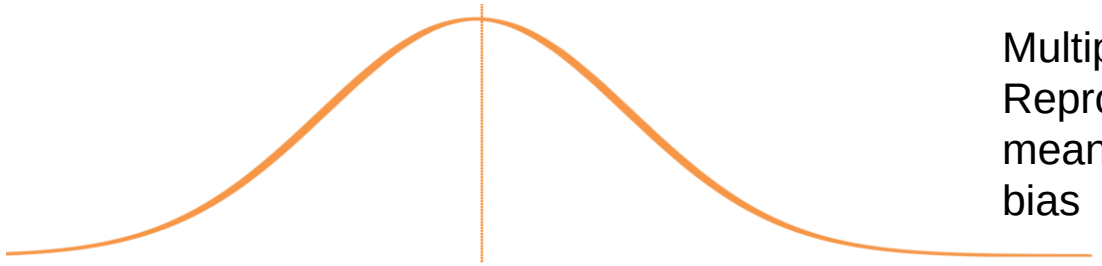


Single lab repeatability (s_x)



Multiple labs same sample = repeatability (s_x), different means

Different means = systematic bias



Multiple labs same sample = Reproducibility (S_x), difference of means from multiple lab mean = bias

Repeatability (single lab)	Reproducibility (multiple lab)
Same method	Same method
Same test material	Same test material
Same apparatus	Different apparatus
Same laboratory	Different laboratories
Same operator	Different operators
Same time	Different times

Reproducibility improved by reducing systematic bias, bias that can only be determined in a multiple lab study

Systematic bias = Subtle changes in method results due to operators, reagents, instruments, techniques.

A single lab cannot evaluate method, or systematic bias. Results MUST be compared to other labs.

A well-defined method, followed without modification, minimizes bias and is reproducible.

- Complete descriptions
- Know what can, and what cannot be modified
- Well defined, tested matrices
- Calibration range and model defined
- Interferences evaluated and compensated
- Reagent preparation and shelf life known and defined
- Sampling and preservation
- Digestion/extraction followed exactly
- Measurements made like in the single lab study
- Calculations defined and followed

Example = various TKN digestions

What steps are so critical that changing them changes the result?

Factor	Nominal	Variation
Portion size	10 ml	25 ml
Evaporation temperature	160 °C	220 °C
Evaporation time	1 hour	30 minutes
Digestion temperature	370 °C	380 °C
Digestion time	15 minutes	1 hour

Questions?

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