

# ASTM International Interlaboratory Detection Estimate (IDE) & Interlaboratory Quantitation Estimate (IQE)

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# Task Group Contact Information

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# ASTM International ([astm.org](http://astm.org))

- 108 years
- 30,000 volunteers
- 130 Technical Committees
- Extensive Tiered Balloting
- Strict Adjudication of Negative Votes

# Committee D19 on Water

- Established in 1932
- Chair: Jack Hall
- 300 Current Standards (two volumes)
  - Subcommittee D19.02 on Quality
    - Chair: John Hubbling
    - Task Group on D and Q
      - Chair: N. Grams



# Universe of D & Q

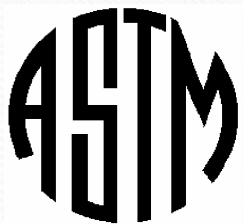
	Detection	Quantification
Among Labs	Interlaboratory Detection (IDE)	Interlaboratory Quantification (IQE)
Within Labs	Intralaboratory Detection (WDE)	Intralaboratory Quantification (WQE)

ASTM.org



Designation: D 6091–97

**99 %/95% Interlaboratory Detection Estimate (IDE) for Analytical Methods with Negligible Calibration Error**



Designation: D 6512–00

**Interlaboratory Quantitation Estimate**

# D/Q Process Overview

## Basic Steps:

- Concentration Design (D2777)
- Optional Outlier Removal (D2777)
- Model Standard Deviation (IDE/IQE)
- Regress (Calibration) Recovery (IDE/IQE)
- Calculate CL/DE (IDE)
- Calculate QE (IQE)



## D2777 – Method Precision & Bias

- First approved in 1969
- Committee recognized importance of validating methods
- Multi-concentration/Multi-lab
- Inter-laboratory SD (not pooled SD)
- Used to establish P&B statements



# Optional Outlier Removal

- Grubbs tests as per D2777 (lab and point)
- IDE & IQE discuss outlier removal
  - Recommend graphing data points
  - Visual examination as well as Grubbs
- Identified potential outliers should be examined
- Removal based on real-world reason

# Model Standard Deviation

(vs. Concentration)

- Use simplest model that fits the data
  - Constant
  - Straight Line Standard Deviation
  - Exponential
  - Hybrid (aka Rock & Lorinzato or General Analytical Model)
- For use in Lst. Sq. vs. Weighted Lst. Sq.
- For use in setting tolerances (DE)
- Used for calculation of IQE (RSD%)



# Conc. Recovery Regression

(True vs. Measured)

- Linear relationship only model
- Least Squares or Weighted Least Squares
- Used to convert measured results to true concentrations (how much in the real world)
- Establishes the bias relationship
- Often termed 'calibration relationship'



# Detection Estimate

- Based on Dr. Currie's theory (IUPAC)
  - Critical Level & Detection Level
- Tolerance Interval
  - 90% Coverage, 95% confidence
- CL: Upper tolerance at zero conc.
  - False Positives (99% control)
- DL: Upper tolerance at CL
  - False Negatives (95% control)

# Statistical Intervals Discussion

- Confidence interval

Used to quantify the uncertainty in a population parameter at a specified confidence level

“With a specified (e.g., 95%) confidence, the interval covers the true [mean] of the population”

# Statistical Intervals

- Prediction interval

Used to quantify the uncertainty in a single future measurement

“With a specified (e.g., 95%) confidence, the next measurement will be within the calculated interval”



# Statistical Intervals

- Tolerance interval

Used to quantify the uncertainty in a certain portion of future measurements

“With a specified confidence (e.g., 95%), a specified portion (e.g., 90%), of future individual measurements will fall within the calculated interval”

# Quantification Estimate

- Known and Controlled Precision (%RSD)
  - Related to significant digits (D. Coleman)
- Can specify %RSD DQO (10%, 20%, etc.)
- Bias addressed as in DE
- Required to be above DE
- No tolerance intervals, no statistical control



# Summary

- IDE and IQE are the wisdom of the majority from a solid consensus process, a diverse and venerable Committee and dedicated and animated Task Group
- Procedures are detailed and technical as well as a consensus balance of practical vs. scientifically correct.
- WDE and WQE in process
- Software makes calculations simple



# Summary

- Using weighted least squares – complex or necessary?
  - Found that very few analytes have constant SD
- Multiple concentrations – complex or necessary?
  - Measurement uncertainty
- Modeling standard deviation – complex or necessary?
  - Constant is not a good assumption
- Dealing with bias (real world) – complex or necessary
  - Measurement uncertainty?

# DQCALC Software:

- Based on Excel
- User friendly and user manual included
- On ASTM.org for \$75
- Put data in simple Excel file
- Open DQCALC and follow prompts
- Must make some decisions
- Output file is saved



# DQCALC Software Use

- Open, opt in or out of outlier removal
  - Set outlier criteria
- Get input file, enter name, comments
- Create output file
- Click to perform initial calculations
- Evaluate Diagnostic/Descriptive Graphics
- Select Model for Standard Deviation
- Software automatically updates calcs based on user's model choice
- Save and exit( or start another calculation)